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Data of Geochemistry

Sixth Edition

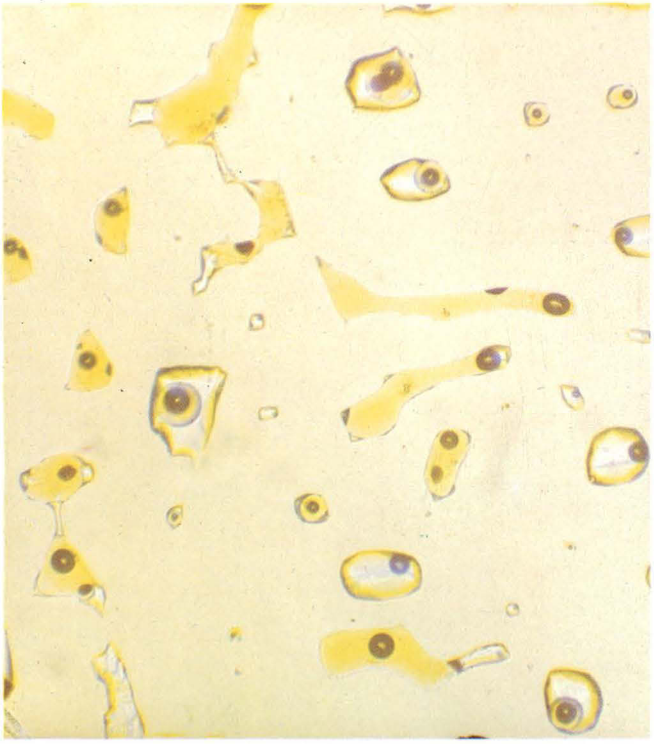
Chapter JJ. Composition of Fluid Inclusions

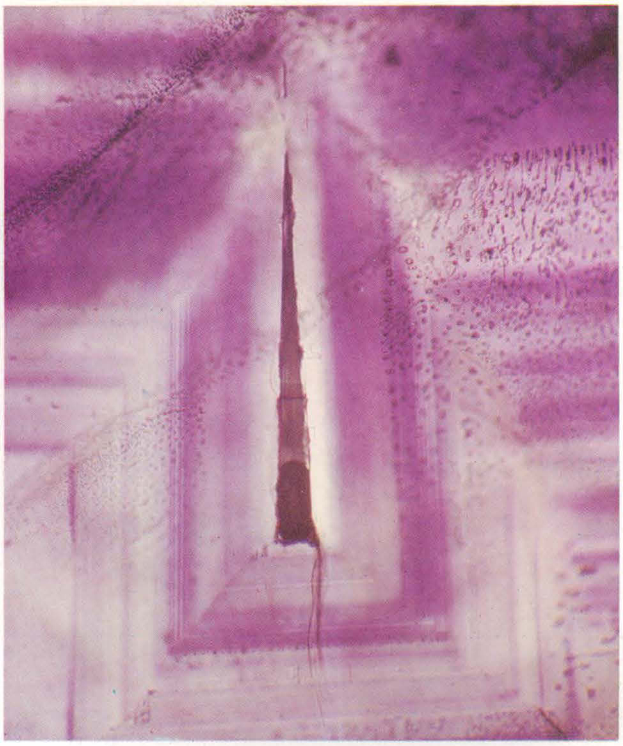
GEOLOGICAL SURVEY PROFESSIONAL PAPER 440-JJ

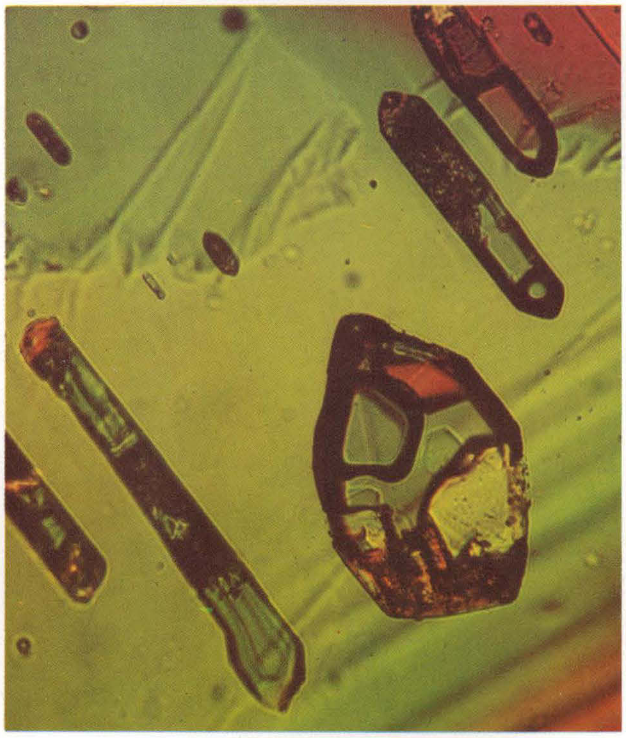
U.S. DEPARTMENT OF THE INTERIOR
March 3, 1849

Roedder—COMPOSITION OF FLUID INCLUSIONS—Geological Survey Professional Paper 440-JJ

COMPOSITION OF
FLUID INCLUSIONS







Relations of inclusions to host mineral.

UPPER LEFT: Photomicrograph of a plane of pseudosecondary inclusions on a {111} cleavage fracture in a pale-colored fluorite crystal from a vug, which trapped an immiscible mixture of liquid oil (yellow) and brine (colorless). Recrystallization of the fluorite walls of the fracture to isolate individual inclusions occurred almost entirely via the brine. As a result the inclusions of brine have become three dimensional and are lined with negative crystal facets, whereas the yellow oil inclusions are still flat and are almost two dimensional. In addition, the index of refraction of the oil is much closer to that of fluorite. Small irregular masses of dark-brown birefringent matter have formed in the oil phase since trapping, and the round gas bubbles (probably methane, under pressure) characteristically occur in the oil rather than in the brine, where both phases are present. Area of photograph is 1×1.2 mm. Plain transmitted light. Sample ER 59–3, Hill mine, Cave-in-Rock, southern Illinois fluorite-zinc district.

UPPER RIGHT: Photomicrograph of a large primary inclusion in zoned purple fluorite crystal from a vug. The crystal grew from bottom to top as a series of subparallel cubic projections, two of which were slightly inclined to each other. These two finally met, trapping a wedge-shaped mass of fluid (a strong brine) as a fluid inclusion. A gas bubble (dark) formed in the thickest part of the inclusion due to shrinkage caused by subsequent cooling. Although not apparent in this two dimensional view, the bubble comprises only about five percent of the volume of the inclusion. Some of the colored growth bands are outlined by tiny primary inclusions. A healed cleavage fracture is revealed by the plane of pseudosecondary inclusions in focus at right, which extend to what was the edge of the crystal at the time of fracturing. Two highly inclined planes of secondary inclusions cut across the crystal from lower left to upper right; one of these intersects the large primary inclusion and probably caused replacement of the fluid in it with new fluid. Area of photograph 2.7×3.4 mm. Plain transmitted light. Sample ER 59–3, Hill mine, Cave-in-Rock, southern Illinois fluorite-zinc district.

LOWER LEFT: Photomicrograph of a multiply-zoned sphalerite crystal, showing the relation of pseudosecondary inclusions to crystal zoning. The red and yellow color bands are not growth zones. The color bands reflect differences in composition between adjacent twin bands, presumably due to preferential uptake of minor constituents such as iron on specific growing crystal faces. The crystal grew from left to right, and successive growth stages are revealed by the nearly vertical hazy gray zones of minute primary inclusions. Fracturing occurred at some time after the growth of this part of the crystal. Healing of a cleavage fracture that was parallel to the plane of this section resulted in the trapping of large numbers of thin, flat, presumably pseudosecondary inclusions, each of which now has a gas bubble (13 volume percent) and several different tiny daughter minerals. One of these is apparently quartz; in the large central inclusion it can be seen as a small prism, holding the bubble away from the wall (lower right edge of bubble). Recrystallization of sphalerite around these inclusions has been controlled in part by the compositional (and twin) zones to yield straight segments in the inclusion borders, and, in many areas in this sample, to yield preferential occurrence along certain compositional zones. Area of photograph is 5.5×7 mm; photograph taken in plain transmitted light. USNM specimen R577, Cananea, Mexico.

LOWER RIGHT: Photomicrograph of a cleavage fragment of topaz in transmitted light between crossed polarizers, showing large amounts of daughter mineral (unidentified) in presumably pseudosecondary inclusions. Most of the daughter minerals are birefringent, although some are so fine grained that their birefringence is not visible here. A few are visibly isotropic, particularly in the large inclusion, where they abut the vapor bubble, making it angular. The different amounts and kinds of daughter minerals in the several inclusions, and the different volume percentages of vapor phase, indicate that at least some of these inclusions were probably interconnected in the past, and recrystallization (necking down) has resulted in isolating material of diverse composition in different inclusions. Occasionally thin tubes can be seen still connecting such inclusions. (See pl. 10, fig. 4). Area of photograph 0.8×1.1 mm. USNM 96595, Rukuba tin mine, Nigeria.

Data of Geochemistry

Sixth Edition

MICHAEL FLEISCHER, *Technical Editor*

Chapter JJ. Composition of Fluid Inclusions

By EDWIN ROEDDER

GEOLOGICAL SURVEY PROFESSIONAL PAPER 440-JJ

Review of world literature on analysis of fluid
inclusions, applicability and limitations of the
methods used, published and unpublished data
obtained, and their geological significance

U.S. DEPARTMENT OF THE INTERIOR
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DATA OF GEOCHEMISTRY, SIXTH EDITION

Michael Fleischer, *Technical Editor*

The first edition of the Data of Geochemistry, by F. W. Clarke, was published in 1908 as U.S. Geological Survey Bulletin 330. Latter editions, also by Clarke, were published in 1911, 1916, 1920, and 1924 as Bulletins 491, 616, 695, and 770. This, the sixth edition, has been written by several scientists in the Geological Survey and in other institutions in the United States and abroad, each preparing a chapter on his special field. The current edition is being published in individual chapters, titles of which are listed below. Chapters already published are indicated by boldface.

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C. Internal structure and composition of the earth
D. **Composition of the earth's crust,** by Raymond L. Parker
E. Chemistry of the atmosphere
F. **Chemical composition of subsurface waters,** by Donald E. White, John D. Hem, and G. A. Waring
G. **Chemical composition of rivers and lakes,** by Daniel A. Livingstone
H. Chemistry of the oceans
I. Geochemistry of the biosphere
J. Chemistry of rock-forming minerals
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L. **Phase-equilibrium relations of the common rock-forming oxides except water,** by George W. Morey
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N. Chemistry of igneous rocks
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DATA OF GEOCHEMISTRY

COMPOSITION OF FLUID INCLUSIONS

By Edwin Roedder

ABSTRACT

Fluid inclusions in minerals and rocks preserve for us tiny samples of the fluids that have been present during, and that have in part caused, a number of geological processes, including ore deposition. Although the original material trapped in them was generally a homogeneous fluid at the time of trapping, most inclusions have undergone changes on cooling to surface temperatures, resulting in the formation of new phases such as a gas bubble, immiscible liquids, or daughter minerals. This report is mainly a compilation of the world literature on the methods and results of study of their composition, but it also includes various unpublished data by the author.

Most inclusions are extremely small and may average less than 10 micrometers in diameter. A single inclusion of this size contains a total of only 10^{-10} gram of material; hence, it cannot be analyzed by ordinary chemical techniques. But a surprising amount of useful *qualitative* and *semiquantitative* compositional data on the nature of solid, liquid, and gaseous phases present can be obtained by a series of relatively simple, nondestructive microscope procedures. These procedures, and their limitations, are discussed in detail and are illustrated with a series of photomicrographs. Additional methods that yield qualitative or semiquantitative data, but require destruction of the samples, are also described.

These various procedures have shown that although inclusions of organic liquids and gases are common in some localities, most fluid inclusions consist of a liquid water solution. At room temperature, the gas bubble usually contains only water vapor at 0.03 atmosphere pressure, but it may consist of CO_2 at pressures up to 70 atmospheres. Where a second, immiscible liquid phase is present, it is generally found to be liquid CO_2. The daughter minerals that form in some inclusions after trapping have been studied intensively, as they are compounds that were sufficiently concentrated in the original fluid that they were precipitated when saturation was reached on cooling. Small, isotropic cubes are by far the most common daughter minerals, as they are found in samples from many environments; they have been identified as halite by several procedures. Many other daughter minerals have been recognized, including particularly sylvite, calcite, and hematite, a number of rare carbonates, fluorides, and fluoborates, and many as yet unidentified species. Large amounts of such daughter minerals are found in inclusions from certain types of pegmatites, from porphyry copper deposits, and from several other environments.

Many igneous rocks and meteorites also contain fluid inclusions, but the "fluid" now consists of silicate crystals or glass, representing a former silicate melt. Such melt inclusions are not covered in this report, although their study involves many of the same techniques.

Quantitative analyses of single, large (~1 millimeter) inclusions have been made by semimicro-, micro-, or ultramicrochemical methods. More commonly, the fluid from a large number of smaller inclusions is extracted by crushing and leaching to provide enough material for analysis by more conventional methods. More than 2,400 partial to relatively complete quantitative analyses of fluid inclusions have been reported in the literature and are summarized here in tables. Many of these are for gaseous constituents only. The significance of most of these data is seriously limited by problems of sample selection and extraction procedure. In addition, analytical procedures adequate to provide quantitative data on the very small samples of fluid usually obtained require considerable care to avoid major contamination, and loss, from a variety of sources. Some of the procedures reported (for example, for pH) cannot fail to give grossly erroneous results except under very limited conditions. There is no known panacea for these problems, and as a result of the many variables involved, it is unlikely that a standardized analytical procedure, suitable for most samples, will be developed. Many of the newer analytical tools have been or soon will be applied to inclusion analysis and considerable progress can be expected, but the nature of the samples and extraction procedures used are the prime considerations in evaluating the precision, the accuracy, and most important, the significance of any measurements obtained.

Representative analyses are given from a number of published papers, and some unpublished data, along with a discussion of their limitations and geological significance. The different types of geologic environment show grossly different compositional ranges.

The many analyses that have been made of the "organic" gases and liquids present as inclusions in the minerals of some igneous rocks show appreciable amounts of a variety of compounds of high molecular weight, in addition to major amounts of H_2, CO, CH_4 and C_2H_6, but the question of inorganic versus organic origin is still open.

Aqueous inclusions have been analyzed by a variety of methods. The concentration of salts in the inclusion fluid as trapped is generally less than 10 percent by weight, but may range from more than 50 to practically 0 percent. The salts consist of major amounts of Na^{+1}, K^{+1}, Ca^{+2}, Mg^{+2}, Cl^{-1}, and SO_4^{-2}, with lesser amounts of Li^{+1}, Al^{+3}, BO_3^{-3}, PO_4^{-3}, $HSiO_3^{-1}$, HCO_3^{-1}, CO_3^{-2}, and many other ions. Many individual ions in this list may predominate, although Na^{+1} and Cl^{-1} are generally the most abundant. Free carbon dioxide, both as liquid and gas, is not uncommon and may be dominant.

The references include those papers concerned directly with various aspects of the composition of inclusions. As many of the methods for inclusion geothermometry are inextricably connected with considerations of inclusion composition, those papers that present geothermometric data also are included and are annotated briefly in a table.

INTRODUCTION AND GENERAL BACKGROUND

GENERAL NATURE AND FREQUENCY OF OCCURRENCE OF FLUID INCLUSIONS

The tiny droplets of aqueous solution, carbon dioxide, oil, or other fluids that occur in most minerals are called fluid inclusions. They are small and frequently remain unnoticed, but as is shown on plates 1 through 12, they may be found in samples from a wide variety of geological environments. The cavities they occupy in the minerals may be flat, roughly spherical, irregular, or faceted "negative crystals." In most natural samples, the shape of the inclusions is the result of recrystallization processes and generally differs greatly from the original shape at the time of trapping.

HOW INCLUSIONS ARE TRAPPED AND THE ENVIRONMENT AT THE TIME OF TRAPPING

Basically, inclusions are samples of fluids that were present when the enclosing minerals grew or recrystallized, usually from these same fluids. Important but relatively rare exceptions are the gas bubbles that occasionally are trapped in minerals growing from gas-saturated or boiling liquids (pl. 9, fig. 4; Ermakov and Kalyuzhnyi, 1957; Kliya and Lemmlein, 1961) and the globules of immiscible fluids, such as oil (frontispiece, upper left; pl. 4, fig. 5), or CO_2 (pl. 4, fig. 6), that are sometimes trapped along with the water phase (Roedder, 1963b). Still another and probably rarer exception would be the formation of inclusions by coalescence of materials formerly distributed through the solid on an atomic or molecular scale. Thus Dolgov (1959) believes that some fluid inclusions in quartz form from the fluids present during later recrystallization of silica gels, and others assume that similar processes have occurred during the formation of "colloform" sphalerite. But Roedder (1968b) has shown that these "colloform" textures are not colloidal in origin. The differences are important in any discussion of the pertinence of fluid inclusion composition to problems of the chemistry of ore fluids.

Mollwo (1941) formed gas inclusions in synthetic crystals of impure KBr by a solid-state reaction near the melting point of the crystal, and Griggs and others (1966; and D. T. Griggs, written commun., 1966) made H_2O inclusions up to a maximum diameter of $0.2 \mu m$ (micrometer) by annealing an experimentally deformed synthetic quartz crystal containing much hydrogen. One obvious parallel is the formation of air inclusions in crystals of ice formed by the compaction of snow (Langway, 1958; Scholander and Nutt, 1960).

Fluid inclusions may be trapped by: (1) Crystal growth irregularities or fluid inhomogeneities during the formation of the enclosing mineral; these are called *primary* inclusions (pl. 1, figs. 1–6; pl. 5, fig. 1; pl. 9, figs. 2, 3; frontispiece, upper right) (Zerfoss and Slawson, 1956; Brooks and others, 1968; Shlichta, 1968), by (2) healing fractures formed in the crystal during its growth—*pseudosecondary* inclusions (frontispiece, all four photomicrographs) (Ermakov, 1949a), or by (3) healing of fractures formed at some subsequent time—*secondary* inclusions (pl. 4, figs. 1–3; pl. 10, figs. 1, 2) (Lemmlein, 1929). Fluid inclusions are found in the minerals of most rocks and ore deposits, and hence constitute evidence of the almost universal presence of a fluid phase during the geological events yielding these materials.

Many papers on inclusions state various criteria that can be used to distinguish between primary, pseudosecondary, and secondary origins, and a discussion of this subject is beyond the scope of this report. However, it should be noted that two of the most commonly used criteria, negative crystal shape, and occurrence in euhedral crystals, projecting into vugs, are generally invalid (Roedder, 1967a, 1968a). (Also see frontispiece).

Care must be used in the interpretation of the composition of inclusions, as they do not always represent the composition of the fluid from which the enclosing mineral actually formed. This is particularly true when minerals form from a fluid containing droplets of another immiscible fluid. Such fluid inhomogeneities may cause the trapping of inclusions of the immiscible droplets in large numbers, with very little or none of the other fluid from which the crystal grew (pl. 4, fig. 5; pl. 9, figs. 2, 4). Examples include (1) vapor bubbles, formed in hydrothermal fluids by sudden release of pressure (Barabanov, 1958a, 1965, 1967; Roedder and Coombs, 1967; Laz'ko and others, 1968); (2) carbon dioxide in hydrothermal fluids (pl. 5, fig. 1; Roedder, 1963b, p. 189); (3) saline brine in granitic melts (pl. 4, figs. 7–9; Roedder and Coombs, 1967); and (4) liquid oil in saline brine (pl. 4, fig. 5; pl. 9, fig. 2; frontispiece, upper left; Roedder, 1963b, 1965a).

Minerals from certain metamorphic rocks, particularly those recrystallized in a water-deficient environment (Yoder, 1955), are generally free of fluid inclusions, as are those from most meteorites and many, but not all, lavas.[1] Thus fluid inclusions are

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[1] Those minerals that have formed from a silicate-rich fluid (magma), as in meteorites, lavas, lunar samples, and many terrestrial intrusive igneous rocks, normally contain "fluid inclusions" consisting of the supercooled silicate fluid, now either a glass or partially crystallized (devitrified) glass. These usually contain also a vapor bubble, formed by differential shrinkage, and hence may be mistaken for ordinary aqueous fluid inclusions. Except for problems of identification, such silicate inclusions are not discussed in this report.

generally not found in materials believed to have formed deep in the earth, such as diamonds and the minerals of the eclogites, with the notable exception of the CO_2 inclusions in the minerals in the olivine nodules brought to the surface with many alkalic basalts (Roedder, 1965d) and in certain meteorites (Yasinskaya, 1967). Bauer and Spencer (1904, p. 119) and Schlossmacher (1932, p. 348–351) refer to fluid inclusions in diamond, containing liquid water, liquid CO_2, and (or) gas. These and similar statements have been widely quoted ever since, but unfortunately, the criteria used for phase identification are never stated. Sutton (1928, p. 42) calls these statements "a favourite myth." The present author has examined many inclusions in diamond, including the sample described and illustrated by Eppler (1961, figs. 19 and 20), and has not seen any recognizable liquid, CO_2 or otherwise.

SIZE, VOLUME, AND NUMBER OF INCLUSIONS

Inclusions are seldom larger than 1 mm (millimeter) (see frontispiece, upper right), although museum specimens with single inclusions containing 10 or even 100 ml (milliliters) of fluid are known (Hidden, 1882; Prikazchikov and others, 1964; Prikazchikov, 1959). The number in any given sample is usually related inversely to inclusion size, for very small inclusions are much more abundant. Inclusions in the size range 1 μm to 10 μm outnumber all inclusions of greater than 10 μm size by a factor of 10 or even 100. Zirkel (1873) reports a hauynite with an estimated 3.6×10^{11} inclusions per cubic centimeter. Electron microscopy of fractured mineral surfaces has shown the existence of large numbers of very small inclusions on mineral grain boundaries. Thus Sella and Deicha (1962a, 1962b, 1963) and Deicha and Sella (1963) show inclusions as small as 0.02 μm, trapped on intergranular surfaces; they number more than 10^8 per square centimeter of surface. Although very numerous, these inclusions are of such small volume individually that they would amount to only about 0.01 volume percent of the rock. Presumably there is no break in the series between these inclusions ($\sim$ 200 A in diameter) and single dislocations ($\sim$ 2 A).[2] Much white quartz contains 10^9 inclusions per cubic centimeter, but as these inclusions average about 1 μm in size, the total fluid content is only about 0.1 weight percent (fig. 1). The white appearance is due to multiple reflections at the inclusion interfaces, and hence the coefficient of transmission of light can be used as a measure of the inclusion content (Ushakovskii, 1966). "Turbid" feldspars also have large numbers of fluid inclusions (Folk, 1955).

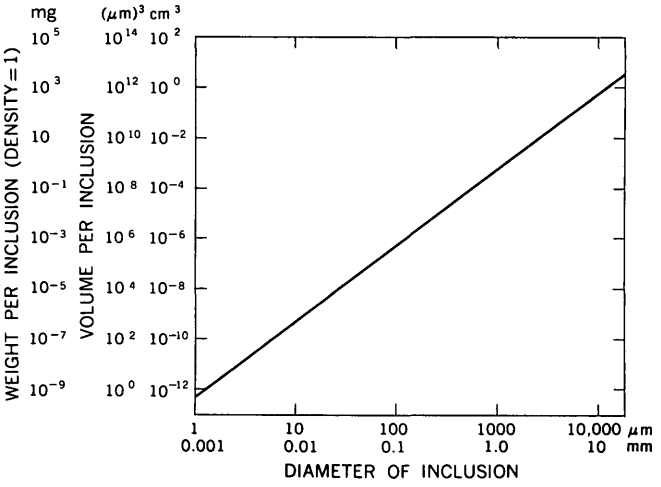

FIGURE 1.—Volume and weight of spherical fluid inclusions, assuming a fluid of density 1.0 g/cm³. (Modified from Roedder, 1958.)

Some samples contain far more than 0.1 percent inclusion fluid, and the volatile constituents present in these inclusions, such as water and CO_2, may cause major ambiguities and errors in analytical determinations of these constituents in rocks and minerals (Faber, 1941; Ermakov and Myaz, 1957). Fluid inclusions may also contribute as much as 100 ppm (parts per million) of each of several nonvolatile constituents to the total analysis of even "pure" mineral separates and may cause serious errors in the determination of the density of minerals (Piznyur, 1957; Ermakov and Myaz, 1957). Thus Hawes (1881) described a quartz with a high concentration of inclusions that has a density of 2.625 g/cm³ (grams per cubic centimeter) (fig. 2), and Langway (1958) used the density of ice to determine the volume of the numerous gas inclusions present. The violent decrepitation of many minerals when heated is due, in large part, to the explosion of the fluid inclusions.[3]

HISTORICAL DEVELOPMENT AND SIGNIFICANCE OF INCLUSION STUDY

Other than brief mention in some of the ancient literature from St. Augustine's time (see Correns, 1954), the first specific description of inclusions is by Abu Reykhan al-Biruni, a central-Asian scholar

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[2] McLaren and Phakey (1965b) report observation of approximately 10^{14} voids or "bubbles" per cubic centimeter of white quartz, using transmission electron microscopy. The size ranged from 200 to 1000 A. At 500 A the quartz would have about 1 percent voids, the walls of which are presumably wet with liquid water (Prof. A. C. McLaren, written commun., 1967).

[3] The internal pressures are occasionally adequate to cause explosion without heating. Thus, a quartz crystal decrepitated spontaneously and broke the glass in its museum display case (Prof. R. Parker, Zurich, oral commun., 1965); similar decrepitation on a much larger scale has occurred in salt mines. (See p. JJ43.)

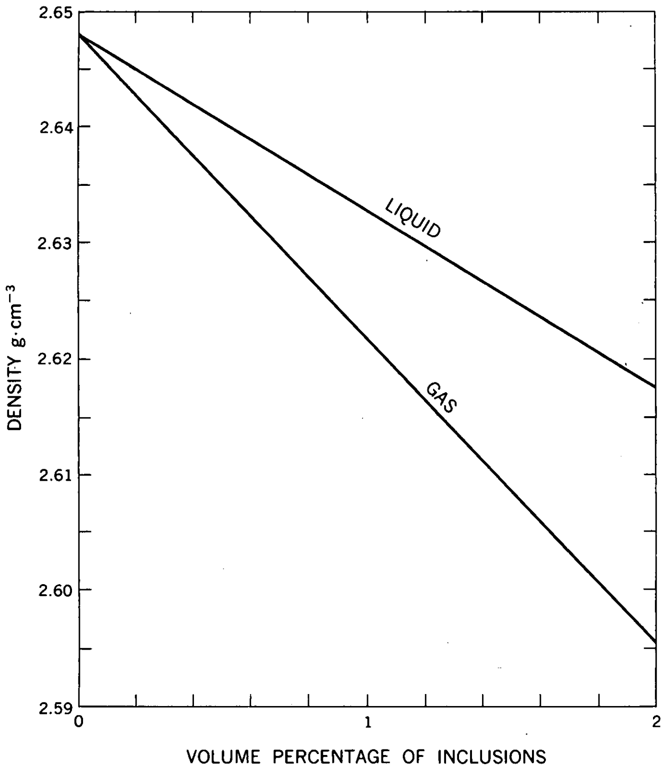

FIGURE 2.—Calculated variation in density of quartz from presence of fluid-filled inclusions containing all liquid solution (density, 1.1 g/cm^3) on all gas (density, 0.0 g/cm^3). Density of pure quartz assumed to be 2.6484 g/cm^3. (From Roedder, 1958.)

of the 11th century (Lemmlein, 1950b). Robert Boyle's description of a large moving bubble in quartz (Boyle, 1672) is apparently the first reference in English. The early naturalists showed considerable attention and interest in these large inclusions (Dewey, 1818; Dwight, 1820; and others). Although there are several earlier reports on the nature of the fluids trapped (Dolomieu, 1792; and others), the first actual "analytical" work to establish the composition of specific inclusions was that done by Breislak (1818), Davy (1822), Brewster (1823a), and Nichol (1828); such work was used as very strong evidence to support the Neptunist theory of the formation of minerals and rocks from water. In the following 140 years a large number of investigations of fluid inclusions were made, for a variety of reasons. Many were aimed at using or disproving a thesis proposed by Sorby (1858) that the gas bubbles present in the fluid of most inclusions—but not all (see pl. 11, fig. 6)—were the result of differential shrinkage of the liquid and the enclosing mineral during cooling from the higher temperature of trapping to the temperature of observation.[4] Sorby showed that the coefficient of expansion on heating (and, conversely, the coefficient of contraction on cooling) for a variety of liquid solutions resembling the fluids in inclusions was one or two orders of magnitude greater than the coefficients for the enclosing or "host" mineral. Hence, he reasoned that the temperature of trapping can be estimated by heating the sample to the point that the bubbles disappear. This use of fluid inclusions is discussed in more detail in chapter II of "Data of Geochemistry," which deals with geologic thermometry. (Also see pl. 8, fig. 1.) Although most of the early work is descriptive only, there are occasional qualitative analytical data (for example, Zirkel, 1870b), and a few quantitative analyses were made without the benefit of modern techniques and apparatus. In spite of these limitations, the work of such men as H. C. Sorby and Ferdinand Zirkel is surprising; many of their conclusions about the significance of inclusions have stood the test of time, and many of their observations are still valid and useful 100 years later.

Inclusions were a subject of intense study and debate by many geologists during Sorby's time, and some of the conclusions from their study were so disturbing to certain schools of geologic thought that many efforts were made to discredit them. Phillips (1875) pointed out that there are large variations in the composition and nature of different inclusions in the same sample and that Sorby had obviously oversimplified the problem. Even today, some believe that proof of the secondary origin of many inclusions, and of the possibility of later addition or loss (leakage), essentially or totally negates their significance. Thus Perrin and Roubault (1952) dismiss them as being secondary features of no consequence, and many others are reluctant to accept inclusion data in the mistaken belief that widespread or universal leakage of fluid inclusions is a proven fact. The writer believes that leakage of fluid inclusions in nature has occurred, but is comparatively rare (for example, see frontispiece, upper right); documentation is given in a separate publication (Roedder and Skinner, 1968). Actually these very ambiguities of origin and subsequent environment may cause the data to have greater significance, provided that the

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[4] Frequently the gas or vapor bubbles in the liquid (which vary from high pressure gas to nearly a vacuum) are termed "libellae" in the older literature, and whole inclusions, regardless of phases present, are termed "vacuoles" or "lacunae;" but because of changing usage, none of these terms is free of ambiguity. Probably the term "bubble" is most appropriate for the gas or vapor phase in a surrounding liquid, but unfortunately some geologists apply this term incorrectly to the whole inclusion of liquid and gas or vapor bubble.

techniques used are adequate to resolve the ambiguities. Error can arise when the conclusions drawn are broader than the available facts and limitations justify, and unfortunately there have been several such papers published, both pro and con, that have caused considerable misunderstanding. Regardless of their origin and history, inclusions do represent actual samples—the only samples we have—of former fluids existing at some time in the history of the earth. As such they are important clues in understanding the geological modus operandi.

Many studies of fluid inclusions involve their use as geological thermometers, in part as an aid in the search for hidden ore bodies (Ermakov, 1966b; 1968b, and other papers in the same volume). As such geothermometric studies are inextricably connected with the problems of the composition of the fluids, and as compositional data on the included fluids are required by many of the methods used in this work (Ermakov and Laz'ko, 1949; Vul'chin and Ermakov, 1965), and for all attempts at correcting the geothermometric data for pressure (Kennedy, 1950a; Lemmlein and Klevtsov, 1956a), such studies are listed in table 1. Many methods have also been proposed for using inclusions to estimate or at least place limits on the pressure at the time of formation, all of which require compositional data. These methods are based on the vapor pressure of the solution (Roedder, 1967a); on the enclosure of fluids assumed to be *totally immiscible* under the conditions of trapping (Johnsen, 1920; Kalyuzhnyi and Koltun, 1953; Kalyuzhnyi, 1955a, Rutherford, 1963; Ypma, 1963; Roedder, 1963b, p. 176–177); or on the enclosure of fluids assumed to be *partially miscible* (that is, exhibiting some mutual solubility) under the conditions of trapping (Smith and Little, 1959). Even the trapping of an originally homogeneous fluid mixture will allow some limits to be set on the range of P-T conditions, if the phase diagram is known, as in system H_2O-CO_2-NaCl (Takenouchi and Kennedy, 1965b).

There have been several attempts to use the types and nature of inclusions—both liquid and solid—as "fingerprints" to identify the provenance of quartz grains in sandstones (Ermakov, 1950a; Keller and Littlefield, 1950; Dolgov, 1954). The author believes that such studies, although difficult, may eventually be very useful in unravelling a number of problems in statigraphy and sedimentation.

Fluid inclusions are especially useful as clues to the nature of the ore-forming fluid. Far too commonly the ore-forming fluid for a given deposit is simply assumed to have been rich in those constituents that were deposited—rich even in proportion to their abundance in the deposit! Noble (1950, p. 244), writing about the Homestake gold deposit, is one of the relatively few who do not make this assumption. But what is known concerning the " * * * very large amounts of volatile constituents and soluble salts [which] must have passed through the formation without leaving any record"? Fortunately his statement is generally not true; fluid inclusions can usually be found (pl. 5, figs. 3–5) and can provide a record of these fluids, albeit complex, fragmentary, and minuscule.

BRIEF SUMMARY OF INCLUSION COMPOSITION

As this chapter deals mainly with the methods of study used and the data obtained on the composition of fluid inclusions, a preliminary summary of their composition may be useful. By far the most abundant type of inclusion consists of a liquid of low viscosity and a gas or vapor bubble having a volume generally less than that of the liquid. The ratio of liquid volume to total volume is sometimes termed the *"degree of fill."* The liquid is generally a water solution with less than 10 weight percent total salts, but concentrations range from more than 50 to practically 0 percent. The salts consist of major amounts of Na^{+1}, K^{+1}, Ca^{+2}, Mg^{+2}, Cl^{-1}, and SO_4^{-2}, with lesser amounts of Li^{+1}, Al^{+3}, BO_3^{-3}, PO_4^{-3}, $HSiO_3^{-1}$, HCO_3^{-1}, CO_3^{-2}, and many other ions. Many individual ions in this list may predominate, although Na^{+1} and Cl^{-1} are generally the most abundant. Carbon dioxide, both as liquid and gas, is not uncommon and may be dominant. The bubble generally results from shrinkage, because the shrinkage of the homogeneous (one-phase) fluid that was originally trapped is much greater than that of the surrounding mineral when cooling occurs. As such, the bubble may consist only of water vapor at the few millimeters of vapor pressure—practically a vacuum—characteristic of most water solutions at room temperature. In other inclusions it is a highly compressed gas that was originally dissolved in the trapped fluid.

If, upon cooling from the temperature of trapping to surface temperatures, the fluid becomes supersaturated with a soluble salt such as NaCl, or any other mineral, these minerals may crystallize from the fluid as one or more new crystals, called *daughter minerals*.[5] These daughter minerals may, in turn,

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[5] In the rather extensive Russian literature on such crystals, they are described by the term "mineraly-uzniki," which may be translated literally as "captive" or "prisoner" mineral; as this may give an incorrect concept of origin, the author (Roedder, 1963b) has introduced the term "daughter" mineral. This change is made particularly necessary by the fact that another term, "zakhvachennye," meaning "trapped," is sometimes used for solid mineral grains trapped *during* the formation of the inclusion.

have inclusions of liquid plus gas in them that were trapped during their crystallization on cooling (Safronov, 1957b). The writer has observed this rather common occurrence in the large halite daughter crystals in inclusions in Colombian emeralds (pl. 2, fig. 6). In addition to the formation of daughter minerals, there is nearly always some further crystallization of the host (enclosing) mineral from the fluid, but this is merely added to the walls and is usually both insignificant and invisible.

Occasionally during the growth of a crystal, solid crystals of the same or other phases become trapped. It is not uncommon for such trapped solid inclusions to cause simultaneous trapping of the surrounding liquid as a funnel-shaped inclusion pointing in the direction of growth, so that the solid inclusion appears partially surrounded by fluid and resembles a daughter mineral (Eppler, 1963; Gübelin, 1964; Pough, 1965; Flanigen and others, 1967; and Khaimov-Mal'kov, 1959). (See also pl. 1, figs. 1–5.). The difference in origin makes it very important that these two cases be clearly distinguished.

Not uncommonly the originally homogeneous fluid breaks up upon cooling to form two immiscible fluids and a gas bubble. Some authors use the general term "three-phase inclusion," without definition, when referring to such inclusions; others use the same term to mean inclusions containing a crystal, liquid, and vapor. The resulting ambiguity is sometimes unresolvable and always undesirable. Usually when there are two liquids and a gas bubble in a given inclusion, one is liquid water solution, one is liquid carbon dioxide, and the gas bubble is carbon dioxide under pressure (pl. 7, figs. 9, 10). More rarely, the second liquid, immiscible with water, is a liquid hydrocarbon; such inclusions may be formed by trapping of an originally inhomogeneous suspension of hydrocarbon fluid in water (pl. 9, fig. 1; frontispiece, upper left).

Many inclusion papers present classification schemes for inclusions, some of which are rather elaborate (Ermakov, 1950a, 1968a). These classifications are mainly compositional, with categories based on the phase composition at room temperature and further subdivisions based on arbitrary divisions of the absolute amounts of the various phases. Other schemes involve factors such as the behavior on homogenization (also a function of gross composition), cause of trapping, origin (time of trapping), degree of crystallinity, presumed state of aggregation at the time of trapping, and geological environment. Most of the objective schemes are not particularly useful as their categories are of no real significance and are based on arbitrary ranges of features that are difficult to measure. The subjective schemes, particularly the one based on origin, are much more significant and useful, but can rarely be applied systematically and without ambiguity.

LITERATURE SUMMARIES

The literature on fluid inclusions is very large and very scattered. F. Gordon Smith (1953a) presents a comprehensive annotated bibliography of more than 400 papers on the subject of fluid inclusions published prior to 1953, but as a result of a considerable increase in interest in fluid inclusions in recent years, the literature has more than doubled since 1953. As little of the Russian literature is covered by Smith, Lemmlein (1956b) presents a short review of much of the Russian inclusion work as an appendix to a Russian translation of Smith's book; this appendix has been translated into English. Short reviews have been published on inclusion research in general (Deicha, 1964), on studies of fluid inclusions in minerals from India (Dekate, 1962), and on the significance of inclusions in ores (Bartholome, 1966).

Other than Smith's book, there are very few general references on the subject in English. Anyone interested in inclusions should read Sorby's classic article (Sorby, 1858), which shows him to be remarkably perspicacious. There have been a number of review articles published in Russian, including a 49-page summary of work on inclusions in hydrothermal minerals (Gurevich, 1961), and reviews of recent work in the U.S. (Bogoyavlenskaya, 1965), France (Aksyuk, 1965), and Europe and Asia (Bogoyavlenskaya, 1968). Several books in English have short summaries (for example, Frondel, 1962), and the author has published a semipopular account (Roedder, 1962b) and a discussion of fluid inclusions as samples of the ore-forming fluid (Roedder, 1967a). Gübelin (1953) published a 220-page book dealing with the identification of gemstones on the basis of the appearance of their inclusions, both solid and fluid. Deicha (1955) published a 126-page book on inclusions, in French, with over 100 references. A 169-page book in Ukrainian on inclusion study (Kalyuzhnyi, 1960) emphasizes the methods of identification and study of the phases present, particularly as used by Kalyuzhnyi and his coworkers. Lesnyak (1964) published a 219-page review (in Russian) of inclusion studies, covering 150 Russian and 42 non-Russian references. Kostyleva (1964) published a 99-page book in Russian, dealing mainly with decrepitation of quartz samples

(66 references). D. N. Khitarov (1965a) published an extensive review, in Russian, of fluid inclusion composition studies (189 pages, 336 references). Two 1967 volumes summarize the very extensive Russian work on organic compounds (in the broad sense) in fluid inclusions in igneous rocks and ore deposits. These are a 121-page summary of the organic compounds in inclusions in the Khibiny alkalic massif (Ikorskii, 1967a), and a 147-page collection of 12 articles by various authors (Dolenko, 1967). At the time of the present writing none of these books was available in English translation.

The first book on the subject was by Ermakov (1950a). (Sometimes this name is transliterated and indexed as "Yermakov.") This 460-page Russian book has been translated into English; it summarizes in particular the extensive Russian work in the field up to 1950 and presents a classification of fluid inclusions. (See also Ermakov, 1965c, 1968a). Since its publication, a series publication, the "Transactions of the All-Union Research Institute of Piezo-optical Mineral Raw Materials" (Trudy VNIIP, Vsesoyuznyy Nauchno-Issledovatel'skiy Institut P'ezoopticheskogo Mineral'nogo Syr'ya), has been established with Ermakov as chief editor. Of this series volume I, part 2 (177 p., Moscow, 1957), consists of 19 papers on inclusions, and volume II, part 2 (134 p., Moscow, 1958) contains 37 papers on inclusions; English translations of these are bound in with the published translation of Ermakov's book. (See headnote to "References".) Additional parts and at least five more volumes of this series are known to have been published, but at the time of writing these volumes were not available to the author.

A symposium dealing mainly with inclusion studies was held in Moscow on May 17–24, 1963, where more than 60 papers were presented. A brief summary of this meeting is given by Ivanova and Zakharchenko (1963) (see also Anonymous, 1963), and a 328-page volume containing 42 papers relating to the symposium was issued under the title "Mineralogical Thermometry and Barometry" (Smirnov and others, 1965). It is not available in English at the time of writing. One of the papers in this volume gives a review of more than 400 analyses of inclusions taken from the literature (Khodakovskiy, 1965). A 264-page volume containing 38 additional papers stemming from this conference was issued in 1966 by the "Nedra" Press of Moscow (Ermakov, 1966a) as volume 9 of the "Transactions of the All-Union Research Institute for the Synthesis of Mineral Raw Materials."

A second symposium on geothermobarometry (mainly by inclusions) was held September 20–25, 1965 in Novosibirsk. The program for this meeting lists titles and authors of 102 papers.[6] The papers from this symposium were issued in two volumes also entitled "Mineralogical Thermometry and Barometry." Volume 1 (Ermakov, 1968c, 368 p.) presents 47 papers dealing with general physical and chemical problems of ore formation and the use of inclusion studies as an exploration tool. Volume 2 (Ermakov, 1968d, 320 p.) presents 53 papers dealing with the thermodynamic regime of ore formation, detailed studies of individual minerals from specific stages of mineralization, and methods of inclusion study. These papers are included in the tables of this report and are mentioned briefly in the text where appropriate, but they have not been translated into English and hence are not discussed in detail.

A third and even larger symposium on geothermobarometry was held September 9–15, 1968, in the Moscow area. A 280-page volume has been issued by the Ministry of Geology of the U.S.S.R. giving abstracts of the 161 papers presented. These papers are not included in this review. A short summary of this symposium is given by Balitskii (1969).

English titles for all and English abstracts for some of the papers presented at both the second and third Soviet symposia on inclusions are presented in the 1968 issue of "Fluid Inclusion Research—Proceedings of COFFI." This yearly abstract volume is issued under the aegis of COFFI, the Commission on Ore-Forming Fluids in Inclusions of the International Association on the Genesis of Ore Deposits. It is privately printed in Washington and available from the author.

In view of the need for a general review in English of all inclusion studies, this report attempts to cover the available literature on the subject of the composition of the fluids and the wide range of methods used for their determination. Particular attention is given to the shortcomings, sometimes rather serious, in the applicability of the methods used and the significance of the data obtained. The following sections on nondestructive and destructive methods contain many examples of data, mainly qualitative or semiquantitative, obtained by the variety of procedures described. The next section, on analytical data, deals mainly with quantitative re-

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[6] A short review of this conference (Naumov and Khodakovskii, 1966) states that 78 papers were presented. Dolgov, Ermakov, and Laz'ko (1965) present a summary of the topics discussed, and a review of the papers on inclusions presented at the 22d International Geological Congress in New Delhi in 1964. Dolgov (1966) presents a 5-page summary of the papers given.

sults and generally does not duplicate the data from the previous sections.

More than one-half of the total world literature on fluid inclusions has been published since the start of 1960. More than two-thirds of these items were from Russian sources. This tremendous increase in publications has made it necessary to establish a cutoff in the literature covered in this report. Although most of the papers published in 1968 are included, as well as some that were published in 1969 and 1970 (mainly the author's own works) the literature search is reasonably complete only up to 1967.[7] Certainly many items have been missed, particularly the gem literature and those larger geologic studies in which the inclusion work is only incidental and is not evident in the title or abstract. The author would welcome information about missing items.

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NONDESTRUCTIVE METHODS OF DETERMINING COMPOSITION

There is no panacea for the problems of determining the composition of fluid inclusions. Except for the very large inclusions, whose analyses are relatively easy, no known method or combination of methods will give an accurate, unambiguous analysis of any given inclusion in any given mineral. Many different techniques have been used in specific cases, but none is universally applicable. Due to the extremely wide range in sample material and in the accuracy of the methods used, the available published analyses of fluid inclusions must be examined with considerable care. The major aim of this report is to describe the methods used, with particular emphasis on their accuracy and applicability, so that the data obtained may be correctly interpreted.

The vagaries of inclusion occurrence make it desirable to obtain as much information as possible from those few inclusions that happen to fulfill more than one of the specifications of an ideal sample, such as optically clear and measurable, large size, and known origin. Not infrequently, good evidence has been presented that the fluids from which a given crystal grew changed very markedly during the growth. As a result, it is almost impossible to obtain truly *duplicate* samples even of primary inclusions, and the widespread occurrence of secondary inclusions provides additional ambiguity. Thus it is important that all possible nondestructive tests be applied to any irreplaceable inclusion before a destructive test is used, even though they yield only qualitative or semiquantitative data. In many cases, qualitative or semiquantitative data are the only type that can be obtained.

As the size of the inclusion to be analyzed decreases, the error and ambiguity in the individual determination increase rather rapidly, simultaneously with an equally rapid decrease in the number of determinable constituents. This is a direct result of the fact that the volume (and weight) of an inclusion are functions of the cube of its radius. (See fig. 1.) It might appear from this that only the large inclusions should be analyzed. Unfortunately, large fluid inclusions (greater than a few millimeters) are relatively rare, and very large fluid inclusions (>1 cm), permitting the use of ordinary analytical methods, are known to occur in only a relatively few localities in the world. If a given type of geological process is to be studied in several occurrences, or if

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[7] As indicated in table 1, reports on solid inclusions and on glass inclusions, with or without daughter minerals, and simple descriptions of the occurrence of liquid, gas, or liquid plus gas inclusions without further identification of composition or thermometric studies have been excluded.

the complexities in a given geological occurrence are to be unraveled by a study of the fluid inclusions present, it is necessary to find them in many samples and parts of samples. Even inclusions 1 mm in diameter are sufficiently uncommon that they cannot be used in most such studies. In addition, a number of studies indicate that the probability of leakage into or out of a given inclusion increases rapidly with size (e.g., see Roedder, 1963b, p. 186). Resolution of the problems of analysis of the composition of fluid inclusions must be based on a compromise between the tremendous abundance of the smaller ones and the ease and accuracy of analysis of the larger ones. The simple expedient of taking a sample containing a large number of small inclusions, to get the same amount of fluid, can be used in those cases where most of the inclusions are presumably of one generation (e.g. Roedder, 1958), but such procedures can introduce many additional problems such as contamination (discussed in "Quantitative Methods"), causing gross errors in some published analyses.

Obviously, methods of analysis are needed that are applicable to individual very small inclusions, and the petrographic microscope, with various accessories, provides a number of them. Most methods are based on optical identification of the phases present, and they use the rather surprising variety of properties that can be measured with the ordinary petrographic microscope. The methods are only qualitative or semiquantitative at best, but they are all nondestructive and generally very quick and easy to use. Vogelsang (1869) pointed out that the identification of the phases present in inclusions can be ambiguous and that the simple explanation is not necessarily the right one. This is still true. There is some ambiguity in most optical identifications, particularly if only one parameter is used, but additional independent methods are generally available. Most significantly, these optical methods are exceedingly sensitive, as they work quite adequately on *single* inclusions weighing less than 10^{-10}g (that is, under a 10–μm diameter). Although these methods are generally monitored by continuous visual observation, standard microcinematography has been found useful in certain situations (Deicha and Taugourdeau, 1952; Roedder, 1965f; Rasumny, 1965), and time-lapse photography, in others (Lemmlein and Kliya, 1952b; Loskutov, 1959b). Mel'nikov and Fratkin (1968) have used time-lapse photography with considerable success.

Although there are several methods of handling irregular fragments (Feklichev, 1965), singly- or doubly-polished plates are by far the most suitable samples. The most appropriate thickness will vary with the opacity of the material, the size of the inclusions in it, and the working distance of the microscope objective to the used, but thickness seldom exceeds the range 0.5 to 5 mm. Preliminary searching can be done by viewing through crystal or cleavage faces or by viewing through ordinary crushed grain mounts in a matching index oil. Low magnification study of larger rough but transparent samples or sawn slabs can be aided by sticking a cover glass on the top surface with matching index liquid or by completely immersing the sample in a flat bottomed glass container of liquid of an appropriate index of refraction on the microscope stage. Trouble from internal total reflection at the inclusion walls can be in part avoided by special adjustable lighting (Roedder, 1962a) although it is frequently easier merely to search for better inclusions. Special care is needed for effective microscopy of high index minerals such as sphalerite (Roedder and others, 1963). Inclined illumination from above or below the stage with a flexible fiber optics illuminator has proven invaluable in the author's laboratory.

Analytical procedures for inclusions in opaque minerals must generally be applied blindly, as the abundance and nature of the inclusions are unknown. Although single inclusions in the millimeter range could be located by X-ray procedures, even in a slab of galena 1 cm thick, the density contrast would not be adequate to differentiate between a full and an empty inclusion, particularly where the third dimension is an unknown variable. Richard L. Sutton, Jr., M.D., (1964; oral commun., 1965), has shown, however, that the actual water level in large (3–5 cm) "fluid inclusions" (water-filled agate geodes, also called water agates or enhydros) may be photographed readily, through the opaque walls, by correct X-ray exposure.

Several additional nondestructive methods of study do not involve microscopy. Nuclear magnetic resonance procedures (Allègre, 1961; Paré and Ducros, 1964) and sensitive differential thermal analysis (see page JJ16) may be used to detect relatively small amounts of water. The large neutron cross section for hydrogen permits the location of even small water inclusions as shadows on neutron radiographs; their position can even be determined in the third dimension through the use of stereo pairs (J. R. Dooley, Jr., and J. R. Shoptaugh, oral commun., 1969). Neutron activation followed by gamma counting may be used on small samples with unopened inclusions. This method has not been tried, but it has some possibilities for certain elements, particularly when present in large concentrations and in large inclusions. Such requirements are necessary to minimize the effect of "contamination" from the same element in the host mineral. It is difficult to obtain an unopened inclusion sample with more than 1 percent fluid by weight, yet contamination from the host mineral in such an optimum sample would be as large as the amount of a given element being sought in the fluid inclusions, even if the concentration in the fluid were 100 times larger than in the host.

COMPOSITION OF LIQUID PHASES

FLUIDITY

Even the rough estimates of viscosity that are possible from observations of bubble movement can be useful to differentiate highly viscous oils from low viscosity water solutions. One common source of trouble in the identification of "liquid" is the differentiation of *glass* from *aqueous liquid.* Sorby 1858), Zirkel (1866), and Ermakov (1950a, p. 80 in translation) list several criteria as follows (somewhat modified):

1. Since the index of refraction of glass is closer to that of the host mineral, the borders of glass inclusions are not as dark from total reflection as those of water inclusions; conversely the relief of the bubble in a glass inclusion is greater than in a water inclusion.
2. Glass inclusions frequently have more than one gas bubble, whereas water inclusions very rarely do and then only because of extremely irregular shape.
3. A bubble in a glass inclusion does not move with gravity and often is not round. Bubbles in water inclusions, if free of the walls, will move like the bubble in a level, and if they are very small (<2 μm), they may be in constant "Brownian" movement.
4. A bubble in a glass inclusion changes only imperceptibly on moderate heating (600°C); generally much higher temperatures are needed for appreciable effects (Deicha, 1955), but Dekate (1963) mentions bubble movement at 590°C.
5. Glass frequently is somewhat colored but water solutions in inclusions are almost always colorless.
6. Glass inclusions maintain their bubbles when truncated in slide preparation, or even when intersected by a crack.

Very tiny inclusions are particularly difficult to identify. Ikorskii (1962) distinguished between minute gas and glass inclusions by putting oils on the surface of the slide; opened gas inclusions were filled with oil and the walls became transparent rather than dark.

The writer does not agree with an additional criterion based on inclusion shape proposed by Ermakov (1950a). He would, however, like to add the following criteria:

7. Glass frequently shows some degree of natural devitrification, recognizable as crystallinity or as a "granular" appearance under well-collimated illumination.
8. The bubble in aqueous inclusions frequently will move in the inclusion when a thermal gradient is impressed on it (as by touching a warm or cool needle to the section).
9. When held at high temperatures, glass inclusions may nucleate new crystalline phases that grow at rates in the range of micrometers per hour (Barrabé and Deicha, 1956).
10. Even when the bubble in water inclusions is held immobile by the walls, small solid particles in the liquid may move with gravity (Brewster, 1823b), in a thermal gradient, or if magnetic, with a changing magnetic field.
11. When cooled, water inclusions eventually freeze. (See page JJ18.)
12. Large glass inclusions may show cracks in the glass.
13. Glass inclusions may have very high temperatures for complete homogenization. Although many aqueous inclusions homogenize at temperatures over 500°C (see table 7), homogenization temperatures over 1,000°C are more likely for glass inclusions. Bazarova and Dmitriyev (1967) report homogenization temperatures as high as 1,670°C for inclusions in leucite.

Sorby (1858), and Zirkel (1868, 1870a, 1870b, 1873, 1876) have described a number of the microscopic features of glass inclusions in some detail, which are not repeated here.

COLOR

With one observed exception, the water solutions in inclusions are not colored. Feklichev (1962) reports that in some very large inclusions in beryl, the liquid phase is seen to have a "notable greenish-blue color." The same samples contained greenish solid matter in some of the larger inclusions. Care must be used in determining the color of very small inclusions at very high magnifications (× 1,000 to × 1,500), as a variety of optical effects yield spurious colors. Many of the various hydrocarbon fluids found in some inclusions are yellow or brown (frontispiece, upper left; pl. 4, fig. 5; pl. 9, figs. 2 and 3). This color may be used to differentiate oil from water in a number of samples and is usually assumed to be a positive test. Unfortunately, however, colorless organic liquids also occur in inclusions (pl. 5, figs. 6 and 7; Murray, 1957; Roedder, 1963b, p. 201–203).

WETTING CHARACTERISTICS

When several different fluids, such as glass, water solution, liquid CO_2, oil, or gas, occur in the same inclusion, they usualy take positions of minimum surface energy. When glass occurs, it is usually in contact with the walls, that is, it wets them, and any other fluids such as "vapor" (essentially a vacuum bubble), or liquid water or CO_2, occur as bubbles within the glass (Roedder, 1965d; Roedder and Coombs, 1967). Ermakov (1950a, p. 80 in translation) has noted that the bubbles in glass inclusions may appear "pressed against the inclusion wall." This is indeed common, and in some glass inclusions dozens of tiny gas bubbles will be seen at the walls. This probably results from a nucleation phenomenon, rather than representing the stable, lowest energy position, as gas bubbles can be seen to nucleate at the inclusion walls in some liquid water inclusions and then move away from the walls into the liquid where they coalesce. Zirkel (1873, p. 69) has described glass inclusions in which the bubble appears to be embedded in the wall of the surrounding crystal. Presumably these result from later recrystallization that changes the shape of the inclusion after nucleation of the bubble. The gas bubbles in glass inclusions which contain daughter minerals frequently are attached to these crystals, and occasionally small daughter minerals in aqueous inclusions will adhere to the liquid-gas interface (Deicha, 1952b).

Where water solution exists together with an immiscible CO_2 fluid (liquid or gaseous), the globule of CO_2 always occurs within the water solution, which, in turn, wets the walls and fills all minor reentrants in the cavity. Hartley (1876c, p. 249) noted that when no water is present, the liquid CO_2 may actually fail to wet the cavity walls. He does not state what criteria he used to verify that no water was present. If a large volume of liquid CO_2 is present it may press against the walls, but freezing data (Roedder, 1963b, p. 192) show that a film of water is usually present. When a gas bubble occurs with liquid water and liquid CO_2, the bubble is always completely surrounded by the CO_2 liquid, an observation first made by Brewster (1826a), many years before the true nature of the liquid CO_2 became known. This simple fact causes considerable difficulty in recognizing the presence of small amounts ($\sim$ 10 volume percent) of liquid CO_2 phase, unless the inclusions are strongly flattened (pl. 5, figs. 2–5; Roedder, 1963b, p. 195). In order to minimize these difficulties in observation of thin films, it is frequently useful to have the sample mounted in a microscope universal stage, although careful centering is required (Prof. W. C. Kelley, Univ. of Michigan, oral commun., 1967). In a very few examples, the interfacial tensions between three different fluids are such that the minimum energy configuration is that of partial engulfing (Torza and Mason, 1969). This is illustrated by the curved plane of contact between H_2S liquid and H_2S gas shown on plate 9, figures 7, 8, and 9.

The same relationship is shown by vapor bubbles in inclusions containing immiscible oil and water. In these, the vapor bubble nearly always occurs *in* the oil phase, which is generally free from the walls of the inclusion if an aqueous phase is present to wet the walls. (See pl. 9, fig. 3, for exception.) Some oil droplets in strong brine inclusions in fluorite wet the walls preferentially (pl 9, fig. 1). An extreme example of preferential wetting is shown by certain multiphase oil inclusions in fluorite, in which one of the liquid organic phases wets only certain spots on the walls of the roughly spherical inclusions, corresponding to specific crystallographic planes in the host fluorite structure (pl. 9, fig. 2; Roedder, 1962b, p. 40).

INFRARED AND ULTRAVIOLET ABSORPTION; FLUORESCENCE

A very few inclusion fluids (organic liquids) fluoresce under ultraviolet illumination, and if the enclosing mineral is transparent to the ultraviolet, this simple test is very effective. It was first used by Reese (1898) to identify petroleum inclusions in quartz. (See also Ikorskii, 1964, 1966b, 1967a, 1967b, 1968.) The test must not be considered definitive, as many organic liquids do not fluoresce. Thus Kühn (1968) has found that not all bitumen inclusions in halite fluoresce. (Also see Wetzel, 1959.)

Many of the phases and constituents present in fluid inclusions show very strong, sharp, and characteristic absorption spectra, particularly in the infrared. For example, the five forms in which carbon dioxide may occur in inclusions—liquid CO_2, gaseous CO_2, and in solution as H_2CO_3, HCO_3^{-1}, and CO_3^{-2}—might be identified or even analyzed nondestructively by means of their absorption spectra, obtained by passing an infrared beam through a polished plate of the mineral. In addition, some of the common minerals in which these inclusions occur, quartz, fluorite, and halite, are transparent to large segments of the infrared spectrum. Except in special samples, however (Cifrulak and Cohen, 1969), this method appears to be unsatisfactory. The major problems are the inherently nonuniform distribution of "sample," that is, inclusion fluid, and the general nature of absorption spectra. Many of the sharp absorption lines occur superimposed on a broad absorption band, and to recognize them, the sample density (path length) must be controlled. Unless the inclusions are very small and uniformly distributed, it is impossible to control the sample density and obtain sharp lines. Only a very small percentage of the beam passing through a mineral plate will traverse inclusions. For this part, the effective path length is so long that the broad band absorption for the ubiquitous constituents, water and CO_2, gives complete absorption. The balance of the beam traverses no inclusions. The author has obtained broad adsorption bands, presumably assignable to liquid water, in infrared spectra of quartz and fluorite containing known inclusions, but the data were not really useful. The method has been of greatest use in distinguishing the various possible modes of occurrence of hydrogen (including liquid inclusions) in minerals such as beryl and quartz (Brunner and others, 1961; Wickersheim and Buchanan, 1959, 1965; Boutin and others, 1964; Wood and Nassau, 1968). It revealed the presence of H_2O, CO_2, and hydrocarbon compounds (as included films and as molecules in clathrate-type cages in the structure) in the mineral melanophlogite (Skinner and Appleman, 1963; Appleman, 1965) and was used by McLaren and Phakey (1966) to show the change in bonding of the hydrogen in quartz on annealing, from presumed OH^{-1} ions in the structure, as formed, to H_2O molecules, presumed to be in newly developed very minute gas inclusions, after annealing.

The strong absorption of infrared light by liquid CO_2, causing local heating and homogenization, has proven to be useful as one criterion to identify liquid CO_2 inclusions in olivine from the olivine nodules of basalts (Roedder, 1965d, and pl. 4, fig. 6). As the infrared present in normal microscope illumination is frequently adequate to homogenize such inclusions, it may prevent their recognition unless special efforts are made to obtain illumination free from infrared.

INDEX OF REFRACTION

The index of refraction of fluids in inclusions may be roughly estimated from the apparent relief against the host mineral or each other. Thus glass inclusions ($n{\sim}1.50$) in quartz ($n{=}1.55$) have much narrower borders (much less total reflection) than do inclusions of water solutions ($n{\sim}1.35$); the same is true for water solutions when compared with liquid CO_2 ($n{\sim}1.18$), and for liquid CO_2 when compared with gas inclusions ($n{\sim}1.00$). This criterion is so extremely sensitive to inclusion shape in the third dimension, however, that it must be used with considerable caution. It is not uncommon to find the apparent relief of two inclusions, of different shape and contents, reversed from that which the true indices of the fluids would indicate. Negative crystals are particularly misleading, as their steeply inclined side facets may cause total reflection with only small differences in index. A close match of the two indices gives very useful information about the inclusions (pl. 9, fig. 1), but may make observations of the inclusions difficult, as in the inclusions of water solutions in cryolite ($n{=}1.34$) in which only the vapor bubble may be visible (Prof. Hans Pauly, oral commun., 1965). Similarly, Ermakov (1965b) reports liquid water inclusions from chambered pegmatites of Kazakhstan so concentrated in salts that they matched or exceeded the index of refraction of the enclosing fluorite ($n{=}1.434$). Some similar inclusions from these deposits, provided through the courtesy of Prof. Ermakov, showed exceedingly strong brines when examined on the freezing stage (Roedder, 1963b, p. 180–181), but the index of refraction of the liquid (at room temperature) was less than that of the fluorite in all of them.

The differences in index are usually so large that phase microscopy is not helpful. In some cases, however, interferometry has been used to advantage (Tolansky and Morris, 1947; Lemmlein and Kliya, 1952a; Loskutov, 1959a). Ingerson (1947) shows that dark-field illumination improves the visibility of small bubbles during homogenization experiments. The writer has found that reflected light, preferably from flexible fiber optics, sometimes helps to distinguish between vague fluid-fluid interfaces and miscellaneous irregularities in the cavity walls. Meyer (1950) devised an ingenious method of combining reflected and transmitted light for high temperature microscopy, by using vertical illumination and a metallic reflecting surface below the polished mineral plate.

As the index of refraction of water solutions varies considerably with the salts present (from 1.33 for pure water to about 1.36 for strong brines), quantitative measurements are desirable. These are possible by several methods, the most elegant of which is based on the angle for total reflection at a flat interface on the inclusion wall between the mineral (of known index or indices) and the fluid (of unknown index). This method, using reflected light, was originally described by Brewster (1823a) to determine the index of refraction of a "strange new fluid" he found in inclusions, which was shown to be liquid CO_2 many years later.[8]

Total reflection can be demonstrated, qualitatively, with any flattened two- or three-fluid-phase inclusion and a hand lens. Viewed in transmitted light from a distant, small source, all fluids will transmit light when the inclusion is perpendicular to the light. As the sample is gradually tipped, the gas phase will become black (totally reflect) first, then the liquids, in sequence. In liquid plus gaseous carbon dioxide inclusions it is even possible to watch the total reflection of the gas phase disappear, at constant tilt, as the warmth of the fingers vaporizes liquid CO_2 and increases the density and hence index of refraction of the dense CO_2 gas.

Stegmüller (1952) gives detailed nomograms for obtaining the index of refraction of inclusion fluids using transmitted light. Wahler (1956) improved the technique considerably and made it more generally applicable, mainly by immersing the crystal in a medium of the same index of refraction. With this technique he measured a number of inclusions to second- and third-place accuracy, permitting estimates of salinity of the water phase. He found the procedure useful also to recognize single-phase gas inclusions, which show an index of 1.00; without such tests it is impossible to identify them. There is a possibility that the change in index of refraction of the liquids with temperature might be an effective parameter for their identification; Bokii and others (1961) have described a method for determining the index of refraction of liquids at temperatures as low as −150°C.

Kalyuzhnyi (1954) described a method, also based on total reflection, for obtaining the index of a liquid or solid inclusion, using a Fedorov (that is, universal) stage. He gives graphs for conversion of stage inclination to liquid index for inclusions in quartz, topaz, and calcite. Since then several papers have been published in which this method has been used (Kalyuzhnyi, 1955b; Lisitsyn and Malinko, 1961; Kalyuzhnyi and Shchiritsya, 1962; and Bakumenko, 1964).

The author has found that the index of the fluid in the larger inclusions can also be measured by comparing the real thickness with the apparent thickness of an inclusion obtained by focusing on the top and the bottom of it, using a calibrated fine focus screw (one division=1 μm actual movement). The real thickness can be obtained from similar measurements of the apparent wall thickness above and below the inclusion, subtracted from the total plate thickness, or by viewing from the side (Roedder and others, 1963, p. 355).

The composition of the fluid in the inclusions will, in general, change as daughter crystals form, and the index should change as well. Although differences in index have not been reported for inclusions with and without nucleated daughter minerals, these should exist and might be useful in estimating composition. The data of Kalyuzhnyi (1965b) on the differences in index for *glass* inclusions in various phenocrysts of a hyalodacite might thus be explained by changes due to crystallization of the host mineral on the inclusion walls.

THERMAL EXPANSION AND HOMOGENIZATION

Ever since Brewster (1823a) found a fluid (shown many years later to be liquid CO_2), in inclusions in topaz, that had a thermal expansion 32 times that of water, this parameter has been used to aid in the identification of the fluids (pl. 7, figs. 9, 10; pl. 8, fig. 1). Sorby (1858) was one of the first to determine experimentally, with some accuracy, the high temperature thermal expansions of some salt solutions pertinent to inclusion studies. He used sealed glass capillaries as "synthetic inclusions." In natural materials, the long, straight tubular inclusions are most suitable for this procedure, as they permit relatively accurate phase ratio determinations from the simple linear intercepts of the phase boundaries on a microscope ocular scale. The areas of individual phases in flattened inclusions can also be used in a similar fashion, but less accurately (pl. 3, fig. 2). The relative volumes of vapor bubble and liquid in equant inclusions can be used (fig. 3), but there may be difficulty in measuring inclusion thickness, and gross underestimates of bubble volume can occur due to a curved upper surface of the inclusion acting as a strong negative lens (pl. 11, figs. 7, 8).

Although the coefficient of thermal expansion of any liquid is a definitive property, application of the principle to the identification of the fluids in inclu-

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[8] Brewster measured the index of refraction of two different fluids (1826a, 1826b) which were later given the names "cryptoline" and "Brewstoline" (or crytolinite and Brewsterlinite). From the indices obtained, and other data, it appears that the latter was liquid CO_2; the nature of the former fluid is obscure, but is generally presumed to be water solution (Julien, 1881). Brewster (1862b) thought both were hydrocarbons. It is important to remember, however, that even though liquid CO_2 will dissolve very little water (Stone, 1943), a number of other compounds, particularly organic ones, will dissolve in it (Auerbach, 1931; Francis, 1954) and may be present in natural liquid CO_2, affecting its index of refraction and other properties.

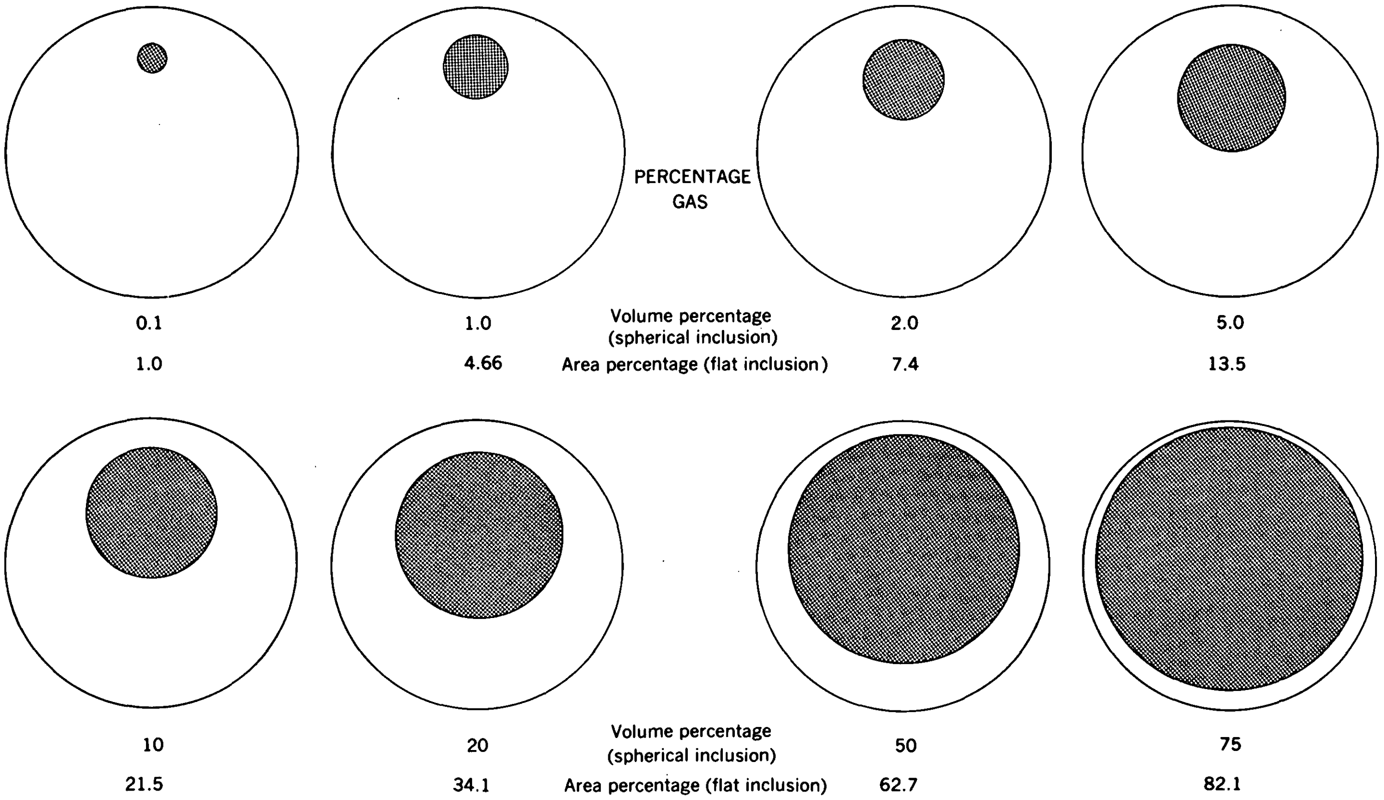

FIGURE 3.—The appearance of inclusions with various volume percentages of gas phase, in which both the gas bubble and the inclusion are assumed to be spherical. The area percentages refer to thin, flat inclusions, where both bubble and inclusion are assumed to be circular disks of negligible thickness, and area percent equals volume percent. Shaded area is gas phase.

sions is not simple.[9] In addition to the problems of measurement of phase ratios mentioned above, the relative phase volumes are involved in determining the apparent expansion. Ostapenko and Khetchikov (1968) have developed a differential equation to calculate these effects. At moderate to high degrees of fill, and low temperatures, where the vapor phase is of low density and volume, the movement of the meniscus toward the vapor with increase in temperature will be a fairly accurate indicator of the thermal expansion of the liquid (pl. 8, fig. 1). At higher temperatures and lower overall density of filling, however, an increasingly significant part of the total mass of the inclusions will be in the vapor phase, and the volume of the liquid phase will *decrease with increasing temperature.* As a result inclusions with certain degrees of fill will, upon heating, first show an expansion and then a contraction and final disappearance of the liquid phase, although the density of the liquid decreases continuously during heating. Ermakov (1950a) has designated this behavior "homogenization with an inversion point" and he shows many graphs of measurements of the degree of fill versus temperature for individual inclusions (that is "homogenization curves"). Kalyuzhnyi (1958c) has calculated the homogenization curves for inclusions of water and of carbon dioxide, and he shows that in water-rich inclusions, inversions occur generally in those with only 25 to 30 percent liquid phase at room temperature.[10] In addition Weis (1953, p. 676) has recorded a range of 200°C in the experimentally determined filling temperatures of two-phase, liquid plus gas inclusions, in a given sample, all with the same apparent degree of fill at room temperature. This might be the result of differences in concentration, but the range indicated

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[9] As examined in detail in chapter II of "Data of Geochemistry," which deals with geologic thermometry, the use of the experimentally determined temperature of homogenization (the filling temperature) of inclusions for geologic thermometry does not require a knowledge of the nature or properties of the fluid. Such data are necessary, however, in estimating filling temperature from the degree of fill at room temperature and in correcting the homogenization temperatures for pressure effects (Klevtsov and Lemmlein, 1959b; Samoylovich and Khetchikov, 1968a, 1968b).

[10] Llambias (1963) has proposed that the temperature of homogenization can be obtained from measurements at several temperatures, extrapolated on the assumption of constant coefficient of expansion, but Ermakov's experimental curves and Kalyuzhnyi's calculated ones both show that the coefficient of expansion is far from constant.

by his freezing data (—3° to —19°C, corresponding to about 5 to 20 weight percent salts) does not appear to be adequate. Unless the geometry is optimum, measurements of percent liquid and gas in an inclusion can be grossly in error (fig. 3).

If data were available on density versus temperature for both liquid and gas phases in the multicomponent systems similar to fluid inclusions, such measurements on inclusions of unknown composition could be used to obtain the composition, or at least limit the possible ranges of composition (Ermakov, 1950a, p. 116 in translation; Klevtsov and Lemmlein, 1959a, 1959b). As shown in this report, however, the composition of fluid inclusions varies widely, and the effects on thermal expansion for two different compositional variables may be equal and opposite. Thus, salts in solution usually reduce the coefficient of expansion, and substances such as CO_2 in solution may increase it. As a result, when measured homogenization curves for inclusions are compared with calculated curves from actual data on known simple systems (Ermakov, 1950b; Cameron and others, 1953; Kalyuzhnyi, 1958c; and work of L. A. Samoylovich, quoted by N. I. Khitarov, 1966), the deviations may be large. Laboratory data on the more complex systems of interest are just now becoming available (Ellis and Golding, 1963; Takenouchi and Kennedy, 1965b; Fabuss and others, 1966; Unterberg, 1966; and Ellis, 1967) and should permit interpretation of many of the phenomenological observations of the past.

The temperatures at which homogenization of fluid inclusions occurs vary widely. If the inclusion has been trapped at near-surface temperatures, and it is small, it may not develop a bubble on cooling to surface temperature (pl. 1, fig. 4; pl. 11, fig. 6). Ermakov (1950a, p. 289 in translation) reports that inclusions trapped in synthetic crystals formed at <70°C seldom have bubbles, but he reports temperatures of filling as low as 38° to 40°C in natural quartz and other minerals. As a result of very large pressure corrections, sufficiently low- or high-pressure gas inclusions and certain organic fluids (see below) may homogenize at subzero temperatures (pl. 5, figs. 6, 7). Carbon dioxide inclusions with an overall degree of filling approximately equal to the critical density homogenize at room temperatures ($\leq +31$°C). If the degree of fill is sufficiently high, "homogenization in the liquid phase" (by expansion of the liquid) may occur at subzero temperatures. Thus, Rutherford (1963) reports that some CO_2 inclusions from the Flin Flon area homogenized on warming to —16°C, and Dolgov, Makagon, and Sobolev (1967) report similar inclusions in kyanite. Conversely, if the degree of fill is sufficiently low (that is, the CO_2 pressure at trapping was relatively low), homogenization in the vapor phase will occur by evaporation of the liquid phase (pl. 5, fig. 1). Roedder (1963b, p. 204) reports this type of homogenization, at <—33°C, for CO_2 inclusions in Colombian emerald; similar behavior, at higher temperatures, is shown by other emerald samples (pl. 5, fig. 2). Inclusions of water solutions may homogenize at any temperature up to a maximum of 374°C for pure water and at much higher temperatures for salt solutions (Sourirajan and Kennedy, 1962).

Although most inclusions homogenize below 500°C, there are many examples in the literature of water-rich inclusions homogenizing at or above 500°C; most of these are listed in table 7. Obviously these require the presence of high concentrations of salts in solution, as pure water inclusions cannot have homogenization temperatures above the critical point of 374°C. One of the first very high homogenization temperatures (>750°C) was reported by Feklichev (1962) for CO_2-bearing water solutions in pegmatitic beryl. As the volume relations of the phases remained nearly unchanged on heating, further verification of these observations would be desirable. Some of the highest recorded homogenization temperatures for water-rich inclusions, in the range 840° to 850°C, were reported for otherwise normal-looking, two-phase, liquid-plus-gas inclusions in nepheline (Kerkis and Kostyuk, 1963; Bazarova, 1965; Bazarova and Feigin, 1966) (See table 7.) At this temperature there was visible evidence of considerable enlargement of the inclusion by solution of the walls, implying an approach of magmatic conditions. This is to be expected, in view of the results of Edgar (1964) who showed that at only 1,000 kg/cm² (kilograms per square centimeter) water pressure, the minimum temperature of melting in the system nepheline-albite-water occurs at 835°C.

Homogenization of inclusions in alkalic rocks at temperatures as high as 890°, and even >950°C has been reported by Panina (1966a). Although these temperatures seem appropriate for homogenization of silicate glass inclusions, her description of critical behavior in inclusions in sodalite and cancrinite (homogenizing at temperatures as high as 630°C) would imply aqueous solutions rather than silicate glass.

The gas bubble (or bubbles) in glass inclusions begins to contract appreciably at about 650°C (Ermakov, 1950a, p. 20 in translation), although this temperature should vary with glass composition. Deicha and his coworkers have reported in detail on the behavior of glass inclusions on heating (Deicha, 1955; Barrabé and Deicha, 1956, 1957; Barrabé, Collomb, and Deicha, 1957, 1959).

If two fluids of considerably different thermal expansion characteristics are trapped in various ratios in simultaneously formed individual inclusions and form vapor bubbles on cooling to lower temperatures, the homogenization temperatures (to achieve the original two-fluid state) will vary with the original phase ratio of the fluids that were trapped. If the two fluids can be identified adequately as to composition, and if the thermal expansions and compressibilities of the two fluids are known, and if the mutual solubilities of the two fluids are either insignificant or are known, it should be possible to obtain *both* pressure and temperature of trapping from such homogenization data. There have been various proposals to use inclusions containing CO_2 and H_2O to determine pressures (Johnsen, 1920; Correns, 1953; Kalyuzhnyi, 1955a, 1965a; Kalyuzhnyi and Voznyak, 1965). Rutherford (1963) used the method to calculate the pressures during deposition of some post-ore quartz at Flin Flon. Similar procedures should be applied to inclusions of oil and water (frontispiece, upper left; pl. 9, fig. 2; Roedder, 1963b, p. 176).

The finite but limited mutual solubilities of two relatively immiscible fluids such as CO_2 and H_2O theoretically provides another geothermometer (Smith and Little, 1959), but serious practical problems in phase identification may preclude its use (Roedder, 1963b, p. 190).

PHASE CHANGES IN THE FLUID UPON HEATING

In addition to geologic thermometry, a variety of interesting and eventually informative observations have been made of phase changes occurring in the liquid or fluid phase upon heating. Certain inclusions show an unmixing of the liquid phase, to form a small amount (<10 percent) of a new, immiscible liquid, on heating to temperatures above 200°C (Kalyuzhnyi, 1956, 1958c; Ermakov and others, 1957; Sobolev and others, 1970). The new liquid has an appreciably higher index of refraction than the surrounding low density liquid and presumably contains a higher concentration of salts. Ermakov, Kalyuzhnyi, and Myaz' (1957, p. 494 in translation) report that the new liquid persisted, with little change in volume, until the disappearance of the gas bubble at 327°C and that the phase changes were reversible on cooling. The significance of this immiscibility, in terms of specific fluid composition, is not known, but in view of the probable shapes of the pressure-temperature-composition diagrams for water-salt systems, such immiscibility with rising temperature under constant volume conditions is expectable. In this connection it might be pertinent to examine the behavior of inclusions in synthetic quartz crystals from those crystal-growth processes where a two-liquid condition in the growth chamber has been proposed (Butuzov and Bryatov, 1956), to see whether such immiscibility can be recognized. Prof. H. Sørensen of Copenhagen reports similar behavior in the fluid inclusions in chkalovite ($NaBeSiO_4$) from the Ilímaussaq nepheline syenite complex in Greenland. The solid daughter minerals in these inclusions dissolve by about 300°–360°, and homogenization occurs in the range 860°–980°, but a second liquid phase is present for a narrow range of temperatures near 700°–800° (H. Sorensen; oral commun., 1968; Sobolev and others, 1970).

Wahler (1956, p. 113) describes an inclusion in quartz from Madagascar that, when heated to about 150°C, developed a "new" gas bubble which vanished again at 220°C; he believes that this bubble probably was CO_2 and that it resulted from the reversal in the solubility of CO_2 with temperature. The data of Tödheide and Franck (1963), Franck (1964), and Takenouchi and Kennedy (1964) on the system CO_2-H_2O show that, for a considerable range in CO_2-H_2O ratio, immiscibility would occur on heating.

An additional interesting nondestructive thermal technique, which has been investigated to determine its potentialities (Hiroshi Ohmoto, Princeton Univ. written and oral commun., 1967), is, in effect, high precision differential thermal analysis. Preliminary results indicate that by the use of highly sensitive calorimetry, it is possible to measure the heat effects resulting from the melting of ice in previously frozen inclusions (and from decrepitation, although this of course is a destructive test) for 0.1-g samples containing $\sim 10^{-5}$ gram water as inclusions. Quantitative calibration involves several variables that may present experimental difficulties, but the large heat of melting of ice, and the inevitable experimental refinements, make the method very promising. MacKenzie (1968) reports similar thermal studies on the warming of samples (0.02 ml) of previously-frozen liquids.

CRITICAL PHENOMENA

Not infrequently the composition and degree of fill of an inclusion are such that it exhibits homogenization at a critical point. This is recognized by the abrupt and complete fading of the meniscus between liquid and vapor. The apparent occurrence of this behavior is made more common by the fact that for many fluids, the liquid and vapor exhibit rather gross changes in density in the last few degrees below the critical temperature, that is, the two-phase field is almost flat on top (fig. 4). There is only one true critical density (degree of fill), but as a result of the flat top to the two-phase field, inclusions grossly different in density—on both sides of the critical density—will appear to go through a critical point on rapid heating or cooling. Thus the more precise the temperature control near the critical point, the smaller the number of inclusions that will appear to exhibit critical phenomena. Inclusions with a degree of fill greater than the critical density will homogenize in the liquid phase (usually with a visible fading of the meniscus just before the bubble shrinks to disappearance; see pl. 6, figs. 5–8); inclusions with less than the critical density homogenize in a similar manner by the evaporation (disappearance) of the liquid phase (that is, they "homogenize in the gas phase"), usually with a visible fading of the meniscus just before the bubble expands to fill the inclusions.

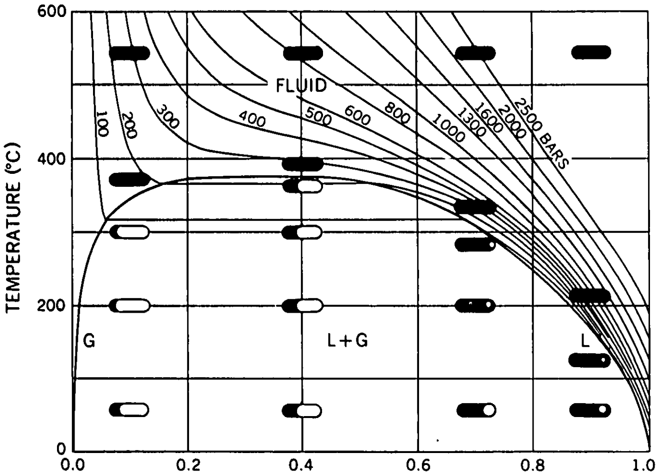

FIGURE 4.—Temperature-density diagram for the system H_2O, plotted from the data of Kennedy (1950b) and of Maier and Franck (1966). The homogenization behavior of four inclusions, all trapped at 540°C (but at different pressures), is indicated. The inclusions of density 0.9 and 0.7 homogenize in the liquid phase (L); that with density 0.4 homogenizes at the critical point; that with density 0.1 homogenizes in the gas phase (G) (from Roedder, 1962b).

The critical temperature is a definitive property for pure substances, and one inclusion fluid, CO_2, is sometimes sufficiently pure in nature to exhibit critical phenomena at the critical temperature (+31.0°C) for the pure material (Kobe and Lynn, 1953; pl. 6, figs. 5–8). In general, however, the fluids of inclusions are multicomponent mixtures, and any critical points (C.P.) they show are for such mixtures. Thus mixtures of methane (C.P., −82.1°C) and ethane (C.P., +32.3°C) have been suggested as the probable major constituents of two monophase inclusions in quartz crystals from Herkimer County, N.Y., showing critical behavior at −5.55°C and −7.85°C (pl. 5, figs. 6, 7). Touray and Sagon (1967) report CH_4 as a major component of similar monophase inclusions in quartz crystals from a marl in the Pyrenees. Inclusions of water solutions may show that the critical temperature for pure water (374.2°C) has been lowered by materials such as CO_2. Cameron, Rowe, and Weis (1953) found inclusions showing critical phenomena at 311°C. More commonly, the salts in solution raise the critical point. Sourirajan and Kennedy (1962) show that 25 weight percent NaCl raises the critical point for water to about 670°C, and many critical point determinations on fluid inclusions fall in the range of 375° to 450°C (Ermakov, 1950a; Bazarov and others, 1968). The temperature of trapping—and hence of formation of the enclosing mineral—cannot lie below these determined critical temperatures, except fortuitously, where a heterogeneous gas-liquid mixture with an overall density equal to the critical density has been trapped. In general, the pressure on the system at the time of trapping was greater than the critical pressure, and hence a pressure correction must be added to these critical temperature determinations. Klevtsov and Lemmlein (1959a) mention the occurrence of homogenization of the gas and liquid phases in inclusions at a critical temperature *below* that at which all solid halide daughter minerals had dissolved.

Even where the density of fill is not near the critical density for the specific fluid, it is important to note the rate of change of the volumes of liquid and gas phase with increase in temperature, in addition to the actual temperature of homogenization. This can provide a crude estimate of the probable critical temperature of fluids trapped at slightly more than the critical density, for as the critical temperature is approached, not only is the thermal expansion of the liquid large, but its rate of increase with rise in temperature becomes progressively greater (fig. 4), and it becomes effectively infinite at the critical temperature. Thus the high thermal expansion of the unidentified liquid (CO_2) found by Brewster (1823a) merely reflects a close approach to its critical temperature of +31°C (pl. 6, figs. 5–8). Similarly, for inclusions homogenizing in the gas phase, the rate of increase in density of the gas phase becomes progressively greater (fig. 4). This cannot be measured as conveniently as can the expansion of the liquid, but the rate of change in density can be estimated from the rate of evaporation of the liquid phase into the gas phase. Thus, Edwin Roedder (U.S. Geological Survey, 1965, p. A156; see also Roedder and Creel, 1966) noted that approximately 12 volume percent (at room temperature) of a phase believed to be liquid H_2S (pl. 7, fig. 11) evaporated into the vapor bubble at temperatures ranging from 64° to 97°C. These temperatures are above the critical temperature for liquid CO_2, excluding this possibility. Both the temperatures and the density increase of the gas phase are appropriate, however, for liquid H_2S near its critical temperature of 100.4°C. Such data are not definitive, but do present strong support to other data which also indicate that this phase is liquid H_2S.

FREEZING DATA

Since the pioneering effort of Sir Humphry Davy in 1822, a number of attempts have been made to use the behavior of fluid inclusions at low temperatures to help in the identification of the materials present. Simple cooling with a drop of alcohol, acetone, ether, or of ethyl chloride (available in handy pressurized tubes as a medical supply), will frequently prove useful as a qualitative test. The experimental difficulties, however, had generally prohibited the accumulation of much quantitative data.[11] As the procedure showed considerable merit, in theory, a microscope freezing stage was designed to avoid some of the troubles (Roedder, 1962a). With this equipment, a variety of data has been obtained on fluid inclusion compositions (Roedder, 1963b; Roedder and others, 1963; Hall and Freidman, 1963; Sawkins, 1963; Buseck, 1966; and Ohmoto, 1968a, 1968b), the most useful of which is the depression of the freezing point of the liquid water solutions, that is the "freezing temperature" (pl. 8, fig. 1). This depression is a measure of the salinity of the fluid, and it permits estimation of the salt concentration in inclusions weighing as little as 10^{-10} g. Other phase changes are observable in fluid inclusions at low temperatures, most of which yield at least qualitative compositional data (Roedder, 1963b). Among these are the triple point of CO_2 at -56.7°C and the "first melting temperature," representing the first development of visible liquid upon heating a solidly-frozen inclusion (pl. 8, fig. 1; Roedder, 1962a, p. 1054). This latter phenomenon, unfortunately, has also been taken to be the "freezing temperature" of the solution (Kormushin, 1965, p. 173). (See also a discussion of the identification of the solid phases formed, page JJ22.) Freezing point depressions for saline fluids were also obtained by Lisitsyn and Malinko (1961), using an unspecified method, and by Rutherford (1963), who determined both the salinity of the water phase and the density of the CO_2 phase in mixed inclusions from the Flin Flon area by this method. A number of more recent applications of the method are noted in table 1.

The freezing stage is particularly useful to detect leakage of fluid inclusions. As most inclusion fluids are far more concentrated than normal ground waters, a single inclusion in which the original fluids have been replaced with ground water will be readily apparent, even in a large group of normal inclusions (Roedder, 1963b, p. 207).[12] Low-temperature microscopy is also helpful in the recognition of homogeneous CO_2 inclusions of either very high or very low filling density. (See p. JJ15.)

Several metastable phenomena occur commonly during the cooling of inclusions. Although these generally cause some experimental difficulty, they are useful indicators that inclusion fluids are generally very clean and free of spurious solid nuclei and hence have presumably been trapped from exceedingly slow-moving solutions (Roedder, 1962a, 1963b). If the expansion on formation of ice eliminates the vapor bubble, sluggishness in its renucleation may lead to high negative pressures, metastable "superheated" ice, and erroneously high apparent freezing temperatures (Roedder, 1963c, 1967b).

BUBBLE MOVEMENT IN THERMAL GRADIENTS

Sang (1873; see also Hunter and Sang (1873) and Tait and Swan (1874)) reported that peculiar movements of the bubbles in some fluid inclusions occurred when a thermal gradient was established across the inclusion.[13] Some bubbles moved *up* a thermal gradient; that is, they appeared to be attracted to a warm probe touching the sample and moved through the fluid as far as the inclusion shape would permit. Other bubbles either moved *down* a thermal gradient, that is, they appeared to be at-

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[11] Ermakov (1950a, p. 344 and 348) mentions the use of a "microfreezer," but he gives no details or data. Lemmlein and Klevtsov (1955) give some experimental data on quartz from Pamir, but they do not give details on experimental technique or equipment.

[12] Although not a usual type of "fluid inclusion," it should be mentioned that the microscopic vesicles in most older pumices become filled with water over geologic time. This is water that has diffused *through* the glass walls, leaving almost all salts behind. Thus the freezing temperature of all of these "inclusions" is almost exactly 0.0°C (Roedder and Smith, 1964; Roedder, 1970a).

[13] This is presumably not the first description of the phenomenon, as William Nichol (1829) mentions that the bubble in an inclusion in halite from Cheshire did not move when a hot wire was placed opposite it, thus implying that other bubbles do move under such conditions.

tracted by a cold probe and repelled by a warm one, or they did not move at all in a thermal gradient even though they were apparently free from the walls. Still others exhibited very peculiar oscillatory movements, either of the bubble itself or of solid particles in the liquid (Brewster, 1845b; Hartley, 1877b; Hawes, 1881).

These phenomena were variously attributed to "unknown cause," "capillarity," or "evaporation and recondensation," until Hartley (1877b) offered a brief explanation (proposed by Stokes) based on changes in surface tension. This seemed to explain some of the apparent conflicts in the data, and the subject was dropped, except for rediscovery by the writer and several others (Hoagland, 1951b; Rush, 1954; Safronov, 1957b; Johnson, 1961a, 1961b; Sutton, 1964).

The basis for all these phenomena appears to be variation in the surface tension over the bubble surface, causing flow of the surface that drags fluid with it. Although the phenomena are still not fully explained, the motions of the bubbles can be detected very quickly and simply by hot or cold probes (Roedder, 1965e, 1966a), and these motions are useful in inclusion studies in a variety of ways. First, the specific behavior is obviously controlled by the composition of the fluid. Even though the nature of the effects of these compositional parameters is complex and not understood, *differences* in the behavior of the bubbles in adjacent inclusions that otherwise seem to be identical indicate differences in composition and presumably origin, and these differences frequently correlate with differences in freezing and homogenization temperatures. Second, the phenomena aid in the determination of freezing temperatures in those inclusions in which the last ice crystal sticks to the bubble-liquid interface. In these, the thermal (and compositional?) gradients set up by slight melting of the ice result in a rapid movement of the bubble and ice crystal about the inclusion. The bubble may push the ice ahead of it, or drag it with it, but in either case the movement is an indication that melting is still taking place, even though the ice may be invisible. Also, many inclusions have only one small clear "window" through which the freezing behavior can be watched; movement of the bubble and ice across this window thus permits more positive identification. Third, the phenomena aid in homogenization experiments. In these, the bubble movement permits an evaluation of the direction of the inevitable thermal gradients in the heating cell, and, by superimposing small thermal gradients on the heated sample near to its homogenization temperature, tiny bubbles may be moved out of dark areas of total reflection. Fourth, as mentioned in "Fluidity" (p. JJ10), bubble movement from thermal gradients may be used to discriminate between glass and liquid inclusions. Fifth, thermal gradients are frequently useful to place the bubble in an inclusion in a position more suitable for photography, and were used in several of the figures in this report.

COMPOSITION OF SOLID PHASES

Daughter minerals in fluid inclusions—solid phases which have crystallized out of the fluid after trapping—indicate saturation of the fluid with respect to these phases, *at the conditions of observation.* Hence they are extremely useful in determining the composition of the fluid, but they have not been given adequate study. Probably the most serious problem in such use lies in the apparent difficulty of identification, but there are a number of methods that can be used, providing a combination of identification criteria that is generally unambiguous.

GENERAL SIGNIFICANCE AND USEFULNESS

The problem of distinguishing between daughter minerals, formed from the fluid of the inclusion, and accidental solid inclusions, trapped along with the liquid, is sometimes difficult.[14] Regularity of phase ratio in a number of inclusions is perhaps the best criterion for a daughter mineral origin (pl. 3, figs. 3, 4), but problems of recrystallization, causing gross changes in the shape of elongated inclusions (frontispiece, upper left, lower left, and lower right; pl. 10, figs. 3–5), and hence isolation of already-formed daughter minerals from their parent liquid (Lemmlein and Kliya, 1952b), can interfere seriously. In addition, small inclusions may not show a daughter mineral that adjacent larger ones have; this is commonly due to failure to nucleate. In NaCl solutions, freezing may cause the nucleation of the stable phase, NaCl crystals, in such supersaturated inclusions (Roedder, 1967a; Touray and Sabouraud, 1970). Any phase with which the solution is supersaturated may be caused to nucleate by freezing, as this withdraws water from the solution and increases the degree of supersaturation (pl. 2, figs. 1, 2).

Fortunately, the distances are so small in fluid inclusions that daughter crystals, at least the water soluble ones, have generally had time to reach minimum surface energy, that is, one single crystal of each phase (Bienfait and Kern, 1965). Hence, the

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[14] Thus Dymkov (1961) describes a case of hematite spherulites growing in negative crystal cavities in calcite, forming complex inclusions.

number of daughter mineral phases is generally equal to the number of crystals. This is not true for very slightly soluble crystals or for those formed during a relatively fast cooling.

Ermakov (1950a, p. 24 in translation) has noted cases in which the sequence of formation of several daughter minerals upon cooling is preserved in the inclusions as a record of the sequence of saturations. He also presents what he considers to be evidence (p. 38 in translation) of the crystallization of quartz from fluids that were saturated with respect to NaCl, so that NaCl occurs both as solid inclusions and as a daughter mineral.

In every case the host mineral should be considered as an additional "daughter mineral" phase, as at least small amounts of it must crystallize out from almost every fluid inclusion, upon cooling. Where this new material on the walls forms a visible layer it has been designated a "bordure of cognate substance" by Ermakov.

In describing certain synthetic minerals and the crystals formed in inclusions of glass, Sorby (1858, p. 457 and 477) used an effective aid in the identification of some daughter minerals that seems to have been forgotten by many later workers. Further crystallization of the host mineral from the trapped fluid will generally occur on the walls of the inclusion, but other phases form separate daughter crystals. Thus, inclusions of a given fluid, saturated with respect to minerals A and B, will show daughter crystals of A in inclusions trapped in B, and vice versa. Kalyuzhnyi and others (1966) report daughter crystals of quartz (?) in inclusions in quartz, and Llambias (1963) reports the occurrence of a daughter crystal of borax, presumably identified as such by crystal shape, in a fluid inclusion in a larger borax crystal. These are the only observed exceptions—almost always the host mineral does not form discrete daughter crystals. Llambias mentions the occurrence of organic matter within the host borax crystal. It is thus possible that a film of such material lining the inclusion prevented crystallization of the material of the daughter crystal directly on the walls.

Solid daughter minerals in fluid inclusions—as well as solid inclusions trapped directly in the crystal—have considerable value in the identification of gemstones (Gübelin, 1950, 1969). Upon cooling, the differences in contraction and compressibility of the solid inclusion and the host gem may set up considerable strain, first recognized as strain birefringence by Brewster (1835, 1845a; pl. 6, fig. 3). Such strain can cause cracking and the development of secondary inclusions in the healed fractures. Even the specific locality producing a given gemstone may sometimes be recognized on the basis of the inclusions. Thus for emerald, the occurrence of fluid inclusions containing NaCl crystals has been used as a fairly reliable criterion that (1) the stone is natural and not synthetic,[15] and (2) that it probably came from the Colombian emerald mines (pl. 2, figs. 1–6; pl. 5, fig. 2). The writer has found NaCl crystals in primary inclusions in Rhodesian (Sandawana) emeralds, however, and Webster (1952) reports seeing NaCl cubes in Australian emeralds. Gübelin (1950) also mentions another identifying feature of the NaCl crystals in Colombian emerald—occasionally these daughter crystals show an irregular bright blue coloration typical of NaCl.

ORDINARY METHODS OF MICROSCOPY

Brewster (1826a, p. 21) was the first to report the occurrence of "squares" (that is, cubes, probably of NaCl) in inclusions. Solid daughter mineral phases in fluid inclusions are generally so small that only optical methods are adequately sensitive for their identification. Many of the usual petrographic techniques may be used, in modified form, but rather severe limitations are imposed by the nature of the samples. Thus, observations of extinction angles and birefringence are limited by the optical properties of the host mineral. Sorby (1858) pointed out that the inclusions in birefringent plates are best studied with a polarizer in, to eliminate troublesome double images. The writer has found that with strongly birefringent minerals such as calcite it is important to use only the image of the *ordinary* ray, which yields much sharper definition than the extraordinary ray. Since these carbonate minerals are optically negative, this procedure also permits an appreciably greater maximum depth of focus into the plate (pl. 3, fig. 3, 4). The thick plates (0.5–5 mm) normally used for inclusion study usually show so much birefringence (even with normally isotropic host minerals such as fluorite and halite) that both extinction angles and birefringence of solid inclusions and daughter minerals can be estimated only by simple statistical methods, using many inclusions. The maximum interference color exhibited by the daughter mineral grains can be used in conjunction with their apparent thickness, if they are equant, to

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[15] There have been several reports of three-phase inclusions, presumably liquid+gas+NaCl crystal, in some synthetic emeralds (Webster, 1952; Wells, 1953). As some of the procedures used to grow synthetic emeralds are still secret, such inclusions could be useful, but it is difficult to verify the true source of any given stone.

obtain the birefringence, but it is particularly difficult to estimate the thickness of thin flat daughter minerals (pl. 12, figs. 1, 2). Eppler (1962b) has shown that the interference color seen in reflected light from flat inclusion crystals can sometimes be used to obtain such an estimate. Crystal habit, interfacial angles, color, pleochroism, etc., all are useful criteria for identification (Mel'nikov, 1968a). Not infrequently the daughter minerals are sharply faceted crystals that permit identification of their crystal system as well. Generally the inclusions are too deeply buried in the sample, or are too small, to obtain interference figures; Kalyuzhnyi (1958a) mentions grinding and polishing to within 10μm or 15μm of the inclusion to permit the use of certain techniques.

Opaque daughter minerals, presumably metallic sulfides and oxides,[16] occur in some inclusions. Their rarity and small size have been used to place limits on the quantities of metal that may be precipitated from the ore-fluids by temperature decrease (Roedder, 1960b; Takenouchi, 1962b). The determination of the size, and hence of the volume and weight percent, of opaque grains is usually limited to measurements of the two visible dimensions and a guess as to the third dimension. In some inclusions the crystal can be moved about for further measurements. There are relatively few available parameters that can be used for the identification of opaque daughter minerals. In a very few inclusions the grain is sufficiently large to permit recognition of the color and crystal habit in reflected light (Roedder, Ingram and Hall, 1963, p. 367). Not infrequently, the external crystal shapes—or rather, silhouettes of them—are visible. Brewster (1853b) was able to estimate the relative *densities* of several different daughter mineral grains by comparing the rates of fall through the liquid when the sample was inverted. One simple but very sensitive test that should be used routinely on opaque grains is for their *magnetic* properties; if they are magnetic, and loose in the cavity, they will move when a small strong magnet is rotated or moved near the microscope objective. As an additional test, Prof. W. C. Kelly (Univ. of Michigan, oral commun., 1967) has been able to determine the Curie temperature of magnetic daughter minerals by observing reversible changes in their properties on the heating stage. (See also Kelly and Turneaure (1969, 1970).)

ESTIMATION OF INDEX OF REFRACTION

One of the most useful and definitive properties of transparent daughter minerals is the index or indices of refraction. These can be estimated by comparison with whatever phase is available. Thus Lemmlein, Kliya, and Ostrovskii (1962) found crystals of cryolite ($n=1.34$ and hence is comparable to or even less than water solutions) in the inclusions of a pegmatitic topaz (pl. 12, figs. 5–7). Ikorskii (1966a) reports inclusions containing rose-colored cubes of villiaumite (NaF, $n=1.336$) and organic matter in many of the alkalic rocks of the Khibina massif. The isotropic cubes (and occasionally octahedrons; see pl. 2, figs. 3, 4) seen in many inclusions are usually NaCl ($n=1.544$), but not infrequently NaCl occurs with KCl ($n=1.490$; see pl. 2, figs. 1, 2). (The vertical sides of such cubes may make estimates of relative relief rather difficult.) Kalyuzhnyi (1958a) notes that NaCl daughter crystals may be pinkish or yellow, but upon recrystallization after heating and cooling, the color is gone. KCl cubes are commonly bright yellow. The writer has observed that daughter crystals of NaCl in inclusions in quartz are sometimes in sufficiently close contact with the host (pl. 2, fig. 5) that a sensitive comparison can be made between the index of the halite ($n=1.5443$) and the ordinary ray of the quartz ($n=1.5443$). (See also pl. 6, fig. 4.) The universal stage may also be useful in identifying such embedded solid crystals (Bakumenko, 1964), and similar comparison can sometimes be made with closely adjoining daughter minerals as well.

Strong birefringence is easy to recognize even without placing the host crystal at extinction (frontispiece, lower right), but if the host crystal has a considerably higher birefringence than the daughter mineral, particularly where the host does not go to complete extinction as in many thick sections of calcite or quartz, this test is inadequate. In such plates, grains with high birefringence can be recognized by the change in relief relative to the liquid on rotation of the stage with only the polarizer inserted. Thus, Slivko (1955, p. 70) shows that one of the many daughter minerals in inclusions in green multicolored tourmaline has one index of refraction very near to that of the solution in which it occurs. It also has a high birefringence and is very soluble, even in refractive index liquids. It is presumably a fluoborate (Slivko, 1958). On very tiny grains, the brightening of the centers on slight change of focus can be used for index comparisons in lieu of an actual Becke line.

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[16] The most common oxide daughter mineral is hematite, but it is frequently in sufficiently small crystals to be bright red (or even yellow) and birefringent.

USE OF THE ELECTRON MICROSCOPE, ELECTRON MICROPROBE, AND X-RAY DIFFRACTION

As inclusions are generally small, electron microscope techniques would seem to be very appropriate for their study, but the very shallow penetation of the beam precludes its use on most unopened inclusions. McLaren and Phakey (1965a, b; 1966) have used transmission electron microscopy to study the development of dislocations and associated spherical and faceted negative crystal cavities in very thin flakes of citrine and amethyst, particularly on annealing at temperatures of 600° to 800°C. Akizuki (1967a) used an acetate peel technique to obtain layers of galena thin enough for transmission electron microscopy. The electron microscope has been used very effectively on replicas of fractured surfaces to study the structure of the walls of inclusions and the changes in shape with recrystallization (Akizuki, 1965a, b, c; 1966; 1967b; Kurshev and Trufanov, 1965) and to study the distribution and nature of very minute inclusions on grain boundaries and within grains (Folk and Weaver, 1952; Iwao and others, 1953; Pelto, 1956; Deicha and Sella, 1962; Sella and Deicha, 1962a, b, 1963).

Similarly, the electron microprobe would be eminently suitable for determining the composition of daughter minerals, if they could be put into the electron beam. Absorption of both the incident beam and the emitted X-rays generally precludes examination of unopened inclusions. Unless the inclusions are very large, however, the mechanical problems of removal of daughter crystals for study are severe. At the time of writing there were only three studies of the use of the electron microprobe on inclusions. The first was by Carron (1961) who used it to study the composition of *glass* inclusions in quartz phenocrysts that were truncated by the polished surface and hence were accessible to the beam. This study was made because such glass inclusions represent samples of the actual magma, complete with volatiles. The second was by Dolomanova and others (1966; 1968) who determined the presence of Fe, Cu, Ti, Ca, Zn, and Cr among the 11 daughter minerals on the walls of a fluid inclusion in an early smoky quartz from an ore deposit in Transbaikal. They believe that the iron and chromium are present as chromite. Dr. Dolomanova states (written commun., 1967) that she analyzed only those solid grains on the upper surface of *unopened* inclusions that were sufficiently close to the polished surface to permit electron beam penetration.

Valyaskho and others (1968) report 41 qualitative electron microprobe analyses of a series of solid, congealed, and gas-liquid inclusions in various minerals from the Khibiny apatite deposits. Some of these were made on specific solid included grains and some on solid phases after homogenization runs.

Identification by X-ray diffraction methods generally requires the extraction of the daughter mineral, unless it is present in large amounts (for example, halite is present in large amounts in inclusions in feldspar from Ascension Island) (Roedder and Coombs, 1967)). Ermakov (1965b) and Lyakhov (1966) extracted and identified a variety of daughter minerals by X-ray diffraction. Lyakhov reports X-ray data on several unknown minerals, and he also verified the presence of halite, sylvite, $FeCl_2 \cdot 2H_2O$, elpasolite, cryolite, and a hydrated magnesium carbonate in inclusions in smoky quartz from pegmatites in Volynia. (See pl. 12.) Kalyuzhnyi and Voznyak (1967) also report $FeCl_2 \cdot 2H_2O$ as a daughter mineral, along with qualitative determinations of both ferrous and ferric iron in quartz from "Zanorysh" type pegmatites. Kalyuzhnyi and Mikolaichuk (1968) report the use of electron diffraction to identify the salts present in the dry residue (NaCl, KCl, and NaF), obtained by evaporation of a solution from multiphase fluid inclusions in topaz. Trufanov and Kurshev (1968) used electron microscopy and both powder and single crystal electron diffraction to identify the residues.

BEHAVIOR ON FREEZING

Upon freezing inclusions, a variety of new solid phases may be formed, and the identification of these frequently yields compositional information otherwise unobtainable (Roedder, 1962a and 1963b). The most common new phase is ice, which can be identified by (1) index of refraction (always appreciably less than that of the solution), (2) very low birefringence, (3) characteristic growth, on cooling, of two parallel flat plates from opposite sides of the round grain obtained by partial melting, (4) parallel extinction and length-fast orientation of these plates, (5) volume increase on freezing and decrease on melting (this registers as an *increase* in bubble size with rising temperature—see pl. 8, fig. 1); and (6) temperature range of stability.[17] Various salts and hydrates may also crystallize on freezing. A particularly significant one is $NaCl \cdot 2H_2O$ (hydrohalite). This forms by reaction of the liquid with daughter NaCl crystals upon cooling. As

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[17] Care must be taken in using this criterion with inclusions whose vapor bubble is eliminated by the expansion on freezing. These may develop high *negative* pressure upon partial melting, which, in turn, may cause ice to exist, metastably, at temperatures as high as +6°C (Roedder, 1967b). Many larger inclusions are broken open by the expansion of freezing, as first recorded by Dwight (1820).

it is strongly birefringent, and melts incongruently at temperatures up to a maximum of $+0.1°C$ (in the pure system $NaCl\text{-}H_2O$), it provides a very good test for distinguishing a cube of NaCl from KCl, which forms no such hydrate. As little as 10^{-12} of NaCl can be recognized by this test (pl. 11, figs. 1–4; Roedder, 1963b, p. 178 and 182; Roedder and Coombs, 1967).

Another useful solid phase formed on cooling certain inclusions is the clathrate compound carbon dioxide hydrate $CO_2 \cdot 5\frac{3}{4}H_2O$[18] (Roedder, 1963b, p. 188–196), which forms by reaction of H_2O and either gaseous or liquid CO_2. Unfortunately, its index of refraction is very close to that of most water solutions, and it is isotropic, so that it is not always visible even though present in rather large amounts. Sometimes its presence is evidenced only by an irregular, jagged interface between gas and "liquid" (pl. 5, figs. 3–5). W. C. Kelly (Univ. of Michigan, oral commun., 1967) has found that the presence of such invisible crystals can also be detected in those inclusions containing a magnetic daughter mineral. When such a magnetic grain is pulled around in the inclusion by manipulating a magnet on the microscope stage, it can be seen to bump into the invisible hydrate crystal. Data on its dissociation pressure (Takenouchi and Kennedy, 1965a) may permit estimates of the CO_2 pressures in some inclusions from the freezing data. One serious source of ambiguity in the use of freezing data on this phase is that the various clathrate structures permit a wide variety of molecules to be trapped, including H_2S, CH_4, C_2H_6, and numerous other organic species (Stackelberg and Müller, 1954; Pauling, 1961; Barduhn, 1963; Jeffrey, 1963; Jeffrey and Mak, 1965).

Occasionally freezing will result in the elimination of metastable equilibria within inclusions, by causing the formation of new phases that should have been present at surface temperatures. Thus freezing commonly permits the formation of a gas bubble in single phase inclusions, formed at temperatures below 100°C, that have persisted for geological time as metastable stretched fluid, under negative pressure (Roedder, 1963b, p. 197; 1967b). Similarly daughter minerals are not always present in inclusions that should contain them. Salt (NaCl) crystals usually nucleate, even in very tiny inclusions at the limit of resolution of the light microscope (Roedder, 1967a, p. 533), but Touray and Sabouraud (1970) have described relatively large supersaturated inclusions that got a NaCl daughter crystal only after freezing and rewarming to room temperature. Daughter minerals present in only small amounts are more apt to remain in (supersaturated) solution. It is thus common for small multiphase inclusions to have fewer solid phases than adjacent apparently cogenetic large inclusions, and the missing phases are almost always those present in minor quantity (for example, Roedder and Coombs, 1967). A very unexpected case of elimination of such metastable supersaturation is shown on plate 2, figures 1 and 2.

BEHAVIOR ON HEATING

Behavior on heating is a very important aid in the identification of daughter minerals. Brewster (1845b) was the first to study this behavior with care and to make use of it to prove the existence of a number of different daughter mineral phases in the same inclusion in topaz. Thus KCl can be distinguished from NaCl by its much higher temperature coefficient of solubility, as pointed out by Ermakov (1950a, p. 57 in translation). (The increase in solubility is approximately eight times greater for KCl than NaCl; see pl. 7, fig. 12.)

NaCl and KCl have relatively limited mix crystal fields at low temperatures, but above 300°C these mutual miscibilities become appreciable, and the solid solution is complete above about 490°C (Thompson and Waldbaum, 1969). Although there apparently has been no mention of it in the fluid inclusion literature, the behavior of NaCl and KCl daughter crystals should be examined in the light of this diagram, as this could yield significant data on the composition of the system.

Yakubova (1952, 1955) used crystal shape and solubility to show the common presence of borax crystals in inclusions in quartz and topaz from a Uralian pegmatite. She verified the identification with microchemical and spectrographic analyses. Ikorskii (1968) describes the effects of heating to 360° and 480° on bitumens in inclusions in eudialyte from the Khibiny apatite deposits.

Many of the recorded heating experiments on inclusions having several daughter minerals show that some of them do not dissolve, even at high temperatures (pl. 3, fig. 1; pl. 7, fig. 1). Thus Feklichev (1962) found that some of the daughter crystals in pegmatitic beryl did not dissolve even at 750°C, and there are numerous references to daughter minerals, particularly ones that are opaque or that have high indices of refraction, persisting to temperatures above the homogenization temperature for the liquid-plus-gas phases (Ermakov, 1950a; Kalyuzhnyi,

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[18] Although usually referred to by this formula, the structural formula is $8CO_2 \cdot 46H_2O$ (Stackelberg and Müller, 1954).

1958a). There are four possible explanations of such behavior:

1. Equilibrium was not attained in the time used. The relatively soluble crystals, such as NaCl and KCl, equilibrate with the solution rapidly on heating (however, see pl. 7, figs. 2, 3 and 4), but Ermakov (1950a, p. 95 in translation) reports that 4 to 5 hours are needed to reach equilibrium, even at high temperatures, for the dissolution of the inclusion walls and of the less soluble daughter minerals.
2. Equilibrium was attained, but the correct temperature for melting of the last grain of the daughter mineral actually does lie above the liquid-plus-gas homogenization temperature. There is no *a priori* reason why a given solution, upon cooling, should not become saturated with a solid phase before it separates into two fluids, liquid plus gas (pl. 7, fig. 12). In such cases, the temperature of homogenization of the daughter minerals would be closer to the true formation temperature than the liquid-gas homogenization temperature would be, as pointed out by Sheftal' (1956) and others.
3. The "daughter" mineral grain was merely a solid inclusion, trapped accidentally at the time of formation of the inclusion. Perhaps the best criterion for distinguishing between an accidental solid inclusion and a true daughter mineral is the constancy of occurrence, and of volume percent, of each daughter mineral in several or many inclusions. Unfortunately, available sample material does not always permit the application of this criterion. If a given mineral occurs also as solid inclusions in the host mineral, it is difficult to be certain that its occurrence in fluid inclusions is truly as a daughter mineral; certainly the solutions *were* saturated with respect to it, and hence it should occur as a daughter mineral.
4. Not infrequently, evidence of necking down of inclusions is found, in which the daughter minerals (and gas bubbles) present at that time are restricted to one of the several smaller inclusions forming at the expense of a larger one; this can cause very diverse phase ratios in different inclusions (pl. 10, figs. 3–5; frontispiece, lower right).

The dissolution of the walls of inclusions upon heating is of considerable importance in understanding the actual composition of the fluids as they formerly existed in the earth. Ermakov (1950a, p. 54 of translation) and Lemmlein (1950a) believe that solution of the walls and the resultant volume change is of great significance to the use of inclusions for thermometry. It appears, however, that the amount of solution of the walls is generally negligible (and the change in volume, ΔV, on dissolving is far less than the volume dissolved) and that the ordinary heating rates used are adequate to permit an approach to equilibrium, except for inclusions, homogenizing at very high temperature, that resemble silicate melts in composition (Kerkis and Kostyuk, 1963; Zakharchenko, 1968) (pl. 7, figs. 5 to 8). It is usually necessary to study the appearance of minute ridges and points on the walls of the inclusion during heating to recognize the existence of *any* solution at all. Actually, it is difficult to separate the effects of a sluggish approach to equilibrium with respect to (1) the solution of daughter (and host) minerals, and (2) the thermal regime in the microscope hot stage at the actual inclusion site.

Lemmlein, Kliya, and Ostrovskii (1962) describe heating experiments on large primary seven-phase inclusions in topaz, containing 70 volume percent of solids at room temperature (pl. 8, figs. 2–4). Each had quartz, muscovite, cryolite, an undetermined mineral in pseudo-hexagonal plates of $n=1.51$, fluorite, liquid, and gas, in the same volume ratios. In order to prevent decrepitation of the inclusions from the internal pressures developed—a common and very troublesome problem in much inclusion work—they heated the samples in a bomb at 3,000 kg/cm^2 external pressure. Their data and photographs (before heating and after quenching) indicate that not only do the daughter minerals dissolve to form a silicate melt (yielding a glass on quenching), but the solubility of topaz in these fluids, at 700°C, is about 10 to 15 volume percent (pl. 8, figs. 5–6). The resulting melt was estimated to contain over 10 percent H_2O.[19] One interesting point they make is that those inclusions that had fractured and lost their volatiles did not dissolve their daughter minerals. Voznyak (1968) presents considerable evidence that at least some of these "daughter minerals" are actually solid inclusions, trapped during the growth of the crystal. However, those phases that occur in uniform ratios in several inclusions (see pl. 12) are probably valid daughter minerals.

Feklichev (1962) eliminated the problem of decrepitation by heating his samples to the maximum temperature of observation (750°C) *before* an in-

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[19] It should be noted that the occurrence of daughter crystals, and not hydrous glass, in the samples as found is evidence of the slow cooling rates in the natural processes. As these inclusions are sealed systems, with all "mineralizers" still present, rate studies on them should be particularly significant.

clusion was selected for high temperature study; by this simple but effective procedure he eliminated those inclusions that were not suitable for high-temperature study. The writer wishes to add that unheated samples should be examined also, because (a) the preheated ones present a biased sample of inclusions present, (b) the inclusions may become stretched, and (c) because many daughter minerals are very slow to recrystallize after dissolving.

Lemmlein and Kliya (1952a) used an ingenious interferometric technique on flat inclusions in topaz to make rough quantitative estimates of the amount of topaz precipitated on the inclusion walls upon cooling. They showed that during the original slow cooling of their topaz samples, the nucleation and growth of the bubble was simultaneous in part with the deposition of topaz on the walls from the inclusion fluids; a record was left in the form of crescent-shaped rings, each showing where a former size and shape of flattened bubble had prevented deposition of topaz on the walls against which it was pressed. The amount of topaz in solution in the inclusions at 200° was estimated to be about 2 volume percent. (See pl. 8, figs. 2–6.)

Kalyuzhnyi (1956) describes several interesting changes in inclusions in topaz. Daughter crystals of elpasolite (K_2NaAlF_6) in multiphase inclusions melted incongruently to form three other phases of lower index of refraction, apparently also fluorides, in the temperature range 135° to 170°C. Other inclusions showed decomposition of elpasolite crystals to still another unknown phase at 250° to 260°C. (See pl. 12.)

Zakharchenko (1955) describes some very interesting polyphase inclusions in quartz from quartz veins in the Pamir, U.S.S.R., which contain as many as nine daughter minerals. They were identified (based in part on destructive tests on large inclusions), in descending order of volume percent abundance, as magnesian calcite (estimated, from Zakharchenko's fig. 15, to be $\sim$20 percent), halite ($\sim$10–20 percent) sylvite ($\sim$1 percent) hematite ($\sim$1 percent), a sericitelike phase ($<$1 percent), clear albite ($<$1 percent), and several other unidentified phases.[20] The fluids present are water solution, liquid carbon dioxide (6–8 percent), and a gas having a very strong odor of H_2S ($\sim$35 percent). During homogenization of the high temperature inclusions (at temperatures of 360° to 420°), very significant amounts of the quartz host dissolve. This is estimated, from measurements of the inclusion size, to be up to 160 g/l (grams per liter) (Zakharchenko, 1955, p. 45; also stated as 6 volume percent or 13 weight percent, p. 42). From these data Zakharchenko calculates that the total volume of mineral matter carried in the solution, at the time of trapping, was 50 to 55 *volume* percent. Apparently even pure CO_2 can dissolve appreciable amounts of unknown solids (pl. 6, fig. 4).

A series of studies of the daughter minerals in large inclusions in pegmatitic quartz, topaz, and fluorite was summarized by Ermakov (1965b). In this work a variety of identification methods were used, as described above, and measurements by two-circle goniometer, X-ray diffraction, and microhardness determinations, were also used on extracted daughter minerals. As many as 30 (presumably all different) daughter minerals were recognized. One-third of these daughter minerals are opaque phases, and many of the other are new minerals. Halite, sylvite, and fluorides make up more than half of the total volume, but the following minerals were also identified: hematite, chlorite, albite, quartz, rutile, zircon, muscovite, staurolite, and sphene (pl. 12). Freezing studies on some of these (Roedder, 1963b, p. 181) have shown that the liquid water solution (at room temperature) contains high concentrations of salts with at least some $CaCl_2$. The samples containing these inclusions came from chambered pegmatites at Bektauata (Kazakhstan) and Volynia, some of which have central crystal-lined cavities up to 200 m^3 in volume.

Lindgren and Whitehead (1914) describe some inclusions in quartz from a hydrothermal ore deposit near Zimapan, Mexico, that have more than 50 weight percent NaCl, as solid daughter mineral and solution, plus unknown amounts of other materials in solution. If it is assumed that only NaCl and H_2O are present, such inclusions would homogenize at about 490° (Sourirajan and Kennedy, 1962). Similar amounts of daughter minerals are found in fluid inclusions in samples from other ore deposits (Sawkins, 1963, 1964, 1965a; Sawkins and Huebner, 1963; Takenouchi, 1962b). (See also pl. 3, fig. 1.)

Glass is a common solid phase in the inclusions in the minerals of volcanic rocks. There have been many interesting studies made of these inclusions, as some, at least, represent true samples of the magma, with all its volatile components (Carron, 1961), but these are beyond the scope of this report. These silicate melt inclusions may be treated in a manner analogous to that used for aqueous inclusions, to obtain the temperatures of melting on heat-

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[20] Spectrographic analyses of the fluid extracts showed small but constant amounts of Al and Fe. The pH of the diluted fluids (a leachate made after loss of gases) was found to be 8.6.

ing (and hence of crystallization on cooling) of the daughter minerals and the homogenization temperature (Roedder and Weiblen, 1970a, 1970b) (pl. 7, figs. 5–8). A few instances are reported of glass occurring in inclusions along with a fluid that is mobile at room temperature and a gas bubble. These probably represent trapping of an "emulsion" of droplets of the fluid phase in a silicate melt. Roedder (1965d) shows that olivine nodules in basalts from widely scattered localities always contain inclusions of liquid and gaseous CO_2 and that many of these contain variable amounts of glass as well (pl. 4, fig. 6). Roedder and Coombs (1967) describe inclusions now containing glass, salt crystal, solution, and gas bubble that represent trapping of various ratios of granitic melt and immiscible droplets of a hydrosaline melt, containing more than 50 weight percent NaCl (pl. 4, figs. 7 and 8).

COMPOSITION OF THE GAS PHASE

Relatively little can be determined about the nature of the gas phase by nondestructive tests.[21] Fluid inclusions give us practically the *only* data we have on the density of the ore fluids (Roedder, 1967a); hence, one of the most important aspects of the gas phase is its volume, as this is the major factor in determining the gross density of the original fluid that was trapped (pl. 12, figs. 3, 4). Ermakov (1950a) categorizes inclusions by their "state of aggregation," based on the manner in which they homogenize (that is, in the liquid phase, in a supercritical fluid phase, or in the gas phase), under the assumption that these states are the states *in which they were trapped.* All that can be definitely determined from most inclusions, however, is the density of the trapped fluid (after corrections for thermal expansion and compressibility of the host mineral) and the minimum temperature of trapping. Depending upon the pressure, all three of Ermakov's "homogenization types" could originate from trapping of supercritical fluids at the same temperature, but at different pressures, and hence different densities (fig. 4.).

Only rarely can the density be determined directly (Petrichenko and Shaydetskaya, 1968). Generally the determination of gross density is based upon the measurement of the volumes of all phases and their identifications as to composition and hence individual density. (Corrections must be made also for the solution of the inclusion walls upon heating to the trapping temperature.) Hence, the volume of the gas phase, with a density near zero, must be measured most accurately. Simple linear intercepts can be used with long tubular inclusions, and various planimetry methods can be used with flattened inclusions (pl. 3, fig. 2). Sketching the projected image of a flat inclusion on heavy paper, followed by cutting out and weighing the pieces of paper representing the phases, is simple, fast, and suprisingly accurate compared to the use of a planimeter. In thicker inclusions, the spherical bubbles can be measured more accurately than any of the other phase volumes, if the image size is not reduced by a curved upper inclusion wall acting as a negative lens (pl. 11, figs. 7 and 8). Unless care is taken, gross errors may also be introduced in the measurement of volume of a small sphere under the microscope due to the production of a falsely focused image associated with wide angle illumination (Saylor, 1965). Measurements of partly flattened or distorted bubbles yield only very crude and inconsistent estimates of phase volumes, and visual estimates can be very far off (fig. 3).

The nature of the gas bubble is shown mainly by destructive tests (see "Destructive methods of determining composition"), but two phenomena may give qualitative evidence of the presence of carbon dioxide. Hartley (1877a) first made the interesting observation that during heating of some inclusions, the "vapor" bubbles evidently achieve a density greater than that of the "liquid" and sink in it. Recent work on the system CO_2–H_2O by Tödheide and Franck (1963) and by Takenouchi and Kennedy (1964) has shown that there is indeed a density inversion of "liquid" and "gas" phases in this system. Such density inversions may thus be used to obtain compositional data.

Roedder (1963b, p. 195) showed that at low temperatures reaction of invisible CO_2 in the gas bubble with water in the surrounding liquid may yield crystals of the clathrate compound carbon dioxide hydrate ($CO_2 \cdot 5\frac{3}{4}\, H_2O$) that are almost invisible. Data on the system H_2O–CO_2–NaCl (Takenouchi and Kennedy, 1965b) may permit the estimation of CO_2 pressures in the gas bubble from the temperature of dissociation of such crystals. Carbon dioxide is the most likely compound to form clathrate crystals in cooled natural inclusions, but some ambiguity remains in the identification, in that many other compounds, both organic and inorganic, form similar clathrates. (See p. JJ23.)

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[21] A novel test, which is applicable only in rare instances, was used by O'Keefe, Dunning, and Lohman (1962) and O'Keefe, Walter, and Wood (1964) to determine the presence of Ne, He, H, and O in the low-pressure gas in large ($\sim$1 cm^3) gas bubbles in tektites. A spectrograph was used to analyze the light produced by an electrodeless gas discharge in the unopened inclusions.

Although of no direct compositional significance, the existence of pressure in the gas bubbles may be determined in a few cases by strain birefringence in the surrounding mineral. This was first described by Brewster (1820) around gas bubbles in amber. Although strain birefringence is common around solid inclusions in minerals (see pl. 6, fig. 3), most crystals apparently require such high pressure to yield visible birefringence that fluid inclusions rarely cause it.

EQUIPMENT FOR HEATING AND COOLING INCLUSIONS

Surprising as it may seem, the development of suitable equipment for heating (or cooling) inclusions, particularly while under the microscope, has been a major hurdle to the progress of inclusion research, and a generally optimum design for such work has not been found. It is very difficult to achieve an adequately high, known, and controllable temperature in the sample under such conditions that high magnification and adequate illumination may be used and yet permit flexibility and speed in operation. Illumination is a particular problem, as the distances involved in such equipment preclude the use of the high-power condenser that is ordinarily so helpful in microscopy of inclusions. A special movable light source has been used by the author (Roedder, 1962a, p. 1056–1057), and Bazarov (1965b) suggests long-focus condensers. Illumination via flexible (and hence adjustable) fiber optics has proven to be the most generally adaptable system in the author's laboratory.

There are many designs of microscope hot stages in the literature, starting with Phillips (1875) who used a simple paraffin bath. Most of the designs have good to excellent precision, but poor accuracy. Ermakov (1944a; 1950a, p. 86 in translation) describes an electrically heated microscope stage, using air as the heat exchange medium, that operates to 650°C. The considerable amount of data that Ermakov (1950a, table 36, p. 249 and elsewhere, in translation) presents on the continuous formation and unidirectional streaming of gas bubbles in some inclusions at high temperatures apparently has not been recognized as being evidence of rather severe, static thermal gradients within the samples on this stage. Kalyuzhnyi (1958b) found that all temperatures determined with the "air-heated thermochambers of the old design" (presumably Ermakov's) were 30° to 60°C too low, and he describes an improved chamber, good to 700°C, using heat-conducting plates to move the heat from the windings to the sample. Bailey (1949), Meyer (1950), Bailey and Cameron (1951), Skinner (1953), Lemmlein (1953), Little (1955), Loskutov (1955), Pomîrleanu (1959), Kormushin (1960), Nadeau (1967), Miller (1968), Pashkov, Timofeev, and Kotov (1968), Bazarov (1968c), Groshenko (1968), Ohmoto (1968a), and Ohmoto and Rye (1970) describe other models. The unit described by Meyer (1950) is particularly novel in that it makes use of vertical illumination, with the sample plate sitting on a heated metal mirror.

Large, static thermal gradients present the most obvious source of trouble in air-conduction heating stages. These are to be expected in any sample that is heated only from the sides but can radiate heat both upward and downward through a large angle of view. Such errors can become much larger at higher temperatures (Bloss, 1964). To avoid at least some of these troubles, Richter and Abell (1953) designed an improved high temperature stage, operating up to 700°C, by surrounding the sample, above and below, with flat heating elements, each with a sufficient gap to permit viewing in transmitted light. The major uncertainties with such equipment are probably in the measurement of the sample temperature. This measurement must be done in such a way that either the value is correct (that is, it is the actual temperature of that part of the sample under observation) or provision must be made for calibration runs, permitting a suitable correction to be applied to cover gradients in sample and thermocouple (for example, Kirkham, 1969) and other sources of error. As both of these procedures are difficult at best, Roedder (1962a) designed a heating stage in which the sample is immersed in rapidly circulating thermostated silicone oil as the heat exchange medium. Although the results are precise and probably also accurate, the procedure is relatively slow, and is limited by the nature of the fluids to approximately 250°C.

Recently several heating stages for use at high temperatures have been described. Barrabé and Deicha (1956, 1957) and Barrabé, Collomb, and Deicha (1957, 1959) heated minute polished spheres, cut from samples of quartz, to study the behavior of glass inclusions. Other hot stages have been described for use up to 1100°C (Brock, 1962) and 1200°C (Kalyuzhnyi, 1960). Dolgov and Bazarov (1965) described a 1,600° stage with which a series of high temperature inclusion studies were made (Kerkis and Kostyuk, 1963; Vartanova and Shcherbakova, 1964; Bazarova, 1965; Valyashko and Kogarko, 1966; Sobolev and others, 1967; Bulgakova and others, 1967; Bazarova and Dmitriyev, 1967; Bakumenko and others, 1969; Bulgakova, 1969; and

Naumov, 1969). A special microscope furnace using a molybdenum sheet as the heating element, *in vacuo*, has been described by Kalyuzhnyi (1965b) for use on glass inclusions, in the range 600° to 1,600°C.

An ingenious microscope heating and crushing stage for the study of inclusions in samples under external hydrostatic pressure has been designed by Ypma (1965). Temperatures up to 250°C may be obtained, with external pressures, controlled by a pressure balance, up to 500 kg/cm^2. Although the heavy glass windows preclude the use of high magnification, the equipment should permit the measurement of filling temperatures on inclusions in minerals that normally decrepitate before homogenization. The most important design feature, however, is that the sample can be crushed while under observation, and the behavior of the gas bubble can be noted, thus permitting evaluation of the vapor pressure of the fluids, and the identification of certain gas constituents. (See p. JJ32–JJ33.)

Entirely different procedures are generally used for heating samples for decrepitation studies (also a destructive test). In these the sample (usually coarsely crushed) is heated gradually and the temperature range of decrepitation noted. This decrepitation may be detected by use of a stethoscope to hear the sounds (Scott, 1948; Smith and Peach, 1949; Ermakov, 1950a, p. 127 in translation), by visually observing the movement of grains (Ermakov, 1950c), or through the use of a microphone, amplifier, and various integrating and recording equipment (Smith and Peach, 1949; Peach, 1949a; Konta, 1951; Dolgov and Raikher, 1953; Kostyleva and Labuntsov, 1958; Zakharchenko and others, 1958; Trufanov and Rodzyanko, 1963; Tugarinov and others, 1963; Malyshev and others, 1964; A. N. Ermakov and Nefedov, 1968; Kostyleva, 1968; Mel'nikov and Ryabov, 1968; and Myaz' and Simkiv, 1968). A special design for use at low temperatures, for example with saline samples, is given by Montoriol-Pous (1967).

Cooling inclusions under the microscope involves a surprising number of seemingly minor but experimentally very troublesome details. The historical development of this method is given by Roedder (1962a), who describes a new cooling stage in which the sample is immersed in rapidly circulating thermostated acetone as the heat exchange medium. Several other cooling procedures have been described in the literature (Kern and Mattern, 1963; Mel'nikov, 1965; Velchev and Mel'nikov, 1965; Myaz' and Simkiv, 1965; Kormushin, 1965; Bazarov, 1966b; and Poty, 1968b). Sawkins (1966a) described a cooling stage based on the use of several thermoelectric (Peltier) cooling units; it will cool to about −18°C and can be controlled to ±1°.

The thermal mass of the 7 liters of acetone in the equipment described by Roedder (1962a) is so large that temperature changes cannot be made rapidly. To permit faster operation over a greater range, a stage is being built that will be cooled by rapidly-moving, single-pass nitrogen gas, cooled by liquid nitrogen, using some features of the cold stages described by Rhodes (1950) and by Monier and Hocart (1950).

One problem inherent in all freezing studies using circulating refrigerated acetone is the possibility that cracking of the host mineral will admit a small amount of acetone which will lower the apparent freezing temperature drastically. Such cracking is most likely in those samples having inclusions with very small vapor bubbles, that are hence subjected to high internal pressures due to expansion on freezing. Valid freezing data on such samples can be obtained by the use of a miniature double-windowed cell, filled with oil, inside the regular cell of the cooling stage (Roedder, 1968d, p. 441). As the oil is immiscible with the inclusion fluid, this technique permits reasonably accurate determinations even on inclusions that do fracture on freezing. The high viscosity of most oils at low temperature precludes using them directly as the circulating fluid.

These cooling devices are generally rather complex in design and operation. A much simpler, very inexpensive cooling procedure, that gives good results over a wide temperature range, at the expense of a little more time, is described by Roedder (1962a, p. 1051). The operator can readily maintain two or three such units, at separate and easily varied temperatures. With a little practice, temperature can be held constant to ±0.5°C by the periodic addition of small lumps of solid CO_2. Simple insulated containers, made of nested pairs of glass beakers with glass wool between, are adequate for the cold baths.

DESTRUCTIVE METHODS OF DETERMINING COMPOSITION

QUALITATIVE AND SEMIQUANTITATIVE METHODS

SOLIDS

If a solid daughter mineral can be removed from its inclusion it can be identified by various normal petrographic and chemical techniques, as well as by single crystal X-ray and electron probe procedures. Brewster (1823b) presented the first account of the identification of loose daughter (?) crystals extracted from a fluid inclusion in quartz. These were identified as calcite from their high birefringence and their solubility, with effervescence, in dilute nitric acid. Minute (~0.002 mm) single daughter crystals are very difficult to find in the crushed debris even when they are strongly birefringent and the host is isotropic (Roedder, 1963b, p. 175). When solid included grains are abundant or large, they can be recognized and their indices can be obtained, for example, petalite in aquamarine (Eppler, 1962b) and magnesian calcite in quartz (Zakharchenko, 1955). Elaborate and detailed studies of daughter minerals in opened inclusions have been reported by Kalyuzhnyi (1958a, 1960, 1961). A tungsten carbide drill (advanced by the microscope fine focus screw) was used to drill two holes into large inclusions in pegmatitic topaz in a humidified environment, after nondestructive optical tests were complete. The index of refraction of the solution was measured, and the indices of the daughter minerals were obtained by inserting index liquids directly into the drained inclusions. Water and other reagents were added in sequence to determine solubilities and to obtain solutions on which various microchemical and spectrographic tests were performed. The solid crystals which Kalyuzhnyi recognized included halite, sylvite, an unnamed new chloride of zinc and aluminum, elpasolite (K_2NaAlF_6), teepleite ($Na_2B_2O_4 \cdot 2NaCl \cdot 4H_2O$), cryolite ($Na_3AlF_6$), caracolite (approximately $PbOHCl \cdot Na_2SO_4$), quartz, and a series of other unidentified phases on which only partial data could be obtained. The new chloride of zinc and aluminum is particularly interesting in that it is present in small amounts (1 to 2 volume percent) in almost all the inclusions studied. Its properties are as follows: apparently "rhombic" (orthorhombic?), showing prism, two pyramids, and probably the pinacoid, $\gamma=1.599\pm0.001$, $\alpha=1.585\pm0.002$, $+2V=50°$, colorless, $H=1$–2, soluable in water with light grayish residue, soluble in concentrated H_2SO_4 with effervescence.

Infrared absorption has proven very useful in characterizing the various bitumens found in the abundant hydrocarbon inclusions in the Khibiny alkalic massif in the U.S.S.R. (Ikorskii, 1967b, 1968).

LIQUIDS

Sorby (1858, p. 470) was the first to prove without doubt that the liquid in common inclusions was water; he decrepitated inclusions in a closed-end tube, condensed the water vapor with low temperatures and determined the crystal form and melting point of the resulting ice crystals. He also noted (p. 471) in this same experiment that another substance was given off, which condensed at a higher temperature. This he found to be KCl or NaCl. Since then there have been many determinations of the water content of fluid inclusions. The quantity of liquid in inclusions can be estimated from weight loss determinations (Ermakov and Myaz', 1957) or from density measurements, as seen in figure 2. (See also Roedder and others, 1963, p. 355.) Many papers have been published in recent years in which the amount of inclusion fluid released during the crushing and leaching of a given sample for analysis is estimated from a count of inclusions in a representative small sample (for example, Zakharchenko, 1950; Vul'chin, 1953; and Ermakov and Myaz', 1957). From this value and the chemical analysis of the leachate, "concentrations" in the inclusion fluid are then calculated. In addition to the problems of multiplicity of origin and uniformity of distribution of small inclusions throughout the sample, the volume of a single inclusion 1 mm in diameter is equal to 10^6 inclusions of 0.01 mm diameter (fig. 1), so the actual volume, composition, and concentration of the inclusion fluid in any given sample may be determined almost completely by a small number of erratically distributed larger inclusions.

Some of the earliest studies on the composition of inclusions made use of simple but effective qualitative tests involving evaporation of the inclusion fluids. Thus Nichol (1828) noted that the evaporation of inclusion fluids yielded new crystals, but that the mass stayed wet for days, indicating the presence of a deliquescent substance.[22] Newhouse (1932) noted similar deliquescence in inclusions from Mississippi Valley-type deposits. Schertel (1878) noted the presence of colorless cubes on evaporation of the leach from inclusions in a "Spanish sphalerite"; these were proven to be NaCl by the spectrum lines of sodium and a strongly positive test for chloride ion with $AgNO_3$. Newhouse (1932) made use of the formation of cubes of NaCl from evaporating inclusion fluids on broken mineral surfaces to obtain a crude but good estimate of the salinity of the fluids; he evaporated similar droplets of known salinity to make the test semiquantitative.

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[22] There is one elegantly simple but notably qualitative test that has only rarely been reported in the literature (Buerger, 1932a; Newhouse, 1932), yet its results present data on the chemical characterization of the ore-forming fluids that are vivid and memorable. This is the *taste* test. The writer has found that large (~1.0 mm) primary fluid inclusions in sphalerite from Creede, Colo., which were accidentally exposed on a new cleavage surface, had a salty but slightly astringent or bitter taste corresponding to a solution rich in NaCl, with small but definite amounts of $MgCl_2$ and (or) $CaCl_2$; other similar open inclusions stayed wet for days, verifying the presence of hygroscopic salts.

Zirkel (1870b) described another elegantly simple but extremely sensitive test for sodium in fluid inclusions. When a sliver of the mineral decrepitates in a flame, the sudden release of sodium-laden steam makes a tiny yellow flash.

In many investigations, qualitative or semiquantitative spectrographic measurements are reported on the solids obtained by leaching crushed samples (for example Vlasenko, 1957; Karyakin, 1958). The elements found are usually Na, K, Ca, Mg, and traces of Si, Al, Ti, Fe, etc. Qualitative tests for the major anions are also usually made and show major Cl^{-1}, with lesser amounts of CO_3^{-2}, SO_4^{-2}, or HCO_3^{-1}. Zakharchenko (1950) and Skropyshev (1957) have analyzed very large inclusions (up to 1.5 cm^3) for six or seven elements by semiquantitative spectrographic techniques, by simply soaking the porous carbon electrodes in the fluid. Several investigators have used semiquantitative spectrographic analyses of samples both with and "without" inclusions to obtain data on the inclusions (for example, Grushkin and Prikhid'ko, 1952; Skropyshev, 1957). Less commonly, hot hydrochloric acid has been used as a leach solution (for example, Saitô, 1951; Shchiritsya, 1960). Unless very considerable efforts are made to avoid even microgram amounts of solid impurities in the sample to be crushed, it is difficult to interpret the trace element analyses made on such leaches, as well as analyses for such materials as ferric iron. Some authors report crushing samples directly in test reagents for qualitative determinations (Machairas, 1963a; Saitô, 1951).

Some of the data reported by Ermakov (1950a) were obtained by ignition at 500° to 600°C to determine H_2O and CO_2 from weight loss, followed by pulverization and extraction with a water leach. The quantity of salts was obtained by the difference in weight between the ignited and the extracted samples.

Huntley (1955) reported on a novel test for the presence of radioactive elements, using a photographic (nuclear) emulsion to detect alpha activity in the inclusions in a polished quartz microscope slide. Unfortunately, the highest activity he records, 42 alpha tracks in 6 months, came from an inclusion that had been opened to the surface during the slide polishing operation,[23] but he does report some tracks from unopened inclusions. Picciotto (1950) suggested that concentrations of U and Th visible in autoradiographs of rocks might represent fluid inclusions, but solid mineral inclusions seem more likely for most such concentrations of radioactivity.

Loskutov (1962) showed that the water present in inclusions in natrolite could move through the open structure of this zeolite and evaporate during months of museum storage, leaving behind crystals of several salts. Thus he records crystals of hydrous sodium ammonium(?) carbonate, nahcolite ($NaHCO_3$), acid phosphate of sodium and ammonium (stercorite?), dehydrated sodium carbonate(?), and sodium phosphate(?). Bergman and Blankenburg (1964) report the identification of the NaCl cubes and solutions in inclusions in Brazilian quartz crystal by X-ray diffraction studies of the recrystallized NaCl obtained by evaporation of a water leach, and Lamar and Shrode (1953) used X-ray diffraction to identify calcium sulfate, calcium carbonate, magnesium sulfate, NaCl, KCl, $MgCl_2$, and basic magnesium carbonate in the solids from evaporation of leachates from ball-milled limestones and dolomites.

The microscope pressure stage of Ypma (1965) permits the determination of the vapor pressure of the fluid in inclusions, at various temperatures, and hence an independent evalution of the gross salinity. By crushing a series of similar inclusions in a sample at a given temperature but varying external pressure, the pressure at which the vapor bubble neither expands nor contracts on release can be determined.

GASES

Many different qualitative tests can be used to determine something about the gases in inclusions. The pressure can be estimated by the simple expedient of opening the inclusion while the sample is immersed in a fluid. This was first reported by Davy (1822) who drilled into inclusions in quartz samples that were under water, oil, or mercury, and found that upon breaking in, the bubble collapsed very markedly, and the external fluid was drawn in. This same simple test was used recently to determine the gas pressure in a large vesicle in a tektite (Rost, 1964). The vapor bubbles in many inclusions are almost solely water vapor, as they collapse instantaneously and completely when exposed to atmospheric pressure. Crushing can be done simply and quickly by pressing on the cover glass while watching the grain with the microscope. If the grain is large or hard, a piece of another slide can be used as a cover. Much more control is possible if a screw thread is used to apply the pressure, as in Deicha's original crushing stage (Deicha, 1950f; Roedder, 1970a).

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[23] The polishing agents used are not stated, but if commercial grade cerium oxide had been used, with its expected thorium activity, it would be very difficult to eliminate slight contamination in opened inclusions.

Davy (1822) also found some inclusions with gas at pressures greater than 1 atmosphere, as it expanded into the surrounding liquid when the drill broke in. Highly compressed gases, such as liquified CO_2 (at more than 70 atmospheres), will practically explode into the surrounding liquid and may even cause minor explosions when the rock is struck with a hammer (Hawes, 1881). The evolved bubbles will dissolve very rapidly if a fluid is chosen in which the gas is known to be soluble; even if the gas is not known to be "soluble" in the fluid chosen, the bubbles will dissolve with surprising rapidity. Thus a solubility of only 1 ppm will permit a 4 μm gas bubble to dissolve completely in the fluid surrounding it within the range of rapid diffusion—a 20 μm radius. The writer has observed that upon releasing some high pressure gas inclusions into fluids that are good solvents for the major gas present, the gas bubble formed is visible only as a black, circular "latent image" on the retina, as the bubble disappears almost instantly after its original expansion. Not infrequently, gas inclusions contain a mixture of gases; when these are released into a solvent for only one of them, there is a rapid shrinkage of the bubble to some intermediate radius as the soluble gas dissolves, followed by a slower rate of shrinkage representing the solution of the less soluble gas. Quick measurements of the bubble diameter can thus be used for rough calculations of the relative volume percentages of the several gases present (Roedder, 1965d).

Chemical reactions of the evolved gas with the fluid in which the crushing is performed can be observed with the microscope. Thus Rasumny (1960) was able to identify the CO_2 in very small (5 μm) inclusions by reaction with a $Ba(OH)_2$ solution, and G. R. Helz was able to determine the presence of H_2S in inclusions by reaction of the gas with an anhydrous glycerol solution of soduim nitroprusside. A green precipitate formed in 1 minute from a 100 μm bubble containing 20 volume percent H_2S (G. R. Helz, written commun., 1964).

When an inclusion containing a liquified gas such as CO_2 is opened, the volume expansion is very large (pl. 11, fig. 5). Thus 1 volume of liquid CO_2 expands to approximately 350 volumes of gas. The specific volume expansion is strongly temperature dependent, but even a crude measure of it can be a useful parameter in phase identification. Such an estimate can be obtained by measurements, made with a vernier micrometer ocular, of the volume of liquified gas before and of the gas bubble or bubbles after crushing (Roedder, 1963b, p. 190). It is frequently difficult to obtain a fragment of the mineral for crushing that is sufficiently small that only a single inclusion is present. When released, the bubble may free itself from the crushed fragments and float to the top to rest against the glass cover. Kalyuzhnyi (1955a) has shown that all bubbles less than 0.4 mm in size are sufficiently close to spherical, even though slightly flattened against the glass, that they can be treated as spheres.

Although obviously only qualitative or at best semiquantitative, the crushing test is very useful, as it is exceedingly sensitive. As little as 10^{-14} g of a relatively nonsoluble, noncondensable gas (less than a billion molecules) may be detected in this manner, as it will form an easily visible gas bubble, several micrometers in diameter at atmospheric pressure. Roedder (1965d) gives some further experimental details on the procedure. Smaller amounts of gas than this cannot be recognized, as the increase in internal bubble pressure as the radius decreases, due to surface tension, causes such small bubbles to "blink out" almost instantly if they are less than a few micrometers in diameter.

Above 100°C, the water in inclusions will also vaporize when the inclusions are opened. Deicha (1952c) described an effective visual test for such gas evolution. He uses a "visual decrepitometer" with a transparent oil as the heating medium so that the tiny bubbles emitted can be seen.

A variety of methods have been used to measure the gas pressure in the numerous gas inclusions present in ice (Nutt, 1961). Of these, the least ambiguous involves opening the inclusions while immersed in a fluid under a controlled, externally applied hydrostatic pressure. Scholander and Nutt (1960) used a solvent for ice (glycerine) as the pressure medium and noted the pressure needed to just balance that in the inclusions at the moment that the slowly advancing solution front first intersected long tubular inclusions. With this method they found that the gas inclusions in Greenland icebergs were under pressures up to 20 atmospheres. A less precise method was introduced by Hamberg (1895), in which the density of the ice with inclusions and the volume of air released on melting were used to obtain the air pressure in the inclusions.

Ypma (1965; oral commun., 1965–68) has shown that several properties of the gas evolved on crushing may be determined with his heating-pressure stage. Thus CO_2, evolved from inclusions in a Brazilian quartz crystal (a part of this sample is illustrated in pl. 7, figs. 9, 10) could be liquefied and revaporized by appropriate adjustments of pressure or temperature. As the bubble gradually became smaller, through solution in the pressure medium (glycerol), the pressure needed for liquefication increased to more than 80 kg/cm^2, greater than the critical pressure of CO_2 (75.3 kg/cm^2). This is believed to be due to differential solution of the gas in the glycerol, presumably increasing the concentration of minor constituents. Similarly, by crushing a series of cogenetic inclusions, held at the temperature of homogenization, at a sequence of external pressures, the vapor pressure of the fluid can be determined. Since both pressure and temperature are then known, the concentration of salts in the fluid can be estimated.

Still another technique was described and used by Vogelsang and Geissler (1869). They decrepitated a sample in an evacuated tube containing electrodes; the presence of CO_2 was proved by the spectra observed upon excitation of the gas.

If strong smelling substances are present in inclusions, the odor on crushing can be used as a crude but effective test. Thus, very small quantities of volatile hydrocarbons in some inclusions (for example, the inclusions in fluorite from the southern Illinois deposits (frontispiece, upper left; pl. 9, figs. 1–3)) yield a strong odor of petroleum.

An exceedingly small amount of hydrogen sulfide may be detected by its odor. Assuming an average minimum level of detectability of 0.025 ppm (Patty, 1962), the release of $\sim 10^{-10}$ g of H_2S should be detectable with the nose. Wright (1881) detected an "unmistakable" odor of H_2S from inclusions in a pegmatitic quartz, but the volume of H_2S was too small to measure. Some limestones yield a fetid odor on scratching or crushing, but this odor is rather rare in well-crystallized minerals (table 1). Notable exceptions are the finding of liquid H_2S in coarse marbles of the Grenville Series (Harrington, 1905), in inclusions in a coarsely recrystallized marble adjacent to a lead-zinc deposit associated with the Bingham copper mine in Utah (pl. 7, fig. 11; Roedder and Creel, 1966), and in a Brazilian quartz crystal (pl. 9, figs. 5–9).

Dons (1956) noted that an odor of H_2S was evolved from a sedimentary barite crystal when it was rubbed, but not when it was decrepitated. The cause for this peculiar fact is not known, but it may lie in the disproportionation of polysulfide ion to form HS^{-1} and SO_4^{-2} at room temperature (Cloke, 1963). Sphalerite sometimes releases a slight odor of H_2S on breaking, but Hosking and Spry (1955) have shown that sphalerite is the one common sulfide that emits H_2S on being scratched with an iron knife. The odor on breaking sphalerite may thus be merely a reaction (unspecified) with the iron of the hammer.

Some fluorite that has been subjected to α-particle bombardment emits a strongly pungent but sweetish odor on crushing. For many years this has been attributed to free fluorine or to ozone liberated by its reaction with water (Becquerel and Moissan, 1890; Sine, 1925). Heinrich and Anderson (1965) report that the gases evolved on crushing a fetid fluorite-bearing carbonatite in a mass spectrometer contained a mixture of C_5 and C_6 hydrocarbons (possible fluorinated?), F_2, HF, and F_2O, and similarly Kranz (1965, 1966, 1967) found a variety of fluorinated hydrocarbons in gas inclusions in "Stinkspat" fluorite from Wölsendorf, Germany.

QUANTITATIVE METHODS

EXTRACTION AND ANALYSIS OF GASES

A variety of methods have been used to extract the contents of fluid inclusions for analysis. All have specific applications, but as the limitations and errors inherent in some of these techniques are large, the resulting data are not always comparable and must always be examined in light of the limitations of the methods used. Many of the analyses for gases from inclusions are reported in the literature as though the gases came from *gas* inclusions, but most of these probably came from the evaporation of liquids such as water or carbon dioxide, from gases dissolved in these liquids, or from various sources other than inclusions.

Khitarov, Rengarten, and Lebedeva (1958) measured the volume of water in large inclusions in calcite by evaporation in a stream of dry air, which was then passed through a weighed absorption tube. A mercury seal around the drill made it possible to open the inclusions directly in the air stream. Karpinskii (1880) proved the presence of high-pressure CO_2 in inclusions by crushing under mercury and absorbing the evolved gas in a barium hydroxide solution. Kalyuzhnyi (1955a) drilled into inclusions under dehydrated glycerol; the evolved bubbles of gas were trapped for measurement as they rose in the fluid. Maslova (1961) crushed samples in dehydrated glycerol and transferred the evolved gas bubbles for analysis by means of a piston ultramicropipet.

The gases (including water) in inclusions may be obtained by simple crushing in a stream of inert gas, followed by absorption and weighing as described by Pfaff (1871); by decrepitation in an inert gas stream followed by absorption (Khitarov and

Rengarten, 1956; Roedder, 1958, p. 263–266; and Rutherford, 1963), or by simple weight loss determinations upon heating. (Using this last method, Sorby (1858) found up to 0.4 weight percent water in quartz from Cornish granites.)[24] Kramer (1965) used a refinement of one of these techniques to determine the volume of water in inclusions in salt, prior to analysis of the materials in solution in the fluids. He weighed selected fragments on a microbalance, after drying to constant weight,[25] cracked open the inclusions and evaporated the water, and reweighed the fragments; the entire operation was done in a dry box. Mironova and Naumov (1967) convert the water evolved on decrepitation to hydrogen with calcium hydride and measure its volume.

Various vacuum techniques have also been applied to determine the water (and other gases) in inclusions. Wright (1881) decrepitated smoky pegmatitic quartz in a vacuum for gas analysis. Chamberlin (1908, p. 39) used a vacuum crushing device to prove that only a very small part of the gases he obtained by heating rock powders in a vacuum came from fluid inclusions. Thus he obtained 0.81 volumes of gas per volume of rock from a quartz sample by heating (his analysis, no. 71) but no measurable gas was evolved upon crushing a portion of the same material (p. 40). Barker (1965b) describes a much more effective vacuum crushing device. Elinson (1968b) gives a detailed description of a device for crushing in an inert gas stream and associated gas-handling equipment.

Wahler (1956) investigated the various sources of error in gas analyses from rocks and minerals, and he developed an elaborate technique for obtaining and analyzing the gases from selected large inclusions, which were evolved by decrepitation upon rapid heating. Entirely apart from the analytical problems such as losses by absorption on the walls of the apparatus, he found that there are many problems inherent in the use of heat to release the gases. For example, diffusion of hydrogen from burner gases through the apparatus walls was a serious source of contamination in some experiments reported in the literature. He also presents evidence, mainly from the literature, that much but not all of the CO, H_2, CH_4, O_2, and H_2S found by earlier workers by heating rocks and minerals came from various chemical or catalytic reactions of original H_2O and CO_2 (and also possibly organic matter) with each other and with the mineral surfaces present, yielding a new assemblage of gases. A cogent argument in this direction is given by the early work of Travers (1898), who compared the gases evolved upon heating various samples (H_2, CO, and CO_2), with the gases evolved upon solution in acids (CO_2 only). After extensive study of the similar problem of volcanic gas analyses, Shepherd (1938) concludes that it is impossible to relate conclusively the gases found by analysis of volcanic gas, or the gases from heated rocks and lavas, to those originally present. (See also Emmons, 1964.) Several studies have shown that the amounts of hydrogen (and carbon monoxide) in the evolved gases increase with increase in the temperature used to extract gases (for example, analyses 23a, 23b, and 64–69, table 2).

Kormushin (1962) and Dolgov (1965b) used decrepitation in vacuo to determine the composition and pressure in inclusions, and Kokubu, Mayeda, and Urey (1961) heated samples to 1,100°C in low pressure oxygen, and then pumped off the gases, to obtain water samples for mass-spectrometric determination of the deuterium/hydrogen (D/H) ratios.

Roedder, Ingram, and Hall (1963) describe another technique for opening specially selected samples, containing a milligram or more of water, by crushing under high vacuum. They place the samples to be crushed in a collapsible metal tube (copper or stainless steel) and squeeze the tube from the outside with a hydraulic press, thus breaking open the inclusions. This technique was also used by Hall and Friedman (1963). Suess (1951) crushed tektites containing gas vesicles in a sealed tube to determine their gas pressure; he measured the density of the samples before and after crushing to obtain the volume of vesicles. Somewhat similar procedures have been used to obtain the volume of the liquid phase in inclusions (Roedder and others, 1963, p. 389) and to obtain the pressure in gas inclusions in ice (Hamberg, 1895).

Vacuum ball milling has been used frequently to open inclusions for gas analyses. Elinson (1949) used a metal ball mill connected to a vacuum line but had considerable difficulty with the rotating seal. Elinson (1956) adopted a simpler technique of grinding in vacuo, with subsequent pumping off and collection of the evolved gases, and made a series of analyses with it (Elinson and Polykovskii, 1961a, 1961b, and 1963). Umova, Glebov, and Shibanov (1957 and 1960) used a similar technique. Although

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[24] Some samples present evidence of natural decrepitation by heating (Deicha, 1961). This may provide useful information on the age relations of dikes and ore mineralization (Lokerman, 1962, 1965; Ermakov and Kholmskii, 1965). Similar reasoning holds for the emptying of inclusions due to cataclasis (Ypma, 1963).

[25] Although the hot plate used was at 125°C (Kramer, 1965, p. 939), the sample temperatures were probably <80°C (written commun. 1965).

not always stated, aparently steel grinding balls were used in all of these studies.

Goguel (1963, 1964) showed that the large quantity of nitrogen found in most of the analyses of gases from ball milling was a contaminant, evolved from the steel grinding balls as they were abraded in the mill. Nitrogen is present in igneous rocks mainly in the form of ammonium ion (Wlotzka, 1961; Stevenson, 1962). It is not known how this will behave on ball milling, but Kranz (1968a) obtained many different nitrogen compounds, including ammonia, amines, and nitriles on analysis of the gases released on vacuum crushing feldspar. Goguel also showed that on grinding carbonate minerals containing no inclusions, considerable amounts of CO_2 may be formed, particularly if silica is present. To minimize or avoid these difficulties, he developed a vacuum microball-milling technique, using a silica glass mill and tungsten carbide balls on $<$1-g samples of $<$2-mm-grain size; this was followed by gas analysis procedures similar to those described by Wahler (1956). In ball milling, absorption of gases on the surfaces of samples, apparatus, and reagents can be an important source of error. Thus Kunkel (1950) found that well-cleaned quartz surfaces normally have one or two layers of water molecules that are not released by heating under 500°C, and absorption of water on olivine and pyroxene is even less reversible (Nelson and Vey, 1968). Even inert gases such as nitrogen and argon are absorbed and held tenaciously (Khodakov, 1966). This absorption can occur almost instantaneously. Ware and Pirooz (1967) have shown that on breaking open gas bubbles in glass in vacuum, absorption on the new surface causes significant losses of CO_2, SO_2, and H_2O.

Goguel (1963) also presents good evidence that much of the hydrogen and helium present in many minerals is lost by diffusion in 1 year at room temperature, once the mineral is crushed. (See also Kovalishin, 1968.) Conversely, diffusion into sample surfaces prior to analysis can contaminate samples with gases (for example, Reynolds, 1960).

Still other methods of extraction and analysis of gas inclusions, appropriate to certain samples only, involve complete fusion or solution. Hoy, Foose, and O'Neill (1962) fused a salt sample containing gas inclusions to obtain a gas sample for analysis. Analysis for argon usually involves fusing of the samples, either with or without a flux, in a vacuum system (Lippolt and Gentner, 1963; Rama and others, 1965). The water-insoluble gases present in salt samples are very easily extracted by dissolving the sample in water (for example, the popping salt of Wieliczka, Poland—see Dumas (1830) and Rose (1839)). Meteorites and rocks have also been dissolved in acids to obtain the gases from them, although much of this gas may not be present as discrete inclusions.

In addition to the serious problems of extraction of gases detailed above, the analytical procedures for use on small amounts of gas mixtures are notoriously poor. Absorption by solid or liquid reagents, combined with barometry or volumetry, provides the basis of many of the analytical procedures, but it is seldom truly specific. Khitarov and Vovk (1963) and Khitarov (1965c) report using an electrical method for carbon dioxide evolved from inclusions; they measure the change in the electrical conductivity of a $Ba(OH)_2$ solution as the CO_2 is absorbed and $BaCO_3$ is precipitated. The method is applicable in the submilligram range.

Dolgov and Shugurova (1966a) claim to be able to extract and analyze for nine constituents (CO_2, NO, O_2, CO, H_2, CH_4, total hydrocarbons, N_2 plus rare gases, and sum of SO_2, NH_3, Cl, H_2S and F) by absorption techniques on gases from inclusions as small as 10 μm or even 1 μm. Assuming liquified gas at a density of 1 g/cm^3, this would correspond to a total sample of only 10^{-9} or 10^{-12} g. The specific methods used were first given in a reference that is not available to the writer (Dolgov and Shugurova, 1965), but Prof. Dolgov indicates (written commun., 1967) that a later paper by the same authors (Dolgov and Shugurova, 1966b) covers essentially the same material. It presents experimental data on the analysis of bubbles of gas mixtures in castor oil or anhydrous glycerol, using absorbers such as KOH and $Cd(C_2H_3O_2)_2 \cdot 2H_2O$. The bubbles analyzed, however, were much larger than those mentioned by Dolgov and Shugurova (1966a), in that they ranged from 1.0 to 0.3 mm in diameter, corresponding to approximately 10^{-6} to 10^{-8} g gas. (See also Shugurova (1968) and Sobolev and others (1970).) Dolgov and Shugurova (1968) present a large number of gas analyses on a variety of minerals, and Dolgov (1968b) reports very appreciable fractionation of gases on gradual release from a punctured inclusion.

Wahler (1956) gives rather complete experimental details on the method he developed for analysis of inclusion gases. His method for gas mixtures, based essentially on fractionation by freezing out various constituents at specific temperatures (plus other manipulations), with pressure measurements before and after each step, can yield analyses accurate to 1 to 2 percent on as little as 1 mm^3 of gas.

The mass spectrometer is particularly suitable for the analysis of inclusion gases. Thus Herzog, Eskew, and Erwin (1962) report the analysis of as little as 10^{-14} cm^3 (approximately 10^{-17} g) of noble gases by the procedure. Mass spectrometry has been used very successfully in the analysis of the gases present in very small bubbles in glass containing only a fraction of a microliter of a gas mixture (Todd, 1956; Wosinski and Kearney, 1966), but only recently has much been done with mass spectrometry of fluid inclusions. In spite of occasional ambiguity due to overlapping masses (for example, CO and N_2; also NO_2 and CO_2 (Craig and Keeling, 1963)), the method is powerful and can be expected to be applied to many inclusion problems. It is particularly useful for obtaining the ratios of gases, for example H_2O and CO_2 (Touray, 1968). Preisinger and Huber (1964; see also Arming and Preisinger (1968)) heated very tiny (0.1 mm) grains of feldspar, from several parts of zoned crystals, in an ultra high vacuum cycloidal mass spectrometer and determined large differences in the contents of H_2, CH_4, N_2, CO, CO_2, and Ar, between the core and rim. The analyses were made on a total volume of 10^{-8} to 10^{-9} cm^3 of gas, as they report finding 0.8 to 3.3 mm^3/g mineral. Ohmoto (1968b) reports extensive studies of the gases in inclusions from the Bluebell mine in British Columbia, made by mass spectrometer on samples released by crushing or decrepitation. He analyzed for H_2O, CO_2, H_2, CH_4, N_2, CO, C_2H_6, and C_3H_8. He was unable to detect H_2S. Bratus', Stasyuk, and Panchishin (1968) used an omegatron mass spectrometer and photographic recording to analyze the gases from individual inclusions in the range 10^{-6} to 10^{-9} g from pegmatitic quartz. Six constituents, H_2, CH_4, H_2O, N_2, Ar, and CO_2 were determined.

C. G. Barker (1965a, 1965b, 1966) used both heating to 400°C and crushing in vacuo to release the gases for mass analysis. By the use of duplicate samples of three fluorites (1965b) he found that all of the CO and most of the hydrogen and methane released upon heating to 400°C were apparently formed by reaction during the extraction process. (See analyses 64–69, table 2.) The mass spectra of the hydrocarbon gases evolved on crushing also differed greatly from those evolved on heating to 400°C (Barker, 1966). Chaigneau (1967b) also reports considerable differences in the gases emitted from quartz on heating or crushing in a mass spectrometer.

Karasev (1958) found evolution of gas and emission of electrons when he broke quartz crystals in a high vacuum, but he got neither on breaking glass or fused quartz. Vanderslice and Whetten (1962) cleaved natural halite from Baden, and synthetic melt-grown crystals, in a mass spectrometer under high vacuum (10^{-9} mm). They obtained bursts of gas, mainly water, which they attribute to very small amounts of individual water molecules in the structure, rather than fluid inclusions. Ryan, Grossman, and Hansen (1968) got similar bursts of gas on cleaving silicates in ultrahigh vacuum, but they reported evidence that the gases were released from the walls of the chamber and the pump, rather than from the samples. Mercer (1967; see also Goldsztaub and others (1966)) found much larger amounts of gas (10^{13} to 10^{14} $molecule/cm^2$) were released on cleaving muscovite in an ultrahigh vacuum. The gas was mainly nitrogen, with minor hydrogen, which he believes was trapped between the silicate layers. Heinrich and Anderson (1965) analyzed the gases evolved from crushed samples of a fetid carbonatite in a time-of-flight mass spectrometer and verified the presence of fluorine and several fluorine compounds.

Kranz (1965, 1966, 1967, 1968b) reported the presence of a variety of fluorinated hydrocarbons in the gases released on crushing uranium-bearing fluorite in vacuum, and in another paper (1968a) he gave full details on the extraction and analysis procedures. The procedure consisted of inclusion release by vacuum crushing, fractionation by freezing and then vaporization at −195, −78, and +20°C and by gas chromatograph followed by a pressure-reduction system, and finally analysis by rapid sweep mass spectrometry. This permitted identification and quantitative or semiquantitative analysis of many hydrocarbon, fluorine, and nitrogen compounds, as well as the normal inorganic gases, in a series of fluorite and feldspar samples.

Great care should be used in the interpretation of these mass spectrometric analyses. In particular, it is easy to let the extreme sensitivity blind one to the gross inaccuracies that can creep into such results. In the author's opinion, the most serious limitation on all such mass spectrometric gas analyses is that of gas fractionation by absorption (and reaction) on the various surfaces exposed. Such problems become acute in the sample size range involved in inclusion studies.

Deuterium/hydrogen ratios have been measured on water from many inclusion samples. Thus Kokubu, Mayeda, and Urey (1961) measured the water which filled amygdaloidal cavities in basalts, and 15 inclusion samples, mainly from various ore deposits, were measured by Roedder, Ingram, and

Hall (1963), using the vacuum extraction method described above. On crushing in vacuo, the evaporated water and CO_2 are condensed in a cold trap at $-196°C$; the pressure of noncondensable gases is measured; the water is converted to hydrogen by reaction with hot uranium metal, and it is separated from the CO_2 by another cold trap. After volume measurement, the hydrogen is measured isotopically by mass spectrometry. Hall and Friedman (1963) report 33 additional measurements, all on Mississippi Valley-type deposits. Rye (1964, 1965) reports D/H ratios for the inclusion waters in a series of samples from Providencia, Mexico. He also has examined the C^{12}/C^{13} and O^{16}/O^{18} ratios in various generations of calcite from this deposit. Eventually, similar measurements on the carbon and oxygen in the fluid inclusions in such samples also may be made, but it is not known how seriously any original isotopic fractionation will be affected by exchange reactions after trapping.

There are several reports of other uses of mass spectrometry on fluid inclusions. Damon and Kulp (1958, p. 449), in discussing the sources for the excess helium and argon they found in beryl crystals (that is, helium and argon in excess of that from radioactive decay since crystallization), state that at least some of it probably is present in the form of fluid inclusions. Undoubtedly some is also present in the relatively large channels in the structure, along with very appreciable quantities of water (as much as 3 percent (Feklichev, 1963)). Lippolt and Gentner (1963) found excess argon in hydrothermal fluorite, presumably from fluid inclusions. Rama, Hart, and Roedder (1965) report large amounts of excess argon in the fluid inclusions of a metamorphic quartz vein, and they used isotope dilution methods to determine the very low total potassium content (sum of that in the inclusions and that in the mineral structure) of the sample. The data presented would give a calculated potassium-argon "age" for the formation of the vein that is too old by a factor of 1,000 (roughly 250 billion years rather than 250 million years). Funkhouser, Naughton, and Barnes (1965) and Funkhouser and Naughton (1968) report similar excessive argon "ages" for ultramafic xenoliths from Hawaii, which they believe result from argon in the fluid inclusions that commonly are present is such rocks (Roedder, 1965d). Zimmermann (1966) presents a very interesting report on the mass spectrometric analysis of the gases given off of quartz samples, with and without inclusions, on heating in vacuum to 200°, 400°, 600°, 800°, 1,000°, and 1,200°C. Touray and Lantelme (1966) report a somewhat similar series of mass spectrometric analyses of gases given off by samples of quartz, fluorite, and dolomite from Tunis, after heating through 50-degree increments from 100° to 550°C.

Gas chromatography is a potentially valuable method for inclusion analysis, and it may be applicable even to relatively nonvolatile components (Giddings and others, 1968). Although it has been used successfully in the determination of $<10^{-5}$ cm^3 (about 10^{-8} g) each of several gases from bubbles in glass (Bryan and Neerman, 1962; Helzel, 1969), and has been widely used in the analysis of a variety of natural hydrocarbon gases, it has seldom been used on inclusion gases. More recently Clarke and Cable (1967), using two absorption columns (SiO_2 gel and 5 A molecular sieve), report a minimum detection range of 10^{-7} to 10^{-8} cm^3. Jeffery and Kipping (1963) used gas chromatography to determine the gases released on fusion of rocks, with a flux, in a stream of carrier gas. They determined H, He, O, N, and Ar and found that the amount of H obtained was less than that which would be formed by reaction of evolved water with the amounts of ferrous iron known to be present. Apparently the first application of gas chromatography to the study of actual inclusion gases was by Ackermann, Schrader, and Hoffman (1964), who studied "popping salt" from the Werra river district in East Germany.

The latest application of gas chromatography has been to the organic gases evolved from fluid inclusions in the alkalic rocks of the Ilímaussaq intrusion in Greenland (Petersil'e and Sørensen, 1970). In this work 300 to 350 g of rock was crushed in a vacuum system and about 0.5 cm^3 of evolved gas was then analyzed by gas chromatography for up to 12 constituents (Prof. H. Sørensen, written commun., 1969). The method is powerful, and it should be applied to many other types of inclusions.

EXTRACTION AND ANALYSIS OF LIQUIDS

Large inclusions present few problems, either on extraction or on analysis. A hole can be drilled, and the fluid pipetted out for analysis (Davy, 1822; Sjögren, 1893).[26] Maslova (1961, 1963) used a piston micropipette on large inclusions. Holser

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[26] "Synthetic" inclusions of large size can be made by drilling holes in the seed crystals used in quartz crystal synthesis (Safronov, 1958). Khetchikov, Balitskii, and Gasparyants (1966) have made chemical analyses of such large "synthetic" inclusions (8 mm^3), as well as the normal small primary inclusions that form in synthetic quartz (using a crushing and leaching technique), for comparison with the original fluid used in the autoclave. The differences reported are probably assignable to the errors inherent in the methods used for volume measurement and microanalysis.

(1963) used a special filter-tipped micropipette to exclude solid particles produced by drilling. Commonly the volume of the inclusion is obtained by refilling the inclusion cavity with a measured amount of mercury or other fluid. Even larger "fluid inclusions," in the form of vug fillings in mines, have been found and occasionally parts have been set aside for analysis (Ingerson, 1947, p. 378–379; Noble, 1950, p. 235; Roedder, 1963b, p. 183; White, Hem, and Waring, 1963). These large vugs are not truly sealed, as they have polycrystalline walls, but the fact that they sometimes have compositions considerably alien to the current local ground or mine waters in the area proves that the rate of leakage has been low.

For the opening of larger numbers of small inclusions, simple crushing, followed by leaching with water of other fluids, is the most common procedure, and more data have been obtained by this method than any other. The optimum procedure would dissolve all the materials from the opened inclusions, including both easily soluble and relatively insoluble daughter minerals, but would dissolve nothing from the host mineral or contaminants. As there is no such procedure, compromises are needed. Relatively insoluble host minerals such as quartz may simply be crushed and leached with water, particularly if the volume of the inclusions extracted is large enough to mask contamination. Sorby (1858) was one of the first to use this method; large samples were crushed in an agate mortar by Zakharchenko (1950) and Skropyshev (1957). Maslova (1958) used a micromanipulator to aid in the crushing and leaching of selected small grains. Kramer (1965) cracked open small single inclusions in weighed samples of salt, determined water by weight loss on evaporation (see previous section), and then dissolved the entire sample with a small amount of water. Corrections were made for the host NaCl dissolved. Similar but much smaller corrections can be applied to leaches made of less soluble minerals such as fluorite (Roedder and others, 1963, p. 360–361; Hall and Friedman, 1963, p. 894; Zakharchenko and Moskalyuk, 1968), but it is difficult to make the crushing and leaching procedure so uniform that the correction for dissolved host mineral is reproducible. Feldspars may release large quantities of alkalies (Govorov and others, 1968). An additional problem is presented by the inherent difference between the correction for host mineral solution to be expected on first contact of a freshly crushed mineral and subsequent leaches. The first leach might be expected to show a higher host mineral solubility due to the presence of very minute or strained particles and fresh surfaces of high energy configuration (points, edges, surfaces with nonrational indices, etc.).

Other workers have opened inclusions, prior to leaching, by decrepitation in a gas stream (Roedder, 1958, p. 264); decrepitation in a vacuum system (Wahler, 1956); crushing in vacuo (Roedder and others, 1963; Hall and Friedman, 1963); and crushing in a ball mill (Faber, 1941; Lamar and Shrode, 1953; Roedder, 1958; Goguel, 1963 and 1964; and Kramer, 1965).[27] Numerous sources of contamination or loss in ball milling (and some other methods) are discussed by Roedder (1958), Goguel (1963), and Suscherskaya (1968). Adequate cleaning of the sample prior to crushing, so that only materials from sealed inclusions are obtained, is an important but often neglected step. Lamar and Shrode (1953) worked with limestones and dolomites in which visible surface efflorescences indicated that some salts were present in connected pores in the rocks, open to the surface. These salts were not removed prior to ball milling in that the purpose of their investigation was not to determine the composition of the fluid inclusions. Such soluble salts are present in fractures in most samples, however, and do constitute a major source of contamination. Simple washing will not suffice to remove them, but extended electrolytic cleaning apparently removes most of them (Roedder, 1958).

One of the most important sources of *contamination* is the solution of ions from the broken surfaces of the host mineral or even very minute amounts of impurity minerals (Correns, 1940); absorption on the large amount of mineral surface may cause a major *loss* of ions from the inclusion fluids. The quantities of salts involved are very small and the volumes of solution are large, so that the amount of contamination from even high grade analytical reagents and apparatus can become a serious problem; ordinary laboratory deionized water is completely inadequate for much of the work, and all contact with glass should be eliminated (Roedder and others, 1963, p. 359–360). Unfortunately, many of the analyses made of leachates, as reported in the literature, do not give sufficient details on technique, such as blank analyses of reagents and water, volumes of leachate used, and analytical accuracy itself, to permit evaluation of the results. Easily made measurements of the electrical conductivity of leach solutions, both before and after leaching, are of great

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[27] One commonly suggested method of opening inclusions—expansion on freezing—has been found to be ineffective except in rare cases (Roedder, 1958, p. 243; 1962a, p. 1054; 1963b, p. 197).

value in monitoring the inevitable contamination during leaching, and they provide very useful data for planning the analytical procedures and checking the results obtained (Roedder and others, 1963).

Once the inclusion salts have been dispersed or dissolved in the leach fluid, the solution must be separated from the crushed mineral. The validity of this separation is a larger problem than it might at first appear to be, and a variety of methods has been used. If the crushing has been coarse, decantation or simple but contamination-free[28] filtering is possible. When ball milling is used, the fine grain size obtained requires special filters. Lisitsyn (1961) used membrane filters (pore diameter 0.04 μm), as did Goguel (1963). Roedder (1958) used an electrodialysis cell to separate the ions from the slurry of water and ground mineral. This is preferable to filtering, in that inclusion ions "plated out" on the rather large amount of mineral surfaces ($\sim 10^4$ cm^2/g) are also removed, and the anions and cations are separated, but as is shown in that paper, several additional problems are introduced. Kalyuzhnyi and Pritula (1968) use pressure filtration.

Ermakov (1950a, p. 57, 61, 64, and elsewhere, in translation) emphasizes that the composition of the fluid obtained by leaching crushed samples is not the same as that of the original fluid as trapped. The difference lies in the additional but unknown amount of host mineral that was in solution at the conditions of trapping. To correct this, he repeatedly recommends heating the inclusions to their homogenization temperature for a length of time adequate to reach equilibrium with the walls, then overheating them so they decrepitate, before leaching the residue. Lisitsyn (1961) and other Russian workers have also used this procedure. The method gives no assurance, however, that (1) any host mineral that does go into solution upon heating (the amount is generally very small) will necessarily be soluble in the leach solution after decrepitation of the inclusion and the resulting precipitation of solutes present, or (2) the solution of the host mineral surface by the much larger volume of leach solution will not put far more host material into solution than the original inclusion fluids precipitated upon cooling. In addition, Roedder (1958, p. 265) found that decrepitation, followed by electrodialysis, gave a very low yield of the normally highly soluble inclusion solutes, presumably owing to chemical reactions and diffusion into the solid sample upon heating.

Many of the techniques suitable for rock and water analyses may be used on the leachates, but in general the quantities of leachate and the concentrations of the individual ions in them are both so small that special semimicro- to ultramicromethods are needed. Although the concentration of salts in inclusions varies widely, if they are assumed to have 10 percent by weight of total salts, the extraction of 10 inclusions, each 1 mm in size (a rather large sample), will yield only 1 mg (milligram) of salts. About half of this milligram will normally be composed of Na and Cl, so that only about 500 micrograms (gammas) are left for all other constituents combined. Larger samples yield more inclusions, but at the cost of more ambiguity as to inclusion origin. A quantitative analytical procedure using flame photometry and several sensitive colorimetric methods particularly appropriate to the 1-mg range has been developed by Blanche Ingram. (See Roedder and others, 1963, p. 361–364.) Other micromethods are described by Gryn'kiv and Kalyuzhnyi (1966) and in a number of the papers listed in table 3.

Special methods such as polarography, quantitative spectrography, and X-ray fluorescence may occasionally be best for certain elements. Arnautov and others (1965; also Bazarov, 1965a) report the first analyses of inclusions made with a multichannel—photoelectric quantometer. Haffty and Pinckney (1967) report a quantitative spectrographic method in which only 0.2 ml of a fluid, containing only 1 to 10 ppm each of Mg, Na, and Ca, may be analysed for these other elements, with results that are in good agreement with atomic absorption spectrography. For sodium, this would correspond to analyzing a single inclusion approximately 0.1 mm in diameter. Neutron activation has particular promise for some fluid inclusion analyses. It is very sensitive for chlorine (5×10^{-8} g), although ordinary wet chemical analyses for chloride usually present few problems. It has actually been used for analyses of inclusions for the heavy metals (Cu, Mn, and Zn) in the 10^{-6}-g range (Czamanske and others, 1963). Ohmoto (1968b) has found atomic absorption analysis to be suitable for Na, K, Ca, Mg, Fe, Zn, and Cl in inclusions. Although none of these methods is universally applicable and perfect, and many are relatively low in precision, the major limitation in the analysis of inclusions, and in the interpretation of the results, still remains in the nature of the samples and the extraction procedures used.

Eh AND pH MEASUREMENTS

It would be very desirable to know the pH of the fluids in inclusions *at the time of trapping.* If these

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[28] Ordinary analytical filter papers may contribute very significant quantities of chloride ion, so that special filters are needed (for example, of asbestos (Roedder and others, 1963, p. 359)).

values could be obtained at room temperature, together with inclusion composition, extrapolations to the temperature and pressure of formation might be possible, and these extrapolations certainly would be valuable in solving problems of ore transport and deposition (Barnes and Ernest, 1961; Wilson, 1961; Barnes 1965). The pH may be obtained either by calculation or measurement. One of the first attempts at calculation was by Uchameyshvili and Khitarov (1965), who used the analytical results on CO_2 and HCO_3^{-1}.

Although many measurements have been made of the pH of fluid inclusions, few of them are at all accurate. Most of the older measurements are merely qualitative—large inclusions were opened and litmus paper was applied. Thus Newhouse (1932) found that the liquid inclusions in galena from Leadville, Colo., and Joplin, Mo., were neutral to litmus paper. Zakharchenko (1950) opened a 1.5 cm^3 inclusion in quartz and found the liquid to be alkaline to litmus (that is, pH between 4.5 and 8.3), but he noted that the inclusion fluid boiled violently on opening. The effects of such gas evolution on the pH of the fluids will vary with the gas and could be large. Kalyuzhnyi (1957, 1960, 1961) gives some results of microcolorimetric pH measurements of large inclusions in which the pH increased as much as one pH unit in the first few seconds after opening. He found pH values as low as 4.3 in multiphase inclusions in pegmatitic topaz. Maslova (1958) reports a pH of 4 for the liquid of a large (0.03 ml) inclusion in fluorite from the vuggy pegmatites of the Kermet-Tas deposit, which contained 69 volume percent liquid and 31 percent gas. Unfortunately, she does not state the method used to measure the pH. Skropyshev (1957) tested the fluid from a large inclusion with three different indicators, and Prikazchikov and others (1964) used a pH meter on the fluid from very large ($\sim$400 cm^3) inclusions. Yushkin and Srebrodol'skii (1965) also used three indicators, bromthymol blue, bromcresol purple, and cresol red, which they introduced into large inclusions in sulfur with a needle; the pH was estimated to be 7 to 7.5. Petrichenko and Shaydetskaya (1968) used three different methods on large inclusions in recrystallized halite; their results ranged from pH 4.95 to 6.2.

Erickson (1965, p. 527, and written commun., 1965) determined the pH of large inclusions in calcite from the Upper Mississippi Valley lead-zinc deposits to be about 7.5, by letting the inclusion fluid wet sensitive pH-indicating paper under a binocular microscope. Although no effervescence was noted, the inclusion fluid escaped almost instantly from the inclusion upon opening. Rather than representing gas evolution from the liquid on pressure release, it may signify merely the expansion of compressed gas in the bubble. The author has noted similar behavior for inclusions in the fluorite-zinc deposits of southern Illinois; the bubbles are extremely soluble in kerosene, and hence they may well be compressed methane or other organic gas.

Not infrequently, the pH of the dilute water leaches is reported as though it were the pH of the actual inclusion fluid (Grushkin and Prikhid'ko, 1952; Zakharchenko, 1955; Grushkin, 1958; Shchiritsya, 1960; Sheshulin, 1961; Dekate, 1961; Polykovskii, 1962a). Kostyleva and Sukhushina (1957) have avoided the variations in pH due to variations in dilution, grinding, etc., by establishing a strictly empirical grinding and leaching procedure for preparing the fluids for pH determination; they believe that the pH of the resulting suspension is a specific value, characteristic of the deposit, and reflects the liquid (and solid) inclusions. L. A. Prikazchikov (as quoted in Ivanova and Zakharchenko, 1963) believes he has evidence that the pH of the leach solutions is the pH of the inclusion fluids, and Skropyshev (1957) states that he obtained similar values for the pH of the actual inclusion fluid (pH, 7.0–7.5) and the leachate (pH, 7.45). However, Khetchikov, Balitskii, and Gasparyants (1966) found that the pH of the fluid in individal inclusions in synthetic quartz corresponded to that of the orginal fluid charge in the autoclave, but the pH of aqueous extractions from the quartz did not. Khetchikov, Balitskii and Dernov-Pegarev (1968) state that the pH of individual (large) inclusions is about the same as that of the mother liquor, but the pH of aqueous extracts has little relationship to that of the mother liquor, even at high alkalinities.

There are several major fallacies in the assumption that the pH of a leach solution is the same as the pH of the inclusions themselves, in addition to the common problem of loss of compressed gases mentioned above. Trufanov (1967) calculates that a thousandfold dilution will cause only minor "5 to 6 percent" changes in the pH of certain types of inclusions. Even if the diluting fluid were pure water at pH of 7.0 (the pH of the water used for leaching is seldom stated), it is very unlikely that the inclusion fluids are sufficiently buffered in composition that they can be drastically diluted without serious changes in pH, yet dilution factors (volume of leach/volume of inclusion fluid) as high as 5,000 are implicit in some of the pH data reported, and these factors are seldom as low as 1,000. In addition, there may be large effects on the pH of a leach solution just from contact with the host mineral and any other solid phases. It has long been known that mineral surfaces react with water, and this reaction affects the pH to the extent of providing a diagnostic field test for certain minerals (Stevens and Carron, 1948), yet the pH values for extracts from a wide range of minerals such as calcite, fluorite, spodumene, microcline, and quartz are frequently assumed to be the pH values for the inclusion fluids themselves, and to be sufficiently accurate that genetic conclusions may be drawn from them—even from small differences between individual samples.

Machairas (1963a, 1963b, 1963c) obtained a measure of the pH and the free CO_2 content of inclusions by a titration procedure, using the decoloration of an alcoholic solution of phenophthalein at pH of 9.3–10. Chukhrov (1960) measured both the pH of leach solutions and the pH of the water condensate obtained during decrepitation of the inclusions. The latter fluid was considerably more acid, as might be expected from the distillation of HCl fromed by high temperature equilibrium between SiO_2, H_2O, and NaCl. If the host mineral is quartz, its effects on the pH will be small. Zakharchenko (1955) found that the pH of a leachate from quartz rich in multiphase inclusions was 8.6 measured electrically. Although this pH cannot result from reaction with the quartz surfaces, very appreciable quantities of carbonates are present as daughter minerals in these inclusions and might act as a buffer in the pH determination. However, considerable liquid CO_2 and some H_2S were lost before leaching.

The writer knows of only one report of the determination of the oxidation potential (Eh) of the fluids in inclusion (Petrichenko and Shaydetskaya, 1968). In this work a pair of electrodes (platinum and calomel, using Zobell's procedure) were inserted in 0.8- to 1-mm holes drilled into large fluid inclusions in water-clear recrystallized halite from the Artemousk rock salt deposit in the Donbass. Atmospheric air was excluded, and a correction of +245 mv (millivolts) was made, following Garrels. The results on 27 single-phase liquid inclusions ranged from —10 to —130 mv, and averaged —70 mv. Two-phase inclusions gave higher Eh readings, from 0 to +150 mv, indicating that their bubbles represented infiltrated air. The acid reducing conditions during halite recrystallization are corroborated by the common occurrence of pyrite in such salt, and contrast with the weakly oxidizing environment of salt basins.

Any large inclusions could be measured with existing equipment, as miniature and subminiature Eh electrodes are available, but the problems of extraction of the fluid precludes most such measurements; in addition, the other problems of pH measurement generally are applicable here as well.

It should be possible to calculate the Eh from detailed studies of the composition of the inclusions. Thus any multivalence state component or group theoretically could be used—Fe^{+2}/Fe^{+3}, CO/CO_2, or HS^{-1}/SO_4^{-2}—together with pH measurements, but it is probable that neither the original high temperature equilibrium will be quenched in, nor that true room temperature equilibrium will be obtained. Miyashiro (1964) has shown that most subsurface waters are low in oxygen, which he attributes to reaction with graphite in sedimentary and metamorphic rocks. Chinner (1960) has pointed out that the oxidizing or reducing *capacity* of the fluid phase compared to that of the surrounding rocks is very important in regional metamorphism, where the system is closed to oxygen, but rocks which form thermal metamorphic aureoles were open with respect to oxygen at the time of metamorphism, as a result of greater movement of water.

Sometimes, daughter minerals may be useful in estimating the state of oxidation. For example, tiny crystals identified as hematite from their optical properties are not uncommon in the fluid inclusions in some porphyry copper deposits (Lindgren, 1905; Butler and others, 1920; Roedder and Creel, 1966). It would be valuable to know the sulfur species present, particularly sulfide versus sulfate sulfur, but there are no known analyses of inclusions that list both. Usually only sulfate sulfur is determined, and not infrequently this is actually total sulfur as sulfate. Except in rather rare instances, the concentration of sulfide sulfur is very low in the ore deposit samples that have been studied, at least as determined by the evolution of H_2S on opening or by calculation from the high contents of heavy metals (Czamanske and others, 1963). Daughter minerals found in inclusions in various Brazilian quartz crystals include sulfides such as pyrite (Roedder and others, 1963, p. 367) and sulfates such as gypsum (Bergman and Blankenburg, 1964). Simultaneous occurrence of sulfates and sulfides in the same inclusions would help to place definite limits on the state of oxidation of the fluids (Barton, 1957). Laz'ko (1958) has shown that both hematite and pyrite are found as primary *solid* inclusions in quartz crystal deposits in the U.S.S.R., nucleating on and growing contemporaneously with the quartz, but never both in the same crystal. Such data should be correlated with the analyses of fluid inclusions that have been made on these same deposits.

Unfortunately, the Eh of an inclusion fluid would be strongly affected by any loss of hydrogen or H_2S from the system. Although the evidence against gross leakage of major constituents from inclusions is commanding (Roedder and Skinner, 1968), there is no proof that small amounts of hydrogen cannot leak out and increase the oxidation state of the remaining fluids. One of the major difficulties in calling on this mechanism to explain the presence of hematite flakes in inclusions[29] is that maintaining an adequate "hydrogen sink" outside the inclusion to drive the diffusion process. As many ore-forming processes apparently take place at low oxidation states, that is, high partial pressures of hydrogen, the most expectable change in the state of the fluid bathing the exterior of the crystal would be toward more oxidizing conditions, particularly as the erosion surface approaches the deposit and oxygenated surface waters are involved. Even this should have only a small effect, however, because diffusion rates are greatly reduced at surface temperatures, and because ground waters penetrating ore deposits are rather effectively buffered with respect to oxygen by reaction with sulfides.

ANALYTICAL DATA

The literature providing data on the composition of fluid inclusions has been tabulated with annotations in tables 1, 3, and 8, and some representative quantitative analyses from this literature are given in tables 2, 4, 5, and 6. Most of the data have been recalculated for ease of tabulation and comparison, as indicated in the footnotes to the tables. In all the tables, only very brief details of localities are given; in many but unfortunately not all of the references cited, complete descriptions will be found. The analyses given are usually only a part of those given in the original reference and were selected to show the range or general nature of the work. The extent of the original work (and of many other works, particularly the most recent ones, that are not represented in the analyses of tables 2, 4, 5, and 6) can be found by referring to table 3, "Summary of quantitative and semiquantitative chemical and isotopic analyses of fluid inclusions."

Not uncommonly the statements of analytical data, as presented in the literature, are somewhat ambiguous. The most common source of ambiguity is the failure to distinguish between constituents that were looked for but were not detected ("n.d." in the tables), and those that were not determined. Other such sources of ambiguity are given in the text and the footnotes to the tables. In particular, the methods used vary so widely that many of these results are not strictly comparable. In addition, the methods used for both extraction and analysis are frequently unstated or stated so inadequately that true evaluation is impossible.

In making use of these analyses a general word of caution is needed. In addition to the problems of selection of methods for extraction and analysis detailed in the preceding sections, the importance of the nature of the samples used cannot be overemphasized. It is the prime consideration in the selection of suitable methods of study, and in evaluating the precision, the accuracy, and most important, the significance of any measurements obtained. All of the many analyses of fluid inclusions that have been made—the author's included—are subject to serious limitations. The chemical manipulations may be reasonably straightforward, but the small sample size and wide variation in composition of both the inclusions and the host mineral make the analyses far from routine and impose large analytical uncertainties. Much more serious, however, are such problems as the possible multiplicity or origin of the inclusions extracted and drastic contamination and (or) loss during extraction. Such factors have not been adequately evaluated in many if not most studies.

ORGANIC GASES AND LIQUIDS[30]

QUALITATIVE AND SEMIQUANTITATIVE DATA

Yellow oils were among the first substances in fluid inclusions to attract attention and study. Dolomieu (1792) reports the occurrence in quartz crystals, presumably from Modena, Italy, of an inflammable yellow fluid that had a bituminous odor. Such oil inclusions were also reported by Breislak (1818), who showed that the simple explanation of fluid inclusions being forwarded by the Neptunists of the day—they indicated the rocks were formed from water—was not necessarily true; the presence of oil inclusions in quartz does not necessitate the quartz having formed from oil.

Quartz crystals from vugs in sedimentary rocks not infrequently have inclusions of organic matter (Reese, 1898; Murray, 1957). The very clear quartz crystals from vugs in dolomite in the Herkimer area, New York, contain several types. A brittle

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[29] It has frequently been noted that small flakes of hematite in some inclusions fail to dissolve during homogenization runs.

[30] The term "organic" is used here in a strictly nongenetic connotation.

black, opaque material called anthraxolite is found in the vugs with the crystals; as it is commonly found also as inclusions in the quartz and as loose, broken fragments in the fluid of the inclusions, it must predate the quartz (Pike, 1949; Dunn and Fisher, 1954). In addition, however, these crystals also show inclusions of a fluorescent yellow oil and of colorless supercritical fluids, possibly mixtures mainly of methane and ethane (Roedder, 1963b, p. 202). Some of the latter inclusions homogenize with fading of the meniscus at critical temperatures of —5.55° and —7.85°C (pl. 5, figs. 6, 7). Murray (1957) presents a very complete analysis of apparently similar inclusions from Canada, detailed on page JJ44.

As the solubility of quartz in these various organic fluids is probably very low and as primary water inclusions do occur as well in these crystals, it is presumed that the oil and dense supercritical organic fluids occurred as immiscible droplets in the water solutions from which the crystals grew. Karpinskii (1880), in explaining the trapping of liquid CO_2, showed that alum crystals, grown from a water solution containing olive oil droplets, developed oil inclusions.

A bituminous odor on breaking or heating samples containing fluid inclusions has often been reported. Forster (1871) heated 4,500 g of smoky quartz from the Tiefen Glacier, Uri, Switzerland. He obtained 0.5–0.6 g of a brownish fluid which showed ammonium and carbonate upon testing; he presumed that a nitrogeneous hydrocarbon in the quartz had been decomposed. Wright (1881) obtained some hydrocarbons and noted a bituminous odor on analyzing the gases (presumably from inclusions) in pegmatitic smoky quartz, but he believed that this merely gave further credence to the concept proposed by Forster that the smoky color was due to organic matter. Organic matter of several kinds has been reported from vugs in various pegmatites. Thus Ellsworth (1932) reports several types of hydrocarbons (p. 173, 252) and numerous occurrences of an organic-rich solid called thucholite in the pegmatites of Canada. If such materials were to be found as inclusions in pegmatite minerals, rather than merely as vug fillings, it would reduce the ambiguity as to their origin.

DATA FROM ORE DEPOSITS

Certain ore deposits are not infrequently associated with organic matter (Germanov, 1961). Organic matter has been found even in minerals from high temperature deposits, such as sheelite (Matveyev, 1947; Beskrovnyy and others, 1967), and mercury deposits, in particular, very commonly have oily or asphaltic matter with them (Bailey, 1959; Fedorchuk, 1963; Krovtsov and Fridman, 1965), as do some of the Russian deposits of Iceland spar (Skropyshev, 1960; Goldberg and Belyayeva, 1965). The organic matter occurs both as inclusions in the crystals and interstitially as vug fillings, etc. Bailey (1959) presents textural evidence of immiscible globules of oil.[31] A similar association is found in some of the lead-zinc-fluorite deposits in sedimentary rocks, such as those of the Mississippi Valley in the United States and of the Pennines in England. Fluorite from both of these occurrences has abundant primary inclusions of yellow oil and a strong bituminous odor on crushing (Bastin, 1931 and 1950; Mueller, 1954c; Roedder, 1963b). The blues and purples of English fluorite (bluejohn) were ascribed to organic matter by Kraatz-Koschlau and Wöhler (1899), and Mueller, 1954a) presents evidence apparently proving that the Derbyshire bluejohn is colored by aromatic hydrocarbons, presumably from hydrothermal alteration of coal-type substances in associated sediments (Mueller, 1954a). Hoering and Abelson (1964) showed that saturated hydrocarbons of low molecular weight are evolved on heating sedimentary organic matter to 180° to 300°C; this type of process is probably the origin of most of the organic compounds reported from inclusions in sedimentary and metamorphic rocks. Bocharova (1964) proposes that the veins of asphaltic material cutting the polymetallic Kurultyken deposit are merely a late stage in the hydrothermal activity and that they represent organic matter extracted from the enclosing rocks by the hot fluids.

Grogan and Shrode (1952) have done geothermometry on primary oil inclusions in fluorite from southern Illinois. They found that the primary oil inclusions have filling temperatures around 100°C, whereas the secondary (they propose the term "subsequent") inclusions of water solution yield temperatures of 112° to 172°C. Freas (1961) obtained similar data, but he pointed out that the pressure correction for the oil inclusions was unknown. It has been suggested that the differences in filling temperature might be a result of a much higher thermal expansion and compressibility for oil than for water, and that if the compositions are known, these very differences might be used to establish *both* tempera-

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[31] Murdoch (1967) reports the occurrence of a new hydrocarbon mineral pendletonite from a California mercury mine. It has a very high melting point (450°C). This and similar compounds should be looked for in the usually unidentified "organic" matter in inclusions from such deposits.

ture and pressure (Roedder, 1963b p. 176–177). Both Grogan and Shrode (1952) and Freas (1961) noted that the oil inclusions have small masses of black opaque matter, which they call bitumen; presumably this is a natural degradation product of the originally homogeneous oil (Roedder, 1962b, p. 40) by a "maturation" process such as that proposed by McIver and others (1963).

The author has observed that the oil inclusions in the samples from southern Illinois are under considerable pressure, as crushing in liquids results in an explosive expansion of the gas bubble (and vaporization of part of the oil). The small vapor bubbles in the brine inclusions in these same samples are under moderate pressure, and they expand about 20 volumes on opening (Roedder, 1970). Roedder, Ingram, and Hall (1963, p. 368) and Hall and Friedman (1963, p. 901) report only very minor amounts (less than a few micrograms) of noncondensable gases in analyses of these samples, but even this small amount would be adequate to cause such behavior. The gas bubble from both types of inclusions dissolves in kerosene so rapidly that its expansion and subsequent disappearance on crushing sometimes is almost too rapid for the eye to register.

POPPING SALT

Considerable attention has been given to the phenomenon of popping salt—salt which seems to explode or crackle on dissolving in water. Dumas (1830) first described such salt from Wieliczka, Poland, and found that gas was evolved, presumably from compressed gas inclusions, upon dissolving the salt. Further details on the occurrence are given by Rose (1839). This type of salt can cause serious mine accidents when large volumes of salt explosively and spontaneously decrepitate into the mine openings. Belchic (1961), Hoy, Foose, and O'Neill (1962), and Thoma and Eckart (1964) have described mine "blowouts" from this cause in which as much as 7,500 tons of salt suddenly decrepitated.

The gas responsible for this is commonly thought to be carbon dioxide, although Dumas (1830) and Rose (1839) found the gas to be inflammable. Bunsen (1851, p. 251) found 84.60 percent CH_4 in the gases from Wieliczka salt. There are probably large variations from place to place in the mine. Thus some samples of halite collected at Wieliczka personally by the author, showing gray zones containing an estimated 2×10^{10} minute primary cubic liquid inclusions per cm^3 and arranged in rows parallel to (100), have no gas pressure at all, as there is not even nucleation of a gas bubble on opening the larger (20 μm) inclusions.

Hoy, Foose, and O'Neill (1962) found CO_2 to make up 46.9 volume percent of the gas in inclusions from the Winnfield salt dome, and Ackermann, Schrader, and Hoffmann (1964) found 84 volume percent CO_2 in the Werra potash deposit gases (plus 14 percent N_2). (See "Sedimentary and Metamorphic Samples," p. JJ44.) Correns (1954) was able to show that one-phase inclusions in salt, from the Saxony Weimar mine, Werra potash deposits, develop a bubble on cooling to $+11°C$; he interprets this to indicate a dense CO_2 liquid, but these data alone do not preclude a dense organic fluid composition.

There have been other explanations. Schmidt (1960) found no (free) CO_2 on chemical analysis of potash ores, but he proposes that the CO_2 explosions in these deposits arise from the instability of solid magnesium chlorocarbonate ($MgCl_2 \cdot MgCO_3 \cdot 7H_2O$). His identification of this compound in one sample high in magnesium is based on electron microscopy. Kegel (1958) proposes that the CO_2 in sylvite which causes explosions occupies structural faults in the KCl lattice, as he believes that such large amounts of CO_2 could not be present as inclusions. Roedder (1963b, p. 183) has shown, however, that at least 0.44 weight percent of inclusions (in this case brines) can be present in bedded salts.

The author has examined the inclusions in popping salt from several additional localities and finds that there are gross differences between them. Thus salt core from the Tuzandeptl salt dome (Jurassic age) in Vera Cruz, Mexico, obtained through the courtesy of Dr. Donald R. Richner of the Diamond Alkali Company showed highly compressed gas inclusions with a strong petroleum odor. The gas evolved was evidently a mixture, as about half is readily soluable in kerosene and the other half is readily soluble in water. Thus, this gas is presumed to be an easily liquefiable mixture of organic compounds, such as ethane, and carbon dioxide. On dissolving the salt in water, each inclusion gave off not only gas bubbles with a volume approximately 250 times that of the inclusion, but it also formed a small amount of a liquid, immiscible in water. As this liquid was soluble in kerosene and had an index of refraction well above water, it probably consists of petroleum-type compounds. Popping salt from the folded salt beds near basalt dikes and sills at Werra, East Germany (obtained through the courtesy of Dr. Herbert Reh, of Jena), gave off 15 cm^3 of an odorless, noninflammable gas, believed to be mainly CO_2, when 700 grams was dissolved in water. (See also Strepetov, 1968.)

In addition to gas inclusions, natural rock salt not infrequently contains inclusions of dark bituminous material (Naumann, 1911) or of yellow-brown oils.

HYDROGEN SULFIDE AND SIMILAR ODORS

Hydrogen sulfide is occasionally reported from fluid inclusions and is generally attributed to organic sources. The odor of fetid limestones or "stinkkalk" is usually assumed to come from H_2S in fluid inclusions (Palache and others, 1951, p. 153). Lamar and Shrode (1963) found about 0.002 weight percent (up to a maximum of 0.012 percent) of H_2S evolved on treating limestones with HCl, and Harrington (1905) found 0.016 weight percent S as H_2S to be present as inclusions in coarse marbles of the Grenville series; it was released by solution in acid.

Not all fetid odors in rocks and minerals can be assigned to H_2S. Lucas (1952) studied three such limestones and presented evidence that their odor was due to a water soluble ammoniacal salt of a volatile acid, containing phosphorus but no sulfur. Hess and Barnabas (1938) describe a large mass of fetid microcline (which they call necrolite) in a tantalum- and tin-bearing pegmatite. They indicate that the vile odor presumably comes from the very numerous liquid inclusions, and the odor has been attributed to a phosphorus compound. An analysis of the microcline shows 0.66 weight percent P_2O_5 and only traces (if any) of Cl and CO_2, but no analysis was made for H_2S.

Hayden (1819) described what he thought was a new mineral from near Baltimore, Md., tentatively called "necronite," which emits a "horrid" smell on breaking. It may be feldspar. Mawson (1906) describes a similar "necronite" feldspar from Australia, later shown by Mawson and Dallwitz (1945, p. 25 and 34) to be from an albite leucogranite containing large crystals of sphene, that contains large numbers of small liquid inclusions yielding a very strong fetid odor on crushing. By crushing under an ammoniacal solution containing lead acetate, Mawson showed that H_2S, if present, must be very low in concentration. Mawson and Dallwitz (1945) mention that the odor suggests a combination of H_2S and CS_2. Both CS_2 and carbon oxysulfide (COS) were found by mass spectrometry in the gases evolved from some quartz crystals (Chaigneau, 1967b). John S. Dickey, Jr., reports that a pyroxenite nodule from the Kakanui volcanic breccia in New Zealand emits an odor similar to CS_2 on crushing, but crushing under solvents for CS_2, followed by sensitive tests for CS_2, gave negative results (written commun., 1966). The author agrees that the odor is indeed very similar to CS_2 and plans to crush some in a mass spectrometer for verification. Eck and others (1966) report calculations showing rather surprising concentrations of both CS_2 and COS at equilibrium in the system C-H-O with 0.0001 percent added S.

Qualitative studies of organic gases from inclusions in igneous and metamorphic rocks have been made (table 8), but as there are many actual analyses, some of which are given below, only one qualitative study will be mentioned here. Heinrich and Anderson (1965) used a time-of-flight mass spectrometer to identify the major constituents in the gases evolved from a crushed but unheated sample of a carbonatite having a disagreeable odor. They found C_5 and C_6 hydrocarbons (fluorinated?), plus lesser amounts of F_2, HF, F_2O, and possibly a small amount of HCl.

QUANTITATIVE DATA

The only extensive quantitative data on organic materials in inclusions are on the gases emitted from inclusions on opening. The first part of table 2 lists representative analyses of gases from sedimentary and igneous rocks that are high in "organic" constituents such as CH_4, C_2H_6, and CO.[32]

SEDIMENTARY AND METAMORPHIC SAMPLES

Nesmelova (table 2, analyses 1 and 2), in her study of the gases in bedded potassium salts, found that their composition varied from bed to bed. She distinguishes between *free* gases in the rock and *inclusion* gases; the former she believes can be related to biochemical processes in the interstratified clay beds. Borshchevskii (1964) suggests that radioactive decay of K^{40} has caused not only the oxidation of ferrous iron to yield the red color of carnallite and sylvite, but also has decomposed bitumens to from the methane and hydrogen in the gas inclusions.

Hoy, Foose, and O'Neill (table 2, analysis 3) found that the gases evolved on fusing a "popping salt" in sealed tubes were mainly CO_2, but appreciable amounts of organic constituents were also found. Ackermann, Schrader, and Hoffman (1964) also analyzed popping salt, from another locality, using gas chromatography. Their analyses show an essentially pure CO_2-N_2 mixture; their average values are entered in table 2 (analysis 4).

An essentially pure organic gas was found by Murray (table 2, analysis 4a) on opening colorless hydrocarbon inclusions (liquid plus gas) occurring in quartz crystals from solution vugs from the gas-productive interval in a well producing natural gas

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[32] For simplicity, those gas analyses high in hydrogen are also included in this part of table 2.

and condensate. The inclusions homogenized in the *gas* phase at 100°±5°C and are believed to be primary. Although the quartz presumably grew from water solution (Füchtbauer, 1961, reports that the waters expelled from oil reservoirs frequently deposit quartz crystals), no water was found in these inclusions, even though sensitive mass spectrometric gas analysis procedures were used and 17 other constituents were determined quantitatively. This may be another case of inclusions trapping only the dispersed phase in a two-liquid system.

It is well known that some methane forms at equilibrium by the interaction of CO_2 and H_2 from H_2O, in completely inorganic systems (French, 1965). Also, methane and higher hydrocarbons are major constituents among the products of low temperature heating of sediments (Hoering and Abelson, 1964), and they form from biochemical processes in sediments (Ivanov, 1960). Thus, as would be expected, many of the partial analyses of inclusion gases reported in the literature, from many types of samples, include appreciable amounts of methane. Chamberlin (1908, analysis 83, p. 19) reports 6.4 volume percent of methane in the small amount of gases other than water that he obtained, 2.9 ml/100 g, on heating clear quartz crystals from a metamorphic terrain. He also obtained small amounts of methane by crushing cavernous quartz crystals from Porretta, Italy, in a vacuum (Chamberlin, 1908, p. 41).

IGNEOUS SAMPLES

A large number of analyses, particularly by Chamberlin (1908), have been made of the gases evolved on heating igneous rocks and on ball milling igneous rocks. (See table 2.) The available data indicate that the gases evolved on heating rocks can come from a variety of sources in addition to inclusions and may be grossly modified by reactions with each other and with the sample. (See Wahler, 1956, and the previous discussion.) Barker (1965a; table 2, analyses 23a, b, and c) obtained two very different compositions of gas from granitic orthoclase by varying the heating temperature and got very large amounts of H_2 and H_2O from biotite (analysis 23c). In this connection it is interesting to compare these analyses with "gas inclusions" from pumice vesicles (Finko, 1964; table 2, analysis 23d), which are one-third hydrogen. Nasedkin (1963) reports a series of similar analyses, but Chaigneau and Bordet (1962) report approximately one-third CO, and about 5 volume percent SO_2.

By far the largest number of analyses of organic-rich gases obtained by ball milling are of alkalic rocks of the several massifs of the Kola Peninsula, U.S.S.R. Most of these are given in a series of papers by the Petersil'e (1958a, 1958b, 1959, 1960, 1961, 1962, and 1963; also Petersil'e and Proskuryakova, 1961), by Kravtsov and others (1967), by Galimov and Petersil'e (1967), and by Ikorskii (1962, 1964, 1965, 1966a, 1966b, 1967a, 1967b, and 1968). These gases are distinctive in that most combustible gases found in igneous rocks are high in hydrogen, but these are high in methane (or CO_2 and methane), have very appreciable concentrations of the higher hydrocarbons C_2H_6, C_3H_8, and C_4H_{10} (see table 2, analyses 11 and 17), and have only a small but variable hydrogen content (Kravtsov and others, 1967). Thus they are very similar to the gases from many ordinary natural gas fields. In addition to those obtained by vacuum ball milling, similar gases, presumably air contaminated, have been obtained from boreholes in the complex (Petersil'e 1959) and are discharged continuously to the atmosphere (Petersil'e, 1961; Kravtsov and others, 1967). The oil fraction in cold chloroform extracts from these rocks consists of pure paraffinic hydrocarbons, and a variety of bituminous compounds, containing nitrogen, oxygen, and sulfur, have been found in small but significant amounts (0.002 weight percent) (Petersil'e, 1959, 1962, 1963; Zezin and Sokolova, 1967). The associated mafic and ultramafic intrusive rocks in the area show in general very low gas contents, without heavy hydrocarbons (table 2, analyses 12 and 15). It is possible that some of the samples showing high CO_2, such as analyses 6 and 7 (table 2), might have had some carbonate with them which can release spurious CO_2 on ball milling (Goguel, 1963) (see p. JJ34), but the organic constituents are probably present as actual inclusions. Although most of these analyses are given on a nitrogen-free basis originally, large quantities of nitrogen were apparently found, but according to Goguel (1963), they represent contamination.

According to Elinson (1959), the numerous rocks analyzed show rather large variations in both the amount and composition of the evolved gases. Petersil'e (1962) says Elinson's statements are based on inadequately checked data and that she is wrong, and he claims that more than 500 analyses show that the gas composition is "nearly constant" from rock to rock and that it differs little from that of oil field gases. The analyses given by Petersil'e himself (1959 and 1963; analyses 5–17, table 2), as well as those of Ikorskii (1962, 1968; analysis 18, table 2), show very great variations.

The *amount* of gas has little real significance; it merely reflects the abundance and size of the inclusions. Tikhonenkov (1963) presents photomicrographs of eucolite from pegmatites related to the sphene foyaites (nepheline syenites) of the Khibinsk massif that show approximately 3×10^{10} gas inclusions per cm^3, and he indicates that these must have formed prior to the formation of late albite and sphene, on the basis that a halo of inclusion-free (recrystallized?) eucolite surrounds these late minerals. Zakrzhevskaya (1964) finds that many of the inclusions in nepheline are of several generations, possibly due to repeated crushing of the rocks, and are present in adequate volume and frequency to yield the gases found on vacuum ball milling. There is however, a gross and inexplicable difference in the hydrogen contents reported in this work for a nepheline-bearing rock and the nepheline separated from it.

Microscopy of these samples is apparently difficult. Both Ikorskii (1962) and Zakrzhevskaya (1964) report the presence of both glass and gas inclusions in these rocks; Ikorskii observed no change in the gas inclusions on cooling to —10°C. Ikorskii and Romanikhin (1964) and Ikorskii (1965, 1968) present evidence that most of the gas inclusions are primary, having formed early, during the crystallization of the nepheline. Dudkin (1964, p. 84) states that liquid-gas inclusions occur in highest concentrations in the borders of the nepheline grains in the Khibinsk pluton; this implies a late-stage, primary origin.

Petersil'e states (1960, 1961) that the combustible gases of these alkalic intrusives do not occur in the effusive, sedimentary and older metamorphic rocks surrounding them and that the geologic position of the massif excludes migration from any sedimentary formation. He believes the gases are of inorganic origin and that the heavier hydrocarbons and the aliphatic ester type of bitumens are a result of a catalytic polymerization, even though the bitumens, in particular, are very similar to those found in sedimentary rocks of oil-producing areas. In part, this concept of polymerization is based on a dependence of the bitumen content on the concentrations of hydrocarbon gases in these rocks. Gol'dberg and Chernikov (1968) believe, however, that these bitumens are from surrounding sedimentary rocks and that they have been separated chromatographically during their migration. In his 1963 paper, Petersil'e indicates that at least some of the complex aromatic hydrocarbons (see also Florovskaya and others, 1966, and Zezin and Sokolova, 1967) and esters of carboxylic acids are formed by an inorganic mechanism. It should be noted that hydrocarbon gas inclusions have also been found in minerals of the similar nepheline syenite complex at Ilímaussaq in Greenland (Prof. H. Sørensen, oral commun., 1968; Sobolev and others, 1970; Galimov and Petersil'e 1967; Petersil'e and Sørensen, 1970).

At present there does not seem to be any explanation for the gross differences in gas composition between the various rock types. Even if one accepts Petersil'e's proposal that aluminum of the rocks acted as a catalyst to permit reaction of hydrogen with carbon (Petersil'e, 1959) there are major differences in the elemental composition of the gases that are left unexplained. One of the most divergent analyses (table 2, analyses 11) is from an otherwise very unusual rock containing appreciable amounts of villiaumite (NaF) and a rather high chlorine content (0.2 weight percent (Kogarko, 1961)). A theory proposed by Borshchevskii (1964) to explain the methane, hydrogen, and other gases as inclusions in potassium salts, as a result of radioactive decay of K^{40} causing decomposition of bitumens, might also be applied to some of the Kola rocks.

Kudriavtsev (1958) made solvent extractions of powdered lavas, including even obsidians. He obtained tens to hundreds of part per million of bitumen, containing a variety of compounds, which he believes are of magmatic origin. Kropotkin and others (1966), using a vacuum crushing technique, found similar amounts of hydrocarbon gases, and oily bitumens, in cores from deep in the Precambrian crystalline basement rocks of the Tuymazy uplift. (See also Kuprin and Ovchinnikova, 1968.) Petersil'e and others (1967) also extracted bitumens from tektites. Slivko and Pavlishin (1967) report the presence of up to 3.9 percent organic matter in late opal from the Volynian pegmatites, which they believe is probably endogenic. Mogarovsky and Markov (1966) report 50–180 ppm of bitumen in a number of hydrothermal fluorite ores from Tadjikistan, and Mogarovsky and others (1966) report bitumens in various granitic rocks, which they attribute to hydrothermal processes that also produced fluorite deposits. Vdovykin (1963) also found 79 ppm of bitumens, which he believes to be of inorganic origin, in the Kokpekta serpentinite massif. Konyukhov and Florovskaya (1963) defend the position that hydrocarbons and bitumens are formed in the crystallizing magma by inorganic reactions (see also Voronoi, 1967), but others believe that both the gases and the bitumens are derived from assimilated sediments. (See also Hoering, 1965, and a series of papers edited by Dolenko, 1967). Studies by Ponnamperuma and Pering (1966) show that alkanes isolated from bitumens associated with certain hydrothermal environments exhibit gas chromatographic distributions that resemble synthetic, inorganic (electrical discharge) hydrocarbons more than they do biogenic, sediment alkanes. However, later studies concerning the carbon isotope ratio and optical activity of these same alkanes indicate that they are otherwise similar to their biologically derived counterparts (Katherine Pering, oral commun., 1968).

Comparatively few other analyses of the gases from inclusions show appreciable quantities of organic constituents, although odorous traces are common (for example, Wright, 1881). Chamberlin (1908) analyzed the gases evolved on heating one rock from the alkalic complex in Ontario, Canada. In this nepheline syenite he found major CO_2 and H_2, with <10 volume percent each of CO, CH_4, and N_2 (table 2, analysis 18a). Elinson and Polykovskii (1961b) found organic constituents in the gases from a number of pegmatite minerals, taken to present a sequence in terms of declining temperature of formation. Three of their analyses are presented in table 2 (analyses 21, 22, and 23). They believe that the methane present in the late smoky quartz was formed by reaction of CO_2 with H_2 during cooling. The large quantities of nitrogen found are presumably contamination, according to Goguel (1963). Sheshulin (1961) found amounts of H_2 and CH_4 comparable to the amounts in the Kola rocks in spodumene crystals from pegmatites cutting bituminous limestones (table 2, analysis 42), but the large amounts of water present make the quantities of H_2 and CH_4 seem small.

Some diamond pipes are known to give off combustible gases; Vasil'ev, Koval'skii, and Cherskii (1961) give an analysis of such a gas (table 2, analysis 19) that had to be burnt off in large flares during the drilling, and Raguin (1961, p. 468) mentions encountering a combustible gas at 330 meters in a South African diamond pipe, but he gives no analysis. $C^{12}/^{13}$ isotope ratios should be made on such gases, to compare with those from diamonds and possibly associated carbonaceous sediments. Beskrovnyy (1958) reports nongaseous organic matter from petroleum to asphaltic bitumens in Siberian kimberlite pipes. Beskrovnyy and Baranova (1963) also report the presence of petroleum-type bitumens in pegmatites and carbonatites.

Inflammable gases have also been reported from dunites. Zavaritskii and Betekhtin (1937) report a hydrogen-rich gas from a Uralian dunite (table 2, analysis 20) and Kuznetsov, Ivanov, and Lyalikova (1963, p. 20) mention a gas with 33.8 volume percent of hydrogen "in the serpentinites of Romania," but they give no other information.

ESSENTIALLY NONORGANIC GASES

Most of the analyses of gases from igneous rocks and pegmatites show mainly inorganic gases, with only relatively small amounts of organic constituents. There are several possible sources for such gases. In addition to gases from inclusions and from various reactions of the inclusion contents, there may be gases in structural holes, in discontinuities within crystals, adsorbed on grain boundaries, or even dissolved in the minerals. Many of the studies reported in the tables merely give analyses of the gases obtained by some method and do not attempt to characterize them as to origin.

IGNEOUS ROCKS AND PEGMATITES

A series of such analyses, mainly from granitic rocks, is presented in table 2 (analyses 24–61). Goguel (1963) analyzed 43 different samples by his vacuum ball-milling method, of which ten are listed in table 2 (analyses 24–33). Analysis 32, of optically clear quartz, is in effect a blank run; note that most of the samples yielded over 10^3 times as much gas as this blank. The great preponderance of water is surprising—even the olivine nodule (analysis 33) shows 300 times more water than CO_2, yet microscopy of similar nodules from this locality indicated small amounts of visible liquid CO_2 inclusions (Roedder, 1965d) and no evidence of water in any visible form. The biotite separated from a granite (analysis 27) shows more gas than the quartz, orthoclase, or plagioclase from the same rock; it is not known how much structural water may be evolved from biotite during vacuum ball milling. Goguel has examined several possible sources for the hydrogen and feels that it may be actually dissolved in the biotite. Fluid inclusions in mica are mentioned briefly by Sorby (1858, p. 487) and were studied by Karskii and Zorin (1968), but they seem to be very rare. Preisinger and Huber (1964; see also Arming and Preisinger, 1968) reported mass spectrometric determinations of the gas evolved on heating very tiny fragments; they showed gross differences in the gas content of the core and rim of a feldspar grain and indicate that this method presents us with a powerful tool for inclusion study.

Some determinations of the gases in inclusions in pegmatite minerals have been made (table 2, analyses 28, 29, 31, and 36–49). M. M. Elinson and V. S. Polykovskii analyzed a series of samples by vacuum ball milling, using steel balls, which presumably yielded most of the nitrogen, according to Goguel (1963). However, other analyses made with a steel ball mill and presumably with steel balls (Umova and others, 1957, 1960) show low to zero nitrogen contents. Very appreciable amounts of argon were found (1–3 volume percent; see footnotes to table 2) in addition to the nitrogen, and it seems doubtful that this could come from the steel grinding balls. Elinson and Polykovskii believe that these gases are from *gas* inclusions, and they present their analyses on a water-free basis. They place genetic significance on the differences in the *amount* of gas between the early and late generations of citrine and conclude on this basis that the early citrine was pneumatolytic and the late, hydrothermal. No explanation is given for the considerable variation in CH_4 and H_2.

Sheshulin (table 2, analysis 42) noted that the small amounts of methane and heavy hydrocarbons he reported from a pegmatite cutting organic-rich marbles were generally absent in similar pegmatites cutting amphibolites. Kovalishin (analyses 49a, b, and c) reported an increase in methane content toward the center of the quartz core of a Volynian pegmatite.

Zhdanov (1959) obtained hydrogen by heating quartz above 500°C. He felt that it came from the dissociation of surface OH^{-1} groups, but Kats (1962) showed that from 10^{17} to 10^{19} H^{+1} ions per cm^3 are present in the structure of natural quartz, compensating for substitutional Al^{+3}. A clear Brazilian quartz contained 5×10^{18} hydrogens, which calculates to 0.003 weight percent H, or 34 ml/100 g. Although this is one or two orders of magnitude more than was found in any of the analyses of quartz listed in table 2, it would not be expected to come out of the structure easily (Brunner and others, 1961). Griggs and others (1966 and written commun., 1966) have shown that an experimentally deformed synthetic quartz crystal having about 10^{20} hydrogens per cm^3 formed discrete inclusions (0.2 μm maximum) when annealed at 300° to 500°C.

Wahler (table 2, analyses 43 and 44) analyzed the gases from the decrepitation of rather large inclusions at about 600°C in a vacuum. Although the other pegmatite analyses presented (analyses 45–49, all made by vacuum heating) show a rather surprising consistency, at compositions that are not unrealistic in terms of the normal phases found in such inclusions, the consistency is probably coincidental, as the author has observed individual samples in such pegmatite minerals with fluid inclusions compositions differing very markedly from these (for example, all water, or all CO_2).

Strutt (1908) extracted rather large quantities of helium from Greenland fluorite (27 cm^3/1,000 g), and he discovered that beryl evolved large quantities of helium (up to 1.68 cm^3/100 g), as well as argon, on heating to red heat in a vacuum. He obtained only 0.9 mm^3 of helium from 1,187 g of galena from Nenthead, Cumberland, England (p. 583, his table III). Aldrich and Nier (1948) found a wide variation in the "excess" He and Ar contents of beryl samples (excess over that to be expected from the decomposition of the uranium and potassium present); presumably these gases occur in the large holes in the beryl crystal structure. Hart and Dodd (1962) and Hart (1966) found excess radiogenic argon in pyroxenes and ultramafic rocks, and Smith and Schreyer (1962) report that in cordierite, argon occurs in the large holes and water (or OH^{-1}) in the smaller ones.

It may be difficult to separate gases evolved from such structural sites from those from inclusions. Probably much but not all of the helium and argon found in beryl by Damon and Kulp (table 2, analyses 46–49) was present in the holes in the structure, but as they heated their samples to temperature of 800° to 1000°C, and even 1400°C, both decrepitation from inclusions and diffusion out of the structural holes may have contributed. The same is true for water from beryl and cordierite (Suguira, 1959). Florensky (1956) reports that considerable quantities of argon can be present in the waters emitted during metamorphism. Rama, Hart, and Roedder (1965) found very large amounts of excess argon in pure white quartz veins from a metamorphic terrain, apparently present in the numerous fluid inclusions. Laughlin (1966, 1967) reports excess argon in pegmatite minerals, yielding high apparent K/Ar age determinations. Funkhouser, Naughton, and Barnes (1965), Naughton, Funkhouser, and Barnes (1966), and Funkhouser and Naughton (1968) report similar findings for ultramafic nodules from basalts; both groups of studies may indicate that excess argon is present in fluid inclusions.

HYDROGEN SULFIDE

The H_2S values given by Damon and Kulp (table 2, analyses 46–49) are among the very few quantitative determinations made for this gas. Maslova (1961) presents a series of 15 analyses of gas bubbles from large inclusions in quartz from Volynia, in which the minimum H_2S content is 10.1 volume percent and the maximum is 94.5 volume percent; CO_2 made up most of the balance. The methods used involved absorption of CO_2 in a glycerol solution of KOH and of H_2S in a glycerol solution of cadmium acetate. As the gas samples were handled under anhydrous glycerol, the analyses are on a water-free basis. Paradoxically, she found *no* HS^{-1}, H_2S, or CO_2 on ultramicrochemical analysis of the associated liquid phase.[33] Kovalishin (1962) reports considerable H_2S in quartz from some of these same pegmatites, and Ermakov (1965b, p. 157 in original) mentions that these quartz samples emit a stench on being crushed.

Mahon (1962a, 1962b) presents analyses of carbon dioxide and H_2S in the steam from Wairakei and Kawerau, New Zealand. He finds CO_2/H_2S ratios of 15 and 56, with about 10 to 50 ppm of H_2S in the total gas. The CO_2 from inclusions in most samples must contain far less H_2S than this, as H_2S is detected only rarely. Goguel (1963) found very small amounts of H_2S (up to a maximum of 0.7 mm^3/g) on ball milling several mica samples in vacuum, but this figure was about at his limit of detection.

ORE DEPOSITS

Very few reasonably complete quantitative analyses have been made of the gases in inclusions in samples from ore deposits (table 2, analyses 50–63) although qualitative observations show CO_2 gas (and even CO_2 liquid—see tables 1 and 3) to be present in many deposits. Garrels and Richter (1955) have even considered the possibility that CO_2 is an ore-forming fluid. Umova, Glebov, and Shibanov (table 2, analyses 50–54) ground quartz in a steel ball mill with unspecified grinding media. They report generally low nitrogen values in the gases obtained, but one analysis (53) shows 41 percent N_2. The chlorine reported was determined turbidimetrically after absorption in an alkaline solution. They state that the CO found in many analyses, including their own (for example, analysis 53), may be fallacious, due to the ammoniacal CuCl solutions used; when another copper absorbent ("persulfuric copper") was used, they found no CO. Chukhrov (table 2, analysis 55–57) lists the composition of the gases evolved, presumably on heating, from various types of vein quartz from the Kounrad molybdenum and tungsten ore deposits. From numerous analyses, he concludes, among other things, that the gases are higher in hydrogen in the ore zones. Kravtsov and Fridman (1965) report that CO_2 gas is sufficiently abundant in the Baley gold deposits (presumably as inclusions) to be useful as a prospecting tool. (See also Boyer and others (1967).) They also report the occurrence of CO_2 and hydrocarbons in the Krasnodar mercury deposits, as "hypogene" gas sealed in pores, with the highest concentrations (144 cm^3/kg) in the richest ore. Liquid CO_2 has been found in inclusions in some mercury mines (Roedder, 1963b, p. 188; see also pl. 5, fig. 1).

Assadi and Chaigneau (1962) and Chaigneau and Assadi (1963) heated 5-g pieces of uranium-bearing fluorite ("antozonite"), and of uranium-free fluorite, to 1,000°C in a low vacuum (10^{-2} mm). There was considerable variation in the analyses of each type (table 2, analyses 58–59 and 60–61), but the only apparent difference that they could relate to the uranium content seemed to be in the SiF_4 content. The uraniferous samples in general had considerably higher SiF_4 content (and higher HCl), but neither was proportional to the uranium content. Greenwood (1964) believes that the odor emitted upon grinding certain fluorites is from the reaction of fluorine atoms (freed by radioactivity) with the water of inclusions to form ozone and hydrofluoric acid. Qualitative verification of this has been found by mass spectrometry (Heinrich and Anderson, 1965).

Chaigneau and Marinelli (1964) analyzed the gases evolved on heating a variety of minerals from a magnetite deposit. (See analyses 62 and 63, table 2.) They report considerable SO_2, CO, and H_2. Barker (1965b) compared the analyses of gases evolved on heating samples of fluorite in a vacuum with those evolved on crushing in a vacuum. (See analyses 64 through 69, table 2.) He found major differences which are attributable to a variety of gas reactions during the heating. (See page JJ35.)

SOLIDS

The solids present in fluid inclusions generally lend themselves only to qualitative studies, that is, identification of the phases themselves, and rather crude measures of the volume percent of each (see p. JJ19 and JJ28). Many of the identifications are subject to some ambiguity, but generally they have been substantiated by other studies of similar samples. In table 1 are tabulated most of the more certain identifications of *daughter* minerals recorded in the literature, as well as other such qualitative phase identifications. Many of these identifications were made by unstated methods, and some have been questioned by their own authors. For convenience, information on inclusion thermometry is also in the table. An attempt was made to eliminate from this

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[33] Dolgov and Shugurova (1966a) showed that the cadmium acetate method I. N. Maslova used for H_2S analysis of gases gives seriously erroneous (high) results, due to absorption of CO_2. Elinson (1968a) used absorption in titrated KOH and iodine solutions to discriminate between these two gases.

table all references to *solid inclusions*, trapped during the growth of the host mineral. This distinction is not always made in the sources stated, but for qualitative considerations it is not too important. It is apparent that the fluids trapped in inclusions in a given host mineral A, crystallizing simultaneously with mineral B and hence containing solid inclusions of B, will also be saturated with respect to B, and hence may well be expected to form daughter crystals of B. The host mineral must always be considered an additional "daughter mineral" as well, as some crystallization of it usually occurs on the inclusion walls upon cooling.

LIQUIDS

There are many qualitative determinations of the phases present in fluid inclusions. Table 1 lists those reports in which phases other than simple aqueous liquid, with or without gas, have been identified. The occurrence of daughter crystals of NaCl, and of liquid CO_2, is so common that there are probably many incidental references to them that are not included in table 1.

A summary tabulation of all actual analyses of aqueous fluid inclusions, both quantitative and semiquantitative, is given in table 3. A number of qualitative analyses have been made by a variety of methods. These are listed in table 8, along with some miscellaneous semiquantitative and quantitative but partial chemical analyses. Representative analyses of liquid inclusions from a number of the reports listed in table 3 are given in tables 4, 5, and 6. Individual reports of analyses have been presented in many different forms, making comparison difficult. There are, however, three general types of analyses: *ratio analyses,* in which only the ratios of several constituent ions are stated and not their absolute amounts (table 4); *leach analyses,* in which the inclusions from a given weight of sample are extracted and the ions present are analyzed, but the amount of water, and hence the concentrations, is unknown or can be only crudely estimated (table 5); and *complete quantitative analyses,* in which both the amounts of water, and of ions, are determined, permitting a statement of analysis in the form of the actual composition of the inclusion fluid (table 6). Many of the Russian analyses are presented in the original papers in terms of a somewhat subjective list of "most probable salts," sometimes under the stated assumption that these "molecules" are actually present in solution. Only the conventional atomic-ionic statement of analyses is used here.

RATIO ANALYSES

Zakharchenko (1950) presented one of the early quantitative analyses of fluids from inclusion-rich pegmatitic quartz ("5–6 percent inclusions"), but the amount of salts found, which may be calculated as less than 3 percent, by weight, of the inclusion liquid, does not agree with the optical evidence he presents of saturated solutions with actual daughter crystals of NaCl and KCl. Vul'chin (1953) presents 38 analyses of inclusions in quartz from a variety of occurrences; an average of six of these from one occurrence, all very similar, is listed (analysis 1, table 4). The analyses were made by a combination of spectrographic and colorimetric methods. The concentrations were estimated to be in the range of 30 to 35 weight percent, presumably on the basis of the apparent volume percent of inclusions and the analyses for salts. Cu, Zn, Pb, and Ag were detected, *in HCl extracts* of the crushed samples. Sharkov (table 4, analysis 2) studied inclusions in four samples of quartz from rock crystal deposits. Some of the inclusions in these samples show as much as 25 volume percent solid daughter minerals, NaCl being rather common. The leaches were made with an unspecified dilution factor and showed, on analysis, about 200 ppm of total salts in the leachate, with weight ratios as given in analysis 2, table 4, but Sharkov (1958, p. 79 in original) concludes that "* * * the liquid inclusions contain relatively dilute alkaline aqueous solutions. They carry insignificant quantities of mineral substances, chiefly chlorides * * * ." Although analysis 2 in table 4 shows sodium greater than potassium, the other three analyses reported by Sharkov show the more unusual relationship of potassium greater than sodium, with K/Na weight ratios of 1.67, 1.13, and 2.56.

Ushakovskii (1966) found considerably different fluids in quartz from various rock crystal deposits of the Southern Urals. He presents results from 19 samples as two analyses—from crystal-bearing veins and from noncrystal-bearing veins (table 4, analyses 4a and 4b). The major differences between the two are in the amounts of Ca and HCO_3, which are relatively low in the analysis of the crystal-bearing veins.

Kramer (1965) and Holser (1963) both present analyses of inclusions from sedimentary halite beds (table 4, analyses 5–17). Kramer's analyses for fluorine show a very low F/Cl ratio, but these inclusions are *very* strong brines with much Ca^{+2}. Roedder (1963b, p. 183) reports first melting temperatures as low as —72°C and freezing temperatures as low as —54°C for similar inclusions in salt from

Goderich, Ontario, indicating the presence of major amounts of calcium salts. In the presence of such high calcium, fluorine would be precipitated out rather completely, in spite of the high concentrations of other salts. The SO_4/Cl ratios are unexpectedly high in view of the high calcium contents, although the solubility of gypsum in chloride brines is increased fourfold by the addition of approximately 2 moles NaCl and 0.2 mole $MgCl_2/l$ (Ostroff and Metler, 1966).

Because very little Br is precipitated during the early stages of normal evaporation of sea water, Holser (1963) used the Br/Cl ratio to indicate the degree of evaporation from the original sea water (given as analysis 17 in table 4 for comparison) for a series of salt samples. He presents one analysis of salt from Goderich (analysis 13) in which the calcium and magnesium may be compared with Kramer's analyses of presumably similar samples; the calcium is comparable, but Holser's Mg/Cl ratio is seven to 15 times that found by Kramer. This is not unexpected, as freezing studies on samples from these beds (Roedder, 1963b, p. 182–184) show that the solutions in the inclusions vary from concentrated sea water brine residues to essentially pure NaCl solutions. These latter are presumably the result of trapping of ground water percolating through pure salt beds. All of Holser's analyses show enrichment in magnesium relative to sea water, but this is in Mg/Cl *ratio* rather than in absolute amounts of magnesium. The Br/Mg ratios for the inclusions are close to those of present day sea water, hence. Holser concludes that this ratio presumably has been constant since Permian time. He also believes that with the possible exception of analyses 14 and 15, the inclusion fluid cannot be derived from re-solution of salt, as then it would be low in Br, Mg, and K, nor can it be connate water, which is high in Mg but low in Br (or low in both). Holser indicates (p. 91) that there were variations of unknown origin "even among inclusions from the same large crystal." In very similar samples from the Goderich area, Roedder (1963b) found that some coarse inclusions in recrystallized salt had formed from abundant minute primary inclusions under isochemical conditions, whereas other nearby inclusions had drastically different compositions and presumably represented new fluids introduced during the recrystallization.

LEACH ANALYSES

Table 5 presents representative analyses of leaches made of fluid inclusions in samples opened by crushing or ball milling. Most of these analyses have been from pegmatites and various types of pegmatitic to metamorphic quartz veins. A few additional reports of quantitative analyses of leachates are not included because they were too incomplete or because internal inconsistencies in the data raised serious questions as to their significance. Many of the most recent analyses (see table 3) are not represented in table 5 because translations are not yet available.

INCLUSIONS FROM PEGMATITES AND QUARTZ VEINS

Lisitsyn and Malinko (table 5, analyses 1–4) investigated the composition of inclusions in quartz from quartz crystal pegmatites and vein-type deposits. For the chemical analyses, they used the method of Lisitsyn (1961), in which the sample is heated for several hours at the filling temperature of the inclusions (independently determined), is overheated 20 to 30 degrees, is rapidly transferred to an agate mortar, water is added, and then the sample is finely ground before filtering. They obtained concentrations for some of these inclusion samples by several procedures: (1) from a visual estimate of the volume percent of inclusions (mostly "1 percent," "2 percent," or "3 percent") and the chemical analyses, (2) index of refraction of the liquid (assuming a mixture of NaCl and KCl), and (3) freezing points, determined by P. V. Klevtsov by an unspecified technique. The data obtained by these three methods, for two of the samples listed in table 5 (analyses 1 and 4), in weight percent salts, are as follows:

Method	Sample 574 (analysis 1)	Sample 601 (analysis 4)
1	9.8	8.8
2	16.6	11.6
3	12	12

A number of elements were found on spectrographic examination of the dry leach residue, including Cu, Ni, Ti, Mo, Pb, Ag, Zn, Sn, Fe, Mn, Ga, Sr, Al, V, Li, and Ba. Some of the samples from these same deposits showed liquid carbon dioxide, or solids such as halite, a carbonate mineral, and less frequently, sylvite, indicating the probable existence of several genetic groups of inclusions.

Sheshulin (table 5, analyses 5–7) has examined the composition of inclusions in spodumene pegmatites, by simple crushing under distilled water followed by filtering. He also estimated the quantity of water present in the inclusions to arrive at concentration values of 6, 7.23, and 16.06 weight percent salts for the three analyses given. The gases present in the spodumene were also analyzed—see analysis 42 in table 2.

In'shin (1958) made a single analysis of an extract from quartz from a vuggy Uralian vein, in which very high calcium and very low magnesium are particularly noteworthy (analysis 8, table 5). The exact methods used were not specified.

Some of Berger and Moskalyuk's analyses from Aldan quartz veins (analyses 50–52 in table 5) are particularly interesting in that they show a threefold excess of calcium over sodium and bicarbonate values near to those of chloride (analysis 50). Moskalyuk (analyses 53–56) reports some similar relationships, including the only analysis in which chloride was not detected. Even more divergent analyses are reported by Arnautov and others (1965) for a fluorite-bearing chambered (vuggy) pegmatite from Bet-Pak-Dal (analyses 60–62), in two of which sodium was not detected, yet no bicarbonate was found. The calcium determinations for inclusions in calcium-bearing host minerals such as fluorite are, of course, maximum values. Ermakov presents a particularly interesting pair of analyses on the same sample, with water and acid leaches (analyses 63 and 64). Inclusions in these samples contained more than 13 different daughter minerals.

INCLUSIONS FROM VARIOUS MINERALS AND ROCKS

Goguel (1963) used a special vacuum ball milling procedure on very small samples, particularly to obtain the gases for analysis. The eight samples for which the gas analyses are given in table 2 (analyses 24–33) were also given a quick leach with water, filtered with a membrane filter and analyzed (analyses 19–25, table 5). Goguel (1964, and written commun., 1963) has revised some of his techniques to reduce contamination and has obtained considerably lower values for boron and chloride. These chloride values are actually the sum of Cl, Br, and I; this is probably true for many of the analyses given, but it is generally not stated.

Koptev-Dvornikov and others (table 5, analyses 26 and 27) report probably the lowest chloride (relative to other constituents such as Na) of any analysis. The analyses, by N. I. Khitarov and F. V. Palei-Rengarten, were presumably made by leaching the sample that was heated to obtain the H_2O, as Koptev-Dvornikov and others (their tables 10 and 12, p. 94 and 95) present information on the water content and the calculated concentrations of salt in some of the inclusion samples. (Analysis 26 is given as 13.5 weight percent.) In view of the high concentration they calculate (up to 27 weight percent salts) and the predominance of HCO_3^{-1} anion in all of their analyses, it is surprising that they do not report crystalline bicarbonate daughter minerals in their inclusions, as the bicarbonates are much less soluble than the chlorides.

Lamar and Shrode (1953) ground samples of limestone and dolomite rocks in distilled water (table 5, analyses 28 and 29), as they were interested in the effects of the total soluble salt content on economic utilization of the rock. They believe that the calcium sulfate in the dried filtrate residue may come from intercrystal deposits, but that the other salts come mostly or entirely from the very tiny (~2 to 4μm) fluid inclusions. They found that small amounts of H_2S (0.002 weight percent to a maximum of 0.012 percent) were emitted when the fetid or oily-smelling limestones were dissolved in HCl.

Evzikova and Moskalyuk (1964) present the only recorded analyses of liquid inclusions from carbonatites (table 5, analyses 65 and 66). As might be expected, bicarbonate greatly exceeds chloride, and potassium exceeds sodium.

INCLUSIONS FROM VARIOUS TYPES OF MINERAL DEPOSITS

Khitarov, Rengarten, and Lebedeva (1958) studied several different Iceland spar deposits. The analyses of inclusions from two of the deposits reported here (analyses 30 and 31 in table 5) were made by simple leaching. Additional analyses were made of samples from other deposits (table 6 lists several) which showed concentrations of salts ranging from 4.7 to 23 g/100 ml of water (that is, as much as 18.7 weight percent).

Analyses 9 through 15 (table 5) were obtained by ball milling followed by electrodialysis; the samples were cleaned first by electrolysis (Roedder, 1958). Li, Na, K, Rb, and Cs were determined by flame photometry, using mixed standards closely resembling the unknowns. The Na/K ratio is near 1.0 for most of these samples (except the chert), and although the total *quantity* of salts extracted from "duplicate" runs varied as much as 20 percent,[34] ratios such as Na/K were much more uniform. Thus, analysis 11 has a Na/K weight ratio of 1.44. Two other runs were made on portions of this sample, and these yielded Na/K ratios of 1.43 and 1.37. The lower values for all constituents on analysis 9 are merely the result of a small amount of inclusions in the transparent quartz crystals. Although only

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[34] It should be noted that these variations are with 1,000-g portions of −10 mesh material containing many tiny inclusions; they probably stem from irregularities in ball milling procedure more than actual sample differences. The variations to be expected in smaller samples with coarser inclusions can be much greater.

rather small amounts of Li, Rb, and Cs were found in these samples, there are very gross differences in their concentrations in the various types of occurrence. The amounts of cations, even without analyses for Ca and Mg, are larger than the sum of Cl^{-1} and SO_4^{-2}; the difference is probably HCO_3^{-1} (possibly also CO_3^{-2}), but the electrodialysis procedure used precluded analyses for these anions. Analyses 14 and 15 are representative of a series of six analyses of inclusions in sheared gold-quartz veins from the Mother Lode of California. Cameron, Rowe, and Weis (1953, p. 259) decry such analyses rather pointedly, in that they are made on obviously secondary inclusions, formed by shearing. It is fairly well accepted, however, that the gold in these veins was introduced during one or more of these late stages of shearing and recementation of quartz, so that these secondary inclusions are more likely to contain the actual gold-bearing fluids that the relatively rare primary inclusions, as described by Goodspeed (1936, p. 405; Howe, 1924, p. 604; Ferguson and Gannett, 1932, p. 43; Roedder, 1958, p. 260). The very high SO_4^{-2} value (analysis 15) may well be due to contamination from finely divided sulfides. Most interesting, however, is the range in cesium values. Although the six veins sampled were all quite similar in general mineralogy and appearance, and showed Na/K ratios that varied only from 0.83 to 1.03, one of the samples was high in cesium, and another (analysis 15) was very high—it contained more than 60 times as much cesium as the minimum value. It is hoped that such gross differences, even though geochemically inexplicable at this time, might be useful in characterizing veins of unknown affiliations as belonging to one or another of multiple vein systems and hence clarify some structural problems.

Ames (1958), using the electrolytic cleaning—ball milling—electrodialysis procedure of Roedder (1958), analyzed four samples from two lead-fluorite mines. These analyses (numbers 32–35 in table 5) are very unusual in several respects. Some of them show eight to 10 times as many milliequivalents of anions ($Cl^{-1}+SO_4^{-2}$) as cations ($Na^{+1}+K^{+1}$). In addition, all the analyses are stated in units of "parts per million per kilogram of sample" (Ames' table 2, p. 478). Although the writer presumes that these units are actually *milligrams* per kilogram of sample for Na, K, Li, Rb, Cs, Cl, and SO_4, most of the values of Mg, Al, and Cu found are recorded in even tens or hundred of units. As these three elements were analyzed by spectrograph, presumably on the residue from evaporation of the electrodialysis cell products, it is assumed that these three are stated in parts per million of dry residue.[35] As no weights are given for the residues, the amount of these constituents cannot be determined from these data.

Roedder, Ingram, and Hall (1963) analyzed eight leaches of crushed samples in connection with the development of methods for quantitative extraction and analysis of single inclusions; three of these analyses are given in table 5 (analyses 36–38). Analyses 36 and 37 are from the rim and core, respectively, of a large single galena crystal. As opposite quadrants of rim, and of core, were combined in each case, there were two different samples of rim, and two of core; thus each pair of analyses of the two parts of rim, and of core, represents an approach to "duplicate" samples. Although the *amounts* of salts found varied by a factor of two, the atomic *ratios* in each pair were very similar. When the atomic ratios are compared, the rim of the crystal is seen to have a lower Ca/Na (or Ca/Cl) ratio. It is also considerably higher in SO_4^{-2}, but possible differences in the case of oxidation of the sample during crushing and leaching may be involved. Analysis 38, a composite sample of planes of presumed pseudosecondary inclusions in many crystals from a shipment of Brazilian radiograde quartz, is of interest in several respects. The Na/K ratio found agrees with the feldspar mineralogy of the Brazilian pegmatitic quartz deposits (Roedder and others, 1963, p. 366), the high calcium agrees with the known occurrence of occasional solid inclusions of calcite in this quartz, and the very high boron content (1 percent of the sodium, by weight) agrees with the well-known occurrence of tourmaline in many of the quasipegmatitic quartz veins. Most of the Brazilian radiograde quartz comes, however, from feldspar-free veins in quartzites (Prof. Earl Ingerson, written commun., 1967). The relationship between such veins and the pegmatite deposits is nebulous at best.

Some analyses of inclusions from Russian ore deposits recorded in table 5 (analyses 39–49) show bicarbonate greater than chloride. Sushchevskaya and Barsukov (analyses 43 and 44) report that bicarbonate increased and chloride decreased during the various stages of mineralization in some sulfide-cassiterite deposits. In an analysis of inclusions from a greisen deposit, Khitarov (analysis 45) reports a bicarbonate-chloride solution with very un-

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[35] If these analyses for Mg, Al, and Cu are simply assumed to be as stated, one of the inclusion samples would have 20 times more copper, and 30 times more aluminum, than sodium.

usual values for sodium and potassium, yielding a weight ratio (Na/K) of 0.16. Calcium was also very high, but as the host mineral was fluorite, this may not be too significant. Balitskii and Lyubofeyev (1962) analyzed inclusions from various stages of a polymetallic deposit (analyses 46–49). Most of these showed bicarbonate much greater than chloride and calcium greater than the sum of sodium plus potassium.

COMPLETE QUANTITATIVE ANALYSES

Table 6 lists representative analyses in which both the amounts of the major solute ions *and* of solvent (water) were determined, thus permitting an estimation of the concentration.[36]

INCLUSIONS FROM IGNEOUS ROCKS, PEGMATITES, AND QUARTZ VEINS

Königsberger and Müller (table 6, analyses 1 and 2) analyzed inclusions in quartz crystals from two metamorphic Alpine-type veins. They did not analyze for HCO_3^{-1} but did find considerable amounts of free CO_2 and of CO_3^{-2}. As CO_2 can occur as a neutral molecule, as monovalent bicarbonate anions, or as divalent carbonate anions, the distinction between these in inclusions is of considerable significance in any attempt to check analyses by the charge balance. The analytical methods used for determination of HCO_3^{-1} and for CO_3^{-2} in inclusions are not always capable of making these distinctions, and, in addition, the specific methods used are seldom stated.

Faber (1941) analyzed the leachates obtained by large-scale ball milling (with flint balls in a flint-lined mill) of 19 rocks and minerals, in connection with a detailed study of the sources of the water determined in various rock analysis procedures and an evaluation of their significance. He used glazed earthenware pots for the long-term leaching (4 to 6 weeks) and in part used decantation in place of filtering. As Correns (1940) has shown that considerable amounts of alkali can be extracted from the feldspar structure by even short-duration leaching (a few hours), at least a part of the alkalies found (analyses 3–6, table 6) are not from inclusions. The sulfate might well come, in part, from oxidation of finely ground sulfides in such rocks and possibly also from sulfates present, and so they are maximum values. The concentrations of salts were obtained by using a value for inclusion water determined by heating grains—not powder—to red heat.

Polykovskii (1962a) reports five analyses, using the method of Lisitsyn (1961), of inclusions from five generations of quartz from "chambered" (vuggy) pegmatites in Maidantal, western Tien-Shan. The concentrations were obtained from optical *estimates* of the volume of inclusions, made by counting representative areas under the microscope. The analyses, in age sequence from earliest to latest, are given in table 6 (analyses 7–11). The homogenization temperatures characteristic of each zone are given, but a later publication (Polykovskii, 1963) gives somewhat different temperatures. The Na/K and Ca/Mg ratios are exceptionally high.

Maslova (1961) extracted a large inclusion (100 mm^3) in a dark-colored quartz crystal believed to have formed as β-quartz (analysis 12, table 6). She found gas bubbles from similar inclusions to contain large quantities of H_2S and CO_2 (see page JJ48), but *no* HS^{-1}, H_2S, or CO_2 was found in the liquid phase, and the discrepancy was not resolved. The composition of this inclusion is comparable in individual constituents to several other analyses that have been obtained on pegmatitic quartz; the concentration of salts found (6.1 weight percent) is a commonly encountered range for such samples and agrees with many of the determinations of freezing temperatures for pegmatitic quartz veins (Roedder, 1963b). Prikazchikov and others (1964) also analyzed the fluid from some giant inclusions (up to 400 cm^3) in a giant quartz crystal from Volynia. They report total concentrations of salt approximately two orders of magnitude lower than Maslova's analysis, with grossly different salt composition (table 6, analysis 12a). Large inclusions are particularly subject to leakage, and it should be noted that the analyses reported by Prikazchikov and others (1964) are very similar to many ground waters.

INCLUSIONS FROM SALINE MINERALS

Kramer (1965) made four ratio analyses of the inclusions in salt beds. (See table 4.) Determinations of the quantity of water were also made in two cases, permitting determination of the concentrations in these strong brines (analyses 13 and 14, table 6). Fluorine was determined on these, and the Cl/F ratio was found to be about 2,000.

Sjögren's (1893) analysis (table 6, analysis 15) of large inclusions in gypsum from the famous Girgenti (now Agrigento) sulfur deposits in Sicily

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[36] Note that the concentrations given in table 6 are in parts per million *of total inclusion fluid;* in some of the original analyses the concentrations are stated in parts per million *of water.* At the high concentrations found in some of these inclusions, the difference is significant, but it was not always possible to tell from the text which method of statement was used.

is one of the first really quantitative analyses of fluid inclusions. In earlier work Silvestri (1882; usually quoted as "von Lasaulx, 1883") determined the composition of a very large (6 cm^3) inclusion in Sicilian sulfur; he reported the fluid to be a very dilute solution (0.1033 weight percent total salts) of NaCl (53.5 percent), Na_2SO_4 (45.1 percent), and $CaCl_2$ (1.3 percent), plus traces of K, Ba, and Sr. Yushkin and Srebrodol'skii (1965) also present an analysis of inclusions in sulfur from the Shorsui sulfur deposit, U.S.S.R. (table 5, analysis 67).

Analysis 16 (table 6), of a Na-Ca-Cl brine from a geothermal well in the Salton Sea area of southern California is included for comparison with data on inclusions from ore deposits, as these waters not only contain very appreciable amounts of ore metals (Helgeson, 1967) but they deposit some of them as rich copper-silver "ore" in the pipes (White, Anderson, and Grubbs, 1963; White, 1965a, 1965b; Skinner, and others, 1967).

Analysis 15a (table 6), by Petrichenko and Shaydetskaya (1968) is particularly complete. It was made on a large inclusion of dense (1.231 $g \cdot cm^{-3}$) fluid in water-clear recrystallized halite from the Artemovsk rock-salt deposit in the Donnbass. The authors discuss the possible source of this fluid on the basis of the ratios Br/Cl and Mg/Cl and the trace constituents. Both pH and Eh were measured. (See pages JJ38 and JJ40).

VESICLES IN LAVAS

Analyses 17–20 in table 6 present some of the data obtained on the fluids found in large (up to 10 cm) vesicles in crystalline basalt or dacite lava flows. These partly liquid-filled vesicles are found irregularly distributed in the dry rock. Upon breaking the rock, the liquid spills out and can be recovered in part by pipetting. The cavities are generally smoothly amygdaloidal, indicating they existed when the rock was still mobile, that is, while it was flowing on the surface (Kokubu and Katsura, 1956). Some of the vesicles (at Nagahama) are lined or filled with zeolites, plus a small amount of calcite, and are angular and irregular in shape. Others contain aragonite and siderite (Imari district), but the mineral (and water) content in individual cavities a few centimeters apart differs considerably (Kokubu and others, 1957). Montmorillonite in distinctly crystalline form is present in the Imazu cavities. The composition and concentration of the fluid is somewhat similar to that of the local ground water, except for relatively small differences in D/H ratio, pH and the concentration of some ions. Larger percentage differences are found in the NH_3 content. No evidence was seen of fissures or cracks to lead meteoric water in nor of leaching of the host rock to yield the amounts of materials necessary to form the quantities of zeolites found. From such considerations, the authors of these papers believe that they represent *magmatic* waters, trapped in vesicles during the fractional crystallization of the magma, and not surface water that has seeped into these cavities. Other geologists have questioned this conclusion, on several grounds: (1) the slow movement of ground water through the intergranular pores of such rocks could cause the differences noted between ground water and inclusion water, (2) once the fluids are at equilibrium with the rock (all excess oxygen removed, etc.), they would not be expected to leave any trace of their movement, (3) it is difficult to visualize a mechanism whereby magmatic waters could exist in such cavities in *surface* flows at normal temperatures of extrusion (>900°C)—even as low as 400°C, 100 bars of pressure are needed to maintain a steam density in the vesicle that would condense to only 4 percent liquid water, and (4) there are large and variable quantities of montmorillonite and zeolites, as well as the local variation in pH and composition of the cavity waters.

INCLUSIONS FROM MINERAL DEPOSITS

Some of the quantitative analyses of inclusions have been of samples from various types of ore deposits. Maslova (1963) and Fedorchuk (1963) give analyses of inclusions in mercury-antimony deposits (table 6, analyses 21–23). Maslova's analysis was made by ultramicrochemical methods on fluid extracted by pipetting out of a hole drilled into the inclusions; as Fedorchuk credits Maslova for the analyses he presents, presumably they were made by the same procedure. These are generally low in total salt concentration and high in bicarbonate, with bicarbonate frequently greater than chloride. Not infrequently these fluids also contain oil or bitumens, H_2S, and even liquid CO_2 (Fedorchuk, 1963; Roedder, 1963b, p. 188–190).

Several studies have been made of the large inclusions that occur in some Iceland spar deposits. Khitarov, Rengarten, and Lebedeva (1958) have analyzed a series of these, varying from 7 to 89 mm^3 in volume, from several types of Iceland spar occurrences. They presumably pipetted out the fluid from the inclusions opened by cleaving and then measured the volume of the cavity with a pipette. These are highly concentrated calcium chloride brines, with relatively low Na^{+1} and HCO_3^{-1} (table 6, analyses 24–26). Skropyshev (1957) made an analysis of a similar large inclusion from an unspecified locality, but apparently no correction was made for leaching of the host calcite (see analysis 27, table 6) ; in a later article (Skropyshev, 1960) he finds that the yellow and red colors of Iceland spar from some deposits in the Siberian platform are due to inclusions of bitumens, which presumably migrated from lower strata during the crystallization. Kiyevlenko (1958) and Il'in (1964) also studied the composition of inclusions in Iceland spar.

The first study to show variations in the nature of the ore-forming fluid during the formation of a given mineral, by analyses of primary inclusions from various zones, was by Grushkin and Prikhid'ko (1952; also reported in Grushkin, 1958). They analyzed inclusion leachates (table 6, analyses 28–30) from crushed samples of fluorite from three differently colored parts of zoned crystals and found that the solutions changed from alkali-chloride and fluoride to sodium-calcium bicarbonate. They report that the contamination, with respect to the listed ions, from the mineral surfaces was essentially undetectable. In order to correct for solution of the host fluorite, they made a second leach and subtracted its analysis from the first. As the first leachates were invariably higher in Ca and F than these "control" leachates, they attribute the difference to the presence of Ca and F ions among the salts of the inclusions.[37] They obtained their water analyses from weight loss on ignition of other parts of the same samples.

Comparatively few of the quantitative analyses of inclusions have been of samples from base-metal deposits. Such analyses, however, have given us most of the information we have on the composition of the ore-forming fluids. A general review of the status of our knowledge of the chemistry of the ore-forming fluids has been published (Roedder, 1965c) ; reviews of the applicable evidence from fluid inclusions, both analytical and otherwise, are given for the Mississippi Valley-type ore deposits (Roedder, 1967d) and for hydrothermal ore deposits in general (Roedder, 1967a).

Roedder, Ingram, and Hall (1963) reported a series of analyses, in which water was determined by vacuum crushing of specially selected and prepared samples, followed by the determination of the salts obtained by water leaching of the same crushed samples (table 6, analyses 31–37). This work showed that there were very appreciable differences between the Mississippi Valley-type ore fluids and those responsible for the formation of the normal magmatic-hydrothermal ore deposits. The former are always much more concentrated brines, with much higher ratios of Na/K, Na/Ca, and Na/Cl. One sample in this series, from Cartagena, Spain (analysis 35), is particularly interesting. Although this sample showed a composition "very similar" to that of the samples from Mississippi Valley-type deposits (Roedder and others, 1963, p. 372), they thought that it came from the hydrothermal lead-zinc-silver veins in andesite and contact metasomatic rocks that have been reported for this district (Berg and others, 1950). It has since been realized however, that data given in another paper (González, 1949) indicate that several widely divergent types of ore deposits occur in the area and that some of them are very similar to the Mississippi Valley-type deposits. As the sample analyzed was a museum specimen labeled simply "Cartagena, Spain," we can only suggest that perhaps it came from the Mississippi Valley-type deposits.

Hall and Friedman (1963) used the same procedure to analyze a series of samples, of known paragenetic positions, from two Mississippi Valley deposits. They found that systematic changes occurred in the total concentration, D/H ratio, and several other constituents during the formation of the Cave-in-Rock deposits (table 6, analyses 38–41) that they believe reflect the complex origins of the fluids. A similar sequence in the Upper Mississippi Valley district (analyses 42–44) also showed variations with paragenetic stage, parallel in part with the other series.

Uchameyshvili (1965) analysed inclusions in a large number of barite samples from various Soviet mines. Many of these solutions were rather concentrated, and some were high in bicarbonate (table 6, analyses 45–49). The Na/K ratios are exceptionally high in a number of these solutions.

The most extensive and thorough studies of the chemical compositions of fluid inclusions in hydrothermal ore deposits are presented in two reports received too late to be included in the tables. They are on the Providencia district in Mexico (Rye and Haffty, 1968), and the Bluebell mine in British Columbia (Ohmoto, 1968b; Ohmoto and Rye, 1970).

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[37] The first leach from a powdered mineral, made under nonequilibrium conditions, would be expected to have a higher apparent solubility even without inclusions; very small grains, corners and edges, and other surface irregularities or points of strain in the lattice would be more soluble, than the bulk of the sample.

DISCUSSION

INERT GASES

The recent developments in gas analysis techniques have given considerable impetus to study of the geochemistry of the inert gases, as an aid in the solution of a variety of geologic problems. Some of these gases can be trapped in specific structural holes in the crystal during its formation, as in beryl (Damon and Kulp, 1958; Feklichev, 1963). They may also be trapped on imperfections and grain boundaries, as in pyroxene (Hart and Dodd, 1962; McDougall and Green, 1964; Lovering and Richards, 1964) and possibly in sodalite (York and others, 1965), or they can be deposited in the structure by radioactive decay after crystallization. However, at least a part of the inert gases found in most rocks occurs in the fluid inclusions. The interpretation of such analyses is even more ambiguous due to multiplicity of origin of the inclusions. Thus Funkhouser, Naughton, and Barnes (1965) and Funkhouser and Naughton (1968) believe that the excess argon they found in xenoliths from basalt, which yielded grossly erroneous (too old) K/Ar "ages," is present in fluid inclusions. Primary, high pressure gas inclusions are common in these samples (Roedder, 1965d) representing the trapping of microscopic droplets of a separate immiscible CO_2-rich gas phase in the melt. It can be expected that argon (and other inert gases) in the system would become highly concentrated in this gas phase. The distribution coefficients to be expected for such gases between a granitic melt and droplets of a separate immiscible hydrous saline melt (as found as primary inclusions in quartz of granites from Ascension Island, Roedder and Coombs, 1967), or between a water-rich phase and droplets of a separate, immiscible CO_2-rich phase (as found as primary inclusions in many pegmatite minerals, see table 1) are not as self-evident. All these "systems" should be examined experimentally.

The K^{40} present in the inclusion fluids forms radiogenic argon with time, and in theory, the K/Ar age of the inclusion could be obtained from such measurements. Radioactive age determinations on sylvite by the K/Ar method are subject to errors in one direction by loss (low retention) of the new radiogenic argon (Borshchevskii and Borisova, 1963) and in the other direction by the presence of primary argon in gas inclusions (Nesmelova, 1959; Thoma and Eckart, 1964; Ackermann and others, 1964). Radiogenic argon can be expected to concentrate in any pore fluids present in rocks, particularly during metamorphism, and such high argon fluids may be trapped as fluid inclusions in metamorphic minerals, giving K/Ar "ages" that are too high by a factor of as much as 1,000 (Rama and others, 1965). Similarly, argon in inclusions may explain some of the discordance between the apparent ages of two minerals from the same pegmatite. Thus Laughlin (1967, 1968) reports excess argon up to a maximum of 0.16 ppm in pegmatitic feldspar, in the same range as that found by Rama, Hart, and Roedder (1965). Gerling, Morozova, and Sprintsson (1968) propose another source of Ar^{40} generation (other than K^{40}) to explain the excess argon they find in some minerals. They propose that this hypothetical element has a half-life of 400 m.y.

The ore-forming fluids can also be expected to become enriched in argon, regardless of their origin; some evidence of this was found by Lippolt and Gentner (1963) and Lippolt (1966) who report up to 5×10^{-7} cm^3 radiogenic "excess" argon in fluorites from the Schwarzwald, which they consider to be primary argon trapped with the fluid of the inclusions. Similarly, Rama, Hart, and Roedder (1965) report excess argon in inclusions in a Mississippi Valley fluorite deposit. They also report that inclusions from an epithermal ore deposit associated with volcanism at Creede, Colo., show no measurable argon. Although ore fluids from such a magmatic source should be enriched in argon, there is independent evidence that these fluids boiled during the deposition of the enclosing minerals (Roedder, 1960a); such boiling would be expected to scavenge argon (and perhaps H_2S) from the fluids rather effectively.

An additional problem in the interpretation of inert gas analyses in inclusions comes in possible contributions of gases to these fluids by former exposure to the atmosphere (Florensky, 1956; Mazor and Wasserburg, 1965). Further studies of the relative abundances of all of the inert gases in inclusions and possibly related natural waters should be fruitful.

ALKALIES

Since the early work of Sorby, Zirkel, and others, showing that the fluid inclusions in many rocks contained strong and even saturated NaCl brines, practically all additional studies of inclusion composition have shown that alkali chlorides are the predominant salts in solution. There are only a very few exceptions, and many of these are represented by analyses in the tables. Sodium is by far the most abundant, generally exceeding potassium by a factor of 1 to 10, but as the analyses in the tables were selected to show both the typical compositions and the more unusual ones, they do not represent a valid cross section. The rarer alkalies are seldom determined, in part due to the small quantities available. The newer techniques are much more sensitive, however, and determinations of all three, lithium, rubidium, and cesium, should become common. Although the absolute amounts present are of comparatively little significance, weight ratios such as Na/Li, K/Rb, and K/Cs may be useful in recognizing different generations of fluids, as, for example in the Mother Lode district (Roedder, 1958, p. 262). The amounts of lithium found in many pegmatitic quartz samples (for example, Stavrov, 1961) could be partly or entirely from fluid inclusions.

As sodium and potassium are among the most abundant constituents present in inclusions and may be determined by flame photometry with relative ease, precision, and accuracy, the Na/K ratio is one of the most useful parameters (Sawkins, 1965b, 1966a, 1966b). For example, the fluids that formed the Mississippi Valley-type ore deposits seem to be characterized by much higher Na/K ratios (about 17, by weight) than those having magmatic-hydrothermal affiliations (Roedder and others, 1963; Hall and Friedman, 1963; Roedder, 1967a). The low ratios for the latter (not infrequently less than 1) presumably reflect interactions with wallrock minerals at the prevailing temperature and acidity (Hemley and Jones, 1964; Ellis and Mahon, 1967); but it is not always certain which changed most, the fluids or the wallrocks. It is possible that careful comparison of inclusion fluids in replacement minerals from wallrocks with those from adjacent vein minerals may help in such problems. The higher Na/K fluids involved in the deposition of the Mississippi Valley-type samples, and the still higher ratios in normal connate waters, probably are derived by other reactions (Roedder and others, 1963, p. 371; Roedder, 1967d, p. 359). Sawkins (1968) believes the extra potassium in the Mississippi Valley-type samples comes from contributions of magmatic waters.

Arnautov and others (1965) present an unusual series of 10 analyses of seven stages of quartz and three of fluorite from a chambered pegmatite in Bet-Pak-Dal. (See analyses 60–62, table 5.) Although the alkalies were determined by flame photometry, a number of these samples showed no detectable sodium. The sensitivity of the specific procedure used is not stated, but ordinarily flame photometry should be able to detect 0.002 mg of sodium under these conditions, even without concentration by evaporation.

The Na/K ratio for the fluids of metamorphic pegmatites and quartz veins fluctuates rather widely on both sides of 1, as might be expected from the diversity of processes involved, but contamination, particularly from feldspar, can introduce serious errors. The original fluids present may have been connate brines or even fresh surface waters, with salts added from the solution of evaporite beds. Engel and Engel (1953) have shown that actual NaCl is almost always present, along with anhydrite and gypsum, in drill cores of the metamorphic marble sequence of the Precambrian Grenville series. They believe this to be of sedimentary origin, though some was redistributed. The composition of the pore fluids in metamorphic rocks is the result of a long series of reactions with the minerals present. Although there is considerable total time available, these reactions occur during changing conditions of temperature and pressure and may not have achieved equilibrium. The fluids may be expelled from the rock to form new minerals, as in pegmatites, at any time during the history of the area, and small portions may be trapped at various stages as inclusions in relatively nonreactive minerals, such as quartz.

The residual fluids from the crystallization of most igneous rocks also are strong sodium chloride brines, as evidenced by the common presence of halite daughter crystals in their inclusions (Sorby, 1858; Zirkel, 1870b, 1873, 1876, and 1893). Much less commonly the halite crystal is accompanied by a sylvite crystal.[38] The ratio of sodium to potassium in such fluids is, of course, related to the ratio in the silicate melts from which they are derived (Gammon and others, 1969).

As a result of such a diversity of possible processes, detailed studies of the inclusions will be needed to piece together the history and significance of the various fluids present at any place. These studies should be of aid in a variety of geologic areas and problems, and conceivably they might help to distinguish metamorphic granites from truly magmatic ones.

ALKALINE EARTHS

Calcium is usually the major cation in those inclusions where sodium is not predominant, and in a very few special cases it greatly exceeds sodium.

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[38] Under some special but at present unknown conditions, a sodium or sodium-calcium carbonate melt might form, presumably as a residual liquid (Knorring and du Bois, 1961; Dawson, 1962; Du Bois and others, 1963).

With but few exceptions it exceeds magnesium, frequently by a large factor. Although there has been little attention given it, the Ca/Mg ratio in the ore-forming fluid is of considerable significance in controlling dolomitization. Care is needed to obtain valid determinations of this ratio in inclusions, however, due to several common sources of contamination. Mississippi Valley-type ores formed from fluids with a rather uniform Ca/Mg weight ratio between 4 and 8, but other types of deposits deviate widely on both sides of this range (table 6). Graf, Meents, and Shimp (1965) propose that the Ca/Mg ratio in saline formation waters is increased during their evolution by exchange of magnesium for calcium, forming dolomite, and by simple extraction of magnesium, forming chlorite.

Strontium is probably present in many inclusions, but is seldom determined. Gerling, Shukolyukov, and Matveyeva (1962) report some age determinations on inclusion-bearing minerals (beryl, tourmaline, and quartz) by the Rb/Sr method that gave absurdly high values. As the quantities of strontium involved are very small it is relatively easy to stipulate conditions whereby an aqueous phase, "sweated" out of a metamorphic rock, could contain significant amounts of strontium enriched in radiogenic Sr^{87} to explain the results obtained. Experimental determinations of the distribution coefficients for strontium between precipitated calcite and the solution have been made over a range of temperatures by Bodine, Holland, and Borcsik (1965), in an effort to delineate the composition of the ore fluids from measurements of the composition of natural crystals of calcite formed by them. Similarly, Helz and Holland (1965) have used natural mineral assemblages and experimental data on the solubility of strontianite to place some limits on the Ca/Sr ratio of the fluids. Eventually, it may be feasible to compare such estimates with direct analyses of inclusion fluids, although it would be necessary to use inclusions in simultaneously precipitated minerals other than calcite to obtain an accurate Ca/Sr ratio. Blount and Dickson (1966) report that celestite exhibits retrograde solubility in water, but the solubilities found (up to 248 ppm) are adequate to permit analytical detection of strontium if such fluids were trapped in fluid inclusions.

Only a very few determinations of barium have been made, but it should be determined more frequently. Barite is a useful gangue mineral in that its presence places some limits on the concentration of the much more important unknown in the ore fluids, sulfate ion (Barton, 1957).

BORON

Although boron would be expected in many types of natural water-rich fluids, particularly residual liquids from the crystallization of metamorphic and igneous rocks, comparatively few quantitative determinations of it have been made. The presence of considerable boron in pegmatitic fluids is evident from fluid inclusions in pegmatitic minerals showing daughter crystals identified as fluoborates (Slivko, 1955, 1958), borax (Yakubova, 1952, 1955), and teepleite ($Na_2B_2O_4 \cdot 2NaCl \cdot 4H_2O$) (Kalyuzhnyi, 1958a). It is also evident in the common presence of tourmaline in the pegmatites, in analyses showing hundreds of parts per million of boron in various pegmatite minerals (although at least some of this boron is attributed to inclusions (Oftedal, 1964, p. 221)), and in the rather common occurrence of vonsenite and other borates, or borosilicates, in various skarn assemblages at the contacts of pegmatites or granitoids (Marakushev, 1963; Utekhin, 1964; Chesterman, 1965). The association of borate minerals, chiefly ludwigite, with certain skarn ores has been used by Geijer (1959) as evidence of a primary (contact metamorphic) origin for the skarn, as opposed to origin by metamorphism of sediments.

Boron is also probably present in many ore fluids. Lindgren and Whitehead (1914) tentatively identified colemanite in fluid inclusions in crystals of the borosilicate danburite from a jamesonite deposit, and datolite is known to occur in skarn iron ores (Yakovleva, 1961). Ellis and Mahon (1967) show that boron would be leached out of many rocks by hot water. From these results, and the occurrence of boron concentrations in some hot springs (Koga, 1957; White and Roberson, 1962; Ellis and Mahon, 1966), one could conclude that some ore fluids may have very appreciable amounts of boron. One analysis of inclusions from sphalerite from Santander, Spain (analysis 32, table 6), shows 500 ppm boron (corresponding to 2,700 ppm BO_3^{-3}), and all others are lower. As the quantities involved are very small, the possibility of contamination is significant. For this reason, normal laboratory borosilicate glassware cannot be used.

CARBON

The compounds of carbon that occur in inclusions are particularly useful in delineating the condition of formation. (See also "Organic Gases and Liquids" under "Analytical Data.") Thus the equilibria between carbon, hydrogen, and oxygen under magmatic conditions will yield various amounts of CO_2, CO, H_2O, and CH_4, in addition to excess graphite, hydrogen, and oxygen (French, 1965; French and Eugster, 1965; Eck and others, 1966; Nokleberg, 1969). Depending on the quantities involved, these reactions may control, or be controlled by other variable valence elements in the environment such as iron, manganese, and sulfur. Thus methane may well be present in significant quantities in hot ore fluids in equilibrium with ordinary sulfide mineral assemblages (Holland, 1965).

Hot gases or fluids from magmas may maintain their high temperature equilibrium composition, in part, if the cooling is rapid. Unfortunately, however, most fluid inclusions in magmatic rocks probably represent fluids that have been cooled rather slowly, with varying degrees of contact with a variety of mineral surfaces, making interpretation difficult. Oró and Han (1966) have shown that pure methane will form rather large amounts of multiring aromatic hydrocarbons with just one pass over silica gel at 1,000°C. Small amounts of aliphatic hydrocarbons are also formed if ammonia and water are present. More than 97 percent of their synthesized hydrocarbons contained even numbers of carbon atoms. Similarly, Giardini, Salotti, and Lakner (1968) have shown that significant quantities of CH_4 and C_2H_6 will form by the reaction of calcite and hydrogen at about 500°C.

Barker (1965a) has attempted to calculate the apparent temperature of final equilibration of the gases from mass spectrometric analysis of gases liberated on heating samples in vacuum. In another publication (Barker, 1966) he indicates that the gases found were not in thermodynamic equilibrium at the temperatures and pressures corresponding to those in the geological or experimental environments and that they must come from several sources. As pointed out in "Destructive Methods of Determining Composition," the problems of unambiguous sampling and extraction of inclusions for analysis of the ratios between the various species are exceedingly difficult.

At still lower temperatures, deviations from the equilibrium ratios are probably more common and persistent (Eck and others, 1966). Hoefs (1965) has found small amounts of free carbon (about 200 ppm) in many igneous rocks; although this is quantitatively adequate to exert considerable control on the gas equilibria, it seems likely that solid-gas equilibrium in particular would not be maintained on cooling.

Reliable data on the ratios between bicarbonate, carbonate, and free carbon dioxide in liquid inclusions would be valuable in calculating the pH (Uchámeyshvili and Khitarov, 1965), and in predicting the concentrations of a variety of ionic species, and hence the solubilities of many solids (for example, Barton, 1957; Brotzen, 1962; Holland and Borcsik, 1965a, 1965b; Krol and Gurkina, 1961). Unfortunately, only rarely are all three determined. Bicarbonate is usually the major anion in those analyses where chloride is not the major one, but the method used for the bicarbonate analysis is seldom stated, and the common presence of free CO_2 (liquid and gaseous) may make at least some of these values too high. Khodakovskiy (1965) found that bicarbonate exceeded chloride in 135 out of 400 analyses of inclusions taken from the literature. Although a number of these analyses are of dubious validity due to sampling and analytical problems, bicarbonate is a major anion that should be determined in all analyses of inclusion fluids.

Although there are some CO_2 inclusions that indicate the existence of immiscibility between H_2O-rich and CO_2-rich fluids at the time of trapping (pl. 5, fig. 1), there are many more that give clear evidence, by their uniform phase ratios at surface temperatures (pl. 7, figs. 9, 10), that a homogeneous CO_2-H_2O fluid was trapped. The ratio CO_2/H_2O is very important in many geological problems such as ore transport, wallrock alteration, and metamorphism (for example, Dobretsov, 1966). It has been determined frequently by analysis of the gases evolved on decrepitation (Vertushkov, 1966; Touray, 1968) (see also tables 2, 3, and 8). However, the difficulties of differentiating primary and secondary inclusions, as well as homogeneous versus heterogeneous fluids at the time of trapping, may make qualitative estimates based on measurements of the relative volume of liquid CO_2 and liquid water phases at room temperature more accurate.

The presence of carbonate in the fluids trapped in inclusions is indicated, qualitatively, by the common occurrence of calcite or other carbonates as daughter minerals. Although calcite shows retrograde solubility in most water solutions, the solubility is apparently normal (nonretrograde) in strong brines. A wide variety of soluble or slightly soluble carbonates of the alkali and alkaline earth metals, for example, dawsonite ($NaAl(OH)_2CO_3$) (see Smith and Milton, 1966), might be expected to occur as daughter minerals in some environments and may account for some of the numerous unidentified phases reported.

Carbon as hydrocarbons and other organic compounds is surprisingly common in inclusions and has been reported even in scheelite crystals (Matveyev, 1947). Small quantities (~0.01 weight percent) of bitumens have been reported in a number of hydrothermal fluorite ores from igneous rocks in Tadjikistan (Mogarovsky and Markov, 1966). Analyses of these compounds may eventually be of great value in placing some limits on the environment of formation of the sample and its subsequent heat treatment, as certain of them are thermally unstable or may distill off (Mueller, 1954b) and some appear to decompose with time, after trapping (Roedder, 1962b, p. 40). At least small amounts of methane are found in many analyses of the gases from inclusions, although new methane can also be formed during the extraction process if heat is used, either by inorganic reactions or by the decomposition of kerogen (Hoering and Abelson, 1964).

Organic matter should, in general, reduce sulfate to sulfide. Although these reactions are apparently very slow, unless aided by bacteria, A. I. Germanov (1965) has proposed that such oxidation of dark organic matter by sulfate, and direct solubility in the hydrothermal fluids, are responsible for much of the bleaching of wallrocks. Barton (1967) has proposed that the Mississippi Valley-type ore metals have been transported by an organic-bearing fluid in which the sulfur was present as sulfate; slow reduction of the sulfate in this nonequilibrium solution avoids the problems of simultaneous transport of sulfide and metal in the same fluid. The solubility of methane alone in 50,000 ppm brines is adequate to provide, at saturation under pressures equivalent to a depth of only 1,000 feet (Duffy and others, 1961), reducing capacity to form about 2 g S^{-2} per liter of brine. In addition, may subsurface waters are saturated with respect to methane (Buckley and others, 1958), and inclusions in Mississippi Valley-type deposits show evidence of a separate immiscible oily liquid; this could have continued to supply reducing agents to the brine as fast as they were consumed.

It has been suggested that organic complexes could also be responsible for transport of part of the sulfur itself, as sulfur-bearing organic compounds (Skinner, 1967). This mechanism may have operated in some deposits, but in the Pine Point lead-zinc deposits, Northwest Territories, Canada, Folinsbee, Krouse, and Sasaki (1966) found that the isotopic composition of sulfur in the pyrobitumens was considerably lighter than that of the ore.

Dombrowski (1960, 1966) (see also Reiser and Tasch, 1960, and Kühn, 1968) has reported the occurrence of bacteria and bacteria spores, in part viable, in inclusions in halite from several deposits, including some of Paleozoic age. Such spores presumably are present but are dormant in the brines as they move underground but would be available to resume normal metabolism whenever a change in the conditions permits. Unfortunately, however, there may be some ambiguity as to the origin of these bacteria, because halite is easily fractured and rehealed. Thus although the halite itself may have a bromine content indicating primary origin (not recrystallized), the occurrence of the inclusions (Dombrowski, 1966, p. 217) indicates fracturing and rehealing at some later time. Furthermore, the aseptic removal of the external parts of the rocksalt sample before bacteriological cultures are attempted, as reported, would not in itself preclude a relatively modern origin for the fluids, and bacteria, in these inclusions.

Although no analyses have been made of inclusions for the various water-soluble organic molecules, it is probable that they have been present in at least some of these environments. As the rates of reaction vary widely for different organic structures, it is possible that some of these may have been more effective reducing agents than either the methane or the oil. For example, glucose and other carbohydrates occur in Devonian sediments (Swain and Rogers, 1966), and solid calcium oxalate, whewellite, is known to occur with fluorite, calcite, dolomite, ankerite, barite, marcasite, quartz, and sphalerite in concretions in Paleozoic sediments (Hyde and Landy, 1966). A basic problem in investigating such a possibility—in addition to the severe analytical difficulties (Hare, 1965)—is that the most effective reductants would be the very ones to be eliminated first from the solutions before trapping. Thus Drozdova, Yakubovich, and Konstantinova (1964) have shown that of the seven amino acids found in fossil organic matter from limestone host rocks for the Pokrovo-Kireev (fluorite) deposits, only three are found in the fluorite, presumably because of oxidation of the others during deposition.

One fascinating aspect of carbon in fluid inclusions is the possibility that it might shed some light on the evolution of the "fluids" that have formed the carbonatites and carbonate lavas. It has been shown (Roedder, 1965d) that the basaltic(?) melts from which the olivine and other minerals of olivine nodules have crystallized throughout the world were all saturated with respect to CO_2 and had minute droplets of a dense, supercritical CO_2 fluid phase in them, even at considerable depths. A coalescence of such droplets could provide the gas phase that obviously has been very important in the emplacement of most carbonatites and carbonate lavas (Dawson,

1962) and may also be the source of the carbonate itself. There has been only one detailed study of the fluid inclusions in the minerals of carbonatites (Evzikova and Moskalyuk, 1964; analyses 65 and 66, table 5). (See also Balashov and Pozharitskaya, 1968.) Evzikova and Moskalyuk found that the liquid water inclusions contained high-potassium, bicarbonate-rich salts, but as water was not determined, concentrations could not be obtained. Liquid CO_2 has been found as inclusions in other carbonatites (Roedder, 1963b, p. 195, and 1965d, p. 1777) and probably will prove to be common when inclusions in these rocks are studied in detail.

NITROGEN AND AMMONIA

A variety of geochemical processes would be expected to cause the concentration of nitrogen, both as elemental gas and as ammonium ion, in water-rich pore liquids and hence in inclusions (Letnikov, 1968). Very few analyses of inclusion liquids list nitrogen compounds, and although nitrogen and ammonia are reported in many analyses of the gases evolved on heating rocks (table 3), there are numerous possibilities for contamination. The prevalence of at least small amounts of both nitrogen and ammonia in those inclusion analyses where these components were determined indicates that they are probably common in inclusions, and it adds to the difficulty of interpreting analyses of nitrogen in rocks and minerals. Much or all of the fixed nitrogen in sedimentary and metasedimentary rocks is undoubtedly of organic origin, and Florensky (1956) feels that the excess nitrogen in natural gas over that originally from the atmosphere as determined from the argon-nitrogen ratio probably comes from biologic materials. However, primary nitrogen does occur in magmatic rocks and minerals. Wlotzka (1961) found tens of parts per million nitrogen as ammonia in igneous rocks, particularly in the micas, where it substitutes for potassium in the interlayer sites (Vedder, 1965). Tovarova (1958) reports analyses of the salts obtained from water leaches of pyroclastic rocks showing that ammonium comprises 5 to 10 percent of the total cations. The occurrence of nitrogen in volcanic gases, however, is generally attributed to atmospheric contamination.

Although the concentration of ammonium ion in hot spring waters is generally only a few parts per million (White, 1957a; Hewett and Fleischer, 1960; Ellis, 1962; White, Hem, and Waring, 1963), Wlotzka (1961) found that chlorite and sericite formed by hydrothermal alteration had higher ammonium than the unaltered rocks, and the ammonium feldspar buddingtonite has apparently been formed by hot spring fluids at several mercury deposits (Erd and others, 1964; Barker, 1964; White, 1967). The ammonium content of the associated waters is high and sometimes exceeds 400 ppm (Roberson and Whitehead, 1961; White and Roberson, 1962). Such ammonia need not originate with the hot spring fluids; Ellis and Mahon (1964, 1966, 1967) find that the ammonia in rocks (along with Cl, B, F, and As) can be removed easily in hydrothermal laboratory experiments that result in only slight alteration, and they believe that it exists, to a large extent, on the surfaces of the rock minerals. Like nitrogen, it can also be derived from the diagenesis and metamorphism of organic matter (Ivanov, 1958, 1960) in contact with hydrothermal fluids.

Only a few determinations of ammonium in inclusions are recorded. Forster (1871), in one of the very early analyses, found ammonium in the distillate from a Swiss smoky quartz. Wlotzka (1961) reports 22 and 27 ppm NH_3 in two pegmatite quartz samples. Loskutov (1962) reports ammonium compounds in inclusions in natrolite. The Japanese analyses of liquids from large vesicles in basalt (table 6, analyses 17–19) present more detailed information on the nitrogen species present than in any other analyses, but as is pointed out in an earlier section, the origin of these fluids is somewhat in question.

The most extensive study of nitrogen compounds in fluid inclusions is that of Kranz (summarized briefly in 1967; reported fully in 1968a). In this work he found that many different nitrogen-bearing compounds were released on crushing various feldspar samples, including many organic amino compounds such as amines and nitriles of low molecular weight. He suggests that these originated from abiogenic chemical processes involving hydrocarbons, ammonia and water in fluid inclusions, as a result of exposure to natural radioactivity. As such they pose serious questions concerning the usual interpretation of a biogenic origin for such compounds.

From the available data, it is apparent that ammonium should be looked for routinely in all inclusion analyses, as it is at least a possible constituent to occur in fluid inclusions from almost any environment. As Haas (1966) has shown that ammonium can help in the difficult problem of transport of iron in hydrothermal fluids, possibly as a ferrous tetraamine complex ($Fe(NH_3)_4^{+2}$), both NH_3 and NH_4^{+1} should be sought.

A series of 17 analyses of fluorite and other vein minerals from various hydrothermal ore deposits reported by Klyakhin and Levitskii (1968) all show surprising amounts of ammonium ion. They report NH_4 in amounts up to 20 percent of the total cations present in the inclusion fluids, and they propose that it may play a significant role in the transport of ore metals. Klyakhin and Levitskii (1968) also quote an analysis by I. S. Gol'dberg (stated as 1963, but without reference) of an extract from galena of the third generation at the Takob fluorite-galena ore deposit, which showed appreciable ammonium. The analysis, stated in percentage equivalents, was Ca^{+2}, 73.6; NH_4^{+1}, 26.4; and Cl^{-1}, 39.1; F^{-1}, 21.1; HCO_3^{-1}, 39.8. Several hydrothermal studies have shown that NH_4^{+1} increases the solubility of various sulfides very greatly. Ptitsyn (1968) reports a solubility of 0.185 mole percent for sphalerite and for pyrrhotite in an ammonium chloride solution at 380°C, and Laudise, Kolb, and DeNeufville (1965) report a solubility of about 60,000 ppm ZnS in a 5 mole NH_4Cl/kg solution at 450°C. This was about 70 times that found for a 4.5 mole NaCl/kg solution at the same temperature.

SULFUR, SULFATE, AND SULFIDE

Although not always stated, the sulfur analyses reported for fluid inclusions are commonly total sulfur, expressed as sulfate. Both sulfide and sulfate sulfur are determined in some analyses of minerals and rocks (Ricke, 1960), but the small size of liquid inclusions has precluded determinations in such samples (Roedder and others, 1963). Except for the negative results of Maslova (1961), sulfide sulfur has not been determined in any liquid inclusion analysis, but the occasional evolution of H_2S gas on opening indicates its presence. No attempts have been made to analyze for other sulfur species, although various sulfur-rich ore fluids have been proposed, for example polysulfides (Cloke, 1963) and bisulfides (Barnes, 1965). The total concentration of sulfur and the ratios between the various sulfur species present in inclusions at room temperature (or better, at the homogenization temperature) would be of great importance to many studies of the mechanism of ore transport and deposition. At present, analytical procedures are not adequate for these determinations on ordinary samples, and considerable effort should be put towards developing more sensitive analytical tools, particularly as some calculations by Eck and others (1966) show that rather surprising concentrations of such unexpected species as CS_2 and COS are present at equilibrium in the system C-H-O with 0.0001 percent added S. (See also page JJ44.) Further indication that a variety of sulfur species are probably present in inclusions is given by the large number of polythionic acids found in some hot spring waters (Uzumasa, 1965, p. 103).

All that can be said with certainty at present is that although free H_2S is known in inclusions from ore deposits and other environments, it occurs rarely, and it usually occurs only in small amounts. The principal evidence for this is the sensitivity of the human nose, which can detect approximately 10^{-10} g of H_2S in 10 cm^3 of air. Even if only 0.1 weight percent of inclusion fluid was present in a sample, and only 0.1 weight percent of this fluid was free H_2S, a recognizable odor of H_2S would be released by the formation of only 1 cm^2 of new surface. Indeed, on mining such an ore, the released H_2S would be a definite health hazard. H_2S is present in some mining operations, particularly from H_2S-bearing mine waters, but even where very strong odors are encountered the concentrations need not be high. The highest concentrations are in oilfield waters, some of which contain hundreds of parts per million H_2S (White, Hem, and Waring, 1963). Some specimens give off an odor resembling H_2S when fractured (see table 1), and it has also been found in the gases evolved from some rocks and minerals by various procedures (see tables 2 and 3), although these gases are not necessarily all from fluid inclusions.

Sulfate is generally present in fluid inclusions, and in some examples it is the major anion; only rarely is it not detected. (See tables 5 and 6.) Although there is an unproven possibility that sulfide sulfur originally trapped in inclusions might be oxidized to sulfate by loss of hydrogen through diffusion, there is ample evidence that many of the fluids in nature were actually sulfate-bearing, at least at the site of ore deposition. Primary sulfates such as barite are common in low-temperature deposits,[39] and there is increasing evidence of the common presence of primary sulfate (anhydrite and (or) gypsum) in high-temperature copper and molybdenum deposits, for example, at El Salvador, Chile (J. P. Hunt, in Roedder, 1965c, p. 1398); Butte, Mont. (Charles Meyer, 1965); Christmas mine, Arizona (Perry, 1969); Hudson Bay Range, Canada (Kirkham, 1969); several Armenian deposits (Karamyan and Faramazyan, 1959); the Noril'sk Cu-Ni ores, U.S.S.R., (Zolotukhin, 1962,

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[39] There is one report of "SO_3 gas" (analytical procedure not stated) in large gas bubbles in celestite crystals associated with calcite, gypsum, and native sulfur in the Dnester region (Lazarenko and Slivko, 1958).

1965); the Alekseyevka copper deposit of the Central Urals (Stoliarov, 1964); the Kal'makyr deposit (Badalov, 1960); and the majority of North American porphyry copper deposits (Guilbert and Lowell, 1968). Primary anhydrite (and gypsum?) apparently crystallized in some of the metamorphic quartz veins, as in Brazil, but frequently was preserved only as hollow casts of quartz retaining the anhydrite crystal shape. Many of the "unidentified birefringent daughter crystals" reported in fluid inclusions have properties appropriate for anhydrite, for example from the Bingham, Utah, porphyry copper deposit (Roedder and Creel, 1966) and possibly also Cornwall, England (Sawkins, 1966b). Sulfate would be expected particularly in fluid inclusions from late stage processes from igneous rocks high in sulfate. Gerasimovskii (1966) reports over 1,000 ppm total sulfur in the agpaitic nepheline syenites of the Lovozero massif; over two-thirds of this was present as sulfate. Kawano (1948) reports the discovery of actual phenocrysts of anhydrite in glassy volcanic rocks from Himeshima, Oita prefecture, Japan.

Although the solubility of anhydrite is generally low (<0.7 weight percent) (Bock, 1961) and retrograde, Blount (1965) and Blount and Dickson (1969) have shown that the idiosyncrasies of the solubility curves for anhydrite even in the simple system $CaSO_4$-NaCl-H_2O make it feasible for anhydrite to crystallize from ascending, cooling hydrothermal solutions, with a wide range of compositions. As such, it might be expected to form both as a daughter mineral and a gangue mineral in some ores.

The presence of primary sulfate minerals and the sulfate in the fluid inclusions of an ore deposit do not, of course, require that the incoming solutions contain sulfate. Holland (1965) has shown that the relatively large sulfate content of fluids in inclusions from low-temperature hydrothermal minerals could be characteristic of the fluids and need not be produced by secondary oxidation, but such oxidation must be common. There are a variety of possible oxidation mechanisms, for example, by mixing with oxygenated ground waters; regardless of the mechanism, the resulting drastic changes in the ore fluids would generally cause precipitation of the ore metals (Barnes, 1965; Raymahashay and Holland, 1969). Similar oxidation of H_2S to sulfate occurs in many hot spring and fumarolic areas, where it has gross effects on the pH of the waters (Ichikuni, 1959) and may yield fluids with a pH of less than one (White and Roberson, 1962). (The reverse process, reduction of sulfate, is described briefly on page JJ61.) Although most hot spring minerals are rather unsatisfactory for fluid inclusion study, analyses of the sulfur species present in their inclusion fluids would be of considerable importance in reconstructing the circulation patterns.

Even if total sulfur is the only sulfur determination that can be made on inclusion samples from ore deposits, the data are useful, as some of these fluids appear to be rather low sulfur or even "sulfur deficient" (total sulfur less than heavy metals). Thus the brines from the Salton Sea geothermal area in California (White, Anderson, and Grubbs, 1963; H. C. Helgeson, in Roedder, 1965c, p. 1385) carry far more heavy metals than sulfur. The low sulfur may be due to a loss of H_2S to a through-streaming gas phase (D. E. White, in Roedder, 1965c, p. 1394; White, 1968a, 1968b). Raymahashay and Holland (1969) calculate that the total dissolved sulfur in solution in the ore fluids at Butte, Boulder, and Ely probably was less than 10^{-2} mole/kg solution (~ 600 ppm). Ingerson (1954) proposed a mechanism (discussed in detail by Lovering, 1961), involving two different fluids in the formation of sulfide deposits, either sequentially or by mixing. One is assumed to be sulfur bearing, and the other contains the metal ions and is sulfur deficient. Beales and Jackson (1966) and Jackson and Beales (1967) have extended this concept to the Mississippi Valley-type ore deposits. While such a mechanism avoids the difficulties inherent in trying to carry both sulfur and the metals in the same solution, the fluid inclusions from two ore deposits (southern Illinois and Creede, Colo.) have been analyzed for *both* sulfur and heavy metals, and in each case, both have been found in significant amounts (Roedder and others, 1963, p. 368; Roedder, 1965a, p. 381; and Czamanske and others, 1963).

Ermakov (1965b) analyzed two leaches made from crushed pegmatitic fluorite containing inclusions with up to 13 recognizable daughter minerals, including some opaque phases. (See Roedder, 1963b, p. 176 and 180–181.) As he found heavy metals but no detectable sulfate in either the first, water leach, or the second, hot, 0.1 percent HNO_3 leach, he assumes (p. 153) that the daughter minerals do not include sulfides. Not uncommonly, however, nitric acid solution of metallic sulfides yields insoluble elemental sulfur instead of sulfate.

FLUORINE

Comparatively few determinations of fluorine have been made on fluid inclusion samples, owing mainly to the analytical difficulties. (See tables 2, 3, 4, 5, 6, and 8.) Some of these determinations are of the gases SiF_4 and F evolved on heating or crushing fluorite (Sine, 1925; Assadi and Chaigneau, 1962; Chaigneau and Assadi, 1963; Kranz, 1965, 1966). Kalyuzhnyi (1961) reports large amounts of SiF_6^{-2} (and F^{-1}) in pegmatitic topaz, quartz, and barite, and occasionally F^{-1} is determined on leach samples. Heinrich and Anderson (1965) and Heinrich and Dahlem (1965) have found a variety of fluorine-bearing compounds in the gases evolved from a fetid, fluorite-bearing carbonatite associated with an assemblage of fluoride minerals such as cryolite and weberite.

Like boron, fluorine would be expected in many types of natural water-rich fluids, particularly residual liquids from the crystallization of metamorphic and siliceous igneous rocks (Kogarko and others, 1968). Fluorine is probably a rather common major constituent in pegmatitic and skarn environments. Detailed studies of daughter minerals in inclusions in such samples (see pl. 12) has resulted in the identification of a variety of fluorides (Kalyuzhnyi, 1956, 1958a; Lemmlein and others, 1962; Ikorskii, 1966a) and fluoborates (Slivko, 1955, 1958). The total concentration of fluorine in such fluids at the time of trapping is unknown, but the volume of fluoride daughter crystals alone requires rather large amounts of fluorine, without even considering the fluorine content of the liquid phase. Fluorite is usually considered to be relatively insoluble, but it has been reported as a daughter mineral in aqueous inclusions (table 1), as well as in glass inclusions (Roedder and Coombs, 1965), and Anikin and Shushkanov (1963) have shown experimentally that up to nearly 0.2 weight percent of fluorite will dissolve in hot brines under pressure. The residual liquids from some alkalic magmas are high in fluorine and may actually crystallize villiaumite (NaF) as a primary phase (Kogarko, 1961, 1964), although later hydrothermal or zeolitizing solutions may dissolve it (Gerasimovskii, 1963). Ikorskii (1966a) reports that daughter crystals of villiaumite, plus organic matter, occur in abundant inclusions in various rock-forming minerals of the alkalic rocks in the Khibina massif, and these same minerals also contain solid inclusions of villiaumite.

Determinations of fluoride on leach solutions have been made from a few samples and are generally very low. Ikorskii (1966a) found up to 200 ppm total water-soluble fluorine in the Khibina rocks. However, this includes villiaumite (NaF) as solid inclusions, as well as that as a daughter mineral in organic inclusions. Chlorine/fluorine ratios are seldom under 100 (Polykovskii, 1962a) except for leaches from fluorite samples, where the solubility of the host mineral precludes a valid determination of the inclusion fluoride content (Grushkin and Prikhid'ko, 1952; Grushkin, 1958). The high calcium concentration of many inclusion fluids would be expected to suppress the concentration of fluoride in solution, and unless considerable care is used to exclude all possibility of sample contamination with calcite, precipitation of part of any fluoride present in the fluid might occur during leaching.

In view of the experimental evidence that fluorine may aid in the transport of tin (Barsukov, 1967), analyses for fluorine in inclusions from tin deposits are important. Sushchevskaya, Barsukov, and Trusikova (1966) report 39 analyses for fluorine on leach samples from various deposits of Myao-Chan; many of these show more than 1 g of fluorine per liter of original inclusion fluid. Although the methods of sample preparation, extraction, and analysis are not stated, it should be mentioned that a small fraction of a milligram of solid fluorite in the original sample could dissolve in the leach water to yield an apparent fluorine content in this range.

It is possible that some fluorite ores have been formed by hot spring waters like those currently associated with the deposits, for example, at the Poncha fluorspar deposits in Colorado (Russell, 1947; Steven, 1960; Edwin Roedder, in U.S. Geological Survey, 1967, p. A160; see also White, Hem, and Waring, 1963, p. F50–F51). Here the waters contain 12 ppm F and 17 ppm Ca, which is not far from normal saturation with respect to CaF_2. A very sensitive method and very careful extraction procedure would be required for fluorine determinations on such solutions, if they are present as fluid inclusions. Thus a selected 10-g sample containing 12 mg of this fluid as inclusions would contain only 10×10^{-7} g fluorine. Contamination from solid fluorite in the sample, in the amount of only 10 parts per billion could double the amount found on analysis. Kalyuzhnyi and Lyakhov (1962) report that minute grains of fluorite occur as solid inclusions in many pegmatite minerals, and hence the fluoride analysis on any leach solution from such minerals would provide only a maximum value.

CHLORINE AND HEAVIER HALOGENS

Chlorine enters the structures of some rock minerals, particularly the micas and amphiboles, and occasionally halite may be found in metamorphic rocks (Engel and Engel, 1953), but the bulk of the chlorine reported in ordinary rock analyses probably occurs in the fluid inclusions (Roedder, 1958, p. 240). Thus a bulk sample of granite blocks in a volcanic breccia from Ascension Island showed only 0.054 percent Cl, but some individual feldspar crystals from this rock had a sufficiently high concentration of daughter crystals of halite in the fluid inclusions (see pl. 4, fig. 4) that the three strongest lines for halite were found in the X-ray powder pattern (Roedder and Coombs, 1967). The residual water-rich fluids in many igneous and metamorphic processes are high in chlorine (Koster van Groos, 1966; Koster van Groos and Wyllie, 1969; Pavlov, 1962), and it is probable that the high chlorine micas and amphiboles that have been found (Lee, 1958; Krutov, 1936, 1960; Geijer, 1961) merely reflect special very high chlorine environments.

Some igneous rocks are particularly high in chlorine, and J. F. Lovering (1966) reports 0.69± 0.03 percent Cl in the glassy matrix of a pantellerite. Magmatic differentiation of such a system must certainly yield high chloride brines, as experimental studies at magmatic temperatures and pressures with granite (Gammon and others, 1967) and albite (Koster van Groos and Wyllie, 1969) have shown a very strong partitioning of chloride into the fluid phase, yielding very strongly saline fluids. At very high chloride concentrations actual silicate-chloride immiscibility can be expected (Delitzyn and Melentiev, 1968; Roedder, 1970b). Fluid inclusions provide evidence that late magmatic brines carrying over 50 percent NaCl by weight are not uncommon (pl. 4, fig. 4 and pl. 11, figs. 1–4), and there is also evidence that such high chlorine solutions have added vast quantities of chlorine to some contact zones, in the form of scapolite and other minerals (Edwards and Baker, 1953; Krutov, 1960). Presumably the bulk of the chlorine in solution passed on through, except for that trapped in the fluid inclusions. The ultimate fate of such sodium chloride brines must be at the surface, but there are many possibilities for chemical change by dilution or by fractionation through boiling en route.

Ellis and Wilson (1960, p. 613) believe that the primary magmatic steam at Wairakei, New Zealand, contained about 1.5 percent chlorine. In the pure system $NaCl-H_2O$ such a vapor composition cannot be obtained below about 400°C (Sourirajan and Kennedy, 1962) but if HCl gas is involved, rather than NaCl, much higher concentrations are possible. Thus Murata (1966) reports a 1.9 *N* HCl solution was formed by the condensation of vapors from the degassing of a basaltic cinder cone at Kilauea Iki. On contact with most rocks, such fluids would rapidly exchange their H^{+1} for Na^{+1}, Ca^{+2}, and Mg^{+2}.

Although chloride is the major anion in most inclusions from ore deposits, and chloride complexes are commonly considered in discussions of ore metal transport (Helgeson, 1964; Helgeson and Garrels, 1968), other anions such as HCO_3^{-1} or SO_4^{-2} occasionally are present in greater amounts. (See tables.) As the analyses listed in the tables were selected in part to show the range in composition, such atypical analyses are overrepresented. Thermal waters associated with volcanism may be very low in chlorine relative to sulfur (White, Hem, and Waring, 1963), but such fluids have not been recognized in inclusions.

In view of the usefulness of the ratios Br/Cl, I/Cl, and Ca/Br in understanding the nature and source of various waters (Ellis and Anderson, 1961; White, Hem, and Waring, 1963; Braitsch and Hermann, 1963; Krasintseva, 1964; Anderson and others, 1966) it is unfortunate that other than those by Holser (1963), almost no determinations of either bromine or iodine have been made on inclusions. The ratios B/Cl and F/Cl are also of some interest, but they are seldom determined. Although bromine, iodine, boron, and fluorine are all rather minor constituents of inclusions, it is hoped that the newer analytical techniques, particularly neutron activation, will soon provide reliable analyses of these even at the levels expected in inclusion samples.

ISOTOPIC STUDIES

The data available on the isotopic composition of inclusion fluids are so very limited that few generalizations are possible. Carbon isotopic ratios have been determined for gases, presumably from gas inclusions, in the Kola Peninsula rocks (Lebedev and Petersil'e, 1964; Galimov and Petersil'e, 1967; Alekseyev and others, 1967; Galimov, 1967) and for carbon dioxide gas from the Laacher See carbonatite district in Germany (Taylor and others, 1964; see also Deines and Gold, 1966). Studies of the isotopic composition of carbon from the bitumens of the Khibiny massif (Zezin and others, 1967) indicate that these substances have not been derived from the surrounding Proterozoic shale. It is probable that measurements of carbon isotopic ratios will do much to clarify the source of several of the forms of carbon in magmatic rocks, including the CO_2 inclusions in olivine nodules, free carbon, carbonatite carbon (for example Vinogradov and others, 1967), hydrothermal vein carbonate, and even diamonds.

Rye (1964, 1965) has studied the carbon and oxygen isotopes in various stages of hydrothermally deposited calcite at Providencia, Mexico. These studies were extended (Rye and O'Neil, 1968) to include these same isotopes in the inclusion fluids, in an attempt to understand the ore-forming process more fully. Such data are complicated by partial isotopic exchange between the hot fluid and the wallrock (White, 1965b; Anderson, 1966; Garlick and Epstein, 1966; Taylor and Epstein, 1968) and by very slow exchange rates between certain species in solution, for example oxygen between H_2O and SO_4^{-2} (Lloyd, 1968). In addition, exchange may occur with at least some of the host minerals, on cooling; Clayton and others (1964, 1966) have shown that even at the temperatures prevalent in sedimentary basins, the connate waters have exchanged oxygen with surrounding carbonates. Clayton, Muffler, and White (1968) have shown such exchange to have occurred at 150°C or even lower between rock and brine in the Salton Sea geothermal area, J. R. O'Neil and R. O. Rye (U.S. Geological Survey, 1967, p. A169–A170) and Rye and O'Neil (1968) have shown it to occur, on cooling, between inclusion fluid and host quartz and calcite at Providencia, Mexico. Such exchange might even be used to estimate relative quantities of solution and rock, but the techniques available now for determining carbon and oxygen are not very adequate to handle the small size of inclusion samples. Improvements can be expected (O'Neil and Epstein, 1966; J. R. O'Neil and R. O. Rye, in U.S. Geological Survey, 1967, p. A169–A170).

The greatest volume of isotopic data on inclusion fluids is on the deuterium-hydrogen ratio (table 3). As most of the minerals studied are anhydrous, isotopic exchange problems are eliminated, but Jensen (1953) has pointed out that if any diffusion of hydrogen occurs out of an inclusion, isotopic fractionation can, of course, be expected. Graf, Friedman, and Meents (1965) have suggested that some isotopic fractionation of hydrogen and oxygen occurs in formation waters as they move through shales. D/H ratios on inclusions have proved useful in following the stage of evaporation of salt brines (Holser and others, 1963), but as the liquid-vapor fractionation has a cross-over point at 223°C, above which normal fractionation is reversed and the heavier species HDO concentrates in the vapor phase (Bottinga and Craig, 1968), uncertainty as to the temperature at which evaporation (or boiling) has occurred will cause ambiguity in any extrapolation to elevated temperatures. D/H ratios have also revealed that the fluids from amygdules in Japanese basalts have isotopic compositions very similar to those of the surface waters of the same region (Kokubu and others, 1961). Hall and Friedman (1963) present 33 D/H analyses of inclusion fluids, from two areas of Mississippi Valley-type deposits. These show a consistent relation of D/H ratio with the stage of deposition. Such data, particularly when tied to other inclusion studies and detailed mineral paragenesis and spatial distribution studies, may help to delineate the circulation patterns and the origin of the ore-forming fluids.

Perhaps the most important problem involved in the use of isotopic studies to understand the circulation patterns in ore deposition lies in the ambiguity as to the origin and initial composition of the water before any exchange occurred. Thus there is isotopic evidence that the ore fluids at some hydrothermal deposits were of magmatic origin, as at Providencia, Mexico (Rye, 1965), whereas others were of meteoric origin, as at the Bluebell mine, British Columbia (Ohmoto, 1968a; Ohmoto and Rye, 1968), but both have presumably been involved in some isotopic exchange. Simultaneous studies of the isotopes of hydrogen, oxygen, carbon, and sulfur, in both inclusions and minerals, may eliminate some of the ambiguity.

HIGH CONCENTRATIONS

Of all the results obtained from fluid inclusion studies, the most unusual and perhaps unexpected is the evidence of the rather common occurrence of exceedingly strong brines in nature—brines that are more concentrated than any now found at the surface. Cubes of halite in secondary fluid inclusions from metamorphic and igneous rocks have been reported so often and are so widespread in occurrence (table 1) that they are generally ignored. Yet they require that fluids containing more than 26 weight percent NaCl were present in these rocks, at some time in their history.[40] (See pl. 4, fig. 4). The volume of fluid was small, but as long as it was present as a separate phase, generally occupying the pores and intergranular films that are now measured as rock porosity, it would continue to approach equilibrium with the mineral assemblage under changing conditions. The high concentrations of chloride

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[40] Where such inclusions are trapped in feldspar or nepheline, particularly if CO_2 is also present, one would expect reaction (retrograde metamorphism) to form scapolite or sodalite during cooling (for example, Wellman, 1968), but this has not been reported. Much higher concentrations are possible without any evidence in the form of daughter minerals, if more highly soluble salts, such as $CaCl_2$, are involved. Thus Maslova and Gurinova (1966) present an analysis of inclusion fluids from an Iceland spar from amygdaloidal Yakutian traprocks that contain 449 g/l.

would have comparatively little effect, other than driving more chlorine into certain rock minerals and lowering the activity of water. But the cations present in this fluid would be of very great significance in controlling, or at least registering, the composition of the mineral surfaces involved; they may greatly affect the solubility of ore elements (Roedder and Coombs, 1967, p. 446; Ryabchikov, 1967); and they may control the structural state of the alkali feldspars (Martin, 1969).

The fact that many—but not all (see, for example, Roedder and Coombs, 1967, and pl. 4, figs. 7, 8)—of these strong brine inclusions in such rocks are secondary features has often been used to degrade their significance, but this very fact makes them that much more likely to be pertinent to the problems of various late-stage magmatic and metamorphic phenomena such as the formation of greisens, pegmatites, and ore deposits, as well as various "deuteric" alteration processes such as albitization, sericitization, chloritization, and argillization. (See pl. 2, figs. 1–6; pl. 3, fig. 1; pl. 4, figs. 1–4, 7–8; pl. 5, fig. 2; pl. 7, fig. 12.) Within any of these categories, inclusion studies may indicate that there have been wide variations in the composition of the fluids present, but the significance of these variations is not yet clear. Studies of rock alteration in hot spring areas (Sigvaldason and White, 1962; White and Roberson, 1962; White and Sigvaldason, 1962) have shown that many of the alteration assemblages common in the wallrocks of ore deposits may also be formed by hot spring waters of low salinity. In some geothermal areas oxygen isotope evidence indicates that boiling of brines has occurred (Craig, 1966a, b); this could lead to a wide range of solution concentrations (pl. 4, figs. 1–3).

Thus in the formation of late-stage miarolitic vugs, vuggy pegmatites, and quartz veins from granitic magmas, there is ample evidence from freezing data (Roedder, 1963b) and from a number of the chemical analyses in which valid water determinations were made (see table 6), that many of the fluids responsible were dilute water solutions, in part carbon dioxide bearing. The total concentration of salts (mainly NaCl) seldom exceeded 1 molar sodium chloride equivalent. Yet there is also a considerable body of evidence, mainly from studies of daughter minerals in inclusions, that during some of these same processes, and even in the same deposits or samples, highly concentrated brines existed, with 30, 40, or even more than 50 weight percent salts in solution. In addition, these salts usually differ, rather grossly, from those of the more dilute solutions and contain, not infrequently, many exotic constituents. (See table 1, and particularly Zakharchenko, 1950; Ermakov, 1950a; Kalyuzhnyi, 1956, 1958a; and Lemmlein and others, 1962.) The fact that most of this work involves qualitative identification and only semiquantitative estimates of the amounts present does not detract from its importance; the difference between the two types of fluids is obvious and real. It is not known whether these differences are a result of phase changes (such as boiling), crystallization path differences (Koster van Groos and Wyllie, 1969), or merely represent spotty sampling of a complete spectrum of such compositions during any given event. In any case, further study of inclusions shows considerable promise in resolving the many complications in these processes.

Similar ranges in total salt concentrations, although apparently with less variation in salt composition, are found for hydrothermal ore deposits. Freezing data and complete chemical analyses of such fluid inclusions (Roedder, 1963b, 1967a; Roedder and others, 1963) show that many ore deposits formed from dilute fluids, generally containing less than 10 weight percent salts. Yet some deposits formed from concentrated brines, as indicated by the listing of daughter minerals in table 1. (See particularly Lindgren and Whitehead, 1914; Koltun, 1958; Takenouchi, 1962b; Sawkins, 1963, 1966b; Roedder and Creel, 1966; and Dolomanova, 1966.) (See also pl. 3, fig. 1; pl. 4, fig. 1).

Although these occurrences may all be related to specific though accidental local conditions, such as boiling, one group of deposits is characterized by solutions of high concentration. The Mississippi Valley-type of deposit seems to have formed from strong, but generally unsaturated[41] sodium-calcium-chloride brines (Roedder, 1963b, 1967a, 1968c, d; Roedder and others, 1963; Hall and Friedman, 1963; Khodakovskiy, 1965; White, 1968b; and the many papers presented at a symposium on these deposits, in New York, 1966 (J. S. Brown, 1967)). Any theory of origin for these important but rather anomalous deposits that is worthy of consideration must be able to explain, or at least not be in conflict with, the data on inclusions. Of the many theories that have been proposed, some would have the deposits formed from relatively fresh ground waters, and others would invoke formation from

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[41] Kuhl (1960) reports the presence of actual halite in barite veins with fluorite, quartz, or sulfides, from northern Silesia, that are similar to the Mississippi Valley-type deposits in many respects.

ocean waters (or formation from the dilution of submarine hydrothermal fluids with sea water—see Ivanov, 1966). Yet, extensive studies of primary inclusions in these deposits indicate formation from brines that are consistently at least five times as concentrated as sea water. The available data seem to favor a nonmagmatic origin for most of the constituents of such brines, although there is much disagreement on the subject.

It is reasonably certain, however, that fluids of this type can and do form, in sedimentary rocks, by normal diagenetic and later processes (White, 1965a; Valyasko and Vlasova, 1965). In particular, high concentrations can be achieved in nature by selective preferential filtering of the water molecules through fine-grained sediments, leaving behind fluids enriched in the dissolved ions, to the extent that some natural brines actually precipitate salts on cooling to surface temperatures (Pinneker, 1964). A more obvious source of high salinites is sometimes found in the solution of evaporite beds along the path of circulation, as has been proposed, for example, for the hot, metal-bearing brines occurring in pockets at the bottom of the Red Sea (Craig, 1966b), and evaporite salts may be present even in metamorphic terraines (Engel and Engel, 1953; Brown and Engel, 1956). Davidson has invoked hot brines, from the solution of evaporites, as the ore-bearing fluids for the formation of various strata-bound copper deposits (1964, 1965a) and the uraniferous and auriferous conglomerates (1965b). In addition to the evidence from fluid inclusions, Davidson (1966) shows that there is a strong correlation between the occurrence of these highly saline brines and known evaporite sequences. Dunham (1966) also uses some of these same data on the composition and homogenization temperature of inclusions in the North Pennine ore field (Sawkins, 1966a) to support a magmatic source for the solutions.

The high salt concentrations in some inclusions in ore deposits are also a factor in the ability of the fluids to form soluble complexes of the ore metals, but here there are also many unanswered questions. (See Roedder, 1965c.) Although considerable progress has been made in the procedures for the calculation of the degree of formation of such complexes (Helgeson, 1964), the bulk of the experimental work on fundamental properties and on solubilities that might be used for checking has been in lower ranges of temperature, pressure, or concentration of solutes. Some unexpectedly high solubilities have been measured, even in brines of simple composition, and other high solubilities will probably be found. (See discussion on "Heavy Metals.")

An additional aspect of the concentration of inclusion fluids that is commonly forgotten is the solubility of the walls of the inclusion itself, on reheating to the temperature of homogenization. Careful studies by Lemmlein (1950a), Lemmlein and Kliya (1952a), and Lemmlein, Kliya, and Ostrovskii (1962) have shown that not only do very large amounts of relatively insoluble daughter minerals dissolve, but also that very significant quantities (tens of weight percent) of the quartz or topaz host mineral dissolve in these complex brines (pl. 8, figs. 2–6). Anderson and Burnham (1962, 1965) have shown that only about 1 weight percent silica is soluble in pure water vapor at 500°C, but much higher amounts dissolve at temperatures over 1000°C (Kennedy and others, 1962). A number of hydrothermal investigations have shown, however, that even at low temperatures, up to several orders of magnitude more silica (and other materials) may dissolve in water vapor if other components, such as the alkalies, are also present.

The salt concentrations in fluid inclusions are of importance in one further regard. Along with the pressure and the temperature of the fluid, the salt concentration controls the *density* of the fluid (Klevtsov, 1959; Lemmlein and Klevtsov, 1956b, 1961; Sourirajan and Kennedy, 1962; Ellis and Golding, 1963; Unterberg, 1966, fig. 2; Fabuss and others, 1966). An example is found in Craig's proposal (1966b) that the hot brines at the bottom of the Red Sea have flowed underground from 1,000 km south, under a hydrostatic head based on differences in salinity. Fluid inclusions permit reasonably accurate and unambiguous estimates of the density of the fluids at the time of trapping, and there does not appear to be any other source for such information. If the relative volumes of crystal, liquid, and gas phases are determined at room temperature, by using tubular, flat, or geometrically regular inclusions, the density of the originally homogeneous fluid can be calculated. Volume changes from external or internal pressure, and from thermal expansion of the host, are negligible compared to the effects of composition and inaccuracies in phase volume measurement (fig. 3 and pl. 11, figs. 7, 8). The composition of the fluid is of considerable importance, not only in the obvious matter of determining its density at room temperature, but also in controlling its thermal expansion and hence density at the temperature of homogenization, as well as its compressibility at still higher pressures.

Some minerals have formed from very low density fluids, less than 0.5 g/cm^3, as evidenced by large numbers of inclusions with uniformly low liquid/gas ratios. Even at these low densities, there apparently can be considerable solubility and dissociation of dissolved salts (for example, Fogo and others, 1954; Quist and others, 1963). The numerous reports of "dry" gas inclusions in some samples do not require, however, that the minerals formed from such low density media, as only the gas phase may have been trapped during boiling or effervescence (pl. 9, fig. 4).

On the other hand, some fluid inclusions with large daughter crystals show densities as high as 1.5 g/cm^3 (for example, 50 percent liquid, 40 percent NaCl crystal, and 10 percent gas, by volume at room temperature; see also pl. 3, figs. 3, 4). Most hydrothermal ore deposits have formed from fluids in the range of 0.5 to slightly over 1.0 g/cm^3. The significance of these density measurements lies in the very great effect the density has in controlling or preventing circulation and mixing of such hot brines with overlying cold fresh waters at density 1.0.

HEAVY METALS

Very few analyses of fluid inclusions list determinations of the heavy metals (tables 3, 4, 5, 6, and 8). Most of these have been qualitative or semiquantitative spectrographic determinations on the residues from the evaporation of water (and even HCl) leaches of large crushed samples, cleaned by only simple washing, and hence they are subject to rather gross contamination from a variety of sources. In addition, many of the results have been sufficiently low so that they were near what might be expected on a "blank," although a truly valid blank determination is difficult to arrange. Balitskii and Lyubofeyev (1962) report spectrographic determinations on powdered quartz from an ore deposit that show a gross drop in the amounts of various heavy metals present, after leaching with water (table 5), indicating water-soluble constituents. As specimens from mineral deposits frequently have minute inclusions of ore minerals, and have presumably been bathed in metal-bearing mine waters for considerable periods of time, rather stringent sample selection and cleaning procedures are needed to avoid ambiguity in such determinations (Roedder, 1958).

Arnautov and others (1965) report very appreciable concentrations of Mn, Fe, Ni, Ti (and Al) in the fluid inclusions of a chambered fluorite-bearing pegmatite (table 5, analyses 60–62). These analyses were made of samples that had been boiled in aqua regia and then washed with doubly distilled water, before crushing, sieving, and leaching for release of the inclusion contents. Unfortunately, however, simple washing is generally inadequate to remove the acid from cracks, along with the impurities it dissolved as it moved into the sample. Samples of acid-leached quartz, of −10 mesh size, were still appreciably contaminated after several days of leaching with water, and they required electrolytic treatment for final cleaning (Roedder, 1958, p. 241).

Lead in particular has been reported in spectrographic analyses of numerous inclusion samples and residues. (See table 3.) Although contamination is very difficult to avoid, it is reasonable to expect that lead was present in appreciable concentrations in at least some late-stage igneous processes, as Zhirov and Stishov (1965) have shown that amazonitization of microcline commonly has resulted from the introduction of lead into the feldspar.

Khitarov and Moskalyuk (1955, table 1) found no lead in leaches from 15 galena samples, but they report one determination (p. 129) in which the amount of lead found (150.9 mg from 100 g of galena or 1,500 ppm) was greater than the sum of all other cations and anions; this analysis was confirmed by several chemists. As the major anion was chloride, solid inclusions of the sparingly soluble lead chloride cotunnite were suspected. The authors believe that this possibility was excluded by certain leaching experiments. But their description (p. 129–130) of the highly irregular distribution of samples showing these high soluble lead determinations, even in a single specimen, would be expectable if cotunnite were present in irregular distribution; it is very difficult to explain (as they do) on the basis of lead chloride in solution in the fluid inclusions. Seeliger (1950) has found tiny solid contunnite inclusions to be deeply embedded in the galena of certain German ore deposits, and depending upon the material, it might be difficult to set up a sample preparation procedure involving grinding and leaching such that all cotunnite present will be exposed and removed, but the fluid inclusions will be left sealed, to be opened on further grinding.

Heavy metals have been determined in a number of thermal waters, some of which may be related to ore deposits. With comparatively few exceptions, Pb, Zn, Cu, Sb, Hg, and Mn show maximum values of about 1 ppm (Minami and others, 1957; Hewett and Fleischer, 1960; Ritchie, 1961; White and Roberson, 1962; Hewett and others, 1963; Cadek and Malkovsky, 1963; Uzumasa, 1965). Arsenic concentrations up to 8.5 ppm are reported by Ritchie for

New Zealand thermal waters, and up to 0.057 ppm Ag was found in some Japanese hot springs (Koga, 1957). White (1965a, 1965b) lists other analyses for these and additional metallic elements from a variety of geothermal fluids. Although fluids of such low concentration are adequate to form large ore deposits, when flowing for geologically reasonable rates and times (Roedder, 1960b, p. 226; White and Roberson, 1962, p. 423; Davidson, 1965a, p. 951), White (1965b, p. 433) believes that the low metal concentration might indicate that these are spent residues, from the precipitation of base-metal ore deposits below currently explored depths of drilling in the thermal areas (50 to 1,200 meters).

The heavy-metal content of the ore-forming fluid is obviously of considerable importance in understanding the mechanism(s) of ore-metal transport and precipitation and in making semiquantitative guesses as to the plumbing system needed to bring the ore in. Any heavy metals present in the ore fluid trapped in an inclusion during the formation of an ore deposit must either (1) precipitate upon cooling to surface temperature or (2) still be in solution.

Inclusions in transparent ore and gangue minerals permit us to estimate the amount of precipitation that has occurred, as simple optical evidence of the occurrence of solid opaque daughter minerals, presumed to be ore minerals, gives us an exceedingly sensitive test (Roedder, 1960b). The generally rare occurrence and small size of these opaque daughter minerals[42] indicate that the amount of precipitation of ore metals from the ore fluids must be rather low, usually less than 1 to 10 ppm, but there are some high temperature inclusions in fluorite from chambered pegmatites in the U.S.S.R. that have precipitated more than 1 percent of sulfides (for example, Roedder, 1963b, p. 181), and ore fluids with over 50 percent iron have been proposed (Ingerson and Mackin, 1961; Ingerson, 1965). Ermakov (1965b) has determined several of the heavy metals in inclusions from the chambered pegmatites, using semiquantitative spectrographic analysis of leach residues from a water and an acid leach, presumably in sequence of the same sample. He found that Cr, Cu, Fe, Mn, Ni, and Pb were one or more orders of magnitude larger in the acid leach.

Not all daughter crystals containing heavy metals need be opaque. Kalyuzhnyi (1958a) recognized caracolite ($PbOHCl \cdot Na_2SO_4$) and a new, unnamed chloride of aluminum and zinc. Lyakhov (1966) used X-ray diffraction to identify a colorless highly birefringent daughter mineral as $FeCl_2 \cdot 2H_2O$ (unnamed), in inclusions in smoky quartz from Volynia, and reports an analysis of over 1 percent iron in solution in the liquid phase. Kalyuzhnyi and Voznyak (1967) also report $FeCl_2 \cdot 2H_2O$ as a daughter mineral in pegmatitic quartz, and they verified the presence of both ferrous and ferric iron by qualitative tests. Cassiterite and hematite inclusions were present in some samples. Kalyuzhnyi and Lyakhov (1962) report the common occurrence of very minute solid inclusions of cassiterite in various pegmatite minerals, and hence these might be expected also as daughter crystals. Many other rare minerals containing heavy metals have yet to be reported as daughter minerals but might be expected, such as the zinc analogue of d'ansite ($ZnSO_4 \cdot 3NaCl \cdot 9Na_2SO_4$) (Burzlaff and Hellner, 1961).

The amount of metal still in solution is more difficult to estimate. Although most of the spectrographic analyses mentioned above show very little, two carefully selected samples, analyzed by neutron activation, showed 100–10,000 ppm (0.01–1.0 percent) each of Cu, Mn, and Zn (Czamanske and others, 1963). The highest values for Cu and Zn were found in inclusions in fluorite from the Cave-in-Rock district, Illinois. These authors examined the various possible sources of contamination and found them to be relatively negligible, but as the values obtained are unexpectedly high, they suggested that the data should be verified by other similar studies. This was done by Pinckney (1966), who reported a series of analyses for Cu and Zn in fluid inclusions, also from the Cave-in-Rock area, using atomic absorption. He found less than 100 ppm to about 1,000 ppm each of Cu and Zn in water leaches of eight inclusions (D. M. Pinckney, oral commun., 1968). Similar results were found by Rye and Haffty (1968) for samples from Providencia, Mexico. Ohmoto (1968b) reports 10–300 ppm Zn in fluid inclusions from the Bluebell mine in Canada. Additional confirmatory evidence that such fluids can exist in nature comes from the discovery of hot, exceedingly saline, metalliferous brines in California (White, Anderson, and Grubbs, 1963; White, 1965b; Helgeson, 1967, 1968). These brines are more saline than even the Mississippi Valley-type inclusions, and although they show $\sim$100 ppm of lead (Doe and others, 1966), and more manganese than found by Czamanske and others in the inclusions, they show considerably lower copper, zinc, and sulfur. All such analyses for heavy metals in trace concentrations

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[42] Moore (1965) has proposed the transportation of heavy-metal sulfides as particulate matter, in suspension. Unless this particulate matter is monomineralic, it should result in the presence of large numbers of solid opaque grains in the primary fluid inclusions.

involve not only difficulties in eliminating or minimizing sources of contamination but also loss by adsorption onto sample containers, even from relatively acid solutions (Chao and others, 1968).

Four additional neutron activation analyses of fluids from inclusions are given by Puchner and Holland (1966). In these, three samples containing secondary inclusions, and one showing a primary inclusion, were analyzed for Na, Mn, Cu, and Zn. Irradiation was performed *before* extraction, and both water and HCl leaches were made. The inclusions were only moderately saline (0.73 to 1.82 weight percent Na, equivalent to 1.9 to 4.6 percent NaCl). Zn and Mn were found to be less than 200 ppm, and Cu was less than 10 ppm in the original fluids. These values are much lower than those reported by Czamanske, Roedder and Burns (1963), but although the quartz that was analyzed was known to be hydrothermal, it was a late-stage, post-ore generation, and the inclusions show considerably lower salinities than primary inclusions in ore minerals from the nearby Providencia ore bodies (Sawkins, 1963).

Hot, strongly saline brines have been found at the bottom of the Red Sea (Brewer and others, 1965; Neumann and Chave, 1965). These waters show heavy-metal concentrations orders of magnitude above ordinary sea water. Miller and others (1966) report the following (converted to ppm): Cu, 0.8; Zn, 2.5; Mn, 72; Fe, 58. They, and Manheim and others (1965), report that the sediments under these waters are very rich in iron and zinc (Fe_2O_3, 56 percent; ZnO, 6 percent) and carry appreciable amounts of Mn, Cu, Pb,[43] and Cd. Several of these papers indicate submarine solution of evaporite beds as a likely origin for these brines, but they also point out similarities to oilfield brines. Craig (1966b) proposes that these brines, driven by density differences, moved great distances at considerable depths.

Davidson (1964; 1965a) has proposed that many of the strata-bound copper ores have formed by slow moving, strongly saline brines (possibly from the solution of saline deposits), which have leached copper out of rocks at depths and redeposited it at higher levels. Somewhat similar origins have been proposed for the extensive strata-bound lead-zinc-fluorite-barite deposits (Roedder, 1966b; Brown, 1967; and also Noble, 1963) and for the uraniferous and auriferous conglomerates close to major unconformities (Davidson, 1965b). Of considerable significance in this connection is the recent report by Lebedev (1967) of numerous occurrences of high-lead brines in sedimentary basins in the U.S.S.R., containing up to 77 ppm lead, at temperatures of only 80°C. Some of these have deposited tons of metallic lead in the boreholes. Similarly, Billings, Kesler, and Jackson (1969) report zinc analyses of 19 mg/l (milligrams per liter) for the "formation water" brines in northern Alberta, and propose that such fluids have deposited the Pine Point ores.

Actually, as yet unknown interactions with the high concentrations of salts in inclusion fluids may be responsible for many unexpectedly high solubilities for heavy metals, particularly in chloride brines. In addition to the possible significance of complexing with ammonium mentioned earlier, Tsusue and Holland (1966) have shown that zinc is very strongly complexed by chloride, and Hemley and others (1967) report very significant solubilities for ZnS in chloride solutions buffered by various silicate minerals. High chloride solutions at 400° to 465°C effectively extracted Fe, Mn, Cu, and Pb from andesite (Ellis, 1968). Gammon, Borcsik and Holland (1967) showed that there was a strong partitioning of Zn and Pb into a saline fluid phase in contact with a granitic melt at magmatic conditions. Borina (1963) measured the apparent solubility of $CaMoO_4$ in solutions of KCl and NaCl; she found that as much as 0.015 weight percent dissolved at 500°C. Bryatov and Kuz'mina (1961; see also Kuz'mina, 1961) dissolved 2,350 ppm lead (from galena crystals) at 400°C, and Anderson and Burnham (1964 and 1967) report in the order of 1,000–2,000 ppm of metallic gold (or platinum) dissolved in chloride brines. At high temperatures many metallic chlorides are volative (Krauskopf, 1964) or form volatile complexes stable at high temperatures, such as $NaCl{\cdot}FeCl_3$, $KCl{\cdot}FeCl_3$, $NaCl{\cdot}CuCl_2$, and $NaCl{\cdot}PbCl_2$ (Ovchinnikov, 1965). Hence, if a chloride phase is permitted to volatilize, it may carry appreciable heavy metals with it, particularly iron (Pavlov and others, 1964). High temperature, supercritical aqueous fluids can dissolve large amounts of uranium, copper, and nickel sulfates (Marshall and Jones, 1963; Jones and Marshall, 1964; and other papers in that series). There is considerable need for further laboratory experimentation in such systems, using these newly developed techniques for experimental measurement of hydrothermal solubility, until valid theoretical procedures are developed for calculating solubilities in the hot, concentrated, multicomponent systems characteristic of nature.

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[43] B. R. Doe (written commun., 1967) has found 0.5 ppm Pb in the brines.

Certainly conclusions concerning the mechanisms of transport and deposition of ore minerals must be based at least in part upon the evidence found in the fluid inclusions.

Graton (1968, p. 1712) has expressed doubts about the validity of laboratory data on the capacity of supercritical fluids to transport solids in solution: "Meniscuses, if they exist, do not disappear or reappear, and the uniform distribution of density throughout the system is likewise unproved, as it might be in a transparent vessel." Nature has provided us with just such transparent vessels or "visual autoclaves" (Ermakov, 1950a) in the form of fluid inclusions, filled with the best available samples of the ore forming fluids.

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[Italic page number indicates most important reference]

TABLES 1–8

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*

Reference: There is much duplication in the data reported by some of these papers, but they could not be eliminated without losing occasional new data or reinterpretations. This tabulation does *not* include descriptions of solid inclusions, simple glass inclusions in igneous rock minerals, with or without daughter minerals, or simple descriptions of the occurrence of liquid, gas, or liquid-plus-gas inclusions without further identification of composition or thermometric studies.

Liquid CO_2: The presence of CO_2 was occasionally established by the formation of carbon dioxide hydrate crystals on cooling.

Organic: The distinction between organic liquid and organic solid is not always clear, particularly when only bitumen is mentioned in the text.

Other phases: Because of its rarity and importance, H_2S is noted here even though it generally does not occur as a separate gas or liquid phase. X in this column indicates presence of one or more unidentified phases.

Thermometry: The method used is not always stated explicitly. H, homogenization method, usually heating stage on microscope; D, decrepitation method (audio or video), regardless of assignment in the original to fluid inclusions or other causes; U, method unspecified or unavailable at time of writing; C, cooling studies, usually on freezing stage on microscope.

Sample notes: B, barite; C, calcite or other carbonates; F, fluorite; G, galena; Q, quartz; S, sphalerite; T, topaz; OD, ore deposit; peg, pegmatite(s); ign, igneous rocks; sed, sedimentary rocks; meta, metamorphic rocks; hyd, hydrothermal.

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Akhvlediani and others, 1968.									H	Q; Alpine veins; Caucasus, U.S.S.R.
Aleksandrovich and others, 1965.					(?)					Halite; potash OD; Starobin, U.S.S.R.
Ames, 1958									H	G, B, F; OD; New Mexico.
Andrusenko, 1958									H	Celestite; veins; U.S.S.R.
Andrusenko, 1966								H_2S	H	Analcime and Iceland spar; U.S.S.R.
Andrusenko, 1968									D	Various minerals; Iceland spar OD; U.S.S.R.
Andrusenko and Il'in, 1960									U	Iceland spar OD; U.S.S.R.
Andrusenko and Kiyevlenko, 1966.									H	Iceland spar OD; U.S.S.R.
Andrusenko and Kiyevlenko, 1968.		X	X					X, hematite	H, C	Iceland spar OD; U.S.S.R.
Andresenko and Lushnikov, 1966.									H	Synthetic Iceland spar.
Andrusenko and Mel'nikov, 1968.									C	Iceland spar OD; U.S.S.R.
Anikin, 1951	X	X		X					CH	Q; hyd; Ural.
Anufriyev, 1966	X							H_2S	D	Q; veins; southern Urals.
Anufriyev, 1968									D	Q; veins; southern Urals.
Arkhipchuk and others, 1968.									H	F, Q, C; Kuray OD; western Transbaikalia.
Arnautov and others, 1965									H	F; peg; U.S.S.R.
Arnold, 1964	X	X							H	Q; Flin-Flon area; Saskatchewan.
Atanasov, 1963									HD	S, Q, B, C; OD.
Atanasov, 1965									H	Q, S, B; Madzharova polymetallic OD.
Auerbach, 1870	X									T.
Awald, 1957							X			Q; Herkimer, N.Y.
Bach, 1940	X							Hematite		Sapphire; Ceylon.
Badalov, 1960									H	Anhydrite; Kal'makyr OD; U.S.S.R.
Bailey, E. H., 1959					X	X	X			Q; Hg OD.
Bailey, S. W., 1949									H	Q; granites.
Bailey, S. W., and Cameron, 1951.									H	S, C; Pb-Zn OD.
Bakumenko, 1963								Viscous liquid	H	Q; peg.
Bakumenko, 1966									H	T; Kazakhstan peg.
Bakumenko, 1968									H	Q; various zoned peg.
Bakumenko and Dolgov, 1966.									H	Microcline, low temperature; peg; U.S.S.R.
Bakumenko and Lysakov, 1965.									H	Q; peg; U.S.S.R.
Bakumenko and others, 1967.								Glass	H	Various minerals, synthetic and ign.
Balashov and Pozharitskaya, 1968.									U	Dolomite, ankerite; carbonatites; U.S.S.R. (quoting M.D. Somina).
Baldwin and Gross, 1967, p. 103.									D	Iron formations.
Balitskii and Lyubofeyev, 1962.	X							X	HD	Q, S; polymetallic OD; U.S.S.R.
Balitskii and others, 1966									H	Q; central Kazakhstan.
Banshchikova, 1958	X								H	Q, T; Kara-Oba rare metal OD; U.S.S.R.
Banshchikova, 1965	X								H	Q, etc; Kara-Oba W-Mo OD; U.S.S.R.
Barabanov, 1958b	X									Albite and adularia; Q-wolframite OD; U.S.S.R.
Bartoshinskii and others, 1967.									H	C, siderite; pegs; Volynia, U.S.S.R.
Bastin, 1931, p. 64					X	X	X			F; Pb-Zn OD.
Bastin, 1950, p. 58						X				F; Pb-Zn OD.
Bauer and Spencer, 1940, p. 118, 119, 236.	X					X				Diamond.
Bazarov, 1964									H	Apatite; veins; U.S.S.R.
Bazarov, 1965a	X								HC	Q, F; chambered peg.
Bazarov, 1966a									H	Apatite; several sources; U.S.S.R.
Bazarov, 1968a									H	Q, F; Kazakhstan pegs.
Bazarov, 1968b									H	Minerals of granites; central Kazakhstan.
Bazarov and Motorina, 1967.		X	X						HC	F; Kazakhstan pegs.
Bazarova, 1965									HC	Nepheline syenites.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Bazarova and Feigin, 1966		(?)						X, ore mineral	H	Nepheline, aegirine; Lovozero massif, U.S.S.R.
Bazarova and Shugurova, 1968.	X					X				Various alkalic ign.
Beaumont, 1846		X								Various OD vein minerals.
Beck and Weed, 1905, p. 293-294.	X								H	Q; Au veins.
Becker, 1882, p. 37		X								Ign.
Belchic, 1961								CO_2 gas		Halite; Winnfield dome, Louisiana.
Bellière, 1919					X	X	X			Q geode; sed.
Berger, 1960									U	C; veins.
Berger and Moskalyuk, 1962.									D	Q; meta veins.
Bergman and Blankenburg, 1964.		X						Gypsum		Q; peg.
Berman and others, 1965									D	Cu-Pb-Zn OD eastern Tuva, U.S.S.R.
Berzina and Sotnikov, 1965.	X								H	Q; Cu-Mo Sorskii OD, U.S.S.R.
Berzina and Sotnikov, 1968.									H	Q, F, Cu-Mo Sorskii OD, U.S.S.R.
Bilgram, 1903							X			Q; meta.
Blais, 1954									D	Q; Au OD.
Bochkarev and Moskalyuk, 1968.									D	Q; rock crystal veins; Urals, U.S.S.R.
Bogolepov and Bocharov, 1968.									D	Various greisens; Kazakhstan.
Bogoyavlenskaya and Blyakhman, 1966.									H	F; Irbinsk OD, U.S.S.R.
Bolman, 1940	X	X		X						C; emerald.
Borcos, 1964									H	S; various Romanian OD.
Borcos, 1965a									H	Q, C, S; Monts Metalliferes, OD, Romania.
Borcos, 1965b									H	Q; various Romania OD.
Borcos, 1966									H	Q, C, dolomite; Monts Metalliferes OD, Romania.
Borcos, 1967									H	Various; Toroiaga-Tiganul OD, Romania.
Borcos and Iosof, 1965									H	S; various OD.
Borcos and Iosof, 1967									H	S; various OD.
Borcos and Manilici, 1965									D	Q, C, B, epidote; various OD.
Boulanger and Deicha, 1960.	X									Q; graphic granite from peg.
Boyer and others, 1967	X									Q; Au OD.
Boyle, 1954	(?)								D	Q; vein OD.
Boyle, 1960		X								Q veins; Au OD.
Bradshaw and others, 1968									D	Halite; various sources.
Bradshaw and Stoyel, 1968									H	Cassiterite, S, Q, F; Cornish Sn OD.
Bragin, 1964									HD	Q, C, F, B; OD.
Bratus', 1969									H	Apophyllite; Korsun-Novomirgorod pluton, U.S.S.R.
Bratus' and others, 1968									H	Q; pegs; Ukraine, U.S.S.R.
Breislak, 1818, p. 370-376						X				Q.
Brewster, 1823a	X	X						X		T, Q, F, etc.
Brewster, 1823b				X						Q; Quebec.
Brewster, 1826a	X	X						X		T, Q, F, etc.
Brewster, 1826b	X				X	X				T, amber, etc.
Brewster, 1827	X							X		Sapphire.
Brewster, 1845b	X	(?)	(?)					X		T.
Brewster, 1849	X	X						X		Beryl, calcite.
Brewster, 1853a					X	X				Amber.
Brewster, 1853b								X (opaque and transparent).		T.
Brewster, 1862	X	X	X					X		Diamond, T, etc.
Brinck, 1956	X							X		Q; Au veins; Surinam.
Brown, C. E., 1967						X				F, Vugs in shale; Iowa.
Budzinski and others, 1959						X				Danburite; Zechstein halite.
Buerger, 1932a								Brine only		G; Joplin, Mo.
Buldakov, 1964	X									Q; huebnerite veins.
Bunsen, 1851					X					Halite; Poland.
Buseck, 1966									HC	Q; Concepcion del Oro OD, Mexico.
Butler and others, 1920, p. 358.		X						Hematite		Q; porphyry Cu OD.
Butuzov and Ikornikova, 1955.									H	Synthetic Q.
Cameron and others, 1951	X								H, D	Q, beryl; pegs.
Cameron and others, 1953	X								H	Q, beryl; pegs.
Chaigneau, 1967a	(?)									Apatite; Oka complex, Canada.
Chaikovsky, 1951	X								H	Q; peg; Volynia.
Chakravarty, 1967									H, D	Various; Bliava OD, Southern Urals, U.S.S.R.
Cifrulak and Cohen, 1969	X							NH_4Cl		Sylvite; Werra district; East Germany.
Cleveland, 1964									H	S; various Mississippi Valley OD.
Comte and Deicha, 1956	(?)								D	Q; W OD.
Correns, 1953	X									General.
Correns, 1954	X								HC	Aquamarine, halite, etc.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Courtis, 1890	X									Q; Au OD.
Coy-Yll and Font-Altaba, 1966.									D	G, S, B, and pyrite; Tarragona, Philippines.
Currie, 1951								Quartz		Apatite; Ontario.
Czamanske and others, 1963.					X	X				F; Pb-Zn OD.
Davy, 1822						X				Q; various localities.
Dawson, 1966						X				B; Nova Scotia.
Deicha, 1948									H	Albite; meta.
Deicha, 1949a									H	Q, C, albite; meta.
Deicha, 1949b									H	Q; meta.
Deicha, 1950a	X									Q; meta.
Deicha, 1950b	X									General.
Deicha, 1950c	X									Q; granite.
Deicha, 1950d	X									General.
Deicha, 1950e	X								HC	Q; meta.
Deicha, 1952a	X									Q; peg.
Deicha, 1952b								X	H	Q.
Deicha, 1954	X								C	Q; Sn and W OD.
Deicha, 1955	X	X	X	X				X, glass	HCD	Various (including villiaumite).
Deicha, 1958	X									Q; meta.
Deicha, 1965									H	OD: Copiapo, Chile.
Dekate, 1961	X								H	B; Pulivendla, India.
Dekate, 1963		X							H	Q, T, F, siderite; W OD.
Dellwig, 1955									HC	Halite.
Dewey, 1818						X			C	Q.
Dmitriev, 1958									HD	Q, F; Kazakhstan pegs.
Dobretsova, 1968									C	Various.
Dolgov, 1954									D	Q; sed; U.S.S.R.
Dolgov, 1955									D	High temperature quartz; U.S.S.R.
Dolgov, 1957a								X	D	Q; peg; U.S.S.R.
Dolgov, 1957b									H	Q; vein OD; U.S.S.R.
Dolgov, 1963								X, glass	H	Q, T, beryl; pegs; U.S.S.R.
Dolgov, 1964a									H	Q; pegs; U.S.S.R.
Dolgov, 1964b	X							X	H	Q, T; pegs; U.S.S.R.
Dolgov, 1965a	X							H_2S (freezing stage).	C	Q, T, beryl, F, tourmaline; pegs; U.S.S.R.
Dolgov, 1965c									D	Various minerals (a review)
Dolgov, 1968a	X							X	H	Q, T; pegs; U.S.S.R.
Dolgov, 1968c	X								HC	T; pegs; U.S.S.R.
Dolgov, 1968d								Many	H	Various minerals.
Dolgov and Bakumenko, 1964.	(?)							H_2S (separate phase?)	H	Q; peg; U.S.S.R.
Dolgov, Bazarov, and Bakumenko, 1968.	X								H, C	Various minerals.
Dolgov, Makagon, and Sobolev, 1967.	X							X (opaque and transparent).	HC	Kyanite; schists and pegs; U.S.S.R.
Dolgov and Popova, 1968	X								H, C	Various minerals.
Dolgov and Serebrennikov, 1968.									D	Q; various sources.
Dolgov and Shugurova, 1966a.								H_2S?	H	Q, F, S; various deposits; U.S.S.R.
Dolgov and Shugurova, 1966b.								H_2S		Q, T; pegs; U.S.S.R.
Dolgov and Shugurova, 1968.									H	Various minerals.
Dolomanova, 1966	X	X						X	D	Q, T, S, tourmaline and molybdenite(?); various OD; U.S.S.R.
Dolomanova and others, 1966	X	X						H_2S, chromite(?), X.		Q; Sherlovogora and Spokoynoye OD; Transbaikal.
Dolomanova and others, 1968.	X	X						H_2S, chromite(?), X.		Q; Transbaikal.
Dolomieu, 1792						X				Q crystals; Italy.
Dombrowski, 1966								Living bacteria		Paleozoic halite; primary inclusions.
Dons, 1956								H_2S	D	B; sed; concretion.
Dontsova and Naumov, 1967.									HD	Q; Various hydrothermal OD.
Doroshenko, 1966									H	B; F OD; Transbaikal.
Doroshenko and others, 1968.									H	F; various OD.
Dreyer and others, 1949									H	Halite.
Drozdova and others, 1964							X		D	F; F OD.
Dumas, 1830					X					Halite.
Dunham and others, 1965, p. 416.									H	Veins near Rookhope (work by F. J. Sawkins).
Dunn, 1929, p. 175								Gold		Q; Au OD.
Dwight, 1920									C	Q.
Dymkin and others, 1967									H	Scapolite; Turgai magnetite deposit, U.S.S.R.
Dzhafarov, 1961									D	Pyrite OD.
Eckermann, 1948, p. 81, 112, 154.	X									Carbonatite; Alnö Island, Sweden.
Efimova, 1966									H, D	S, G, C, Q; Tetyukhe Sn-Pb-Zn OD; U.S.S.R.
Elinson and Polykovskii, 1961a, 1961b.									H	Q; pegs; Maidantal.
Elinson, Polykovskii, and Shuvalov, 1969.									D	Q; Q-Wolframite veins; Maidantal.
Ellsworth, 1932						X	X	Fluorine (antozonite).		Various Canadian pegs.
Engel, 1946							X			Q; western Arkansas.
Eppler, 1962a		X	X					X		Emerald, T, etc.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Eppler, 1963		(?)		X				X		Emerald; Burbar, Colombia.
Eppler, 1966								X (birefringent and opaque).		Blue spinel; Ceylon.
Erd and others, 1957									HD	C; sed; Indiana.
Erhard and Stelzner, 1878	X								H	T.
Erickson, 1965									H	C; Mississippi Valley Pb-Zn OD.
Ermakov, 1943									H	F; hyd. OD.
Ermakov, 1944a									HD	Q, F, B, C; hyd. OD.
Ermakov, 1944b									H	Q, R, C, B; various OD.
Ermakov, 1945a									H	C (Iceland spar).
Ermakov, 1945b	X	X	X					Hematite	H	Q; meta veins.
Ermakov, 1949b								Glass, graphic granite.	H	T; pegs. Also saline minerals.
Ermakov, 1950a	X	X	X	X	X	X	X	Many	DH	Various samples.
Ermakov, 1950b									H	Q; meta; Briestenstock (Alps), etc.
Ermakov, 1950c									H, D	Various.
Ermakov, 1957	X	X	X					X	H	S; various OD, general.
Ermakov, 1965a	X								H	Q.
Ermakov, 1965b	X	X	X					X, hematite, chlorite, albite, quartz, rutile, zircon, muscovite, staurolite, sphene, magnetite.	H	Q, T, F; chambered peg; Volynia.
Ermakov, 1966c									D	Review of use for exploration.
Ermakov, 1968b									D	Various OD.
Ermakov and Gromov, 1968.									D	Halo in country rock of peg; U.S.S.R.
Ermakov, Kalyuzhnyi, and Myaz', 1957.	X	(?)	(?)					X, second liquid.	H	Q; pegs; Volynia.
Evzikova and Moskalyuk, 1964.							X	X	H	C; carbonatites.
Fahey, 1962								X		Northrupite; Green River Formation.
Faiziev, 1965									HD(?)	F, Q, pyrite; Krasnye Kholmy OD, U.S.S.R.
Fedorchuk, 1963							X	Dolomite, H_2S		Hg-Sb OD.
Fedorchuk, 1965									H	Q; Hg OD.
Feklichev, 1962	X	X		X				X	H	Pegmatitic beryl.
Ferguson and Gannett, 1932.									"H"	Q; Au veins.
Flanigen and others, 1967								Crystallized and glassy flux, etc.		Synthetic emerald.
Florovskaya and others, 1966.						X	X	Various aromatics.		Ign; Khibiny and Ilmen massifs, U.S.S.R.
Font-Altaba, Montoriol-Pous, and Amigo, 1966.									D	F; Spain.
Ford and King, 1965, p. 1692, 1698.							X			C; Derbyshire Pb-Ba OD.
Fosberry, 1963								H_2S		Q and C; South-West Africa.
Frank-Kamenetsky, 1951									U	B OD.
Freas, 1961					X	X	X		H	S, F, Q: Zn OD.
Fryklund and Fletcher, 1956.									D	S; Coeur d'Alene district, Idaho.
Galkiewicz, 1965									H	S; Silesia-Krakow Zn OD.
Gapon, 1962									"D"	Native gold.
Garbuzov and Khetchikov, 1965.									D	S, G; Tetyukhe OD, U.S.S.R.
Gerlach and Heller, 1966						X			H	Halite; natural and synthetic.
Germanov, 1946						X				Q, F; Cu-pyrite OD; Armenia.
Girault, 1966	X							X	D	Apatite; Oka, Canada.
Girault, 1967	(?)	(?)						X	D	Apatite; Oka, Canada.
Girault and Chaigneau, 1967.	X									Apatite; Oka, Canada.
Giuscă and others, 1968									U	Various Romanian OD.
Gol'dberg, 1967										Alkalic and ultramafic dike rocks; U.S.S.R.
Gol'dberg and Belyayeva, 1965.						(?)	(?)			C; Lower Tunguska OD, U.S.S.R.
Golovchenko, 1966									H	Cinnabar; Hg OD; U.S.S.R.
Go-Tsin, 1965									D	S, G, pyrite; OD.
Govorov and others, 1968								X	HD	Various minerals; Sn-W OD; Far East (U.S.S.R.).
Grigorchuk, 1962									U	Q, C, S, G; Pb-Zn OD.
Grigorchuk, 1964									H	Q; Krasnoyarovo-Zolinsk OD, U.S.S.R.
Grigorchuk and others, 1964.								X	H	Q, C, S; Krasnoyarovo-Zolinsk OD, U.S.S.R.
Grogan and Shrode, 1952					X	X	X		H	F; F-S OD.
Gromov, 1968a								Many minerals, glass.		Various minerals from granites.
Gromov, 1968b									D	Q, F, molybdenite, davidite; peg; U.S.S.R.
Gromov and Krestin, 1967								Glass, gas, and crystals.		Olivine and plagioclase; Siberian trap rocks; U.S.S.R.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Groves and Solomon, 1969	X	X	X					X, Sulfides	H, C	Q, F; Sn OD; Mount Bischoff, Tasmania.
Grubb, 1964								Quartz, fluorite		Beryl; pegs.
Grushkin, 1948a									H	Q; Aurakhmat polymetallic OD, U.S.S.R.
Grushkin, 1948b									H	F, Q; Aurakhmat polymetallic OD, U.S.S.R.
Grushkin, 1949									H	F, Q; Aurakhmat polymetallic OD, U.S.S.R.
Grushkin, 1950									H	F, Q; Aurakhmat and Chatmazar OD, U.S.S.R.
Grushkin, 1953	X								H	F, Q; OD; U.S.S.R.
Grushkin, 1954									H	C, F; OD; U.S.S.R.
Grushkin, 1958									H	F; Aurakhmat polymetallic OD, U.S.S.R.
Grushkin and Bykov, 1952									H	Peg minerals; Oigainga, U.S.S.R.
Grushkin and Khel'vas, 1950.									H	F, Q, C; Chatmazar and Bulatsay, U.S.S.R.
Grushkin and Khel'vas, 1951.									H	F.
Gübelin, 1943	X	X								Various gems.
Gübelin, 1945	X									Emerald.
Gübelin, 1950		X		X						Emerald.
Gübelin, 1953	X	X								Various gems.
Gübelin, 1957	X	X								Various gems.
Gübelin, 1964								Phenacite		Synthetic emerald.
Guilhaumou and Ognar, 1969.	X	X	X					Hematite(?)	HC	Dolomite; Tunisia.
Gurevich, 1961	X	X					X			Various minerals.
Gurova, 1968									H	F; Podolian sandstones; U.S.S.R.
Gurova and Galetskii, 1968.									H	F; various ign.
Gurova and Marchenko, 1968.									H	F; various ign.
Gurova and Val'ter, 1968									H	F; OD; Podolia.
Hall, 1967									HC	F; southern Illinois OD.
Hammerschmidt, 1883		X								Gypsum and anhydrite.
Harrington, 1905	X							H_2S(?)	H	F; OD; Podolia Marble from Grenville Series.
Hartley, 1876a	X								H	Q, T.
Hartley, 1876b	X								H	Q.
Hartley, 1876c	X								H	Various.
Hartley, 1877a	X								H	T, various.
Hartley, 1877b	X								H	Q, and other minerals.
Hartley, 1877c	X								H	Q; ign.
Hawes, 1878	X	X							H	F, Q, beryl, hornblende.
Hawes, 1881	X				X					Q, peg.
Hayden, 1819								H_2S(?)		Feldspar(?); near Baltimore, Md.
Hegel and Schlossmacher, 1956.	X									T.
Hidden, 1882	X									Q; meta.
Hoagland, 1951a	X	X		X						Emerald.
Holden, 1925	X								H	Q.
Holland, T. H., 1900	X									Massive Q veins associated with periodotites; India.
Huebner, 1962, p. 27		X						X, CaF_2(?)	HC	S, Q; Pb-Zn OD; Providencia, Mexico.
Hunt, 1894	X	X								Q; granite.
Hunt, 1908	X	X								Q; granite.
Hunt, 1909		X								Q; Sn OD.
Hutchison, 1966									D	Various micas.
Iddings, 1892, p. 345		X								Q; ign; Nevada.
Idriceanu, and others, 1965.									H	Q; OD Apuseni Mountains, Romania.
Ikorskii, 1962					X			X	HC	Lovozero alkalic massif, U.S.S.R.
Ikorskii, 1964					X	X			H	Various minerals; Khibinsk massif, U.S.S.R.
Ikorskii, 1965					X	X		X	D	Nepheline; Khibinsk massif, U.S.S.R.
Ikorskii, 1966a						X	X	Villiaumite, NaF.	H	Eudialyte; Khibinsk massif, U.S.S.R.
Ikorskii, 1966b					X	X			H	Various minerals; Khibinsk massif, U.S.S.R.
Ikorskii, 1967a, b					X	X	X	Villiaumite, NaF	H	Various minerals; Khibinsk massif, U.S.S.R.
Ikorskii, 1968					X	X	X	Villiaumite, NaF.	H	Various minerals; Khibinsk massif, U.S.S.R.
Ikorskii and Romanikhin, 1964.					X	X				Nepheline; Khibinsk massif, U.S.S.R.
Il'in, 1964									H	Iceland spar OD.
Imai, 1961									D	Q; Au vein OD.
Imai, 1966									D	Q, F, S, wolframite, scheelite, cassiterite, etc; various Japanese OD.
Imai and Ito, 1959									H	Q; W-Cu OD.
Ingerson, 1947									H	Q; peg.
Ingerson, 1955, p. 398									H	C; Mexico.
In'shin, 1958	X								H	Q crystal OD.
In'shin, 1959	X	X						X	H	Q.
Ivanov, 1963									D	Cassiterite, Q, S; Sn greisens.
Ivantishin, 1955		X	X					Cassiterite(?)	H	T; peg; Korostenj, U.S.S.R.
Iwao and others, 1953									D	Q; veins.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Janković, 1968	--------	--------	--------	--------	--------	--------	--------	--------------	D	Q; OD; Yugoslavia.
Jepsen, 1964	--------	--------	--------	--------	--------	--------	--------	--------------	H	C, Q; dinosaur bones.
Johnsen, 1920	X	--------	--------	--------	--------	--------	--------	--------------	H	Q.
Johnson, 1961a	--------	X	--------	--------	--------	--------	--------	--------------	----------	Emerald.
Johnston and Butler, 1946	--------	--------	--------	--------	--------	--------	--------	H_2S	----------	Q; meta veins; Xique-Xique, Brazil.
Jolly and Heyl, 1968	--------	--------	--------	--------	--------	--------	--------	--------------	C	F; Zn OD; middle Kentucky.
Judd, 1881	X	X	X	--------	--------	X	--------		----------	Various minerals.
Judd, 1889	--------	X	--------	--------	--------	--------	--------	--------------	----------	Scapolite.
Julien, 1881	X	--------	--------	--------	--------	--------	--------	--------------	H	T and various rocks.
Julien, 1884	X	--------	--------	--------	--------	--------	--------	--------------	H	Various minerals.
Julien, 1885	X	--------	--------	--------	--------	X	--------	--------------	H	Q, Herkimer, N.Y.; T, Brazil, and others.
Kalita, 1963	--------	--------	--------	--------	--------	--------	--------	--------------	D	Q, F, peg; U.S.S.R.
Kalita and Kudakova, 1966.	--------	--------	--------	--------	--------	--------	--------	--------------	D	Q, F, microcline; peg; U.S.S.R.
Kalyuzhnyi, 1955a	X	X	--------	--------	--------	--------	--------	--------------	H	Q; meta veins; U.S.S.R.
Kalyuzhnyi, 1956	--------	--------	--------	--------	--------	--------	--------	X, elpasolite	H	T; peg; Volynia.
Kalyuzhnyi, 1957	X	--------	--------	--------	--------	--------	--------	X, goethite	----------	Q, T, F, etc; peg; U.S.S.R.
Kalyuzhnyi, 1958a	--------	X	X	--------	--------	--------	--------	X, teepleite, cryolite, quartz.	----------	T; peg; Volynia.
Kalyuzhnyi, 1960	X	X	X	X	--------	--------	--------	X	H	Various samples.
Kalyuzhnyi, 1961	X	X	X	--------	--------	--------	--------	Many	----------	Q, B, T.
Kalyuzhnyi, 1965b	--------	--------	--------	--------	--------	--------	--------	Glass, with liquid water.	H	Phenocrysts in ign.
Kalyuzhnyi and Koltun, 1953.	X	--------	--------	--------	--------	--------	--------	--------------	H	Q, S, ankerite; veins, meta; U.S.S.R.
Kalyuzhnyi and others, 1966.	X	--------	--------	--------	--------	--------	--------	Quartz(?)	H	Q; peg; Volynia.
Kalyuzhnyi and Pritula, 1967	X	X	--------	--------	--------	--------	--------	X	----------	T; peg
Kalyuzhnyi and Shchiritsya, 1962.	X	--------	--------	--------	--------	--------	--------	--------------	H	Nalgol'nyi Ridge OD, U.S.S.R.
Kalyuzhnyi and Voznyak, 1967.	X	--------	--------	--------	--------	--------	--------	$FeCl_2 \cdot 2H_20$, topaz, K-feldspar.	H	Q; Zanorysh type peg; U.S.S.R.
Kantor and Eliáš, 1968	--------	--------	--------	--------	--------	--------	--------	--------------	D	Q; various stibnite deposits.
Kapchenko, 1963	--------	--------	--------	--------	X	--------	--------	--------------	----------	Various samples—a review.
Kapustin, 1966	--------	--------	--------	--------	--------	--------	--------	--------------	U	Apatite; Kola, U.S.S.R.
Karamyan and Faramazyan, 1959.	--------	--------	--------	--------	--------	--------	--------	--------------	U	Gypsum; Cu-Mo OD; U.S.S.R.
Karamyan and Madanyan, 1968.	--------	--------	--------	--------	--------	--------	--------	--------------	H	Q; Kadzharan Cu-Mo OD, U.S.S.R.
Karpinskii, 1880	X	--------	--------	--------	--------	--------	--------	--------------	H	Q; peg; Urals.
Karskii and Zorin, 1968	--------	--------	--------	--------	--------	--------	--------	--------------	H	Muscovite; peg; Mamsk.
Karyakin, 1954	--------	--------	--------	--------	--------	--------	--------	--------------	H	Q; Urals.
Karyakin and Piznyur, 1965.	--------	--------	--------	--------	--------	--------	--------	--------------	H	Q, C; near-polar Urals.
Kashiwagi and others, 1955.	--------	--------	--------	--------	--------	--------	--------	--------------	HD	Q; OD.
Kashkai and others, 1968	--------	--------	--------	--------	--------	--------	--------	--------------	H, D	Garnet; Dashkesan Fe OD, U.S.S.R.
Kazanskiy and others, 1968.	--------	--------	--------	--------	--------	--------	--------	--------------	HC	Q; Precambrian sed; U.S.S.R.
Keith and Tuttle, 1952	--------	--------	--------	--------	--------	--------	X	--------------	H	Q; sed (two types of organic solids).
Kelly and Goddard, 1966	--------	--------	--------	--------	--------	--------	--------	--------------	H	Telluride ores; Colorado.
Kelly and Turneaure, 1969.	X	X	--------	--------	--------	--------	--------	Magnetite plus 23 other phases.	H, C	Q, cassiterite, etc.; various OD; Bolivia.
Kennedy, 1950a	--------	--------	--------	--------	--------	--------	--------	--------------	H(D)	F.
Kerkis, 1966	--------	--------	--------	--------	--------	--------	--------	--------------	H	Nepheline; eastern Sayan, U.S.S.R.
Kerkis and Kostyuk, 1963	--------	X	--------	--------	--------	--------	--------	--------------	H	Nepheline; Botogol deposit, U.S.S.R.
Khairetdinov and others, 1965.	--------	--------	--------	--------	--------	--------	--------	--------------	D	Pyrite and marcasite OD.
Khakimov, 1966	--------	--------	--------	--------	--------	--------	--------	--------------	H	Q; hyd veins in porphyry; Pamir.
Khakimov, 1968	--------	--------	--------	--------	--------	--------	--------	X (colloidal)	----------	Q; Idzhebanskii OD, Armenia.
Khel'vas, 1959	--------	--------	--------	--------	--------	--------	--------	--------------	U	Q; Fe OD.
Khel'vas, 1964	--------	--------	--------	--------	--------	--------	--------	--------------	H	F; Pb-Zn skarn OD; U.S.S.R.
Khetchikov, 1965	--------	--------	--------	--------	--------	--------	--------	--------------	D	G, S; Tetyukhe OD, U.S.S.R.
Khetchikov, Balitskii, and others, 1966.	--------	--------	--------	--------	--------	--------	--------	--------------	D	Synthetic quartz.
Khetchikov, Dorogovin, and Samoilovich, 1968.	--------	--------	--------	--------	--------	--------	--------	--------------	HD	Synthetic quartz.
Khetchikov, Efimova, and others, 1966a.	--------	--------	--------	--------	--------	--------	--------	--------------	H	Q; Sn-sulfide OD; U.S.S.R.
Khetchikov and Garbusov, 1964.	--------	--------	--------	--------	--------	--------	--------	--------------	D	S, G; polymetallic skarn OD; Tetyukhe, U.S.S.R.
Khitarov, N. I., 1965	--------	X	--------	--------	--------	--------	--------	--------------	H	Granite peg.
Kholmskiy, 1965	--------	--------	--------	--------	--------	--------	--------	--------------	H	Q, C; Fe OD in KMA; U.S.S.R.
Khundadze, 1965	--------	--------	--------	--------	--------	--------	--------	--------------	U	B, C; Chordsk barite OD; U.S.S.R.
Kinoshita, 1924	X	--------	--------	--------	--------	--------	--------	--------------	H	Gypsum.
Kirkham, 1969	--------	X	--------	--------	--------	--------	--------	X, ore mineral	H	Q; Mo OD; British Columbia.
Kirsch, 1956	--------	--------	--------	--------	--------	--------	--------	--------------	H	F (octahedral and cubic); Erzgebirge.
Kiyevlenko, 1958	--------	--------	--------	--------	--------	--------	--------	--------------	H	Iceland spar; Siberia.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Kiyevlenko, 1959									DH	C; Iceland spar OD; Siberia.
Klevtsov and Lemmlein, 1958.	X	X							CH	Q; southern Urals.
Klevtsov and Lemmlein, 1959a.		X							H	Q; veins, meta; U.S.S.R.
Kliya and Lemmlein, 1961	X								H	Q; veins, meta; U.S.S.R.
Koizumi and Hosomi, 1953									D	Q.
Kokorin, 1966									D	Various minerals; Amur tin OD, U.S.S.R.
Kokorin and Kokorina, 1966.									H	Various minerals; Amur tin OD, U.S.S.R.
Kokorin and Kokorina, 1968.									H	Q; Sn OD; U.S.S.R.
Kokta, 1956									H, D	F; Kožlí and Vrchoslav, Czechoslovakia.
Koltun, 1953									H	Colored tourmaline.
Koltun, 1957	X								H	Q; Tourmaline; Au-W OD; U.S.S.R.
Koltun, 1958	X	X	X					X	H	Au OD; Lebedino, Aldan, U.S.S.R.
Koltun, 1966		X							H	Q; Aldan, U.S.S.R.
Koltun and Golovchenko, 1962.									H	Q; Hg-As-Sb OD; U.S.S.R
Koltun and Lokerman, 1962.									H	G, S, garnet, etc.; OD.
Koltun and others, 1961									H	Axinite; various OD.
Koltun and others, 1963									H	Q, F, G, etc.; OD.
Komarov and Parshin, 1965.									D	Cosalite, Q; Karaoba OD, U.S.S.R.
Komarov and Parshin, 1966.									D	Q, wolframite; OD; Kazakhstan.
Königsberger, 1901, p. 210									U	Q; Alpine veins.
Königsberger and Müller, 1906a.	X								H	Q; meta veins; Bächistock.
Königsberger and Müller, 1906b.	X								H	Q; meta veins; Aar massif.
Konta, 1951									D	Q, F; Sn-W OD; Zinnwald, Czechoslovakia.
Konta, 1962	X	X							D	Q, F, T, beryl; Sn-W OD.
Konta and others, 1952									D	Q, feldspar; peg.
Koplus and Kudakova, 1968.									D	Q vein OD; Yakutia.
Kormushin, 1965									C	F; Keremettas OD, U.S.S.R.
Kornilov, 1962									D	Q, Pechenga Cu-Ni OD, U.S.S.R.
Korostelev and Chainikova, 1966.									D	S; Maritime OD, U.S.S.R.
Koster van Groos, 1966		X						Glass		Synthetic, in system albite-NaCl-H_2O.
Koster van Groos and Wyllie, 1969.		X						Glass		Synthetic, in system albite-NaCl-H_2O.
Kostov, 1963									H	Epidote, Q; peg.
Kostyleva, 1964									D	Q; various OD.
Kostyleva, 1965									D	Q, F, etc; OD and peg; U.S.S.R.
Kostyleva, 1968									D	F, apatite; various sources.
Kostyleva and Labuntsov, 1958.									D	Q; various.
Kostyuk and Bazarova, 1965.									H	Nepheline; Botogol deposit, U.S.S.R.
Kotov and others, 1968									H, D	Q-Mo OD; U.S.S.R.
Kovalishin, 1962								H_2S		Q; peg ("stinkrock"); Volynia.
Kovrigo and Izyumskii, 1968.									H	Various; Leninogorsk OD, U.S.S.R.
Kravtsov and Fridman, 1965.								H_2S, SO_2		Chalcopyrite OD; north-central Caucasus.
Krendovskiy, 1879		X	X					X, alum(?)		Granites.
Krol and Gurkina, 1957	X								U	F; rare earth peg.
Krylova, 1962									D	Q; granite peg; Aldan, U.S.S.R.
Kudenko and Stetsenko, 1964.									D	S; various OD.
Kühn, Peter, 1959									H	S; Bánská Stiavnica and Stříbro OD, Czechoslovakia.
Kühn, Peter, 1962									H	S; Zn OD.
Kühn, Peter, 1966									H	S; Zn OD.
Kühn, Robert, 1968						X	X			German potash deposits.
Kukharenko and Dontsova, 1962.									D	Various minerals from carbonatites.
Kupka, 1961									D	General.
Kurovets and Piznur, 1961									H	Monazite; peg.
Kurshakova, 1961									D	Skarn minerals.
Kuznetsov, 1968									D	Halo around polymetallic OD; U.S.S.R.
Kuznetsov and Kobilev, 1967.	X								H	Q; volcanic rocks of northern Osetia, U.S.S.R.
Lamar and Shrode, 1953					X				D	C; sed.
Lapukhov, 1968									H	Q, B; Salair OD, U.S.S.R.
Laspeyres and Kaiser, 1895.	X									Q; ign.
La Vallée and Renard, 1876.		X							H	Q; ign.
Lavrenko, 1961									U	Q; Fe OD.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Lawrence, 1938	--------	--------	--------	--------	--------	--------	X	H_2S	D	B, black; east Tennessee.
Lazarenko, E. E., 1967	--------	--------	--------	--------	--------	--------	--------	X	H	Q; Volynia; U.S.S.R.
Lazarenko, E. K., 1955, p. 112–114.	X	--------	--------	--------	--------	--------	--------	--------	HD	S; pyrite OD.
Lazarenko and others, 1963.	--------	--------	--------	--------	--------	--------	--------	--------	U	Realgar, metacinnabar B, C, S, Q, zircon, tridymite.
Lazarenko and others, 1967.	--------	--------	--------	--------	--------	--------	--------	--------	H	Pegs; Volynia.
Lazarenko and Slivko, 1958.	--------	--------	--------	--------	--------	--------	--------	"SO_3 gas"	--------	Celestite (with sulfur); Dnester region, U.S.S.R.
Laz'ko, 1949	X	--------	--------	--------	--------	--------	--------	--------	U	Various.
Laz'ko, 1957a	--------	X	--------	--------	--------	--------	--------	--------	--------	Q.
Laz'ko, 1957b	X	X	--------	--------	--------	--------	--------	--------	H	Q; Kurumkan OD, U.S.S.R.
Laz'ko and others, 1965	X	--------	--------	--------	X	--------	--------		--------	Various OD and peg.
Lea, 1870	X	X	(?)	--------	--------	--------	--------	--------	--------	Various gems.
Lebedev, 1965 (p. 175 of translation).	--------	--------	--------	--------	--------	--------	--------	H_2S	--------	G; Iokun'zh Pb-Zn OD, U.S.S.R.
Lemmlein, 1950a	--------	--------	--------	------	--------	--------	--------	Quartz, topaz on walls.	--------	Q, T.
Lemmlein, 1956a	--------	--------	--------	--------	--------	--------	--------	Topaz on walls	--------	T.
Lemmlein and Klevtsov, 1955.	X	X	X	X	--------	--------	--------	--------	C, H	Q, T, beryl; Pamir, U.S.S.R.
Lemmlein and Kliya, 1952a.	--------	--------	--------	--------	--------	--------	--------	X, topaz on walls.	H	T; peg.
Lemmlein and Kliya, 1952b.	--------	--------	--------	--------	--------	--------	--------	X	--------	T and other minerals.
Lemmlein and Kliya, 1954.	--------	--------	--------	--------	--------	--------	--------	Glass	U	Q; ign phenocrysts.
Lemmlein and others, 1962.	--------	--------	--------	--------	--------	--------	--------	X, quartz, muscovite, cryolite, fluorite.	H	T; peg.
Lesnyak, 1954a	--------	--------	--------	--------	--------	--------	--------	--------	H	Q, S, C; Pb-Zn OD.
Lesnyak, 1954b	--------	--------	--------	--------	--------	--------	--------	--------	H	Q, S, C; Pb-Zn OD.
Lesnyak, 1955	--------	--------	--------	--------	--------	--------	--------	Up to 8 crystals, pyrite, hematite; glass.	H	Ign (granitoid).
Lesnyak, 1957a	--------	X	X	--------	--------	--------	--------	X (pyrite?)	H	Various skarn minerals.
Lesnyak, 1957b	--------	--------	--------	--------	--------	--------	--------	--------	H	Gypsum; sed OD.
Lesnyak, 1958	--------	X	--------	--------	--------	--------	--------	--------	--------	Q; ign.
Lesnyak, 1961	X	X	--------	--------	--------	--------	--------	X	U	Various skarn minerals.
Lesnyak, 1962	--------	(?)	--------	--------	--------	--------	--------	--------	H	Q; Sb OD in skarn.
Lesnyak, 1963	--------	X	--------	--------	--------	--------	--------	X, glass	H	Various OD.
Lesnyak, 1965	--------	X	X	--------	--------	--------	--------	X, pyrite(?)	H	Q, F, C; Tyrny-Auz skarn OD, U.S.S.R.
Lesnyak and Uskov, 1965	--------	--------	--------	--------	--------	--------	--------	--------	H	F; Abagaitui F-B OD, Transbaikal.
Levenshtein and others, 1969.	--------	--------	--------	--------	--------	--------	--------	--------	H	F; Donets basin, U.S.S.R.
Levitskiy, Yu. F., and Moskalyuk, A. A. in Khitarov, 1966.	--------	--------	--------	--------	--------	--------	--------	--------	U	F; Transbaikal, U.S.S.R.
Lézier, 1966	(?)	--------	--------	--------	--------	--------	--------	--------	--------	Peridotite; Auvergne.
Lhamsuren, 1968a	--------	--------	--------	--------	--------	--------	--------	X	--------	T; greisen OD; Baga-Gazaryi-Chulu, central Mongolia.
Lhamsuren, 1968b	--------	X	X	--------	--------	--------	--------	Many	--------	T; greisen OD; Baga-Gazaryi-Chulu, central Mongolia.
Lhamsuren, 1968c	--------	--------	--------	--------	--------	--------	--------	--------	H	Q; Zuun Bayan OD, central Mongolia.
Lindgren, 1905, p. 213–218	--------	X	--------	--------	--------	--------	--------	Hematite(?) and wall lining.	--------	Q; veins and ign; Cu OD.
Lindgren and Whitehead, 1914.	--------	X	--------	--------	--------	--------	--------	X, colemanite(?) in danburite.	--------	Q and danburite, Pb OD.
Lisitsyn and Malinko, 1957.	X	X	X	X	--------	--------	--------	--------	--------	Q; meta veins.
Lisitsyn and Malinko, 1961.	X	X	X	X	--------	--------	--------	--------	HC	Q; meta veins.
Little, 1955	X	X	--------	--------	--------	--------	--------	X (silicates?)	HCD	Q, cassiterite; Sn OD.
Little, 1960	X	X	--------	--------	--------	--------	--------	X	HCD	Q, cassiterite; Sn OD.
Litvinovskii, 1967	--------	--------	--------	--------	--------	--------	--------	X	H	Nepheline; Bambuisk intrusion, U.S.S.R.
Llambias, 1963	--------	--------	--------	--------	--------	X	X	Borax in borax	--------	Borax and inderite.
Loskutov, 1962	--------	--------	--------	X	--------	--------	--------	Na, NH_4 carbonates-phosphates	H	Natrolite; ign.
Lovering, T. G., 1958	--------	--------	--------	--------	--------	--------	--------	--------	H	Q; polymetallic OD.
Lyakhov, 1963	--------	--------	--------	--------	--------	--------	--------	--------	H	Q; pegs; Volynia.
Lyakhov, 1966	--------	X	X	X	--------	--------	--------	X, $FeCl_2 \cdot 2H_2O$, elpasolite, cryolite, hydrated magnesium carbonate.	H	Q; pegs; Volynia.
Lyakhov, 1968	--------	--------	--------	--------	--------	--------	--------	--------	H	Various minerals; Baleisk OD, eastern Baikal.
Lyakhov and Myaz', 1966	--------	--------	--------	--------	--------	--------	--------	--------	H	Q, C; Baleisk OD, eastern Baikal.
Lyakhov and Piznyur, 1964	X	--------	--------	--------	--------	--------	--------	--------	H	Q; Au and Q deposits, U.S.S.R.
McCulloch, 1959	--------	--------	--------	--------	--------	--------	--------	--------	H	Synthetic NaCl.
Macdonald and Katsura, 1965.	--------	--------	--------	--------	--------	--------	--------	Fluorite(?), glass.	--------	Phenocrysts from volcanics; Lassen Peak, Calif.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Machairas, 1963a	X	--------	--------	--------	--------	--------	--------	--------	--------	Various peg minerals.
Machairas, 1963b	X	--------	--------	--------	--------	--------	--------	--------	D	Q; Au OD.
Machairas, 1963c	X	--------	--------	--------	--------	--------	--------	--------	--------	Various peg minerals.
Machairas, 1963d	--------	--------	--------	--------	--------	--------	--------	--------	D	Quartz.
McMahon, 1884a	X	--------	--------	--------	--------	--------	--------	X	--------	Beryl, feldspar, garnet; Indian granites.
McMahon, 1884b	X	X	--------	--------	--------	--------	--------	Opaque	--------	Q; granite from Araváli, India.
McMahon, 1902	--------	--------	--------	--------	--------	--------	--------	X	--------	Beryl; Himalayas.
Madaliev, 1957	--------	--------	--------	--------	--------	--------	--------	--------	D(?)	Sb deposit; Gissar range.
Maiskii, 1968	--------	--------	--------	--------	--------	--------	--------	--------	H	T; pegs; southeastern Mongolia.
Maiskii and Trufanof, 1967.	X	--------	--------	--------	--------	--------	--------	--------	H	Various; Q-C veins; northwestern Caucasus.
Maleev, 1967	--------	--------	--------	--------	--------	--------	--------	--------	H	Various minerals; Hg OD; U.S.S.R.
Malinko, 1963	--------	--------	--------	--------	--------	--------	--------	--------	D	Various Ca-Mg borates.
Malinko, 1966	--------	--------	--------	--------	--------	--------	--------	--------	D	Various skarn borates.
Malyshev and others, 1964.	--------	--------	--------	--------	--------	--------	--------	--------	D	C, B, F, G, S, Q; Pb-Zn OD.
Manilici and others, 1965	--------	--------	--------	--------	--------	--------	--------	--------	H, D	Q; Baia Sprie OD.
Maslova, 1961	--------	--------	--------	--------	--------	--------	--------	H_2S	--------	Q; peg; Volynia.
Maslova, 1963	--------	--------	--------	--------	--------	--------	X	--------	--------	Q; Sb-Hg OD.
Maslova and Morozov, 1968.	--------	--------	--------	--------	--------	--------	--------	--------	H	Q; Pamir; U.S.S.R.
Maslyakevich, 1967	X	--------	--------	--------	X	X	--------	--------	H	Various postmagmatic rocks.
Matveyev, 1947	--------	--------	--------	--------	--------	X	X	--------	--------	Scheelite, Q, amazonite; Urals.
Mawson, 1906	--------	--------	--------	--------	--------	--------	X	CS_2(?)	--------	Feldspar and quartz; Australia.
Mawson and Dallwitz, 1945, p. 25 and 34.	--------	--------	--------	--------	--------	--------	--------	Liquid H_2S+CS_2(?).	--------	Albite leucogranite; Australia.
Mehnert, 1949	--------	--------	--------	--------	--------	--------	--------	--------	D	Q, F, C, B; Cu-Bi OD.
Mel'nikov, 1965	--------	X	--------	--------	--------	--------	--------	X	C	Q.
Mel'nikov, 1968a	--------	X	X	--------	--------	--------	--------	Many	H	Q, F.
Mel'nikov, 1968b	--------	--------	--------	--------	--------	--------	--------	--------	HC	Q, F.
Mel'nikov and Fratkin, 1968.	--------	X	--------	--------	--------	--------	--------	X	HC	Various minerals.
Mel'nikov and Ryabov, 1968.	--------	--------	--------	--------	--------	--------	--------	--------	D	Various sulfides (in argon).
Merlich, 1961	--------	--------	--------	--------	--------	--------	--------	--------	U	Hg-Pb-Zn and As-Sb OD.
Meyer, 1950	--------	--------	--------	--------	--------	--------	--------	--------	H	Q; monzonite, peg, and Mo veins; Butte, Mont.
Meyer and others, 1968	--------	--------	--------	--------	--------	--------	--------	--------	H	Q; Butte, Mont.
Miller, J. D., 1968, 1969	--------	--------	--------	--------	--------	--------	--------	--------	H	F; Zn OD; east Tennessee.
Mincheva-Stefanova and others, 1967.	--------	--------	--------	--------	--------	--------	--------	--------	H	S; Gradishte-Madansk, Pb-Zn OD.
Mirochnikov, 1968a	--------	--------	--------	--------	--------	--------	--------	--------	H, D	Q; vein OD.
Mirochnikov, 1968b	--------	--------	--------	--------	--------	--------	--------	--------	H, D	Q; various sources.
Miyazawa, 1967a, 1967b	--------	--------	--------	--------	--------	--------	--------	--------	H	Q; Japanese OD.
Miyazawa and Nozaki, 1957.	--------	--------	--------	--------	--------	--------	--------	--------	H	Q, C; OD.
Mogarovskii, 1963	--------	--------	--------	--------	--------	--------	--------	--------	H	Celestite, C, Q; sed.
Morgante, 1940	--------	--------	--------	--------	--------	X	--------	--------	H	F; vein with G and S.
Morozov, 1964	--------	--------	--------	--------	--------	--------	--------	--------	HD	Q; rock crystal veins; central Asia.
Morozov, 1966a	X	X	X	X	--------	--------	X	H_2S, hematite, hydromica.	--------	Q; rock crystal veins; central Asia.
Morozov, 1966b	--------	--------	--------	--------	--------	--------	--------	--------	H	Q; rock crystal veins; western Pamir.
Morozov, 1967	X	X	--------	--------	--------	--------	--------	--------	H	Q, crystal veins; Pamir.
Moskalyuk, 1961	--------	--------	--------	--------	--------	--------	--------	--------	DH	Q, F; OD, peg and veins.
Moskalyuk and Zakharchenko, 1966.	--------	--------	--------	--------	--------	--------	--------	X	DH	F, Q; Kayib massif, U.S.S.R.
Motorina, 1967	X	X	X	--------	--------	--------	--------	X, some opaque.	H	T, pegs; Volynia.
Motorina and Bakumenko, 1968.	X	--------	--------	--------	--------	--------	--------	--------	H	Tourmaline; peg; U.S.S.R.
Mueller, 1954a	--------	--------	--------	--------	--------	X	X	--------	--------	F; Pb-Zn vein OD.
Mueller, 1954b	--------	--------	--------	--------	X	X	X	--------	--------	F-bearing veins; British Isles.
Müller and Heymel, 1956	X	--------	--------	--------	X	--------	--------	--------	--------	Halite; potash deposits.
Murray, 1957	--------	--------	--------	--------	X	X	--------	--------	H	Q; sed.
Myaz', 1957	--------	--------	--------	--------	--------	--------	--------	X	H	Q; meta veins(?) (low temp).
Myaz', 1961a	--------	X	--------	--------	--------	--------	--------	--------	H	Q; meta veins.
Myaz', 1961b	--------	--------	--------	--------	--------	--------	--------	--------	H	Q crystal veins; Kazakhstan.
Myaz' and Koltun, 1960	--------	--------	--------	--------	--------	--------	--------	X	H	Epidote; OD.
Myaz' and others, 1968	--------	--------	--------	--------	--------	--------	--------	--------	H	Q, F; various sources.
Myaz' and Yasinskaya, 1965.	--------	--------	--------	--------	--------	--------	--------	--------	H	Fe-Mn OD; Kazakhstan.
Nacken, 1921	X	--------	--------	--------	--------	--------	--------	--------	--------	General.
Nadeau, 1967	--------	--------	--------	--------	--------	--------	--------	--------	H	F; Sweetwater barite OD, Tennessee.
Narseev and others, 1968	--------	--------	--------	--------	--------	--------	--------	--------	D	Various peg minerals; eastern Kazakhstan.
Nash, 1969	--------	--------	--------	--------	--------	--------	--------	--------	H, C	Q; various Au OD; Western United States.
Nauchitel', 1968	--------	--------	--------	--------	--------	--------	--------	--------	D	Cassiterite; U.S.S.R.
Naumann, 1911	--------	--------	--------	--------	--------	X	--------	--------	--------	NaCl; sed.
Naumov, 1968	--------	--------	--------	--------	--------	--------	--------	--------	D	Various minerals.
Naumov, Balitskii, and Khetchikov, 1966.	--------	--------	--------	--------	--------	--------	--------	--------	HD	Synthetic quartz, calcite, and villiaumite.
Naumov and Ivanova, 1967.	X	--------	--------	--------	--------	--------	--------	--------	H, D	Q, wolframite, huebnerite various OD; U.S.S.R.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample not
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Naumov and Khodakovskii, 1968.									D	Various minerals (a review).
Naumov and Malinin, 1968.	X								H, D	Q; various OD; U.S.S.R.
Naumov, Panov, and Mayshev, 1964.									D	Q; Zambarat OD, U.S.S.R.
Naumov and Suschevskaya, 1966.									D	Q, G, F, tourmaline, cassiterite, arsenopyrite, chalcopyrite; Myao-Chan.
Nedashkovskii, 1961									D	Albite, beryl, Q; ign and veins.
Nesmelova, 1959					X					Sylvite and carnallite; sed.
Newhouse, 1932								X		S; Pb-Zn OD.
Newhouse, 1933								H_2S	H	S; Pb-Zn OD.
Nichol, 1829									H	Halite; Cheshire, England.
Nicholas, 1951									H(?)	Q; kaolinized granites.
Nikanorov and Mikhailov, 1964a.		X		X				X, apatite	D	Peg minerals.
Nikanorov and Mikhailov, 1964b.									D	Q; peg.
Nikanorov and others, 1966.									D	Q; various.
Nikanorov and others, 1968.									H, D	Q; various peg; U.S.S.R.
Nikolayeva, 1954									D	Placer gold.
Nikulin, 1967									H	Q; Khingan tin OD; U.S.S.R.
Nishio and others, 1952									D	Q, C etc; various OD.
Nordenskiöld, 1886	(?)					X				T; peg.
Ódor and Szeredai, 1964									D	F; (by weight loss).
Oesterling, 1952, p. 337									D	S; Hutson mine; Kentucky.
Ohmoto, 1968a	X								H, C	Q, C, S; Bluebell mine, Canada.
Ohmoto and Rye, 1970				X				X	H, C	Q, C, S; Bluebell mine, Canada.
Oppenheim, 1948						(?)	X			Emerald; Muzo, Colombia.
Outerbridge, 1957									H	B, C, celestite; Colorado Plateau uranium OD.
Ovchinnikov and Chelishchev, 1967.									HD	Many OD.
Palache and others, 1951, p. 153.								H_2S		Fetid calcite (general).
Panina, 1966a		(?)						X	H	Various rock minerals; Synnyr alkalic massif, U.S.S.R.
Panina, 1966b								X	H	Nepheline syenite; Borgoi massif, U.S.S.R.
Panina and Lysakov, 1968									H	Pseudoleucite; Synnyr massif, U.S.S.R.
Papunen, 1967, p. 64								X, some opaque.		B; Finland.
Parsons and Soukup, 1961		X								Emerald; various gemstones.
Peach, 1949a									D	S, Kansas; Q, synthetic.
Peach, 1949b									D	Synthetic halite.
Peach, 1951									D	Peg minerals.
Pelletier, 1956									D	F; Pb-Zn OD.
Penn, 1951	X	(?)		(?)						Emerald.
Perhac and Heinrich, 1962									U	Rare earth vein OD.
Perna, 1968									H, D	Various OD.
Petersil'e, 1960					X	(?)				Alkalic ign.
Phillips, J., 1868									H	Q; Au veins; California.
Phillips, J., 1875									H	Various minerals; Sn OD.
Phillips, W., 1823, p. 4, 171							X			Q; Pb-Zn OD.
Pinckney, D. M., in U.S. Geological Survey, 1962, p. A4–A5 [1].									H	Q, etc; Butte, Mont., and Mississippi Valley-type OD.
Pinckney, 1965									H	Q; veins in Boulder batholith, Montana.
Pinckney, 1966									H	Cave-in-Rock CaF_2-zinc OD; Illinois.
Piotrovskiy, 1953									U	B (synthetic).
Piznyur, 1957	X								H	Q; meta veins.
Piznyur, 1959									D	Q; meta veins.
Piznyur, 1960									H	Q; meta veins.
Piznyur, 1968		X							H	Q; Zhireken Cu-Mo OD U.S.S.R.
Podgornova, V. N., 1966									H	Q; Nagol'nyy ridge, U.S.S.R.
Podgornova, V. P., 1958								X	H	F; veins in Sn-skarn.
Polykovskii, 1962a		X							H	Q; peg in skarn.
Polykovskii, 1962b									H	Q; peg in skarn.
Polykovskii, 1963									H	Q; peg; western Tien Shan.
Polykovskii, 1964									H(?)	Q; Q-feldspar veins; U.S.S.R.
Polykovskii, 1965									H	Q; pegs; western Tien Shan.
Polykovskii, Grushkin, and Podovil'skii, 1960.	X	X							H	Q; pegs in skarn.
Polykovksii and Malikov, 1960.									H	C; sed.
Polykovskii, Merkulov and Bozhko, 1968.									D	Halo in country rock near Q veins; Pamir.

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[1] Data by Pinckney on p. A4 here are incorrectly attributed to "Roedder and Pinckney."

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Polykovskii, Neklyudov, and Idrisova, 1966.									H	F; Bazardarin, U.S.S.R.
Polykovskii and Roizenman, 1965.									H, D	Q; pegs: Maidantal.
Polykovskii and Roizenman, 1966.	X								H	Q; pegs; Maidantal.
Polykovskii, Roizenman, and others, 1963.	X								H	Q; peg.
Pomîrleanu, 1957									H	Q; Herja OD, Romania.
Pomîrleanu, 1959									H	General.
Pomîrleanu, 1965									H	Diopside; Muntii Rodnei.
Pomîrleanu, 1968a									H	Diopside; Romanian Carpathians.
Pomîrleanu, 1968b	X								H	Scheelite; various OD.
Pomîrleanu, Apostoloiu, and Maieru, 1965.									H, D	Kyanite; meta.
Pomîrleanu and Barbu, 1964.									D	OD; Valea Ilba basin.
Pomîrleanu, Barbu, and others, 1967.									H	Spodumene; peg.
Pomîrleanu and Movileanu, 1966.									D	S; Capnic OD; Romania.
Pomîrleanu and Movileanu, 1967.									H	S; various Romanian OD
Pomîrleanu and Movileanu, 1968a.				X					H	Muscovite; various pegs.
Pomîrleanu and Movileanu, 1968b.									H	S, scheelite; various Romanian OD.
Pomîrleanu, Movileanu, and others, 1968.									HD	S; Capnic, Romania.
Pomîrleanu and Petreus, 1968a.									H	F; Capnic and Herja, Romania.
Pomîrleanu and Petreus, 1968b.									HD	C, F; Capnic, Romania.
Popov, 1963									U	Pb veins OD.
Poty, 1968a									H	Q; vein; Dauphine.
Poty, 1968b	X								H, C	Q; La Gardette and Mont Blanc.
Pough, 1965								Phenacite		Synthetic emerald.
Prikazchikov, 1966									H	Q; pegs; Volynia.
Prikazchikov and others,								X, kaolin(?)	H	Q; peg.
Prinz, 1882	X							X		Various gems.
Prokhorov, 1965									D	Pyrite; various sources.
Prokhorov and Khayretdinov, 1965.									D	Pyrite.
Prokhorov, Miroshniko, and Khayretdinov 1968.									D	Q; various Au OD.
Puchner and Holland, 1966.									H	Q; Noche Buena, Zacatecas, Mexico.
Pulou and de Croizant, 1965.									D	B and F.
Pushkina and Yakovleva, 1957.		X							H	Q; meta veins.
Puzanov, 1958									H	Q; W-Mo-Sn skarn OD.
Puzanov, 1960									H	Q; veins in skarn.
Puzanov and Kudakova, 1964.									D	Q veins, quartzite, granites.
Puzanov and Kudakova, 1966a.									D	Q, quartzites and granites; Aldan, U.S.S.R.
Puzanov and Kudakova, 1966b.									D	Q; various crystal OD; Aldan, U.S.S.R.
Rakhmanov, 1963									U	Various skarn minerals.
Rakhmanov, 1965									H	Scheelite, C, granite; Maikhura skarn OD, U.S.S.R.
Rakhmanov, 1968	X								H, C	Q; peg; southern Gissar.
Rakhmanov and Ryabov, 1968.									D	Halo in country rocks near F OD; U.S.S.R.
Ramenskaya, 1960						X				C (Iceland spar).
Rasumny, 1960, 1965	X									Various minerals.
Raumer, 1967									H	Q; Alpine veins.
Reese, 1898					X	X				Q; sed.
Renard, 1876		X							H	Q; Quenast diorite; Belgium.
Renard, 1877, p. 226	X	X							H	Q, from Quenast diorite; Belgium, and CO_2 from S; Santander, Spain.
Renard, 1889, p. 64		(?)						Glass		Q; Ascension Island granitic blocks.
Richter and Ingerson, 1954.									H	Synthetic Q.
Rodzyanko, 1967									H, D	Tourmaline, axinite, and datolite.
Rodzyanko and Trufanov, 1964.									D	Datolite, C, G, S, etc.; sulfide OD; U.S.S.R.
Roedder, 1958, p. 265									D	Q; Au vein; California.
Roedder, 1960a									HC	S; Pb-Zn OD; Creede, Colo.
Roedder, 1960b								X (ore minerals).		S, F; various OD.
										Colo.
Roedder, 1962b	X	X			X	X	X	X	HC	Various (a review).
Roedder, Edwin, in U.S. Geological Survey, 1962, p. A4.	X								HC	Q; Red Devil mercury OD, Alaska.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Roedder, Edwin, in U.S. Geological Survey, 1962, p. A89.	--------	--------	--------	--------	--------	--------	--------	--------------	C	Halite; Goderich, Ontario.
Roedder, 1963a	X	--------	--------	--------	--------	--------	--------	X, glass, spinel	HC	Olivine nodules; ign.
Roedder, 1963b	X	X	X	X	X	X	X	X	HC	Various minerals and OD.
Roedder, 1963c	--------	--------	--------	--------	--------	--------	--------	--------------	C	Various minerals and OD.
Roedder, Edwin, in U.S. Geological Survey, 1963, p. A178.	--------	X	--------	--------	--------	--------	--------	--------------	C	Q; Bingham, Utah.
Roedder, 1965a	X	X	X	X	X	X	--------	X	HC	Various OD.
Roedder, 1965d	X	--------	--------	--------	--------	--------	--------	X, glass, spinel	HC	Olivine nodules; ign.
Roedder, Edwin, in U.S. Geological Survey, 1965, p. A156.	--------	--------	--------	--------	--------	--------	--------	H_2S (liquid)	C	C; Pb-Zn OD; Lark, Utah.
Roedder, 1966b	--------	--------	--------	X	X	X	X	H_2S	HC	Various Mississippi Valley-type OD.
Roedder, 1966c	--------	--------	--------	--------	--------	--------	--------	--------------	HD	Various; a review.
Roedder, 1967a	--------	--------	--------	--------	--------	--------	--------	--------------	HD	Various OD (a review).
Roedder, 1967b	--------	--------	--------	--------	--------	--------	--------	--------------	C	F, Q; various OD.
Roedder, 1967c	--------	--------	--------	--------	--------	--------	--------	--------------	HC	S, C; Pine Point OD, Canada.
Roedder, 1967d	--------	--------	--------	--------	X	X	X	--------------	HC	S, F; various Mississippi Valley-type OD.
Roeder, Edwin, in U.S. Geological Survey, 1967, p. A159 (see also Roedder, 1967d).	--------	--------	--------	--------	X	X	X	--------------	HC	Various OD.
Roedder, Edwin, in U.S. Geological Survey, 1967, p. A160.	--------	--------	--------	--------	--------	--------	--------	--------------	HC	Various saline minerals; Chile.
Roedder, Edwin, in U.S. Geological Survey, 1967, p. A160.	--------	--------	--------	--------	--------	--------	--------	--------------	HC	Fluorite; Colorado.
Roedder, 1968b, p. 461	--------	--------	--------	--------	--------	X	--------	--------------	----------	S, Silesia.
Roedder, 1968c	--------	--------	--------	--------	--------	--------	--------	--------------	HC	S, F, B, Q, C; Pb OD; Laisvall, Sweden.
Roedder, 1968d	--------	--------	--------	--------	--------	--------	--------	--------------	HC	S, C; Pine Point OD, Canada.
Roedder, 1969a	--------	--------	--------	--------	--------	--------	--------	--------------	HC	Celestite, F; Clay Center, Ohio.
Roedder 1969b	--------	X	--------	X	X	X	X	X, H_2S(?)	HC	S, F, B, Q, dolomite; southern Appalachian Valley OD.
Roedder, 1970a	X	--------	--------	--------	X	X	--------	--------------	----------	Various.
Roedder, 1970b	X	X	X	X	--------	--------	--------	X, glass	H, C, D	Q, feldspar; ign; Ascension Island.
Roedder, 1971	X	X	X	X	--------	--------	--------	X, H_2S, hematite, anhydrite(?).	HC	Various; Bingham Cu OD, Utah, and other porphyry Cu OD.
Roedder and Coombs, 1965.	X	X	--------	X	--------	--------	--------	X, glass	HC	Q, feldspar; ign; Ascension Island.
Roedder and Coombs, 1967.	X	X	X	X	--------	--------	--------	X	HCD	Q, feldspar; ign; Ascension Island.
Roedder and Creel, 1966	(?)	X	X	--------	--------	--------	--------	X, H_2S, hematite, anhydrite(?).	CH	Various; Bingham Cu OD, Utah.
Roedder, Edwin, and Heyl, A. V., in U.S. Geological Survey, 1965, p. A155 (see also next entry).	--------	--------	--------	X	--------	X	X	--------------	HC	F, B, Q; Pb-Zn-CaF_2 OD; Hansonburg, N. M.
Roedder, Heyl and Creel, 1967, 1968.	--------	--------	--------	--------	--------	--------	--------	X, barite(?)	HC	F, B, Q; Pb-Zn-CaF_2 OD; Hansonburg, N. M.
Roedder, Ingram, and Hall, 1963.	--------	--------	--------	X	--------	--------	--------	X	----------	Various minerals and OD.
Roedder, Edwin and Skinner, B. J., in U.S. Geological Survey, 1967, p. A159–A160 (see also Skinner and others, 1967).	--------	--------	--------	--------	--------	--------	--------	--------------	HC	S; Salton Sea geothermal area.
Roehl, 1968	--------	--------	--------	--------	X	X	X	--------------	----------	Limestone; Mexico.
Rogers and Sperisen, 1942	--------	X	--------	--------	--------	--------	--------	--------------	----------	Emerald; Colombia.
Roizenman, 1965	X	--------	--------	--------	--------	--------	--------	--------------	----------	Various.
Rose, 1839	--------	--------	--------	--------	X	--------	--------	--------------	----------	Halite; sed.
Rouse, 1952	X	--------	--------	--------	--------	--------	--------	--------------	H	Sapphire; Ceylon.
Ruchkin and Nikolaichuk, 1968.	--------	--------	--------	--------	--------	--------	--------	--------------	D	Various minerals; Blyava OD, Urals, U.S.S.R.
Rutherford, 1963	X	X	--------	(?)	--------	--------	--------	X, gypsum(?)	HC	Q; Flin Flon area, Saskatchewan.
Rutherford and Arnold, 1963.	X	X	--------	(?)	--------	--------	--------	--------------	HC	Q; Flin Flon area, Saskatchewan.
Ryabov, 1962	--------	--------	--------	--------	--------	--------	--------	--------------	U	Q; low temp. Mo-W OD.
Ryabov, 1966	--------	--------	--------	--------	--------	--------	--------	--------------	D	Cassiterite; various Far Eastern Sn OD; U.S.S.R.
Ryabov, 1968	--------	--------	--------	--------	--------	--------	--------	--------------	D	Various minerals; Sn OD; U.S.S.R.
Ryabov and Ruchkin, 1968.	--------	--------	--------	--------	--------	--------	--------	--------------	D	Various ore minerals; Southern Urals.
Rye, 1965	--------	X	--------	--------	--------	--------	--------	--------------	H	C, Q, S: Providencia OD, Mexico.
Safronov, 1957a	--------	--------	--------	--------	--------	--------	--------	X	----------	Synthetic quartz.
Safronov, 1957b	--------	X	--------	--------	--------	--------	--------	--------------	----------	Q; Pamir, U.S.S.R.
Safronov, 1958	--------	--------	--------	--------	--------	X	--------	--------------	----------	Synthetic quartz.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Safronov and Khadzhi, 1957.									H	Synthetic quartz.
Saha, 1959									D	Paragneisses; Ontario.
Sainsbury, 1968	X									F; beryllium OD; Alaska.
Sarcia and others, 1965									D	S, Q; Pb-Zn OD.
Savul and Pomîrleanu, 1957a.									H(?)	Q; Niculitel OD.
Savul and Pomîrleanu, 1957b.									H	Q; secondary; Dobrogea du Nord.
Savul and Pomîrleanu, 1958.									H	Q; various OD.
Savul and Pomîrleanu, 1960.									H	Q; schists.
Savul and Pomîrleanu, 1961a.									D	Q; polymetallic OD.
Savul and Pomîrleanu, 1961b.									H	Q; Felsobanya OD.
Savul and Pomîrleanu, 1961c.									D	Q; Baia Sprie OD.
Savul and Pomîrleanu, 1963a.									H(?)	Q; Fundul Moldovei OD
Savul and Pomîrleanu, 1963b.									H	OD; Carpathians.
Savul and Pomîrleanu, 1965a.									H	S, Q, diopside; Eastern Carpathian OD.
Savul and Pomîrleanu, 1965b.									H	Q; various OD.
Sawkins, 1963		X	X	(?)				X	HC	S, Q, C, F; Pb-Zn OD.
Sawkins, 1964		X						X	HC	S, Q, C, F; Pb-Zn OD.
Sawkins, 1965a		X						X	HC	S, Q, C, F; Pb-Zn OD.
Sawkins, 1966a									HC	F, Q, B, C: North Pennine OD.
Sawkins, 1966b		X	X					X (sulfate?)	HC	Q, F, S; OD; Cornwall, England.
Sawkins and others, 1964									H	Q, F; North Pennine orefield.
Sawkins and Holland, 1965.									HC	F, B; North Pennine orefield.
Sawkins and Huebner, 1963.		X						X	HC	S, Q, C, F; Pb-Zn OD.
Sayar, 1953								Magnetite(?)		Q; dolomitic marble; Turkey.
Sazonov, 1957									D	F; Pb-CaF_2 OD.
Sazonov, 1961									H	S; various OD.
Sazonov, 1962									H, D	Garnet and ore minerals; Kurusaisk orefield, U.S.S.R.
Schlossmacher, 1932, p. 348–451.	X									Diamond.
Schlossmacher, 1955		X								Various gemstones.
Schmidt, A., 1881					X	(?)				S; Wiesloch Zn OD.
Schmidt, R. A., 1962									H	S, C; Pb-Zn OD.
Schröder, 1925, p. 270	X									Miarolitic cavities in granite.
Scott, 1948									D	Q and various minerals.
Seeliger, 1950									H	S, etc; Pb-Zn OD; Ruhr.
Senchilo and Komarov, 1962.									D	Q; Mo OD.
Shamrai and Trufanov, 1968.	(?)								H	Various minerals; Hg OD; northern Caucasus.
Shaposhikov and Ermakov, 1968.									H	Q, Synthetic.
Sharkov, 1958	X	X	X	(?)				X		Q; meta veins.
Shcherba, 1960									D	Various Kazakhstan OD.
Shcherba and others, 1964									D(H?)	Q, etc.; Mo-W OD.
Shchiritsya, 1960									H	Q; veins in meta.
Sheshulin, 1961	X	(?)			X				D, H	Spodumene; peg.
Shestakov and Prokhorov, 1965.									D	Magnetite; Eastern Sayan Mountains.
Shiobara, 1961									D	Various; Kamioka mine, Japan.
Shugurova, 1967a, 1967, b	(?)							X	H	Q, F; peg.
Sidorenko, 1951									H	B, Q; Transcarpathia.
Sidorenko, 1958					X	X				Authigenic Q; brown coal deposit.
Silliman, 1880	X									Q; with tourmaline in Au OD.
Sims and Barton, 1961									H	S; Central City district, Colorado.
Sims and Barton, 1962, p. 394.								X (0.8 volume percent, opaque).	H	S; polymetallic Zn OD.
Singewald, 1932, p. 58	X	X						Glass(?)		Q; granodiorite; Buckskin Gulch, Colo. (personal observation, E. Roedder, 1964).
Sinyakov, 1967									H	Monticellite; from skarn magnetite OD.
Sinyakov, 1968									H	Monticellite; skarn.
Sivoronov, 1968									H	Taconite and jaspilite iron OD; U.S.S.R.
Sjögren, 1905							X			Quartz.
Skinner, 1953									H	Q; Brazil.
Skinner and Appleman, 1963.						(?)	(?)	H_2S(?)		Melanophlogite; Sicily.
Skinner and others, 1967, p. 325.									HC	S; Salton Sea geothermal area.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Skropyshev, 1957									H	C (Iceland spar).
Skropyshev, 1960						X				C (Iceland spar).
Slivko, 1952									H	Tourmaline; peg.
Slivko, 1955, p. 66–74		X	X					X, fluoborates, fluorides.	H	Tourmaline, Q; peg.
Slivko, 1958	X							X, alkali fluoborates.		Tourmaline; peg.
Slivko, 1966								X	H	Tourmaline; peg.
Slivko, 1969	X								H	Tourmaline; Korets peg; U.S.S.R.
Smirnov, 1967									H	Stratiform Pb-Zn OD; U.S.S.R.
Smith, 1948									D	Q, C, pyrite; McIntyre OD, Ontario.
Smith, 1949									D	Q, cassiterite, tourmaline; Sn OD.
Smith, 1950									D	Q vein; Au OD.
Smith, 1951									D	Q vein; Au OD.
Smith, 1952a									D	Garnet, skarn OD and meta.
Smith, 1952b									D	S; Mississippi Valley-type, and others.
Smith, 1953b								X, silicates		Peg minerals.
Smith, 1953c									D	Meta, high-grade.
Smith, 1954a									D	Q; Au veins.
Smith, 1954b	X	X								Various.
Smith, 1957									D	Ign, various.
Smith and Little, 1953									DH	General.
Smith and Little, 1959	X								H	General.
Smith and others, 1950									DH	Various minerals.
Sobolev and Bakumenko, 1961.									H	Albite peg.
Sobolev and Bazarova, 1963.								X	H	Kyanite; peg.
Sobolev and others, 1964									H	Q, T, etc; peg and granite.
Sobolev and others, 1967	X								H	Various; nepheline basalt; Hungary.
Sobolev and others, 1970		X	X		X			X	H, C	Various minerals; Ilímaussaq alkalic intrusion, Greenland.
Sokolov, 1960									DH	Q vein.
Sokolov and Zakharchenko, 1961.									H	Q; peg.
Sorby, 1858		X						X	HC	Various minerals and rocks.
Sorby, 1869	X	X							H	Various gems.
Sorby, 1877	X								H	Granite.
Sorby and Butler, 1869	X	X				X		X		Various gems.
Spencer, 1917, p. 100–101		(?)	(?)							Q; ign and porphyry Cu OD.
Spezia, 1904	X								C	Anhydrite.
Spezia, 1907	X								HC	Calcite; Traversella, Italy.
Spotts and Silverman, 1966.					X	X				Dolomite crystals in tar; California.
Stalder, 1964a	X								HD	Q, F, ankerite, adularia; Alpine.
Stalder, 1964b	X								H	Q; Taminser Calanda.
Stalder, 1967	X	X						Sulfur		Alpine vein minerals.
Stephenson, 1952									D, H	Various minerals; OD.
Steven, 1960, p. 410									H	F; Northgate district, Colorado.
Steward, 1967								Glass (hydrous melt).		Synthetic quartz.
Stewart, D. B., and Roedder, Edwin, in U.S. Geological Survey, 1963, p. A145.								Glass (hydrous melt).		Synthetic quartz.
Steyn, 1962									D	Various; Sn-bearing pipes.
Stoiber and Davidson, 1959.									H, D	Q, C; OD; Michigan copper district.
Stronskaya, 1955									H	Q; tourmaline-muscovite peg.
Sukhorskaya and Sukhorskiy, 1967.						X	X		H	Q; veins in quartzite; Aldan, U.S.S.R.
Sukhorskiy, 1953a	X								H	Q.
Sukhorskiy, 1953b									H	C.
Sukhorskiy, 1954									H(?)	Q; meta veins.
Sukhorskiy, 1965a	X								H	Q; veins in quartzites; Aldan, U.S.S.R.
Sukhorskiy, 1965b									H	Q; pegs and veins.
Sushchevskaya and others, 1966.									D	Q; various Myao-Chan Sn-W deposits, U.S.S.R.
Sushchevskaya and Ivanova, 1967.	X								HD	Q, wolframite; OD; eastern Transbaikal.
Sutton, 1964		X			X	X	X	X		Various.
Taber, 1950								Clay	HC	Q; meta(?).
Takahashi and others, 1955.									D	Various minerals, OD.
Takenouchi, 1962a									HD	Q, S; various OD.
Takenouchi, 1962b		X		(?)				X	H	Q; OD.
Tammann and Seidel, 1932.			X		X			$CaSO_4$, $MgCl_2 \cdot 6H_2O$ (solid inclusions?).	D	Halite.
Tender, 1967									H	C; Hg OD; U.S.S.R.
T'eng and others, 1965									HD	Q; quartz crystal deposits.
Thompson, 1953	X								D(?)	S, Q; Pend d'Oreille OD, Idaho.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Touray, 1965		X						X	H	Gypsum; Sahara.
Touray, 1967		X	X		X			X, pyrite		Various minerals.
Touray, 1968	X								H, D	Q; various localities.
Touray and Deicha, 1967	X	X				X				F; various.
Touray and Jauzein, 1967					X	X			HC	Q; geodes from sed.
Touray, Lantelme and Vogler, 1966.									D	Q, B, halite.
Touray and Poirot, 1968	X	X						X	HC	Emerald; Colombia.
Touray and Sabouraud-Rosset, 1970.		X						Sulfides(?)	HC	F; Ouezzane, Morocco.
Touray and Yajima, 1966	X								HC	Q; Alpine.
Touray and Yajima, 1967		X				X	X	X(barite?)	HD	F; Tunisia.
Traveria-Cros and Montoriol-Pous, 1967.									D	F, G; F OD; Spain.
Trdlicka and Coufal, 1966									D	C; Pribram, Czechoslovakia.
Trdlicka and Kupka, 1964									D	Metasomatic magnesite OD.
Trdlicka and Kupka, 1966									D	Siderite; Goemer OD, Czechoslovakia.
Trufanov, 1962		X	X					X	H	Q; peg.
Trufanov, 1966		X	X	X					HD	Q, T, apatite; pegs; central Kazakhstan.
Trufanov, 1967	X	X	X					S, fluorite, apatite.	HD	Q, F, apatite, tourmaline; peg; Kazakhstan.
Tschermak, 1903							X			Quartz.
Tsusue, 1967									H	Q, rhodochrosite; Mn OD; Japan.
Tugarinov and Naumov, 1969.	X							X	H, D	Q and many others; hyd U OD; U.S.S.R.
Tugarinov and others, 1963.									D	Q; dolomite, calcite; Krivoi Rog, U.S.S.R.
Turba, 1962									D	B ores, in processing.
Turovskii and others, 1966.									D	OD containing colloidal malacon.
Twenhofel, 1947									H	F.
Uchameyshvili, 1965, p. 149–150.									H	B; various OD.
Uchameyshvili and others, 1966.									HD	B; various OD.
Ushakovskii, 1966									D	Q; Southern Urals.
Usol'tsev, 1966									D	S; Barvinski OD, U.S.S.R.
Usol'tsev and Troshin, 1967.									D	S; eastern Transbaikal, U.S.S.R.
Vakhromeev, 1968									D	F, S; Mississippi Valley type OD; Siberia.
Val'ter and Gurova, 1966									H	F; cementing sandstone.
Val'ter and Gurova, 1968									H	F; Dniester River area, U.S.S.R.
Valyashko and Kogarko, 1966.								X (glass)	HD	Apatite; Kola Peninsula, U.S.S.R.
Valyashko and others, 1968.								Many	H	Apatite; Khibini OD, U.S.S.R.
Van Alstine and others, 1962.					X			H_2S (odor)		F, various Mexican OD.
Varček, 1965									H	S; various OD.
Vartanova and Shcherbakova, 1964.									H	Q; in granites.
Velchev and Mel'nikov, 1965.									CH	F.
Venediktov, 1958a									HD	Q; meta veins.
Venediktov, 1958b									U	Q; meta veins.
Verbichev, 1959								X	H	Q, C, F; garnet skarn.
Vertushkov, 1966	X								H	Q; various OD.
Vian, and others, 1964									D	Pyrite ores, in processing.
Vlasenko, 1957	X									Nepheline; Il'men Mtns., U.S.S.R.
Vogelsang, 1869	X	(?)							CH	Various ign and meta.
Vogelsang and Geissler, 1869.	X								H	Q and other minerals.
Vovk and Voznyak, 1967	X				X					Albite; pegs; Mamsk.
Vujonovic, 1963									D	Q veins.
Vul'chin, 1951									H	General.
Wahler, 1956	X	X				(?)				Q, T, beryl.
Walker, 1935		(?)						X		Q.
Wardlaw and Hartzell, 1963.									H	Halite.
Weber, 1908								H_2S		S; Santander, Spain.
Webster, 1952		X								Synthetic and Australian emeralds.
Webster, 1953	X								H	Chrysoberyl.
Webster, 1954	X								H	Beryl; Madagascar.
Webster, 1955		X	(?)					Pyrite (daughter?).		Emerald.
Webster, 1956	X							Fluorite(?) cubes.		T; Nigeria.
Webster, 1962	X	X								Beryl; Madagascar.
Weis, 1953	X								CH	Zoned peg minerals.
Weller and others, 1952					X	X			H	F; Zn-CaF_2 OD.
Wells, 1953		(?)								Synthetic emerald.
Westervelt, 1960									H, D	Q, S, Knebelite; Bluebell mine, Canada.
Wetzel, 1959						X	X			Dolomite; Zechstein salt deposits.
Wichmann, 1880	X	X								Granite and schist; Michigan iron ranges.

TABLE 1.—*Summary of qualitative phase identification and thermometry of fluid inclusions*—Continued

Reference	Phase identification								Thermometry	Sample notes
	Liquid CO_2	Solid NaCl	Solid KCl	Solid carbonates	Organic			Other phases		
					Gas	Liquid	Solid			
Williams, A. F., 1932, p. 422.	X									Diamond.
Williams, H., 1932								Fluorite		Phenocrysts from vocanics; Lassen Peak, Calif.
Wimmenauer, 1963		X	X	X						Apatite from carbonatite; Kaiserstuhl, West Germany.
Wimmenauer, 1966, p. 197		X	X	X						Apatite from carbonatite; Kaiserstuhl, West Germany.
Winchell, 1910	(?)	X								Q; OD vein.
Wright, A. W., 1881	(?)				(?)			H_2S, SO_2(?)		Q; Branchville, Conn.
Wright, C. E., 1880, p. 249	X									Granite gneiss; Penokie Iron Range.
Yajima and Touray, 1967		X							H	F; Morocco.
Yajima, Touray, and Iiyama, 1967.		X						X	H	Albite; Modane.
Yakovlev and Lebedev, 1968.									H	Various minerals; Deputat Sn OD, U.S.S.R.
Yakubova, 1952	X							X, borax	H	T, Q; pegs; Murzinka, Urals, U.S.S.R.
Yakubova, 1955	X					X		X, borax	H	Various minerals; peg.
Yakubovich, 1964	X								H	F; OD; East Azov region, U.S.S.R.
Yasinskaya, 1967	X								H, C	Several chondritic and achondritic meteorites.
Ypma, 1963	X	X							H, D	Q, etc; Pb-Zn OD.
Yudin, 1968									D	Various minerals; Kounrad OD, U.S.S.R.
Yushkin and Srebrodol'skii, 1965.								H_2S, X		Sulfur; Shorsui OD, U.S.S.R.
Zabarina, 1965									D	S, axinite, arsenopyrite; OD.
Zabarina and Kuts, 1964									D	Various; Far Eastern Sn OD, U.S.S.R.
Zakharchenko, 1950	X	X	X	X				X, hematite, H_2S.	H	Q; meta veins and peg.
Zakharchenko, 1955	X	X	X	X				X, hematite, albite, H_2S.	H	Q; meta veins.
Zakharchenko, 1961									H, D	Q; meta veins.
Zakharchenko, 1964	X	X	X					X, glass (on heating).	H	Zoned peg.
Zakharchenko, 1968									H	Q; granites and peg; U.S.S.R.
Zakharchenko and others, 1966.		X	X					X, hematite	H	Q, F; various Kazakhstan veins and peg.
Zakharchenko and Trufanov, 1964.									H	Q; giant crystal peg; Akzhailyau, U.S.S.R.
Zakrzhevskaya, 1964					X				H	Ign phosphate OD.
Zakrzhevskaya, 1968					X	X	X		H	Nepheline; Khibiny apatite OD, U.S.S.R.
Zakrzhevskaya and Elinson, 1966.					X				U	Nepheline; Khibiny apatite OD, U.S.S.R.
Zaritskii and others, 1966	X								U	F; Donets Basin-Azov area, U.S.S.R.
Zatsikha and others, 1967									H	Various minerals; Donbass-Azov area, U.S.S.R.
Zirkel, 1868		X								Q; trachite.
Zirkel, 1870a, p. 21, 33, 60	X									Basalt minerals.
Zirkel, 1870b		X							H	Many ign minerals.
Zirkel, 1873	X	X						X		Various rocks.
Zirkel, 1876	X	X	(?)					X, hematite		Various ign and meta.
Zirkel, 1893	X	X								Various ign and meta.
Zolotukhin, 1962		X								Anhydrite; Noril'sk OD, U.S.S.R.
Zolotukhin, 1965, p. 133-134.									H	Anhydrite; Noril'sk OD, U.S.S.R.
Zolotukhin and others, 1966.									D	Pyrrhotite, chalcopyrite; trap intrusions; U.S.S.R.
Zwaan, 1958	X									Aquamarine.

TABLE 2.—*Analyses of gases from fluid inclusions in rocks and minerals*

[N.d., not detected; ----, not reported; tr., trace]

Analysis No.	Reference	Material	Volume percent of evolved gases									Volume (ml per 100-gram sample)	Sample notes
			H_2O	CO_2	CO	O_2	H_2	N_2[1]	CH_4	C_2H_6	H_2S		
						GASES HIGH IN "ORGANIC" CONSTITUENTS							
						Sedimentary rocks							
1	Nesmelova, 1959.	Carnallite	-------	[2] 22.0	-------	-------	32.6	38.3	[2] 7.0	-------	-------	---------	Bedded potash salts; Solikamsk and Bereznikovsk mines, U.S.S.R.[3]
2	do	Sylvite	-------	14.5	-------	-------	4.6	78.5	1.9	-------	-------	---------	Do. Sample B.[4]
3	Hoy and others, 1962.	Halite	17.3	46.9	4.8	4.4	1.8	18.4	1.5	-------	-------	---------	Inclusions under pressure; Winnfield salt dome, Winn Parish, La.[5]
4	Ackermann and others, 1964.	Halite-sylvite.	-------	84.0	<.05	.5	.4	14.0	1.0	<0.01	<0.1	3.9	Folded sediments; Werra potash deposits, East Germany.[6]
4a	Murray, 1957.[7]	Quartz	.00	7.53	-------	-------	-------	1.60	64.9	10.7	-------	---------	Vug in dolomite from Mississippian Rundle formation, Alberta, Canada.
						Igneous rocks							
5	Petersil'e, 1959.[8]	Khibinite	-------	25.20	0.89	-------	3.81	(9)	68.94	0.38	-------	[10] 6.746	Rocks from nephelin syenite intrusive complex, Khibina massif, Kola Peninsula, U.S.S.R.[11]
6	do	Apatite-nepheline rock.	-------	59.63	5.57	-------	3.80	(12)	29.06	.88	-------	[10] 1.132	Do.[13]
7	do	do	-------	91.52	6.98	-------	.67	(12)	.45	.38	-------	[10] 1.333	Do.[14]
8	do	Roscherite	-------	66.21	2.22	-------	5.38	(12)	25.58	.19	-------	[10] 2.119	Do.
9	do	Foyaite	-------	-------	10.44	-------	2.79	(12)	86.36	.24	-------	[10] 2.0785	Do.
10	do	do	-------	33.84	1.36	-------	9.51	(15)	52.87	.80	-------	[10] .8096	Rocks from nepheline syenite intrusive complex, Lovozero massif, Kola Peninsula, U.S.S.R.
11	do	Villiaumite-foyaite.	-------	0	0	-------	30.17	(15)	62.28	3.77	-------	[10] .0928	Do.
12	do	Peridotite	-------	88.25	6.24	-------	5.05	(15)	.45	0	-------	[10] 2.869	Ultrabasic intrusive complex, Monchegory massif, Kola Peninsula, U.S.S.R.
13	do	Iolite-urtite	-------	41.6	1.46	-------	3.62	(12)	52.0	.67	-------	[10] 4.95	Paleozoic intrusive complex, Khibina massif, Kola Peninsula, U.S.S.R.
14	Petersil'e, 1963.	do	-------	.19	4.13	-------	3.45	-------	89.70	2.06	-------	3.390	Paleozoic intrusive complex (author's average analysis), Khibina massif, Kola Peninsula, U.S.S.R.[16]
15	do	Ultrabasic rocks.	-------	.60	64.90	-------	22.60	-------	11.90	.00	-------	.168	Paleozoic intrusive complex (author's average analysis), Kovdorsk massif (main) Kola peninsula, U.S.S.R.[17]
16	do	Potassium feldspar.	-------	0.007	3.05	-------	2.22	-------	92.97	1.46	-------	3.02	Mineral from nepheline syenite intrusive complex, Khibina massif, Kola Peninsula, U.S.S.R.[18] [19]
17	do	Nepheline	-------	.11	1.76	-------	1.81	-------	92.74	3.04	-------	5.29	Mineral from iolite-urtites and khibinites; Khibina massif, Kola Peninsula, U.S.S.R.[19] [20]
18	Ikorskii, 1962.	Roscherite	-------	.00	1.31	-------	.35	-------	97.45	.85	-------	24.4	Rocks from nepheline syenite intrusive complex, Khibina massif, Kola Peninsula, U.S.S.R.[21]
18a	Chamberlin, 1908.	Nepheline syenite.	-------	42.42	8.76	-------	36.33	7.00	5.49	-------	-------	.27	Alkalic intrusive complex, Methuen Township, Ontario, Canada.[22]
19	Vasil'ev and others, 1961.	Kimberlite	-------	.0	-------	1.9	50.90	12.8	30.4	[23] 3.70	-------	---------	Gas from drill hole; Udachnaya pipe, Siberian diamond field, Yakutia, U.S.S.R.[24]
20	Zavaritskii and Betekhtin, 1937.	Dunite	-------	-------	-------	3.8	66.5	[25] 20.7	9.5	-------	-------	---------	Nizhyn Tigil dunite massif, northern Urals, U.S.S.R.

TABLE 2.—*Analyses of gases from fluid inclusions in rocks and minerals*—Continued

Analysis No.	Reference	Material	Volume percent of evolved gases									Volume (ml per 100-gram sample)	Sample notes
			H_2O	CO_2	CO	O_2	H_2	N_2[1]	CH_4	C_2H_6	H_2S		
						GASES HIGH IN "ORGANIC" CONSTITUENTS—Continued							
						Igneous rocks—Continued							
21	Elinson and Polykovskii, 1961b.[26]	Feldspar	-------	27.5	-------	-------	4.60	64.60	0.00	-------	-------	8.29	Quartz crystal pegmatites from leucocratic porphyritic granites in Paleozoic sediments; western Tien Shan, U.S.S.R. Sample 250.
22	do	Honeycomb quartz.	-------	20.6	-------	-------	40.84	37.66	.00	-------	-------	62.30	Same pegmatites as analysis 21. Sample 109a.
23	do	Smoky quartz.	-------	10.2	-------	-------	3.70	74.40	10.75	-------	-------	16.30	Same pegmatites as analysis 21. Sample 109b.
23a	Barker, 1965a.[27]	Orthoclase	67.21	18.11	4.37	-------	9.72	.29	.29	-------	-------	131.7	From granite; Shap Fell, England. Sample 41.
23b	do	do	82.32	14.77	.67	-------	1.93	.09	.18	-------	-------	105.8	Granite, Shap Fell, England. Sample SN9.
23c	do	Biotite	67.80	4.82	.12	-------	26.91	.11	.22	-------	-------	1,940	Granite, Shap Fell, England. Sample NS15.
23d	Finko, 1964	Pumice	-------	[28] 25.75	.0	.0	36.35	37.90	.0	-------	-------	1.84	Young unaltered pumice (closed pores); Pacific region, U.S.S.R. Sample 2.[29]
						GASES LOW IN "ORGANIC" CONSTITUENTS							
						Igneous rocks							
24	Goguel, 1963.[30]	Quartz	94.41	2.02	0.29	-------	2.97	0.27	(0.04)	-------	0.00	148.3	Youngest Variscan granite; Schluchsee (Schwarzwald), West Germany.
25	do	Orthoclase	97.98	1.12	.04	-------	.71	.14	.00	-------	.00	285.8	Do.
26	do	Plagioclase	97.03	1.35	.08	-------	1.25	.28	(0.02)	-------	.00	288.6	Do.
27	do	Biotite	98.40	.34	.00	-------	1.24	.03	.00	-------	.00	437.0	Do.
28	do	Quartz	97.40	.98	.08	-------	1.32	.18	(0.02)	-------	.01	193.0	Pegmatite associated with granite; Schluchsee (Schwarzwald), West Germany.
29	do	Orthoclase	99.70	.08	.02	-------	.13	.08	.00	-------	.00	611.9	Do.
30	do	Quartz	90.56	1.74	.35	-------	6.71	.64	.00	-------	.00	59.6	Youngest Variscan granite; Andlau, 1, Vosges, France.
31	do	do	98.23	.15	.06	-------	1.45	.07	.05	-------	.00	213.8	Pegmatite; Landsverk, Evje, Norway.
32	do	do	.00	27	18	-------	13	22	11	-------	9	.112	Optically clear quartz; locality unknown.
33	do	Olivine	99.01	.31	.23	-------	.31	.13	.00	-------	.00	38.4	Olivine nodule in Tertiary basalt; Hoher Hagen, West Germany.
						Pegmatites							
36	Elinson and Polykovskii, 1963.[31]	Granite	-------	25.3	N.d.?	-------	11.2	60.7	1.4	N.d.	-------	9.95	Vuggy quartz veins bordered by microcline in Upper Permian granite; Maidantal quartz-feldspar veins, U.S.S.R. Sample 125.[32]
37	do	Granite-porphyry.	-------	3.1	N.d.?	-------	6.3	84.7	3.1	N.d.	-------	15.95	Maidantal quartz-feldspar veins, U.S.S.R. Sample 128.
38	do	Greisen I	-------	5.2	N.d.?	-------	.0	93.7	.0	N.d.	-------	4.44	Maidantal quartz-feldspar veins, U.S.S.R. Sample 154.
39	do	Microcline zone.	-------	12.8	N.d.?	-------	.0	85.1	.0	N.d.	-------	3.76	Wallrock has been sericitized and chloritized ("greisen I"); Maidantal quartz-feldspar veins, U.S.S.R. Sample 129.
40	do	Citrine I (early).	-------	8.0	N.d.?	-------	1.6	88.8	.0	N.d.	-------	41.83	Do.[33]
41	do	Citrine II (late).	-------	4.5	N.d.?	-------	.9	93.5	.0	N.d	-------	9.85	Do.[34]

TABLE 2.—*Analyses of gases from fluid inclusions in rocks and minerals*—Continued

Analysis No.	Reference	Material	Volume percent of evolved gases									Volume (ml per 100-gram sample)	Sample notes
			H_2O	CO_2	CO	O_2	H_2	N_2[1]	CH_4	C_2H_6	H_2S		
			GASES LOW IN "ORGANIC" CONSTITUENTS—Continued										
			Pegmatites—Continued										
42	Sheshulin, 1961.[30]	Spodumene	[35] 98	0.26	0.00	-------	0.24	0.14	[36]0.29	-------	-------	545	Spodumene pegmatite in Proterozoic graphitized marbles; unspecified locality.
43	Wahler, 1956.	Beryl	74.4	[37] 24.3	.6	0.0	.3	-------	[38].4	-------	-------	11	Rio Guanhães, Brazil. Sample 16.
44	do	do	95.4	[37] 4.2	.06	.0	.03	-------	[38].3	-------	-------	77	Spitzkopje, South-West Africa. Sample 18.
45	Wright, 1881.	Quartz	69.02	30.48	-------	-------	-------	.50	-------	-------	Tr.	36.0	Pegmatite; Branchville, Conn.[39]
46	Damon and Kulp, 1958.[40]	Beryl	75.3	18.7	.67	.1	.50	1.51	.074	-------	.005	398	Pegmatite, 2,750 m.y. age; Beartooth, Mont. Sample B1.[41]
47	do	do	[42] 79.7	12.6	.37	.02	4.17	1.97	.69	-------	.014	376	White beryl from 1,630 m.y. pegmatite; Keystone, S. Dak. Sample B7.
48	do	do	73.8	19.7	.66	.11	3.42	2.32	.053	-------	.005	407	Aquamarine from 320 m.y. pegmatite; Beryl Mtn., N.H. Sample B11a.[41]
49	Damon, P.E., written commun., 1963.	do	64.4	17.1	(43)	-------	(43)	11.1	3.6	-------	.1	---------	Same pgematite as in analysis 47. Sample SS–D–3C.[44]
49a	Kovalishin, 1965.[45]	Quartz	-------	76.3	.0	.3	13.2	4.8	5.4	-------	-------	7.24	Milky white quartz from center of quartz zone; pegmatite; Volynia, U.S.S.R.
49b	do	do	-------	58.6	.0	6.1	6.1	27.2	2.0	-------	-------	5.80	Smoky quartz from healed fractures in quartz zone; pegmatite; Volynia, U.S.S.R.
49c	do	do	-------	73.28	.0	.95	15.07	4.22	6.49	-------	-------	4.00	Smoky quartz from zone of fracturing at "hole 1, depth 16M"; pegmatite; Volynia, U.S.S.R.
			Ore deposits										
50	Umova and others, 1957.[46]	Quartz	70.6	27.6	0.0	0.3	0.6	0.9	-------	-------	-------	51	Primary inclusions from scheelite vein; Berezovskoe deposit, U.S.S.R. Sample 16.
51	do	do	75.5	22.1	.0	1.0	1.5	.0	-------	-------	-------	49	Primary inclusions from sulfide vein; Berezovskoe deposit, U.S.S.R. Sample 14.[47]
52	do	do	66.7	13.3	.0	2.7	16.7	.0	-------	-------	-------	3.6	Many secondary inclusions; Khrustal Mtn., U.S.S.R. Sample 12.
53	do	do	(48)	14.0	[49] 2.0	5.0	38.0	41.0	-------	-------	-------	24	Possibly primary inclusions with pyrite ores; Belorechensky deposit, U.S.S.R. Sample 6.
54	do	do	75.0	1.5	.0	2.0	12.0	9.5	-------	-------	-------	16	Inclusions similar to those of analysis 53. Sample 11.
55	Chukhrov, 1960.[50]	do	-------	23.4	.0	-------	58.5	18.1	.0	-------	-------	.33	Molybdenum deposit; late banded quartz; Eastern Kounrad, U.S.S.R.
56	do	do	-------	12.2	.0	-------	13.2	.6	.0	-------	-------	.40	Molybdenum and tungsten deposits; Eastern and Northern Kounrad, U.S.S.R. *Minimum* of 30 analyses.
57	do	do	-------	84.4	.0	-------	84.2	14.6	.0	-------	-------	4.69	Same samples as in analysis 56; *maximum* of 30 analyses.
58	Chaigneau and Assadi, 1963.[51]	Fluorite	-------	14.9	45.9	-------	31.1	3.5	4.6	-------	-------	---------	Nonuraniferous fluorite; Italy. Sample I.
59	do	do	-------	70.8	13.1	-------	.0	5.9	.0	-------	-------	---------	Nonuraniferous fluorite; Hungary. Sample VI.

TABLE 2.—*Analyses of gases from fluid inclusions in rocks and minerals*—Continued

Analysis No.	Reference	Material	Volume percent of evolved gases									Volume (ml per 100-gram sample)	Sample notes
			H_2O	CO_2	CO	O_2	H_2	N_2[1]	CH_4	C_2H_6	H_2S		
GASES LOW IN "ORGANIC" CONSTITUENTS—Continued													
Ore deposits—Continued													
60	Assadi and Chaigneau, 1962.	do	-------	71.17	3.58	-------	14.31	2.59	-------	-------	-------	198.5	Uraniferous fluorite ("antozonite"); Vendée, France. Sample 4.[52]
61	do	do	-------	91.20	.78	-------	.0	8.02	-------	-------	-------	3,350	Uraniferous fluorite ("antozonite"); Velence-Szüzvar, Hungary.[53]
62	Chaigneau and Marinelli, 1964.[54]	Magnetite	-------	91.3	3.2	-------	.9	.3	-------	-------	-------	599	Calamita magnetite deposit, Elba, Italy. Sample 1.
63	do	Ilvaite	-------	56.9	4.7	-------	30.2	1.1	-------	-------	-------	127	Calamita magnetite deposit, Elba, Italy. Sample 2.
64	Barker, 1965b.[55]	Fluorite	-------	61.43	16.34	-------	18.58	1.02	2.60	-------	-------	25.2	Groverake mine, North Pennines, England. Heated sample.
65	do	do	-------	82.41	.00	-------	5.32	12.18	.01	-------	-------	15.2	Groverake mine; crushed sample.
66	do	do	-------	72.53	9.04	-------	11.90	1.23	5.29	-------	-------	15.9	Stottfieldburn mine, North Pennines, England. Heated Sample.
67	do	do	-------	81.17	.00	-------	4.27	12.13	2.41	-------	-------	27.9	Stottfieldburn mine; crushed sample.
68	do	do	-------	70.59	9.45	-------	10.93	1.26	7.74	-------	-------	41.9	Menheniot, east Cornwall, England, Heated sample.
69	do	do	-------	51.14	.00	-------	2.38	46.48	.00	-------	-------	55.2	Menheniot; crushed sample.

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[1] Probably includes noble gases in most analyses.

[2] CO_2 values are sum of (CO_2+H_2S); CH_4 values are (CH_4+higher hydrocarbons).

[3] (Ar+Kr+Xe), 0.954; (Ne+He), 0.003.

[4] Rare gases not determined.

[5] Ar, 0.4; SO_2, 3.7; C_2H_2, 0.4; other hydrocarbons, 0.4.

[6] These are mainly averages of 47 samples run by gas chromatography. (Minimum and maximum values are also given.) The O_2, Ar, H_2, He, C_2H_6 and H_2S determinations are the average of one to three determinations. Ar, 0.2; He, <0.5; C_2H_6 and CO values represent limit of detection; H_2S was detected but was below the limit of analysis.

[7] Analysis in molecular percent, *not* volume percent, determined by mass spectrometer. Other major constituents are: propane, 4.66; N-butane, 2.45; pentanes, 3.32; hexanes, 1.57; I-butane, cyclopentane, benzene, methylcyclopentane, toluene, methylcyclohexane, heptanes, xylene, and di-methylcyclohexane, 0.03 to 0.81 percent each.

[8] Analyses 5 through 9 are recalculated from Petersil'e's averages for five, 15, three, eight and eight analyses, respectively. Analyses 10, 11, and 12 are averages of unspecified numbers of analyses. Analysis 13 is the average of Petersil'e's averages of eight urtites and 10 iolites. Some of these same analyses are given by Petersil'e (1961) with minor differences. Analyses for C_3H_8 and C_4H_{10}, respectively, in the sequence given: 0.77, 0; 0.88, 0.18; 0, 0; 0.28, 0.14; 0.14, 0.02; 1.62, 0; 1.51, 2.26; 0, 0; 0.71, 0.0.

[9] Analysis given by Petersil'e on a nitrogen-free basis; some similar(?) rocks from the same complex showed up to 90 percent N_2.

[10] Original analyses given in cc/kg; these values are the sum of listed constituents.

[11] For analysis of bitumens in same rock, see text.

[12] No nitrogen analysis stated but khibinites from the same complex showed as much as 90 percent N_2.

[13] "Lenticular" type apatite-nepheline rock.

[14] "Spotty" type apatite-nepheline rock.

[15] No nitrogen analysis stated, but khibinites from the related Khibina massif showed as much as 90 percent N_2.

[16] C_3H_8, 0.38; C_4H_{10}, 0.09.

[17] C_3H_8, 0.00; C_4H_{10}, 0.00.

[18] Average of 13 analyses. C_3H_8, 0.10; C_4H_{10}, 0.20.

[19] The mineral data table headings include "C_2H_3" and "C_3H_4," which presumably are misprints for the C_2H_6 and C_3H_8 given here.

[20] Average of 17 analyses. C_3H_8, 0.45; C_4H_{10}, 0.07.

[21] Quoted by Ikorskii (1962) from an unspecified work of I. A. Petersil'e. Includes: C_3H_8, 0.05; C_4H_{10}, 0.00.

[22] Evolved on heating in vacuum. The volume of gas per 100 g is estimated from the stated 0.68 volume percent gas.

[23] Includes others higher in series.

[24] Undersaturated hydrocarbons, 0.3; He, 0.0316, Ar, 0.0156.

[25] N_2 includes rare gases; rare gases, 0.22; He, absent.

[26] Analyses 21, 22, and 23 are arranged in order of descending temperature and time of formation. Earlier aplite border showed only a very small amount of gas, all CO_2. Data originally in cm³/kg. Argon (Ar+Kr+Xe) and helium (He+Ne) values, in the sequence given: 3.30, 0.0030; 0.90, 0.0420; 0.95, 0.0037. Elinson and Polykovskii (1961a) list these same analyses, with minor differences, and indicate CO and higher hydrocarbons as 0.0 percent in analyses 22 and 23.

[27] In analyses 23a, 23b, and 23c the heating temperature (°C) and argon (percentage of Ar), in sequence, are: 975°, 0.01 percent; 601°, tr; 975°, 0.01 percent. The CO contents are subject to larger uncertainty. H_2S and SO_2 were found in the gas from the biotite.

[28] CO_2 includes other acid gases if present.

[29] (He+Ne), 0.0000 percent; (Ar+Kr+Xe), 0.2786 percent.

[30] Data for N_2 include noble gases; data for CH_4 include C_2H_6, and are uncertain where placed in parentheses.

[31] In analyses 36 through 41 Ar and He values, in the sequence given, are: 1.40, ----; 2.82, ----; 1.10, 0.0036; 2.13, ----; 1.6, 0.0065; 1.12, 0.0104. Original analyses are in milliliters per kilogram. The authors state in a footnote (p. 774) that the analytical method used (by difference) might give N_2 values that are too high.

[32] The sum given by the authors does not equal the sum of the constituents given; the latter are assumed correct for this recalculated analysis.

[33] Average of the two analyses presented by the authors (their numbers 495 and 195 1-1).

[34] Average of the three analyses presented by the authors (their numbers 496, 195 1-2, and 195).

[35] Recalculated from estimated data.

[36] Includes heavy hydrocarbons.

[37] The data for CO_2 include NH_3, SO_2, and H_2S, if present.

[38] The data for CH_4 include N_2, F_2, noble gases, and higher hydrocarbons, if present.

[39] Included also traces of SO_2, NH_3, F, Cl(?), and hydrocarbons(?).

[40] In analyses 46, 47, and 48 He and Ar values, in the sequence given, are: 2.41, 0.80; 0.34, 0.031; 0.018, 0.0011. The authors imply (p. 446) that the H_2, CO, and O_2 might be from the decomposition of additional H_2O and CO_2.

[41] Recalculated from data of Damon and Kulp (1958, table 6) plus 0.24 weight percent H_2O (Prof. P. E. Damon, written commun., 1963).

[42] H_2O was assumed to be 0.24 weight percent to compare with other two analyses.

[43] The H_2 and CO found were assumed to be formed from the decomposition of H_2O and CO_2.

[44] He, 3.0; Ar, 0.3; SO_2, 0.2 volume percent.

[45] CO_2 includes H_2S if present; CH_4 includes C_nH_{2n+2} series; N_2 includes rare gases. C_nH_m, 0.0 percent.

[46] Analyses 50 through 54 recalculated from their data. Analyses for Cl_2, in the sequence given are: 0.006, 0.005, 0.67, n.d., 0.05.

[47] Kovalishin (1962) quotes this analysis also, but he lists the CO_2 determination as "CO_2+H_2S."

[48] Relative volume of water not stated.

[49] Assumed by Umova and others (1957) to be in error (see text).

[50] Only one analysis as such is given, and for each of three other types of quartz, by Chukhrov. Analyses 56 and 57 are based on a total of 30 individual sample analyses; only the extremes are reported here. CH_4 includes heavy hydrocarbons.

[51] In analyses 58 and 59 SiF_4 is reported as 0 and 2.4, respectively; HCl, 0, 1.0; NO, 0, 6.8. N_2 includes rare gases.

[52] SiF_4, 6.86; HCl, 1.49; U, 20,000 ppm; loss on ignition, 1.52 weight percent; N_2 includes rare gases.

[53] SiF_4 ,0.0; HCl, 0.0; U, 80 ppm; loss on ignition, 8.06 weight percent; N_2 includes rare gases.

[54] SO_2 is reported as 4.3 and 7.1 percent in analyses 62 and 63, respectively. N_2 includes rare gases.

[55] Duplicate samples were heated to 400°C in vacuum or crushed in vacuum to release gases. Water analyses in the given sequence (analyses 64–69), respectively, are: 99.25, 99.74, 98.74, 99.89, 99.67, and 99.84. The data in the table are on a water-free basis.

TABLE 3.—*Summary of quantitative and semiquanti-*

(X indicates quantitative analyses were made on all samples, or number in parentheses indicates number of samples on which analyses were made. Other symbols: S, of presumably quantitative analysis; N.d., not detected; tr, trace. An attempt was made to report only the original statements of analyses, but it is not always

Reference	Total number of samples analyzed	Na	K	Ca	Mg	Cl	SO$_4$	Li	Rb	Cs	Al^1	Cu	F	PO$_4$	B^1	Si^1	Br	HCO$_3^{-1}$
Ackermann and others, 1964	?	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Ames, 1958	4	X	X	S	S	X	X	X	X	X	S	S	----	----	----	----	----	----
Andrusenko, 1966	4	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Andrusenko, 1968	2	X	X	X	X	----	----	----	----	----	----	----	----	----	----	----	----	----
Andrusenko and Il'in, 1960	5	X	(3)	X	(3)	X	(3)	----	----	----	----	S	----	----	----	----	----	(2)
Andrusenko and Kiyevlenko, 1966.	37	X	X	X	X	X	X	----	----	----	(1)	----	(1)	----	----	----	----	X
Andrusenko and Kiyevlenko, 1968.	1	X	X	X	----	X	----	X	----	----	----	----	----	----	X	----	----	----
Anufriyev, 1966	19	X	X	X	X	X	X	X	----	----	----	----	X	----	----	X	----	X
Anufriyev, 1968	19	X	X	X	(9)	X	X	(10)	----	----	----	----	(5)	----	----	----	----	X
Arnautov and others, 1965	10	X	X	X	X	X	X	X	----	----	X	----	X	----	----	X	----	X
Assadi and Chaigneau, 1962	6	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Bakumenko and others, 1969	7	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Barker, 1965a	13	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Barker, 1965b	3	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Bazarov, 1968a	18	X	X	X	X	X	X	----	----	----	X	----	X	----	----	X	----	----
Berger and Moskalyuk, 1962	11	X	X	X	----	X	X	----	----	----	----	----	X	----	----	X	----	X
Berzina and Sotnikov, 1968	5	X	X	X	X	----	----	----	----	----	X	----	X	----	----	----	----	----
Bogomolov and Krasovskii, 1967.	>4?	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Bratus' and Kovalishin, 1968	11	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Bratus' and others, 1968	7	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Bunsen, 1851	1	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Chaigneau, 1967a	3	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Chaigneau, 1967b	6	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Chaigneau and Assadi, 1963	6	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Chaigneau and Marinelli, 1964.	7	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Chamberlin, 1908	112	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Chukhrov, 1960	31	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Czamanske and others, 1963	2	----	----	----	----	----	----	----	----	----	----	X	----	----	----	----	----	----
Damon and Kulp,[16] 1958	4	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Dinisenko, 1967	14	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Dolgov, 1968d	25	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Dolgov, Pogrebnyak, and Shugurova, 1969.	20	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Dolgov and Shugurova, 1966a.	100	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Dolgov and Shugurova, 1968	192	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Dolomanova, 1966	19	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Dolomonova and others, 1966.	2	S	----	S	S	----	----	S	----	----	S	S	----	----	S	----	----	----
Doroshenko and others, 1968.	18	X	X	X	X	X	----	(7)	----	----	----	----	(7)	----	----	X	----	X
Elinson, 1949	38	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Elinson, 1956	27	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Elinson and Polykovskii, 1961a and 1961b.	14	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Elinson and Polykovskii, 1963.	13	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Elinson and Polykovskii, 1967.	13	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Elinson, Polykovskii, and Shuvalov, 1969.	9	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Elinson and Sazonov, 1966	7	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Emmons, 1964	32	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Ermakov, 1965b	1	X	X	X	X	X	X	X	----	----	X	S	X	----	X	X	----	X
Evzikova and Moskalyuk, 1964.	2	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Faber, 1941	19	X	X	X	----	X	X	----	----	----	X	----	----	----	----	X	----	----
Fedorchuk, 1963	>1	X	X	----	----	X	----	----	----	----	----	----	----	----	----	----	----	X
Fedorchuk and others, 1963	5	X	X	----	----	X	----	----	----	----	----	----	----	----	----	----	----	X
Feklichev, 1962	1	X	X	X	----	----	----	X	----	----	----	----	----	----	----	----	----	----
Finko, 1964	2	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----

tative chemical and isotopic analyses of fluid inclusions

spectrographic determination (qualitative or semiquantitative); R, ratios only were given in original; E, estimate only, qualitative analysis, or qualitative statement possible to recognize duplication where credits are not given or are not available because of partial translation)

CO_3^{-2}	Fe[1]	H_2O	CO_2[2]	CO	H_2	N_2[3]	CH_4	C_2H_6	H_2S[4]	Other	D/H	pH	Sample notes
------	------	------	X	X	X	X	X	X	X	O_2, Ar, He	------	------	Halite and sylvite (popping salt).
------	------	------	------	------	------	------	------	------	------	------	------	------	Galena, barite, and fluorite (2).
------	------	X	------	------	------	------	------	------	X	------	------	------	Iceland spar and analcime.
------	------	------	------	------	------	------	------	------	------	------	------	------	Celestite and barite; Lower Tungusk.
------	------	------	------	------	------	------	------	------	------	Pb, Zn, Mo, Ni, Pt, Os, Rn(Rh?), La, V.	------	------	Iceland spar.
------	------	------	------	------	------	------	------	------	------	------	------	(2)	Do.[5]
------	------	------	------	------	------	------	------	------	------	------	------	X	Iceland spar, from pegmatite.[6]
------	------	------	E	------	------	E	------	------	E	O_2(E)	------	X	Quartz; southern Urals, U.S.S.R.
------	------	------	------	------	------	------	------	------	------	Sr(5)	------	------	Quartz from southern Urals, U.S.S.R.
X	X	------	------	------	------	------	------	------	------	Mn, Ni, Ti	------	------	Quartz, fluorite; chambered pegmatite.
------	------	------	X	X	X	X	------	------	------	SiF_4, HCl	------	------	Antozonite (uraniferous fluorite).
------	------	------	X	X	X	X	------	------	------	O_2, acid gases	------	------	Quartz phenocrysts from porphyry.
------	------	X	X	X	X	X	X	------	------	Ar, H_2S(1), SO_2(1)	------	------	Various, from igneous rocks.[7]
------	------	X	X	X	X	X	X	------	------	Hydrocarbons	------	------	Fluorite; England.[8]
------	X	------	------	------	------	------	------	------	------	Ni, Ti, Mn	------	------	Quartz and fluorite from pegmatite.[9]
------	------	------	------	------	------	------	------	------	------	Sr, Ba	------	X	Quartz crystals; Aldan, U.S.S.R.
------	X	------	------	------	------	------	------	------	------	Mn, Ni, Ti	------	------	Quartz from Sorskii ore deposit.
------	X	------	------	------	------	------	------	------	------	------	------	------	Sphalerite and galena.
------	------	------	X	------	X	X	X	------	------	O_2	------	------	Quartz from graphite-bearing veins.[10]
------	------	X	X	------	X	X	X	------	------	Ar	------	X	Quartz from pegmatites.
------	------	X	X	X	X	X	X	------	------	O_2	------	------	Halite; Wieliczka, Poland.[11]
------	------	X	X	X	X	X	X	------	------	SO_2, HCl	------	------	Apatite; Oka complex, Canada.
------	------	------	X	X	X	X	X	X	------	HCl, SO_2, CS_2, CSO	------	------	Quartz, various localities.[12]
------	------	------	X	X	X	X	X	------	------	SiF_4, HCl, NO	------	------	Nonuraniferous fluorite.
------	------	------	X	X	X	X	------	------	------	SO_2	------	------	Minerals of Calamita magnetite deposit.
------	------	------	X	X	X	X	X	------	X	SO_2, He, NH_3	------	------	Rocks.[13]
------	------	------	X	X	X	X	X	------	------	------	------	------	Quartz.[14]
------	------	------	------	------	------	------	------	------	------	Mn, Zn	------	------	Fluorite and quartz.[15]
------	------	X	X	X	X	X	X	------	X	O_2, He, Ar, SO_2(1)	------	------	Beryl.
------	------	------	X	------	X	X	X	------	------	Bitumen, Ar	------	------	Alkaline rocks and minerals.
------	------	------	X	X	X	X	------	------	X	O_2	------	------	Various.[17]
------	------	------	X	X	X	X	------	------	------	O_2, acid gases	------	------	Gas bubbles in tektites.
------	------	------	X	------	------	X	------	------	X	SO_2, NH_3, Cl	------	------	Quartz, topaz, fluorite, galena, sphalerite, and calcite.[18]
------	------	------	X	(49)	(49)	X	------	------	X	O_2	------	------	Various minerals.[19]
------	------	------	X	X	X	X	X	------	------	O_2, (He+Ne), (Ar+Kr+Xe).	------	------	Quartz, granite, etc.[20]
------	S	------	------	------	------	------	------	------	------	Ba, As, Mn, Sn, W, Zr, Ti, Pb, Nb, Ga, Cr, Ni, Ag, Be(S).	------	------	Quartz.[21]
------	------	------	------	------	------	------	------	------	------	------	------	X	Fluorite from various deposits.
------	------	------	X	------	X	X	X	------	------	------	------	------	Metamorphic and sedimentary rocks.
------	------	------	X	X	X	X	X	------	------	------	------	------	Igneous rocks and ore deposits.
------	------	------	X	------	X	(8)	X	------	------	Ar, He	------	------	Pegmatite minerals.
------	------	------	X	X	X	X	X	X	------	Ar, He	------	------	Mainly quartz.
------	------	------	X	------	X	X	X	------	------	Ar, He	------	------	Quartz from skarns.
------	------	------	X	------	X	X	------	------	------	------	------	------	Granite and greisen from tungsten deposits.
------	------	------	X	------	X	X	X	------	------	(Ar+Kr+Xe)	------	------	Various minerals from Kurusai.
------	------	------	X	X	X	X	X	------	------	O_2	------	------	Various igneous rocks.
X	X	------	------	------	------	------	------	------	------	Mn, SiF_6^{-2}, Ba, Cr, La, Ni, Pb, Ti, Zn, Zr(S).	------	X	Fluorite; Bektauata pegmatite.[22]
X	X	------	------	------	------	------	------	------	------	------	------	------	Calcite and dolomite from carbonatites.[23]
------	------	X	X	------	------	------	------	------	------	------	------	------	Granite (14), basalt (1), quartz (2), and feldspar (2).
------	------	------	------	------	------	------	------	------	X	Bitumens	------	X	Cinnabar.[24]
------	------	------	------	------	------	------	------	------	------	------	------	X	Quartz from an antimony-mercury deposit.[25]
------	X	------	------	------	------	------	------	------	------	------	------	------	Beryl.
------	------	------	X	X	X	X	X	------	------	O_2, (He+Ne), (Ar+Kr+Xe).	------	------	Pumice.

TABLE 3.—*Summary of quantitative and semiquantitative*

Reference	Total number of samples analyzed	Na	K	Ca	Mg	Cl	SO_4	Li	Rb	Cs	Al[1]	Cu	F	PO_4	B[1]	Si[1]	Br	HCO_3^{-1}
Girault, 1966	3	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Goguel, 1963	43	X	X	X	----	X	X	----	----	----	----	----	----	(2)	----	----	----	----
Goguel, 1965	22	X	X	X	----	X	X	----	----	----	----	----	----	----	----	----	----	----
Govorov and others, 1968	20	X	X	X	X	X	X	----	----	----	----	----	X	----	----	----	----	X
Grushkin and Prikhid'ko, 1952; and Grushkin, 1958.	3	X	X	X	X	X	----	----	----	----	----	----	X	----	----	----	----	X
Haffty and Pinckney, 1967	3	X	----	X	X	----	----	----	----	----	----	----	----	----	----	----	----	----
Hall and Friedman, 1963	20	X	X	X	X	X	X	----	----	----	----	----	----	----	(5)	----	----	----
Holser, 1963	8	----	----	R	R	R	R	----	----	----	----	----	----	----	----	----	R	----
Hoy and others, 1962	1	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Ikorskii, 1962	1	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Ikorskii, 1964	17	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Ikorskii, 1968	89	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
In'shin, 1958	1	X	X	X	X	X	X	----	----	----	X	S	----	----	----	X	----	X
Ivantishin, 1947	1	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	----
Iwasaki and others, 1956	12	(1)	(1)	(1)	(1)	X	(1)	----	----	----	(1)	----	----	----	----	----	----	----
Jeffrey and Kipping, 1963	9	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Kalyuzhnaya and Kalyuzhnyi, 1963.	2	X	X	X	X	X	X	X	----	----	X	----	X	----	X	----	----	X
Kalyuzhnyi, 1961	?	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	----
Kalyuzhnyi and Kovalishin, 1967.	48	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Kalyuzhnyi and others, 1966.	7	X	X	X	X	X	X	X	----	----	X	----	X	----	X	----	----	X
Kalyuzhnyi and Pritula, 1967.	40	X	X	X	X	X	X	X	----	----	X	----	X	----	X	----	----	X
Kalyuzhnyi and Pritula, 1968.	4	X	X	X	X	X	X	X	----	----	X	----	X	----	X	----	----	X
Kalyuzhnyi and Shchiritsya, 1962.	2	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Khetchikov, Efimova, and others, 1966a.	5	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Khetchikov, Efimova, and others, 1966b.	10	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Khitarov, D., 1965b	3	X	X	X	X	X	----	----	----	----	----	----	X	----	----	X	----	X
Khitarov, D., 1968	3	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Khitarov and Moskalyuk, 1955.	15	X	X	X	X	N.d.	X	----	----	----	----	----	----	----	----	----	----	X
Do	1	X	----	X	X	X	X	----	----	----	----	----	----	----	----	----	----	N.d.
Khitarov and Rengarten, 1956.	[35] 9	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Khitarov and others, 1958	8	X	----	X	X	X	----	----	----	----	----	----	----	----	----	----	----	X
Khitarov and Uchameyshvili, 1962.	9	X	X	X	----	X	X	----	----	----	----	----	----	----	----	----	----	X
Khodakovskiy, 1965	>400	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Khundadze, 1965	2	X	X	X	----	X	X	----	----	----	----	----	----	----	----	----	----	X
Kiyevlenko, 1958 and 1959	6	X	----	X	X	X	----	----	----	----	----	----	----	----	----	----	----	X
Klyakhin and Levitskii, 1968.	17	X	X	X	X	X	X	----	----	----	----	----	X	----	----	----	----	X
Kokubu and Katsura, 1956	18	(1)	(1)	(1)	(1)	X	(1)	----	----	----	(1)	----	----	----	----	(1)	----	----
Kokubu and others, 1957	5	X	X	X	X	X	X	----	----	----	X	----	X	X	----	X	----	----
Koltun, 1957	6	X	X	X	X	X	X	----	----	----	X	----	----	----	X	X	----	----
Königsberger and Müller, 1906a, 1906b.	2	X	X	X	----	X	X	X	----	----	----	----	----	----	----	----	----	----
Koptev-Dvornikov and others, 1962.	7	X	X	X	X	X	X	----	----	----	----	----	(3)	----	----	----	----	X
Kostyleva, 1964	15	(6)	(6)	(6)	(6)	(6)	(6)	----	----	----	----	----	----	----	----	(6)	----	----
Kovalishin, 1962	4	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Kovalishin, 1965	15	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Kovalishin, 1966	24	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Kramer, 1965	30	R	R	R	R	R	R	----	----	----	----	----	R	----	----	----	----	----
Kranz, 1965 and 1966	15	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Kranz, 1968a and 1968b	27	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Krol and Gurkina, 1957	1	E	E	----	----	E	----	----	----	----	----	----	----	----	----	----	----	E
Lamar and Shrode, 1953	19	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Lazarenko, 1967	7	X	X	X	X	X	X	X	----	----	X	----	X	X	----	----	----	X
Lindgren, 1895	1	X	X	X	X	X	X	----	----	----	X	----	----	----	----	X	----	----
Lisitsyn and Malinko, 1957	>1	E	E	E	E	E	E	----	----	----	S	S	----	----	----	S	----	E
Lisitsyn and Malinko, 1961	12	X	X	X	X	X	X	S	----	----	S	S	----	----	----	X	----	X
Malyshev and Khodakovskii, 1964.	3	X	X	X	----	X	----	----	----	----	----	----	----	----	----	----	----	X
Maslova, 1961	1	X	X	X	N.d.	X	N.d.	----	----	----	N.d.	----	N.d.	N.d.	----	N.d.	----	X
Do	15	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Maslova, 1963	5	X	X	----	----	X	----	----	----	----	----	----	----	----	----	----	----	X
Maslova, 1965	4	X	X	X	X	X	X	----	----	----	X	----	----	----	----	X	----	X

chemical and isotopic analyses of fluid inclusions—Continued

CO_3^{-2}	Fe[1]	H_2O	CO_2[2]	CO	H_2	N_2[3]	CH_4	C_2H_6	H_2S[4]	Other	D/H	pH	Sample notes
		X	X	X	X	X	X						Apatite; Oka Canada.[26]
		X	X	X	X	X	(X	X)	X				Minerals from eight granites, olivine (2), kyanite (1), and topaz (1); quartz blank.
		X	X	X	X	X	X						Various minerals.
		(1)										X	Feldspar, fluorite, quartz, and galena.
		E										X	Fluorite.
													Do.[27]
		X	E				E				X		Fluorite (10), quartz (1), calcite (3), sphalerite (2), galena (2), barite (1), and witherite (1).
													Halite.
		X	X	X	X	X	X			Ar, O_2, SO_2, C_2H_2, and other hydrocarbons.			Do.
			X	X	X		X	X		C_3H_8, C_4H_{10}			Roscherite (nepheline rock).
			X		X		X	X		C_3H_8, C_4H_{10}			Alkalic rocks and minerals.[28]
			X		X		X	X		C_3H_8, C_4H_{10}			Various minerals and rocks; Khibiny massif, U.S.S.R.
	S		E						X	Mn, Ti, Sr, and Ba; all S	X		Quartz.
													Galena.[29]
	(1)									NH_4, NO_2, NO_3			Cavities in basalt.
					(4)	(4)	(4)	(4)		He, O_2, Ar			Minerals and rocks.[30]
X	X		X	X	X	X	X			SiF_6^{-2}, Mn, O_2, C_nH_m			Beryl and topaz.
			X	X	X	X	X			O_2, SiF_6^{-2}, F^{-1}		X	Quartz, topaz, and barite; Volynia, etc.
			X		X	X	X			O_2			Granite and quartz from pegmatites.
X	X		X	X	X	X	X	X		Mn, SiF_6, O_2		X	Quartz and pegmatites; Volynia, U.S.S.R.
X	X									Mn^{+2}, SiF_6^{-2}		X	Various pegmatite minerals.
X	X									Mn^{+2}, SiF_6^{-2}		X	Quartz from Volynia, U.S.S.R.
			X									X	Quartz.[31]
	X												Quartz from tin-sulfide deposits.
	X												Sphalerite and galena from Maritimes, U.S.S.R.
												X	Fluorite from greisens.
													Quartz from greisens.[32]
	X									Pb (n.d.)			Galena; Transbaikal ore deposits, U.S.S.R.[33]
	N.d.									Pb			Galena; Kazakhstan ore deposits, U.S.S.R.[34]
			X										Quartz (3), feldspar (3), and biotite (3).
		X											Calcite.[36]
										Ba + Sr			Barite, Choida deposit.
													Many, from ore deposits.[37]
										Ba + Sr		X	Barite; Chordsk deposit, U.S.S.R.
		X											Calcite.[38]
										NH_4			Fluorite, calcite, barite, quartz, and galena.
	(1)									NH_3, NO_2, NO_3		X	Cavities in basalts.
	X									NH_4^{+1}, NO_2, NO_3	X[39]	X	Do.
													Quartz.
X		X	X										Do.
			X										Granite.[40]
(6)			X		X	X	X			O_2, (Ar, Kr, Xe)			Quartz; Kazahkstan ore deposits.[41]
			X	X	X	X	X			O_2			Quartz, topaz; pegmatites from Volynia.[42]
			X	X	X	X	X	X		O_2			Pegmatite minerals and granites.[42]
			X	X	X	X	X			O_2			Pegmatite minerals and rocks.[43]
													Halite and cherts.[44]
		X	X		X	X	X		X	Ar, He			Various fluorites.[45]
		X	X		X	X	X	X	X	C_2H_4, NH_3, He, Ar			Fluorite and feldspar.[46]
			E										Fluorite from greisen.[47]
									X				Limestone and dolomite.
X	X									Mn^{+2}			Quartz; Volynia.[48]
	X												Quartz.[49]
	S									Mn, Ni, Pb, Zn, Sn, and Sr; all S.			Quartz.
	S	E	E							Ga, Sr, Ba, Sn, Mo, Ti, V, Ag, Pb, Zn, Mn, Ni (S).			Do.
										F (tr.)			Galena; Zambarak deposit.
N.d.	N.d.		N.d.						N.d.	HS^{-1} (N.d.)		X	Quartz.
			X						X				Do.
												X	Quartz from Sb-Hg deposit. See footnote 25.
X	X											X	Iceland spar.

TABLE 3.—*Summary of quantitative and semiquantitative*

Reference	Total number of samples analyzed	Na	K	Ca	Mg	Cl	SO_4	Li	Rb	Cs	Al[1]	Cu	F	PO_4	B[1]	Si[1]	Br	HCO_3^{-1}
Maslova and Gurinova, 1966.	4	X	X	X	X	X	X	----	----	----	X	----	----	----	----	X	----	X
Maslova and Morozov, 1968	2	X	X	X	X	X	X	----	----	----	X	----	----	----	----	X	----	X
Matsuo and Miyake, 1966	18	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Mirochnikov, 1968a	8	X	----	X	X	X	X	S	----	----	S	S	----	----	----	S	----	X
Morozov, 1966a	16	X	----	X	X	X	X	----	----	----	S	S	E	----	S	X	----	X
Morse, 1905	20	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Moskalyuk, 1959	25	X	X	X	X	X	X	----	----	----	X	----	X	----	X	X	----	X
Moskalyuk, 1961	41	X	X	X	X	X	X	----	----	----	X	----	X	----	X	X	----	X
Moskalyuk and Zakharchenko, 1966.	19	X	X	X	X	X	X	----	----	----	X	----	X	----	X	X	----	X
Motorina and Bakumenko, 1968.	8	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Müller and Heymel, 1956	?	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Murray, 1957	1	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Nesmelova, 1959	20	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Nikanorov and others, 1968	17	X	X	X	X	X	X	----	----	----	----	----	X	----	----	X	----	X
Ohmoto, 1968a and 1968b	56	(41)	(41)	(41)	(41)	(41)	----	----	----	----	----	----	----	----	----	----	----	----
Petersil'e, 1958b	9	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Petersil'e, 1959 and 1961	32	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Petersil'e, 1960	36	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Petersil'e, 1962 and 1963	76	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Petersil'e and others, 1965	26	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Petersil'e and Sorensen, 1970.	28	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Petrichenko and Shaydetskaya, 1968.	1	X	X	X	X	X	X	X	X	X	X	----	X	----	X	----	X	X
Polykovskii, 1962a	5	X	X	X	X	X	X	----	----	----	N.d.	----	X	----	N.d.	X	----	X
Polykovskii, 1962b	5	X	X	X	X	X	X	----	X	----	N.d.	----	X	----	N.d.	X	----	X
Polykovskii, 1964	?	X	X	X	X	X	X	----	----	----	----	----	X	----	----	----	----	X
Polykovskii, 1965	9	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Preisinger and Huber, 1964	2	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Prikazchikov, 1966	13	X	----	X	X	X	X	----	----	----	----	----	----	----	----	X	----	X
Prikazchikov and others, 1964.	5	X	----	X	X	X	X	----	----	----	X	----	----	----	----	X	----	X
Puzanov, 1960	2	X	X	X	X	X	X	----	----	----	----	----	----	----	----	X	----	X
Rakhmanov, 1968	1	X	----	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Roedder, 1958	11	X	X	----	----	X	X	X	X	X	----	----	----	----	----	----	----	----
Roedder and others, 1963	21	X	X	X	X	X	X	----	----	----	----	----	----	----	X	----	----	----
Rutherford, 1963	1	R	R	R	R	----	----	----	----	----	----	----	----	----	----	----	----	----
Rye, 1964	15	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Rye, 1965	~50	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Rye and Haffty, 1968	22	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	----
Saitô, 1951	5	----	----	----	E	----	----	----	----	----	----	E	----	----	----	----	----	----
Sharkov, 1958	4	X	X	X	X	X	X	----	----	----	----	S	----	----	X	----	----	X
Shchiritsya, 1960	2	X	X	X	X	X	X	S	----	----	X	----	----	----	X	S	----	----
Sheshulin, 1961	3	X	S	X	X	X	X	S	----	----	S	----	----	----	----	S	----	X
Do	4	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Shugurova, 1967b	26	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Silvestri, 1882	1	X	----	X	----	X	X	----	----	----	----	----	----	----	----	----	----	----
Sjögren, 1893	1	X	X	X	X	X	X	----	----	----	----	----	----	----	----	----	----	----
Skropyshev, 1957	1	X	X	X	N.d.	X	X	----	----	----	X	----	----	----	----	X	X	----
Smirnova, 1958	2	X	N.d.	X	N.d.	X	N.d.	----	----	----	----	----	----	----	----	----	X	----
Sobolev and others, 1968	7	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Sokolov and Zakharchenko, 1961.	4	X	X	X	X	X	X	----	----	----	----	----	X	X	X	X	----	X
Sugawara and others, 1944	1	X	X	X	X	X	X	----	----	----	X	----	----	----	----	X	----	----
Sushchevskaya and Barsukov, 1965.	[78]12	X	X	X	----	X	----	----	----	----	----	----	(25)	----	----	----	----	X
Sushchevskaya and others, 1966.	58	(7)	(7)	(7)	----	(39)	----	(11)	----	----	----	----	(39)	----	----	----	----	(7)
Sushchevskaya and Ivanova, 1967.	7	X	X	X	----	X	----	----	----	----	----	----	X	----	----	----	----	X
Tammann and Seidel, 1932	1	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Tender, 1967	>2?	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Tokhtuyev and Frantsuzova, 1963.	12	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Touray and Lantelme, 1966	3	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----

chemical and isotopic analyses of fluid inclusions—Continued

CO_3^{-2}	Fe[1]	H_2O	CO_2[2]	CO	H_2	N_2[3]	CH_4	C_2H_6	H_2S[4]	Other	D/H	pH	Sample notes
------	------	------	------	------	------	------	------	------	------	------	------	X	Iceland spar.[50]
X	X	------	------	------	------	------	------	------	------	------	------	X	Quartz; Pamir, U.S.S.R.
------	------	------	X	------	------	X	------	------	------	O_2, Ar	------	------	Antarctic ice.[51]
------	X	------	------	------	------	------	------	------	------	NH_4^{+1}(4), NO_3(4); Ti, Cr, Ni, Mn, Sr, Ba, Pb, Zn, Ag, Sn, Cd, Ge; all S.	------	------	Quartz from rock crystal veins.
------	S	------	------	------	------	------	------	------	E	Mn, Ba, Sr, Ti, Cr, Ni, Zn, Mo, Pb, V, Ag, Cd; all S.	------	------	Quartz; Western Pamirs.[52]
------	------	------	X	X	X	X	------	------	------	O_2	------	------	Fluorite.[53]
------	X	------	------	------	------	------	------	------	------	------	------	X	Quartz, fluorite, etc., from granite pegmatites.[54]
------	X	------	(4)	------	------	------	------	------	(4)	------	------	X	Do.
------	X	------	------	------	------	------	------	------	------	------	------	------	Quartz and fluorite; Kayib massif.[55]
------	------	------	X	X	X	X	------	------	X	NO, O_2	------	------	Tourmaline from pegmatites.
------	------	------	X	X	X	X	X	X	------	O_2, heavy hydrocarbons	------	------	Potash salts; Harz and Werra districts.
------	------	------	X	------	------	X	X	X	------	Plus 13 other hydrocarbon compounds.	------	------	Quartz.
------	------	------	X	------	X	X	X	------	X	(Ar+Kr+Xe), (Ne+He)	------	------	Carnallite and sylvite.[56]
------	------	X	------	------	------	------	------	------	------	------	------	X	Quartz from pegmatites.
------	(41)	X	X	X	X	X	X	X	------	C_3H_8, Zn (41)	(4)	------	Various minerals; Bluebell mine.[57]
------	------	------	X	X	X	------	X	X	------	C_3H_8, C_4H_{10}	------	------	Rocks, Kola Peninsula.[58]
------	------	------	X	X	X	------	X	X	------	------	------	------	Do.[59]
------	------	------	X	X	X	(4)	X	X	------	O_2, C_3H_8, C_4H_{10}, He (1), Ar (1).	------	------	Igneous rocks, Kola Peninsula, U.S.S.R.
------	------	------	X	X	X	------	X	X	------	------	------	------	Minerals and rocks, Kola Peninsula.[60]
------	------	------	(16)	(16)	(16)	------	(16)	(16)	------	C_3H_8 (16)	------	------	Alkalic rocks, Kola Peninsula.[61]
------	------	------	X	X	X	------	X	X	N.d.	He, C_3H_8	------	------	Alkalic rocks, Kola Peninsula.[62]
X	X	------	------	------	------	------	------	------	------	Mn, Ti, La, V (S), and SiF_6^{-2}.	------	X	Large inclusion in recrystallized halite.[63]
------	X	E	E	------	E	E	E	N.d	------	Ar, He	------	X	Quartz.[64]
------	------	X	(3)	------	(3)	(3)	(1)	------	------	Sr, He (2), Ar (3)	------	X	Quartz; skarns of Tien-Shan.[65]
------	------	------	X	------	X	X	------	------	------	He, Ar	------	X	Quartz-feldspar veins.[66]
------	------	------	X	------	X	X	X	------	------	Ar, He	------	------	Quartz; skarns of Tien-Shan.[67]
------	------	------	X	X	X	X	X	------	------	Ar	------	------	Feldspar from granite.
------	------	------	------	------	------	------	------	------	------	------	------	X	Quartz from pegmatites of Volynia.[68]
------	X	------	------	------	------	------	------	------	------	Nb, Be, Cu, Pb, Sn, Ga, V, Y, Mn, Ni, Ti, Cr, Mo, Zr, Sc (S).	------	X	Quartz; giant inclusions giant crystal.[69]
------	------	------	------	------	------	------	------	------	------	------	------	X	Quartz; Soviet Far East.[70]
------	------	------	------	------	------	------	------	------	------	------	------	X	Quartz from pegmatite; southern Gissar.
------	------	(1)	(1)	------	------	------	------	------	------	------	------	------	Quartz: hydrothermal (8), metamorphic (2), chert (1).
------	------	(14)	E	------	------	------	E	------	------	------	(14)	------	Fluorite (7), galena (4), quartz (2), sphalerite (7), and calcite (1).
------	------	------	------	------	------	------	------	------	------	------	------	------	Quartz.
------	------	------	------	------	------	------	------	------	------	------	X	------	Sphalerite, calcite, and quartz.[71]
------	------	------	------	------	------	------	------	------	------	------	X	------	Do.[71]
------	------	------	------	------	------	------	------	------	------	Zn, Cu	------	------	Do.[72]
------	E	------	------	------	------	------	------	------	------	Mn, Ni, Co, Pb, Zn, As, Ti, V, Mo, Be, Ce, Zr, Rh, Ru; all E.	------	------	Fluorite.
S	------	------	------	------	------	------	------	------	------	Mn, Ni, Ti, Pb, Zn; all S	------	------	Quartz.
X	------	------	E	------	E	E	E	E	------	O_2 (E), V, Mn, Cu, Zn, Pb, Ag, Ni, Ti, Cr, Sr; all S.	------	X	Do.
S	------	E	------	------	------	------	------	------	------	Mn, Ni, Pb, Ag, Zn, Sr; all S.	------	X	Quartz, feldspar, and spodumene.
------	------	------	X	(3)	(3)	(3)	(-----3-----)		------	------	------	------	Spodumene.
------	------	------	X	X	X	X	------	------	------	O_2	------	------	Fluorite and quartz from pegmatites.[73]
------	------	X	------	------	------	------	------	------	------	------	------	------	Sulfur; Sicily.[74]
------	------	------	------	------	------	------	------	------	E	------	------	E	Gypsum.
X	------	------	------	------	------	------	------	------	------	Ti, Sr (S)	------	X	Calcite.
------	------	------	------	------	------	------	------	------	------	------	------	------	Quartz from vein in marbleized limestones.[75]
------	------	------	X	X	X	X	------	------	X	NO, O_2	------	X	Chkalovite, nepheline, and tugtupite; Greenland.[76]
------	X	------	------	------	------	------	------	------	------	------	------	------	Pegmatites; Tarbagatai Range.[77]
X	X	------	------	------	------	------	------	------	------	NH_4^{+1}, NO_2, NO_3	X	X	Cavities in basalt.
(15)	------	X	(28)	------	------	------	------	------	------	------	------	------	Sulfide-cassiterite deposits.
------	------	(7)	------	------	------	------	------	------	------	------	------	------	Quartz; Myao-Chan Sn-W deposits.[79]
------	------	X	X	------	------	------	------	------	------	------	------	(3)	Quartz and wolframite.
------	------	------	X	------	------	X	X	------	------	O_2	------	------	Halite.
------	------	------	X	------	X	X	------	------	------	------	------	X	Calcite; mercury deposits.
------	------	------	X	N.d.	X	X	N.d.	------	------	O_2	------	------	Metamorphic rocks and iron ores.[80]
------	------	X	X	------	------	------	X	------	X	------	------	------	Quartz, fluorite, and dolomite.[81]

TABLE 3.—*Summary of quantitative and semiquantitative*

Reference	Total number of samples analyzed	Na	K	Ca	Mg	Cl	SO_4	Li	Rb	Cs	Al[1]	Cu	F	PO_4	B[1]	Si[1]	Br	HCO_3^{-1}
Trufanov, 1966	4	X	X	X	X	X	X	----	----	----	----	----	X	X	X	X	----	X
Trufanov, 1967	24	X	X	X	X	X	X	----	----	----	----	----	X	X	----	X	----	X
Uchameyshvili, 1965	30	X	X	X	----	X	X	----	----	----	----	----	----	----	----	----	----	X
Uchameyshvili and Khitarov, 1965.	17	X	(12)	X	----	----	----	----	----	----	----	----	(1)	----	----	----	----	(12)
Umova and others, 1957	12	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Ushakovskii, 1966	2	X	X	X	X	X	X	X	----	----	----	----	X	----	----	----	----	X
Valyashko and others, 1968	1	X	X	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Vasil'ev and others, 1961	1	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Vul'chin, 1953	38	S	X	X	S	X	X	----	----	----	(2)	S	----	----	X	----	----	----
Wahler, 1956	9	S	(3)	S	----	E	E	----	----	----	----	----	----	----	----	----	----	----
Wright, 1881	1	S	----	----	----	E	----	----	----	----	----	----	X	----	----	----	----	----
Yushkin and Srebrodol'skii, 1965.	3	X	----	X	X	X	X	----	----	----	----	----	----	----	----	----	----	X
Zakharchenko, 1950	1	E	----	S	S	X	X	----	----	----	S	----	----	----	----	X	----	X
Zakharchenko, 1961	13	X	X	X	X	X	X	----	----	----	----	----	----	----	----	X	----	X
Zakharchenko and Moskalyuk, 1968.	6	X	X	X	X	X	X	N.d.	N.d.	N.d.	N.d.	----	X	----	----	----	----	X
Zakharchenko and Trufanov, 1964.	4	X	X	X	X	X	X	----	----	----	X	----	X	----	----	X	----	X
Zakharchenko and others, 1966.	15	X	X	X	X	X	X	----	----	----	----	----	X	X	----	X	----	X
Zakrzhevskaya, 1964	4	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Zakrzhevskaya and Elinson, 1966.	4	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Zatsikha and others, 1967	6	X	X	X	X	X	X	X	----	----	X	----	X	----	X	----	----	X
Zavaritskii and Betekhtin, 1937.	1	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Zimmermann, 1966	4	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----
Zolutkhin, 1959	1	X	X	X	X	X	X	----	----	----	----	----	----	----	X	----	----	X

---

[1] Variously stated in original analyses, as element, oxide, etc.

[2] Usually includes "other acid gases."

[3] Usually includes rare gases.

[4] The absorbents used in many analyses for CO_2 also absorb H_2S, and hence yield only total (CO_2+H_2S), plus other acid gases. An "X" in this column signifies specific indications of the presence of H_2S, or (H_2S, SO_2, NH_3, and Cl), as opposed to CO_2.

[5] Includes seven analyses of individual inclusions, six apparently quoted from earlier papers of Khitarov, Rengarten, and Lebedeva (1958) and Kiyevlenko (1958), with some gross changes (misprints?). The other 30 are of aqueous extracts.

[6] Not found: Rb, Cs, Sr, Mg, Fe^{+2}, Fe^{+3}, Al, Si, F^{-1}, SO_4^{-2}, HCO_3^{-1}.

[7] CO analyses are subject to some uncertainties—see the original reference.

[8] Mass spectrometric determinations were made using two different extraction procedures on each sample—heating to 400°C, or crushing, both in vacuum. Hydrocarbons of mass numbers 77, 49, and 47 were prominent in the crushed samples.

[9] Very high values for Mn, Fe, Ni, and Ti were reported.

[10] CH_4 is sum of C_nH_{2n+2} series. C_nH_m and CO were not detected.

[11] H_2, CO, and ethylene all were 0.00 percent (Bunsen, 1851, p. 251).

[12] Each sample analyzed by mass spectrograph using two extraction procedures—heating and crushing.

[13] Not all rocks were analyzed for all constituents; "hydrocarbons" and "heavy hydrocarbons" were determined on some samples.

[14] Only one analysis as such as given, and for each of three other types of quartz "minimum" and "maximum" values are given for each constituent.

[15] Cu, Mn, and Zn were determined by neutron activation analyses of samples very similar to those analyzed for major constituents by Roedder, Ingram, and Hall (1963).

[16] Includes one sample from Prof. P. E. Damon (written commun., 1963). (See analysis 49, table 2.)

[17] H_2S is sum of (H_2S+SO_2); in part from literature.

[18] Gases stated only in graphical, ratio form. Only the sum of ($SO_2+H_2S+NH_3+Cl+F$) is plotted, although SO_2 and NH_3 were most abundant.

[19] Includes 34 analyses for heavy hydrocarbons.

[20] CH_4 includes heavy hydrocarbons. Also gives 58 analyses of rocks and minerals for F, B_2O_3, H_2O, S, and CO_2.

[21] Fe, Cu, Ti, Ca, Zn, and Cr were also detected, by electron microprobe, among the 11 daughter minerals lining the fluid inclusions. The spectrographic determinations listed were on the quartz itself, presumably with fluid inclusions.

[22] Both an acid leach and a water leach were analyzed. Some of the inclusions contained 15 recognizable phases.

[23] Also analyzed for Sr^{+2} and an anion listed as Fe^{-1} (n.d.), which was probably F^{-1}.

[24] Fedorchuk (1963) quotes the range of analyses "by Maslova" of gas-liquid inclusions in cinnabar-containing crystals of quartz from mercury deposits of "the concordant type." The ranges given are so close in many respects to those given by Maslova (1963) and by Fedorchuk, Kostyleva-Labuntseva, and Maslova (1963), for inclusions in quartz "from mercury-antimony deposits," that they are probably the same analyses; there are some very significant differences, however, in that Fedorchuk (1963) gives a wider pH range (6.0–7.4) and lower potassium values (0.09–0.12 g/1), as well as other more minor differences.

[25] The analyses given in Fedorchuk, Kostyleva-Labuntseva, and Maslova (1963) are the same as those in Maslova (1963), and presumably they are the same as those given by Fedorchuk (1963). See footnote 24.

[26] Mass spectrometric analyses of gases evolved on sequential heating to 1,000°C. Traces of HCl were found, but no SO_2 or HF were found.

[27] Made by both atomic absorption spectroscopy and a new method of optical emission spectrography.

[28] Also six analyses of bitumens for C, H, and (N+O+S).

[29] "Na_2O+K_2O" only given in original. The analyses are presented in two forms—as "percent of aqueous extract," and as "residues, as percent in galena"; these data are mutually contradictory.

[30] Determinations were made by fusion with flux followed by gas chromatography. Hydrocarbons up to C_4H_{10} were found in a granite; these and the hydrogen were believed to be from interaction of evolved H_2O and CO_2 with ferrous iron-bearing minerals.

[31] (Na+K) determined.

[32] Three leachates analyzed from each sample; no chlorine found in any leach.

[33] Only Mg and SO_4 were found in 12 of the analyses; all other constituents were "not determined" or "not detected."

[34] Pb^{+2} was found to exceed the sum of all other constituents.

[35] Also 21 analyses of rocks for CO_2 only. Goguel (1963) believes some of the CO_2 found came from the dissociation of carbonates on heating to ~520°C.

[36] Six of these analyses are also reported by Kiyevlenko (1958, 1959).

[37] This is a review article in which various chemical relationships for various types of deposits are plotted. Seventeen new analyses are listed, however, from unpublished reports that are "in press."

[38] These six analyses are also reported by Khitarov, Rengarten, and Lebedeva (1958).

[39] See Kokubu, Mayeda, and Urey (1961).

[40] Analyses by N. I. Khitarov.

[41] Kostyleva (1964, table 10) lists analyses for CO_2 plus other acid gases, O_2, H_2O, CO_2, etc. From the text it appears that "H_2O" may be H_2, and the second "CO_2" may be CH_4. The six analyses are ultramicrochemical determinations by I. N. Maslova.

[42] "CO_2" is (H_2S+CO_2); "CH_4" is C_nH_{2n+2} series; C_nH_m series was not found.

[43] By vacuum ball milling. C_nH_m and CO were not found.

[44] J. R. Kramer (oral and written commun., 1963) has provided two unpublished quantitative analyses—see table 6.

[45] Includes some mass spectrometric analyses of the gases for a variety of fluorinated hydrocarbons.

[46] By gas chromatography-mass spectrometry of gases from vacuum grinding.

chemical and isotopic analyses of fluid inclusions—Continued

CO_3^{-2}	Fe[1]	H_2O	CO_2[2]	CO	H_2	N_2[3]	CH_4	C_2H_6	H_2S[4]	Other	D/H	pH	Sample notes
------	X	------	------	------	------	------	------	------	------	------	------	X	Stages from pegmatite.[82]
------	------	X	(15)	------	(15)	(15)	(15)	------	------	O_2(15)	------	X	Pegmatite minerals and rocks.[83]
------	------	X	------	------	------	------	------	------	------	Sr	------	(4)	Barite from many deposits.[84]
------	------	------	(12)	------	------	------	------	------	------	------	------	(10)	Barite; Caucasian deposits.[84]
------	------	------	X	X	X	X	------	------	------	O_2, Cl_2	------	------	Quartz.
------	------	------	X	------	X	X	------	----	------	O_2	------	------	Quartz; Southern Urals.[85]
------	X	------	------	------	------	------	------	------	------	------	------	------	Apatite; Khibiny deposits.[86]
------	------	------	X	------	X	X	X	X	------	Undersaturated hydrocarbons, O_2, He, Ar.	----	------	Gas from kimberlite.
------	(2)	------	------	------	------	------	------	------	------	Mn(10), Zn, Pb, Ag; all S	------	------	Quartz.
------	------	(6)	------	(6)	(6)	------	------	------	------	He and Ar (S)	------	------	Quartz, beryl, and topaz.
------	------	X	X	------	------	X	------	------	X	SO_2, NH_3, Cl_2, H_2S, F (?)	------	------	Quartz.
------	X	------	E	------	------	------	------	------	E	------	------	E	Sulfur; Shorsu and Rozdol deposits.
------	S	------	------	------	------	------	------	------	E	Mn(S)	------	X	Quartz.
------	------	------	------	------	------	------	------	------	------	------	------	X	Quartz veins; Kazakhstan, U.S.S.R.
------	N.d.	------	------	------	------	------	------	------	------	------	------	X	Quartz from pegmatites of Kola Peninsula.[87]
------	------	------	------	------	------	------	------	------	------	------	------	X	Quartz and fluorite from Kazakh pegmatites. See footnote 54.
------	X	------	(12)	(12)	(12)	(12)	(12)	------	------	Sr, O_2, (He+Ne), (Ar+Kr+Xe).	------	X	Quartz and fluorite from Kazakh pegmatites.[88]
------	------	------	X	------	X	------	X	------	------	------	------	------	Nepheline-apatite ores.
------	------	------	X	------	X	------	X	------	------	------	------	------	Do.
X	X	------	(4)	(4)	(4)	(4)	(4)	------	------	Mn, C_nH_m, O_2, SO_2	------	------	Fluorite; near-Azov area.
------	------	------	------	------	X	X	X	------	------	O_2, rare gases	------	------	Borehole in dunite.
------	------	X	X	X	X	X	X	------	X	------	------	------	Quartz.[89]
------	------	------	------	------	------	------	------	------	------	------	------	X	Cavity in dacite.[90]

A variety of amines, nitriles, and fluorinated hydrocarbon compounds were determined.

[47] As reported in "Referativnyi Zhurnal Geology," abstract 4492, 1958, K and Na bicarbonates were much greater than chlorides, and CO_2 "was present." The original article may give quantitative data.

[48] SiF_6^{-2} was not found.

[49] Al_2O_3 and Fe_2O_3 combined; Na_2O and K_2O combined.

[50] Total concentrations of salts in the inclusions ranged from 96 to 449 g/l. The same analyses were given by Maslova (1965).

[51] Made by mass spectrometry on gases evolved on melting in vacuo.

[52] Na is the sum of (Na+K), obtained by difference between anions and cations.

[53] Helium group gases were looked for by spectroscopy in the residue from a 10-kg sample and were not found. The amount of water obtained was about 10 times the weight of all other gases together.

[54] A number of individual determinations were listed as not detected, trace, or not determined. Moskalyuk (1961) includes some analyses of granites and sulfide ores. Two of the analyses in Moskalyuk (1961) are also given by Zakharchenko and Trufanov (1964) with considerably different values for some constituents.

[55] B was detected in only one sample. No Al or Fe^{+2} was detected.

[56] Sum of CO_2+H_2S reported only.

[57] Ionic constituents were determined by atomic absorption on both water and acid leaches; gases were determined by mass spectrometry after extraction by vacuum crushing and by decrepitation. H_2S was not detected.

[58] Each is an average of several samples. Also includes two analyses of bitumens extracted with chloroform.

[59] Some of these analyses are given, with slightly different numbers, by Petersil'e (1961 and 1992). Most are averages for up to 15 samples. Analyses are also given for C_3H_8 and C_4H_{10}. The bitumens in three rocks were also analyzed; Petersil'e (1963) describes complex aromatic hydrocarbons and esters of carboxylic acids.

[60] Includes 69 mineral analyses, as eight averages, and seven rock averages. The mineral data table headings include "C_2H_3" and "C_3H_4" which presumably are misprints for C_2H_6 and C_3H_8.

[61] Sixteen analyses of gases from rocks and 10 analyses of bitumens (for C, H, and N+O+S) are reported; all are averages for from one to seven samples of given rock types.

[62] Analysis by vacuum crushing and gas chromatography. Appreciable amounts were also reported for iso-C_4H_{10}; n-C_4H_{10}; iso-C_5H_{12}; and n-C_5H_{12}.

[63] Also determined density (1.231 g/ml) and oxidation-reduction potential.

[64] No Fe^{+2} was detected, only Fe^{+3}. See also footnote 65.

[65] No Fe^{+2} was detected, only Fe^{+3}. These analyses, by A. A. Moskalyuk, apparently are the same five as presented by Polykovskii (1962a), but there are considerable differences both in calculated concentrations and ratios; the sum of the stated ion concentrations agrees with the sum of the calculated salts in Polykovskii (1962b, table 2) but does not agree in Polykovskii (1962a, p. 474). The data on calculated salts from Polykovskii (1962a), however, are duplicated in Polykovskii (1965).

[66] From original presentation (in abstract) of composition as salts.

[67] Five analyses of leaches, stated as assumed salt concentrations, are duplicated from Polykovskii (1962a).

[68] Includes four previously published analyses quoted from Prikazchikov and others (1964). "Na" is sum of Na+K. Traces of Sr, Ba, Li, and Rb are reported.

[69] One analysis was of a leach from crushed quartz; the others are from multimilliliter inclusions. "Na"=Na+K.

[70] Also semiquantitative spectrographic analyses for Al, Fe, Mn, Ni, Ti, Cu, Pb, Ag, Bi, Zn, Be, Sr, Li, V, Sn, and Ga.

[71] Inclusion samples similar to those on which Sawkins (1963) determined freezing behavior and filling temperatures, from Providencia, Mexico.

[72] Atomic absorption for all but Cl and SO_4, which were determined by X-ray fluorescence.

[73] SO_2, H_2S, NH_3, HCl, and HF were not found.

[74] Traces of K, Ba, and Sr also found. The total concentration of salts in this large inclusion (6 cm^3) was 0.1033 percent.

[75] As quoted by Khodakovskiy (1965).

[76] H_2S is the sum of acid gases; heavy hydrocarbons were not found.

[77] Al^{+3} and Fe^{+3} were not detected.

[78] Includes an additional 48 analyses for Na/K ratio and 38 analyses for HCO_3^{-1}/Cl^{-1} ratio.

[79] All 58 analyses are presented as 15 averages, in the form of the ratios Na/K, Na/Ca, Cl/HCO_3, and Cl/F. The methods of extraction and analysis are not stated.

[80] All 12 are multiple samples, with maximum, minimum, and average values given for unspecified numbers of samples in each category; a total of 57 samples were analyzed.

[81] Mass spectrometric analyses of the gases evolved on heating through 50-degree increments from 100° to 550°C.

[82] Only traces of Fe^{+2}, Fe^{+3}, B, and F were found.

[83] H_2O was determined by loss in weight after 2 hours at 800° to 900°C.

[84] pH values are calculated from CO_2 and HCO_3^{-1}.

[85] Averaged (?) data from 19 samples. N_2 was determined by difference.

[86] Analyses for Ca, HCO_3^{-1}, and $H_2PO_4^{-1}$ showed equal values for blank and sample.

[87] Trace of B found in one sample.

[88] Rather large quantities of rare gases were found. No O_2, CO, or CH_4 could be detected. Li, B, Sr, Fe^{+3}, and Cs were detectable in some samples, but there was no detectable Fe^{+2}.

[89] Samples from two localities, each consisting of a pair, with and without inclusions. Analyses by mass spectrometry of gases evolved on heating in vacuum to 200°, 400°, 600°, 800°, 1,000°, and 1,200°C. CO and N_2 were assigned to contamination. Some hydrocarbons of mass 28 were obtained.

[90] "Na+K" only was given in original.

TABLE 4.—*Analyses of fluid inclusions originally stated only as ratios*

[N.d. not determined]

Analysis No.	Reference	Material	Ratios by weight								Sample notes
			HCO_3/Cl	F/Cl	K/Cl	Na/Cl	Mg/Cl	Ca/Cl	Br/Cl	SO_4/Cl	
						INCLUSIONS FROM QUARTZ VEINS					
1	Vul'chin, 1953 [1]	Quartz	-------	---------	0.243	0.741	0.016	0.374	--------	0.172	Quartz-gold-pyrite vein associated with granite dikes; Berezovka, Ural, U.S.S.R.
2	Sharkov, 1958 [2]	do	0.276	---------	.155	.404	.066	.230	--------	.552	Quartz veins in metamorphosed sediments; highlands of Dagestan, U.S.S.R.
3	do	do	1.624	---------	.601	.234	.303	1.165	--------	2.092	Do.
4	Rutherford, 1963.[3]	do	-------	---------	.052	.50	.003	.28	--------	-------	Vein quartz; Coronation mine, Flin Flon area, Saskatchewan.
4a	Ushakovskii 1966.[4]	do	.063	0.011	.096	.495	.0014	.122	--------	.020	Crystal-bearing veins; Southern Urals, U.S.S.R.
4b	do	do	.450	.007	.060	.367	.030	.324	--------	.052	Noncrystal-bearing veins; Southern Urals, U.S.S.R.
						INCLUSIONS FROM SALINE BEDS					
5	Kramer, 1965 [5]	Halite	-------	0.00053	0.030	0.63	0.015	0.124	--------	0.41	Salt beds of Salina Group (Silurian); Goderich, Ontario.
6	do	do	-------	.00043	.032	.61	.014	.113	--------	.26	Salt beds of Salina Group (Silurian); Cleveland, Ohio
7	do	Chert	-------	---------	----------	.51	.031	.170	--------	.35	Uppermost Niagara (Middle Silurian) cherts; Niagara escarpment, Ontario.
8	do	do	-------	.00036	.086(?)	.56	.075	.136	--------	.03	Uppermost Niagara (Middle Silurian) cherts; Manitowoc, Wis.
9	Holser, 1963 [6]	Halite	-------	---------	----------	-------	.219	.000	.00122	N.d.	Wellington Formation of Permian age; Carey mine. Hutchinson, Kans.
10	do	do	-------	---------	----------	-------	.237	.000	.0138	N.d.	Do.
11	do	do	-------	---------	----------	-------	.247	.00	.016	.034	Do.
12	do	do	-------	---------	----------	-------	.22	.00	.017	N.d.	In core at 546.5 ft; Wellington Formation of Permian age; Naval Air Station, Hutchinson, Kans.
13	do	do	-------	---------	----------	-------	.21	.13	.013	N.d.	Core No. 2, 1,505 ft; Salina "B" salt (Silurian); Goderich, Ontario.
14	do	do	-------	---------	----------	-------	.068	.000	.0034	N.d.	Salado, first ore zone (Permian); U.S. Borax mine, Carlsbad, N. Mex.
15	do	do	-------	---------	----------	-------	.171	.000	.0035	N.d.	Do.
16	do	do	-------	---------	----------	-------	.258	.001	.0133	N.d.	Recent salt; salt flats, Laguna Ojo de Liebre, Baja California.
17	do	-----------	-------	---------	----------	-------	.0671	.0211	.0034	.139	Sea water.

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[1] Analyses for six samples (Nos. 20 through 25) by Vul'chin, all very similar, are averaged and recalculated here. The method of statement of these analyses in the original is obscure; presumably they were normalized to 2 "percent" Al+Fe+Mn (in the filtrate?). Some mutually exclusive data in the original (apparently misprints) were changed in the quotation of these data by Koltun (1957), who reviews the inclusion studies on the deposit; the newer data are used here. Other weight ratios: BO_3/Cl, 0.581; Al+Fe+Mn/Cl, 0.405.

[2] Recalculated from Sharkov's samples 166 (2) and 284(3). The actual concentrations of chloride in the two leach solutions (analyses 2 and 3) were 61.98 and 12.77 mg/l, respectively. SiO_2/Cl weight ratios in the sequence were 0.926 and 3.892. Spectrographic results for additional elements on leach residues, in the sequence given, were: Al, ----, 0.001–0.01; Fe, 0.001, 0.001; Mn, 0.001–0.01, 0.001–0.01; Ni, 0.01, 0.01; Ti, 0.001, 0.001; Cu, 0.001–0.01, 0.001–0.01; Pb, 0.01 0.01; Zn 0.001–0.01, 0.01.

[3] As a chloride analysis was not reported, a weight ratio of 0.5 for Na/Cl was *assumed* here to permit comparison. Different ratios are stated in the original thesis and its abstract (Rutherford 1963).

[4] Averaged(?) analyses based on data on 19 samples. Original data given as percentages of cations and of anions; recalculated assuming charge neutrality and that these data were weight percentages. Both analyses show a trace of Li. Gas analyses were also made.

[5] Recalculated from original molar ratios. Analyses 5 and 6 were made by a microchemical leaching procedure; analyses 7 and 8 were made by ball milling.

[6] Isotopic data on these samples are reported by Holser, Kaplan, and Silverman (1963).

TABLE 5.—*Analyses of fluid inclusions made on leach solutions after crushing or ball milling*

[Data for analyses are as given in original reference except where they are recalculated to 1,000 g and are rounded for uniformity. Tr., trace; ----, not reported; n.d., not detected]

Analysis No.	Reference	Material	Milligrams per kilogram of sample Na^{+1}	K^{+1}	Ca^{+2}	Mg^{+2}	Cl^{-1}	SO_4^{-2}	Li^{+1}	HCO_3^{-1}	Sample notes
INCLUSIONS FROM PEGMATITES AND QUARTZ VEINS											
1	Lisitsyn and Malinko, 1961.[1]	Quartz	10.40	8.20	30.06	3.64	26.94	20.74	--------	61.00	Quartz crystal deposit in granite; Southern Urals, U.S.S.R. Sample 574.
2	do	do	15.20	32.00	47.54	2.68	48.22	29.88	--------	20.74	Quartz crystal deposit in granite; Southern Urals, U.S.S.R. Sample 100–11.
3	do	do	68.80	20.19	39.63	26.69	87.94	46.66	--------	18.30	Quartz crystal deposit in gneiss; Southern Urals, U.S.S.R. Sample 75–83–140.
4	do	do	15.80	17.00	23.34	3.48	36.88	25.60	--------	51.24	Quartz crystal deposit in quartz porphyry; Southern Urals, U.S.S.R. Sample 601.
5	Sheshulin 1961[2]	do	64.4	--------	26.86	12.64	81.56	--------	--------	175.74	Spodumene pegmatite in Proterozoic graphitized marble; unspecified locality.
6	do	Microcline	25.8	--------	37.68	16.30	65.24	--------	--------	274.58	Do.
7	do	Spodumene	168.0	--------	33.26	15.07	138.28	Tr.	--------	384.40	Do.
8	In'shin, 1958[3]	Quartz	11.5	4	54	.27	85	15	--------	55	Outer zone of quartz crystals from vuggy vein; Astaf'evskii, Urals, U.S.S.R.
11	Roedder, 1958[4]	do	130	90	--------	--------	50	5	1.45	--------	Core of pegmatite pod in hornblende schist; Farmington Canyon, Utah, Sample 16a.[5]
12	do	do	125	155	--------	--------	151	13	.83	--------	Three-foot quartz pod in chlorite schist; Antelope Island, Utah. Sample 26.[6]
INCLUSIONS FROM VARIOUS MINERALS AND ROCKS											
16	Goguel, 1963[7]	Quartz	43	120	7	--------	109	42	--------	--------	Youngest Variscan granite; Schluchsee (Schwartzwald), West Germany.[8]
17	do	Orthoclase	--------	--------	--------	--------	256	35	--------	--------	Do.[8] [9]
18	do	Plagioclase	--------	--------	--------	--------	230	56	--------	--------	Do.[8]
19	do	Biotite	--------	--------	--------	--------	102	28	--------	--------	Do.
20	do	Quartz	53	102	8	--------	140	26	--------	--------	Pegmatite associated with youngest Variscan granite; Schluchsee (Schwartzwald), West Germany.[8]
21	do	Orthoclase	--------	--------	--------	--------	390	46	--------	--------	Do.[10]
22	do	Quartz	100	110	--------	--------	96	13	--------	--------	Youngest Variscan granite. Andlau, 1, Vosges, France.[8]
23	do	do	112	25	6	--------	160	20	--------	--------	Pegmatite, Landsverk, Evje, Norway.[8] [11]
24	do	do	40	5	1.4	--------	47	6	--------	--------	Optically clear; locality unknown.[8]
25	do	Olivine	0–6	0–5	11	--------	5–25	16–25	--------	--------	Olivine nodule in Tertiary basalt; Hoher Hagen, West Germany.[12]
26	Koptev-Dvornikov and others, 1962.	Granite	57	79	31	1.3	.8	39	--------	220	Paleozoic intrusive complex; Betpakdal, U.S.S.R. Sample 20.[13]
27	do	do	32	41	33	4.5	5.8	19	--------	192	Paleozoic intrusive complex; Betpakdal, U.S.S.R. Sample 75.[14]
28	Lamar and Shrode, 1953.[15]	Dolomite	130	40	40	260	450	280	--------	510	Paleozoic dolomites; Illinois. Average of seven samples.
29	do	Limestone	80	30	170	30	120	380	--------	140	Paleozoic limestones; Illinois. Average of 12 samples.
13	Roedder, 1958[16]	Chert	85	445	--------	--------	120	140	0.70	--------	Nodules in Paleozoic limestone; Provo Canyon, Utah. Sample 23a.
65	Evzikova and Moskalyuk, 1964.[17]	Calcite	1.08	1.41	1.96	N.d.	2.1	.42	--------	12.8	Carbonatite of Gulinsk alkalic intrusive; northern Siberian platform, U.S.S.R
66	do	Dolomite	.60	2.30	1.64	.84	1.06	.48	--------	10.63	Do.
67	Yushkin and Srebrodol'skii, 1965.[18]	Sulfur	108.5	--------	44.9	6.0	160	55.5	--------	85.4	Large primary inclusions; Shorsui sulfur deposit, U.S.S.R.
INCLUSIONS FROM VARIOUS TYPES OF MINERAL DEPOSITS											
30	Khitarov and others, 1958.[19]	Calcite	0.05	N.d.	1.44	<0.0005	0.62	N.d.	--------	3.6	Iceland spar deposit in tuff beds associated with trap rock intrusions; Markhal, Siberia, U.S.S.R. Sample 8.
31	do	do	.16	N.d.	1.04	<0.0005	1.2	N.d.	--------	2.3	Iceland spar deposit in tuff beds associated with trap rock intrusions; Dzhekinde, Siberia, U.S.S.R. Sample 7.
32	Ames, 1958[20]	Galena	8.8	3.8	--------	--------	116.1	--------	--------	--------	Crystals from vugs in Pennsylvanian limestones; Portales lead mine, Hansonburg district, near Bingham, N. Mex.[21]

TABLE 5.—*Analyses of fluid inclusions made on leach solutions after crushing or ball milling*—Continued

Analysis No.	Reference	Material	Milligrams per kilogram of sample								Sample notes
			Na^{+1}	K^{+1}	Ca^{+2}	Mg^{+2}	Cl^{-1}	SO_4^{-2}	Li^{+1}	HCO_3^{-1}	
			INCLUSIONS FROM VARIOUS TYPES OF MINERAL DEPOSITS—Continued								
33	do	Barite	5.0	2.8	--------	--------	25.4	--------	--------	--------	Do.[22]
34	do	Fluorite	1.3	15.0	--------	--------	16.8	242.1	--------	--------	Crystals from vugs in Pennsylvanian limestones (presumably later than barite); Portales lead mine, Hansonburg district, near Bingham, N. Mex.[23]
35	do	do	6.3	50.0	--------	--------	16.8	560.0	--------	--------	Deposit similar to that in analysis 34; Alamo fluorite mine, 3.5 miles northeast of Derry, N. Mex.[24]
36	Roedder and others, 1963.	Galena	7.87	.41	1.58	0.34	15.4	5.29	--------	--------	Rim of crystal; Upper Mississippi lead-zinc district, Platteville, Wis. Sample ER 59–62a.[25]
37	do	do	9.32	.49	2.45	.46	19.9	2.10	--------	--------	Core of same crystal as analysis 36; Upper Mississippi lead-zinc district, Platteville, Wis. Sample ER 59–62d.[25]
38	do	Quartz	5.0_4	1.1	1.4_7	.20	10.6	.68	--------	--------	Oscillator plate reject; Brazil. Locality details unknown. Sample ER 61–3/4.[26]
39	Kostyleva, 1964 [27]	do	40.0	2.90	1.10	3.05	70.0	.35	--------	0.325	Ore-bearing quartz; Eastern Kounrad, U.S.S.R. Sample 520.
40	do	do	23.0	6.00	.40	--------	35.0	.01	--------	2.10	Quartz-wolframite vein; Northern Kounrad, U.S.S.R. Sample 1.
41	Puzanov, 1960 [28]	do	3.35	2.24	1.57	2.07	7.8	5.76	--------	12.2	Rock crystal from skarn deposit. Soviet Far East.
42	do	do	3.69	3.58	1.48	3.93	6.4	6.99	--------	27.5	Vein quartz from skarn deposit; Soviet Far East.
43	Sushchevskaya and Barsukov, 1965.[29]	do	7.0	1.8	2.0	Tr.	8.0	Tr.	--------	24.6	White quartz, with cassiterite and arsenopyrite; sulfide-tin deposit, Yagodyni district, U.S.S.R. Sample 105.
44	do	do	14.2	9.0	4.0	Tr.	66.8	Tr.	--------	3.0	White comb quartz; sulfide-tin deposit, Preryvistyi district, U.S.S.R. Sample 573.
45	Khitarov, D., 1965b.[30]	Fluorite	6.0	36.5	274	10.8	130.5	N.d.	--------	190.6	Greisen deposit, Urals, U.S.S.R. Sample G–1.
46	Balitskii and Lyubofeyev, 1962.[31]	Rock crystal.	5.52	--------	39.06	7.29	7.44	21.60	--------	129.93	Vein 2, drift 8 of polymetallic deposit (stage 1); northwestern Caucasus, U.S.S.R.
47	do	Quartz (coarse).	45.54	--------	21.03	7.29	106.38	N.d.	--------	38.43	Polymetallic deposit of analysis 46 (stage 2).[32]
48	do	Sphalerite	Tr.	--------	24.63	8.37	9.57	7.20	--------	91.50	From sulfide-carbonate vein; crosscut 265, polymetallic deposit of analysis 46 (stage 2).
49	do	Calcite (coarse).	28.29	--------	72.12	12.75	59.55	43.23	--------	201.30	Stage 3 of polymetallic deposit of analysis 46; crosscut 501.
9	Roedder, 1958	Quartz	85	100	--------	--------	27	6	1.85	--------	Euhedral crystals in specularite tactite; Quitman Mountains, Tex. Sample "A".[4, 33]
10	do	do	32	30	--------	--------	29	22	~.1	--------	Euhedral crystals in mesothermal vein; Maxfield mine, Utah. Sample 9.[4, 5]
14	do	do	100	100	--------	--------	140.9	28.8	1.2	--------	Empire vein, Grass Valley, Calif. Sample 45.[34]
15	do	do	87	105	--------	--------	88.9	81.6	.90	--------	Wentworth vein, Empire mine, Grass Valley, Calif. Sample 48.[35]
50	Berger and Moskalyuk, 1962.[36]	do	31.2	2.4	69.0	--------	117.1	2.4	--------	73.2	Columnar recrystallized quartz vein; vein 1, upper zone, Aldan shield, U.S.S.R. Sample 146.
51	do	do	36.3	1.9	14.3	--------	63.9	3.6	--------	18.3	Rock crystal; vein 2, intermediate zone, Aldan shield, U.S.S.R. Sample 39.
52	do	do	15.4	1.4	20.9	--------	55.3	1.8	--------	9.1	Rock crystal; stockwork 59, lower zone, Aldan shield, U.S.S.R. Sample 53.
53	Moskalyuk, 1961.[37]	do	5	N.d.	15	.1	N.d.	3.6	--------	27	Honeycomb quartz from pegmatite; Keremet-tas, U.S.S.R. Sample 14.
54	do	do	120	42	28	.6	277	5	--------	N.d.	Crystal-bearing pegmatite veins; Pamir, U.S.S.R. Sample 55b.
55	do	Fluorite	16	2.0	31	N.d.	42	N.d.	--------	N.d.	Dark violet fluorite from pegmatite; Keremet-tas, U.S.S.R. Sample 87n.
56	do	do	4	Tr.	26	1.0	5	18	--------	73	Pegmatite body No. 72, Aktailyau, U.S.S.R. Sample 17z.
57	Sokolov and Zakharchenko, 1961.[38]	Quartz	33	2	24	N.d.	58	12	--------	27	Smoky crystal from albitized pegmatite; northwest Tarbagatai Range, U.S.S.R Sample 15g.

TABLE 5.—*Analyses of fluid inclusions made on leach solutions after crushing or ball milling*—Continued

Analysis No.	Reference	Material	Milligrams per kilogram of sample								Sample notes
			Na^{+1}	K^{+1}	Ca^{+2}	Mg^{+2}	Cl^{-1}	SO_4^{-2}	Li^{+1}	HCO_3^{-1}	
			INCLUSIONS FROM VARIOUS TYPES OF MINERAL DEPOSITS—Continued								
58	Zakharchenko, 1961.[30]	----do-----	34	3	154	12	60	N.d.	--------	36	Massive fine-grained quartz vein (No. 3); Baikonur River basin, Kazakhstan. U.S.S.R.
59	----do----------	----do-----	51	10	24	.5	63	N.d.	--------	55	Columnar vein quartz; Teke-Zhugurgun, Kazakhstan, U.S.S.R.
60	Arnautov and others, 1965.[40]	Rose quartz.	N.d.	N.d.	140.5	3.2	428.9	160.6	N.d.	N.d.	Fluorite-bearing chambered pegmatite; Bet-Pak-Dal, U.S.S.R. Sample IV.
61	----do----------	Green quartz.	N.d.	N.d.	217.5	N.d.	451.1	N.d.	N.d.	N.d.	Pegmatite of analysis 60. Sample V.
62	----do----------	Vein fluorite.	3.2	N.d.	--------	9.1	645.4	N.d.	N.d.	N.d.	Pegmatite of analysis 60. Sample II.
63	Ermakov, 1965b.[41]	Fluorite----	7.48	3.31	6.44	1.70	17.40	N.d.	.35	10.61	Water leach, chambered pegmatite; Bektauata intrusive, Kazakhstan, U.S.S.R.
64	----do----------	----do-----	6.79	1.57	147.9	4.00	.70	N.d.	.52	N.d.	Acid leach, chambered pegmatite of analysis 63.

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[1] Data recalculated to 1,000-gram samples for uniformity. Data on SiO_2 in analyses 1–4 were: 132.20, 62.60, 135.40, and 91.40 mg/kg, respectively. pH ranged from 6.2 to 7.8. The concentration of salts, determined by several different methods (in poor agreement), ranged from 2 to 40 weight percent.

[2] Spectrographic analysis shows $K \approx Na$. Concentrations were calculated from estimated amounts of H_2O, but they apparently contain internal inconsistencies.

[3] Original data were for a 100-gram sample. The pH of the extract was 6.5. Optical study shows the presence of liquid CO_2 and of halite daughter crystals. Many samples emitted the odor of H_2S when broken. SiO_2 was 60 mg/kg; R_2O_3 was not found. Additional elements by spectrography were: Al, 0.01–0.1; Fe, 0.01–0.1; Mn, 0.001–0.01; Ti, 0.001; Cu, 0.001–0.01; Sr, 0.01–0.1; Ba, 0.1–1.0.

[4] Cs was $\leq$0.1 mg/kg in analyses 11 and 12.

[5] Rb, $\sim$0.1 mg/kg.

[6] Rb, 0.45 mg/kg.

[7] Gas analyses for the same samples are given in table 2. Some of the chloride values may be too high by as much as 50 ppm (Reiner Goguel, written commun. 1963). All chloride analyses are actually (Cl+Br+I).

[8] PO_4, 0 mg/kg.

[9] BO_3, 30 mg/kg.

[10] PO_4, 30 mg/kg.

[11] BO_3, 20 mg/kg.

[12] PO_4, 20 mg/kg.

[13] F, 6.8 mg/kg; gaseous CO_2, 260 mg/kg.

[14] F, 11.3 mg/kg, gaseous CO_2, 450 mg/kg.

[15] Analyses 28 and 29 include some salts from connected intergranular pores in the rocks, as well as sealed inclusions.

[16] Rb, 1.23 mg/kg; Cs, 0.2 mg/kg. Contamination may be responsible for part of the potassium that was found.

[17] In analyses 65 and 66, values for Sr^{+2} were 0.74 and 0.14 mg/kg, respectively; F^{-1} ("Fe^{-1}") was not detected in either analysis; CO_3^{-2}, ----, 4.80 mg/kg; pH, 7.1, 9.3. Some inclusions contain 0.01 percent organic carbon.

[18] Extraction was made on 200 g. The values given here are rounded from the original which gives up to seven significant numbers. H_2S odor was detected on grinding. The pH of the liquid inclusions themselves was 7–7.5. Na is the sum of Na+K. Fe total was 0.28. From an estimate of the inclusion volume the authors calculate a value of about 6 percent solids in solution in the inclusion fluid, but the data given seem to indicate only about 4 percent.

[19] Data are milligrams found in unspecified sample weight, corrected for solution of calcite on leaching. The value for Mg^{+2} ($<$0.0005 mg in each) is incorrectly stated as "$>$0.005 mg" in the English translation.

[20] Analyses were originally stated as "ions in parts per million per kilogram of sample" (Ames, 1958, table 2, p. 478); presumably milligrams per kilogram of sample is meant. Spectrographic determinations for Ca, Al, Mg, and Cu, presumably in parts per million of the dried residue from the electrodialysis (see Ames, 1958, p. 478), in analyses 32–35, respectively, are: Ca, tr., tr., ----, ----; Al, 100.0, 150.0, 100.0, 80.0; Mg, 70.0, 60.0, 60.0, 50.0; and Cu, 50.0, 100.0, 7.0, 6.0.

[21] Li, 0.1 mg/kg; Rb, tr.; Cs, tr.

[22] Li, 0.1 mg/kg; Rb, tr.; Cs, tr.; H_2O (weight loss at 700°C), 0.18 percent of sample.

[23] Li, tr.; Rb, tr.; Cs, tr.; H_2O (weight loss at 700°C), 0.17 percent of sample.

[24] Li, 0.1 mg/kg; Rb, tr.; Cs, tr.; H_2O (weight loss at 700°C), 0.19 percent of sample.

[25] B, 0.003 mg/kg.

[26] B, 0.05- mg/kg. Numbers in subscript are uncertain.

[27] Ultramicrochemical analyses on 20 grams, by I.N. Maslova, recalculated to 1,000 grams. Analyses 39 and 40 also show SiO_2, 0.03 and 0.01 mg, respectively.

[28] A sample weight of 100 grams is assumed. The pH for both solutions (analyses 41 and 42) was 7.3, and each had 15 mg SiO_2.

[29] The authors made direct determinations of the water contents, but they state only (p. 225, footnote) that these varied from 0.3 to 1 percent and averaged 0.61 percent. They assume 0.61 percent is correct. To permit comparison of the original leachate values with other data in table 5, the samples are assumed to have been 50 grams, and the leach volumes are assumed to have been 100 ml. B = tr. in both analyses. F was not determined in sample 105 (analysis 43) and was 1.2 mg/kg in sample 573 (analysis 44). Both primary and secondary inclusions were found in samples.

[30] Recalculated from data given as milliequivalents, assuming an 80-gram sample was used. Includes 162.5 mg F and considerable silica.

[31] Originally given as mg/l of water extract, from an unspecified size of sample, at a solid-liquid ratio of 1:3. Several samples show a large excess of anions over cations. Na is the sum of Na+K. Leach solution pH determinations, in analyses 46–49, respectively, are 6.9, 6.2, 6.9, and 7.2.

[32] Spectrographic analysis of this quartz show the following maxima (in percent): Pb, 0.03; Zn, 0.03; Cu, 0.01; Fe, 0.03; Ag, 0.003; Ba, 0.03; Ti, 0.01; Co, 0.003; and Mn, 0.03. All of these metals, except Ti and Co, are either absent or present only in very small traces in pure quartz powder after water leaching. Stages 1 and 3 showed much lower metal contents.

[33] Rb, 0.21 mg/kg.

[34] Rb, 0.45 mg/kg; Cs, $<$0.05 mg/kg.

[35] Rb, 0.55 mg/kg; Cs, 3.2 mg/kg.

[36] Values, in analyses 50–52, respectively, are: Sr^{+2}, 3.0 mg/kg, 0.3 mg/kg, tr.; Ba^{+2}, tr., n.d., tr.; SiO_2, 6.0 mg/kg, n.d., tr.; F^{-1}, 2.2 mg/kg, 2.2 mg/kg, n.d.; pH, 7.2, 6.2, 6.4. Li, Rb, and Cs were detected. Fluorite and gypsum occur in some veins. Data originally obtained on 150-gram samples.

[37] Values, in analyses 53–56, respectively are: SiO_2, 3 mg/kg, ----, n.d., 6 mg/kg; F^{-1}, 0.3 mg/kg, n.d., 27 mg/kg, 9 mg/kg, pH, 6.6, 6.5, 6.1, 7.2. Analysis 56 is also quoted in a later paper by Zakharchenko and Trufanov (1964), with different values for Ca (1.0 mg/kg) and Mg (4.0 mg/kg).

[38] Fe^{+2}, Fe^{+3}, Al^{+3}, and B^{+3} were not determined; SiO_2, 14 mg/kg; F^{-1}, 1 mg/kg; pH, 6.58.

[39] In analyses 58 and 59, SiO_2 was 21 and 30 mg/kg, respectively; pH, 6.0 and 6.8.

[40] Recalculated from analyses made on 50- to 100-g samples. Alkalies by flame photometer; other cations by multichannel photoelectric quantometer. CO_3^{-2} was not detected. In analyses 60–62, in miligrams per kilogram, respectively: Si, ----, ----, 68.7; Mn, 16.1, 2.8, 21.5; Al, 16.1, 0.5, 21.4; Fe, 84.3, 17.4, 69.8; Ni, 1.6, 1.6, 1.1; Ti, 2.4, n.d., 5.4; F, 2.1, 2.7, ----. The samples were boiled in aqua regia and then washed in doubly distilled water before grinding, sieving, and leaching (on steambath). The final grinding was to a particle size of "0.1μ". The rose quartz is colored by hematite flakes; the green quartz (deposited on the rose) is colored by chlorite flakes.

[41] Inclusions in these samples show a large volume of daughter minerals. More than 13 different solid phases in each include halite, sylvite, hematite, chlorite, albite, quartz, rutile, zircon, muscovite, staurolite, sphene, magnetite, and several unidentified opaque phases. The two analyses (63 and 64) are of leaches made with water at room temperature, and with 0.1 percent HNO_3 at 90° to 95°C, apparently in sequence on the same 115-gram sample. Data recalculated to 1 kilogram. In analyses 63 and 64, respectively: Fe^{+3}, n.d., 0.35; SiO_2 1.04, 1.39; BO_3^{-3} 1.74, not determined; F^{-1}2.96, 139.2; pH 5.5, ----. Mn^{+2}, Al^{+3}, SO_4^{-2}, CO_3^{-2}, and SiF_6^{-2} were not detected. Semiquantitative spectrographic analyses of the dry residues showed Al, Ba, Ca, Cr, Cu, Fe, La, Mg, Mn, Na, Ni, Pb, Si, Ti, and Zn; Zr was not detected in either leach.

TABLE 6.—*Quantitative analyses of inclusion fluids*

[Data for analyses are as given in original reference except where they are recalculated, and are rounded for uniformity. N.d., not detected; ----, not reported; tr., trace]

Analysis No.	Reference	Mineral	Parts per million of total inclusion fluid									Isotopic ratio (D/H)	Sample notes
			Na^{+1}	K^{+1}	Ca^{+2}	Mg^{+2}	Cl^{-1}	SO_4^{-2}	B	HCO_3^{-1}	Concentration		
INCLUSION FLUIDS FROM IGNEOUS ROCKS, PEGMATITES, AND QUARTZ VEINS													
1	Königsberger and Müller, 1906a.	Quartz	20,000	7,000	3,000	----------	16,000	5,000	----------	----------	71,000	----------	Alpine vein quartz; Bächistock, Switzerland.[1]
2	Königsberger and Müller 1906b.	do	25,000	15,000	3,000	----------	15,000	7,000	----------	----------	100,000	----------	Alpine vein quartz; Aarmassif.[2]
3	Faber, 1941 [3]	Granite	44,500	18,800	Tr.	----------	30,100	75,600	----------	----------	169,000	----------	Precambrian Rönne granite; Bornholm, Denmark.
4	do	do	30,900	42,300	5,900	----------	110,700	13,800	----------	----------	203,600	----------	Precambrian Vang granite; Bornholm, Denmark.
5	do	do	26,600	17,900	3,400	----------	22,800	55,500	----------	----------	126,200	----------	Paleozoic granite; St. Breward, Cornwall, England.
6	do	do	21,400	13,800	2,500	----------	50,700	11,000	----------	----------	99,400	----------	Paleozoic granite; Princeton, Devon, England.
7	Polykovskii, 1962a [4]	Quartz	66,300	Tr.	13,700	----------	101,000	5,300	N.d.	18,900	199,170	----------	Core (earliest) quartz, "chambered" pegmatite; western Tien-Shan, U.S.S.R. (central Asia).
8	do	do	56,500	4,350	10,100	300	107,000	17,400	N.d.	----------	186,900	----------	Honeycomb quartz; homogenization temperature, 480° to 510°C. Same locality as in analysis 7. Sample 2.
9	do	do	48,800	1,050	12,640	Tr.	84,000	1,050	N.d.	24,400	171,070	----------	Honeycomb quartz of analysis 8. Sample 3.
10	do	do	23,900	180	19,700	----------	41,300	280	N.d.	50,000	127,560	----------	Smoky quartz; homogenization temperature, 330° to 460°C. Same locality as in analysis 7. Sample 4
11	do	do	2,750	Tr.	1,850	30	2,100	820	N.d.	3,150	11,390	----------	Outer amethyst zone; homogenization temperature, 80° to 150°C. Same locality as analysis 7. Sample 5.
12	Maslova, 1961	do	23,300	1,150	310	----------	36,140	----------	----------	265	61,200	----------	Pegmatite; Volodar-Volynia, U.S.S.R.[5]
12a	Prikazchikov and others, 1964.[6]	do	92.8	----------	81.0	2.7	17.7	75.0	----------	244	513	----------	Giant (7 tons) quartz crystal in pegmatite; Volynia, U.S.S.R. Sample III.
INCLUSION FLUIDS FROM SALINE MINERALS AND BRINE													
13	Kramer, 1965 [7]	Halite	69,100	3,300	13,700	1,660	110,000	44,800	----------	----------	242,600	----------	Salina salt beds (Silurian); Goderich, Ontario.[8]
14	do	do	88,200	4,700	16,400	2,050	145,000	38,100	----------	----------	294,500	----------	Salina salt beds (Silurian); Cleveland, Ohio.[9]
15	Sjögren, 1893	Gypsum	11,980	680	1,140	920	18,830	6,680	----------	----------	40,230	----------	Large crystals associated with sulfur deposits, Cianciana, Girgenti, Sicily.[10]
15a	Petrichenko and Shaydetskaya, 1968.[11]	Halite	60,500	9,700	1,040	13,000	142,000	1,100	36	1,370	230,000	----------	Large inclusions in clear, recrystallized halite; Donbass, U.S.S.R.
16	White, 1965a and 1965b.[12]	Brine	51,000	25,000	40,000	730(?)	185,000	56	520	----------	309,877	----------	Strong brine from 5,232-ft depth at temperature of 270° to 370°C; Niland, southern California.
INCLUSION FLUIDS FROM LAVA FLOWS (LARGE VESICLE FILLINGS)													
17	Sugawara and others, 1944.	Basalt	511	54.7	92.0	14.0	869	167	----------	----------	[13] 1,842	(14)	Amygdaloidal nepheline basalt; Nagahama, Hamada-si, Simane Prefecture, Japan.[15]
18	Iwasaki and others, 1956.	do	30.2	29.0	126	.0	10.1	4.2	----------	----------	[16] 280	----------	Amygdaloidal trachyandesitic basalt; Imari district, Saga, Japan.[17]

19	Kokubu and others, 1957.	----do-----	115	53.8	15.5	5.1	170	66	----------	----------	433	----------	Amygdaloidal olivine blasat Imazu, Hukuoka, Japan.[18]
20	Zolotukhin, 1959-----	Dacite-----	3.00	----------	32.00	8.51	5.31	13.16	N.d.	122.00	184	----------	Cavity in intrusive olivine dacite; Tisen quarry, near Vinogradov, U.S.S.R. Sample 2.[19]

INCLUSION FLUIDS FROM MINERAL DEPOSITS

21	Maslova, 1963[20]------	Quartz-----	1,300	380	N.d.	N.d.	660	N.d.	----------	1,510	3,850	----------	Antimony-mercury deposit in the Far East (Pacific region), U.S.S.R. Sample 1.[21]
22	----do--------------	----do-----	140	40	N.d.	N.d.	40	N.d.	----------	320	540	----------	Antimony-mercury deposit of analysis 21. Sample 2.[22]
23	Fedorchuk, 1963[23]----	Quartz-----	1310–1320	90–120	----------	----------	580–620	----------	----------	1470–1510	3470–3540	----------	Quartz containing cinnabar, from "concordant type" deposit, central Asia mercury-antimony province, U.S.S.R.
24	Khitarov and others, 1958.[24]	Calcite-----	8,200	N.d.	42,000	----------	83,600	----------	----------	2,100	135,900	----------	Iceland spar deposit in pillow lavas; Gonchak, Siberian shield, U.S.S.R. Sample 1.[25][26]
25	----do--------------	----do-----	830	N.d.	70,400	----------	158,800	----------	----------	N.d.	230,000	----------	Iceland spar deposit of analysis 24. Sample 2.[25][27]
26	----do--------------	----do-----	12,500	----------	13,900	----------	47,700	----------	----------	N.d.	74,000	----------	Iceland spar deposit in pillow lavas; Nidym, Siberian shield, U.S.S.R. Sample 3.[28]
27	Skropyshev, 1957-----	----do-----	15,427	6,333	57,209	N.d.	65,890	15,600	----------	120,290	290,995	----------	Unspecified Iceland spar deposit. Sample 1–50.[29]
28	Grushkin and Prikhid'ko, 1952, and Grushkin, 1958.	Fluorite (early dark violet).	21,000	23,000	[30] 4,000	----------	53,000	N.d.	----------	----------	[31] 106,200	----------	Hypogene fluorite replacing limestone, Aurakhmat, central Asia.[32]
29	----do--------------	Fluorite (violet).	25,000	3,000	[30] 3,000	3,000	49,000	N.d.	----------	5,100	[31] 89,500	----------	Do.[33]
30	----do--------------	Fluorite (late green).	7,300	1,400	[30] 7,300	----------	4,000	N.d.	----------	32,000	[31] 53,900	----------	Do.[33]
31	Roedder and others, 1963.	Sphalerite--	57,100	2,700	18,000	2,400	124,600	<3,300	30	----------	208,130	+3.5	Sphalerite-galena Mississippi Valley-type deposit; Tri-State district, Oklahoma. Sample ER61-1.
32	----do--------------	----do-----	48,000	2,400	13,000	<1,300	91,000	<19,000	500	----------	175,000	+1.4	Replacement zinc deposit in limestone; Santander, Spain. USNM C-437.
33	----do--------------	----do-----	25,000	4,600	15,000	2,100	63,600	<6,400	100	----------	116,800	+.5	Au-Ag-Cu deposit in volcanic rocks; Ani Ugo, Japan. USNM 18557.
34	----do--------------	----do-----	7,200	6,500	10,400	3,600	17,600	<18,000	<360	----------	63,660	−4.6	Pb-Zn-Ag deposit in volcanic rocks; Rico, Colo. USNM R552a.
35	----do--------------	----do-----	71,600	3,100	17,000	2,800	129,600	<3,000	<80	----------	227,000	+.9	Sphalerite-galena Mississippi Valley-type deposit?; Cartagena, Spain, USNM 18309c–d. (See text.)
36	----do--------------	Quartz-----	470	110	60	<100	630	630	----------	----------	2,000	−5.2	Barren quartz vein in rhyolite; Oatman, Ariz. Sample H-1.
37	----do--------------	Calcite-----	120	30	<1,100	280	<140	670	<3	----------	2,300	----------	Barren calcite vein in latite; Chihuahua, Mexico. Sample F-1.
38	Hall and Friedman, 1963.	Fluorite (yellow).	45,200	2,400	6,000	2,100	84,000	3,400	----------	----------	143,100	[34] +3.2	Fluorite and sphalerite, as vuggy bedded replacements; Cave-in-Rock district, Illinois.[35][36]
39	----do--------------	Fluorite (early purple).	41,000	3,200	4,400	6,100	78,000	5,900	----------	----------	138,600	[34] +2.3	Do.[35][36]
40	----do--------------	Quartz-----	17,300	4,300	8,400	2,800	47,000	12,900	----------	----------	92,700	[34] −.2	Do.
41	----do--------------	Galena-----	55,400	3,100	20,600	4,000	115,000	([37])	----------	----------	>198,100	[34] +1.8	Do.
42	----do--------------	Sphalerite--	53,400	2,500	20,400	2,200	120,000	1,000	40	----------	199,540	[34] +4.0	Sphalerite-galena Mississippi Valley-type deposit; Amelia mine, upper Mississippi Zn-Pb district.
43	----do--------------	Galena-----	39,000	3,200	18,500	3,400	83,000	N.d.	100	----------	+147,200	[34] +2.7	Sphalerite-galena Mississippi Valley-type deposit (later than sphalerite); same locality as analysis 42.
44	----do--------------	Calcite (stage 3).	16,800	600	1,600	1,200	21,400	3,600	40	----------	45,200	[34] −.1	Sphalerite-galena Mississippi Valley-type deposit (later than galena); same locality as analysis 42.[36]

TABLE 6.—*Quantitative analyses of inclusion fluids*—Continued

Analysis No.	Reference	Mineral	Parts per million of total inclusion fluid									Isotopic ratio (D/H)	Sample notes
			Na^{+1}	K^{+1}	Ca^{+2}	Mg^{+2}	Cl^{-1}	SO_4^{-2}	B	HCO_3^{-1}	Concentration		
			INCLUSION FLUIDS FROM MINERAL DEPOSITS—Continued										
45	Uchameyshvili, 1965.[38]	Barite	17,900	1,000	6,300	----------	42,900	4,300	----------	1,900	83,200	----------	Kopet-Dag barite area, Arpaklen deposit, U.S.S.R. Sample 3.
46	do	Barite-calcite.	2,400	200	14,600	----------	18,400	17,200	----------	24,500	76,300	----------	Upper Racha, Georgian Soviet Socialist Republic, Lesora section, Chorda, U.S.S.R. Sample 22.
47	do	Barite with galena.	6,800	500	5,700	----------	13,400	11,000	----------	4,000	45,900	----------	Upper Racha, Georgian Soviet Socialist Republic, upper Gvalvana section, Chorda, U.S.S.R. Sample 25.
48	do	Barite	9,900	30	38,200	----------	88,200	33,600	----------	N.d.	173,700	----------	Center of vein 11; Okureshi deposit, western Georgia, U.S.S.R.
49	do	Barite polymetallic ore.	8,200	100	42,500	----------	88,800	35,800	----------	29,800	210,200	----------	Margin of vein 12; same locality as analysis 48.

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[1] Free CO_2, 95,000 ppm; CO_3^{-2}, 18,000 ppm; Li, 2,000 ppm. The alkalies and calcium were verified by spectrograph.

[2] Free CO_2, 50,000 ppm; CO_3^{-2}, 35,000 ppm. K value includes Li.

[3] Recalculated. Water determinations, on which all concentrations are based, were made on separate samples. The alkalies given are those equivalent to the Cl^{-1} and SO_4^{-2} found; all extra alkali (about two to four times as much) was assumed by Faber to be from the leaching of feldspar surfaces.

[4] The water determinations, on which all concentrations are based, were *estimated* optically. Other determinations in analyses 7 through 11, respectively, are: Fe^{+3}, ----, ----, ----, ----, tr.; F^{-1}, ----, 2,900 ppm, ----, 5,050 ppm, tr.; $HSiO_3^{-1}$, 12,600 ppm, ----, 2,630 ppm, 8,720 ppm, tr.; pH, 6.64, 6.35, 6.70, 7.3, 6.99. Fe^{+2} and Al^{+3} were not found. It is not known whether the determinations marked by dashes are "not reported" or are "not detected." Crystals of NaCl were seen in some of the inclusions. The gases evolved—highest values in samples 2 and 3 (analyses 8 and 9)—included CO_2, N_2, H_2, Ar, He, CH_4, and no heavy hydrocarbons. See also footnote 65, table 3.

[5] Inclusion volume, 100 mm^3.

[6] There is some ambiguity in the original tabulation, presented in "mg per 100 ml of solution," in that 8 ml of liquid from the very large inclusion was diluted to 40 ml before analysis. The data given here assume the "mg per 100 ml of solution" refers to the original inclusion fluid. "Na" is Na+K; it was listed as 2.8, but the summation indicates a misprint. SiO_2, tr.; pH, 6.9, Sr, 0.18; Ba, 0.3; Li, 0.5; Rb, 0.01, by flamephotometer. Spectroscopy on residue showed Si, Al, Mg, Ca, Fe, and Zr, with traces of Mn and Ti.

[7] Unpublished analyses, corrected for leach blank. Na and Cl were determined, even though the entire sample was dissolved, by calculation from the values for K, SO_4, and F, and the assumption of a sea water composition; the requirement of electroneutrality provided a check (Kramer, 1965; oral and written commun., 1963, 1965).

[8] Average of four analyses. Includes F, 58 ppm.

[9] Includes F, 63 ppm.

[10] A single large (3 cm^3) inclusion in transparent gypsum crystals up to 30 cm long. A moderate but unmeasured amount of H_2S was evolved on opening the inclusion. The pH was about neutral.

[11] Data recalculated from the original in grams per liter, using measured density of 1.231. Li, 45 ppm; Rb, 2 ppm; Cs, Mn^{+2}, Fe^{+3}, Al^{+3}, CO_3, F, and SiF_6^{-2} were not found. pH (determined by three methods), 4.95–6.2; Eh, −10 to −130 mV (average, −70 mV). Semiquantitative spectrographic analysis also gave Mn, 0.005 ppm; Fe, 0.005 ppm; Ti, 0.008 ppm; La, 0.005 ppm; Al, 0.01 ppm; V was not found.

[12] Data included here for comparison. Also includes, in parts per million, SiO_2, >110(?); Al, 450; Fe, 3,200; Mn, 2,000; As, 15; Pb, 104; Zn, 970; Sr, 750; Ba, 200; Cu, 10; Ag, 1; Li, 300; Rb, 169; Cs, 20; NH_4, 482; F, 18; Br, 146; I, 22; NO_3, 35; H_2S, ~1; Sb, 0.5; Sn, 0.65; Hg, 0.008; Ni, tr.; Cr, tr.; V, W, HCO_3, S_2O_3, NO_2, and PO_4 were not detected. Original pH probably 5 to 6; density at 20°C, 1.264 g/ml. Concentration of salts was obtained by evaporation at 180°C. Includes some data from White, Anderson, and Grubbs, (1963).

[13] This is residue on evaporation; analysis total 1,792 ppm.

[14] 0.8γ heavier than Tokyo city water.

[15] NO_3, 4.4; NO_2, 0.69; SiO_3, 24.6; NH_3, 0.13; Al, 5.4; Fe, 2.18; pH, 8.1.

[16] This is residue on evaporation; analysis total 202 ppm.

[17] NO_3, 0.033; NO_2, 0.005; NH_4, 0.63 (also given as 0.8 in table 5 of Iwasaki and others, 1956); Fe, 0.67; Al, <0.6; pH, 7.0. See also Kokubu and Katsura (1956).

[18] In parts per million: NO_3, 0.48; NO_2, 2.4; SiO_3, 1.4; P, 0.01; F, 0.8; NH_4, 0.1; Al, 0.05; and Fe, 2.2. pH was 5.8.

[19] "Na+K" only in original; pH, 7.0.

[20] The pH was found to be 6–6.5 in each inclusion, presumably by colorimetry (Maslova, 1961). Qualitative analysis showed no Ca, Mg, Al, Fe, F, CO_3, SO_4, or SiO_2. Sample 1 (analysis 21) contained dolomite and(?) bitumen on the surface of the bubbles. It also shows an unexplained large excess of cations.

[21] Author's average of four analyses of inclusions of total volumes, in cubic millimeters: 20, 2.5, 2.2+2.5, and 25. This is presumably the same analysis as reported by Fedorchuk (1963), although there are significant differences in the data given. Fedorchuk, Kostvleva-Labuntseva, and Maslova and others, (1963) list the same analyses.

[22] Inclusion volume, 35 mm^3.

[23] Analysis "by Maslova." See analyses 21 and 22, by Maslova (1963). See also footnotes 24 and 25 to table 3.

[24] Recalculated. These analyses are also given by Kiyevlenko (1958). No free CO_2 was found. All analyses were corrected for solution of calcite during leach.

[25] Two crystals from same deposit. pH was measured to be 5–5.5, using the "universal indicator."

[26] Volume of inclusion 89 mm^3. (Water was determined and the volume of the cavity was measured.)

[27] Volume of inclusion 63 mm^3. (Water was determined and the volume of the cavity was measured.)

[28] Volume of inclusion 7 mm^3 (water estimated from assumed 90 percent filling and measured volume of cavity).

[29] SiO_2, 7,346 ppm; Al^{+3}, 2,900 ppm. (Note: The volume of the inclusions was only estimated. No correction was made for leaching of the calcite, although the leach volume was 1600 times the inclusion volume. The second "control" leach, which was added to the first for the calculations in the original, showed amounts of Ca, K, SO_4, SiO_2, and Al_2O_3 nearly equal or greater than the first leach.) Spectrographic determination showed also Mg, 0.001–0.01 ppm; Ti, 0.001–0.01 ppm; Sr, ~0.1 ppm; Fe, small traces. The original statements of concentration (386 g/l on p. 321 and 410 g/l in table 6) are in disagreement, but they apparently refer to grams per liter of water in the inclusion fluid. Hence, the 410 g/l has been converted to 29.1 weight percent salts, with an assumed liquid density of 1.4, for purposes of comparison here.

[30] Corrected for solution of crushed host mineral.

[31] Recalculated from data of tables 4 and 6 in the original 1952 references. H_2O determinations were made on separate samples. The concentrations are recalculated, as the "concentrations, in percent" of Grushkin and Prikhid'ko (1952, table 6) are apparently grams per 100 grams water.

[32] F, 4,000 ppm; SiO_2, CO_3, NO_3, R_2O_3, and Fe^{+2} were not detected.

[33] F, 1,400 ppm; SiO_2, CO_3, NO_3, R_2O_3, and Fe^{+2} were not detected.

[34] D/H data scaled from graphical plots in Hall and Friedman (1963, figs. 3 and 4).

[35] About 0.02 weight percent of fluid was found in sample.

[36] Corrections were made to the calcium values for solution of crushed host mineral on leaching (W. E. Hall, oral commun., 1963).

[37] Sulfate was not determined but is probably high in view of the deficiency of analyzed anions.

[38] Sr determinations in analyses 45–49 (in parts per million), respectively: 8,900 ,n.d., 4,600, 3,800, and 5,000. CO_2 was very low or was not detected. H_2O (percent of sample evolved at 1,200°C): 0.19, 0.28, 0.15, 0.11, 0.16. "Less than 20 percent" secondary inclusions were seen in the samples analyzed (p. 136).

TABLE 7.—*Recorded fluid inclusion homogenization temperatures of 500°C or higher*

[The numerous reports of peaks at 500°C, and higher, on decrepigrams are not included, nor are homogenization determinations on glass inclusions]

Mineral and locality	Maximum temperature or range (°C)	Reference
SKARN ORE DEPOSITS		
Hedenbergite; Maikhura deposit, U.S.S.R.	550–600	Rakhmanov, 1963, p. 69; 1965, p. 274.
Calcite from skarn; Lebedinoye uranium-gold deposit, U.S.S.R.	590	Tugarinov and Naumov, 1969.
Various minerals from tin-tungsten greisens in the Far East, U.S.S.R.	650	Govorov and others, 1968.
Monticellite; Lespromkhozny deposit, U.S.S.R.	890	Sinyakov, 1967, 1968.
Quartz, from datolite-bearing rock associated with a lead-zinc deposit in the Far East, U.S.S.R.	>540	Ermakov, 1950a, p. 227 in translation.
Fluorite and quartz; Tyrny-Auz skarn ore complex, U.S.S.R.	510–575	Lesnyak, 1957a, 1961, 1965.
Tourmaline and axinite from contact zones of small intrusions in U.S.S.R.	602–625	Rodzyanko, 1967.
OTHER ORE DEPOSITS		
Quartz veins with molybdenite (multiphase inclusions); Bingham porphyry copper deposit, Utah.	650–725	Roedder, 1971.
Cassiterite; Cornwall, England	600	Little, 1955.
Cassiterite and quartz; Bolivia	530	Kelly and Turneaure, 1969.
Greisen minerals from a molybdenum-tungsten deposit, U.S.S.R.	600–700	Shcherba and others, 1964.
Quartz from cassiterite-sulfide ore deposit, Mount Bischoff, Tasmania.	530	Groves and Solomon, 1969.
Anhydrite from chalcopyrite ores of Noril'sk deposit, U.S.S.R.	>500	Zolotukhin, 1962.
Quartz and albite of granite and quartz-feldspar bodies of Sorskii Cu-Mo-deposits, U.S.S.R.	520, 730, and 800	Berzina and Sotnikov, 1965, 1968.
Garnet; Kurusaisk ore field, U.S.S.R.	500–730	Sazonov, 1962.
Quartz; Krasnoyarovo-Zolinsk orefield, U.S.S.R.	530	Grigorchuk and others, 1964.
IGNEOUS ROCKS		
Nepheline (normal-appearing, liquid-plus-gas inclusions); Botogol nepheline syenite, U.S.S.R.	840	Kerkis and Kostyuk, 1963; Kerkis, 1966.
Do	850	Bazarova, 1965.
Nepheline; Bambuisk alkalic intrusion	>850	Litvinovskii, 1967.
Do	680–850	Kostyuk, and Bazarova, 1965.
Nepheline, aegirite; Lovozero alkalic massif, U.S.S.R.	700–850	Bazarova and Feigin, 1966.
Multiphase inclusions in granitoid rocks, North Caucasus, U.S.S.R.	570	Lesnyak, 1955.
Nepheline syenite of the Borgoi massif, Buryat-Mongol, U.S.S.R.	500–750	Panina, 1966b.
Pseudoleucite; Synnyr alkalic massif, U.S.S.R.	750–>910	Panina and Lysakov, 1968.
Chkalovite (Na_2BeSiO_4), and nepheline, from alkalic complex, Ilímaussaq, Greenland.	1,040	Sobolev and others, 1970; and H. Sørensen, oral commun., 1968.
Quartz from granite; central Kazakhstan, U.S.S.R.	900	Bazarov, 1968b.
Quartz from granite and pegmatites (heated under external pressure); U.S.S.R.	850–900	Zakharchenko, 1968.
Leucocratic granites of eastern Transbaikal, U.S.S.R.	860	Vartanova and Shcherbakova, 1964.
Various magmatic deposits	800	Lesnyak, 1964 p. 207–208.
Apatite (Inclusions contain large amounts of crystals.); Khibina and Lovozero alkalic massifs, U.S.S.R.	890	Valyashko and Kogarko, 1966; Valyashko, and others, 1968.
Apatite; various sources, U.S.S.R.	630	Bazarov, 1966a.
Nepheline, feldspar, sodalite, cancrinite, tremolite, and scapolite; Synnyr alkalic massif, northern Transbaikal, U.S.S.R.	630–950	Panina, 1966a.
Quartz in ejected granitic blocks; Ascension Island	590–612	Roedder and Coombs, 1967; Roedder, 1970b.
QUARTZ VEINS AND PEGMATITES		
Quartz crystals; metamorphic veins of Kurumkan deposit, U.S.S.R.	500	Laz'ko, 1957b.
Quartz crystals; metamorphic veins of Barsukchi deposit, U.S.S.R.	515	Piznyur, 1957.
Quartz, pegmatitic (multiphase inclusions)	>500	Dolgov, 1957a.
Quartz, pegmatitic	580–620	Dolgov, 1964a.

TABLE 7.—*Recorded fluid inclusion homogenization temperatures of 500°C or higher*—Continued

Mineral and locality	Maximum temperature or range (°C)	Reference
QUARTZ VEINS AND PEGMATITES—Continued		
Quartz, zoned pegmatites of western Tien-Shan, U.S.S.R.	510–520–530	Polykovskii, 1962a, 1963, 1965.
Quartz from graphic pegmatites	500	Bakumenko, 1963.
Quartz and spodumene from pegmatites (homogenization in gas phase).	550–650	Sheshulin, 1961.
Primary minerals of pegmatites from Akzhailyau massif, U.S.S.R.	500	Trufanov, 1962.
Do	600	Trufanov, 1967.
Giant quartz crystals, Akzhailyau pegmatites, U.S.S.R.	600	Zakharchenko and Trufanov, 1964.
Quartz-fluorite pegmatites, Kazakhstan	780	Shugurova, 1967b.
Quartz-fluorite veins, greisens, and pegmatites; Kent massif, Kazakhstan.	>600	Zakharchenko and others, 1966.
Quartz, pegmatitic; Volynia, U.S.S.R.	510, 600	Moskalyuk, 1961.
Various chambered fluorite-bearing pegmatite and granite minerals (14 samples); Bet-Pak-Dal, etc., U.S.S.R.	500–860	Sobolev and others, 1964.
Quartz from fluorite-bearing pegmatite; Bet-Pak-Dal, U.S.S.R.	570–780	Bazarov, 1965a.
Quartz crystals, metamorphic veins	520–550	Venediktov, 1958a.
Quartz; pegmatites of central Kazakhstan, U.S.S.R.	510–620	Balitskii and others, 1966.
Do	880	Bazarov, 1968a.
Quartz; pegmatites of Kazakhstan and Zabaykal'ye, U.S.S.R.	>600	Bakumenko, 1968.
Quartz; pegmatites of Tarbagatai Range U.S.S.R.	500	Sokolov and Zakharchenko, 1961.
Quartz, pegmatitic; Volynia, U.S.S.R.	540–600	Ermakov, Kalyuzhnyi, and Myaz, 1957, p. 499 in translation.
Do	500–600	Kalyuzhnyi and others, 1966.
Multiphase inclusions in pegmatitic quartz; Volynia, U.S.S.R.	>500	Lyakhov, 1966.
Do	500–570	Motorina, 1967.
Crystal-gas inclusions in honeycomb quartz from pegmatites; Volynia, U.S.S.R.	650	Ermakov, 1965b.
Honeycomb quartz from pegmatites; Volodarsk-Volynsk, U.S.S.R.	>573	Lazarenko, 1967.
Quartz, pegmatitic; Maidantal, U.S.S.R.	620	Elinson and Polykovskii, 1961a, 1961b.
Honeycomb quartz; Maidantal pegmatites, U.S.S.R.	540	Polykovskii and Roizenman, 1965, p. 70.
Quartz; Pamir, U.S.S.R.	>550	Zakharchenko, 1955.
Quartz, topaz-bearing pegmatite; Korostenj, U.S.S.R.	500	Ivantishin, 1955.
Quartz(?) of granite pegmatites; U.S.S.R.	650–700	Khitarov, N. I., 1965.
Topaz; Bayan-Aul pegmatites, U.S.S.R.	500	Bakumenko, 1966.
Topaz, Il'men Mountains, U.S.S.R.	580	Ermakov, 1950a, p. 291 in translation.
Topaz, pegmatitic; U.S.S.R.	>500	Lemmlein and Kliya, 1952a.
Do	>700	Lemmlein and others, 1962.
Kyanite from pegmatite	>575–600	Sobolev and Bazarova, 1963.
Beryl; pegmatites of Connecticut	600	Cameron and others, 1953, p. 252.
Beryl; pegmatites of Black Hills, S. Dak.	515	Weis, 1953.
Beryl, pegmatitic (CO_2-bearing inclusions)	>750	Feklichev, 1962.
Quartz from pegmatites of central Kazakhstan	600	Trufanov, 1966.
Quartz from pegmatites of the "Zanorysh" type, U.S.S.R.	550	Kalyuzhnyi and Voznyak, 1967.

TABLE 8.—*Qualitative, semiquantitative, and partial quantitative chemical analyses of inclusions*

[Not all constituents found necessarily were present as inclusions]

Reference	Constituents and sample notes
Akhvlediani and others, 1968	Na, Ca, Mg, K, and Cl on quartz from rock crystal veins, U.S.S.R.
Aleksandrovich and others, 1965.	H_2O and gases by heating halite from potash deposit.
Anikin, 1951	Na, K, Cl, and SiO_2 in Uralian hydrothermal quartz.
Arming and Preisinger, 1968	H_2O and CO_2 by mass spectrometry; 16 samples.
Bakumenko, 1968	H_2S, CO_2, and other gases in 19 samples of pegmatitic quartz.
Bazarova and Shugurova, 1968	CO_2 and other gases in various igneous rocks.
Becquerel and Moissan, 1890	Free F distilled from fluorite.
Beskrovnyy and others, 1967	Bitumens in schleelite-bearing skarns, Uzbek Soviet Socialist Republic.
Boyer and others, 1967	CH_4/CO_2 and CO_2/H_2O ratios in quartz by mass spectrometry.
Bradshaw and others, 1968	H_2O and CO_2 from decrepitation of halite.
Bratus', 1969	pH of inclusions in apophyllite.
Buerger, 1932a	NaCl in galena.
Chemla and others, 1968	CO_2, H_2O, CH_4, H_2, SO_2 in rock minerals by mass spectrometry.
Cifrulak and Cohen, 1969	NH_4Cl, liquid CO_2, liquid H_2O, and vapor in KCl by infrared analysis.
Davy, 1822	(Na, K), SO_4^{-2}, Cl, CO_3^{-2}, N_2, H_2O, and naphtha in quartz.
Dekate, 1961	Na, Ca, Mg, Fe^{+2}, Cl, HCO_3^{-1}, SO_4^{-2}, and pH, in barite from Pulivendla.
Dekate, 1963	Na, K, Ca, Mg, Cl, F, CO_3^{-2}, SO_4^{-2}, Al, Fe^{+2}, and pH, in quartz and fluorite, Rewat Hill tungsten deposit.
Dekate, 1967	K/Na and HCO_3/Cl ratios in inclusions from tungsten deposits in India discussed but no data presented.
Dolomanova and others, 1966, 1968.	Fe, Cu, Ti, Ca, Zn and Cr (by electron microprobe) in solid daughter minerals in smoky quartz.
Dons, 1956	CO_2; H_2S by odor only (could not be verified chemically) in barite concretion.
Dunton, P. J., in U.S. Geological Survey, 1965, p. A184.	Cl and S in eight samples from Zacatecas, Mexico.
Elinson, 1968a	CO_2, H_2S, H_2, He, in various samples.
Fermor, 1909, p. 213	Mn in H_2SO_4 extract of inclusion-rich rose quartz.
Forster, 1871	NH_3, CO_2 from metamorphic smoky quartz and CO_2 from fluorites heated in oxygen.
Fosberry, 1963	Total S in H_2S-bearing quartz and limestone.
Funkhouser and Naughton, 1968.	Ar, He in ultramafic inclusions, Hawaii.
Girault, 1967	CO_2, CO, H_2, N_2, H_2O, HCl, and hydrocarbons, by mass spectrometry on apatite from Oka, Canada.
Gol'dberg and Belyayeva, 1965	Large variety of complex organic compounds, including haloidorganic and sulfurorganic aliphatics, in Iceland spar.
Gol'dberg and Chernikov, 1968	Eighteen analyses of bitumens from alkalic and ultramafic rocks.
Greenwood, 1964	Ozone and fluorine (by odor) in pegmatitic fluorite.
Grogan and Shrode, 1952	Major Cl, Na, and Ca and lesser F, S, Si, Mg, Fe, and Al, in fluorite from Illinois.
Groves and Solomon, 1969	Eight analyses for ratios Na/K and Na/Li, Mount Bischoff, Tasmania.
Harrington, 1905	H_2S in Grenville limestone.
Hart, 1968	He and Ar from inclusions (?) in ultramafic rocks.
Heinrich and Anderson, 1965	C_5 and C_6 hydrocarbons, F_2, HF, F_2O, and possibly HCl, from fetid, fluorite-bearing carbonatite.
Holland, 1966	Na, Mn, Cu, and Zn in quartz near Providencia, Mexico, by neutron activation (reporting work by H. F. Puchner).
Holser and others, 1963	D/H ratios on halite analyzed by Holser (1963).
Huntley, 1955	Alpha-emitters in inclusions in clear quartz.
Il'in, 1964	Cl, K, and Na in Iceland spar.
Judd, 1889	Cl in scapolite.
Julien, 1881	NaCl in anorthosite, Labrador.
Kalyuzhnyi and Voznyak, 1967	BO_3^{-3}, Fe^{+2}, Fe^{+3}, Mg^{+2}, Mn^{+2}, and pH, in quartz from "Zanorysh" type pegmatites, U.S.S.R.
Karyakin, 1958	Na, K, Ca, Mg, Al, Fe, Mn, and Si in metamorphic quartz veins.
Kazanskiy and others, 1968	N_2, CO_2, O_2 in sedimentary quartz.
Khel'vas, 1959	Ca, Mg, Fe, Al, and Cu in liquid and solid inclusions.
Khetchikov, Balitskii, and Gasparyants, 1966.	Na, Li, Cl, S^{-2}, SiO_2, and pH in synthetic quartz crystals.
Kokubu and others, 1961	D/H ratios on H_2O from various igneous rocks.
Königsberger, 1900	CO_2 and H_2O in rose quartz.
Kraatz-Koschlau and Wöhler, 1899, 1900.	C, H, CO_2, and H_2O in fluorite from England.
Kropotkin and others, 1966	Bitumens, Ar, He, H, CO_2, N_2, and hydrocarbons up to butane from Precambrian basement rocks.
Langway, 1958	Composition (by mass spectrograph), and pressure of air bubbles in Greenland ice.
Laughlin, 1966, 1967, and Laughlin and Yoder, 1968.	Potassium and excess argon, probably from inclusions, in pegmatite minerals.
LaVallée Poussin and Rénard, 1876.	Na and Cl in quartz from a diorite.
Lyakhov, 1966	Fe, Mg, pH, and possibly BO_3 in quartz from Volynia.
Maslova, 1958	Na, K, Cl, Ca, F, and pH (but no Mg, Al, Fe^{+3}, SO_4^{-2}, CO_3^{-2}, PO_4^{-3}) in fluorite, Kermet-Tas.
Matsuda, 1963	H_2O from inclusions in beryl by heating to 500°–700°C.
Meyer, H., 1965	H_2, N_2, O_2, and CO_2 from melting of feldspars.
Mogarovsky and Markov, 1966	Bitumen in Tadjikistan hydrothermal fluorite deposits.

TABLE 8.—*Qualitative, semiquantitative, and partial quantitative chemical analyses of inclusions*—Continued

Reference	Constituents and sample notes
Motorina, 1967	CO_2, N_2, and sum of (H_2S, SO_2, NH_3, HCl, Cl_2) in topaz, Volynia.
Motorina and Bakumenko, 1968.	H_2S, CO_2, NO, O_2, CO, and H_2 from inclusions in pegmatites.
Mountain, 1942	CO_2, O_2, N_2, and pH, in "geode" from pegmatitic tin deposit.
Myaz' and others, 1968	Quartz and fluorite: ratios Na/K, Ca/Mg, and HCO_3/Cl.
Naughton and others, 1966	Potassium and excess argon in olivine nodules.
Newhouse, 1932	Na, Ca, and Cl (but no Mg, K, S^{-2}, SO_4^{-2}, or CO_2) in various sphalerites.
Nichol, 1829	Cl, Mg, and Ca, but no SO_4^{-2}, in halite, Cheshire.
Ohmoto, 1968a	D/H ratios on H_2O from inclusions in quartz and carbonates.
Osipov, 1968	Cl, F, Br, I, and B_2O_3 on water leach from rock.
Petersil'e and others, 1967	Gases and bitumens in tektites.
Pfaff, 1871	Cl and H_2O in various minerals and rocks.
Pirovskiy and Gusev, 1963	Elemental and chromatographic analyses of four bitumens from volcanic vent rocks.
Pinckney, 1966	Cu and Zn in Mississippi Valley-type deposits (<100 to $\sim 1{,}000$ ppm in water leaches, oral commun., 1968).
Polykovskii and Roizenman,	CO_2, H_2O, in 10 samples of pegmatitic quartz.
Poty, 1968b	CO_2, H_2O, and hydrocarbons in quartz from La Gardette and Mont Blanc.
Puchner and Holland, 1966	Na, Mn, Cu, and Zn in hydrothermal quartz, Zacatecas, Mexico, by neutron activation.
Rama and others, 1965	Potassium and excess argon in quartz and fluorite.
Renard, 1876, 1877	Na and Cl in quartz, Quenast diorite.
Roedder, 1958	CO_2 and H_2O in quartz, Grass Valley, Calif.
Roedder, 1963a, 1965d	CO_2 and H_2O in olivine nodules.
Rose, 1839	N_2, CO_2, and CH_4 in Wieliczka halite.
Rosset, 1965	Na and Cl in Oligocene gypsum.
Rye and O'Neil, 1968	O^{18} and C^{13} in fluid inclusions, Providencía, Mexico.
Salm-Horstmar, 1853	K, Na, Cl, Ca, and SO_4^{-2} in Dauphiné and New York quartz.
Sandberger, 1889	Cl and SO_4^{-2} in light sphalerite.
Sawkins, 1966a	Na/K ratios, North Pennine orefield, England.
Sawkins, 1966b	Na/K ratios, Cornwall ore field, England.
Schaffhäutl, 1843	Chlorine monoxide in fluorite from Wolsendorf.
Schertel, 1878	Na, Cl, Zn, and SO_4^{-2} in sphalerite from Spain.
Scholander and Nutt, 1960	CO_2 in air inclusions in Greenland ice.
Shugurova, 1967a	CO_2 and N_2 in 16 samples of pegmatitic quartz.
Sine, 1925	F, in antozonite, Ontario.
Sorby, 1858	Na, K, Ca, Mg, Cl, SO_4^{-2}, and pH in calcite from Vesuvius (p. 480), quartz (p. 471–473), etc.
Sorby and Butler, 1869	(Na, K) and Cl in emerald.
Stalder, 1967	CO_2, H_2O, CH_4, N_2, and H_2S in Alpine quartz by mass spectrometry.
Tammann and Seidel, 1932	CO_2, O_2, N_2, and hydrocarbons in halite.
Touray, 1968	CO_2/H_2O ratios in 17 samples by mass spectrometry.
Touray and others, 1966	CO_2/H_2O ratios by mass spectrometry of gases evolved on decrepitation.
Touray and Sagon, 1967	H_2O, CH_4, and hydrocarbons by mass spectrometry.
Touray and Yajima, 1966	CO_2, by mass spectrometry of gases from heating Alpine quartz.
Valyashko and others, 1968	Fe, Si, Al, K, Mn, Cl, S, Ca, TR, Ti, Cu, and Mg by X-ray spectrometry in solid and congealed inclusions from the Khibiny apatite deposits, U.S.S.R.
Vertushkov, 1966	CO_2 and H_2O, Uralian vein quartz.
Vlasenko, 1957	Na, K, Ca, Si, Al, Ti, Fe, Mg, Cl, SO_4, CO_3^{-2}, in nepheline syenite.
Wardlaw and Schwerdtner, 1966.	Br in primary inclusions in halite.
Weber, 1908, p. 223	NH_3, S^{-2}, and H_2S, in sphalerite from Santander.
Weinschenck, 1896	CO_2, from heating fluorite.
Wimmenauer, 1963	K and Na in apatite, Kaiserstuhl carbonatite.
Wlotzka, 1961	NH_3 in pegmatitic quartz.
Wyrouboff, 1866	CO_2 from heating fluorites in oxygen (amount proportional to color).
Zakharchenko, A. I., Lazarevich, N. S., and Elinson, M. M., in Khitarov, 1966.	Halides, halogens, H_2, N_2, CO_2, H_2S, and H_2O in granitic series.
Zezin and Sokolova, 1967	Twenty-three analyses of solid and liquid organic compounds, Khibiny massif, U.S.S.R.
Zirkel, 1870b	Na and Cl in minerals from various rocks.

PLATES 1–12

[Except as otherwise indicated, all photomicrographs are of doubly polished plates, taken in transmitted white light. Highly birefringent minerals were photographed in polarized light (one polarizer only), adjusted to yield a single, sharp image. All others were photographed in plain light]

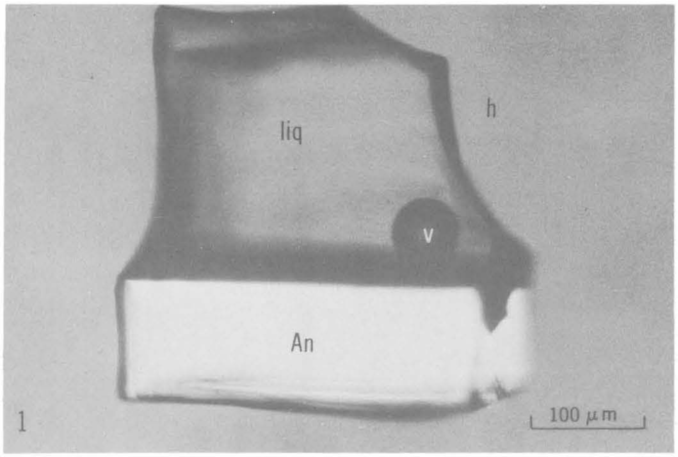
PLATE 1

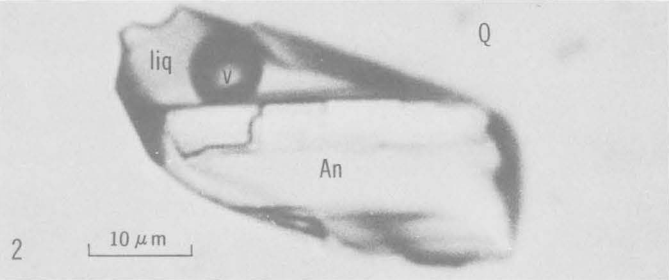
FIGURES 1–5. Photomicrographs of primary fluid inclusions formed by imperfect closure of host mineral around solid crystal inclusions. In each case the direction of growth was probably upward as shown.

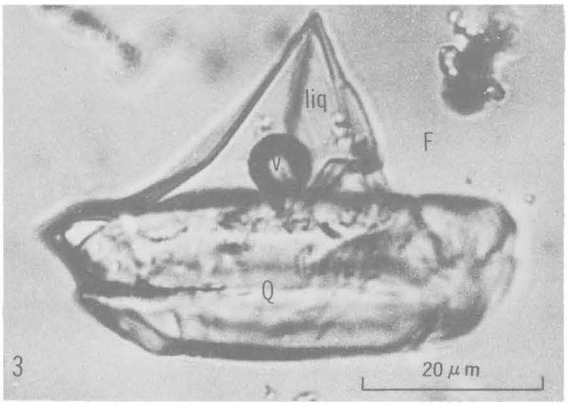
1. Solid inclusion of anhydrite (**An**) in halite (**h**). The liquid (**liq**) is a saturated water solution with vapor bubble (**v**) and presumably differs in origin from the high pressure gas inclusions also present in this salt. (See p. JJ43.) ER 64–157, Winnfield salt dome, Louisiana.
2. Unidentified solid inclusions, probably anhydrite (**An**) in quartz (**Q**). Since the anhydrite crystal is surrounded by solution (**liq**), the shrinkage bubble (**v**) would represent the sum of the shrinkage of anhydrite and liquid, minus that of the quartz host. ER 62–24, quartz geode, Keokuk, Iowa, courtesy of Richard L. Sutton, Jr., M.D.
3. Solid inclusion of quartz (**Q**) in fluorite (**F**). ER 63–170, Hansonburg, N. Mex.
4. Opaque cube (probably pyrite) in calcite (**C**), with resultant inclusion of liquid (**liq**). This is one of hundreds of such pyrite(?) crystals outlining a "ghost" or "phantom" within the calcite. The "stretched" liquid in this is a strong brine (approximately 3-molar NaCl equivalent), under negative pressure (Roedder, 1963c), that is the result of a failure to nucleate a vapor bubble on cooling from the temperature of formation; on supercooling to −30°C, this and other similar inclusions nucleated vapor bubbles that were eliminated by heating to 45±2°C. ER 62–105, Farrey mine, near Leadmine, Illinois (Upper Mississippi Valley district).
5. Solid inclusions of chalcopyrite(?) along a growth plane in banded sphalerite, each of which has resulted in trapping a primary fluid inclusion. The three fluid inclusions in the plane of focus each show liquid and a gas bubble. Providencia, Mexico (Sawkins, 1964, p. 905).

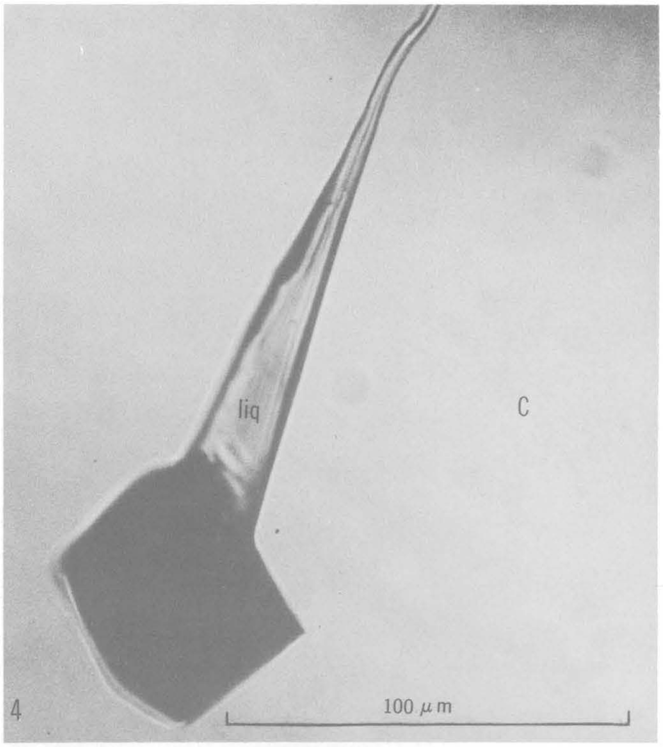
6. Primary inclusions in yellow, color-zoned sphalerite crystal from a vug. The large inclusions (containing mildly saline brine and a small vapor bubble) lie in a plane parallel to and only about 0.3 mm under the striated crystal surface, so that both the inclusions and the crystal face are in focus. Such inclusions must be primary. Presumably they form as a result of covering over of irregularities (stepwise growth) on the crystal face. Sample from P. M. Bethke, U.S. Geological Survey, his number PMB–107, from the OH vein at the Pb-Zn-Cu-Ag deposit at Creede, Colo.

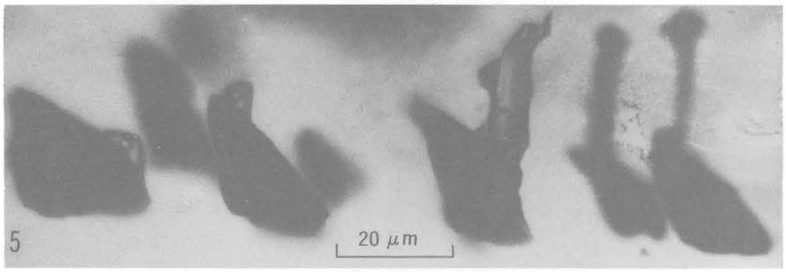
7, 8. Deep etching as a cause for the formation of primary inclusions. In figure 7, a green gemmy tourmaline crystal from Minas Gerais, Brazil (ER 63–142, courtesy of Richard L. Sutton, Jr., M.D.), is almost cut in half by a series of deep etch pits through the original striated prism faces. If later growth had occurred, it would probably have covered over these etch pits. Figure 8 is a photomicrograph of a polished plate of sphalerite from Creede, Colo. (PMB J–47, courtesy of P. M. Bethke, U.S. Geological Survey; see Barton and others, 1963, fig. 4C), which grew from left to right as a series of color zones. Solution of the light-yellow zone A occurred at the stage now marked by the layer of small fluid inclusions (**x**), opaque in this lighting, and caused a deep etch pit at the center. Succeeding growth of dark zone B trapped a large primary fluid inclusion (**y**) which was opened by the sawing of the sample.

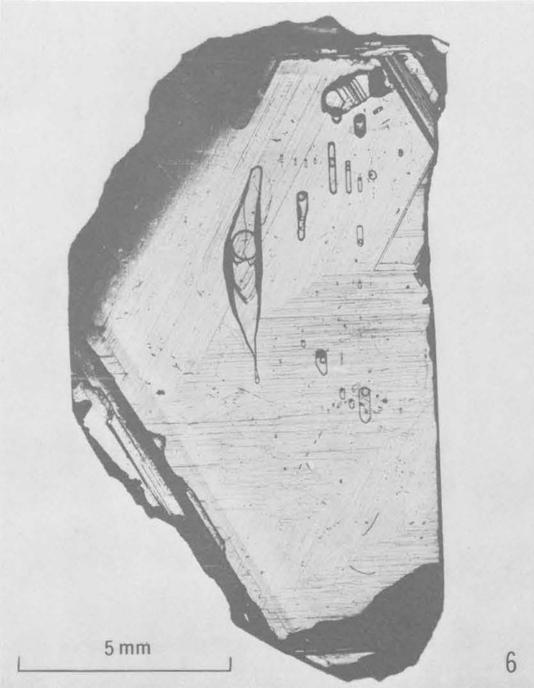


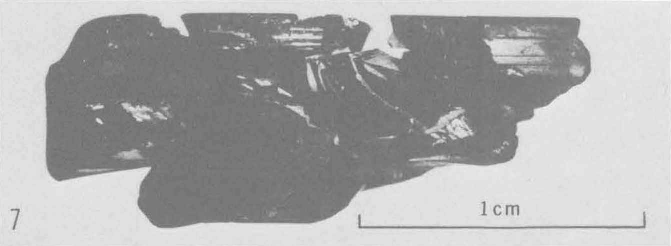












MECHANISMS OF TRAPPING OF PRIMARY INCLUSIONS

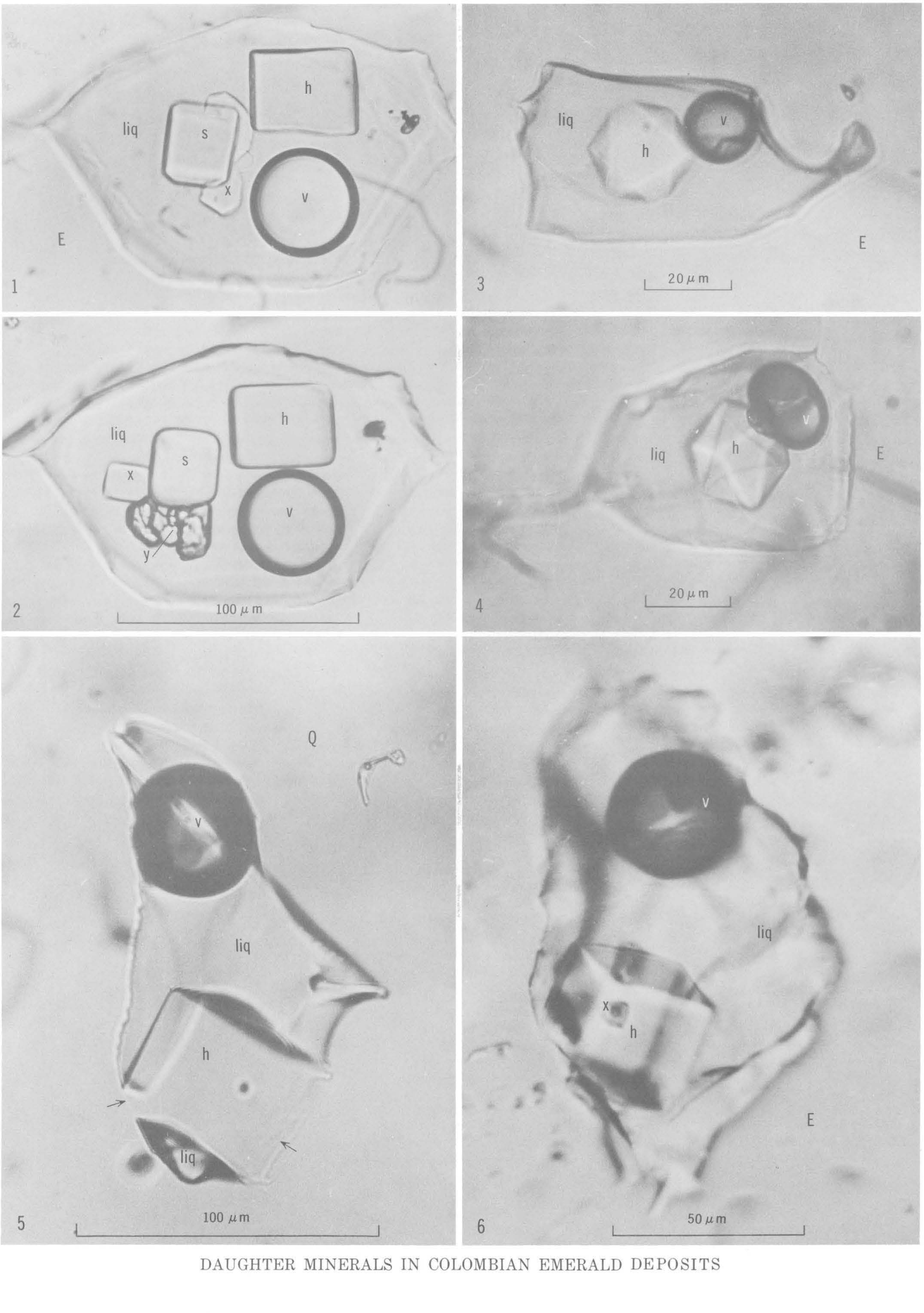
PLATE 2

FIGURES 1, 2. Photomicrographs of a fluid inclusion (**liq**) in emerald (**E**) containing vapor bubble (**v**), and several daughter minerals, before (fig. 1) and after (fig. 2) freezing, both taken at room temperature. This is one of many inclusions containing the same assemblage. Cube **h** is halite, cube **s** is probably sylvite, and **x** is an unidentified isotropic phase that recrystallized after freezing. (Dark lines in lower right of fig. 1 are photographic artifacts.) Granular mass with high relief (**y**), which nucleated on freezing, is believed to be parisite ($(\text{Ce, La})_2\text{Ca}(\text{CO}_3)_3\text{F}_2$) on the basis of its optical properties and the frequent occurrence of parisite crystals in the same veins (Walton, 1950). Most of the other inclusions in this sample showed such parisite(?) crystals before (and after) freezing. ER 63–139b, Muzo emerald mine, Colombia, courtesy of Banco de la Republica, Bogotá.

3, 4. Photomicrographs of inclusions showing isotropic octahedral daughter crystals (**h**) believed to be halite, plus vapor (**v**) and liquid (**liq**) in emerald (**E**). ER 63–139b, Muzo emerald mine, Colombia, courtesy of Banco de la Republica, Bogotá.

5. Photomicrograph of large three-phase inclusion in quartz. In addition to the dark vapor bubble (**v**), a large daughter crystal of halite (**h**) grew from the liquid (**liq**), in very close contact with the quartz walls, sealing off a small part of the liquid phase (at bottom). The index of refraction of the cube of halite (1.544) is very close to that of the surrounding quartz (**Q**) in this orientation, making the contacts almost invisible. (See arrows.) ER 63–84Q, Muzo emerald mine, Colombia, courtesy of Banco de la Republica, Bogotá.

6. Photomicrograph of two-phase liquid-gas inclusion (**x**), in cubic halite daughter crystal (**h**) in liquid inclusion (**liq**) in emerald (**E**). The inclusion in the daughter mineral preserves a record of the conditions at the time of growth of the daughter mineral; the larger inclusion preserves a record of the conditions at the time of growth of the enclosing emerald crystal. ER 63–139b, Muzo emerald mine, Colombia, courtesy of Banco de la Republica, Bogotá.



DAUGHTER MINERALS IN COLOMBIAN EMERALD DEPOSITS

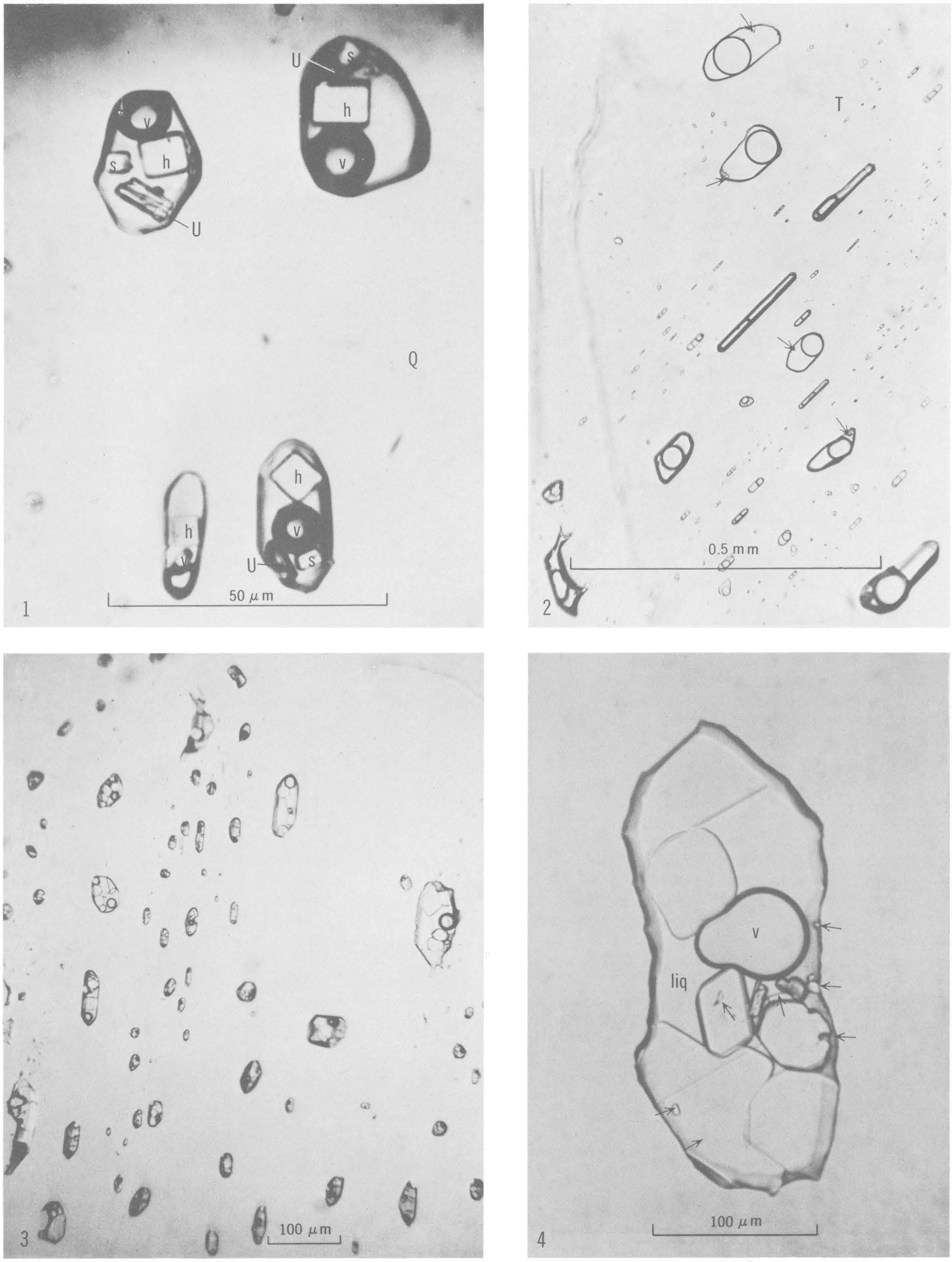
PLATE 3

FIGURE 1. Photomicrograph showing daughter minerals in inclusions in quartz (**Q**) from a vein of quartz, cassiterite, chalcopyrite, arsenopyrite, pyrite, and marcasite, associated with the Land's End Granite, Penlee quarry, near Penzance, Cornwall, England. The smaller cubes, probably sylvite (**s**), dissolve at 145°C; the larger halite cubes (**h**) dissolve at 400°C; the vapor bubbles (**v**) disappear at 440°C. The unidentified prismatic daughter crystals (**U**, possibly a sulfate) have strong birefringence and parallel extinction; they show no signs of solution even at 440°C (Sawkins, 1966b, his sample 64–C–21).

2. Photomicrograph of plane of probably pseudosecondary inclusions in topaz (**T**), parallel to {001}, each containing a large vapor bubble (approximately 35 percent by volume) in the liquid, and three tiny daughter crystals. (For examples, see arrows; in other inclusions they are hidden.) These crystals are unidentified, but are recognizably different phases (Roedder, 1963b, p. 173). USNM specimen 96595, Rukuba tin mine, Nigeria.

3. Photomicrograph of plane of pseudosecondary(?) multiphase fluid inclusions in transparent magnesite crystal, Brumado, Bahia, Brazil (Bodenlos, 1954; see also Rosenberg and Mills, 1966). Each contains at least seven different daughter crystals and a small bubble. (See fig. 4.) Within the limits of available observational techniques, each inclusion other than the very smallest ones visible here appears to contain the same assemblage, indicating trapping of an originally homogeneous fluid containing over 50 percent solids by weight. ER 62–4.

4. Photomicrograph of a single multiphase inclusion from same sample as figure 3, showing liquid (**liq**), vapor bubble (**v**), and 14 daughter crystals, at least seven of which are different phases. Two of the daughter crystals are isotropic, but six of the small and at least one of the large crystals are anisotropic (see arrows).



CONSTANCY OF RATIOS OF DAUGHTER MINERALS

PLATE 4

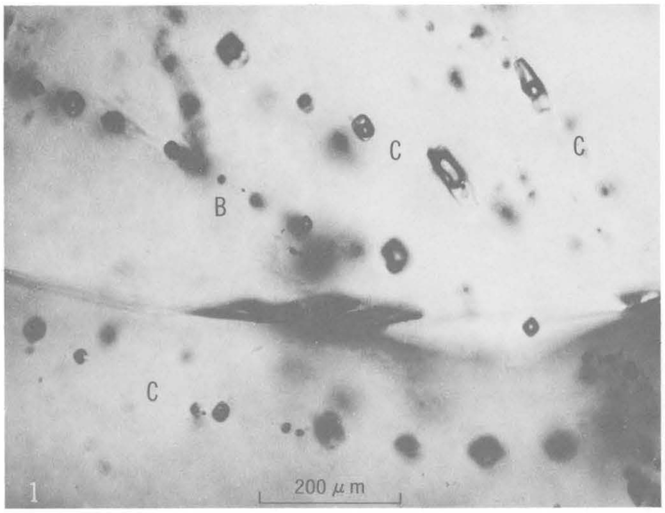
FIGURES 1–3. Photomicrographs of two stages of secondary inclusions in quartz from a quartz-molybdenite vein, Bingham porphyry copper pit, Bingham, Utah. Figure 1 shows a plate of quartz containing four curved, healed fractures, now outlined by trains of fluid inclusions (plus one more recent crack that has not been healed). The fracture labeled **B** contains only inclusions corresponding to low density, low salinity fluids high in CO_2, a typical one of which is shown enlarged in figure 2. It is a faceted negative crystal in quartz (**Q**), now containing a small amount of low salinity liquid (**liq**), a large vapor bubble (**v**) containing CO_2 under pressure, and a small red to opaque grain, possibly specularite (**S**). On warming after freezing, the last ice crystal melts at −4.9°C, leaving only crystals of $CO_2 \cdot 5\frac{3}{4}\ H_2O$ (melting at about 0°C) and the specularite. The other three fractures labeled **C** all contain an entirely different type of fluid inclusion, illustrated in figure 3. It was a very dense brine and formed inclusions which now contain saturated solution (**liq**), a large daughter crystal of halite (**h**), a birefringent prism, possibly anhydrite (**An**), a small red to opaque grain, possibly specularite (**S**), and a low pressure vapor bubble (**v**). It is possible that inclusions of fracture **B** might represent the dense vapor of solutions of the type trapped in fracture C, boiling under slight pressure release. ER 63–209 (Roedder, 1971).

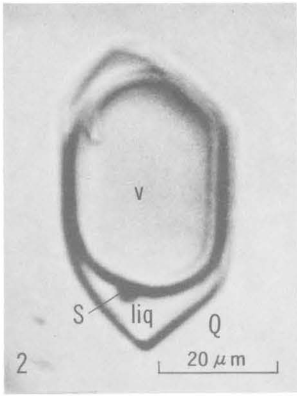
4. Photomicrograph of alkali feldspar from granitic block in lavas of Ascension Island, South Atlantic, showing very high concentration of multiphase inclusions. These presumably primary inclusions are believed to have formed as a result of immiscible droplets of a highly saline fluid sticking to the feldspar crystal surface as it grew from a silicate melt. Each contains vapor, liquid, and a large daughter crystal of halite (see pl. 11, figs. 1–4); the larger inclusions have nucleated crystals of several other phases as well. The inclusions are so abundant (about $10^{10}/cm^3$) that single-crystal X-ray photographs of such feldspar show the three strongest powder diffraction lines of NaCl. ER 63–135b (Roedder and Coombs, 1967).

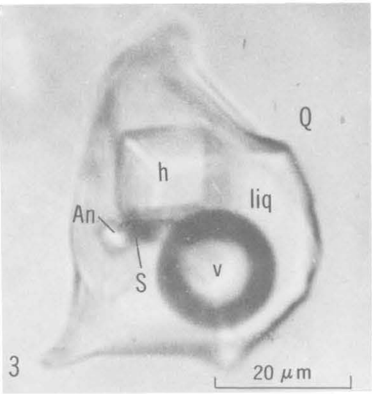
5. Photomicrograph of a cleavage fragment of fluorite showing very large numbers of almost spherical primary inclusions of dark-brown oil, each with a vapor bubble. These occur in specific crystal growth zones in the fluorite and are believed to represent the preferential trapping of droplets of oil suspended in the hot brines from which these fluorite crystals grew. Sample from unknown locality in southern Illinois, courtesy of Anthony Denson, U.S. Geological Survey.

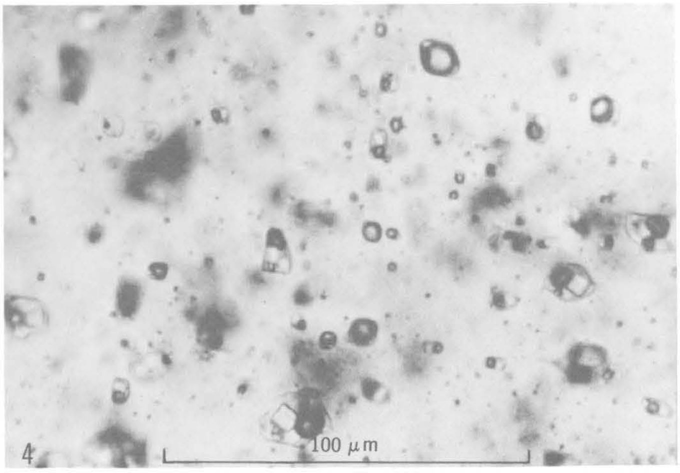
6. Photomicrograph of fluid inclusions in olivine from ultrabasic nodule in basalt, showing basaltic(?) glass (**gl**), liquid CO_2 (**lc**), and gaseous CO_2(**v**). The two CO_2 phases homogenize in the liquid phase by the slight warming caused by absorption of infrared (IR) light on removal of the IR filter on the microscope light. During the growth or later fracturing and healing of the host olivine, at an estimated depth of 8–16 km and 1,200°C, the CO_2 was present as homogeneous supercritical gas bubbles in the basaltic, CO_2-saturated, melt. Most olivine nodule occurrences examined, from many localities, show such inclusions, implying worldwide saturation of the basaltic magmas from which nodule minerals are presumed to have crystallized (Roedder, 1965d). ER 63–33, 1801 Kaupulehu flow, Hualalai, Hawaii.

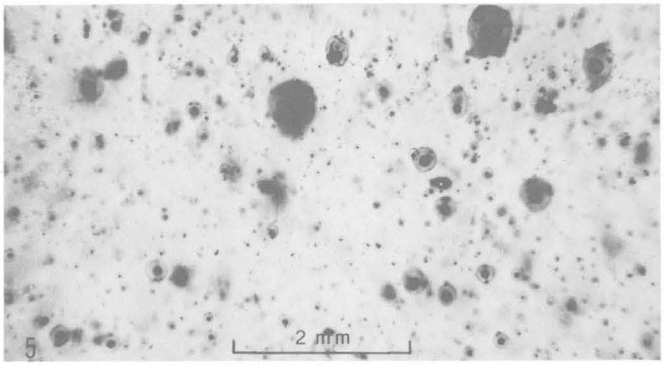
7–9. Photomicrographs of inclusions showing evidence of immiscibility between silicate melt and hydrous, saline fluids (Roedder and Coombs, 1967). During the growth of the host quartz crystal (**Q**) from a saturated hydrous silicate melt of rhyolitic composition, some parts of this melt were trapped as primary inclusions. There were immiscible globules of highly saline aqueous fluid in the melt, which were also trapped in these inclusions. On cooling, the melt formed a glass (**gl**), and the saline fluid formed at least three phases. In figures 8 and 9 it formed an isotropic cube, presumably NaCl (**h**), a thin, almost invisible layer of saturated liquid water solution (**lw**), and vapor (**v**). In figure 7 (lower right) it formed a wet mass of crystals (**x**) lining the cavity, with one larger cube, presumably NaCl (**h**); several other droplets of the saline fluid were trapped without silicate melt, forming inclusions now containing a large crystal of halite (**h**), vapor (**v**), saturated liquid water solution (**lw**), and several unidentified crystals. ER 63–134, Ascension Island, South Atlantic Ocean.

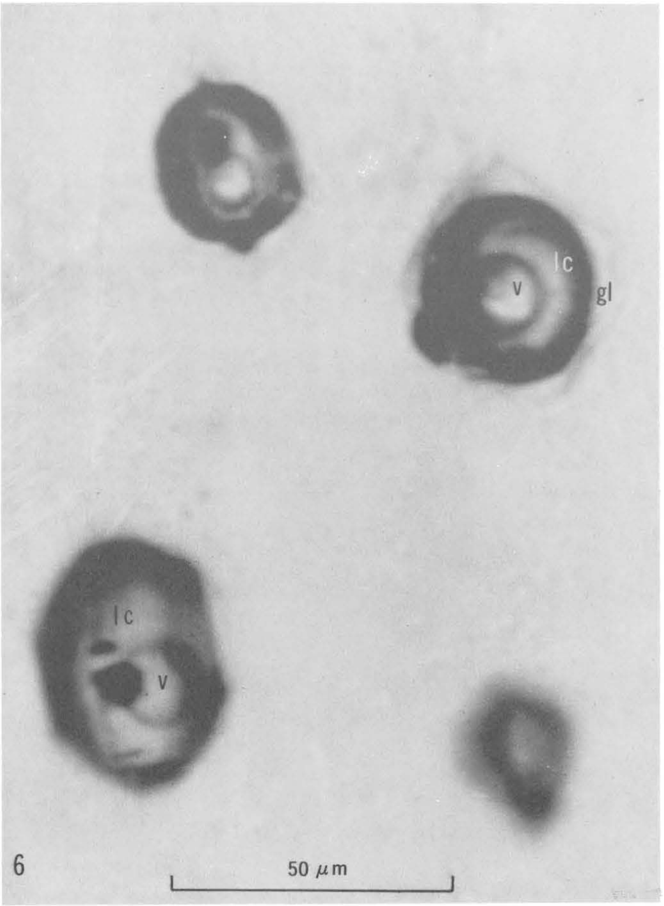


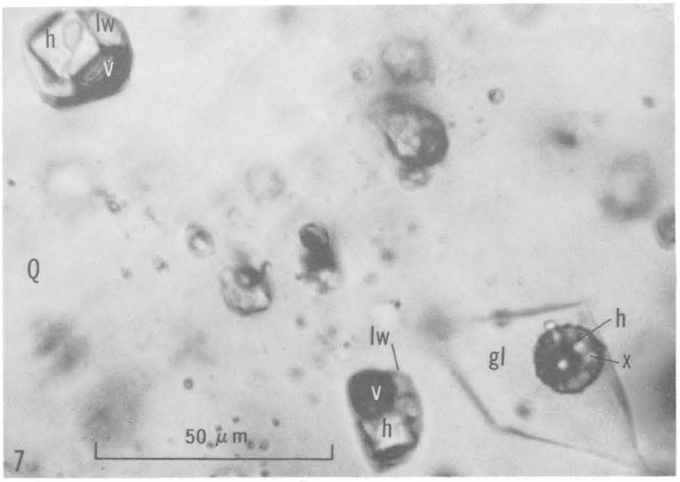


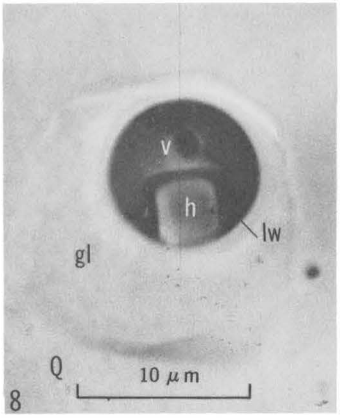


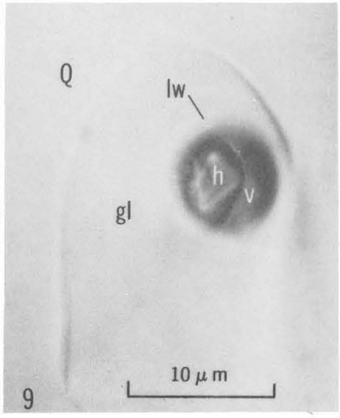












EVIDENCE OF IMMISCIBLE FLUIDS IN NATURE

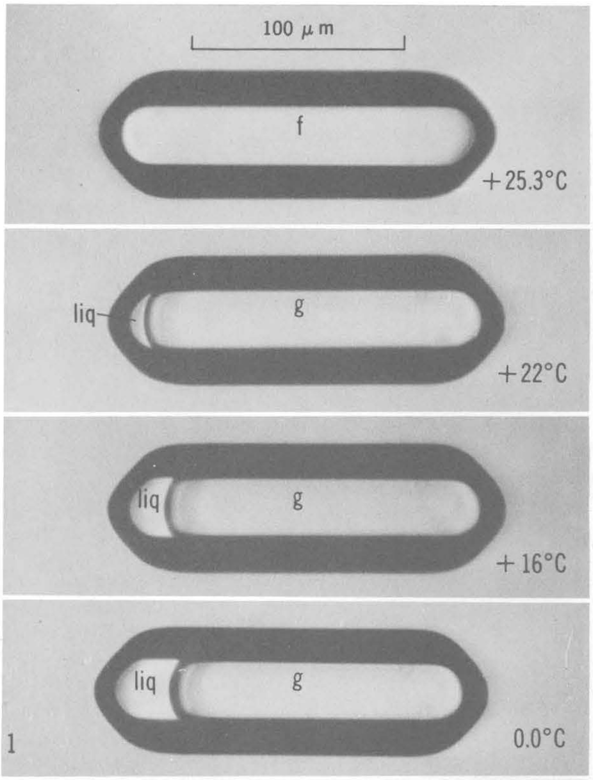
PLATE 5

FIGURE 1. Serial photomicrographs of a primary CO_2 inclusion, in the form of a negative crystal, in a quartz crystal from a vug in the Red Devil mercury mine, central Kuskokwim area, Alaska, taken at the temperatures indicated. Inclusion evidence indicates that this CO_2 was present as globules of a separate, immiscible, dense CO_2 fluid in the mildly saline, water-rich fluids forming this quartz crystal and the coprecipitated cinnabar. Although at the temperatures of trapping (probably over 100°C) the CO_2 was present as a homogeneous fluid phase (**f**), its separation to form liquid CO_2 (**liq**) and gaseous CO_2 (**g**) on cooling can be used to verify the density (and hence pressure) of filling. ER 61–5 (Roedder, 1963b, p. 189).

2. Photomicrograph, taken at room temperature, of multiphase inclusions in emerald (E), showing relatively small vapor bubbles (**v**) and large daughter crystals of halite (**h**) in saturated brine (**lw**). The bubbles contain CO_2 gas at high pressure. The inset shows another inclusion in this same group, photographed at 10°C, containing a crescent-shaped fillet of liquid CO_2 (**lc**) that evaporates into the bubble on warming to room temperature. ER 63–139b, Chivor emerald mine, Colombia, courtesy of Banco de la Republica, Bogotá.

3–5. Serial photomicrographs of a flattened inclusion taken at different temperatures to show the identification of CO_2 by the formation of invisible crystals of $CO_2 \cdot 5\frac{3}{4}\ H_2O$ on cooling. Figure 3, taken at room temperature, shows liquid water solution (**lw**), probably about 1 molar NaCl equivalent in salinity, vapor (**v**), and a small film of liquid CO_2 (**lc**). When cooled quickly, more liquid CO_2 condenses from the gas to yield the metastable assemblage shown in figure 4, taken at about −5°C. When the inclusion is first frozen at −78°C, then equilibrated at −8°C (fig. 5), the liquid CO_2 slowly reacts with the water solution to form a mass of crystals of $CO_2 \cdot 5\frac{3}{4}\ H_2O$ (**x**). The "meniscus" between this and the vapor bubble (**v**) is a jagged, irregular border (arrow); on heating, the last invisible hydrate crystal melts at 7.60°±0.20°C to form liquid CO_2 and water solution again, with a smoothly curved meniscus. During the melting process, the CO_2 hydrate crystals are usually invisible as they are isotropic, with an index of refraction almost identical with that of the solution. ER 64–187, quartz, from a quartz-anhydrite-calcite vein, Homestake mine, South Dakota.

6, 7. Photomicrographs of a negative crystal "single phase" or "gas" inclusion, in quartz (**Q**). At room temperature and down to −5.50°C, it appears to be filled with colorless "gas," as seen in figure 6 (taken at −5.50°C). On slight further cooling (fig. 7, taken at −5.55°C), the "gas" is seen to have been a supercritical fluid, as it separates abruptly into liquid (**liq**) plus vapor (**v**). It is believed to be a mixture of organic compounds, probably mainly methane and ethane. ER 61–26, "Herkimer County diamond," Herkimer County, N.Y. (Roedder 1963b, p. 202).



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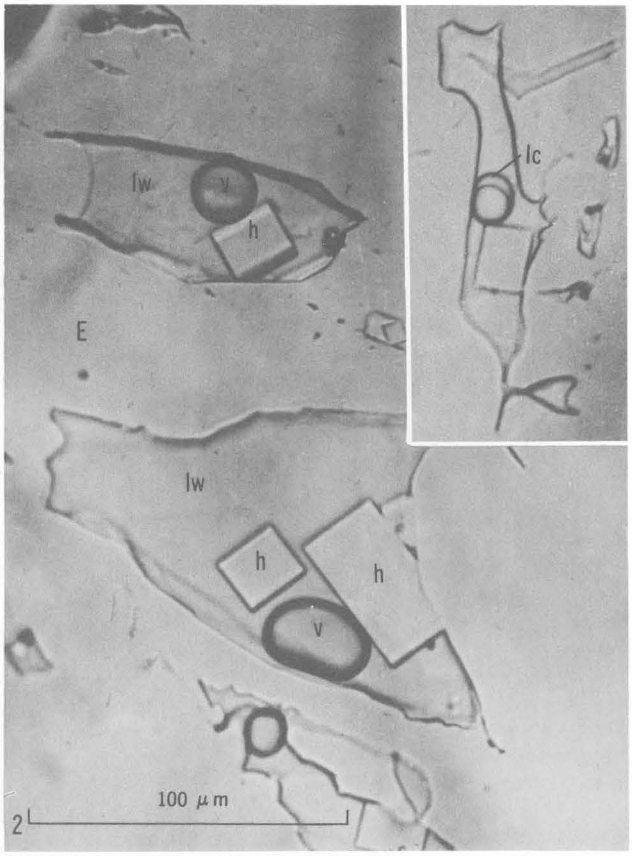

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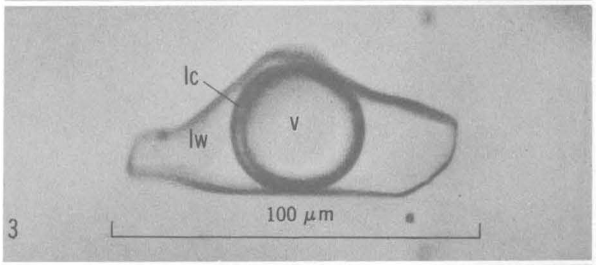

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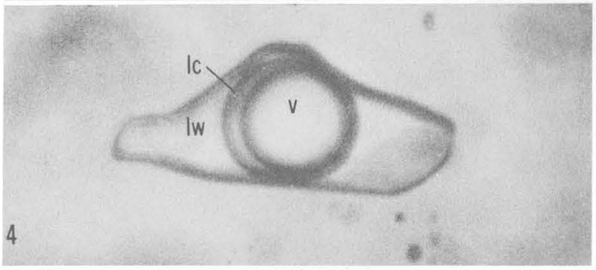

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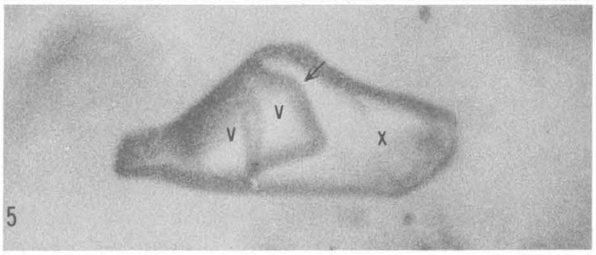

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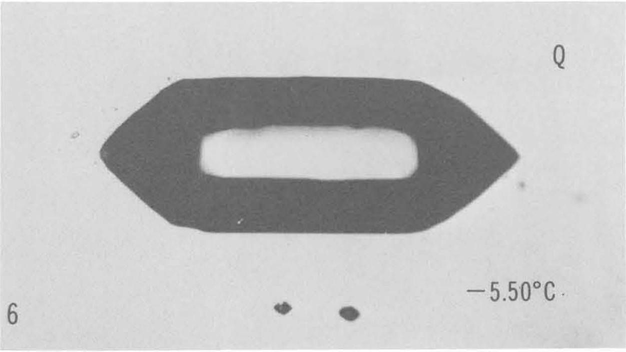

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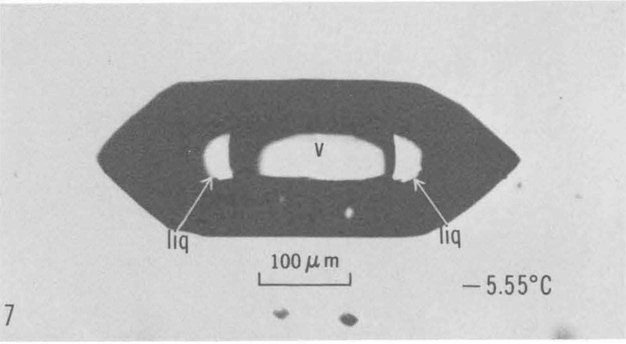


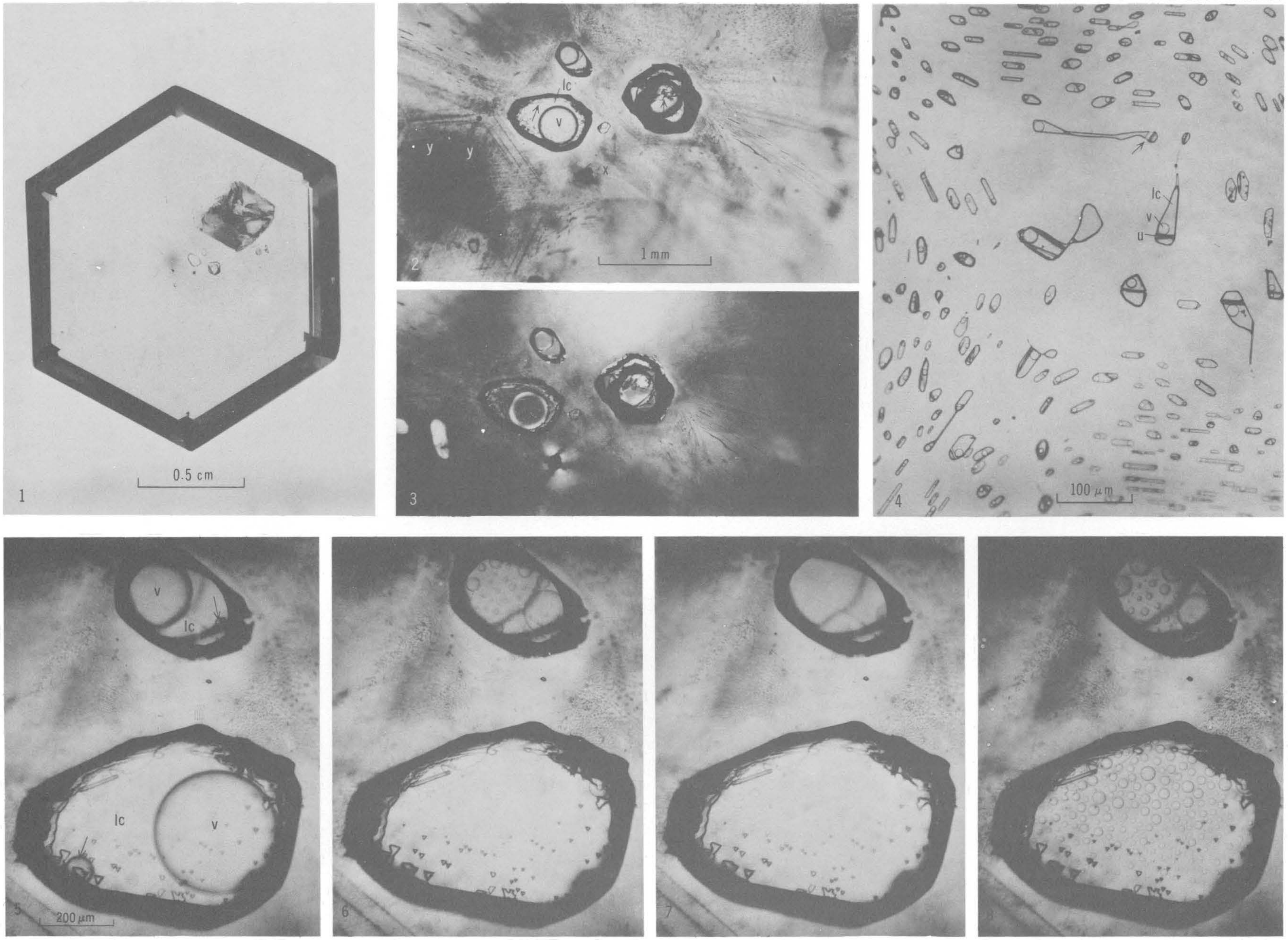
7

IDENTIFICATION OF GAS COMPOSITION BY COOLING PROCEDURES

PLATE 6

FIGURES 1–4. Inclusions in a very large (10.2 carat) cut gem sapphire from the famous locality at Yogo Gulch, Mont. All but figure 3 were taken in ordinary light. This flat, blue, hexagonal tablet, 3 mm thick, shows several large inclusions near its center (fig. 1). In figure 2, these show liquid CO_2 (**lc**) and a large bubble of gaseous CO_2 (**v**), plus two types of daughter crystals, equant and acicular (see arrows); both of these are birefringent (compare with fig. 3, taken with crossed polarizers; see also fig. 5). Although these CO_2 inclusions are under high pressure (approximately 70 atmospheres at room temperature), the sapphire host is not stressed enough to cause visible strain birefringence. The small opaque solid inclusion (**x**) causes such birefringence, but some faintly visible transparent solid inclusions (**y**) do not. A series of "feathers" extend several millimeters from the central inclusions. These consist of thousands of tiny secondary inclusions, tapering down in size with distance from the central inclusions. Figure 4 shows a small area of feather from the upper right of figure 2. Each inclusion has liquid CO_2 (**lc**) and vapor that homogenize at about 31°C and an unidentified birefringent plate or blade (**u**) with an index of refraction not very much less than that of the enclosing sapphire (see arrow). These sapphires occur in an altered pyroxene-biotite-analcite dike cutting limestone. Clabaugh (1952) believes the sapphire crystallized directly from the magma at depth. The inclusions indicate that the sapphire formed under high pressure, possibly in the presence of immiscible globules of dense CO_2 fluid carrying unknown solids in solution; later, under a lower confining pressure, the internal pressure caused the inclusions to crack and let some of the fluid escape into the fracture, where it caused healing of the fracture and the trapping of many tiny secondary inclusions. USNM specimen.

5–8. Typical behavior of large inclusions of essentially pure CO_2, at nearly the critical density of filling. These are enlargements of two of the inclusions shown in figure 2. The small triangles are markings on the wall of the inclusion. Because of absorption of infrared light from the microscope light (fig. 5), the meniscus between CO_2 liquid (**lc**) and CO_2 gas (**v**) becomes fainter as their densities approach each other at about 31°C. (Compare with fig. 2.) Such light absorption obviously cannot be perfectly uniform, and so even under static conditions, new bubbles of vapor form continuously in the liquid and move to the cooler main vapor bubble. (See arrows in fig. 5.) In figure 6 the large inclusion has homogenized in the liquid phase by a fading and shrinkage of the vapor bubble, indicating the fluid density to be slightly greater than the critical density; the faint menisci in the small inclusion simply fade away, indicating the fluid in it to be almost exactly at the critical density. The difference in filling density responsible for such a difference in behavior may be very small. After complete homogenization (fig. 7), slight undercooking of the stretched fluid (now a supercooled, subcritical fluid) results in a sudden splitting into faint vapor bubbles and liquid (fig. 8).



BEHAVIOR OF LARGE CO_2 INCLUSIONS IN SAPPHIRE

PLATE 7

FIGURE 1. Inclusion containing some daughter minerals that do not dissolve on reheating. The large cube is halite, the smaller rounded crystal is sylvite, and the two smallest crystals are unidentified. From its optical properties, the dark hexagon (actually bright red) is identified as a flake of hematite; when such inclusions are heated, the hematite and several of the tiny crystals do not dissolve even though the other major phases in this and similar inclusions become a fluid at temperatures above 400°C. Sample ER 63–211A, quartz, Bingham porphyry copper mine, Utah.

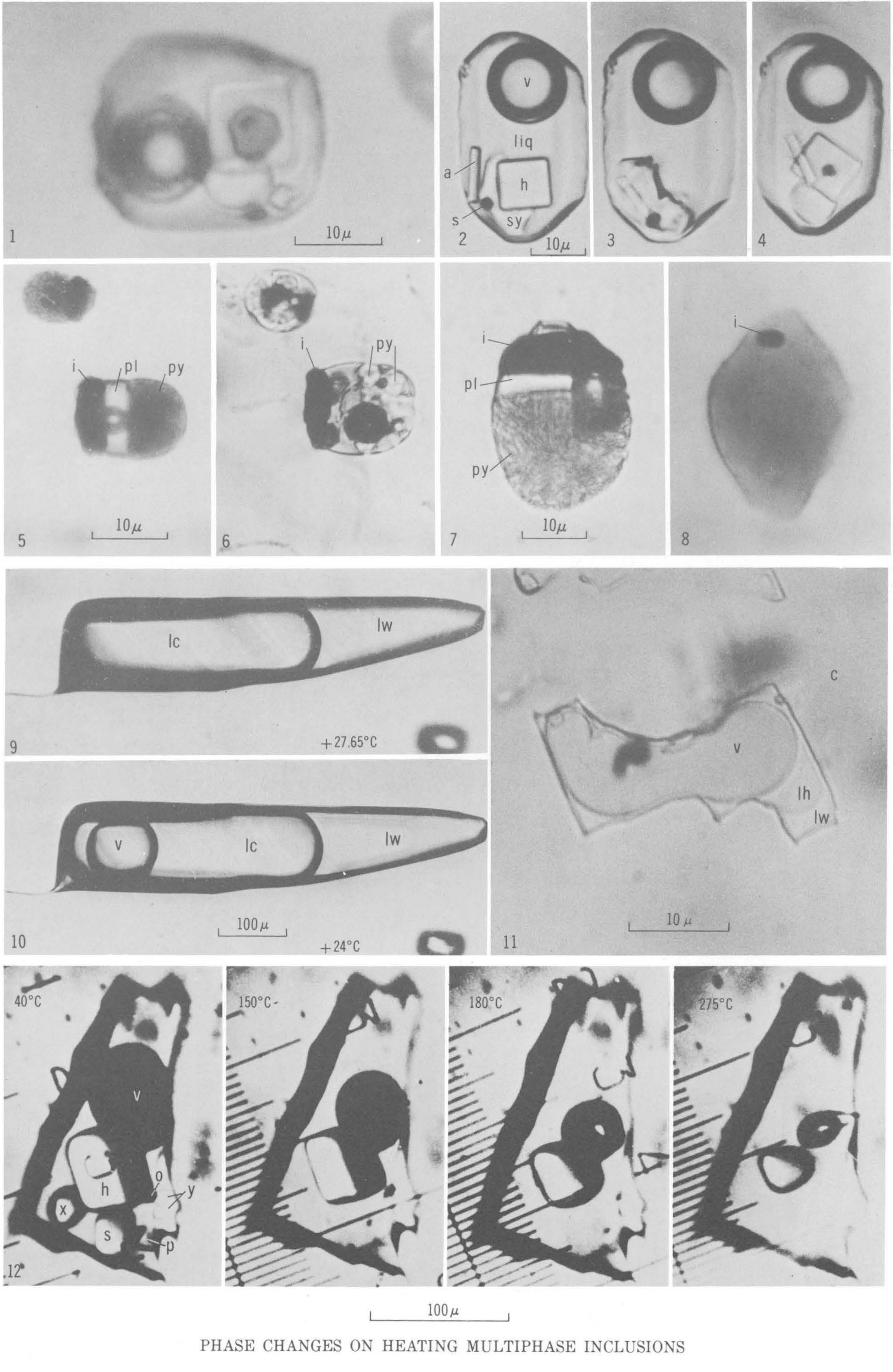
2–4. A multiphase inclusion in quartz similar to that in figure 1, from the same sample, showing recrystallization of daughter minerals. Figure 2 was photographed at room temperature after heating to 155°C (Roedder and Skinner, 1968); it shows halite (**h**), sylvite (**sy**), vapor (**v**), liquid (**liq**), hematite? (**s**), and possibly anhydrite (**a**). The inclusion was then heated to 410°C for 96 hours, was cooled and held at room temperature for several days (fig. 3), and then was examined after a period of 8 months (fig. 4). Note that the halite recrystallized to a euhedral crystal, and the nucleation and growth of sylvite at the bottom moved the other crystals about.

5–8. Examples of the homogenization of daughter minerals in silicate melt inclusions, in olivine from Apollo 11 lunar rocks, in a manner analogous to that used in aqueous inclusions. Figures 5 and 7 show the inclusions as returned from the moon, containing epitaxially oriented daughter crystals of plagioclase (**pl**) and ilmenite (**i**) (both parallel to {100} of the enclosing olivine) and feathery (quench?) crystals of pyroxene (**py**). Figures 6 and 8 show these same inclusions, after heating (in vacuo) to 1,110°C for 2 days and 1,200°C for 4 hours, respectively, and subsequent quenching. Note that at 1,110°C all of the plagioclase, most of the pyroxene, and part of the ilmenite is gone, and at 1,209°C only a trace of ilmenite is left, and the bubble is gone. This ilmenite melted at 1,210°C. Note that there has been solution of the walls of the inclusion as well. Both from lunar rock 10020–41.

9, 10. Photomicrographs of one inclusion of a large group of probably pseudosecondary inclusions in pegmatitic quartz, all showing a uniform ratio (at 24°C) of liquid water solution (**lw**) (about 0.7 molar NaCl equivalent, estimated from the freezing temperature of −2.50°C), liquid CO_2 (**lc**), and gaseous CO_2 (**v**). Homogenization of the two CO_2 phases occurs in the liquid phase at 27.65°C; at much higher temperatures, probably above 350°C, only one homogeneous "gas" phase is present, containing all the CO_2, H_2O, and salts. ER 61–28, Volta Bala, Teófilo Otoni, Minas Gerais, Brazil, courtesy of W. D. Johnston, Jr., U.S. Geological Survey, his number 52.

11. Photomicrograph of inclusion containing liquid H_2S, from a coarse-grained fetid marble. Although the density of filling varies between individual groups, a number of inclusions in this calcite (**c**) show the same three fluid phases, identified as strong and in part saturated aqueous brine (**lw**), liquid H_2S (**lh**), and gaseous H_2S (**v**). This particular inclusion shows homogenization of the H_2S liquid and gas, in the gas phase (that is, by evaporation), at 64°C. ER 63–232, U.S. mine, Lark, Utah, adjacent to Bingham porphyry copper pit (Roedder, 1971).

12. Serial photomicrographs of a multiphase inclusion in topaz from Volynia, U.S.S.R., taken at the temperatures indicated. At 40°C there is a vapor bubble (**v**, 19.6 percent by volume), and daughter crystals of halite (**h**, 14.9 percent), sylvite (**s**, 5 percent), an unknown colorless phase (**x**, 3 percent), several small hexagonal crystals of another unknown phase (**y**), a prismatic bluish crystal (**p**, about 1 percent), and an opaque mineral (**o**, about 1 percent). On heating, the sylvite dissolved very rapidly, and at 150° and 180°C all of the sylvite and the bulk of the other phases have dissolved; at 275°C all phases except halite (3.4 percent) and vapor (2.5 percent) are gone. The vapor bubble disappears at 300°C and the last of the halite dissolves at 310°C. The volume percentages were obtained from an adjoining long tubular inclusion containing the same assemblage. From Ermakov (1950a, pls. 21–24).



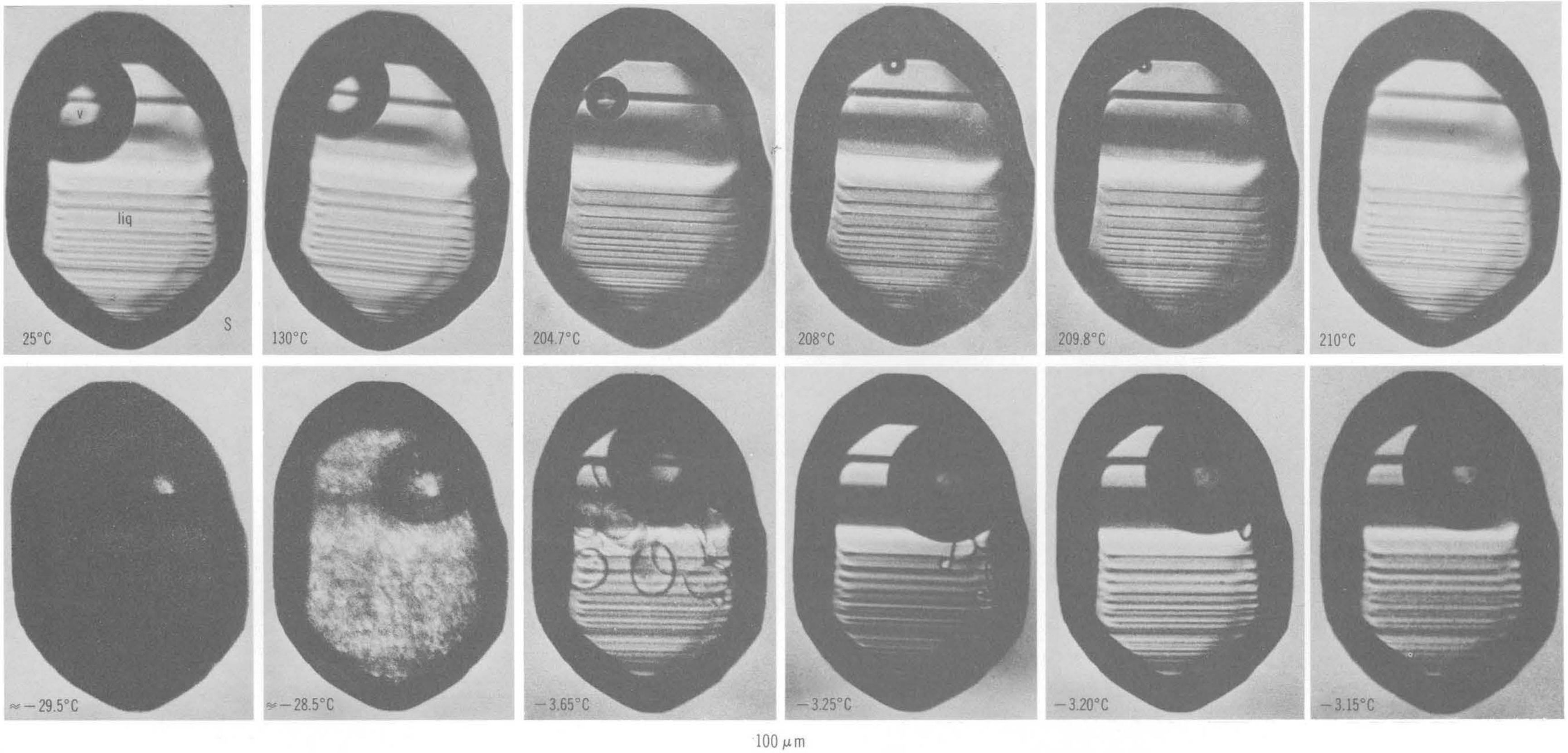
PHASE CHANGES ON HEATING MULTIPHASE INCLUSIONS

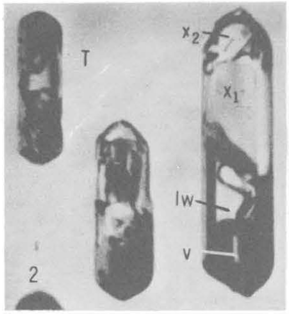
PLATE 8

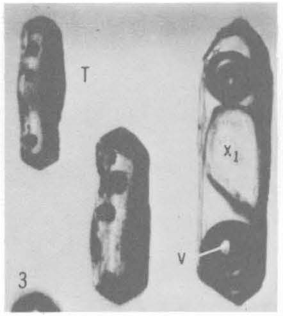
FIGURE 1. Serial photomicrographs of a large primary inclusion in sphalerite (**S**) from Creede, Colo. (sample ER 57–34), taken on the freezing stage (bottom row) and heating stage (top row), after equilibration at the temperatures indicated. The horizontal bars are oscillatory stria on the cavity walls. After freezing at −78°C, the inclusion looked the same as at ≈ −29.5° and consisted of an opaque mass of solid ice and salts. Between −29.5° and −28.5°C, enough melting occurred to form liquid films between the grains, making the inclusion translucent (that is, the "first melting temperature" is −29°C). At higher temperatures, more of the ice melted, the remainder of the ice recrystallized to larger crystals, and the bubble increased in volume due to the volume decrease on melting of ice. The last ice crystal melted, under equilibrium conditions, at −3.15°C (that is, the "freezing temperature"). Slight cooling did not cause ice to nucleate again, as the solution remained as supercooled liquid to temperatures below −35°C (Roedder, 1962a). On heating, the bubble (**v**) decreased in volume to the homogenization temperature (or filling temperature); in this, the inclusion homogenized in the liquid phase.

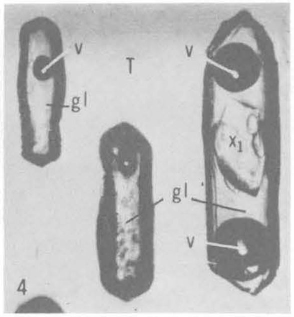
2–4. Sequence of photomicrographs, at room temperature, of a group of inclusions in topaz (**T**), showing vapor (**v**), liquid water solution (**lw**), and large amounts of daughter crystals (**x**) before heating (fig. 2), after heating to 740°C (700°?) for 3½ hours (under external pressure to prevent decrepitation) (fig. 3), and after unspecified "repeated heating" (fig. 4). The largest daughter crystals are quartz ($\mathbf{x}_1$) and muscovite ($\mathbf{x}_2$). Smaller amounts of fluorite(?), cryolite, various fluorides and chlorides of K, Na, and Ca, and an unidentified phase are present in figure 2. The large amounts of daughter minerals (about 70 percent by volume), and part of the topaz host, dissolved slowly on heating to form a hydrous glass (**gl**). The vapor bubbles (**v**) in the heated samples probably formed on cooling, as the vapor bubble shown in figure 2 was found to disappear at 500°C, before most of the daughter crystals had dissolved. From Lemmlein, Kliya, and Ostrovskii (1962). (See also Voznyak (1968).)

5, 6. Photomicrographs of a pair of primary inclusions in topaz taken at room temperature, before heating (fig. 5), and after heating to 700°C, under external pressure to prevent decrepitation (fig. 6). Not only have all of the daughter crystals (**x**) dissolved to form a hydrous silicate melt, now a glass (**gl**), but very appreciable amounts of the topaz walls of the inclusions dissolved at elevated temperatures and reprecipitated, in part, changing the shape and size of the inclusions. See figures 2–4 for details of daughter crystals. From Lemmlein, Kliya, and Ostrovskii (1962). (See also Voznyak (1968).)

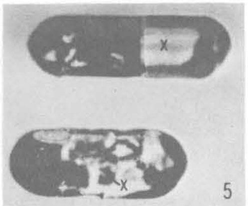


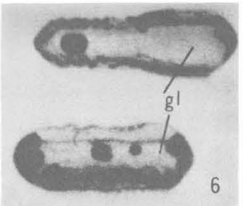






0.2 mm





100 μm

PHASE CHANGES ON HEATING AND COOLING

PLATE 9

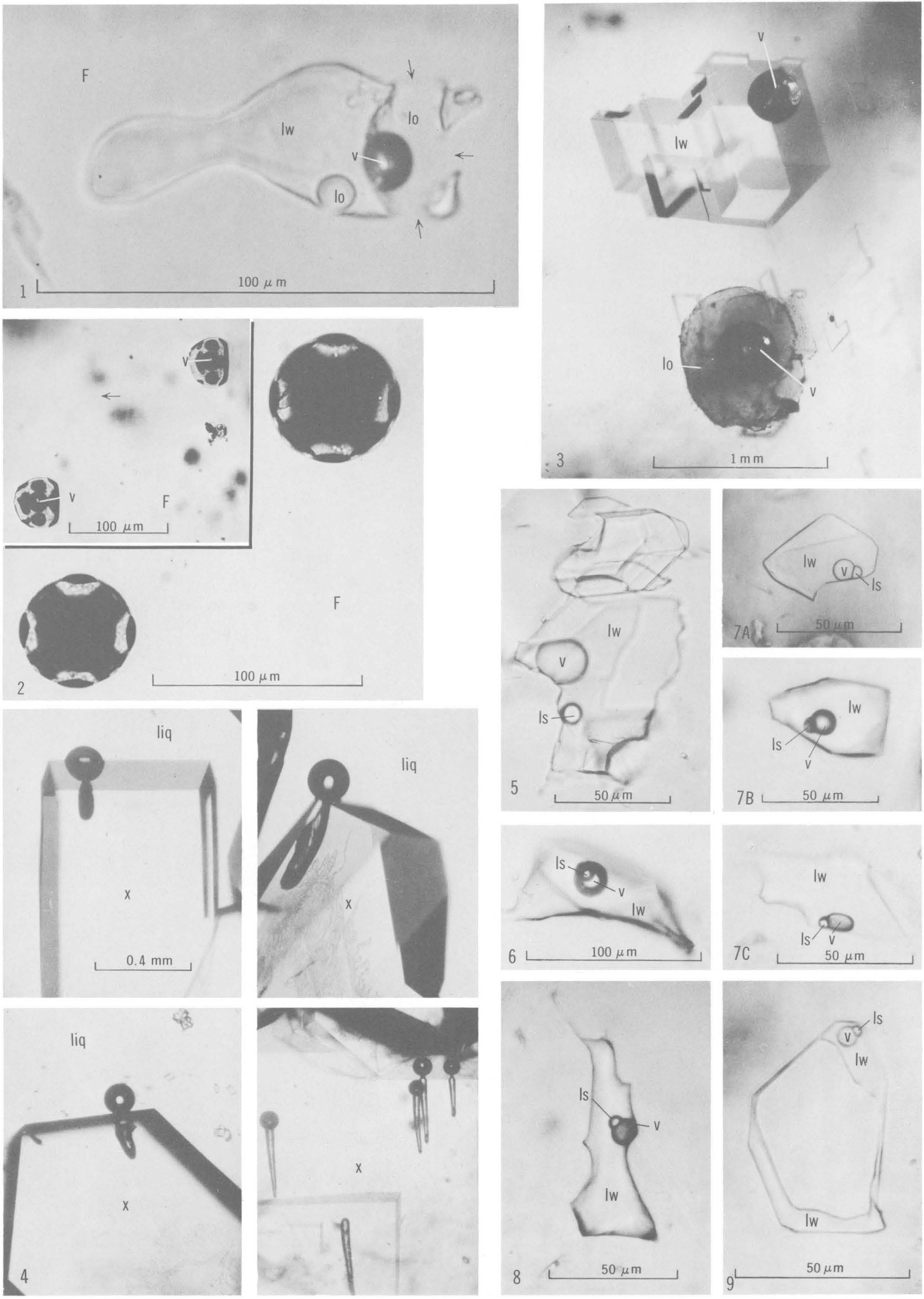
FIGURE 1. Photomicrograph of large primary inclusion in yellow fluorite (**F**) showing dark vapor bubble (**v**), strong brine solution (**lw**), and two globules of oil (**lo**) with an index of refraction almost identical with that of the enclosing fluorite. The oil wets the fluorite host in three zones (arrows) thus isolating two small segments of brine. The oil droplet at the left was not present before freezing in the laboratory; presumably it was dislodged from the main mass by rapid volume changes during the almost instantaneous freezing of the strongly supercooled brine. ER 64–13al, Koh-i-Maran Range, Kalat Division, Pakistan. Sample provided by Omar B. Raup, U.S. Geological Survey.

2. Photomicrographs of primary oil inclusions in fluorite (**F**). These originally homogeneous droplets of oil were suspended in the saline brines from which the fluorite grew and adhered to the surface of the crystal. After trapping, the oil underwent degradation to form a small amount of a dark phase that accumulated on crystallographically controlled parts of the fluorite walls. Shrinkage of the oil after trapping formed the central vapor bubbles (**v**), now containing methane(?) under high pressure. The inset is a cross section of a fluorite cube (**F**), which grew in the direction of the arrow. The two oil droplets first stuck to the growing fluorite crystal along the flat surfaces on the bottom of each inclusion, now coated with dark material. The larger photomicrograph is a plan view taken perpendicular to the cube face of two oil inclusions similar to those in the inset. ER 59–57e, West Green mine, southern Illinois fluorite-zinc district. Sample courtesy of Dr. James Bradbury, Illinois Geoolgical Survey.

3. Photomicrograph of large primary inclusions of colorless brine (**lw**) and yellow oil (**lo**) in color-zoned purple fluorite. These are believed to have retained essentially the shape that they had at the moment of trapping. The brine inclusion (top) represents the covering of a group of negative crystal reentrants on the surface of the growing cube, as are commonly seen on the present surface of such crystals. It is considered primary from its position in the crystal, and not from its shape, as recrystallization can also form such negative crystal faces, although usually only on much smaller inclusions. (See frontispiece, upper left.) The oil was present as a rounded droplet suspended in these brines, which adhered to the growing fluorite surface, and was enclosed by growth of the fluorite without change in shape. Note that there is a larger volume percentage of vapor bubble (**v**) in the oil than in the brine inclusion, although the two inclusions were probably trapped at almost identical temperatures; this results from differences in compressibility and thermal expansion characteristics for the two fluids. The two different apparent radii on the bubble in the brine arise from prism effects at the sloping inclusion walls. Note also that this bubble is actually adhering to the wall (small light-gray oval at right). This only occurs in very strong brines, where the salinity causes gross changes in surface wetting characteristics. Taken in deep purple transmitted light, with added lateral light for reflection from bubbles. ER 59–3, Hill mine, Cave-in-Rock, Illinois.

4. Evidence of trapping of fluid inclusions that are *not* representative of the fluid from which crystal growth occurs. Synthetic sucrose crystals (**x**) growing in saturated water solution (**liq**) at room temperature by slow evaporation of water (all at same scale as upper left photomicrograph). Exsolution of air, as described by Powers (1958), formed gas bubbles which adhered to the growing crystal surface and formed tubular gas-filled inclusions, all of which (in these photomicrographs) are still connected to the surface. (In the lower right photomicrograph the bubbles are in a layer of solution above the crystal, except for the inclusion at the bottom, which lost its bubble.) Some crystals show hundreds of such tubular gas inclusions, sealed off by further crystal growth, and not a single inclusion of the syrupy water solution from which the crystals actually formed. Similar phenomena probably occur in nature, whenever crystals grow from systems of two immiscible fluids such as water plus oils or gases such as CO_2 or steam.

5–9. Behavior of immiscible globules of liquid H_2S in inclusions. In this sample, each inclusion contains liquid water solution (**lw**), a small vapor bubble (**v**), and a small immiscible globule of a fluid that is probably liquid H_2S (**ls**). As the phase ratios of many inclusions are rather uniform (approximately 4:1 by volume, vapor:liquid H_2S), it is assumed that a single, homogeneous fluid was trapped originally and has separated on cooling into three phases. A few such inclusions show separate, spherical (or circular, flattened) globules of vapor and liquid H_2S as in figure 5. This accidental configuration does not have the lowest surface energy. Apparently the system has the lowest surface energy when the H_2S liquid occupies the interface between liquid water and vapor as in figure 7 (Torza and Mason, 1969). In more three-dimensional inclusions, the H_2S globule sticking to the interface may appear, due to foreshortening, to be inside the vapor bubble as in figure 6. In figures 5 and 8, the plane of focus has been adjusted to show that the globule of H_2S liquid has a considerably higher index of refraction than the vapor bubble. Figure 9 shows a somewhat unusual ring-shaped inclusion containing the same assemblage. ER 67–7, yellow quartz, from Xique-Xique, Bahia, Brazil (Johnston and Butler, 1946, p. 615), courtesy of Prof. Earl Ingerson, University of Texas.



EVIDENCE OF PREFERENTIAL WETTING PHENOMENA AMONG IMMISCIBLE FLUIDS IN NATURE

PLATE 10

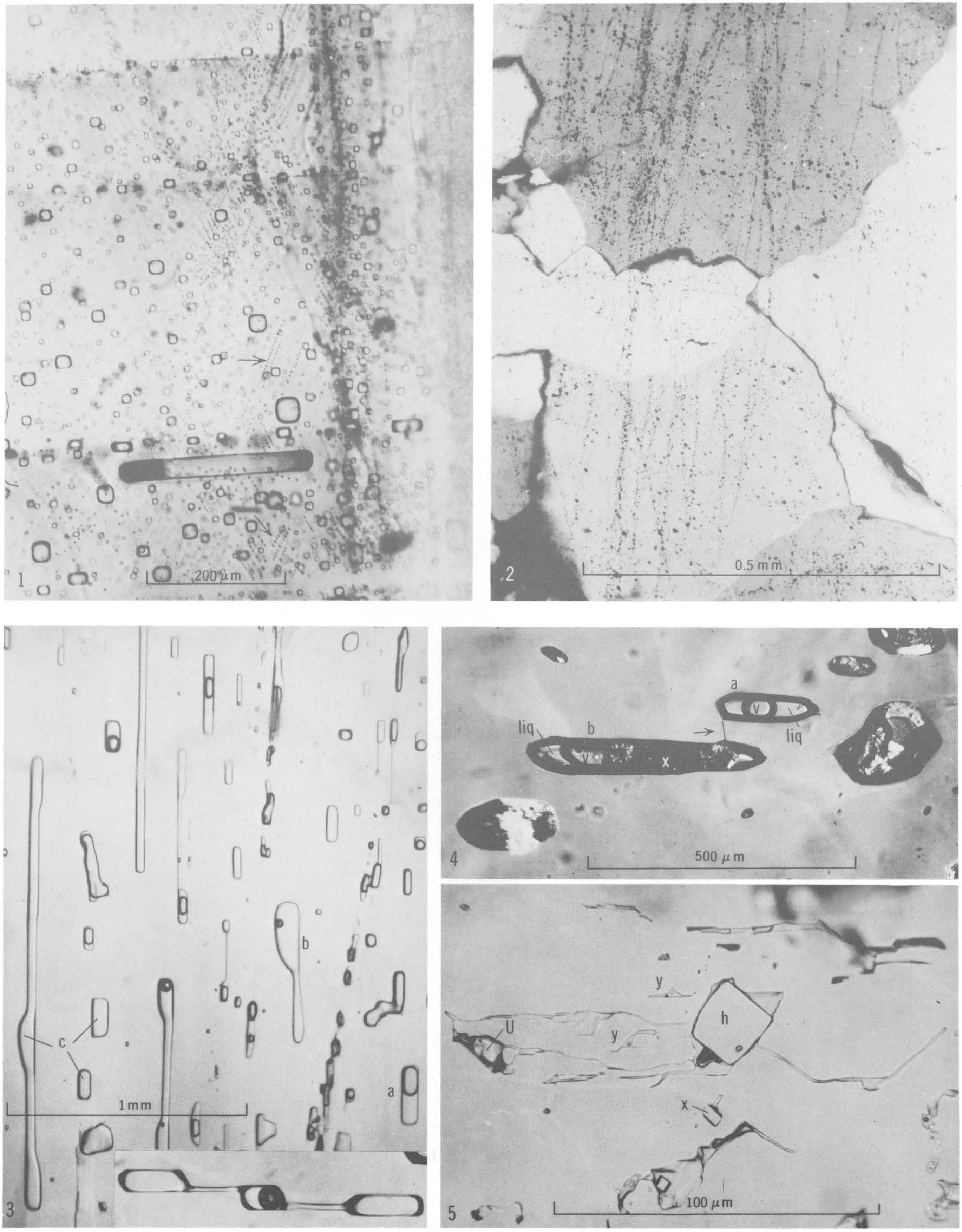
FIGURE 1. Secondary inclusions in healed cleavage cracks in halite that show three features normally classed as evidence of primary origin. These features are: (1) faceted negative crystal shape, (2) occurrence as three sets of mutually perpendicular planes (one in the plane of the photograph), parallel to normal crystal growth faces {100}, and (3) peculiar diagonal rows of tiny, uniformly-sized inclusions (see arrows). The first actually is characteristic of any inclusions, regardless of origin, that have had adequate time to recrystallize; with soluble salts this time can be measured in hours. The second is a logical consequence of the cubic cleavage of halite being parallel to the common gorwth face. The third, although commonly seen to be the result of growth steps on the crystal surface, is believed in this sample to be the result of tear lines (Buerger, 1932b) on the original fracture. The clear zone crosscutting all zones of inclusions at the bottom may be a fracture that healed free of inclusions. Note also that these inclusions are free of bubbles, yet this is in the same sample as in figure 3. Presumably these inclusions formed at a lower temperature. ER 64–112, large halite cleavage mass, Salar Grande, Chile, provided by George Erickson, U.S. Geological Survey.

2. Photomicrograph of typical planes of secondary inclusions in quartz from a gold-quartz vein, Grass Valley, Calif., taken with partly crossed polarizers. Note that many of the planes of inclusions cut across grain boundaries, outlining former throughgoing shear fractures.

3. Photomicrograph of a healed fracture (not parallel to {100}) in halite, showing nonuniform phase ratios caused at least in part by necking down of an original large, thin, flat, secondary inclusion into many smaller inclusions of lesser total surface area. The vapor bubbles formed before and during the necking down, so that individual inclusions now have a wide range of gas/liquid ratios (**a** and **b**) or no gas at all (**c**). As the "gas" in the bubbles is merely water vapor in this sample, the composition of the fluid in all will be essentially the same, but the homogenization temperatures will differ widely. It should be noted that similar variation in gas/liquid ratios can also result from partial leakage of inclusion contents, as is also common in halite. The insert photograph shows a long, tubular, possibly primary inclusion in the same sample, in the process of necking down to form three smaller inclusions, only one having the bubble. Same sample as fig. 1.

4. Photomicrograph of plane of pseudosecondary or possibly primary inclusions in topaz, parallel to {100}, showing results of necking down by recrystallization after daughter minerals have precipitated. Inclusions **a** and **b** were originally a single, larger inclusion, which has since split up, leaving a very thin tube between (arrow) (unretouched). Although both parts contain vapor and liquid, all of the large mass of unidentified, birefringent daughter crystals (**x**) were left in inclusion **b**. The other inclusions in the field all contain the same assemblage of vapor (**v**), liquid (**liq**), and crystals. Coalescence of thin, flat, or long, tubular inclusions by such recrystallization processes commonly leaves small daughter crystals behind, embedded in the host mineral as apparent solid inclusions but frequently connected with a fluid inclusion by almost invisible tubes that may be open or blocked. Transmitted light, partly crossed polarizers. USNM 96595, Rukuba tin mine, Nigeria.

5. Plane of pseudosecondary inclusions in quartz, showing an early stage in the process of necking down after daughter minerals have precipitated. Highly birefringent daughter crystals (**U**) (possibly parisite, see pl. 2, figs. 1, 2) and a large halite daughter crystal (**h**, coated with a film of brine) are nearly isolated from each other and have been isolated from the bulk of the fluid of the inclusion (out of this field of view). Necking down has caused individual parts of the original inclusion to be sealed off at various stages in the process, resulting in another isotropic cube with a coating of liquid (**x**) and two apparently "low temperature" inclusions, with very small vapor bubbles (**y**). ER 63–84Q, Muzo emerald mine, Colombia, courtesy of Banco de la Republica, Bogotá.



SECONDARY INCLUSIONS AND NECKING DOWN OF INCLUSIONS

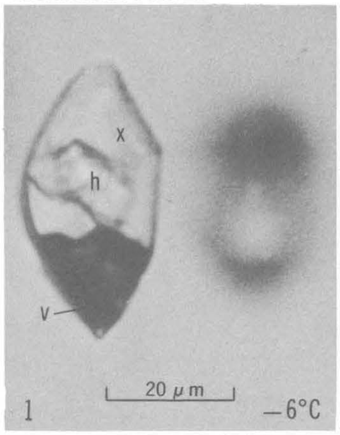
PLATE 11

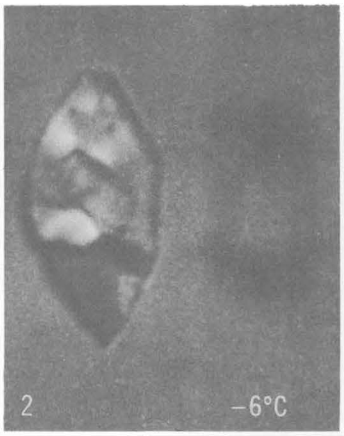
FIGURES 1–4. Serial photomicrographs of an inclusion, taken at the temperatures indicated, to illustrate the identification of halite by the formation of crystals of hydrohalite ($NaCl \cdot 2H_2O$) on cooling. At room temperature the inclusion appears almost identical to figure 4 taken at −1.5°C. It contains (see fig. 4) saturated brine (**liq**), vapor (**v**), and a large daughter crystal (cube) of halite (**h**). After freezing at −78°C, and then equilibrating at −6°C (fig. 1), all available solution reacts with the halite to form a solid mass of crystals of hydrohalite (**x**), causing the "meniscus" between the former liquid and vapor (**v**) phases to be irregular and jagged. A small corroded but unreacted mass of halite remains. The new phase is birefringent, as seen in figure 2 also taken at −6°C, but with partly crossed polarizers. On warming, the hydrohalite decomposes (melts incongruently) to form halite plus solution (fig. 3); the last hydrohalite crystal disappears at −1.5°C (fig. 4). ER 63–133, quartz, from granite block in volcanic breccia, Ascension Island, South Atlantic Ocean (Roedder and Coombs, 1967).

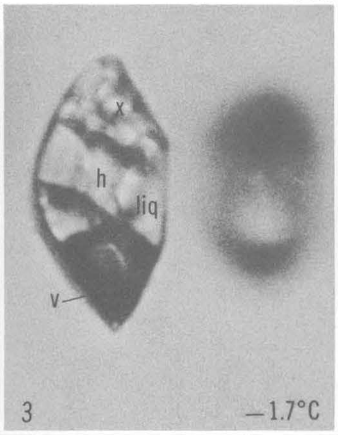
5. Serial photomicrographs of an olivine grain, embedded in oil of $n = 1.64$, to show the use of the crushing stage to prove the presence of highly compressed gases (CO_2) in the inclusions. Pressure was applied, perpendicular to the plane of the photograph, by glass plates. This caused cracking and release of gases between frames **a** and **b**, **c** and **d**, and **e** and **f**. The explosively evolved bubbles come from the inclusions indicated by arrows in frames **a**, **c**, and **e**. One minute elapsed between taking frames **b** and **c**, and between **d** and **e**, permitting most of the gas to dissolve in the index liquid. ER 63–33a, olivine nodule from 1801 Kaupulehu flow, Hualalai, Hawaii (Roedder, 1965d).

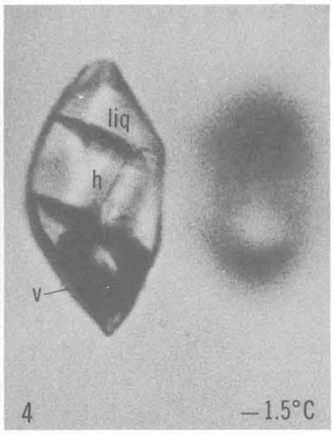
6. Photomicrograph of single-phase inclusions in a large (10 cm) single crystal of melanterite ($FeSO_4 \cdot 7H_2O$), formed as a stalactite in a mine opening by evaporation of mine water, OH vein, Creede, Colo. These contain only the liquid phase (a saturated solution of melanterite) as they formed at essentially surface temperatures. Identification of such single-phase inclusions is sometimes ambiguous, in that the relief shown by negative crystals filled with liquid (as here) may be almost duplicated by thinner, gas-filled inclusions. ER 65–123.

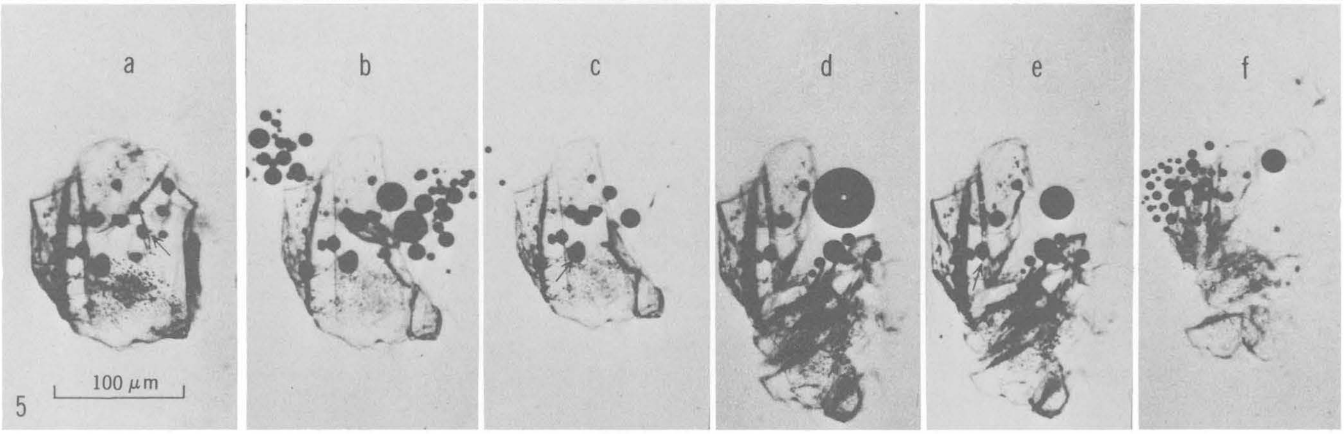
7, 8. Photomicrographs of a large primary fluid inclusion in sphalerite, illustrating one of the hazards in estimating the volume of a gas bubble and hence the degree of fill. In figure 7, the plane of focus cuts through the center of the bubble, and two apparently different bubble radii are visible. The volume of the bubble calculated from the larger radius is over twice that calculated from the smaller radius. The two apparent radii are a result of the shape of the inside of the inclusion cavity. Over the upper part of figure 7, the wall consists of a relatively flat negative crystal facet, set perpendicular to the line of sight (see fig. 8, where the plane of focus is on this facet); this yields the larger, essentially true image. Over the lower part of the bubble the curving surface of the sphalerite wall (toward the observer) acts as a strong negative lens, reducing the apparent size of the bubble. As most inclusions have such curved inner surfaces, estimates of the volume of the gas bubble—or any other phase within the inclusion—will be low. The effect is particularly large for a mineral with a high index of refraction, such as sphalerite ($n = 2.368$). Synthetic sphalerite crystal, grown in 67 hours by a thermal gradient technique, at about 430°C and 6,500 psi (pounds per square inch), in a 10 molal NaOH solution. Sample grown and provided through the courtesy of Steven D. Scott, Pennsylvania State University, his run 195–2.

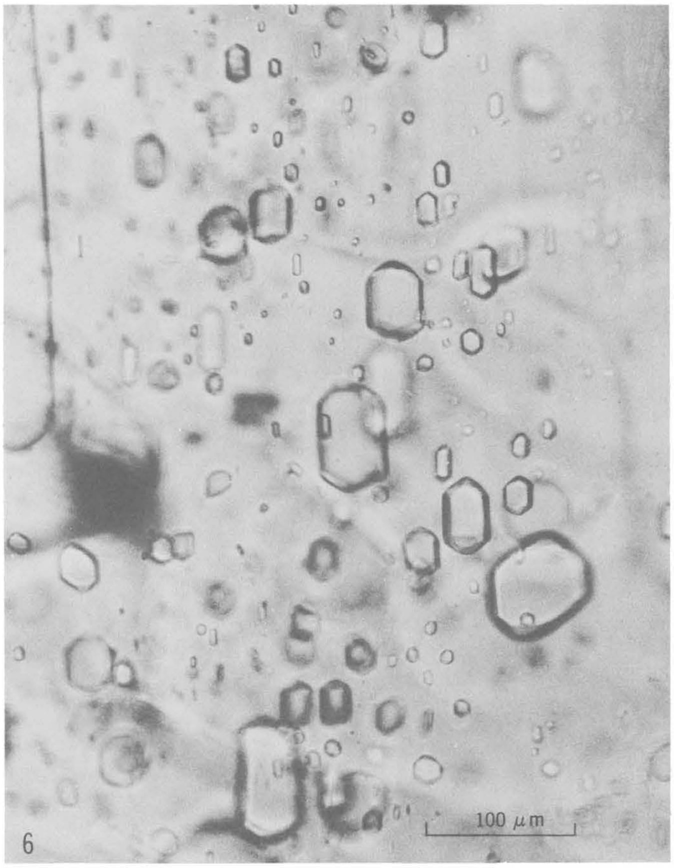


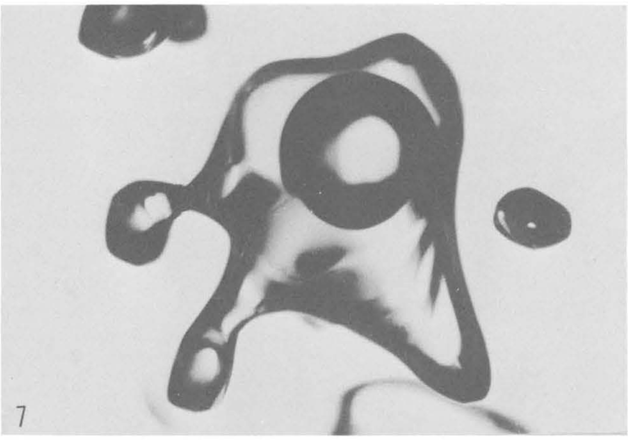


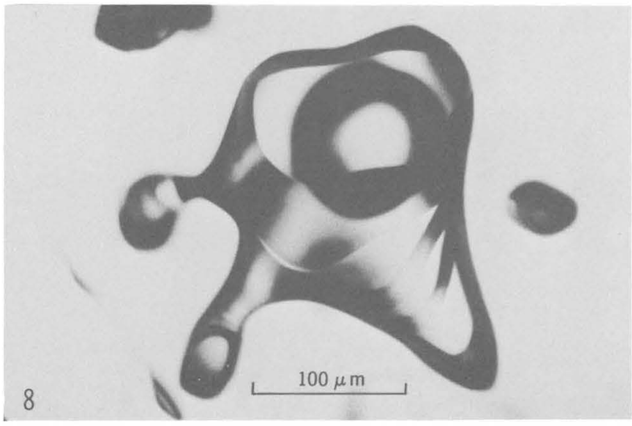












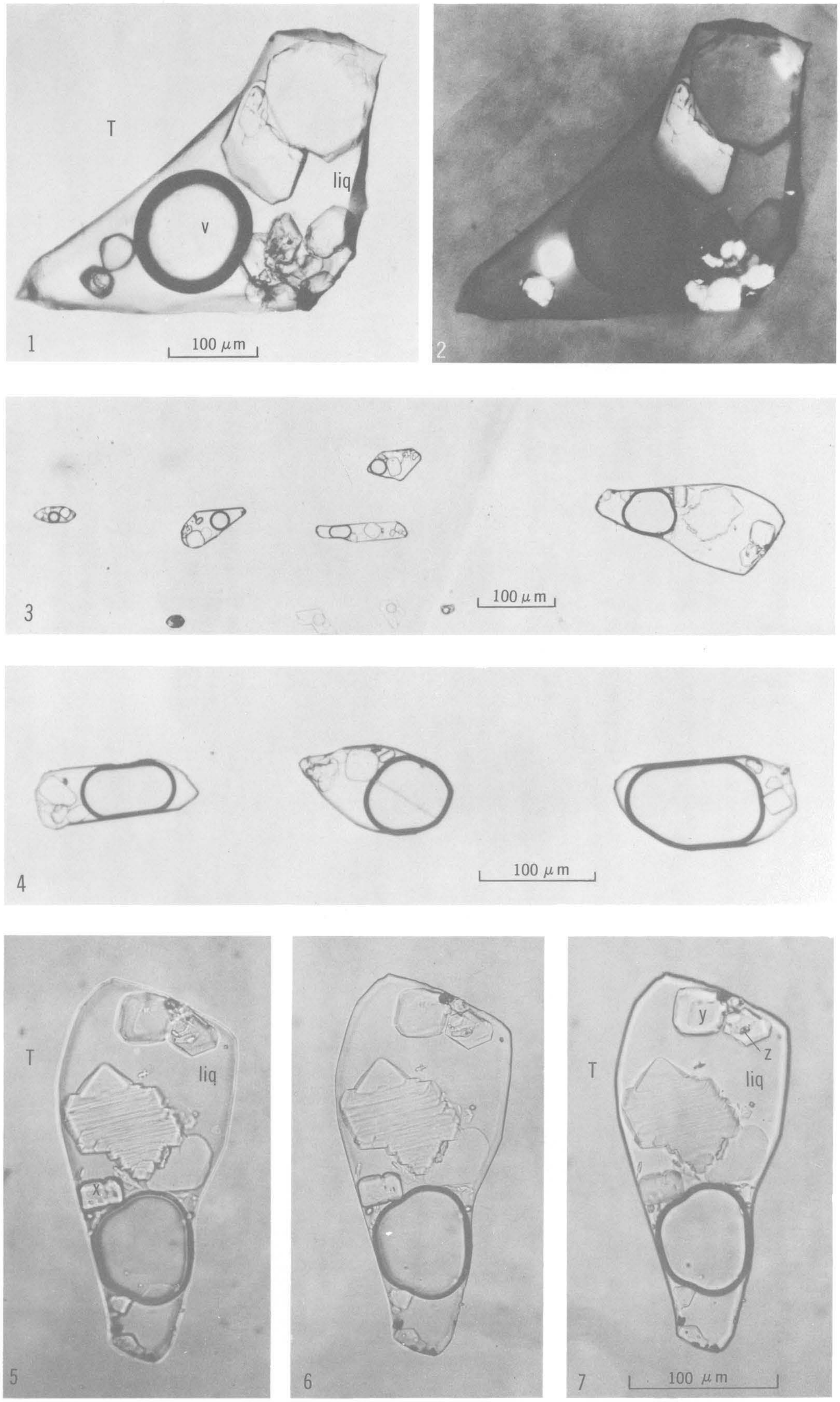
INCLUSION MICROSCOPY TECHNIQUES AND PROBLEMS

PLATE 12

FIGURES 1, 2. Photomicrograph of a large multiphase inclusion in plain light (fig. 1) and with almost crossed polarizers, set to place the enclosing topaz (**T**) almost at extinction (fig. 2). This and other inclusions in the sample contain at least 16 daughter minerals, presumably all different phases, plus liquid (**liq**) and vapor (**v**). Ten daughter crystals are seen to be birefringent (fig. 2); presumably some of those that appear isotropic are not but merely have their extinction positions parallel with those of the enclosing topaz. There may be as many as five opaque phases. All photomicrographs on this plate are from a single cleavage flake approximately 2 cm in diameter, loaned by Dr. Bernard Poty, Centre de Recherches Petrographiques et Geochimiques, 54-Vandoeuvre, France. All the inclusions are strongly flattened parallel to the (001) cleavage and may be primary or pseudosecondary in origin. Lemmlein, Kliya, and Ostrovskii (1962) reported on the homogenization of similar inclusions in topaz from Volynia (see pl. 8, figs. 2–6) and record the presence of large daughter crystals of quartz and muscovite, lesser cryolite, and still smaller amounts of various fluorides and chlorides of Na, K, and Ca. Lyakhov (1966) gives X-ray powder diffraction data on six of the 14 different solid phases he extracted from inclusions in morion from these same pegmatites, including hydrous ferrous chloride, $FeCl_2 \cdot 2H_2O$. Some stages in the solution of the daughter minerals are shown on plate 7, figure 12.

3, 4. Photomicrographs of two planes of inclusions, only a few millimeters apart, that have trapped two entirely different densities of fluids. In each plane, all inclusions apparently have a uniform ratio of phases, and all appear to have the same phases, but the ratio of gas bubble to other constituents differs grossly between the two planes. The small but plainly visible differences in gas bubble ratio between adjacent inclusions (particularly fig. 4) may be only apparent and come from irregularities in the third dimension, or they may be real and stem from bubble nucleation before necking down occurred. The differences between figures 3 and 4 may be a result of differences in the confining pressure at the time of trapping.

5–7. Detailed photomicrographs of one inclusion from figure 3, taken with the plane of focus at three different levels. In figure 6, the plane of focus cuts through most of the phases in this very flat inclusion. In figure 5 the plane of focus is raised, and in figure 7 it is lowered relative to figure 6. Note that the movement of the Becke lines in figure 5 shows that many of the daughter minerals are higher in index then the enclosing liquid (**liq**), and some are very much higher (**x**). In figure 7, however, note that the two larger crystals at the top (**y** and **z**) have an index of refraction very appreciably less than that of the enclosing liquid (**liq**) which, in turn, has an index of refraction less than the enclosing topaz (**T**). These specific daughter minerals have not been positively identified, but various low index fluoride minerals such as cryolite ($n = 1.34$) and avogadrite ($(K,Cs)BF_4$, $n = 1.32$) have been reported in the extensive Russian work on such daughter minerals.



MULTIPHASE INCLUSIONS IN PEGMATITIC TOPAZ, VOLYNIA, U.S.S.R.