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Zinc Oxide Neutral

ZnO 81.39

DEFINITION

Zinc Oxide Neutral, freshly ignited, contains NLT 95.0% and NMT 98.0% of zinc oxide (ZnO).

IDENTIFICATION

- **A.** When strongly heated, it assumes a yellow color that disappears on cooling.
- **B.** The retention time of the zinc peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay.

ASSAY

PROCEDURE

Proceed as directed in [Zinc Determination \(591\), Ion Chromatographic Method](#).

Diluent: 0.2% (w/v) [hydrochloric acid](#)

Standard solution: 20 µg/mL of [USP Zinc Oxide RS](#) in *Diluent*, prepared as directed in the chapter

Sample solution: 20 µg/mL of Zinc Oxide Neutral from the freshly ignited Zinc Oxide Neutral in *Diluent*, prepared as directed in the *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of zinc oxide (ZnO) in the portion of Zinc Oxide Neutral taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of zinc from the *Sample solution*

r_S = peak response of zinc from the *Standard solution*

C_S = concentration of [USP Zinc Oxide RS](#) in the *Standard solution* (µg/mL)

C_U = concentration of Zinc Oxide Neutral in the *Sample solution* (µg/mL)

Acceptance criteria: 95.0%–98.0%

IMPURITIES

- **CHLORIDE AND SULFATE (221), Sulfate**

Standard solution: 0.020 N [sulfuric acid](#)

Sample: 0.1 g

Acceptance criteria: The *Sample* shows no more sulfate than corresponds to 2.3 mL of the *Standard solution* (2.2%).

Change to read:

- **▲ARSENIC (211), Procedures, Procedure 1▲** (CN 1-JUN-2023) : NMT 2 ppm

LEAD

Sample solution: Add 2 g to 20 mL of water, and stir well. Add 5 mL of [glacial acetic acid](#), and warm on a steam bath until the solution is dissolved.

Analysis: Add 5 drops of [potassium chromate TS](#).

Acceptance criteria: No turbidity or precipitate is produced.

Change to read:

MERCURY

Diluent A: Carefully add 50 mL of [nitric acid](#) to 450 mL of water, and mix.

Diluent B: Carefully add 10 mL of [nitric acid](#) to 490 mL of water, and mix.

Aqua regia: [NOTE—Prepare immediately before use.] [Hydrochloric acid](#) and [nitric acid](#) (3:1)

Stannous sulfate solution: [NOTE—The mixture is a suspension and should be stirred continuously during use.] Add 25 g of stannous sulfate to 250 mL of [0.5 N sulfuric acid](#).

Sodium chloride–hydroxylamine sulfate solution: 120 mg/mL each of [sodium chloride](#) and hydroxylamine sulfate in water

Potassium permanganate solution: 50 mg/mL of [potassium permanganate](#) in water

Standard stock mercury solution: [NOTE—Use of a commercially prepared mercury standard is recommended.] 1.0 mg/mL of mercury from mercuric chloride in *Diluent A*

Standard working mercury solution: 0.5 µg/mL of mercury in *Diluent B*, from *Standard stock mercury solution*

Standard solutions: Transfer 1-, 2-, 3-, and 4-mL aliquots of *Standard working mercury solution* to four separate 300-mL biological oxygen-demand (BOD) bottles. To each bottle add 5 mL of water and 5 mL of *Aqua regia*. Heat the bottle for 2 min in a water bath at 95°. Cool, and add 50 mL of water and 15 mL of *Potassium permanganate solution*. Mix thoroughly, and place in a water bath for 30 min at 95°. Cool, add 5 mL of *Sodium chloride–hydroxylamine sulfate solution*, and dilute with water to 200 mL. These solutions contain the equivalent of 2.5, 5, 7.5, and 10 ng/mL of mercury, respectively.

Sample solution: Transfer 2.0 g of Zinc Oxide Neutral into a 300-mL BOD bottle. To the bottle add 5 mL of water and 5 mL of *Aqua regia*. Heat the sample in a water bath for 2 min at 95°. Cool, and add 50 mL of water and 15 mL of *Potassium permanganate solution*. Mix thoroughly, and place in a water bath for 30 min at 95°. Cool, add 5 mL of *Sodium chloride–hydroxylamine sulfate solution*, and dilute with water to 200 mL.

Blank solution: To a 300-mL BOD bottle add 5 mL of water and 5 mL of *Aqua regia*. Heat the solution for 2 min in a water bath at 95°. Cool, and add 50 mL of water and 15 mL of *Potassium permanganate solution*. Mix thoroughly, and place in a water bath for 30 min at 95°. Cool, add 5 mL of *Sodium chloride–hydroxylamine sulfate solution*, and dilute with water to 200 mL.

Mercury detection instrument and Aeration apparatus: Proceed as directed in [▲Mercury \(261\), Procedures, Procedure 2 and Procedure 3▲](#)

(CN 1-Jun-2023) ·

Analysis: Add 5 mL of *Stannous sulfate solution* to a *Standard solution*, and immediately insert the bottle into the *Aeration apparatus*. Measure the absorbances of the *Standard solutions*. Repeat with the remaining *Standard solutions* and *Sample solution*. Perform a blank determination using the *Blank solution*, and make any necessary corrections. Plot the absorbances of the *Standard solutions* versus concentrations, in µg/mL, and draw the straight line best fitting the plotted points. From the graph so obtained, determine the concentration, in ppm of mercury, in the *Sample solution*.

Acceptance criteria: NMT 1 ppm

• IRON AND OTHER HEAVY METALS

Sample solution: Use the solution from the test for *Carbonate and Color of Solution*.

Analysis: Cool two separate 5-mL aliquots of the *Sample solution*. Add [potassium ferrocyanide TS](#) to the first aliquot, and add [sodium sulfide TS](#) to the second aliquot.

Acceptance criteria: White precipitates are formed in both aliquots.

• MAGNESIUM OXIDE

Blank: Carefully add 10 mL of concentrated [nitric acid](#) to 490 mL of water, and mix.

Standard solution: [NOTE—Use of a commercially prepared magnesium–inductively coupled plasma standard solution is recommended.] 25 µg/mL of magnesium in *Blank*

Sample solution: 4 mg/mL of Zinc Oxide Neutral in *Blank*

Instrumental conditions

(See [Plasma Spectrochemistry \(730\)](#).)

Mode: Inductively coupled plasma–atomic emission spectrometry

Analytical wavelength: 279.1 nm

Analysis

Samples: *Standard solution*, *Sample solution*, and *Blank*

Calculate the percentage of magnesium oxide in the portion of Zinc Oxide Neutral taken:

$$\text{Result} = (C_U/C_S) \times (M_{r1}/M_{r2}) \times 100$$

C_U = concentration of magnesium in the *Sample solution*, determined from the instrument (µg/mL)

C_S = concentration of Zinc Oxide Neutral in the *Sample solution* (µg/mL)

M_{r1} = molecular weight of magnesium oxide, 40.30

M_{r2} = molecular weight of magnesium, 24.31

Acceptance criteria: NMT 0.7%