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Supplementary components and operation of the U.S.

Geological Survey gas-flow heating/freezing stage

By

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Abstract

The supplementary components and operating procedures for the U.S. Geological Survey gas-flow heating/freezing stage have been developed to enhance the rapid, accurate measurement of fluid inclusions homogenizing between -150°C and 500°C. Doubly polished mineral slabs as thick as 2mm are carefully photographed at several magnifications to provide the optimum samples for large-scale growth history-fluid inclusion studies such as those pursued in our laboratory. Long-focal-length condensers and fiber-optic illuminators provide optimal viewing conditions and the large sample chamber volume saves both documentation and sample-changeover time. Nitrogen gas, chilled by passage through a tank of liquid nitrogen, and air, heated by passage through a glass-encased, nichrome-wire heating coil, are used for freezing and heating runs, respectively. Precise control of temperatures and heating rates, as well as careful stage and thermocouple calibration, make it possible to achieve accuracies of +2.0°C for heating runs and +0.2°C for freezing runs. The use of gas to control temperature (1) permits rapid accumulation of abundant, accurate data, (2) ameliorates thermal gradient problems so often encountered in stages using convection and/or conduction for heat transfer, and (3) permits the use of a cyclical procedure to measure temperatures that would otherwise be unmeasurable.

INTRODUCTION

A novel gas-flow fluid inclusion stage designed at the U.S. Geological Survey (Werre, Bodnar, Bethke, and Barton, 1979) permits rapid measurement of both the temperature of homogenization (Th) and the depression of the freezing point (that is, the melting of ice; Tm) of fluid inclusions in relatively large samples. The use of flowing gas to cool and heat the fluid inclusions allows rapid changeover between heating and freezing modes and also minimizes thermal gradients in the stage.

The purpose of this report is to describe the supplementary components and the overall operation of the system as set up in a U.S. Geological Survey laboratory in Reston. The description of the procedures we have developed for measuring Th and Tm illustrates some of the advantages of the gas-flow stage and documents ways to circumvent or minimize the problems we have encountered. We also describe the key steps in sample preparation and documentation that we have found to yield the most useful samples and to enhance efficient data collection and recording. Figure 1 shows the entire system and Table 1 lists the specific equipment used in our laboratory.

Paul B. Barton, Jr., and Tom Casadevall made significant contributions to the development of this system for studying fluid inclusions.

SAMPLE PREPARATION AND DOCUMENTATION

Doubly polished mineral slabs are necessary to provide the optimum optical conditions for fluid inclusion studies. Singly polished slabs, although requiring less preparation time, provide such poor visibility that greatly increased measuring time, or the inability to measure the inclusions at all, negates the time saved in the preparation step. Many inclusions measurable in a doubly polished slab are unmeasurable or barely detectable in a singly polished slab.

The slabs are mounted for preparation on glass slides with a thermoplastic cement. When the polishing is complete, the samples can be easily removed from the slide by soaking overnight in methanol or acetone.

The prepared samples may be as thick as ≈2mm when used in the standard stage. Thicker sections, sometimes required for large inclusions, significantly disrupt gas flow in the shallow stage. This disruption necessitates the use of a high-volume modification to the stage, which sacrifices some precision of temperature estimation. This high-volume modification restricts the user to lower power microscope objectives having working distances greater than 12mm (for example, Leitz UMK 32X; magnification 20X when used in the normal orthoscopic mode), but this limitation is not a problem when large inclusions are examined.

After sample preparation, the documentation process begins. A series of photographs at various magnifications is used to (1) document the relationship of fluid inclusions to indicators of growth history such as color banding and ghost crystal faces, (2) insure recognition of particular fluid inclusions at a later date, and (3) document the criteria used to classify inclusions as primary, pseudosecondary, and secondary.

The entire thin section is first photographed with either a stereo microscope-camera apparatus or some other low magnification photographic system for Polaroid black and white photographs, and/or a slide copier for 35mm color or black and white film. Individual or groups of fluid inclusions are then identified, classified as to origin, and assigned to

Table 1. Equipment used in the U.S. Geological Survey laboratory in conjunction with the gas-flow heating/freezing stage

Item	Trade name	Identification	Supplier of our equipment
Thermoplastic cement	Lakeside 70C		Hugh Courtright & Co., Ltd. 6135 W. 65th St. Chicago, Illinois 60638
Stereo microscope-camera apparatus	Wild stereo microscope	Model M7S	Wild-Heerbrugg Ltd. CH-9435 Heerbrugg, Switzerland
Low magnification camera apparatus	Leitz Aristophot with macro-dia attachment		E. Leitz, Inc. Rockleigh, N. J. 07647
Petrographic microscope-camera system	Leitz Orthoplan microscope with Leitz camera system		"
Slide copier	Honeywell Repronar	Model 805A	Honeywell, Inc. Denver, Colo.
Diamond-embedded wire saw	Diamond microwire saw		Gryphon Corp. 101 East Santa Anita Ave. Burbank, Calif. 91502
Dental drill			Kerr Manufacturing Co. 28200 Wick Rd. Romulus, Mich. 48174
Diamond cutting wheels	Starlite line		Star Dental Mftg. Co., Inc. Philadelphia, Pa. 19139
Fiber optics ring illuminator	Intralux	Model 150H	American Volpi Corp. 26 Aurelius Ave. Auburn, N. Y. 13021
Fiber optics illuminator with single flexible light guide		Model 11-80	American Optical Corp. Fiber Optics Division Southbridge, Mass. 01566

Table 1. Equipment used in the U.S. Geological Survey laboratory in conjunction with the gas-flow heating/freezing stage

Item	Trade name	Identification	Supplier of our equipment
Gas pressure regulators Building air Tank nitrogen		E11-9-N570M E19-7-125A03	Air Products & Chemicals, Inc. Specialty Gas Department P. O. Box 351, RD#2 Tamaqua, Pa. 18252
Single tube flowmeters (2 needed)		E21-C-150MM12	"
Flowmeter tubes		E29-R-150MM4 E29-G-150MM5	"
Gas heater	Flameless torch	SGH110226	Sylvania Emissive Products Box 220 Exeter, N. H. 03833
Variable voltage transformer	Powerstat		The Superior Electric Comp. Bristol, Conn.
Thermocouples Chromel/alumel (O.D. ≈ .02") Iron/Constantan	Type K Type J	SCASS-020G-6 SICSS-020G-6	Omega Engineering, Inc. 1 Omega Dr. Box 4047 Stamford, Conn. 06907
Digital temperature indicator	Instrulab	DTI 5000	Instrulab, Inc. 1205 Lamar St. P. O. Box 426 ND STA. Dayton, Ohio 45404
Digital voltmeter	Dana	6900	Dana Laboratories, Inc. 2401 Campus Dr. Irvine, Calif. 92713
Electronic ice point reference cell		Model TRC	Omega Engineering, Inc. (See address above)
Thermocouple calibrator	Temptron freezing point standards	Models 503 & 505	Temptron Engineering, Inc. 8744 Remmet Ave. Canoga Park, Calif. 91304

Table 1. Equipment used in the U.S. Geological Survey laboratory in conjunction with the gas-flow heating/freezing stage

Item	Trade name	Identification	Supplier of our equipment
Temperature recording device	Datel miniature digital panel printer	DPP-7A1	Datel Systems, Inc. 1020 Turnpike St. Canton, Mass. 02021
Constant voltage transformer	Sola		Sola, Unit General Signal 1717 Busse Rd. Elk Grove Vill., Il. 60007
Liquid nitrogen Dewar		Model LAB-10	Minnesota Valley Engineering New Prague, Minn. 56071

a "stratigraphic" zone or otherwise related to growth history wherever possible. Higher magnification photographs are then taken of the individual groups of fluid inclusions with the petrographic microscope-camera system (either 4"x5" black and white Polaroid, 35mm color or black and white, or both), and the locations of these inclusions are marked on the low-magnification photographs.

The slab is then removed from the glass slide and cut into chips ($\leq \approx 1$ cm^2; the diameter of the stage is 22mm) in such a way as to minimize the breakage of fluid inclusions. A diamond-embedded wire saw (wire diameter .008") cuts very slowly, is very gentle, and creates only a narrow kerf (<.25mm). This wire is vertically mounted between a counter-weighted wire pulley above and a rotating threaded drum below. The cutting motion is provided as the wire is continuously wound off one end of the drum and onto the other end. In addition to the gentleness, a second advantage of this type of saw is that it permits cutting along a curved boundary because the wire is very thin and flexible. For samples not requiring such precise cutting, a "dentist's" drill with diamond cutting wheels is much faster.

Because flowing gas is used to transfer heat, the samples must be stabilized within the stage. For low- to moderate-temperature runs, samples may be epoxied to glass cover slips (25mm diameter) after final cutting; however, many epoxide formulations darken significantly when heated to >240°C for long periods of time, causing serious visibility problems. We have found that, in most cases, the chip can be stabilized by pressure from the shielded thermocouple.

COMPONENTS OF THE SYSTEM USED TO STUDY FLUID INCLUSIONS
Magnifying/Illuminating System

Illumination

The heating/freezing stage is placed on the stage of a petrographic microscope whose standard illumination system is often used. A foot switch permits easy activation of this light source, as needed. Since standard high-power microscope condensers have too short a working distance to use with this stage, the illumination problems accompanying fluid inclusion studies are much more severe than those encountered during normal transmitted- and reflected-light microscopy. For example, the edges of the fluid inclusions often appear dark because of total reflection of the rather collimated incident light. The vapor bubbles and ice crystals typically move to the edges just before homogenization and can seldom be illuminated by merely increasing the intensity of the normal substage illumination. Also, inclusions having a negative crystal shape commonly have faces oriented in such a way as to cause total reflection of the incident light. The severity of these problems increases as the difference between the refractive index of the inclusion fluid and that of the host mineral increases. These problems are exacerbated because the sample, inside the heating/freezing stage, lies above the plane of the microscope stage thus precluding the use of a short-focal-length condenser cap. Long-focal-length condenser caps, or long-working-distance objectives used in place of the condenser cap, can provide Koehler illumination, but the angle of convergence is then much less than that when the standard 2mm-focal-length condenser cap is used. Consequently, problems of shadowing due to total reflection are only partially eliminated. Use of fiber-optic illuminators held at higher angles to the optic path provides the best solution to total-reflection problems.

Two types of fiber-optic illuminators can also be used to light dark fluid inclusions that would otherwise be unmeasurable. The first type, a ring illuminator (Figure 1, #5 and Table 1), is made to fit around a long-focal-length microscope objective attached to the substage assembly in place of the swing-in condenser cap. It provides light entering at ≈45° to the optical axis from four ports equally spaced around a metal ring at the end of a flexible light guide. The ring illuminator can be used alone or in combination with the microscope illumination system. The second type of fiber-optic illuminator, an illuminator having one flexible light guide, is hand-held beneath or above the stage at the desired angle and can illuminate many inclusions that the ring illuminator does not. We have tried several methods of fixing this illuminator at the proper position to provide the best illumination; although all are easier than hand-holding, none are as effective.

A supplementary infrared filter is necessary in many optical systems to avoid heating of the fluid inclusions by the illuminator. This heat can increase the temperature in the inclusion by 0.5°C or more owing to adsorption of infrared radiation by the water. This increase is especially critical during freezing runs when temperatures are measured to a few tenths of a degree. The infrared effect is even more noticeable in CO_2 inclusions than it is in water inclusions. Roedder (1965) found that this effect could cause measuring errors as great as 1.28°C in the Th of CO_2 inclusions. The fiber-optic ring illuminator has an effective filter built in, and we have adapted an extra filter to the standard microscope illuminator to minimize the heating effects when using that illumination system.

High-power objectives

We use a Leitz 50X UTK universal-stage objective for maximum magnification. This has a magnification of 32X when used without the U-stage hemispheres, as in our application. This objective was chosen because, at the time the stage was designed, it had the longest working distance (6.3mm) of any available high-resolution objective. The dimensions of the stage were designed for the working distance of this objective.

A cooling coil has been designed for this objective because its working distance places it very close to the upper window of the heating stage and it could be severely damaged by long exposures to high temperatures. We use the cooling coil when operating the stage at temperatures much greater than 250°C. The coil is made of copper tubing (O.D.=3mm) that is wrapped to fit snugly around the objective (Figure 2, #2). Water from an overhead reservoir flows, by gravity feed, through plastic tubing into the coil and down to a catch basin on the floor. This catch basin is fitted with an electric pump, activated by an automatic float switch, which pumps the water back up to the overhead reservoir.

Optimal viewing conditions (that is, the best resolution) are achieved if the condenser and the objective(s) have the same numerical aperture.

Gas-Flow System

A photograph of the gas delivery/control system and a schematic diagram of its operation in both the heating and freezing modes are shown in Figures 1 and 3. We use building-supplied compressed air for heating runs and purified nitrogen gas for cooling, although nitrogen can be used for both modes if compressed air is not available. The rate

of gas flow into the stage is controlled by two 150mm single-tube flow-meters. The two flowmeters are of different diameters to provide flow rates of 873 to 8,730 cc/min and 2,322 to 23,220 cc/min. The flowmeters are arranged so that they may be operated in parallel for freezing runs, in which cold nitrogen gas and room-temperature nitrogen gas are mixed to control the temperature.

Heating Procedure

For heating runs, a single-stage line regulator reduces the pressure of compressed building air from 80 psi to about 15 psi; the air is then passed through the control/delivery system and into the heating stage. Only one flowmeter is used for heating (Figure 3). The flow rate affects several aspects of the measuring procedure: higher flow rates decrease the thermal gradients within the stage but require more power for a given temp-erature and cause sample instability. We have found the optimum flow rate to be about 15,000 cc/min. For high temperature runs (>500°C), nitrogen gas should be used to prevent oxidation of the Invar alloy stage components.

Freezing Procedure

During freezing runs, purified nitrogen gas is used instead of com-pressed air. The flowmeter having the larger diameter controls the flow of the tank nitrogen that is fed directly into the stage; the smaller dia-meter tube controls the flow of tank nitrogen to be chilled into the Dewar (Figure 3). The nitrogen enters the Dewar through a length of copper tubing (O.D.≈0.4 cm.), coiled at its lower end, and the chilled nitrogen gas passes out of the Dewar through another length of tubing, which is also coiled at the end. Both coils are immersed in the liquid nitrogen. For ease of operation, these two tubes and coils are fastened together (Figure 4). The gas flow through the liquid nitrogen Dewar is designed to provide nearly constant temperature chilled nitrogen regard-less of the level of liquid nitrogen in the Dewar. Foam and insulating tape wrapped around the plastic tubes that run from the liquid nitrogen to the stage reduce the flow of room heat into the tubes (Figure 1). Good insulation around these tubes is critical.

After the inclusion fluid has been frozen by means of chilled gas only, warming of the fluid is controlled by mixing chilled gas and room-temperature nitrogen gas before passing it through the heating system and into the stage. The optimum flow rate is about 10,000 cc/min (3,000 cc/min of chilled and 7,000 cc/min of unchilled gas). This mixing of chilled and unchilled gas provides the primary and gross control of the warming or cooling rates and of the temperatures; precise control re-quires the use of the heating coil, as discussed below.

The cold nitrogen gas exiting from the stage is split into two flows, which are directed onto the top and bottom glass plates of the stage to prevent condensation or frosting on the glass plates (Figure 5). A 38-liter, 175-kg/cm^2 cylinder of nitrogen gas and a 10-liter Dewar of liquid nitrogen will supply enough cold gas for about 16 hours of freezing runs.

As currently designed, the system can produce temperatures as low as -150°C in about 10 minutes by means of just chilled nitrogen gas.

Electrical System

Heating Procedure

During heating runs, the air passes from the flowmeter through a gas heater (a coil of nichrome wire within a silica glass tube) encased in insulating material, which is inserted directly into the side of the heating stage (Figure 2, #1). This heating coil controls the temperature of the gas stream entering the stage, either to maintain a constant sample temperature or to establish a desired heating or cooling rate. The gas heater is regulated by two variable voltage transformers, wired in series, which control current flow to the wire heating coil. This series arrangement provides fine control of the voltage because the first transformer controls the fraction of total possible voltage (that is, 115 V) passing into the second transformer, which in turn controls the percentage of that fraction of 115 V which passes into the heating coil. The series arrangement of the variable transformers is not important in heating runs, where a relatively high percentage of the line voltage is used, but for Tm determinations, where the heater is used to warm the chilled nitrogen gas, only a small percentage of the line voltage is used, and the series arrangement allows very precise control of the warming rate. By means of the specified heater (Table 1), temperatures as high as 550°C can be reached with 115-V transformers, but a 220-V transformer and line is required for higher temperatures.

Freezing Procedure

The electrical system used for freezing runs is the same as that used for heating runs, except that the heating coil functions mainly as a mechanism for the fine control of temperatures and heating rates and is complementary to the coarse control provided by gas mixing.

Temperature Measurement and Recording System

Temperature measurement

The heating stage has been carefully calibrated and the pattern of thermal gradients within the stage has been precisely mapped at various temperatures. Therefore, when one thermocouple is located in the center of the stage, temperatures throughout the stage can be estimated by means of the thermal gradient maps. We use either chromel/alumel or iron/constantan thermocouples. In our setup the chromel/alumel thermocouple, which we use most frequently, is attached to a 6-channel digital temperature indicator reading in tenths of degrees (Figure 1, #7), and the iron/constantan thermocouple is connected to a digital voltmeter reading in millivolts. An electronic ice point reference cell is used with the voltmeter (neither of these is shown in Figure 1).

The chromel/alumel and iron/constantan thermocouples were calibrated in a thermocouple calibrator at the melting points of zinc (419.6°C) and tin (231.9°C). These calibrations showed that at 419.6°C, the chromel/alumel thermocouples deviated by 0.4°C to 1.4°C (average, 1.0°C) and at 231.9°C, they deviated by 0.0°C to 0.8°C (average, 0.4°C) from the reference temperature. The chromel/alumel thermocouples were also calibrated in a pure water-ice bath, in several ice-brine mixtures against a precision

mercury thermometer, and at the melting point of mercury (-38.87°C). In the ice bath, the thermocouples deviated by 0.0°C to 0.4°C (average, 0.2°C). At the melting point of mercury, the thermocouples deviated by 0.0°C to 0.6°C (average, 0.3°C). The amount of deviation varies slightly with the channel of the temperature indicator to which the thermocouple is connected; therefore, data are collected by means of the most accurate combination of thermocouple and channel, and the necessary correction is applied.

Our digital temperature indicator is made to operate only with chromel/alumel thermocouples, which may have hysteresis problems due to order-disorder annealing at high temperatures (Kollie and others, 1975). For that reason, either, iron/constantan thermocouples should be used for measurements of Tm or one chromel/alumel thermocouple should be reserved for freezing runs and never altered by heating to high temperatures.

Temperature Recording

A printer is connected to the digital temperature indicator and, when activated, records on paper tape the temperature indicated on the display (Figure 1, #8); then, the analyst doesn't need to look away from the inclusions being measured. The buss-interface to connect the printer to the digital temperature indicator was fabricated for us by the Instrulab company which supplied the temperature indicator. A footswitch, of the type mentioned before for activating the light source, can also be used to hold the display at the temperature indicated at the instant when the switch is pressed. This is available as a special-order option with the Instrulab temperature indicator and is very useful if the printer is not used.

MEASURING PROCEDURES

Careful control of heating rates is the critical factor that insures rapid, accurate measurements. During heating and freezing runs, the rate of gas flow and the gas temperature are regulated 1) to control heating rates properly, 2) to reduce thermal gradients, and 3) to prevent blowing an unsecured sample around in the stage. A slow rate of temperature increase (no greater than 0.2°C/min, when within a few degrees of the Th) must be maintained to minimize thermal conductivity problems that result in Th and Tm measurements that are higher than the true values. Some initial experimentation with the rate of gas flow, the mixing of room-temperature and chilled nitrogen gas, and the current flow to the gas heater was necessary to master effective control of heating rates.

Occasional variations in building voltage and lack of precision in the transformer dials can increase the difficulties of precise temperature control. These problems are especially critical at certain times, such as near the Th and Tm. An AC line regulator (that is, constant voltage transformer) greatly reduces the effects of variations in building voltage, which in Reston are sizeable enough to be bothersome. Also, a voltmeter or ammeter in the circuit would enhance the reproducibility of current settings and therefore heating rates; however, we have not yet introduced one into our system.

Rapid freezing of the fluid is accomplished by a flow of chilled nitrogen gas, without mixing and without heating. Decreasing the temp-

erature in the stage to -100°C will freeze most water-dominated fluids; this can be achieved within about 5 minutes. After freezing, room-temperature gas is mixed with chilled gas to heat the sample quickly to within about 10°C of the freezing temperature (that is, until the ice be-gins to break up significantly). When the temperature rises to within a few degrees of the freezing temperature, the flow of room-temperature gas is further decreased to slow the heating rate to the desired value; however, a minimal flow of nitrogen gas must be maintained to prevent the formation of frost on the outer glass windows of the stage. During the critical few minutes before last melting, a little power is supplied to the gas heater for fine control. This yields a faster and more precise response than can the gas mixing mechanism alone.

Problems with visibility of vapor bubbles in dark inclusions and of moving ice fronts in many inclusions often necessitate use of a cycling procedure to measure Th and Tm. The physical basis for this cycling pro-cedure is the significant undercooling required to renucleate vapor bubbles and ice crystals once they have been eliminated by heating to the Th and Tm. If the inclusion is heated or allowed to warm to a temperature just below the Th or Tm, respectively, and rapid cooling is initiated by turning the transformer power off or increasing the flow of chilled nitrogen gas, then the vapor bubble or ice crystals will gradually in-crease in size as cooling proceeds. If, however, the Th or Tm is slight-ly exceeded before rapid cooling is initiated, then the vapor bubble will suddenly reappear (often jumping all over the inclusion when it does) at ⩾30°C below the Th, or the fluid in the inclusion will dramatically and instantaneously refreeze at ⩾20°C below the Tm.

For example, if only one small corner of a dark fluid inclusion can be illuminated, chances are the last traces of the vapor bubble or ice will not stay in that small visible area and it will not be possible to deter-mine their temperature of final disappearance. Therefore, a cycling procedure using these dramatic renucleation events may be the only way to measure such dark inclusions. The sudden reappearance of the bubble or refreezing of the fluid upon cooling is usually visible throughout the inclusion and is therefore detectable, even if only a small area of the inclusion can be illuminated. The procedure involves heating the fluid inclusions to successively higher temperatures before initiating rapid cooling, in order to determine the temperature at which the vapor bubble or ice crystals dramatically reappear instead of gradually growing back. By incrementally changing the final temperature before "cooldown", the Th and Tm can be bracketed within the desired precision range. Each cycle of heating followed by rapid cooling can be done in 5-10 minutes.

ADVANTAGES OF SYSTEM

The gas-flow fluid inclusion measurement system has several signifi-cant advantages over commercially available stages. Firstly, rapid, pre-cise measurements of inclusions with Th up to 500°C permit accumulation of abundant, accurate data in a short time. Secondly, the large viewing area saves both documentation and sample-changeover time. Thirdly, the use of gas to control temperature helps to ameliorate the thermal gradient problems so often encountered in stages using convection and/or conduction for heat transfer. Fourthly, the rapidity with which the temperature of the sample can be changed permits use of a cycling procedure to measure

the Th and Tm for inclusions that would otherwise be unmeasurable. Even
for fluid inclusions without visibility problems, the precision of
measurement is improved by such cycling, and the rapid response allows
proof of reversibility during heating and freezing runs. Finally, the
use of a single stage for both heating and freezing runs permits rapid
changeover from one mode to the other without disassembly or rearrange-
ment. This thermometric measurement system, along with the documentation,
sample-preparation, illumination, and calibration procedures that we
have developed, is ideal for detailed growth-zone by growth-zone studies
of fluid inclusions from large numbers of mineral specimens.

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Figure Captions

Figure 1 Photograph showing the components of the system used to analyze fluid inclusions: cylinder of nitrogen gas with pressure regulator (1), single-stage pressure regulator (2), gas-flow control panel with transformers (3), petrographic microscope with heating/freezing stage (4), fiber optic light source (5) and ring illuminator (6) in place on the substage assembly, digital temperature indicator (7) and temperature recording device (8). The liquid nitrogen Dewar (9) is visible under the counter. The Polaroid camera apparatus (10) is attached to the top of the microscope. Note the tubing, which is wrapped with insulation, leading from the Dewar to the stage.

Figure 2 Close-up photograph of the heating/freezing stage showing the heating coil (1) encased in insulating material inserted in the left side of the stage and the coil of copper tubing wrapped around the high-power objective to protect it at temperatures ≥250°C (2). The thermocouple (3) is inserted in the right side of the stage.

Figure 3 Schematic diagram of the air/nitrogen flow control manifold. The coils of tubing shown inside the Dewar to the right of the figure are shown in detail in Figure 4. The openings of the two tubes are always above the level of liquid nitrogen in the Dewar.

Figure 4 Photograph showing the copper coils that carry room-temperature nitrogen gas into the Dewar of liquid nitrogen (1) and carry chilled nitrogen gas (2) out of the Dewar and into the fluid inclusion stage.

Figure 5 Photograph showing the T-shaped tube assembly used to direct the exiting cold gas onto the upper and lower glass plates of the heating/freezing stage in order to prevent frosting on the plates during freezing runs.
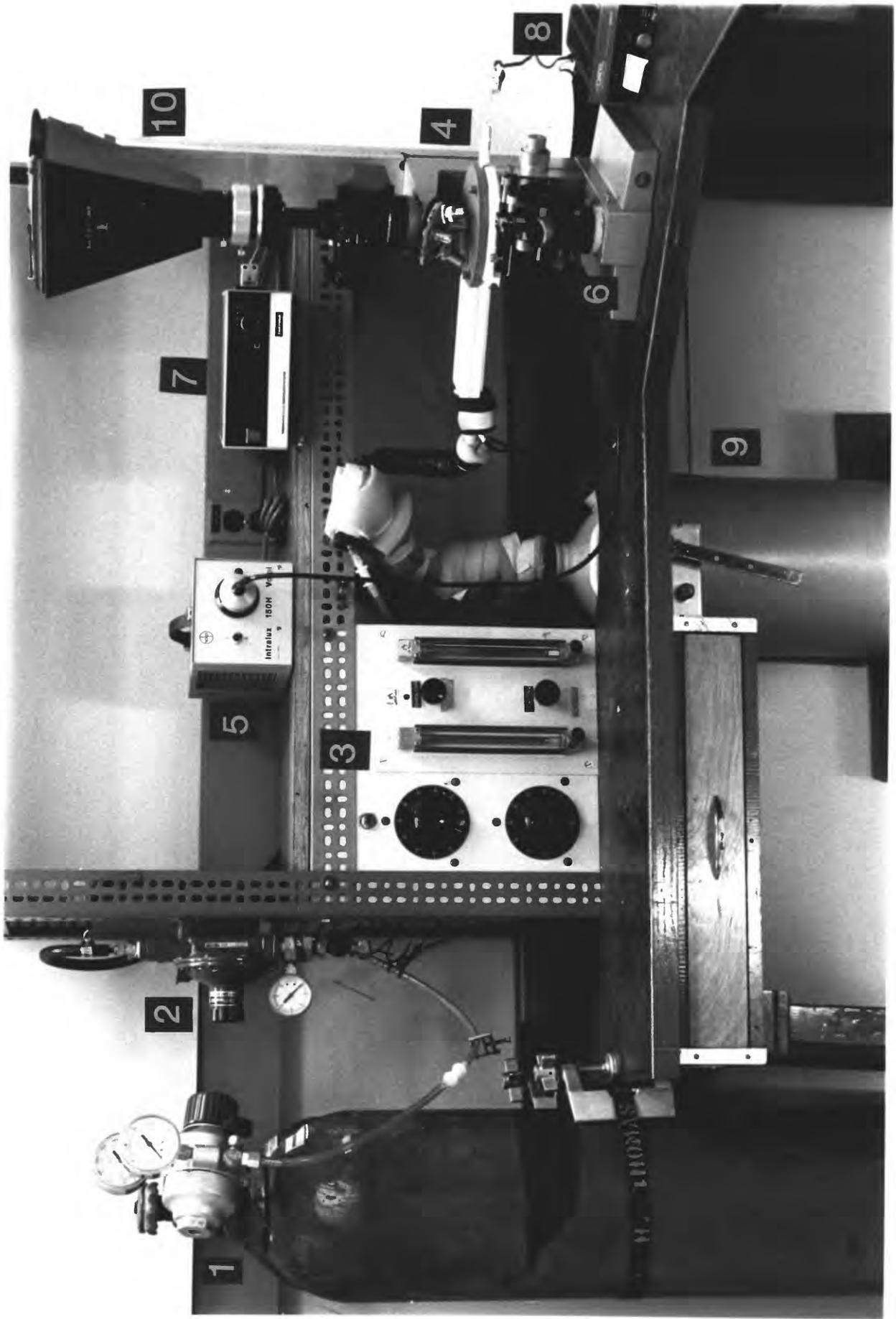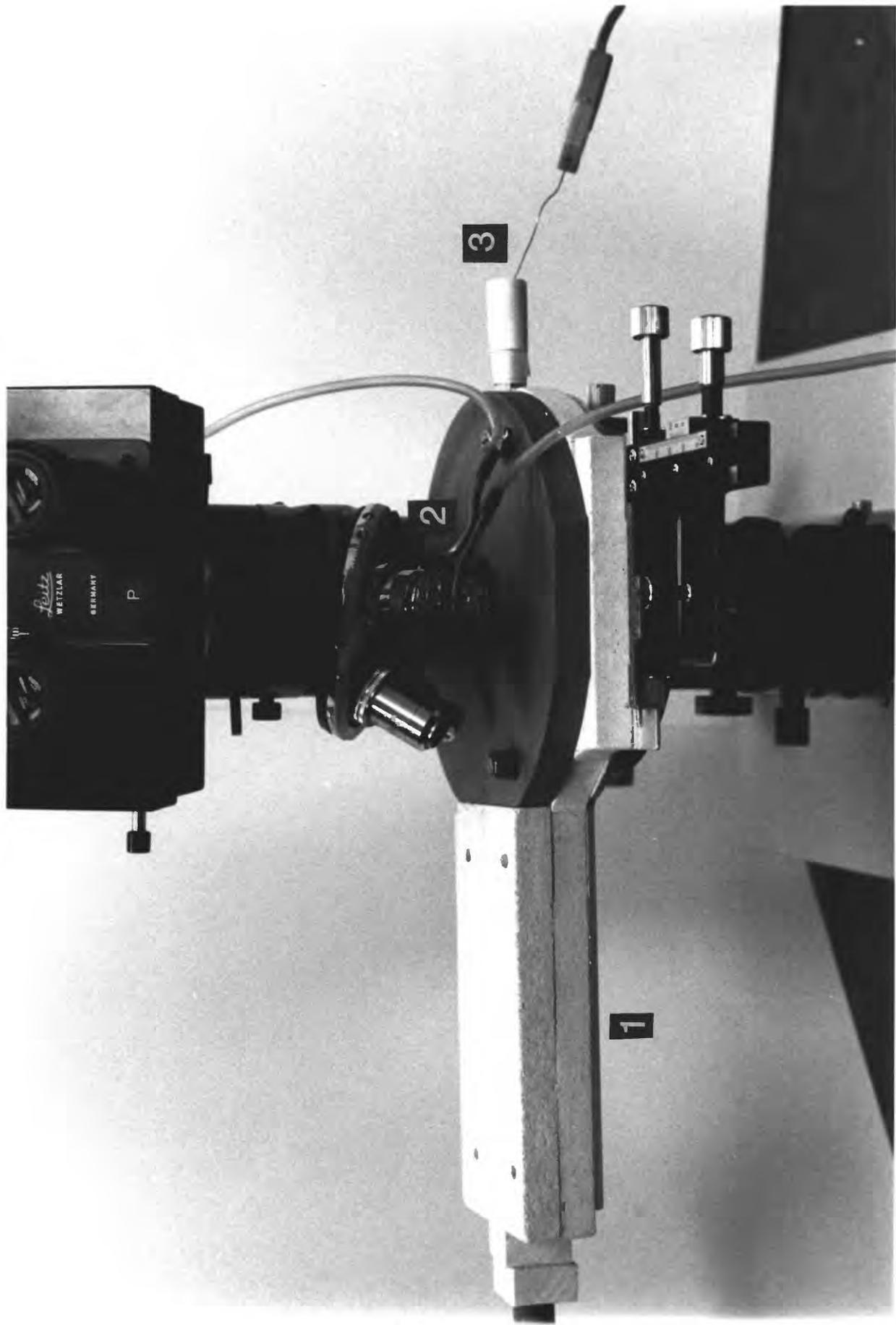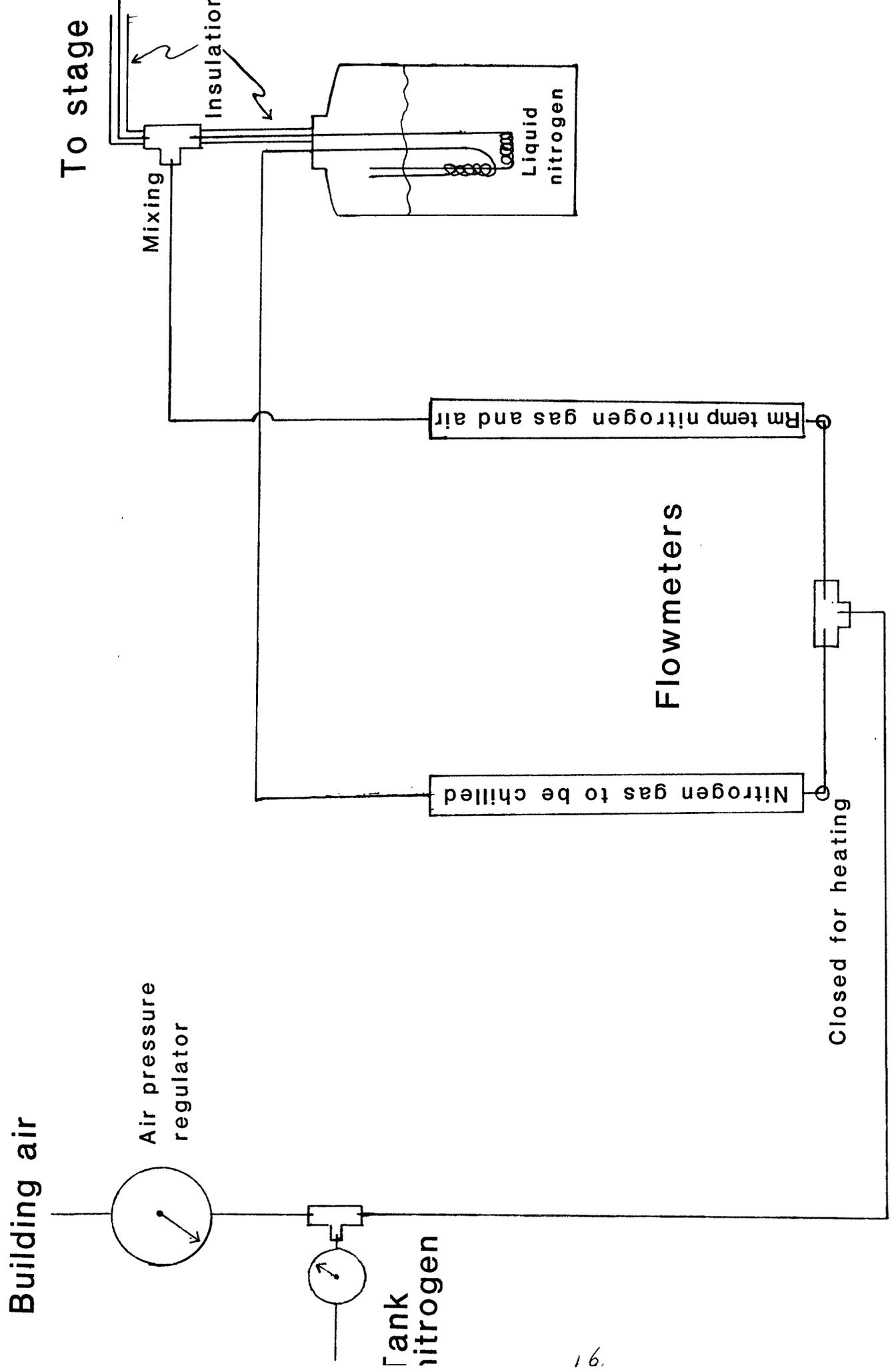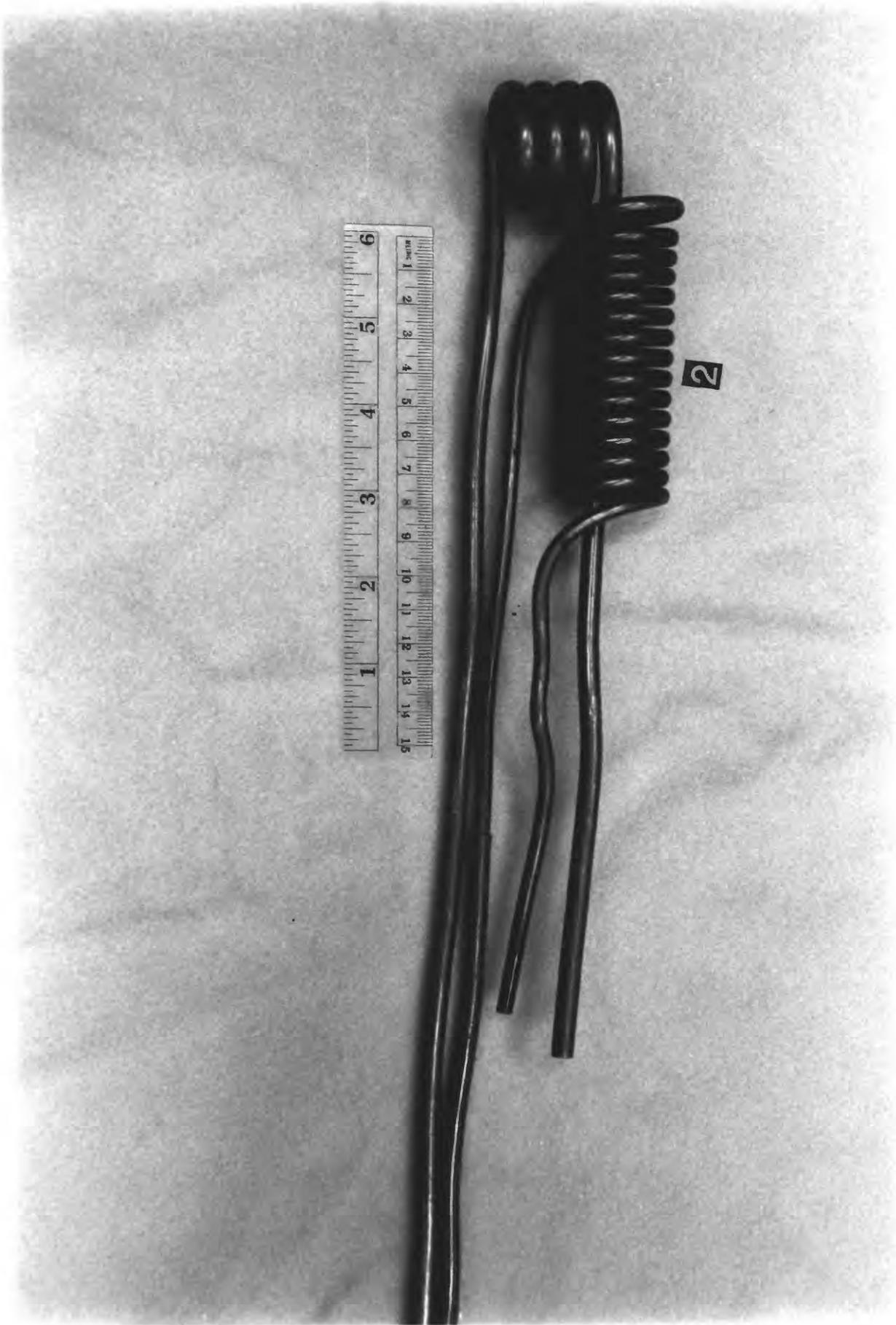
Figure 1

Figure 2

Building air

To stage

Insulation

Mixing

Liquid nitrogen

Air pressure regulator

Rm temp nitrogen gas and air

Flowmeters

Nitrogen gas to be chilled

Closed for heating

Tank nitrogen

Figure 3

16.

Figure 4

Figure 5

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