

U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 915

Calcium Carbonate

This Standard Reference Material 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.

The sample consists of highly purified calcium carbonate, and chemical assay as well as analysis for specific impurities indicate that the material may be considered to be essentially pure.

Purity	99.9+ percent
Water	0.01 ± 0.005 percent

Replicate samples taken from a randomly selected region of the undried material were assayed by a coulometric acidimetric procedure. The results from nine independent determinations, based on expression of the assay as calcium carbonate, indicate a purity of 99.99+ percent with a standard deviation of 0.003 percent. Samples equilibrated at a relative humidity of 90 percent and assayed by this coulometric procedure showed a maximum moisture adsorption of 0.02 percent as compared to samples that were dried for 6 hours at 210 °C. The moisture content, similarly measured, on samples equilibrated at 75 percent relative humidity, was found to be 0.01 percent. The water content was determined by the Karl Fischer method.

The calcium carbonate used for this Standard Reference Material was obtained from the J. T. Baker Chemical Company, of Phillipsburg, New Jersey. Analyses were performed by C. E. Champion, E. R. Deardorff, G. Marinenko, O. Menis, T. C. Rains, T. A. Rush, W. P. Schmidt, B. F. Scribner, V. C. Stewart, J. K. Taylor, and D. W. Vomhof.

The overall direction and coordination of technical measurements leading to the certification were under the chairmanship of R. Schaffer.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J. L. Hague.

Washington, D. C. 20234
March 4, 1969
Revised November 21, 1973

J. Paul Cali, Chief
Office of Standard Reference Materials

The material was examined for compliance with the specifications for reagent grade calcium carbonate as given in *Reagent Chemicals*, 4th edition, published by The American Chemical Society. The material was found to meet or exceed the minimum requirements in every respect. Examination by thermal gravimetric analysis indicated the loss of a minute proportion of weight below 175 °C (volatile matter) and the composition was stable above this temperature until a temperature of 625 °C, above which decomposition (evolution of CO₂) set in.

A semi-quantitative survey for trace contaminants by emission spectroscopy indicated the presence of less than 0.001 percent 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 2.1 parts per million (ppm); potassium was less than 0.4, lithium less than 0.05, and barium much less than 10 ppm. Neutron activation analysis indicated copper 0.9, manganese 0.6 and sodium 0.5 ppm. Copper was determined at 1 ppm by spectrophotometry.

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

This material is for use as a standard in clinical chemistry. It may be used to prepare calcium standard solutions for either atomic absorption or titrimetric methods of analysis.

Standard stock solutions of calcium for atomic absorption (Reference Method) [1]: Prepare a minimum of three concentrations at 2.00, 2.50, and 3.00 mmol of calcium per liter, with each to contain 140 mmol of sodium chloride and 5.0 mmol of potassium chloride per liter. To each of three 1-liter volumetric flasks, add 8.18 g NaCl and 373 mg of KCl. To the first flask (2.00 mmol of Ca per liter) add 200.2 mg of SRM 915 (dried at 200 °C for 4 hours), to the second flask (2.50 mmol of Ca per liter) add 250.2 mg of SRM 915, and to the third flask (3.00 mmol of Ca per liter) add 300.3 mg of SRM 915. To each flask add a few milliliters of water and 1 ml of concentrated HCl. Make sure that all the calcium carbonate is in solution before diluting with water up to the neck. When at ambient temperature, dilute each flask to the calibrated volume and mix by inverting the flask 30 times. Pipettings and dilutions of this material should follow the instructions given in reference [1].

Stock solution of calcium for titrimetric procedure: Place 0.250 g of dried SRM 915 into a 1000-ml volumetric flask. Add approximately 9 ml of deionized water and 1 ml concentrated HCl. Shake until dissolved. Fill to the mark with deionized water and store in a pyrex bottle. This solution contains 10.0 mg per 100 ml or 5.00 meq. per liter.

This Standard Reference Material should be stored in a well-stoppered bottle (preferably the original bottle) at room temperature. Calcium carbonate is a stable material. Under proper storage, experience at NBS indicates this material to be stable for at least 10 years. If the material purity degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that the material not be used after 5 years from the date of purchase.

Solutions prepared from SRM 915, Calcium Carbonate, are stable indefinitely when stored in an glass-stoppered bottle. All such solutions should be clear and display no turbidity.

References:

- [1] J. P. Cali, G. N. Bowers, Jr., and D. S. Young, Clin. Chem. 19, 1208-1213 (1973).
- [2] N. W. Tietz, Fundamentals of Clinical Chemistry, pp. 636-648, W. B. Saunders Co., Philadelphia, Pa., 19105 (1970).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
B311, Chemistry Building
National Bureau of Standards
Washington, D. C. 20234

The date of issuance and certification of this Standard Reference Material was March 4, 1969.