National Institute of Standards and Technology
U.S. Department of Commerce

Date of Issue:
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Standard Reference Material® 924a
Lithium Carbonate
CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended for use as a chemical of known purity in the calibration and standardization of procedures and for the routine critical evaluation of the daily working standards used in these procedures.

Description: A unit of SRM 924a consists of a single glass bottle containing 30 g of material.

Certified Value: 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 [1]. The measurand is the total mass fraction for lithium carbonate. Metrological traceability is to the International System of Units (SI) derived unit for mass fraction (expressed as a percent). This certified value is calculated from the results of independent coulometric assays. The stated uncertainty is the expanded uncertainty, U, expressed at a level of confidence of approximately 95 % and calculated as $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guide [2]. The value of u_c is intended to represent, at the level of one standard deviation, the combined effects of material variability as well as random and systematic components of uncertainty. The value of the coverage factor, k, is 2. 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.

Certified Value for SRM 924a Lithium Carbonate

Lithium Carbonate (Mass fraction): 99.867 % ± 0.017 %

Period of Validity: The certified value delivered by **SRM 924a** is valid within the measurement uncertainty specified until **07 January 2029**. The certified value is nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Additional Information: Additional information is provided in Appendix A.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM at the time of purchase. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available free of charge through the NIST SRM website.

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Certificate Revision History on Page 2

Safety: SRM 924a is intended for research use. See the material safety data sheet for additional safety information.

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.

Use: 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. A minimum sample of 200 mg should be used to relate analytical determinations to the certified value and its associated uncertainty.

Standard Stock Solutions: Traceability to the SI is best maintained by preparing lithium standard stock solutions prepared from SRM 924a on a mass fraction basis (dilute to a known mass rather than a marked volume). Solutions are stable when stored, but 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.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication (NIST SP) 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Jan 2022).

[2] 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 https://www.bipm.org/en/publications/guides (accessed Jan 2022); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Jan 2022).

[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*; National Bureau of Standards Special Publication (SP) 260–69; U.S. Government Printing Office: Washington, DC (1980); available at https://www.nist.gov/system/files/documents/srm/SP260-69.PDF (accessed Jan 2022).

[4] *ACS 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).

Certificate Revision History: 21 January 2022 (Change of expiration date; updated format; editorial changes); 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).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Homogeneity: Small, but statistically significant differences in the mass fraction of lithium carbonate 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.

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 (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 $^6Li/^7Li$ 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 μg/g is calcium at 15 μg/g. The detection limit of several other species was above 10 μg/g and these are not necessarily present in the sample. These include the following elements: bromine at ≤45 μg/g, iron at ≤16 μg/g, sodium at ≤13 μg/g, and mercury at ≤12 μg/g.

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