

# Certificate of Analysis

## Standard Reference Material 984

### Rubidium Chloride

RbCl, rubidium assay, weight percent . . . . . 99.90 ± 0.02  
Absolute abundance ratio,  $^{85}\text{Rb}/^{87}\text{Rb}$  . . . . . 2.593 ± 0.002

This lot of rubidium chloride was prepared to ensure material of intermediate purity and high homogeneity. The material is somewhat hygroscopic, absorbing approximately 0.6 percent moisture in a 75 percent relative humidity at room temperature, but can be dried to the original weight by desiccation over freshly exposed  $\text{P}_2\text{O}_5$  or  $\text{Mg}(\text{ClO}_4)_2$  for twenty-four hours. The material should therefore be stored with a desiccant such as  $\text{P}_2\text{O}_5$ .

The assay of this material is based on the determination of rubidium by a combination of gravimetry and isotope dilution analysis on eight samples of about 2 g each of the dried RbCl. More than 99% of the rubidium was precipitated, filtered and weighed as rubidium perchlorate. The weight of  $\text{RbClO}_4$  was corrected for potassium and cesium perchlorate. The soluble rubidium was determined by isotope dilution mass spectrometry. The total rubidium was the sum of the rubidium from the rubidium perchlorate and the rubidium from the filtrate. All weighings were corrected to vacuum and the atomic weights used in the calculations were from the 1969 Table of Atomic Weights. The indicated tolerance is at least as large as the 95 percent confidence level for a single determination.

Chloride was determined by silver coulometry to be 29.32 weight percent. Preferential oxidation of iodide and bromide showed that the material contains <0.001% I and <0.003% Br. Flame emission spectrometry indicated lithium, <0.02 ppm; sodium, 2.3 ppm, potassium, 420 ppm; and cesium 24 ppm. Emission spectrographic examination indicated, in addition, calcium, <10 ppm; magnesium, <10 ppm; silicon, <10 ppm; and aluminum, detection questionable. The loss on ignition at 500 °C (20 hours) was 0.010% and the insoluble matter was 0.0001%. A materials balance shows that 99.99 ± 0.02 weight percent of the material is accounted for.

The absolute abundance ratio of  $^{85}\text{Rb}/^{87}\text{Rb}$  was determined by triple-filament solid-sample mass spectrometry. Mixtures of known  $^{85}\text{Rb}/^{87}\text{Rb}$  ratio, prepared from nearly isotopically pure separated rubidium isotopes, were used to calibrate the mass spectrometers. The indicated uncertainties are overall limits of error based on 95 percent confidence limits for the mean and allowances for the effects of known sources of possible systematic error. Details of the preparation and measurements are described in a published paper [J Res NBS, 73A, 511-516 (1969)].

The following members of the Analytical Chemistry Division contributed to the characterization of this material: T. J. Murphy and P. J. Paulsen - rubidium assay; G. Marinenko - chloride assay; T. C. Rains and T. A. Rush - flame emission determinations; E. K. Hubbard - emission spectrographic analysis; and E. J. Catanzaro and E. L. Garner - absolute ratio determination.

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

The technical and support aspects involved in the preparation, certification, and issuance of this standard reference material were coordinated through the Office of Standard Reference Materials by J. L. Hague.