



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 924a

Lithium Carbonate

This Standard Reference Material (SRM) is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures employed in clinical analysis and for the routine critical evaluation of the daily working standards used in these procedures. SRM 924a is supplied in crystalline form as a 30 g unit.

Certified Purity

Lithium Carbonate, mass fraction..... 99.867 (\pm 0.017) %

This certified value is calculated from the results of independent coulometric assays. The uncertainty was computed according to the CIPM approach [1], and is at the 95 % level of confidence. It includes material variability as well as within method sources of uncertainty which were statistically evaluated (Type A) or evaluated by other means (Type B). The relative molecular mass for lithium carbonate used in the calculations is 73.8915 and is based on the lithium isotopic abundance as determined by mass spectrometry in the SRM material.

NOTICE AND WARNINGS TO USERS

This is intended for "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 attached registration card to facilitate notification.

Stability and Storage: This SRM should be stored in its original bottle at room temperature. It must be tightly re-capped after use and protected from moisture and light.

Homogeneity: Small, but statistically significant, differences in purity between the bottles were detected in the 200 mg samples used for the coulometric measurements. This material variability is included in the uncertainty of the certified value. Therefore, a minimum sample of 200 mg should be used to relate analytical determinations to the certified value and its associated uncertainty.

Coulometric analyses were performed Division by K.W. Pratt; trace element analyses were performed by K.E. Murphy and P.J. Paulsen; lithium isotopic analysis was performed K.E. Murphy and R.D. Vocke, all of the NIST Analytical Chemistry Division.

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

The technical and 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
June 12, 1995

Thomas E. Gills, Chief
Standard Reference Materials Program

INSTRUCTIONS FOR USE

Drying Instructions: Samples of SRM 924a should be oven-dried at 200 °C for 4 h then cooled to room temperature in a desiccator prior to use.

Standard Stock Solutions: Lithium standard stock solutions prepared from SRM 924a are stable when stored in stoppered glass bottles, but concentration changes can occur due to evaporation. All such solutions should be clear and display no turbidity. The preparation of solutions specifically for use in either atomic absorption spectrometry or flame emission spectrometry is described in [2].

Source of Material: The lithium carbonate used for SRM 924a was obtained from the Aithaca Chemical Co., Uniondale, NY. The material was examined for compliance with the specification for reagent grade lithium carbonate as specified by the American Chemical Society [3] and was found to meet or exceed the minimum requirements in every respect. Mass spectrometric analyses indicate a $^6\text{Li}/^7\text{Li}$ ratio corresponding to that found in natural material.

Coulometric Assay: The assay value for this material was obtained by automated coulometric back-titration [4], to a strong acid endpoint (pH 7), of weighed Li_2CO_3 samples after addition of excess coulometrically standardized HCl and elimination of the product CO_2 . The certified value represents the result of 16 such titrations of samples from 8 randomly selected bottles from the entire lot of SRM 924a.

Trace Metal Analyses: Semiquantitative inductively coupled plasma mass spectrometric (ICP-MS) analyses performed to obtain general information on impurities indicate that the only species present in this SRM at a level greater than 10 $\mu\text{g/g}$ is calcium at 15 $\mu\text{g/g}$. The detection limit of several other species was above 10 $\mu\text{g/g}$ and these are not necessarily present in the sample. These include: bromine at $\leq 45 \mu\text{g/g}$, iron at $\leq 16 \mu\text{g/g}$, sodium at $\leq 13 \mu\text{g/g}$, and mercury at $\leq 12 \mu\text{g/g}$.

REFERENCES

- [1] "Guide to the Expression of Uncertainty in Measurement", ISBN 92-67-10188-9, 1st Ed., ISO, Switzerland, (1993).
- [2] Velapoldi, R.A., et.al., A Reference Method for the Determination of Lithium in Serum, NBS SP260-69, (July 1980).
- [3] *Reagent Chemicals*, 8th Ed., American Chemical Society, Washington, DC, (1993).
- [4] Pratt, K.W., *Anal. Chim. Acta*, 289, 135, (1994).