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INNOVATIVE METHODS FOR INORGANIC SAMPLE PREPARATION

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MASTER

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ABSTRACT

Procedures and guidelines are given for the dissolution of a variety of selected materials using fusion, microwave, and Parr bomb techniques. These materials include germanium glass, corium-concrete mixtures, and zeolites. Emphasis is placed on sample-preparation approaches that produce a single master solution suitable for complete multielement characterization of the sample. In addition, data are presented on the soil microwave *digestion* method approved by the Environmental Protection Agency (EPA). Advantages and disadvantages of each sample-preparation technique are summarized.

I. INTRODUCTION

Sample preparation is a critical step in chemical analysis. The preparation handling frequently establishes the lower quantitation limit in an elemental analysis by contributing to the level of contaminants in the analytical blank. Acid decompositions that are necessary prior to instrumental analysis for both major and minor constituents are time consuming and are usually the slowest step in the overall analysis process. Instruments capable of simultaneous multielement analyses have provided the impetus to prepare large numbers of samples in less time and with greater efficiency. Yet, although significant advances have been made in analytical instruments, sample preparation methods have not changed significantly in many

years. Thus, sample dissolution systems that will deliver a valid master solution for multielement analyses are a weak link in the analytical chain.

Sample preparation techniques need to provide (1) complete sample dissolution, (2) retention of volatile analytes, (3) minimal contamination from the environment, added salts, or the digestion vessel, (4) low reagent blanks, and (5) speed. We evaluated three methods of inorganic sample preparation with respect to these criteria. These methods involve sample dissolution by use of lithium tetraborate fusion, Parr acid bomb, and closed-vessel microwave oven. All three methods were carried out using equipment available in the Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL). This report describes our experience in applying these methods to samples encountered in provision of analytical support to ANL programs. Samples included corium-concrete mixtures from the ANL Molten Corium-Concrete Interaction (MCCI) studies, portland cement, zeolites, and glasses. All of these materials are complex matrices that present a dissolution challenge. In addition, the microwave digestion and Parr bomb methods were tested for environmental soil preparation. The preparation schemes used and data obtained are presented. Advantages and disadvantages of each preparation method are summarized to aid in selecting the appropriate procedure for inorganic samples different from those addressed in this work.

II. EXPERIMENTAL

A. Apparatus

1. Parr Bombs - Parr Teflon Acid Bomb, Model No. 4745 - This is a general purpose bomb used with a 23-mL Teflon cup with cover, obtained from Parr Instrument Co., Moline, IL.

2. Microwave System

- a. Microwave Oven - Commercial 1.4-ft³ (0.04-m³) microwave oven (Sears Kenmore), with a spring-powered carousel turntable. The oven has variable power and was modified to vent into a fume hood.
- b. Vessels - Capped Teflon vessels, 120-mL capacity, with relief valves designed for use with a laboratory microwave system (CEM Corp., Indian Trail, NC). The relief valve in the vessel cap is designed to remain static, while pressure and temperature affect the dimensions of the vessel. If the pressure reaches 75 to 85 psi (0.5 to 0.6 MPa), the valve will open and vent vapors.
- c. Capping Station (CEM Corp.) - The capper is a necessary part of the digestion system. It ensures reproducible torque on the vessels, guaranteeing sufficient torque for a tight seal and providing appropriate stress on the cap to achieve proper operation of the relief valve.

B. Reagents and Samples

1. Mineral Acids

- a. Hydrochloric acid (HCl)
- b. Nitric acid (HNO₃)
- c. Hydrofluoric acid (HF)

Instra-Analyzed acids (Baker Chemical Co.) and/or reagent-grade acids were used in all tests.

2. Lithium Tetraborate (Li₂B₄O₇) - High purity lithium tetraborate (Spex Industries, 99.99% purity).

3. Samples

- a. Molten corium-concrete mixtures containing uranium oxide, zirconium, steel, fission-product elements, and concrete. These mixtures were generated at temperatures simulating reactor meltdown conditions as part of the MCCI studies, which were performed to determine the vaporization behavior of the elements present in the corium-concrete mixtures and to understand the release of refractory fission products during a degraded-core accident. Typically, solidified melt and aerosol samples produced in MCCI tests are analyzed for Ba, Sr, La, Ca, Al, Mg, Na, K, Fe, Cr, Ni, Zr, Ag, Ce, and U.
- b. EPA soil reference material, referred to as a Solid Laboratory Control Sample (LCS 0287). This soil sample is used in multielement environmental analysis for quality assurance purposes and was prepared by the University of Nevada at Las Vegas - Quality Assurance Laboratory (UNLV-QAL).
- c. Portland Cement NBS SRM-1016 for silicon analysis. This material is generally fused with sodium carbonate to effect dissolution for silicon determination. Mineral acid treatment was tested in the present study.
- d. Zeolites. Various zeolites are dissolved to provide a single solution that can be used for major and minor cation analysis.
- e. Glasses. Germanium glass samples are prepared for sodium, aluminum, and rubidium analyses in support of a program studying diffusion mechanisms in alkali glasses.
- f. Rocky Flats Soil No. 1, NBS SRM-4353. This soil is a high silicon material used in uranium analysis for quality assurance purposes.

C. Preliminary Sample Preparation

If the sample material to be dissolved is chunky, it is pulverized using shatterbox grinding equipment. Moisture content may be determined after drying at 105 °C for at least four hours.

III. SAMPLE PREPARATION AND ANALYSIS

A. Lithium Tetraborate Fusion

1. Preparation Method and Analysis Results

The steps followed in the lithium tetraborate fusion were as follows.

- Weigh about 150 mg of lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$) and place it into a platinum crucible.
- Add 25 to 100 mg of weighed sample.
- Mix the sample with the $\text{Li}_2\text{B}_4\text{O}_7$ using a platinum rod.
- Weigh out another 100 mg of $\text{Li}_2\text{B}_4\text{O}_7$ and add to the crucible so it covers the mixture already present. (Note: If the sample material is voluminous, 150 mg of $\text{Li}_2\text{B}_4\text{O}_7$ is required.)
- Cover the crucible with a platinum lid and place in a muffle furnace preheated at 1000 to 1100 °C.
- Heat the sample for 0.5 to 3 h, as needed, judged by a visual inspection. The flux should look clear and glassy.
- When the fusion is complete, remove the crucible from the furnace and allow it to cool to room temperature.
- Add a small Teflon-coated stirring bar and 10 mL of 1% HCl to the crucible.
- Heat the crucible on a hot plate/stirrer to dissolve the melt.

- Cool the solution and transfer it to an appropriate volumetric flask so that, on dilution to volume, the concentration is about 1 mg sample per mL of solution.

After this procedure was finished, the resulting solution was submitted for multielement analysis with inductively coupled plasma/atomic emission spectroscopy (ICP/AES). This procedure was tested with MCCI aerosols from high-silica concretes that had been subjected to temperatures approaching 2600 K. These materials are resistant to simpler dissolution techniques. The average fusion time needed was 2 h. Standards for the ICP/AES measurements were prepared in the $\text{Li}_2\text{B}_4\text{O}_7$ flux matrix. Fusion blanks analyzed with the samples were found to contain negligible amounts of the elements of interest.

Data obtained following the fusion procedure with four samples are shown in Table 1. Included in this table are samples that are high and low in refractory elements (Al, Si, U, Zr, etc.), which are known to be difficult to dissolve. The ICP/AES measurements for major components in these samples have an estimated uncertainty of $\pm 10\%$, relative to the reported values. The data in Table 1 indicate that the mass recovery attained for these difficult-to-dissolve samples is very good, from 96.1 to 101.5%.

Lithium tetraborate fusion of samples is widely used for ICP/AES analyses. However, the following properties of the $\text{Li}_2\text{B}_4\text{O}_7$ fusion solution should be considered before choosing this preparation technique: (1) the high dissolved solids content of the fusate solution requires that the sample be diluted, which limits sensitivity for minor elements; (2) the high boron concentration in the fusate results in boron being deposited in the ICP nebulizer system, requiring long flushing (days) or disassembly and cleaning to bring the boron background

Table 1. Sample Characterization Following
Lithium Tetraborate Fusion Preparation^a

Element (Oxide Form)	Conc., wt %							
	Sample 1		Sample 2		Sample 3		Sample 4	
	Element	Oxide	Element	Oxide	Element	Oxide	Element	Oxide
Al(Al ₂ O ₃)	2.27	4.29	28.7	54.2	1.38	2.61	0.03	0.06
Ba(BaO)	0.222	0.248	0.012	0.013	0.386	0.431	<0.002	<0.002
Ca(CaO)	3.07	4.30	0.07	0.10	6.76	9.46	0.01	0.01
Ce(CeO ₂)	0.252	0.309	<0.02	<0.02	0.328	0.403	<0.02	<0.02
Cr(Cr ₂ O ₃)	0.02	0.03	0.05	0.07	0.12	0.18	<0.01	<0.01
Cu(CuO)	0.02	0.02	0.03	0.04	0.13	0.16	0.03	0.04
Fe(Fe ₂ O ₃)	0.18	0.26	0.15	0.21	0.51	0.73	0.06	0.09
K(K ₂ O)	2.18	2.63	0.34	0.41	--	--	<0.05	<0.6
La(La ₂ O ₃)	0.163	0.191	<0.005	<0.006	0.244	0.286	<0.005	<0.006
Mg(MgO)	0.20	0.33	0.15	0.25	1.10	1.82	<0.01	<0.02
Mn(MnO)	0.02	0.03	0.02	0.03	0.03	0.04	<0.01	<0.01
Mo(MoO ₃)	0.07	0.10	0.01	0.02	0.03	0.04	<0.2	<0.3
Na(Na ₂ O)	<0.2	<0.2	0.24	0.32	<0.2	<0.3	<0.02	<0.02
Ni(NiO)	<0.02	<0.03	<0.02	<0.02	<0.02	<0.02	0.05	0.06
Si(SiO ₂)	2.60	5.56	12.4	26.6	5.06	10.8	46.0	98.4
Sn(SnO ₂)	<0.05	<0.06	<0.05	<0.06	<0.05	<0.06	<0.05	<0.06
Sr(SrO)	0.125	0.148	0.005	0.006	0.202	0.239	<0.001	<0.001
Te(TeO ₂)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
U(UO ₂)	56.6	64.2	0.87	0.99	41.2	46.7	0.29	0.33
W(WO ₃)	0.56	0.77	0.17	0.24	0.18	0.25	0.94	1.30
Zn(ZnO)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Zr(ZrO ₂)	13.4	18.1	9.37	12.7	17.0	23.0	0.04	0.05
Cl ^{-b}	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	--	--
NO ₃ ^{-b}	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	--	--
SO ₄ ^{=b}	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	--	--
Total	101.5		96.1		97.1		100.3	

^aUnless otherwise noted, data obtained with ICP/AES.

^bDetermined by ion chromatography.

down to a level suitable for trace boron measurements; and (3) use of the fusate requires standards prepared in the lithium tetraborate matrix and may contribute to increased uncertainty in the values measured.

2. Elimination of Boron after Tetraborate Fusion

The second problem listed above (ICP boron memory) can be eliminated by removing the bulk of the boron from the lithium tetraborate solution before ICP/AES analysis. For this purpose, we considered treatment with HF, methyl alcohol, or boron-specific resin IRA-743. To evaluate the effectiveness of each approach, a synthetic solution was prepared: 1 mg each of Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Ni, Pb, Sn, Sr, Tl, Zn, Zr, and V, plus ~155 mg B, were diluted to 50 mL.

Table 2 shows the recovery of the cations of interest for separately treated duplicate portions of the synthetic solution after two successive treatments with HF. In the first treatment, 10 mL HF was added to the mixture in a Teflon beaker and heated to gentle dryness. The residue was dissolved in 2 mL of 6M HCl, 2 mL of HF was added, and the solution was again heated to dryness. This residue was dissolved in 5 mL of 6M HCl and diluted to 50 mL for ICP/AES analysis. Results of a tetraborate reagent blank taken through the entire procedure are also shown.

The results from treatment with HF to eliminate the bulk of the boron from solution after fusion were very encouraging, with 2/3 of the boron being removed and all other elements in the mixtures being recovered. The removal can probably be increased to <250 $\mu\text{g B/mL}$ (acceptable for ICP/AES) if more HF (10 mL) is used in the second treatment and/or a third treatment is added before analysis. We note that determination of certain elements is not

Table 2. Recovery of Cations from Tetraborate Solution^a after HF Treatment

	Recovery, ^b $\mu\text{g/mL}$		
	Reagent Blank	Solution A1	Solution A2
B	926	910	992
Al	8.4	28.2	25.8
Ba	<0.1	20.0	20.0
Be	<0.05	18.7	18.8
Ca	0.2	20.1	20.2
Co	<0.1	19.9	19.9
Cr	<0.1	19.0	19.1
Cu	<0.1	18.9	19.0
Fe	0.49	20.0	20.0
Cd	<0.1	19.4	19.5
Mg	<0.1	19.2	19.3
Mn	<0.1	19.3	19.4
Ni	<0.2	19.4	19.5
Pb	<0.3	19.6	19.6
Sn	2.0	23.2	23.3
Sr	<0.05	19.5	19.6
V	0.2	18.7	18.4
Zn	<0.1	18.6	18.7
Zr	0.35	20.7	21.0

^aStarting solution contains 20 $\mu\text{g/mL}$ cations, 3100 $\mu\text{g/mL}$ boron.

^bDetermined by ICP/AES with uncertainty of $\pm 10\%$.

compatible with the HF treatment. Silicon, for example, is lost during treatment by volatilization as SiF_4 ; rare earth elements may be lost from solution as insoluble fluorides. Consequently, separate provision is required for measuring these elements, either with an instrument tolerant to high levels of boron or with a different preparation of a separate sample portion.

Two treatments of the same cation mixture with methyl alcohol, 25 mL each time, showed >50% of the boron still remaining. It was therefore decided not to pursue this method.

The possibility of using the boron-specific resin of Rohm and Haas, IRA-743, was also considered. However, this material has a theoretical capacity for boron of less than 5.7 mg/mL, depending on the flow rate.¹ It seems best suited for removing smaller amounts of boron from water and, therefore, did not appear useful for routine use in this application.

B. Microwave Dissolution

1. Calibration of Microwave Oven²

Before sample preparation by the microwave oven method, the oven was calibrated by the following procedure.

- Weigh a Teflon beaker (1 liter or larger) to the nearest 0.01 g. (Glass beakers absorb microwave energy and must not be used.)
- Add 1 kg of water (or any suitable amount) to beaker at ambient temperature.
- Weigh the mass of water. Measure the water's temperature to $\pm 0.1^\circ\text{C}$.
- Put the beaker containing the water on the rotating carousel in the microwave oven and expose to microwave radiation (heat) for 2 min, recording the power level.

- After heating, immediately remove the beaker and contents; weigh the beaker and measure the temperature of the heated water.

The power absorbed (P_{watts}) was calculated from the following equation:

$$P_{\text{watts}} = \frac{(K) (C_p) (m) (DT)}{t}$$

where K = conversion factor for thermochemical calories/second to watts (-4.184)

C_p = heat capacity, thermal capacity, or specific heat of water (-1.0 cal g⁻¹ • C⁻¹)

m = mass of the water sample in grams

DT = final temperature minus initial temperature in degrees Celsius

t = time heated in seconds

If 1 kg of water is taken and heated for 2 min, the calibration equation simplifies to:

$$P_{\text{watts}} = (DT) (34.87)$$

With this calibration, P_{watts} can be related to the percent power setting of the microwave (see Table 3).

Table 3: Calibration of Sears Kenmore Microwave Oven

% Power	Water Mass, g	Temperature, °C		Time, min	Power, W
		Initial	Final		
50	699.98	24.8	34.8	2	244
100	710.38	25.0	45.6	2	510

Ideally, the microwave unit will have a linear relationship between the output power and the scale used in controlling the unit. The power available for heating can be evaluated so that the absolute power setting (watts) may be compared from one microwave unit to another (see Fig. 1).

Fig. 1. Percent Power vs. Absorbed Power for Two Microwave Units

2. Sample Preparation and Analysis Results

Six types of samples were prepared by the microwave-oven method³: portland cement, silicon dioxide, germanium glass, zeolites, corium-concrete, and soil.

a. Portland Cement

- Weigh 100 mg of portland cement (NBS SRM-1016) and place in the special 120 mL Teflon vessel.
- Add 2 mL HNO_3 and 5 mL of HCl-HF (7:3 volume ratio).
- Close the vessel with the cover containing a relief valve. Be sure that the ring seal surface is facing upward with the flat side down, and screw down the cover so that it is finger tight.
- Place the vessel in the capping station so that the lugs on the cap nest in the grooves of the inside ring of the tightening collar. (See the procedure manual for assembly.)

- Heat in the microwave oven for 2 min at 100% power.
- Let cool.
- Open vessel by using the capping station.
- Add 50 mL of 3% H_3BO_3 .
- Recap.
- Heat 5 min at 100% power.
- Cool. Transfer the clear solution to an appropriately sized volumetric flask and dilute to volume [200 mL if atomic absorption analysis (AA) is to be done for silicon determination]. Note: Our laboratory will not use this sample preparation method for ICP/AES analysis because of the high boron content.

Results from AA analysis for SiO_2 in Portland cement are given in Table 4.

The data show that the microwave dissolution is reliable and, therefore, can be used as an alternative to the more time-consuming standard fusion technique of sample preparation for determining silicon in cement.

Table 4. Atomic Absorption Analysis of
Portland Cement (NBS SRM-1016)
Prepared by Microwave Dissolution

	Wt. % SiO_2
Certificate Value	21.1
Measured Value	21.5

b. Silicon Dioxide Standard

The material used was silicon dioxide, Lot 08771 (99.9999% purity), from Spex Industries.

- Weigh 100 mg of this material and place in the 120-mL Teflon vessel.
- Add 3 mL HNO_3 plus 3 mL HCl-HF (7:3 volume ratio). Cap the vessel.
- Heat the vessel in the microwave oven for 3 min on 100% power.
- Let cool.
- Transfer the solution to an appropriately sized Nalgene polypropylene volumetric flask and dilute to volume. The concentration is 467 $\mu\text{g Si/mL}$ of solution.

This silicon standard solution (when needed) can be diluted and used for calibration of the ICP/AES or the AA instruments prior to the analysis of unknown samples. The above is a quick and easy method of preparing a silicon standard solution.

c. Germanium Glass

- Weigh 50 mg of germanium glass and place in the 120-mL Teflon vessel.
- Add 5 mL HCl . NOTE: Most likely, HNO_3 would be just as effective; HCl was used because our standard solutions are in an HCl matrix.
- Prepare up to four samples at one time.
- Cap the vessels and place them on the rack together; heat in the microwave oven for 10 min at 100% power.

- After cooling, check each sample for undissolved residues using a flashlight. (When this check was done, if we found that not all the samples were totally dissolved, the vessels were recapped, heated 10 min more at 100% power, and cooled. Without exception, complete dissolution was attained after this second heating.)

Solutions prepared by the above method were transferred to appropriately sized volumetric flasks and diluted to volume. The glass samples were diluted to 100 mL, and further dilutions of 5 to 25 mL were prepared. Duplicate portions of three different samples were dissolved and analyzed by ICP/AES. Results are given in Table 5.

Table 5. Results from Germanium Glass Replicate Analysis after Microwave Dissolution

	Conc., wt %		
	Na ^a	Al ^a	Rb ^b
19201	9.88	10.57	N/A ^c
	9.92	10.67	
19202	7.36	7.82	N/A
	7.47	7.77	
19301	3.26	N/A	11.24
	3.23		11.25

^aDetermined by ICP/AES with uncertainty of $\pm 3\%$.

^bDetermined by AA with uncertainty of $\pm 3\%$.

^cN/A = Not applicable.

These analyses of germanium glass are nonroutine and infrequently requested in the ACL. In preparation of such samples for analysis, the beaker/hot-plate treatment used previously was found to be very slow (taking days), whereas microwave dissolution was expedient and complete in minutes.

Because no like-matrix glass was available as a standard, the samples were prepared and analyzed in duplicate to assess the completeness of dissolution. Table 5 shows that the duplicate results are well within instrumental measurement capabilities, and thus, dissolution is apparently complete and reliable.

d. Zeolites

- Rapidly weigh 50 to 100 mg of zeolite sample material (powder, approximately 2- μ m particle size) and place in the special 120-mL Teflon vessel.
- Add 5 mL H₂O, 2-3 mL HNO₃, and 250 μ L HF.
- Cap the vessel and heat in the microwave oven for 2 min at 100% power.
- After cooling, transfer the solution to a Nalgene polypropylene volumetric flask and dilute to volume so that the final sample concentration is 1 mg/mL.

The above sample preparation scheme has successfully provided complete dissolution. However, occasionally, the solution appears turbid in the microwave vessel after the mixed acid treatment. When this occurs, 2 mL more of H₂O is added, and the solution is heated again for 2 to 3 min in the microwave oven at 100% power.

To assess the performance of the aforementioned dissolution conditions, data from replicate measurements of two zeolite samples are shown in Table 6. Precision of replicate samples is very good. However, experience shows that zeolite analyses can be deceiving with regard to accuracy. Because no like-matrix zeolite standard is available, it is difficult to assess accuracy.

Table 6. Weight Percent of Cations Found after Microwave Dissolution of Zeolites

	Concentration, wt %			
	Sample 1 ^a		Sample 2 ^a	
	A	B	A	B
Al	9.32	9.33	9.58	9.48
Ba	1.91	1.92	0.77	0.76
Ca	0.10	0.10	0.54	0.52
Fe	0.97	0.97	0.96	1.09
K	8.55	8.72	9.09	9.15
Li	2.41	2.44	2.56	2.52
Mg	0.30	0.30	0.29	0.29
Na	0.56	0.57	0.74	0.73
Si	19.1	19.1	17.8	18.8
Sr	0.872	0.880	0.125	0.122
Ti	0.07	0.07	0.06	0.06

^aDetermined by ICP/AES. Uncertainty of $\pm 5\%$ for major components, $\pm 20\%$ for minor components.

After this study, a zeolite was standardized by classical fusion and gravimetric analysis for aluminum and silicon. Comparison of the data in Table 6 with the standard values revealed some discrepancies for silicon determinations, and caution is advised in using the above procedure at this time for determining silicon in the zeolite matrix.

e. Corium-Concrete Mixture

- Weigh 50 to 100 mg of corium-concrete sample (pulverized if necessary) from the MCCI experiments and place in the special 120-mL Teflon vessel.
- Add 2 mL H₂O.
- Add acids, considering the expected composition. Some guidelines are:

1. Add 2 mL HNO_3 along with 2-3 mL HCl-HF (7:3 volume ratio).
  2. If Ag is present, add 3 mL HNO_3 and 500 μL HF only.
  3. If low in Si, add 250 μL HF, 2 mL HNO_3 , and 1 mL HCl .
  4. If no Ag and low in U, add 2 mL HNO_3 and 2-3 mL HCl-HF (7:3 volume ratio).
  5. If high in U, add 2 mL HNO_3 , and 1 mL HCl plus 2 drops HF (can always add a little more HF if needed). (Note: If too much HF is added, a green UF_4 precipitate is formed.)
- Cap the vessel and heat in microwave for 3 min at 100% power.
  - After cooling, open the vessel and inspect the solution (using a flashlight) for undissolved residues. If dissolution is not complete, adjust the acid mixture according to the guidelines above and try again. If ultimately unsuccessful, another approach to dissolution is required.
  - Transfer the solution to a Nalgene polypropylene volumetric flask and dilute to volume so that the final sample concentration is 1 mg/mL.

Dissolving these types of solidified aerosols and melts for multielement analysis by ICP/AES is challenging. No one technique is always successful because the samples are produced under different conditions and may be refractory. High temperatures are used in the MCCI experiments to study the behavior of the elements present under reactor meltdown conditions. To assess the effectiveness of the sampling and the analytical technique used for sample preparation, data are presented in Table 7. The analysis results are presented as weight percent of each measured element along with a calculated oxide value. The mass recovery

Table 7. Characterization of Corium-Concrete Sample Following Microwave Dissolution

Element (Oxide Form)	Concentration, ^a wt %			
	Sample 1		Sample 2	
	Element	Oxide	Element	Oxide
Al(Al ₂ O ₃)	0.33	0.62	0.38	0.72
Ba(BaO)	0.21	0.23	0.22	0.25
Ca(CaO)	2.99	4.18	2.93	4.10
Ce(CeO ₂)	0.26	0.32	0.27	0.33
Cr(Cr ₂ O ₃)	0.05	0.07	0.15	0.22
Cu(CuO)	0.01	0.01	0.12	0.15
Fe(Fe ₂ O ₃)	0.20	0.29	0.89	1.27
K(K ₂ O)	<0.3	<0.4	<0.3	<0.4
La(La ₂ O ₃)	0.170	0.199	0.193	0.226
Mg(MgO)	0.17	0.28	0.18	0.30
Mn(MnO)	<0.01	<0.01	<0.01	<0.01
Mo(MoO ₃)	0.11	0.16	0.14	0.21
Na(Na ₂ O)	<0.1	<0.1	<0.1	<0.1
Ni(NiO)	<0.01	<0.01	0.09	0.11
Si(SiO ₂)	2.30	4.92	2.37	5.07
Sn(SnO ₂)	<0.05	<0.06	<0.05	<0.06
Sr(SrO)	0.12	0.14	0.12	0.14
Te(TeO ₂)	<0.3	<0.4	<0.3	<0.4
U(UO ₂)	59.5	67.5	57.1	64.8
W(WO ₃)	0.80	1.11	0.94	1.30
Zn(ZnO)	<0.01	<0.01	<0.01	<0.01
Zr(ZrO ₂)	14.2	19.2	15.5	20.9
Cl ^{-b}	<0.01	<0.01	<0.01	<0.01
NO ₃ ^{-b}	<0.05	<0.05	<0.05	<0.05
SO ₄ ^{=b}	<0.05	<0.05	<0.05	<0.05
Total	99.2		100.1	

^aUnless otherwise noted, determined by ICP/AES. Uncertainty of ±3% for major components, ±10% for minor.

^bDetermined by ion chromatography.

achieved is very close to 100%. This suggests that no major sample components were overlooked in the analyses, and that the major elements were present in the assumed oxide form.

By varying the proportions of the HCl, HNO₃, and HF used in the dissolution and microwave heating in the closed Teflon vessel, the corium-concrete sample can almost always be completely dissolved, and separate preparations and measurements for Si and U are not necessary. Preparation blanks are negligible.

f. Soil

- Weigh 0.5 ± 0.050 g of soil (LCS 0287) into a special 120-mL Teflon vessel.
- Add 10 ± 0.1 mL Instra-Analyzed HNO₃. When any reaction subsides, cap the microwave vessel with the relief valve disk in place.
- Weigh the vessel assembly to the nearest 0.01 g.
- Heat two vessels at a time at 344 W for 10 min.
- Cool and again weigh the vessel assembly to the nearest 0.01 g. (Note: The final weight should be within 10% of the original weight. If not, the vessel has vented; the contents should be discarded and the sample redigested.) Pour the solution through 11-cm Whatman #40 filter paper and collect the filtrate in a 100-mL volumetric flask. Use water to rinse the digestion vessel, cap, and residue.
- Dilute the filtrate to 100 mL. It is now ready for metals analysis.

This digestion procedure has been approved by the U.S. Environmental Protection Agency (EPA).⁴ The method is not a total dissolution, but a digestion comparable to

the hot-plate treatment in use for environmental sample preparation in the EPA's Contract Laboratory Program (CLP).⁵ To avoid subsequent sample dilution that may be necessary for some analyses, the above procedure immediately dilutes the filtrate to 100 mL rather than 50 mL, as specified in the EPA procedure. The EPA LCS 0287 solid reference material was digested using both the Sears Kenmore and the CEM microwave units. Results are given in Tables 8 and 9. The elements of interest all fall within the control limits, except antimony.

Table 8. Digestion of EPA Laboratory Control Soil Sample (LCS 0287) Using Sears Kenmore Microwave Oven

Element	Element Concentration in Dry Soil, mg/kg				
	Expected Value	Control Limits		Measured Values	
		Lower	Upper	Sample A ^a	Sample B ^b
Al	325	225	424	317	329
Sb	211	127	294	(38)	(34)
As	917	635	1199	990	1050
Ba ^c	(4.8)	0	40	3	3
Be	19.4	16.5	22.3	22	22
Cd	45.4	35.7	55.1	43	44
Ca	196200	166800	225600	187000	189000
Cr	99.6	79.2	120	109	110
Co	144	125	162	153	163
Cu	6910	6006	7820	6900	6980
Fe	22430	17770	27080	24400	25400
Pb	236	188	285	225	233
Mg	118100	100400	129900	118000	118000
Mn	208	177	239	219	221
Ni	60.9	49.2	72.6	64	62
K ^c	(50)	0	1000	--	--
Ag	22.2	15.5	29.0	33	27
Se	39.2	19.1	59.4	45	48
Na ^c	(50)	0	1000	--	--
Tl	39.0	24.6	53.5	--	--
V	65.8	51.7	79.9	69	73
Zn	187	138	236	196	193

^aSample A mixed with 10 mL HNO₃ and heated for 10 min at 50% power (250 W).

^bSample B mixed with 10 mL HNO₃ and heated for 10 min at 70% power (350 W).

^cValues for these elements are below the Contract Required Detection Limit (CRDL), and the windows are given for advisory purposes only. Parentheses indicate that accuracy is uncertain.

Table 9. Digestion of EPA Laboratory Control Soil Sample (LCS 0287) Using CEM Microwave Oven

Element	Element Concentration in Dry Soil, mg/kg				
	Expected Value	Control Limits		Measured Values	
		Lower	Upper	Sample C ^a	Sample D ^a
Al	325	225	424	320	367
Sb	211	127	294	29	27
As	917	635	1199	974	948
Ba ^b	(4.8)	0	40	6.1	5.8
Be	19.4	16.5	22.3	21	20
Cd	45.4	35.7	55.1	41	40
Ca	196200	166800	225600	175000	173000
Cr	99.6	79.2	120	102	101
Co	144	125	162	149	150
Cu	6910	6006	7820	6620	6510
Fe	22430	17770	27080	22100	22100
Pb	236	188	285	215	211
Mg	118100	100400	129900	110000	111000
Mn	208	177	239	209	205
Ni	60.9	49.2	72.6	60	60
K ^b	(50)	0	1000	59	72
Ag	22.2	15.5	29.0	24	22
Se	39.2	19.1	59.4	28	21
Na ^b	(50)	0	1000	80	92
Tl	39.0	24.6	53.5	--	--
V	65.8	51.7	79.9	58	59
Zn	187	138	236	177	173

^aSamples C and D mixed with 10 mL HNO₃ and heated for 10 min at 55% power (340 W).

^bValues for these elements are below the CRDL, and the windows are given for advisory purposes only.

C. Parr Bomb Dissolution⁶

1. Sample Preparation and Analysis Results for Zeolites

The procedure used for preparation of zeolite samples was as follows.

- Rapidly weigh 50 to 100 mg of zeolite sample material and place in 23 mL Teflon cup.

- Add 2 mL H₂O, 2 mL HNO₃, 1 mL HCl, and 250 μ L HF.
- After any reaction subsides, cover the cup and place in the Parr bomb metal jacket.
- Fit the jacket with the spring and metal caps. Seal by turning down the knurled cap until hand tight.
- Put the Parr bomb into a preheated oven, 140 to 150° C, and keep at this temperature for three or more hours (overnight is often convenient).
- Wearing insulated gloves, remove Parr bomb and allow to cool to room temperature.
- Open the bomb. (When cool, the metal jacket is easily opened by hand.)
- Transfer the sample solution to a polypropylene volumetric flask and dilute to a concentration of about 1 mg/mL.

The same dissolution scheme used in the microwave preparations is applicable to the Parr bomb. The temperature and pressure should be held below 150° C and 1200 psig (~8 MPa). In a 23 mL bomb, the maximum inorganic sample loaded should be less than 1 g, and the acids used should not exceed 12 mL so that adequate free vapor space is present above the surface of the bomb contents. If these limits are observed and precautions are taken to avoid overheating, there should be no unusual hazards.⁵

Because of the elevated temperature and pressure attainable with this technique, granular samples (or especially stubborn material) are more successfully dissolved in the Parr bomb. The design of the cup uses a tapered closure between the Teflon cup and cover. A long sealing face tends to self-seal and minimizes the tendency for acid migration through the joint.

An acid digestion bomb has been developed for heating in the microwave. It is sealed using an O-ring.

Data from zeolite Parr bomb preparations are not presented here because they are analogous to those shown in Table 6. As mentioned before, precision of replicate samples is very good; however, experience shows that zeolite analysis can be deceiving with regard to accuracy, especially in silicon determinations. Because no like-matrix zeolite is available, it is difficult to assess accuracy. After this study, a zeolite was standardized by classical fusion and gravimetric analysis for aluminum and silicon. It does appear that some discrepancies exist for silicon determinations, and caution is advised in using the above procedure at this time for determining silicon in the zeolite matrix.

2. Sample Preparation and Analysis Results for Soil

- The procedure used for preparation of soil samples was as follows.
- Weigh 0.1 to 1.0 g (usually 0.25 g) of soil and place in 23-mL Teflon cup.
- Add 2 mL H_2O , 4 mL HNO_3 , and 1000 μL HF.
- After any reaction subsides, cover the cup and place in the Parr bomb metal jacket.
- Fit the jacket with the spring and metal caps. Seal by turning down the knurled cap until hand tight.
- Put the Parr bomb into a preheated oven, 140 to 150°C, and keep at this temperature for three or more hours (overnight is recommended).
- Wearing insulated gloves, remove Parr bomb from oven and allow to cool to room temperature.
- Open the bomb. (When cool, the metal jacket is easily opened by hand.)

- Transfer the sample to a Teflon beaker. Heat to dryness on a hot plate to eliminate most of the Si^{4+} and F^- that are present.
- Add 2 mL HNO_3 and heat to dryness twice more.
- Add 500 μL HNO_3 and let stand for 10 min, swirling a few times.
- Add 10 mL H_2O and warm on the hot plate for 10 min.
- Cool. Transfer the solution to a volumetric flask and dilute to a concentration of about 5 mg/mL.

This sample preparation typically gives total dissolution and is well suited for total uranium and/or uranium isotopic abundance measurements using thermal ionization mass spectrometry. A soil standard (Rocky Flats Soil No. 1, NBS SRM-4353) was taken through this scheme and analyzed for total uranium by kinetic phosphorimetry. Results are given in Table 10.

Table 10. Total Uranium Analysis after Parr Bomb Dissolution of NBS SRM-4353 Rocky Flats Soil

Element	Total Uranium, $\mu\text{g/g}$		Recovery, %
	Accepted Value	Found	
Total U	3.15	2.99	94.9
		3.07	97.5
		3.04	96.5
		3.14	99.7

As shown in Table 10, the dissolution and phosphorimetry measurements for total uranium provide nearly complete recovery and very reliable uranium results. In addition, complete dissolution of high silica soils using only mineral acids, as attainable with the Parr bomb procedure described, is a definite advantage for any subsequent analyses. Fusion methods commonly utilized for dissolution of such samples add considerable salts to the already complicated matrix and usually introduce a need to eliminate the added salts before analysis.

Thus, the mineral acid dissolution produces a cleaner solution matrix and often avoids additional sample-preparation effort.

IV. CONCLUSIONS

Sample preparation is often the most time-consuming aspect of chemical analysis. Fusions and bombs have been around for some time, but microwave techniques are new and are the most significant innovation in sample preparation. Closed-vessel microwave digestion/dissolution has provided a faster, cleaner, and more efficient method of generating a solution for multielement analysis of many different kinds of inorganic samples. Challenging samples require different methods of preparation, each optimized to the material which has to be decomposed. There is no single system that answers all the needs for sample preparation. The techniques being used in our laboratory have made it possible to prepare certain demanding sample types that would be impossible, or at least extremely difficult, to prepare by conventional methods. The preparation phase of analysis has moved from the dark ages of the beaker/hot plate and into the high tech age of the microwave oven. However, each technique offers advantages and disadvantages, as listed below.

<u>Preparation Method</u>	<u>Advantages</u>	<u>Disadvantages</u>
Hot Plate/Glass or Teflon Beakers	No real limit on sample size. Easy to handle and observe. Traditional. Inexpensive.	May be slow. Acids are depleted and require replenishing. Dissolution temperature is limited to the acid boiling point. Volatile elements may be lost. When larger quantities of acids are needed, impurities introduced are increased. Blanks from acid impurities increase the detection limit.

<u>Preparation Method</u>	<u>Advantages</u>	<u>Disadvantages</u>
Li ₂ B ₄ O ₇ Fusion/Pt Crucible	Easy. Dissolves samples to acid dissolution that resist beaker/hot plate treatment. Moderately fast.	Requires a muffle furnace for best results. Introduces a high solids concentration into the sample. For analysis, requires matrix matching. High boron "contaminates" the ICP/AES. May add unwanted impurities.

<u>Preparation Method</u>	<u>Advantages</u>	<u>Disadvantages</u>
Parr Bomb/ Teflon Cup in Steel Jacket	Temperatures above normal boiling point attainable. Pressure up to 1200 psig. Small amount of Acid. Acid does not evaporate. No contamination. Volatile elements retained. Teflon cups are replaceable.	Sample size is limited. Slow to heat and cool. Teflon is somewhat porous and loss can occur by diffusion. Can be dangerous. Requires an oven. Chlorine can diffuse into the Teflon cups.

<u>Preparation Method</u>	<u>Advantages</u>	<u>Disadvantages</u>
Microwave Oven/ Closed Vessel (special Teflon vessels with relief valve)	Easy and fast. Mineral acids heat rapidly. Pressure achieved aids dissolution. Small amount of acid required. No salts added. Volatile elements retained. No contamination. Relief valve safety feature. Vessels are translucent so dissolution observable. Can be automated. Usually one preparation accommodates multielement analysis.	Sample size is limited. Special equipment is needed--oven with exhaust, vessels, and capping station. Definitive procedures not established.

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