National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 915a

Calcium Carbonate

(Clinical Standard)

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 for calcium determinations employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures. It may be used to prepare calcium standard solutions for either atomic absorption or titrimetric methods of analysis. The SRM consists of highly purified calcium carbonate in a 20-g unit.

Certified Purity: 99.965 ± 0.015^{a} Wt %

[a]See Table 2, Summary of Uncertainties; and Table 3, Components of Uncertainty

The certified value is calculated from the results of independent coulometric assays as described below. The uncertainty interval represents the expanded uncertainty, U, calculated according to the ISO Guide [1] as twice the combined standard uncertainty and gives the 95% level of confidence for the certified value.

Coulometric Assay: The assay value for this material was obtained by automated coulometric back-titration [2], to a strong acid endpoint (pH 7), of weighed $CaCO_3$ samples after addition of excess coulometrically standardized HCl and elimination of the product CO_2. The certified value represents the result of six such titrations of samples from three randomly selected bottles from the entire lot of SRM 915a.

Expiration of Certification: This certification will be 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.

Coulometric analyses were performed in the NIST Analytical Chemistry Division by K.W. Pratt.

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
January 13, 1995

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

The original trace element analyses on this lot of material (included in the Supplemental Information section) were performed in the NIST Analytical Chemistry Division (formerly the Inorganic Analytical Research Division) by T.C. Rains, T.A. Rush, V.C. Stewart, and D.W. Vomhof (all retired).

NOTICE AND WARNINGS TO USERS

This Standard Reference Material is intended for "in vitro" diagnostic use only.

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: This SRM was homogeneous within the uncertainty limits for the 280-mg sample size used for the coulometric assays. Samples less than 250 mg are not recommended in order to avoid possible inhomogeneities with smaller sample sizes.

INSTRUCTIONS FOR USE

Standard Stock Solutions of Calcium for Reference Method for Atomic Absorption Spectrophotometry [3]: Using class A volumetric flasks, prepare three stock solutions using the quantities given in Table 1. Add the specified initial volume of water before adding the specified volume of concentrated HCl. After all the SRM 915a is dissolved, add the specified amounts NaCl and KCl, dilute with water up to the neck, and invert until the salts are totally dissolved. Then dilute each solution to the calibrated volume and mix by inverting the flask 30 times. The pipettings and dilutions of these standard stock solutions follow the same instructions as given in reference [3].

Table 1. Specifications for Stock Solutions

Final Concentration (mmol/L)			Final Volume (L)	Mass SRM 915a (mg)	Initial Volume Water (mL)	Volume 37% (12 mol/L) HCl	Mass NaCl (g)	Mass KCl (mg)
Ca	Na	K						
2.00	140	5.0	2.000	400.5	18	2	16.36	746
2.50	140	5.0	1.000	250.3	9	1	8.18	373
3.00	140	5.0	1.000	300.4	9	1	8.18	373

Drying Instructions: To achieve the certified value, this material should be dried at 200-210 °C for 4 h.

Stock Solution of Calcium for Titrimetric Procedure: Place 0.2503 g of dried SRM 915a into a 1-L volumetric flask. Add approximately 9 mL of deionized water and 1 mL concentrated (12 mol/L; 37%) HCl. Make sure that all the calcium carbonate is in solution before diluting with water up to the neck. When at ambient temperature, dilute to the calibrated volume and mix by inverting the flask 30 times. Store in a borosilicate glass bottle. This solution contains 10.0 mg Ca^{2+} per 100 mL or 5.00 meq/L.

Stability of Prepared Solutions: Solutions prepared from SRM 915a, Calcium Carbonate, are stable indefinitely when stored in a glass-stoppered bottle, excepting concentration changes due to evaporation during sampling. All such solutions should be clear and display no turbidity.

Source of Material: The calcium carbonate used for this SRM was obtained from the J.T. Baker Chemical Co., Phillipsburg, NJ.

The material was examined for compliance with the American Chemical Society [4] specification for reagent grade calcium carbonate. The material was found to meet or exceed the minimum requirements in every respect. Examination by thermal gravimetric analysis indicated a minute loss of weight below 175 °C (volatile matter). The composition was stable above 175 °C to a temperature of 625 °C, above which material decomposition (evolution of CO_2) began to occur.

SUPPLEMENTAL INFORMATION

A semi-quantitative survey for trace contaminants by emission spectrometry indicated the presence of less than 0.001 wt % of copper, iron, magnesium, manganese, and silicon in the material. By atomic absorption, magnesium was evaluated at 1.0, sodium at 0.4, and strontium at 2.1 μg/g; potassium was less than 0.4, lithium less than 0.05, and barium much less than 10 μg/g. Neutron activation analysis indicated copper at 0.9, manganese at 0.6, and sodium at 0.5 μg/g. Copper was determined at 1 μg/g by spectrophotometry.

Table 2. Summary of Uncertainties for SRM 915a

SRM 915	$CaCO_3$, %
Combined Type A[a], (see Table 3)	0.0042
Combined Type B[b], (see Table 3)	0.0062
Combined Standard Uncertainty, u_c	0.0075
Coverage Factor	2.0
Expanded Uncertainty, U	0.015

Overall effective degrees of freedom = 91.6. The combined standard uncertainty represents the root sum of squares of the components of uncertainty. The coverage factor is based on the Student's t distribution for >30 degrees of freedom and a two-sided coverage probability of 95%.

[a] Type A uncertanties are evaluated by statistical methods [1].
[b] Type B uncertanties are evaluated by other means [1].

Table 3. Components of Uncertainty for $CaCO_3$ in SRM 915a

Type A

Source	c_i	units	u_i	units	c_iu_i	Degrees of Freedom
$CaCO_3$ Assay: standard error of the mean	1	--	0.0033	%	0.0033 %	5
HCl standardization: standard error of the mean	0.241	$\%*g_{HCl}/\mu mol_{HCl}$	0.0111	$\mu mol_{HCl}/g_{HCl}$	0.0027%	5
Combined Type A Uncertainty					0.0042%	

Table 3. Components of Uncertainty, Continued

Type B

Source	c_i	units	u_i	units	c_iu	Degrees of Freedom
Incomplete elimination of CO_2	1	--	0.006	%	0.006%	∞
Cathodic side-reactions	10^{-4}	%/ppm	0.5	ppm	5×10^{-5} %	∞
Pt electrode nonideal behavior	1	--	6×10^{-4} %	%	6×10^{-4}%	∞
HCl weighing	8.1×10^{-3}	%/mg	0.12	mg	9.7×10^{-4} %	∞
$CaCO_3$ weighing	3.3×10^{-4}	%/μg	2.9	μg	9.6×10^{-4}%	∞
Constant current supplies	10^{-4}	%/ppm	7.2	ppm	7.2×10^{-4}%	∞
Combined Type B Uncertainty					0.0062%	

REFERENCES

[1] "*Guide to the Expression of Uncertainty in Measurement*," ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993).

[2] Pratt, K.W., Anal. Chim. Acta, **289**, 135-142 (1994).

[3] Cali, J.P., Bowers, Jr., G.N., and Young, D.S., Clin. Chem. **19**, 1208-1213 (1973).

[4] *Reagent Chemicals*, 8th Ed. American Chemical Society, Washington, DC, (1993).