National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 955b

Lead in Bovine Blood

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

Certified Concentration Values: The certified concentration values of lead were determined using isotope dilution, inductively coupled plasma mass spectrometry (ID-ICPMS) and confirmed using graphite furnace atomic absorption spectrometry (GFAAS). 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 955b. The certified concentration values and their expanded uncertainties are given in Table 1 in μg/dL and μmol/L [1,2].

Table 1. Certified Lead Concentration Values and Uncertainties in SRM 955b

Concentration Levels	μg/dL	μmol/L
Level 1	4.04 $\pm$ 0.15	0.1951 $\pm$ 0.0072
Level 2	10.30 $\pm$ 0.10	0.4971 $\pm$ 0.0048
Level 3	20.59 $\pm$ 0.21	0.9937 $\pm$ 0.0101
Level 4	39.36 $\pm$ 0.36	1.899 $\pm$ 0.017

Expiration of Certification: The certification of SRM 955b is valid, within the measurement uncertainties specified, until **30 March 2003**, provided the SRM is handled and stored in accordance with the instructions given in the certificate. However, the certification is invalid if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The ID-ICPMS analyses were performed by E.S. Beary, S.E. Long, K.E. Murphy, and L.L. Yu of the NIST Analytical Chemistry Division and density determinations by R.D. Vocke of the NIST Analytical Chemistry Division. Confirmatory analyses using GFAAS were performed by M. Chaudhary-Webb of the Division of Environmental Health Laboratory Sciences of the Centers for Disease Control and Prevention (CDC).

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

Experimental design and statistical analysis of the data were provided by M.G. Vangel of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
Certificate Issue Date: 10 April 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

NOTICE AND WARNING TO USERS

Warning: SRM 955b is intended for "in vitro" diagnostic use only. This product was derived from bovine blood collected at a USDA licensed establishment. The supplier of this material has reported that all donor animals were sourced in the United States, a country in which Bovine Spongiform Encephalopathy is not known to exist.

Storage: The SRM should be kept in its original vials and stored frozen at or below -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.

Instructions for Use: 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 one-third of the original blood volume remains. For the certified concentration to be applicable to an analytical determination, a minimum sample of 100 μL must be used.

SOURCE, PREPARATION, AND ANALYSIS

Source of Material: This SRM was prepared in collaboration with the Division of Environmental Health Laboratory Sciences, National Center for Environmental Health of the CDC under the direction of D.T. Miller, D.C. Paschal, and S. Nesby. M. Chaudhary-Webb was responsible for the homogeneity testing of the prepared materials. The source of blood for this SRM was three steers that had been fed gelatin capsules containing lead nitrate at the CDC animal facility in Lawrenceville, GA.

Preparation of Material: At the CDC, the blood was collected, analyzed for lead by GFAAS [3] 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 1.5 mg/mL as anticoagulant, and dispensed into polyethylene vials. The bottles were tightly capped and stored at -20 °C. Ten vials were randomly selected from each of the four pools to test for homogeneity. One 100 μL aliquot was taken from each vial, diluted with matrix modifier, and analyzed in duplicate. The analytical results indicated satisfactory homogeneity for each vialed whole blood pool within the limits of precision of the method. Pools were then shipped frozen to NIST.

Analytical Methods: At NIST, ten randomly selected vials at each concentration level were analyzed by a high accuracy method based on ID-ICPMS [4]. In this method, the entire content of a vial was weighed and analyzed. The results on a per weight basis were converted to per volume basis using the experimentally determined density of the material. The density at 19 °C of all four concentration levels is 1.052 g/mL $\pm$ 0.002 g/mL. Certification was entirely based on the results of the ID-ICPMS measurements, a method for which potential sources of error have been explicitly evaluated. Confirmatory analyses using GFAAS were performed in the Division of Environmental Health Laboratory Sciences of the CDC.

REFERENCES

[1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).

[2] *Guide to the Expression of Uncertainty in Measurement,* ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994).

[3] 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**, pp. 1701-1704, (1987).

[4] Murphy, K.E., Paulsen, P.J., "The Determination of Lead in Blood Using Isotope Dilution Inductively Coupled Plasma Mass Spectrometry," Fresenius J. Anal. Chem., **352**, pp. 203-208, (1995).

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the internet http://ts.nist.gov/srm.