Designation: D 7442 – 08

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 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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 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

[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.

[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, p. 25.