National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 928

Lead Nitrate

(Clinical Standard)

This Standard Reference Material (SRM) is certified for use as an assay standard for lead. It is intended primarily for use in the calibration and standardization of procedures employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures. It is supplied in a unit of 30 g.

Lead Nitrate Certified Value . . . . . 100.00 ± 0.03 percent

The certified value shown is based on the determination of lead in the material as received; drying being unnecessary. Lead is precipitated as the chromate using a slight excess of potassium dichromate (SRM 136c). The lead chromate is removed by filtration and the excess chromate ion determined spectrophotometrically. Details of this method are reported elsewhere.[1] The molecular weight of lead nitrate employed in the calculation is 331.219. This value is based on a mass-spectrometrically determined value of 207.209 for the atomic weight of lead in this sample. The uncertainty shown represents two standard deviations of a single measurement based on 16 determinations with allowances for known sources of possible error.

A semi-quantitative survey for trace metals by emission spectroscopy indicated the following: silver, 2 µg/g, chromium, 3 µg/g, nickel, 3 µg/g. No other metals were detected.

Source of Material: The lead nitrate used for this SRM was obtained from the J.T. Baker Chemical Co., Phillipsburg, NJ. This material was examined for compliance with the specifications for reagent grade lead nitrate as given in Reagent Chemicals, 5th edition, published by the American Chemical Society. The material met or exceeded the requirements in every respect.

Chemical analyses were performed in the NIST Inorganic Analytical Research Division by T.J. Murphy and J.W. Gramlich. Spectroscopic analyses were performed by C.S. Annell and D. Golightly of the United States Geological Survey.

The overall direction and coordination of technical measurements leading to certification were under the chairmanship of I.L. Barnes of the NIST Inorganic Anlytical Research Division.

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 T.W. Mears. Revision of the certificate was coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
April 5, 1994
(Revision of certificate dated 2-27-76)

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

NOTICE AND WARNING TO USERS

This SRM is intended for use as a standard for lead determination in clinical chemistry and "in vitro" diagnostic use only.

Expiration of Certification: This certification is valid for five years from the date of shipment from NIST. Periodic reanalysis of representative samples from this SRM lot will be performed, and if significant changes are observed within the five-year period, the purchaser will be notified by NIST. Please return the enclosed registration card to facilitate notification.

Storage of Crystalline SRM 928: This SRM should be stored in the tightly-closed original bottle under normal laboratory conditions. Tests show the material to be dry as-received and will not adsorb appreciable water when exposed to a 90% relative humidity atmosphere for 5 days.

INSTRUCTIONS FOR USE

Drying Instructions: No additional drying is required.

Preparation and Stability of Prepared Solutions: The solutions of SRM 928 are stable as described below. At the time of use, these solutions should be clear and display no turbidity.

Because of the instability of non-acidified aqueous lead solutions at the working levels it is recommended that three levels of concentration be used.[2]

(1) A stock standard solution containing 50 mmol/L is prepared by dissolving 1.6561 g of SRM 928 in ion-free water. If the solution is cloudy or a precipitate forms, add a few drops of ammonium hydroxide. Mix, dilute to 100 mL in a calibrated volumetric flask and transfer immediately to a previously acid-washed, water-rinsed, dry polyethylene bottle. This solution is stable for six months.[2]

The above directions are directly quoted from the reference given. Users are cautioned that carbonate-free ammonium hydroxide, not in excess of 0.2 mL, should be added. Otherwise, insoluble basic lead salts will form. However, experience at NIST indicates that this material will easily dissolve without cloudiness when high-purity water is used.

(2) An intermediate solution containing 500 µmol/L is prepared by a 1:100 dilution of the above stock solution. This solution may be stored in a capped polyethylene bottle at room temperature for one month.[2]

(3) Working standard solutions of 0.5, 1.0, 2.5, and 5.0 µmol/L should be prepared each time an analysis is performed.[2]

Note: Dilute aqueous lead standards remain stable for less than 3 h. Lead is readily adsorbed on the surfaces of glass and plastic containers and this reaction is accelerated by exposure to light, particularly ultraviolet light.[3] It is recommended that very dilute aqueous lead solutions be prepared in a darkened room and protected from light.[4]

REFERENCES

[1] Catanzaro, E.J., Murphy, T.J., Shields, W.R., and Garner, E.L., J. Res., NBS 72A, 261-267, (1968).

[2] Kopito, L., and Shwachman, H., Measurement of lead in blood, urine, and hair by atomic absorption spectroscopy, Standard Methods of Clinical Chemistry, Vol. 7, G.R. Cooper, editor-in-chief, pp. 151-162, Academic Press, Inc., New York, NY, (1972).

[3] Kopito, L., and Shwachman, H., J. Lab. Clin. Med. 70, 326-332, (1967).

[4] Fundamentals of Clinical Chemistry, N.W. Tietz, editor, pp. 852-857, W.B. Saunders Co., Philadelphia, PA, (1970).