Standard Practice for Microwave Digestion of Industrial Furnace Feed Streams and Waste for Trace Element Analysis[1]

This standard is issued under the fixed designation D5513; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes the multi-stage microwave digestion of typical industrial furnace feed stream materials using nitric, hydrofluoric, hydrochloric, and boric acids for the subsequent determination of trace metals.

1.2 This practice has been used successfully on samples of coal, coke, cement raw feed materials, and waste-derived fuels composed primarily of waste paint-related material in preparation for measuring the following trace elements: Ag, As, Ba, Be, Cd, Cr, Hg, Pb, Sb, and Tl. This practice may be applicable to elements not listed above.

1.3 This practice is also effective for other waste materials (for example, flyash, foundry sand, alum process residue, cement kiln dust, etc.).

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

2.2 *Other Document:*

40 CFR 266 Subpart H, Hazardous Waste Burned in Boilers and Industrial Furnaces, Latest Revision[3]

3. Summary of Practice

3.1 A weighed portion of the feed stream material is combined with concentrated nitric acid in a polytetrafluoroethylene-lined digestion vessel, and heated in a microwave digestion unit. Following a programmed heating cycle, the vessel is vented and specified quantities of hydrofluoric and hydrochloric acids are added, and the mixture undergoes further microwave heating. Following this heating cycle, the vessel is vented and a specified quantity of boric acid solution is added, and the mixture undergoes a final microwave heating. Following this final heating cycle, the vessel is vented, the contents are quantitatively transferred to a volumetric flask and brought to volume. Typically, the only undissolved material is particulate carbon. If particulate matter is observed, filtration or centrifugation may be needed. The digested sample is ready for analysis.

4. Significance and Use

4.1 The U.S. Environmental Protection Agency Regulations, 40 CFR 266 , require that boilers, cement kilns, and other industrial furnaces utilizing waste-derived fuel adhere to specific guidelines in assessing potential metals emissions. A common approach for estimating potential emissions is performing total metals analysis on all feed stream materials. This practice describes a multi-stage microwave-assisted digestion procedure that solubilizes trace elements for spectroscopic analyses.

5. Apparatus

5.1 *Microwave Digestion Unit*—Equipped with an automatic turntable, pressure and/or temperature controller, and closed perfluoroalkoxy (PFA)-lined digestion vessels equipped with pressure relief/rupture membrane fittings or equivalent pressure relief device. The unit should comply with applicable regional, federal, or state standards for microwave leakage. The user must follow specific manufacturer's instructions for system installation.

NOTE 1—The digestion unit used in developing this practice was equipped with a pressure controller, automatic turntable, exhaust fan, and programming capacity. The unit delivered 1000 W of power at 100 % output. The lined digestion vessels consisted of a high-strength polymeric vessel body and cap, inner PFA liner and rupture membrane housing, and PFA vent stem. These vessels have a maximum operating pressure of 200

[1] This practice is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

psig. There are a number of suitable lab grade microwave systems available to the user that meet these minimum specifications. The user must follow specific manufacturer's instructions for using digestion vessels.

5.2 *Analytical Balance*—Capable of weighing to 0.001 g (1 mg).

5.3 *Labware*—High-density volumetric polyethylene or polypropylene flasks/sample containers are recommended for this practice. The user should be mindful of the quality limitations associated with volumetric non-glass labware.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean meeting the numerical requirements of Type II water as defined by Specifications D1193.

6.3 *Boric Acid Solution* (20 g/L)—Dissolve 20 g of boric acid (H_3BO_3) in water and dilute to 1 L. It may be necessary to place solution on a combination hot plate/magnetic stirrer and with the aid of a stir bar, allow the solution to mix under gentle heat until boric acid is fully in solution.

6.4 *Hydrochloric Acid,* 37 %, (sp. gr. 1.200), HCl.

6.5 *Hydrofluoric Acid,* 48 %, (sp. gr. 1.150), HF.

6.6 *Nitric Acid,* 70 %, (sp. gr. 1.400), HNO_3.

7. Hazards[5]

7.1 It is recommended that all operations involving concentrated acids be performed in a laboratory fume hood.

7.2 Hydrochloric acid is a highly corrosive chemical that is reactive with metals and most alkaline chemicals. Impervious gloves and chemical goggles are required for handling. See safety data sheet (SDS) for additional information.

7.3 Hydrofluoric acid is a highly corrosive chemical that is reactive with metals and water or steam. Additionally, HF specifically attacks silicate glass making certain fume hood enclosures susceptible to damage. Impervious gloves and chemical goggles are required for handling. See SDS for additional information.

7.4 Nitric acid is a highly corrosive chemical that is reactive with metals and most organic materials. Impervious gloves and chemical goggles are required for handling. See SDS for additional information.

7.5 Some samples undergoing microwave digestion can exhibit a rapid pressure rise within the digestion vessel. The potential exists for this type of sample to rupture the rupture membrane and liberate corrosive gases. Because of this, the microwave unit must be vented to a fume hood for proper evacuation of vapors.

8. Sample

8.1 Although feed stream materials are generally pulverized powders or liquids, the homogeneity of some feed stream materials can be uncertain. The laboratory sample should be thoroughly mixed or homogenized prior to withdrawing a portion for analysis. This practice assumes that non-liquid feed streams are pulverized powders at the time of sample preparation.

NOTE 2—If a non-liquid feed stream material is not in the form of a pulverized powder, it may need to be reduced in particle size to pass through a No. 100 sieve.

9. Calibration and Standardization

9.1 Although equipment manufacturers specify general power output ratings for microwave digestion units, it is important to verify the actual power output of a specific unit. It is recommended that this microwave power check procedure be performed monthly.

9.1.1 *Power Check Procedure at 100 % Instrument Power:*

9.1.1.1 Remove from the instrument cavity the turntable, drive lug, and all vessels.

9.1.1.2 Adjust the instrument cavity exhaust to minimum air flow (refer to the manufacturer's instructions).

9.1.1.3 Program the instrument for 4-min time and 100 % power.

9.1.1.4 Transfer 2000 ± 2 mL of room temperature (19 to 25°C) water into a 2-L polypropylene beaker.

9.1.1.5 Measure and record the initial water temperature (T_i) to the nearest 0.1°C.

9.1.1.6 Place the beaker in the right front corner of the instrument cavity (as you face the front of the instrument). This position closely approximates the position of a digestion vessel during processing.

9.1.1.7 Heat the water for the programmed time.

9.1.1.8 When the heating cycle is complete, immediately remove the beaker from the cavity, thoroughly stir the water to ensure even heat distribution, and measure the final temperature (T_f) to the nearest 0.1°C.

9.1.1.9 Calculate the delivered power in accordance with the following equations:

$$\text{Power (watts)} = \Delta T \times (35\ W/{}^\circ C) \quad (1)$$

where:

ΔT = $T_f - T_i$.

W/°C = $$\frac{K \times Cp \times M}{t}$$

where:

W = watts,

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[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[5] This information taken from the *NIOSH Guide to Chemical Hazards*, U.S. Depart. of Health and Human Services, June 1990.