~~Designation:D7442–08~~ Designation: D 7442 – 08a

Standard Practice for Sample Preparation of Fluid Catalytic Cracking Catalysts and Zeolites for Elemental Analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy[1]

This standard is issued under the fixed designation D 7442; 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 covers uniform dissolution techniques for preparing samples of fluid catalytic cracking catalysts (FCC) and exchanged zeolitic materials for analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). These techniques describe standardized approaches to well-known, widely used laboratory practices of sample preparation utilizing acid digestions and borate salt fusions. This practice is applicable to fresh and equilibrium FCC catalysts and exchanged zeolite materials.

~~1.2~~

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

1.3 *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.*

2. Terminology

2.1 *Acronyms:*

2.1.1 *FCC*—Fluid Catalytic Cracking

2.1.2 *FCCU*—Fluid Catalytic Cracking Unit

2.1.3 *ICP-AES*—Inductively-Coupled Plasma-Atomic Emission Spectroscopy

3. Summary of Practice

3.1 Three preparation techniques are presented for converting solid, power samples into clear, dilute acid solutions suitable for analysis by ICP-AES. The three techniques presented are Perchloric Acid Digestion, Sulfuric Acid Digestion, and Lithium-Borate Fused Dissolution. Other techniques may be possible; however, these three approaches are established, widely used laboratory techniques for preparing FCC catalyst and catalyst-like samples.

3.2 Powder samples are heat-treated for 1 to 3 h to remove volatile components prior to further preparation by any of these three techniques.

3.3 The Perchloric Acid and Sulfuric Acid techniques involve dissolving small aliquots of heat-treated sample in the respective acid liquors and diluting the resulting solutions to the appropriate analytical volume. These techniques require boiling acid solutions in platinum or polytetrafluoroethylene (PTFE) labware and shall be used in appropriate fume hoods. The Perchloric Acid Digestion shall *never* be used in a standard fume hood.

3.4 The Lithium Borate Fused Dissolution technique involves dissolving small aliquots of heat-treated sample in a molten flux of lithium metaborate and lithium tetraborate salts, dissolving the resulting flux solution in a dilute nitric acid solution, and diluting the clear, concentrated specimen solution to an appropriate analytical volume. This technique must be performed in an operational fume hood and can be performed manually or may utilize the advantages of an automated fluxer. The optimal ratio of flux to sample, as well as fusion temperature needed, will vary depending on sample matrix.

4. Significance and Use

4.1 The chemical composition of catalyst and catalyst materials is an important indicator of catalyst performance and is a valuable tool for assessing parameters in a FCCU process. This practice will be useful to catalyst manufacturers and petroleum refiners for quality verification and performance evaluation, and to environmental authorities at the state and federal levels for

[1] This practice is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.03 on Chemical Composition .
~~Current edition approved April 1, 2008. Published April 2008.~~
Current edition approved Sept. 1, 2008. Published September 2008. Originally approved in 2008. Last previous edition approved in 2008 as D 7442–08.

*A Summary of Changes section appears at the end of this standard.

evaluation and verification of various compliance programs.[2, 3, 4]

4.2 Catalysts and catalyst type materials are difficult to prepare for analysis by ICP, and although the techniques presented in this practice are common, there is wide variation among laboratories in sample pretreatment and digestion recipes. This practice is intended to standardize these variables in order to facilitate the utility of comparative data among manufacturers, refiners, and regulatory agencies.

5. Apparatus

5.1 *Muffle Furnace*— at 1000 to 1100°F (538 to 593°C). — at 538 to 593°C.
5.2 *Analytical Balance*.
5.3 *Digestion Vessels*—platinum dish or PTFE beaker.
5.4 *Volumetric Flasks*—Class A glass, 250 mL.
5.5 *Automated Fusion Machine*—alternate to manual procedure.
5.6 *Crucible*—Pt $_{95\%}$/Au$_{5\%}$ high-form.

6. Reagents

6.1 All reagents should conform to American Chemical Society (ACS) specifications.[5] Ultra high purity standards and reference materials are commercially available from recognized vendors.
6.2 *Perchloric Acid*, concentrated, 69 to 72 %.
6.3 *Hydrofluoric Acid*, concentrated, 48 %.
6.4 *Sulfuric Acid*, H_2SO_4, concentrated, 94 %.
6.5 *Nitric Acid*, HNO_3, concentrated, 65 %.
6.6 *Hydrochloric Acid*, 1:1 HCl (concentrated HCl, 38 %, diluted 1:1).
6.7 *Hydrogen Peroxide*, 3 %.
6.8 *Lithium Borate Fluxes*, lithium tetraborate, or metaborate, or both.
6.9*Boric Acid Solution*
6.9 *Boric Acid Solution, 2 to 3 %*.

7. Preparation of Powder Samples

7.1 Catalysts and catalyst type sample powders contain small amounts of moisture and other volatile materials that must be removed to eliminate potential error in the analysis. Typically, 50 g of powder sample are heated in air in a laboratory furnace at 1000538 to 1100°F (538 to 593°C) 593°C for 1 to 3 h to remove volatile components prior to further preparation by any of these three techniques.

7.2 The bed depth of catalyst during the heat treatment should typically be 1 in.25.4 mm or less. The heat-treated specimen should be thoroughly blended upon cooling, since some particle size segregation normally occurs during the heat treatment step.

7.3 The heat-treated specimen should remain in a desiccator until use to prevent re-adsorption of ambient moisture.

8. Hazards

8.1 *Hazards Common to All Mineral Acids*:

8.1.1 Wear suitable gloves, eye protection, and proper protective clothing to protect in the event of splashes and spills. Dilutions shall be performed by adding acid to water, not the other way around. Limit quantities in storage to what is needed for the next few weeks.

8.1.2 Boiling acid solutions can be particularly dangerous, and the elevated temperature typically increases the severity of the hazardous properties. Particular care and advance preparation shall be given to work with tasks involving acid solutions under these conditions.

8.2 *Hazards Specific to Perchloric Acid*:

8.2.1 When not handled properly, perchloric acid can be a very dangerous reagent. Digestions with perchloric acid should be performed only in a fume hood specifically designed for its unique hazards and properties. This hood shall have a water washdown system, operated according to the manufacturer's specifications and instructions. This system is required to prevent buildup of explosive perchlorate salts in the duct work.

8.2.2 Solutions with perchloric acid shall never be boiled to dryness. Careful, attentive observation of techniques using perchloric acid is imperative for safe use.

8.2.3 Perchloric acid should not be mixed or used with organic materials if there is a possibility that the temperature will become elevated beyond ambient levels.

---

[2] Dean, John R., *Practical Inductively Coupled Plasma Spectroscopy*-, John Wiley, New York, 2005.
[3] Gaines, Paul, "ICP Operations," at www.ivstandards.com/tech/icp-ops .
[4] Segal, Eileen B., "First Aid for a Unique Acid: HF," *Chemical Health and Safety*, Sept/Oct 1998, Vol 5, Sept/Oct 1998, p. 25.
[5] *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.