

Trace Digestion Method

1. Introduction

The Trace Digestion Method is a dissolution process that is conducted in order to chemically dissolve solid sample materials, such as coal combustion byproducts and rocks, into a solution which contains trace elements in preparation for analysis on the ICP-MS. This process also eliminates any remaining organics in the samples.

This method is similar to the dissolution described in the ASTM Method D 6357-04 section 9.2

2. Interfaces with Other Methods

This method relies on:

EGL Method 29, Calibration of Laboratory Scales and Analytical Balances and
EGL Method 25, Method for Sample Login, Control, and Disposition.

This method prepares digested samples for:

EGL Method 16, The determination of Trace Elements in Samples by Inductively-Coupled Plasma Mass Spectrometry.

3. Materials and Equipment

Hotblock¹ capable of heating up to 120°C, 50 ml polypropylene digestion tubes, deionized water (DI), nitric acid (HNO₃), hydrochloric acid (HCl), hydrofluoric acid (HF), disposable chemical resistant gloves, eye protection, and lab coat.

4. Procedure

Weigh out approximately 0.2 grams of the sample material (laboratory prepared ash 525°C or LOI 525°C depending on original sample type) into a 50 ml digestion tube. Record the mass of the sample for future reference and for use in the ICP-MS method.

Add 4 ml of concentrated HNO₃ to the samples followed by 10 ml of a premixed 70/30 HCl/HF solution. Mix the digestion tube well by gently shaking and then cap it securely. Place the tubes in a Hotblock preheated to approximately 90°C and allow them to set for approximately 2.5 hours. After the allotted time, remove the tubes and cool for around 15 minutes. During this time raise the temperature of the Hotblock to approximately 120°C.

Remove the caps (lay out to ensure proper order for later in the method) and place the digestion tubes back into the hotblock which is at approximately 120°C and allow the samples to go to dryness without burning them (around 3 hours). Rinse the sides of the

¹ Any use of trade names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

tubes with DI water and allow the samples to go to dryness once again. Remove the tubes and allow enough time for cooling. At this time lower the temperature of the hotblock back to 90°C.

Add 2 ml of concentrated HNO₃ and 38 ml of DI water (for a final volume of 40 ml). Recap the tubes and place back into the Hotblock heated to roughly 90°C and heat for approximately one hour. Remove the digestion tubes from the Hotblock and let the samples sit overnight before analyzing.

5. Calibration and Quality Control Samples

Check the temperature of the Hotblock periodically with a thermometer. Adjust the temperature control on the front of the Hotblock if necessary. Digest current, approved lab standards, blanks, and duplicate samples in the same manner as described above.

6. Limits, Precautions, and Interferences

As a precaution, place the Hotblock under a hood to help dissipate heat and fumes. Place all acids under a hood and perform all the dispensing of the acids under a hood. During the entire process, all safety gear should be worn.

7. Acceptance of Data

This procedure does not produce any data and therefore acceptance of data does not apply.

8. Data Handling and Transfer

The sample masses are transferred from the balance to an Excel™ template electronically which is saved on the shared network drive.

9. References

American Society for Testing and Materials International [ASTM], 2007, Annual book of ASTM standards, section five, petroleum products, lubricants, and fossil fuels, Gaseous fuels; coal and coke: West Conshohocken, Pennsylvania, American Society for Testing and Materials International, v. 05.06, p. 608.

10. Attachments

None

11. History of Changes

Revision 0: initial issue