

5.1.13

AOAC Official Method 957.22 Arsenic (Total) in Feeds

Colorimetric Test
First Action 1957
Final Action 1960

A. Reagents

- (a) *Arsenic trioxide*.—NIST As₂O₃ SRM 83, or equivalent.
- (b) *Magnesium oxide–magnesium nitrate slurry*.—Suspend 75 g MgO and 105 g Mg(NO₃)₂·6H₂O in enough H₂O to make 1 L. Agitate vigorously before addition to test sample. (Freshly prepared slurry gives ash which is easily disturbed by air currents.)
- (c) *Stannous chloride solution*.—Dissolve 40 g As-free SnCl₂·2H₂O in HCl and dilute to 100 mL with HCl. Effective as long as it discharges yellow color in test extract.
- (d) *Absorbing solution*.—Transfer with graduated cylinder 25 mL 1.5% HgCl₂ solution, and with pipet 3.75 mL 3M H₂SO₄ and 3.75 mL 0.006M KMnO₄, into 250 mL graduated cylinder. Dilute to 250 mL with H₂O and mix. Prepare fresh daily.
- (e) *Ammonium molybdate reagent*.—Dissolve 1 g (NH₄)₂MoO₄ in 100 mL 2.7M H₂SO₄. Solution keeps several weeks. (Prepare 2.7M H₂SO₄ by diluting 3M [9 + 1].)
- (f) *Hydrazine sulfate reagent*.—0.15%. Dissolve 0.15 g N₂H₄·H₂SO₄ in 100 mL H₂O. Solution keeps several weeks.

B. Apparatus

(Do not clean apparatus and glassware with detergents, as they interfere with color development. Haemo-Sol, available from Haemo-Sol, Inc., 7301 York Rd, Baltimore MD 21204, USA; Tel: +1-800-821-5676; www.haemo-sol.com; or equivalent, is satisfactory.)

- (a) *Evaporating dishes*.—70 mL; Coors No. 430, size 00A, or equivalent.
- (b) *Arsine evolution apparatus*.—Bend 6 mm id glass tubing at 120° angle ca 10 cm from one end and at 60° angle ca 15 cm from other end. Plug shorter end with glass wool impregnated with saturated Pb(CH₃COO)₂ solution and insert in rubber stopper, placed in top of 125 mL Erlenmeyer, so that end of tube projects just below stopper. Plug other end with unimpregnated glass wool and connect through rubber tubing to glass tube, constricted at lower end, that reaches to bottom of 50 mL large neck volumetric flask, or if preferred, 50 mL centrifuge tube, marked exactly at 50 mL and approximately at 20 mL.

C. Preparation of Test Solution

Weigh ground test portion containing 50 g As (unless aliquot is to be taken from digested solution) into 70 mL ashing dish. If >2.5 g test portion is used, increase amount of slurry and size of ashing dish. Add ca 10 mL well-mixed slurry, A(b), and enough H₂O to permit thorough mixing with stirring rod. Rinse stirring rod, and dry mixture at 100 °C. Ash 2–4 h at 550–600 °C. (Slight C residue does not interfere. Use care to avoid loss of ash.)

Cool and moisten residue with H₂O. Cover dish with watch glass and add ca 15 mL HCl (1 + 1). Let stand overnight, or heat on water

bath with agitation until ash dissolves. Filter through Whatman No. 30 paper into 125 mL Erlenmeyer. Rinse filter with enough hot H₂O, in several portions, to obtain ca 60 mL filtrate.

D. Preparation of Standard Curve

Dissolve 0.660 g As₂O₃ in 25 mL 10% (w/v) NaOH solution, dilute to 1 L with H₂O, and mix. Dilute 10 mL aliquot to 1 L with H₂O (1 mL = 5 g As). Transfer 0, 2, 4, 6, 8, 10, 12, and 14 mL aliquots from buret into 125 mL Erlenmeyers. Dilute each to ca 60 mL with H₂O and proceed as in E. Plot *A* against g As.

E. Arsine Evolution

Add ca 10 mL HCl, 2 mL KI solution, (15%: keep in dark: discard when solution turns yellow), and 0.5 mL SnCl₂ solution, A(c). Swirl, heat in water bath 5 min, and cool. Have all parts of evolution apparatus ready for immediate assembly, with ca 20 mL absorbing solution, A(d), in 50 mL volumetric flask or centrifuge tube marked at 50 mL. Add 5–6 g Zn, 30 mesh, to digested solution; quickly insert stopper containing glass tubing into Erlenmeyer and place delivery tube against bottom of volumetric flask or centrifuge tube so that bubbles will be small. Use few drops of H₂O to test for leaks between rubber stopper and Erlenmeyer. Connecting glass tube must be large enough so bubbles will not carry over Pb compounds from impregnated glass wool plug into absorption flask.

F. Color Development

After 30 min, disconnect rubber tubing, leaving delivery tube in receiving vessel so that any Hg arsenide on tube will be exposed to color-developing reagents. Add 1.0 mL ammonium molybdate reagent, A(e), and mix by forcing air through delivery tube. Add 1.0 mL hydrazine sulfate reagent, A(f), and again mix. Heat in boiling water bath 20 min. Rinse delivery tube with H₂O and remove. Cool to room temperature, dilute to 50 mL, and mix. Filter through tight glass wool plug in funnel or centrifuge. (Do not use filter paper, as color will be adsorbed.) Read *A* against H₂O at 750 nm. Maximum *A* is at 840 nm. Determine g As from standard curve.

- As 2.90 = arsanilic acid; As 2.24 = arsenosobenzene;
As 3.51 = 3-nitro-4-hydroxyphenylarsonic acid.
As 3.30 = 4-nitrophenylarsonic acid; As 3.47 = *p*-ureidobenzearsonic acid.

Arsenical drug, mg/kg = g As conversion factor/W

W = g test portion

- References: *Ind. Eng. Chem. Anal. Ed.* **15**, 408(1943);
24, 1821 (1952).
Sandell (1959) “*Colorimetric Determination of Traces of Metals*,” 3rd Ed.
JAOAC **40**, 455(1957).

CAS-7440-38-2 (arsenic)

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