

UNCONTROLLED

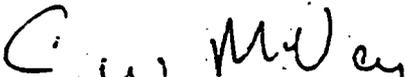
WEST VALLEY NUCLEAR SERVICES CO., INC.

ANALYTICAL CHEMISTRY METHOD
ANALYTICAL AND PROCESS CHEMISTRY

ACM-MICRO-1901, Rev. 2
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SAMPLE DIGESTION USING MICROWAVE DISSOLUTION

Approved by:


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Part I

1.0 PURPOSE

- > This method is used to rapidly digest samples of glass, melter feed, glass formers, and simulated melter waste for elemental analysis. It will add no contamination to the sample from fluxes or crucibles, as from using fusions, and yet provide a complete digestion with no losses from volatile compounds.

2.0 APPLICATION

The majority of materials to be analyzed in the production of glass are able to be digested with this method. It is primarily intended for analysis of glass and components used in the production of a glass product. Sample preparation of radioactive glass and melter feed must be done in cell.

3.0 DISCUSSION

3.1 Application

- > Analysis of glass products, after digestion in sealed vessels, can provide accurate results with a minimum amount of time spent on sample preparation. A sample size of about 50 mg diluted to 100 mL is used for glass samples. A sample size of about 100 mg diluted to 100 mL is used for melter feed, glass formers, and simulated melter waste. These sample sizes allow even trace elements (0.01 through 0.1 percent) to be analyzed while keeping projected dose rates at less than 1 mR.

3.2 Potential Errors Using This Method:

- > 3.2.1 Contamination: All beakers, flasks, digestion vessels, and sample grinding apparatus must be cleaned prior to use. All vessels are cleaned with an acid rinse (6.5) followed by a rinse with nanopure water.
- 3.2.2 Incomplete Dissolution: If samples are not prepared to a fine mesh, they may not completely dissolve using the parameters listed in the method. A longer digestion time may be necessary or a finer sample prepared.
- 3.2.3 Low values for certain elements such as silicon and boron may result if the dissolution vessels allow release of steam during the digestion.
- 3.2.4 Some elements (e.g., Ti) may not be completely dissolved using the microwave oven. An alkali fusion (or other) approach may be necessary to digest these type samples.
- > 3.2.5 slurry samples must be thoroughly mixed (stirred and/or shaken) prior to aliquoting. All aliquots are weighed to the closest 0.1 mg. Wet aliquots are taken using Eppendorf pipettors with disposable tips. Cut approximately 1/4 inch off the Eppendorf tips to insure a representative aliquot is taken.
- > 3.2.6 Simulated melter waste samples must be dried to a stable weight prior to digestion. Consistent results can not be obtained using a wet aliquot for these samples. Dry ~1 g wet sample 1 hour on plastic cover in the oven at 103°C. Dessicate 5 minutes and weigh sample. Replace in oven 15 minutes. Dessicate 5 minutes and weigh. Repeat until weight is stable.
- > 3.2.7 After adding Hydrogen Peroxide to digestion vessels, immediately recap vessels to prevent splattering of the sample.

4.0 REFERENCES

- 4.1 CEM Microwave Instruction Manual
- 4.2 PRD 5.0

Part II

5.0 EQUIPMENT

- 5.1 Plastic volumetric glassware.
- 5.2 Microwave Oven.
- 5.3 Vented digestion vessels for microwave use.
- 5.4 Analytical Balance.

6.0 REAGENTS AND STANDARDS

- 6.1 Nitric Acid (Specific Gravity 1.42)
- > 6.2 Hydrogen Peroxide (30 percent)
- 6.3 Hydrofluoric Acid (48 percent)
- 6.4 Scandium Solution (1,000 µg/mL)
- > 6.5 Acid Rinse: 200 mL nitric acid and 100 mL hydrogen peroxide diluted to 5 litres with nanopure water

7.0 SAFETY PRECAUTIONS

- 7.1 Care must be taken when handling concentrated acids, especially hydrofluoric acid. Plastic gloves should be worn when handling concentrated acids and a face shield worn when transferring the acids from stock containers to reagent bottles.
- 7.2 Standard laboratory safety precautions.
- 7.3 Do not use any sealed vessels in the microwave oven which do not have a safety release.
- 7.4 Microwave must be vented to a fume hood.

8.0 RECORDS

All measurement data and sample identification shall be recorded on the work sheet attachment A. The final result shall be recorded on the analytical request sheet (PRD 5.0).

9.0 CALIBRATION AND CONTROL

A visual inspection to assure that all solid matter has dissolved is the only requirement needed to insure a good dissolution of sample. No calibration or control of microwave oven is required.

10.0 PROCEDURE

- 10.1 Feed samples are first dried and calcined at about 700°C to remove organic materials, nitrates, water, and other volatile fractions which will be expected to off-gas in the melter. Feed samples may also be digested as a slurry.
- 10.2 All dried samples are ground to a fine powder using a ball mill or mortar and pestle. If the material is not a fine enough powder it may not go into solution with the parameters listed in this method. A longer digestion time may be required to achieve complete dissolution.
- > 10.3 Glass samples or slurry samples when solids loading is <450 kg: Approximately 50 mg (100 mg for slurries) of the finely ground sample are weighed on plastic weighing boats to the closest 0.1 mg. The sample is transferred into the vented digestion vessel by washing the weighing dish with deionized water. A volume of about 3 to 5 mL is used. One mL of nitric acid is added followed by 2 mL of hydrofluoric acid. The vessels are sealed and placed into the microwave oven.
- > 10.4 Program the microwave oven to a power setting of 90 percent and a time of about 1 minute (increase the time about 0.5 minutes for each additional samples placed in the oven). Turn on the exhaust blower and turntable, close the door and start the microwave oven. After the cycle is completed, remove the digestion vessels and allow them to cool. Add 1 mL of hydrogen peroxide and immediately recap digestion vessel. Sample should remain capped approximately 5 minutes to complete dissolution. (Addition of hydrogen peroxide improves manganese dissolution). Visually check to verify complete dissolution.
- > 10.5 Add 0.500 mL of the Sc stock solution* to the digestion vessel and transfer the contents quantitatively to a plastic volumetric flask. Dilute to the mark with deionized distilled water and mix. The sample is now ready for analysis by Inductively Coupled Plasma (ICP) or Atomic Absorption Spectroscopy (AAS).
- > 10.6 When solids loading increases to 450 kg or greater, solids may remain undissolved in the previous digestion method. The following method applies when solids loading is \geq 450 kg.
- > 10.7 Slurry samples or glass samples when solids loading \geq 450 kg: Weight approximately 100 mg (50 mg for glass) of slurry directly into digestion vessel. Add 5 mL nitric acid followed by 5 mL of hydrofluoric acid. The vessels are sealed and placed into the microwave oven.

* Scandium is added only if it is to be used as an internal standard for the ICP

- > 10.8 Program the microwave oven to a power setting of 40 percent and a time of about 5 minutes (increase the time about 2 minutes for each additional sample placed in the oven). Turn on the exhaust blower and turntable, close the door, and start the oven. After the cycle is completed, remove the digestion vessels and allow them to cool. Add 1 mL hydrogen peroxide and immediately recap the digestion vessel. Sample should remain capped approximately 5 minutes to complete dissolution. Visually check to verify complete dissolution.
- > 10.9 Add 0.500 mL of the Sc stock solution* to the digestion vessel and transfer the contents quantitatively to a plastic 100 mL volumetric flask. Dilute to the mark with deionized distilled water and mix. The sample is now ready for analysis by Inductively Coupled Plasma (ICP) or Atomic Absorption Spectroscopy (AAS).
- > 10.10 Although there is no actual time limit after the digestion for the analysis to take place, it is recommended to be done within a day or two. The sample should be shaken well just prior to analysis.

11.0 CALCULATIONS

Not Applicable

12.0 ATTACHMENTS

- > Attachment A.- Digestion Log Sheet

* Scandium is added only if it is to be used as an internal standard for the ICP

