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

Date of Issue:
14 November 2022

Standard Reference Material® 915c
Calcium Carbonate
CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for calibration and standardization of procedures for calcium (Ca) determinations employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures. Additionally, the certified values can be used to validate analytical methods for determining calcium and carbonate.

Description: A unit of SRM 915c consists of a single glass bottle containing approximately 20 g of material.

Certified Values: A NIST certified value is a value for which NIST has the highest confidence in that all known or suspected sources of bias and imprecision have been considered and any contributions they may make to measurement uncertainty have been quantified and are expressed in the reported uncertainty [1]. Certified mass fraction values for elements in SRM 915c, reported on a dry-mass basis, are provided in Table 1. The measurands in Table 1 are total mass fractions for each analyte reported and metrological traceability is to the International System of Units (SI) derived unit for mass fraction expressed as a percentage [2].

Table 1. Certified Mass Fraction Values for SRM 915c

Quantity	Mass Fraction[(a)] (%)	Coverage Factor, k
Calcium	40.023 ± 0.016	1.78
Carbonate	59.944 ± 0.023	1.80
Calcium Carbonate	99.947 ± 0.040	1.87

[(a)] Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty associated with the half width of a symmetric 95 % coverage interval for the mean of all bottles of SRM 915c because the underlying mass fraction is assumed to be the same for each bottle. To propagate this uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/k$ [3–4].

Non-Certified Values: Non-certified values are provided in Appendix A.

Additional Information: Additional information is provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 915c** are valid within the measurement uncertainty specified until **01 August 2042**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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. Registration will facilitate notification. 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 through the NIST SRM website (https://www.nist.gov/srm).

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

Safety: SRM 915c is intended for research use; not for human consumption.

Storage: The original unopened bottles of SRM 915c should be stored at room temperature (25 °C ± 10 °C). An open bottle must be protected from moisture and acid vapors and can be reused until the material reaches its expiration date, provided that the open bottle is tightly re-capped and stored at room temperature (25 °C ± 10 °C).

Use: Before use, the contents of the unopened bottle should be mixed thoroughly by inverting and rolling. Homogeneity of the material has not been evaluated for sample sizes smaller than those used by NIST. Therefore, the certified values may not be valid for test portions smaller than 150 mg.

Drying Instructions: Dry the material at 200 °C to 210 °C for 4 h. After the SRM has been dried, store it in a desiccator over anhydrous magnesium perchlorate.

Source of Material: The $CaCO_3$ used for this SRM was obtained from a commercial supplier. The material was examined for compliance with the specification for reagent grade $CaCO_3$ as specified by the American Chemical Society [5]. The material was found to meet or exceed the minimum requirements in every respect.

Assay Techniques: The coulometric assay value was obtained by automated back-titration [6] using coulometrically-standardized hydrochloric acid (HCl) as the excess added substance, with potentiometric detection of the strong acid endpoint after removal of evolved carbon dioxide (CO_2) and correcting for trace impurities. The gravimetric assay value was obtained by conversion to $CaSO_4$ and correcting for trace impurities in the $CaSO_4$ (gravimetric procedure based on [7]). Impurity values were reported by the manufacturer and by a semi-quantitative analysis by inductively coupled plasma mass spectrometry (ICP-MS).

Possible Interfering Species: It is the responsibility of the user to evaluate which species may interfere with the application of this SRM and to apply any necessary corrections that affect the given application. The list of non-certified values of significant impurities in Appendix A may be useful in this evaluation.

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 Nov 2022).

[2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at https://www.nist.gov/pml/special-publication-811 (accessed Nov 2022).

[3] JCGM 101:2008; *Evaluation of Measurement Data — Supplement 1 to the "Guide to the Expression of Uncertainty in Measurement" — Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Nov 2022).

[4] 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 (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Nov 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 Nov 2022).

[5] ACS; *Reagent Chemicals*; Online edition., American Chemical Society: Washington, DC (2017).

[6] 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).

[7] Moody, J.R.; Vetter, T.W.; *Development of the Ion Exchange-Gravimetric Method for Sodium in Serum as a Definitive Method*; J. Res. Natl. Inst. Stand. Technol., Vol. 101, pp. 155–164 (1996) available at https://nvlpubs.nist.gov/nistpubs/jres/101/2/j2mood.pdf (accessed Nov 2022).

[8] Vetter, T.W.; Waters, J.F.; Pintar, A.; *Certification of Standard Reference Material® 915c Calcium Carbonate*; NIST Special Publication 260210 (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-223.pdf (accessed Nov 2022).

If you use this SRM in published work, please reference:

Vetter TW, Waters JF, Pintar A (2022) Certification of Standard Reference Material® 915c Calcium Carbonate. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-223. https://doi.org/10.6028/NIST.SP.260-223

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.

* * * * * * * End of Certificate of Analysis * * * * * * *

APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the SI or other higher-order reference system [1]. Non-certified mass fraction values for significant impurities are provided below.

Table A1. Non-Certified Mass Fraction Values for Significant Detected Impurities (≥1 µg/g) in SRM 915c[a]

Impurity	Assumed Form[b]	Mass Fraction (µg/g)[c,d]
Barium (Ba)	$BaCO_3$	1 ± 0.2
Chromium (Cr)	$Cr_2(CO_3)_3$	1.3 ± 0.5
Phosphate (HPO_4)	$CaHPO_4$	15 ± 0.6
Magnesium (Mg)	$MgCO_3$	20 ± 0.8
Potassium (K)	K_2CO_3	10 ± 0.4
Sodium (Na)	Na_2CO_3	16 ± 0.7
Strontium (Sr)	$SrCO_3$	125 ± 5

[a] Values assigned based on values reported by manufacturer or semi-quantitative analysis. Additional impurities might be present between 10 µg/g and 50 µg/g, but could not be quantified because of detection limitations. See NIST Special Publication (SP) 260-223 [8] for details.

[b] Assumed form of compound in $CaCO_3$ that was used to make corrections for impurities in SRM 915c.

[c] Value and standard uncertainty based on the mass fraction of the impurity (not the assumed compound form).

[d] The uncertainty of the non-certified value is a combined standard uncertainty, u_c, consistent with the ISO/JCGM Guide [4].

* * * * * * * * * * * End of Appendix A * * * * * * * * * * *

APPENDIX B

Acquisition and preparation of this SRM were coordinated by T.W. Vetter. Analytical measurements were performed by T.W. Vetter and J.F. Waters of the NIST Chemical Sciences Division.

Consultation on the statistical design of the experimental work and evaluation of the data was provided by A.L. Pintar of the NIST Statistical Engineering Division.

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

* * * * * * * * * * * End of Appendix B * * * * * * * * * * *