



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 924a

Lithium Carbonate

This Standard Reference Material (SRM) is intended for use as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures and for the routine critical evaluation of the daily working standards used in these procedures. A unit of SRM 924a consists of a single glass bottle containing 30 g of the material.

Certified Purity

Lithium Carbonate (Mass fraction): 99.867 % \pm 0.017 %

Certified Value and Uncertainty: This certified value is calculated from the results of independent coulometric assays. The uncertainty was computed according to the ISO/JCGM Guide [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. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [2].

Expiration of Certification: The certification of **SRM 924a** is valid, within the measurement uncertainty specified, until **01 May 2022**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). The certification is nullified 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 the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Metrological Traceability: The measurand is the total mass fraction for lithium carbonate. Metrological traceability is the SI derived unit for mass fraction (expressed as a percent).

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

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

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 09 February 2016
Certificate Revision History on Last Page

Steven J. Choquette, Acting Director
Office of Reference Materials

NOTICE AND WARNINGS TO USERS

This is intended for research use.

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 acid vapors.

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.

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 reference 3.

Source of Material⁽¹⁾: 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 (Li_2CO_3) as specified by the American Chemical Society [4] 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 [5], 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 carbon dioxide (CO_2). The certified value is the result of 16 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 the following elements: 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 $\leq 12 \mu\text{g/g}$.

⁽¹⁾ Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

REFERENCES

- [1] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Feb 2016); see also Taylor, B.N.; 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); available at <http://www.nist.gov/pml/pubs/index.cfm> (accessed Feb 2016).
- [2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000); available at <http://www.nist.gov/srm/upload/SP260-136.PDF> (accessed Feb 2016)
- [3] Velapoldi, R.A.; Paule, R.C.; Schaffer, R.; Mandel, J.; Machlan, L.A.; Garner, E.L.; Rains, T.C.; *A Reference Method for the Determination of Lithium in Serum*; NBS Special Publication 260-69, U.S. Government Printing Office: Washington, DC (1980); available at <http://www.nist.gov/srm/upload/SP260-69.PDF> (accessed Feb 2016).
- [4] *Reagent Chemicals*, 8th ed., American Chemical Society, Washington, DC (1993).
- [5] Pratt, K.W.; *Automated, High Precision Coulometric Titrimetry. Part II. Strong and Weak Acids and Bases*; *Anal. Chim. Acta*, Vol. 289 (2), pp. 135–142 (1994).

<p>Certificate Revision History: 09 February 2016 (Editorial changes); 24 January 2012 (Extension of certification period; editorial changes); 08 June 2006 (Technical and editorial changes); 26 April 2006 (Update of expiration date and editorial changes); 23 April 1996 (Revised); 12 June 1995 (Original certificate date).</p>

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.