



Designation: D5513 – 22

Standard Practice for Microwave Digestion of Industrial Furnace Feed Streams and Waste for Trace Element Analysis¹

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, fly ash, foundry sand, alum process residue, cement kiln dust, etc.).

1.4 The values stated in SI units are to be regarded as standard. Other units of measurement in parentheses in this standard are informational.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 8.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

D5681 Terminology for Waste and Waste Management

2.2 *Other Document:*

40 CFR 266 Subpart H, Hazardous Waste Burned in Boilers and Industrial Furnaces, Latest Revision³

3. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology D5681.

4. Summary of Practice

4.1 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.

5. Significance and Use

5.1 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.

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.