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Standard Test Method for Total Nickel in Fresh Alumina-Base Catalysts[1]

This standard is issued under the fixed designation D4481; 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 test method covers the determination of nickel in fresh alumina-base catalysts and has been tested at nickel concentrations from 2.5 to 60 weight %, expressed as nickel oxide (NiO).

~~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 its use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D7442 Practice for Sample Preparation of Fluid Catalytic Cracking Catalysts and Zeolites for Elemental Analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy

E105 Practice for Probability Sampling of Materials

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E456 Terminology Relating to Quality and Statistics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 The test specimen (as received) is treated with concentrated hydrochloric acid to solubilize the nickel. If necessary, nickel is recovered from any insoluble residue by potassium pyrosulfate fusion, after hydrofluoric-sulfuric acid treatment to remove silica. Ammonium citrate is added to complex the aluminum and buffer the solution. Nickel is precipitated as nickel dimethylglyoxime, Ni $(C_4H_{\ 7}O_2N_2)_2$, at a weakly alkaline pH. The precipitate is washed and weighed as Ni $(C_4H_7O_{\ 2}N_2)_2$ after drying at 120°C.

3.2 A separate test specimen is taken to determine loss on ignition (LOI) at 550°C. The value is used to calculate the nickel as percent nickel oxide (NiO) on a 550°C dry basis.

4. Significance and Use

4.1 This test method sets forth a procedure by which catalyst samples can be compared either on an interlaboratory or intralaboratory basis. It is anticipated that catalyst producers and users will find this method of value.

5. Interferences

5.1 Cobalt, molybdenum, and aluminum do not interfere. Interferences by elements that precipitate as hydroxides, such as iron, chromium, aluminum, lead, tin, manganese, titanium, and zirconium, are avoided by the addition of ammonium citrate before making the solutions ammoniacal. Copper, present in the 2 to 10 % range, tends to be co-precipitated with the nickel dimethylglyoxime. The only other metal ions precipitated by dimethylglyoxime are palladium, gold, and bismuth.

6. Apparatus

6.1 *Beakers*, 600-mL, 150-mL.

6.2 *Hotplate*.

6.3 *Furnace*, electric muffle. Calibrated and capable of maintaining temperatures of 550 ± 25°C, and 950 ± 25°C.

[1] This test method is under the jurisdiction of Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.03 on Chemical Composition.

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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.

6.4 *Platinum Dishes*, 100-mL[3] capacity.
6.5 *Mortar and Pestle*, agate, or equivalent mechanical grinder.
6.6 *Crucibles*, sintered-glass, 30-mL, medium porosity frit.
6.7 *Fiberglass Filter*, 3.2 cm.
6.8 *Drying Oven* capable of maintaining a temperature of 120°C.
6.9 *Vacuum Filtering Flask*, 500-mL.
6.10 *Filter Holder* and *Filter Disk*, Millipore 0.65 μm-47-mm diameter.
6.11 *pH Paper* to detect a value of 9.
6.12 *Screen*, 250-μm openings, 60-mesh.
6.13 *Analytical Balance*, capable of weighing to nearest 0.1 mg.