DEPARTMENT OF THE INTERIOR

U.S. GEOLOGICAL SURVEY

A MICROWAVE-OVEN SYSTEM FOR THE DISSOLUTION OF GEOLOGIC SAMPLES

by

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Open-File Report 86-189

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1986

ABSTRACT

A total of 51 geologic reference materials, including rocks, stream sediments, sulfide ores, and soils, were analyzed in evaluating a procedure for the rapid decomposition of geologic samples. The decomposition procedure consists of an HCl-HNO_3-HF polycarbonate-bomb dissolution technique, using microwave heating. The resulting solutions were analyzed by inductively-coupled-plasma optical-emission spectroscopy (ICPOES); the residues remaining were analyzed by dc arc spectrography. The dissolution technique provides recoveries greater than 95% of most elements in most of the materials studied; low recoveries of Al, Cr, Li, Pb, Si, and Zr are observed when refractory-mineral phases are present. Using this method, as many as 100 samples per person per day can be prepared for analysis.

INTRODUCTION

The modern analytical laboratory is frequently called upon to perform multielement analyses of widely diverse sample types, particularly geologic samples. Now that such techniques as graphite-furnace atomic absorption and plasma-emission spectroscopy are widely accepted (1-4), there has been an increasing need to dissolve various geologic solids, including rocks, mineral separates, soils, and marine sediments. When automated atomic-absorption spectrometry (AAS) or inductively-coupled-plasma optical-emission spectroscopy (ICPOES) instruments are used for the analysis of geologic materials, the most time consuming step in the procedure is dissolution of the sample.

The search for a rapid total-dissolution technique has led several workers to investigate the use of a microwave oven as a heat source in a low-temperature digestion system. Abu-Samra et al. (5) and Barrett et al. (6) used a microwave oven with an HNO_3-$HClO_4$ mixture for wet-ashing biologic samples. Matthes et al. (7) reported on a microwave-oven system for the acid dissolution of metal and mineral samples, using plastic bottles. The method of Matthes et al. uses an HCl-HNO_3-HF mixture sealed in polycarbonate bottles that act as pressure vessels when heated in a microwave oven. Recently, Nadkarni (8) reported favorable results for the decomposition of coal, fly ash, shale, and biologic materials, using a microwave oven with aqua regia and HF in covered Teflon* beakers.

The present work was initiated to evaluate the microwave-oven technique for decomposing various geologic samples in preparation for major-, minor-, and trace-element determinations. In this report, we describe some modifications of Matthes et al.'s method and present data for 51 standard geologic reference samples to illustrate the features of this type of digestion system.

EXPERIMENTAL SECTION

Apparatus. Samples were decomposed in 250-mL polycarbonate bottles fitted with polypropylene screw caps (Nalge #3122-0250). The threads of the polycarbonate bottles were double-wrapped with Teflon tape to ensure a tight seal.

Heating was by means of a 650-W, 0.040-m^3-capacity microwave oven equipped with a removable revolving carousel. To purge the oven with compressed air during and after the heating cycle, 14 lengths of small-diameter (3 mm OD) plastic tubing were inserted through the existing ventilation holes in the side of the microwave-oven cavity. These small tubes were brought out of the oven through the rear ventilation slots and were inserted into two lengths of 9-mm-ID tubing. All exposed metal parts inside the microwave oven were covered with plastic tape to prevent corrosion. The entire microwave oven was placed in a fume hood to provide for adequate ventilation.

The bottle rack used in this study was a polyethylene food container (Tupperware #1257-6 cake carrier), with a top that seals to prevent the release of any fumes escaping from the polycarbonate bottles. The rack will accommodate 12 bottles arranged annularly. Because the microwave energy inside the oven cavity is distributed inhomogeneously, no sample bottles are placed in the center of the rack. This arrangement assures that all samples are subjected to the same microwave flux.

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Instrumentation. An ARL 35000 sequential inductively-coupled-plasma optical-emission spectrometer was used for the analysis of solutions. Options allowing saturation of nebulizer argon with water and washing of the nebulizer tip were used to reduce clogging of the nebulizer during analyses of solutions containing high total dissolved solids. An optional cutoff filter was used to eliminate spectral-order overlap for wavelengths above 320.0 nm. Additional instrumentation included a peristaltic pump for sample delivery and a mass-flow controller for control of the argon flow to the nebulizer. Instrument operating conditions have been previously described (9).

A Jarrell-Ash 3.4-m Ebert spectrograph was used for the dc arc spectrographic analysis of residues. Procedures and operating conditions were described by Myers *et al.* (10).

Reagents. All reagents were of analytical grade or better. The 1.5% boric acid solution was prepared by dissolving 30.0 g of orthoboric acid (99.9%) in 2 L of water. This solution was dispensed by using a Repipet (#3100-A-U) 100-mL bottle-top dispenser.

The HCl-HF mixture (7:3 v/v) was made up with concentrated acids.

Baker Instra-Analyzed reagent-grade HCl and HNO_3 were used.

Standard solutions were prepared from serial dilutions of 1,000 mg L^{-1} solutions obtained from commercial suppliers and had final concentrations of 1.5% H_3BO_3.

Standards used in spectrographic analysis were prepared from 99.999% pure compounds, as described by Myers *et al.* (10).

Standard Reference Materials. Validation of the method presented in this study was performed by using standard reference materials. Sample descriptions and sources are given in Table I. Values for elemental concentrations were obtained from the certificates of analysis or from the compilation of usable values by Abbey (11).

Dissolution Procedure. A 0.100-g sample, ground to pass 100 mesh, was placed in a 250-mL polycarbonate bottle. Then, 2.0-mL of concentrated HNO_3 was added to the bottle, followed by 5.0 mL of the 7:3 mixture of HCl-HF. All bottles were tightly capped and placed in the bottle rack. The covered rack, containing 12 samples, was placed on the carousel in the microwave oven, the compressed air was turned on, and the samples were heated at high power (650 W) for 2.5 min. After completion of the heating cycle, the bottles were allowed to stand in the closed oven, and compressed-air purging of the system was continued for 2 min. The rack was then removed from the oven, and the bottles were cooled in an ice-water bath for 10 min. When cool, the bottles were uncapped, and 93.0 mL of 1.5% H_3BO_3 solution was added to each bottle from a precision liquid dispenser. The bottles were recapped, returned to the oven, and heated at high power for 10 min. After cooling to room temperature in a water bath, the solutions were ready for analysis.

Caution: During the dissolution process, some acid fumes may leak around the caps of the bottles. This leakage will create a health hazard unless the microwave oven is properly vented to a fume hood. The melting point of polycarbonate is 135°C. Care must be taken to ensure that the bottle contents remain below this temperature if the heating time is increased or if the volume of solution in the microwave cavity is decreased. Therefore, if fewer than 12 samples need to be decomposed, bottles containing water or reagent blanks must be included to maintain a constant volume of solution in the oven during each run.

RESULTS AND DISCUSSION

The microwave-oven acid-dissolution method used in this study is a modified version of the method of Matthes et al. (7). We have found that compressed air is a convenient and effective alternative to compressed carbon dioxide for purging the oven during the digestion process, and that the covered bottle rack is an efficient system for containing occasional acid leaks that occur during the procedure. Also, the covered rack helps to attenuate the release of acid fumes inside the microwave oven and thus reduces potential corrosion.

Polycarbonate is a high-tensile-strength, acid-resistant plastic that is microwave transparent. Using the recommended acid mixture and heating time, bottle lifetime is typically four runs. Yellowing of the plastic and gradual frosting of the bottle walls are normal with use. Internal bottle pressure is insufficient to cause an explosion, provided that both the heating time and the total volume of solution inside the microwave oven are maintained at their recommended values.

Volatility Study. Earlier work (8) indicated that substantial amounts of Cr and Pb are volatilized during a microwave-oven digestion procedure when the dissolution is carried out in covered beakers. To check the volatility of these elements under the dissolution conditions used in this study, an experiment was carried out in which known amounts of aqueous standard solutions were carried through the entire digestion procedure. Ten replicate solutions, each containing known amounts of B, Cr, Pb, and Si, were prepared from stock solutions of these elements. Five of these replicates were carried through the microwave-oven digestion procedure, and the remaining five were treated identically, except that the heating step was omitted. All 10 solutions were then analyzed by ICPOES. Analysis of the results listed in Table II, using a two-sided t-test, indicates no significant difference between the heated and unheated samples regarding the recovery of B, Cr, Pb, or Si. We attribute our success in retaining these volatile elements to our use of sealed bottles during the digestion procedure.

Recovery Studies. To check for completeness of decomposition, the solution from each sample was filtered through Whatman #44 filter paper, and the residue was collected and analyzed by dc arc spectrography (10). The dc arc technique was chosen because small amounts of undissolved material are detected even when the amount of the element present in the residue is only a small percentage of the total present in the sample. In addition, this technique allows multielement analyses, using as little as 1 mg of sample.

The dc arc technique also allowed us to gather data on elements for which accepted values are not available. In the absence of accepted values, it is impossible to detect incomplete dissolution of an element by analyzing the resulting solution. However, incomplete dissolution can be detected by careful examination of any residue remaining after digestion. Although analysis of residues does not allow quantifying recoveries of elements without accepted values, it does provide a qualitative evaluation of the decomposition procedure for those elements.

Because the material used as a spectroscopic buffer for the dc arc technique contains Si, Al, and K, the recoveries of these elements cannot be determined by analysis of the residues. Therefore, the recoveries of these three elements are based on analysis of solutions by ICPOES. Li was also determined by ICPOES because the detection limit for Li in the dc arc technique would have prevented us from obtaining information in the recovery range 60-95%.

Analysis of Rocks and Soils. Results of the dissolution of 33 rocks and soils are listed in Table III. Li, Al, and K recoveries, measured by ICPOES, agree well with the accepted values. The low results for Al in the three GXR samples are due to incomplete attack of the α-Al_2O_3 minerals present. The presence of corundum in sample GXR-5 was established by obtaining an X-ray-diffraction powder pattern on the residue left after the dissolution step. Allcott (12) indicated that corundum-lined grinding vessels were used during preparation of the GXR standards, and so α-Al_2O_3 is present in these standards as a contaminant.

The Si recoveries listed in Table III also agree well with the accepted values, except for the samples containing quartz. For example, granites and granodiorites typically contain 20-50% quartz (13). In Table III, the SiO_2 content is 9% low for sample G-2 (a granite) and 11% low for sample GSP (a granodiorite). Although Si recovery from samples of pure natural quartz increases with decreasing particle size (Fig. 1), less than 50% of the quartz in a sample will dissolve by this technique, even when the sample is ground finer than 325 mesh. By contrast, sample RGM-1 (an obsidian), which contains no quartz, exhibits excellent Si recovery.

The dc arc emission spectra of the residues were examined for the 42 elements listed in Table IV. The mean weight of 4.5 mg of the residues from decomposition of the 33 rocks and soils indicates better than 95% decomposition for most of the samples studied. Evaluation of the spectra from the residues, however, in conjunction with the known mineralogy of the samples, reveals that the main components of the residues are quartz (SiO_2), corundum (Al_2O_3), chromite ($FeCr_2O_4$), zircon ($ZrSiO_4$), and rutile (TiO_2). We note that less than 20% of the Zr in samples GSP, GSD-8, W-1, and G-2 was dissolved by this technique because the Zr in these samples is present in the form of zircon. Likewise, the low Cr recovery from ultramafic-rock types, such as samples PCC and DTS, is due to the Cr content of these rock types in the form of chromite. Poor Cr recovery in samples W-1 and BCR-1 was noted by Nadkarni (8). The compositions of all the residues are listed in Table V. We note that elements with better than 95% recovery are omitted from Table V. These results indicate that analysts must be concerned about the presence of acid-resistant minerals when decomposing rocks and soils by this technique.

Analysis of Ores and Minerals. A total of 18 standard ores, concentrates, and mineral samples (Table I) were decomposed by the microwave-oven procedure. The ICPOES results listed in Table VI again indicate incomplete Si and Al recoveries from refractory minerals. We also note incomplete Li recovery from the sample of spodumene ore.

Residues from this suite of samples were collected and analyzed by procedures identical to those used for the rocks and soils. Of the 18 samples, 9 had residues weighing more than 20% of the original sample weight, and the elements detected in these residues (Table VII) are characteristic of such minerals, as cassiterite (SnO_2) and magnetite ($Fe^{2+}Fe_2^{3+}O_4$), which are notoriously acid resistant. A residue of elemental sulfur remains after dissolution of the ore samples that are composed primarily of sulfide minerals.

To check the effect of sample size on the recovery of selected elements from sulfide ores, five of the Canadian Certified Reference Materials Project (CCRMP) ore samples were decomposed a second time, starting with 0.5 g of sample. Comparison of the results obtained by using different-size sample aliquots (Table VIII) reveals that Ag and Pb recoveries depend on sample size. The low Pb recovery from the sample of Pb ore is caused by the precipitation of $PbSO_4$ resulting from oxidation of the galena (PbS) present in this sample. Fig. 2 plots the amount of Ag in the sample aliquot versus the amount of Ag in the final solution. The curve reflects the anticipated limited solubility of Ag in the dilute-acid medium employed in this technique. We note that reliable Ag values can be obtained from samples containing less than 0.1 mg of Ag. However, repeated decompositions of the same sample, using different sample weights, must be used to verify the validity of the Ag determinations.

Low Sn recovery is evident from the data listed in Table VIII. Complete mineralogic data are available for samples KC-1 and MP-1A. The presence of Sn as cassiterite in these two samples results in the poor recoveries observed.

CONCLUSIONS

The microwave-oven, mixed-acid-digestion system is a suitable dissolution technique for a wide range of geologic sample types. It has proved to be a rapid, inexpensive method for decomposing rocks and soils in preparation for multielement determinations. This method is particularly useful for volatile elements, which are normally lost during decomposition procedures using open vessels. The solutions prepared by this procedure are compatible with analysis by ICPOES and AAS, although the resulting 1:1,000 dilution factor may preclude determination of certain elements by these techniques. Approximately 100 samples can be prepared for analysis in a single day. Refractory minerals, such as chromite, corundum, quartz, rutile, and zircon, and other materials known to be resistant to attack by mineral acids and HF are only partially dissolved by this technique. Except for Ti and Cr, the transition elements contained in most geologic samples can be completely dissolved by this technique.

ACKNOWLEDGMENTS

We are grateful to R. C. Erd, who performed the X-ray-diffraction analyses. We also thank L. Jassie and H. M. Kingston, U.S. National Bureau of Standards, Gaithersburg, Md., for many helpful discussions and enthusiastic support.

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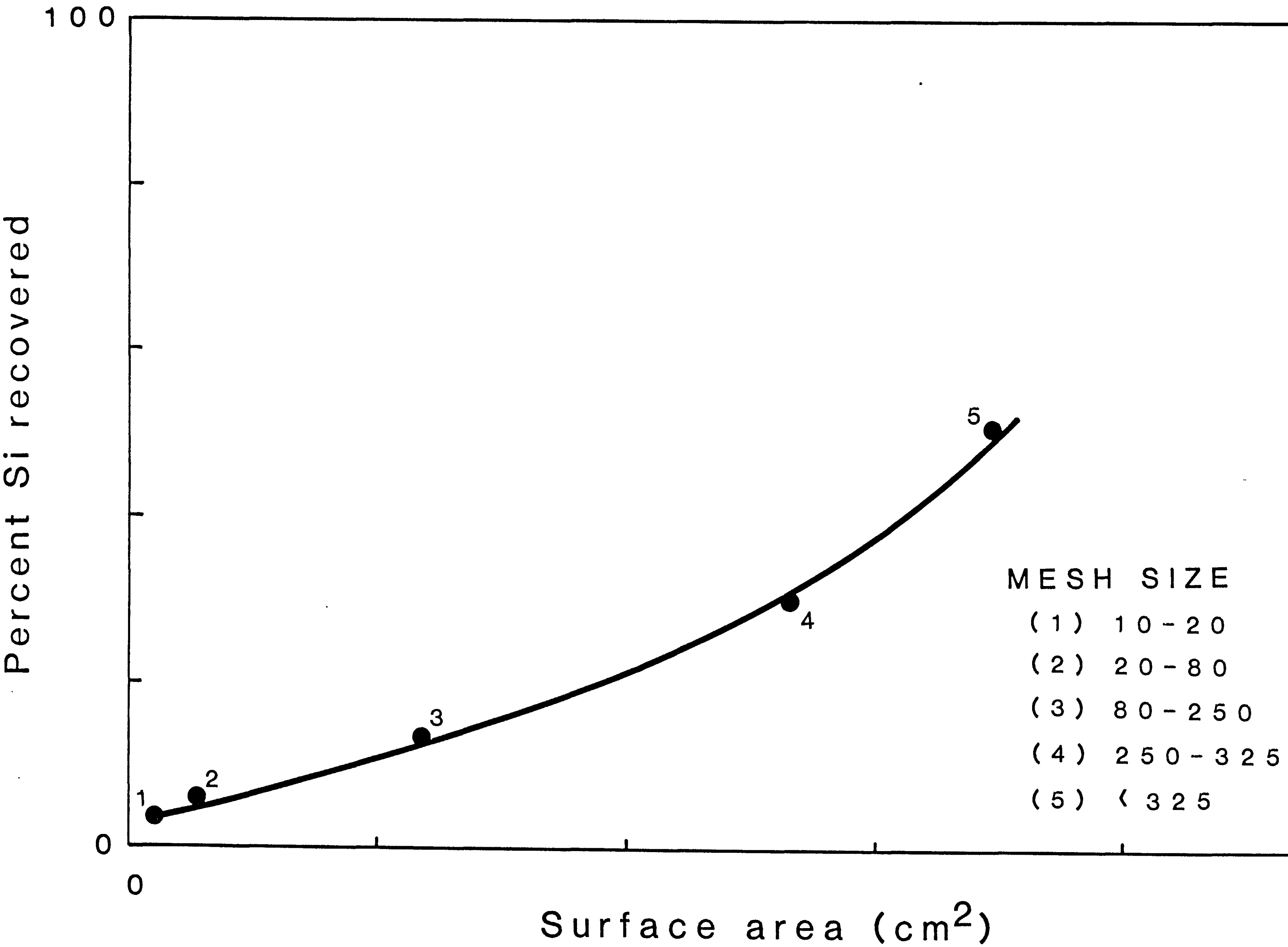
FIGURE CAPTIONS

FIG. 1. Effect of mesh size on Si recovery from quartz, using a 100-mg sample.

FIG. 2. Ag recovery from solution, as determined by ICPOES after microwave heating/acid dissolution.


100
0
Percent Si recovered
0
80
Surface area (cm²)
1
2
3
4
5
MESH SIZE
(1) 10-20
(2) 20-80
(3) 80-250
(4) 250-325
(5) ‹ 325

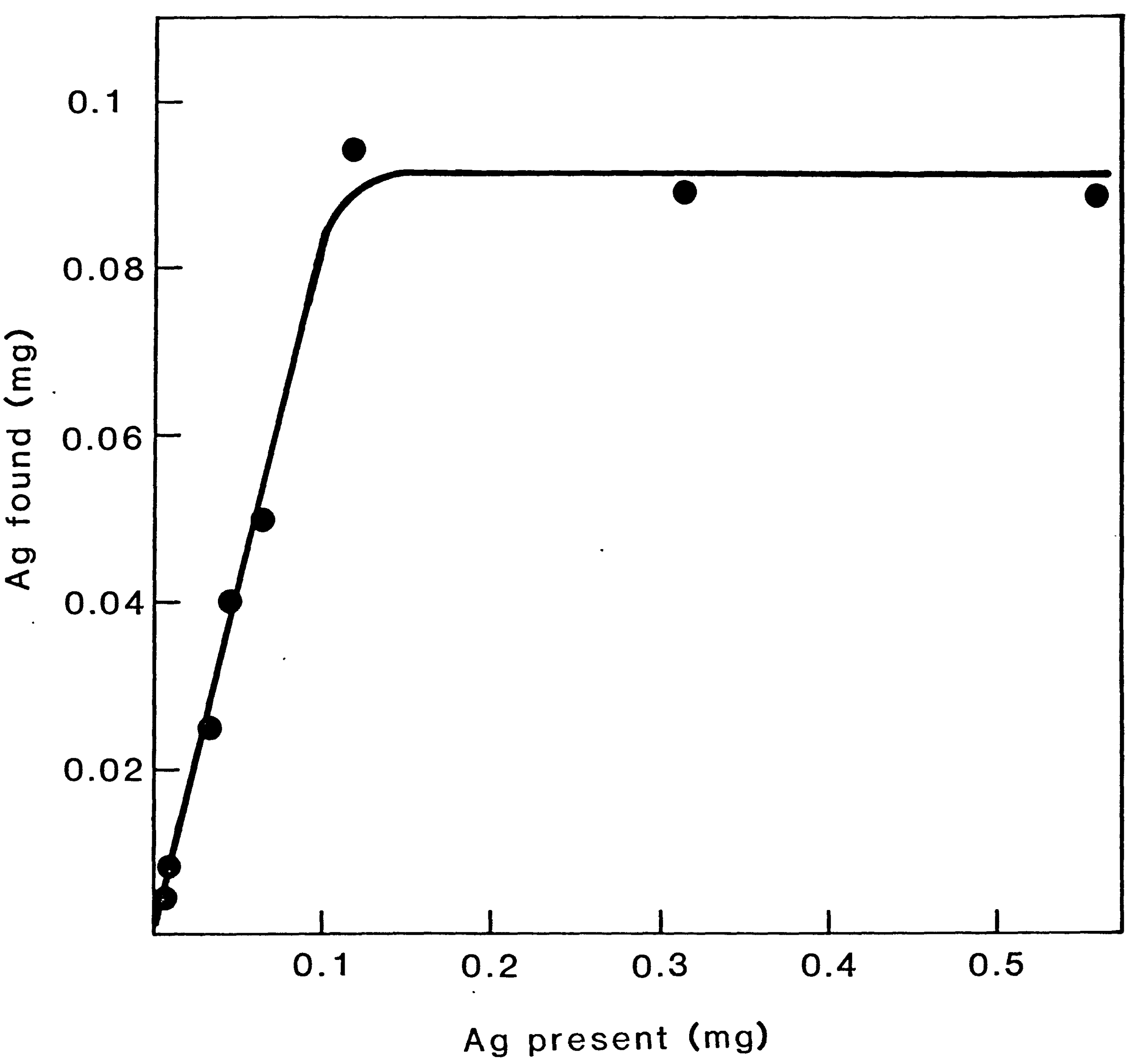

0.1
0.08
0.06
0.04
0.02
Ag found (mg)
0.1
0.2
0.3
0.4
0.5
Ag present (mg)

Table I. Listing of Standard Reference Materials

Rocks and Soils

Sample	Description	Organization[a]
A-1	Mn nodule	USGS
AGV-1	Andesite	"
BCR-1	Basalt	"
DTS-1	Dunite	"
G-2	Granite	"
GSD-2	Stream sediment	IGGE
GSD-3	Stream sediment	"
GSD-8	Stream sediment	"
GSP-1	Granodiorite	USGS
GXR-1	Jasperoid	"
GXR-2	Soil	"
GXR-3	Fe-Mn-rich hot-spring deposit	"
GXR-5	Soil	"
MAG-1	Marine sediment	"
MESS-1	Marine sediment	NRC
Mica-Fe	Biotite	CRPG
MRG-1	Gabbro	CCRMP
P-1	Mn nodule	USGS
PCC-1	Peridotite	"
RGM-1	Rhyolite	"
SGR-1	Shale	"
STM-1	Syenite	"
SY-3	Syenite	CCRMP
TB	Slate	ZGI
UB-N	Serpentine	ANRT
W-1	Diabase	USGS
1A	Limestone	NBS
56B	Phosphate rock	"
88A	Limestone, dolomitic	"
97	Clay, flint	"
99A	Feldspar, soda	"
120B	Phosphate rock	"
1645	River sediment	"

Ores and Minerals

BX-N Bauxite	ANRT	
CCU-1	Cu concentrate	CCRMP
CPB-1	Pb concentrate	"
CZN-1	Zn concentrate	"
KC-1	Zn-Pb-Sn-Ag ore	"
MP-1A	Zn-Sn-Cu-Pb ore	"

25C	Mn ore	NBS
27E	Fe ore	"
69A	Bauxite	"
79	Fluorspar	"
103A	Chrome refractory	"
113	Zn ore	"
138	Sn ore	"
181	Spodumene Li ore	"
182	Petalite Li ore	"
183	Lepidolite Li ore	"
333	Mo ore	"
692	Fe ore	"

[a] ANRT, Association Nationale de la Recherche Technique, France; CCRMP, Canadian Certified Reference Materials Project; CRPG, Centre de Recherches Petrographiques et Geochimiques, France; IGGE, Institute of Geophysical and Geochemical Exploration, People's Republic of China; NBS, U.S. National Bureau of Standards; NRC, National Research Council of Canada; USGS, U.S. Geological Survey; ZGI, Zentrales Geologisches Institut, East Germany

Table II. Effect of Heating on the Recovery of Volatile Elements

Element (μg/g)	Unheated		Heated	
	$\bar{x}$	s	$\bar{x}$	s
B (%)	1.48	± 0.01	1.48	± 0.02
Cr	11.2	± 0.1	11.2	± 0.2
Pb	12.3	± 0.2	12.5	± 0.2
Si	20.6	± 0.4	21.1	± 0.3

s = standard deviation of five replicate determinations.

Table III. Analysis of Rocks and Soils by ICPOES

	Al_2O_3 (%)		K_2O (%)		Li (ug/g)		SiO_2 (%)	
Sample	Found	Present	Found	Present	Found	Present	Found	Present
A-1	3.14±.02	3.36	0.50±.01	0.55	68±2	64	3.39±.02	3.31
AGV-1	16.5±.1	17.19	2.83±.03	2.92	11±2	12	58.0±.3	59.61
BCR-1	13.2±.1	13.72	1.74±.02	1.70	13±1	14	54.9±.4	54.53
DTS-1	<1.0	0.25	<0.10	0.001	<10	2	39.2±.2	40.61
G-2	15.3±.1	15.40	4.41±.07	4.46	30±4	35	60.1±.4	69.22
GSD-2	15.0±.1	15.75	4.90±.09	5.18	105±1	100	62±1	69.94
GSD-3	11.9±.2	12.09	2.42±.01	2.44	33±3	33	63±1	71.34
GSD-8	7.2±.2	7.70	2.71±.02	2.75	12±2	13.5	71.6±.7	82.92
GSP-1	14.6±.2	15.28	5.3±.1	5.51	27±2	30	56.0±.4	67.32
GXR-1	1.32±.02	6.69	<0.10	0.064	<10	7.6-8.5	47.8±.5	49.2
GXR-2	12.2±.1	35.1	1.56±.01	1.70	56±4	--	46.3±.4	49.2
GXR-3	5.10±.06	11.7	0.90±.02	0.89	144±4	100-131	12.0±.1	13.0
GXR-5	11.0±.2	39.3	1.12±.02	0.99	44±1	39-47	41.6±.5	42.1
MAG-1	16.4±.1	16.46	3.81±.03	3.72	83±4	78	49.0±.3	51.19
MESS-1	10.9±.2	11.03	2.29±.01	2.24	41±2	(60)	48.4±.2	66.1
MICA-Fe	19.2±.3	19.58	8.8±.2	8.79	1,160±30	1,400	34.2±.1	34.55
MRG-1	7.8±.1	8.50	0.17±.01	0.18	<10	4	38.2±.5	39.32
P-1	3.91±.02	4.19	1.22±.01	1.17	138±6	122	11.5±.1	12.11
PCC-1	<1.0	0.73	<0.10	0.004	<10	3	39.2±.4	42.10
RGM-1	13.3±.2	13.80	4.10±.05	4.35	60±4	50	74±2	73.47
SGR-1	3.53±.04	6.49	1.82±.04	1.63	152±6	--	28±1	28.30
STM-1	18.0±.2	18.44	4.07±.03	4.29	34±1	30	59.2±.9	59.66
SY-3	11.4±.3	11.80	4.39±.06	4.20	91±6	92	57.4±.7	59.68
TB	20.2±.4	20.60	3.85±.06	3.85	114±5	115	53.4±.2	60.24
UB-N	1.86±.02	2.94	<0.10	0.02	27±3	28	38.8±.5	39.93
W-1	14.6±.1	15.02	0.66±.02	0.64	12±3	14.5	50.8±.3	52.72
NBS-1A	3.86±.06	4.17	0.77±.03	0.71	16±2	--	11.4±.1	14.12
NBS-56B	3.23±.02	--	0.18±.02	--	<10	--	9.89±.09	10.1
NBS-88A	<1.0	0.19	0.120±.002	0.12	<10	--	1.23±.02	1.20
NBS-97	40.3±.6	38.77	0.49±.06	0.54	1,120±40	1,068	42±1	42.87
NBS-99A	19.5±.5	20.5	5.01±.01	5.20	<10	--	63.4±.5	65.2
NBS-120B	1.10±.01	1.06	<0.10	0.12	<10	--	2.94±.04	4.68
NBS-1645	4.38±.09	4.27	1.21±.03	1.52	<10	--	20.1±.2	--

Table IV. Detection Limits for Elements Determined by dc Arc Spectrography

Element	Limit (%)	Element	Limit (μg/g)	Element	Limit (μg/g)
Si	0.007	Ag	0.7	Li	100
Al	0.002	As	100	Mo	2
Fe	0.0003	Au	7	Nb	10
Mg	0.00015	B	2	Ni	0.7
Ca	0.001	Ba	1	Pb	7
Na	0.01	Be	0.7	Sb	20
K	0.02	Bi	7	Sc	0.7
Ti	0.0001	Cd	7	Sn	2
P	0.15	Ce	50	Sr	1
Mn	0.0001	Co	1	Tl	3
		Cr	0.7	V	1
		Cu	0.7	W	10
		Ga	0.7	Y	7
		Ge	7	Yb	0.7
		In	1.5	Zn	15
		La	7	Zr	3

Table V. Recovery of Elements Based on dc Arc Analysis of Residues from the Decomposition of Rocks and Soils.

Sample	95-90%	89-80%	80%	Uncertified Elements Detected
A-1[a]	--	--	--	--
AGV-1	Zr	--	--	--
BCR-1[a]	--	--	--	--
DTS-1	Ca	--	Cr	--
G-2	--	Ti	Zr	--
GSD-2	--	--	Ti Be Cr La	Ce Yb
			Nb Sn Y Zr	--
GSD-3	V	--	Ti Cr La Y Zr	--
GSD-8	--	Nb Y	Ti Sn Zr	--
GSP-1	--	Y	Ti La Zr	--
GXR-1	Sb	Cr	Mg Ti	--
GXR-2	--	--	Ti Zr	--
GXR-3	Mg	--	Ti	Zr
GXR-5	Cr	--	Ti Zr	--
MAG-1	Y	--	Ti Zr	--
MESS-1	--	Cr	Ti Zr	Yb
Mica-Fe	--	La Y	Ce Zr	--
MRG-1	Co	Zn	Cr	--
P-1[a]	--	--	--	--
PCC-1	Mg Ca Ti	--	Cr V	--
RGM-1	--	--	Cu	--
SGR-1	Co	--	Ti Zr	--
STM-1	Fe	Mg Ca Ti	Na Zr	--
SY-3	Co	--	Zr	--
TB	--	Sr	Ce Ti Y Zr	--
UB-N	V	Cr Zn	--	--
W-1	Ti	--	Zr	--
1A	--	--	Ti	Sc Zr
56B[b]	--	--	--	Mo Sn Zr
88A	--	--	Ti	--
97	Ca	Mg Zr	Ti Cr	Y
99A	--	Ti	--	--
120B	--	--	Ti	Sn Zr
1645[c]	--	--	--	--

[a] All certified elements exhibit >95% recovery. [b] Only four accepted values listed in certificate of analysis. [c] Analysis of undiluted residue indicates presence of Si.

Table VI. Analysis of Ores and Minerals by ICPOES

	Al_2O_3 (%)		K_2O (%)		Li (μg/g)		SiO_2 (%)	
Sample	Found	Present	Found	Present	Found	Present	Found	Present
BX-N	34.2±0.5	54.45	<0.10	0.05	26±1	--	7.5±.2	7.43
CCU-1	<1.0	0.247±0.007	<0.10	--	<10	--	2.46±0.03	2.61±0.08
CCU-1^a	0.260±0.005	0.247±0.007	<0.02	--	<2	--	2.76±0.05	2.61±0.08
CPB-1	<1.0	0.28±0.02	<0.10	--	<10	--	0.83±0.02	0.74±0.04
CPB-1^a	0.28±0.01	0.28±0.02	<0.02	--	<2	--	0.77±0.04	0.74±0.04
CZN-1	<1.0	0.25±0.01	<0.10	--	<10	--	0.90±0.02	1.0
CZN-1^a	0.250±0.003	0.25±0.01	<0.02	--	<2	--	1.13±0.01	1.0
KC-1	1.42±0.02	1.5	<0.10	0.12	<10	--	15.0±0.1	23
KC-1^a	1.46±0.02	1.5	0.09±0.01	0.12	<2	--	12.4±0.1	23
MP-1A	3.44±0.05	9.8	<0.10	--	22±7	--	23.8±0.2	41.4
MP-1A^a	3.3±0.2	9.8	0.09±0.01	--	24.1±0.3	--	19.6±0.3	41.4
NBS-25C	1.75±0.01	--	<0.10	--	<10	--	2.23±0.02	2.36
NBS-27E	<1.0	--	<0.10	--	<10	--	3.25±0.03	3.64
NBS-69A	57±1	55.0	<0.10	<0.01	<10	--	4.56±0.03	6.01
NBS-79	<1.0	0.02	<0.10	0.01	<10	--	1.55±0.02	1.88
NBS-103A	1.69±0.02	29.96	<0.10	--	<10	--	4.96±0.03	4.63
NBS-113	<1.0	--	<0.10	--	<10	--	4.14±0.08	--
NBS-138	<1.0	--	<0.10	--	<10	--	0.38±0.01	--
NBS-181	16.8±0.1	--	<0.10	0.3	2.10 ±0.02	2.97 (%)	42.7±0.7	--
NBS-182	16.6±0.2	--	<0.10	0.1	2.14 ±0.05	2.02 (%)	79.4±0.8	--
NBS-183	25.0±0.3	--	8.4±0.2	8	2.09 ±0.09	1.91 (%)	48.5±0.5	--
NBS-333	1.24±0.02	--	<0.10	--	<10	--	1.74±0.03	--
NBS-692	1.43±0.02	1.41	<0.10	0.04	<10	--	6.59±0.05	10.14

^{a} 0.5-g sample size.

Table VII. Recovery of Elements Based on dc Arc Analysis of Residues from the Decomposition of Ores and Minerals.

Sample	95-90%	89-80%	80%	Uncertified Elements Detected
BX-N	--	Ni	Ba Ti Y Zr	Mo Sn Zn
CCU-1	Pb Ca	Cu Fe	--	Co
CPB-1	Mg Sn	--	--	--
CZN-1	As	Sn	--	--
KC-1	Ag	--	Sn	Y
MP-1A	--	--	Sn	Nb W Y Zr
25C[a]	--	--	--	Ba Cr Zr
27E	--	--	Fe	--
69A	--	--	Mg Ti Zr	--
79[b]	--	--	--	Ba Mo Sn
103A	--	--	Fe Mg Mn Cr	Co Ni
113[b]	--	--	--	Ag Cd Pb
138	--	--	Sn	Nb Sc V W Zr
181	--	--	Li	Be Cr Sn Zr
182[b]	--	--	--	Cr Sn
183[b]	--	--	--	Be Sn
333	--	--	Mo	Sn V W
692	--	--	Fe Mg	--

[a] All certified elements exhibit >95% recovery. [b] Only one accepted value listed in certificate of analysis.

Table VIII. Effect of Sample Size on Percent Recovery of Certified Elements from Sulfide Ores, Based on Analysis of Residue by dc Arc

	CCU		CZN		MP-1A		KC-1		CPB	
Sample Weight (g)	0.1	0.5	0.1	0.5	0.1	0.5	0.1	0.5	0.1	0.5
Ag	--	--	91	86	95	73	86	16	82	28
As	--	--	99	92	99	99	--	--	99	99
Bi	--	--	--	--	99	99	--	--	99	95
Cd	100	99	100	100	--	--	--	--	99	99
Cu	88	88	100	100	100	99	99	99	100	100
Pb	91	98	99	99	99	99	100	96	100	80
Sn	--	--	81[a]	73[a]	45	58	70	71	95[a]	99[a]
Zn	100	100	99	99	100	99	100	99	99	99

[a] Recoveries are based on noncertified Sn values and are listed for information purposes only.