



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 955a

#### Lead in Blood

This Standard Reference Material (SRM) 955a is intended primarily for use in evaluating the accuracy of lead determinations in blood and for use in validating working or secondary reference materials for lead-in-blood analysis. A unit of SRM 955a consists of four vials of frozen bovine blood, one each of four different lead concentrations. Each vial contains approximately 2 mL of blood.

**Certified Concentration Values:** The certified concentration values of lead were determined using isotope dilution, inductively coupled plasma mass spectrometry (ID-ICPMS). The certified values of lead and their associated uncertainties are given below. The uncertainties are 95%/95% statistical tolerance intervals and reflect the combined effects of measurement imprecision and variability of actual lead concentration among vials. The intervals are constructed so that at a confidence level of 95%, they will include the concentrations for 95% of all vials of SRM 955a.

#### Lead Concentration at 22 °C

Vial No.	$\mu\text{g/dL}$	$\mu\text{mol/L}$
955a-1	$5.01 \pm 0.09$	$0.242 \pm 0.004$
955a-2	$13.53 \pm 0.13$	$0.653 \pm 0.006$
955a-3	$30.63 \pm 0.32$	$1.478 \pm 0.015$
955a-4	$54.43 \pm 0.38$	$2.627 \pm 0.018$

#### NOTICE AND WARNINGS TO USERS

SRM 955a is intended for "in vitro" diagnostic use only.

**Expiration of Certification:** This certification expires one year from the date of shipment from NIST. NIST will continuously monitor this SRM and should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Please return the attached registration form to facilitate notification.

**Use and Cautions:** Before use, a frozen sample should be allowed to thaw at room temperature (22 °C). The sample should be mixed by gently rolling, not shaking, the vial to remix any water that may have separated on freezing. Shaking will cause bubbles to form at the top of the sample. Do not use if clotted. The contents of a vial may be thawed, sample withdrawn and refrozen. Due to possible evaporative losses, it is advised that the contents of a vial not be used if less than 1/3 of blood remains.

For the certified concentration to be applicable to an analytical determination, a minimum sample of 100  $\mu\text{L}$  must be used.

Gaithersburg, MD 20899  
December 7, 1994  
(Revision of certificate dated 12-4-91)

Thomas E. Gills, Chief  
Standard Reference Materials Program

(over)

The technical and support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by R. Alvarez. Revision of this certificate was coordinated through the Standard Reference Materials Program by J.C. Colbert.

The ID-ICPMS analyses were performed by K.E. Murphy and P.J. Paulsen of the NIST Analytical Chemistry Division and density determinations by J.R. Moody of the NIST Analytical Chemistry Division. Confirmatory analyses using graphite furnace atomic absorption spectrometry (levels 3 and 4) and laser-excited atomic fluorescence spectrometry (levels 1 and 2) were done by R.D. Elms, G.C. Turk, and M.S. Epstein, IARD.

Statistical analysis of the experimental data was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The overall direction and coordination of the analyses were under the chairmanship of R.D. Vocke of the NIST Inorganic Analytical Research Division.

**Storage:** The SRM should be kept in its original vials and stored frozen at -20 °C. The vials should be stored in the box and aluminized bag supplied. Frost-free freezers should not be used because of temperature fluctuations.

#### PREPARATION AND ANALYSIS

This SRM was prepared in collaboration with the Division of Environmental Health Laboratory Sciences, National Center for Environmental Health and Injury Control, Centers for Disease Control (CDC). D.C. Paschal, E.W. Gunter, and D.T. Miller were responsible for its preparation. The source of blood for this reference material was from two cows that had been fed gelatin capsules containing lead nitrate at the CDC livestock facility in Lawrenceville, GA. At CDC, the blood was collected, analyzed for lead by an atomic absorption method, and blended under clean conditions to produce four pools at the desired lead concentrations. The four pools were treated with tripotassium EDTA at a concentration of approximately 1.5 mg/mL, and dispensed into polyethylene vials. The vials were then stored at -20 °C. Twenty vials were selected randomly from each of the four pools to test for homogeneity [1]. Two 100  $\mu$ L aliquots were taken from each vial, diluted with a matrix modifier, and analyzed in duplicate. The results indicated satisfactory homogeneity for each lot within the limits of precision of the method. The vials were shipped frozen to NIST.

At NIST, ten randomly selected vials at each concentration level were analyzed by a high accuracy method based on ID-ICPMS. In this method, the entire contents of a vial was weighed, spiked, and then analyzed, and the results, in ng/g were converted to  $\mu$ g/dL using the density of the material. The density at 22 °C of all four concentration levels is  $1.050 \pm 0.002$  g/mL. Because ID-ICPMS methods are inherently more accurate for the determination of lead in blood than other analytical methods, the certified concentrations are the means of the IDMS results.

#### REFERENCE

- [1] Miller, D.T., Paschal, D.C., Gunter, E.W., Stroud, P.E., D'Angelo, J., Determination of Lead in Blood Using Electrothermal Atomization Atomic Absorption Spectrometry with a L'vov Platform and Matrix Modifier, *Analyst* **112**, 1701-1704, (1987).