Zn MW: 65.37 (Zn); 83.17 (ZnO) CAS: 7440-66-6 (Zn), 1314-13-2 (ZnO) RTECS: ZG8600000 (Zn), ZH4810000 (ZnO)

METHOD: 7030, Issue 2 **EVALUATION: PARTIAL** **Issue 1: 15 February 1984**
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OSHA : 5 mg/m^3 (ZnO fume & resp. fraction); 10 mg/m^3 (ZnO dust)
NIOSH: 5 mg/m^3 (ZnO); 15 mg/m^3/15 min (ZnO dust); STEL 10 mg/m^3 (ZnO Fume)
ACGIH: 5 mg/m^3, STEL 10 mg/m^3 (ZnO fume) 10 mg/m^3 (ZnO dust)

PROPERTIES: metal; valence 2; MP 419°C (Zn)

SYNONYMS: vary depending upon the compound.

SAMPLING

SAMPLER: FILTER (0.8-µm cellulose ester membrane)

FLOW RATE: 1 to 3 L/min

VOL-MIN: 2 L @ 5 mg/m^3
-MAX: 400 L

SHIPMENT: routine

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: not studied

BIAS: not measured

OVERALL PRECISION ($\hat{S}_{rT}$): not measured

ACCURACY: not determined

MEASUREMENT

TECHNIQUE: ATOMIC ABSORPTION, FLAME

ANALYTE: zinc

ASHING: conc. HNO_3, 6 mL; 140 °C

FINAL SOLUTION: 1% HNO_3, 100 mL

FLAME: air-acetylene, oxidizing

WAVELENGTH: 213.9 nm

BACKGROUND CORRECTION: D_2 or H_2 lamp, if needed

CALIBRATION: Zn^{2+} in 10% HNO_3

RANGE: 10 to 100 µg per sample [1]

ESTIMATED LOD: 3 µg per sample [2]

PRECISION ($\hat{S}_r$): 0.03 [1]

APPLICABILITY: The working range is 1 to 10 mg/m^3 for a 10-L sample. This is an elemental analysis, not compound specific. Aliquots of the samples can be analyzed separately for many additional metals.

INTERFERENCES: None known.

OTHER METHODS: This method is a revision and replacement of P&CAM 173 for Zinc [1,3]. Method 7300 (plasma emission) is an alternate analytical method. Method 7502 (X-ray diffraction) is specific for ZnO.

REAGENTS:

1. Nitric acid, conc.
2. Nitric acid, 1% (v/v). Add 10 mL conc. HNO_3 to 500 mL water; dilute to 1 L.
3. Calibration stock solution, 1000 µg/mL Zn. Commercial standard or dissolve 1.00 g Zn metal in minimum volume of (1+1) HCl; dilute to 1 L with 1% (v/v) HNO_3.
4. Air, filtered.
5. Acetylene.
6. Distilled or deionized water.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: cellulose ester membrane filter, 0.8-µm pore size, 37-mm diameter; in cassette filter holder.
2. Personal sampling pump, 1 to 3 L/min, with flexible connecting tubing.
3. Atomic absorption spectrophotometer with air-acetylene burner head and zinc hollow cathode lamp or electrodeless discharge lamp.
4. Regulators, 2-stage, for air and acetylene.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, With watchglass covers.*
6. Volumetric flasks, 100-mL.*
7. Micropipets, 1 to 100 µL.*
8. Hotplate, surface temperature 140 °C.

* Clean with conc. HNO_3 and rinse thoroughly with distilled or deionized water before use.

SPECIAL PRECAUTIONS: Perform all acid digestions in a fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 3 L/min for a sample size of 2 to 400 L. Do not exceed a filter loading of ca. 2 mg total dust.

SAMPLE PREPARATION:

NOTE: The following sample preparation gave quantitative recovery (see EVALUATION OF METHOD). Steps 4 through 9 of Method 7300 or other quantitative ashing techniques may be substituted, especially if several metals are to be determined on a single filter.

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 6 mL conc. HNO_3 and cover with a watchglass. Start reagent blanks at this point.
5. Heat on hotplate (140 °C) until sample dissolves and a slightly yellow solution is produced. Add additional acid as needed to completely destroy organic material.
6. When the sample solution is clear, remove watchglass and rinse into the beaker with 1% HNO_3.
7. Place the beakers on the hotplate and allow to go to near dryness (ca. 0.5 mL liquid remaining).
8. Rinse walls of beaker with 10 mL 10% HNO_3. Reheat 5 min to dissolve the residue, then allow to air cool.
9. Transfer the solution quantitatively to a 100-mL volumetric flask and dilute to volume with distilled or deionized water.

   NOTE: Dilute to a smaller volume, e.g., 10 mL, if required for sensitivity of analysis for other metals in the sample.

CALIBRATION AND QUALITY CONTROL:

10. Add known amounts, covering the range 0 to 100 µg Zn per sample, of calibration stock solution to 100-mL volumetric flasks and dilute to volume with 1% HNO_3.
11. Analyze the working standards together with the samples and blanks (steps 16 and 17).
12. Prepare a calibration graph of absorbance vs. solution concentration (µg/mL).
13. Aspirate a standard for every 10 samples to check instrument drift.
14. Check recoveries with at least one spiked media blank per 10 samples.
15. Use method of standard additions occasionally to check for interferences.

MEASUREMENT:

16. Set spectrophotometer according to manufacturer's recommendations and to conditions on page 7030-1.
    NOTE: Non-atomic absorption may require the use of D_2 or H_2 continuum background correction in some samples.
17. Aspirate standards and samples. Record absorbance readings.
    NOTE: If the absorbance values for the samples are outside of the range of the standards, dilute the solutions with 1% HNO_3, reanalyze, and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

18. Using the measured absorbances, calculate the corresponding concentrations (µg/mL) of zinc in the sample, C_s, and average media blank, C_b, from the calibration graph.
19. Using the solution volumes (mL) of the sample, V_s, and media blanks, V_b, calculate the concentration, C (mg/m^3), of zinc in the volume of air sampled, V (L):

$$C = \frac{(C_s V_s - C_b V_b)}{V}, \ mg/m^3.$$

EVALUATION OF METHOD:

Estimated LOD was 3 µg Zn per sample [2]. Since only the analytical procedure was studied, bias, overall precision, and accuracy were not determined.

REFERENCES:

[1] NIOSH Manual of Analytical Methods, 2nd ed., V. 5, P&CAM 173, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).
[2] User check, UBTL, NIOSH Seq.# 3990-P (unpublished, November 29, 1983).
[3] Criteria for a Recommended Standard...Occupational Exposure to Zinc Oxide, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 76-104 (1976).

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