

#### 14.1.04

### AOAC Official Method 991.11 Zinc in Serum

#### Flame Atomic Absorption Spectrophotometric Method First Action 1991 Final Action 1995

(Applicable to determination of 0.6–6.0 g Zn/mL animal serum.)

Results of the interlaboratory study supporting acceptance of the method:

$s_r = 0.05\text{--}0.07$ ;  $s_R = 0.07\text{--}0.39$ ;  $RSD_r = 1.2\text{--}11.4\%$ ;  $RSD_R = 6.1\text{--}12.9\%$

#### A. Principle

Serum is diluted with 0.03% aqueous Brij 35 and zinc is determined by flame AAS using standard solutions prepared in same matrix.

#### B. Apparatus

(a) *Atomic absorption spectrophotometer*.—Equipped with nebulizer assembly and 10 cm C<sub>2</sub>H<sub>2</sub>/air burner head. Flame lean blue. Zinc hollow cathode lamp operated with background correction (Smith–Hieftje or D<sub>2</sub> arc).

(b) *Polypropylene tubes*.—Disposable. 7 mL, with caps.

(c) *Automatic pipets*.—With disposable tips, delivering 0.40 mL.

(d) *Volumetric flasks*.—100 mL. Rinse first with 20% HNO<sub>3</sub>, then with water.

#### C. Reagents

(a) *Brij 35*.—(Polyoxyethylene 23 lauryl ether.) 30% solution w/v. Dilute 1 mL to 1000 mL with water to prepare 0.03% solution for diluting samples and standards.

(b) *External control*.—NIST SRM Nonfat Powdered Milk 1549. Certified (95% confidence interval) concentration 46.1 ± 2.2 mg Zn/kg. Dissolve 1.00 g in 50 mL H<sub>2</sub>O to prepare Zn concentration of 0.92 g/mL.

(c) *Zinc standard solutions*.—(1) *Stock standard solution*.—1000 mg/L. Obtain commercially or prepare by dissolving 1.000 g Zn metal in 30 mL HNO<sub>3</sub> (1 + 1) in 1 L volumetric

flask. Dilute solution to volume with water. (2) *Intermediate standard solution*.—10 g/mL. Dilute 1.0 mL stock standard solution to 100 mL with Brij 35 solution. (3) *Working standard solutions*.—Dilute 0.0, 1.0, 5.0, and 10.0 mL portions of intermediate standard solution to 100 mL with Brij 35 solution to prepare solutions containing 0.0, 0.10, 0.50, and 1.0 g Zn/mL, respectively.

#### D. Preparation of Test Solution

Thoroughly mix sera. Transfer 0.40 mL of each serum and external control solution to separate polypropylene tubes. Add 2.0 mL Brij 35 solution to each tube. Cap tubes and invert several times to mix contents. Analyze one external control for each 10 sera or fraction of 10.

#### E. Determination

Use the following AA parameters: wavelength 213.9 nm (background corrected); bandwidth 1.0 nm; flame air–C<sub>2</sub>H<sub>2</sub> (lean blue). Optimize system so that 0.50 g Zn/mL standard solution produces response of ca 0.200 AU. To do this, adjust aspiration rate and burner orientation to maximize signal. Operate Zn lamp at 1/3 maximum rated current to deliver optimum absorbance and signal-to-noise ratio.

Aspirate series of working standard solutions followed by external control solution. Repeat analysis if external control is not within range of 0.14–0.17 g Zn/mL. Aspirate solutions and external control. Prepare standard curve by plotting concentration, g/mL, vs *A* and determine concentration of solution. Dilute (1 + 1) with matrix solution and reanalyze any diluted sera that exceed standard curve. Multiply results by 6 (or 12) to account for dilution. Express results in g Zn/mL.

Reference: *JAOAC* 73, 619(1990).

CAS-7440-66-6 (zinc)