U. S. Department of Commerce
Peter G. Peterson
Secretary

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 999
Potassium Chloride
(Primary Chemical)

This Standard Reference Material is intended for use as an analytical standard of known potassium and chloride content. This lot of potassium chloride was prepared to ensure a material of high purity and homogeneity, and has been assayed for both potassium and chloride after ignition at 500 °C.

Potassium assay, weight percent	52.435 ± 0.004
Chloride assay, weight percent	47.551 ± 0.003

The indicated uncertainties are at least as large as twice the standard deviation for a single determination based on 18 determinations for potassium and 12 determinations for chloride. Potassium was determined by a combination of gravimetry and isotope dilution – mass spectrometry. Chloride was determined by coulometric argentimetry.

This potassium chloride as received contains about 0.1 percent occluded water. The certified values of the assay for use as a primary standard are based on material dried at 500 °C for 4 hours in a platinum or Vycor crucible (Pyrex is unsatisfactory). The certified values are based on the 1971 atomic weight values: potassium – 39.098, chlorine – 35.453, and oxygen – 15.999_4. The value for the faraday is 96486.70 $C \cdot mol^{-1}$. The value of the electrochemical equivalent for this material is 0.772724 ± 0.000019 $mg \cdot C^{-1}$.

The potassium chloride used for this Standard Reference Material was obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey. Analyses were performed by G. Marinenko, T. J. Murphy, T. C. Rains, T. A. Rush, W. P. Schmidt, and V. C. Stewart.

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

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 and T. W. Mears.

Washington, D.C. 20234
September 6, 1972

J. Paul Cali, Chief
Office of Standard Reference Materials

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Purity: This material has been found to conform to the specifications of the American Chemical Society for a reagent grade chemical. The only impurities detected at greater than 1 μg/g are rubidium (27 μg/g) and sodium (9 μg/g). Based on the potassium determination, the assay of this material is (after drying) $99.98_1 \pm 0.00_7$ weight percent; on the basis of the chloride determination the assay (after drying) is $99.99_1 \pm 0.00_6$ weight percent. Therefore, this SRM conforms to the definition of a primary-grade chemical according to the IUPAC classification for high-purity chemicals [Analyst, 90, 251-255 (1965)].

Homogeneity: The sample of potassium chloride was received in one unit. This unit was divided into 12 sub-units. Each sub-unit was assayed for potassium and for chloride. Each set of data indicated the sample to be homogeneous within the limits of uncertainty reported.

The methods used at NBS for the assay of potassium and chloride are described below. All weights were corrected to mass in vacuum.

Potassium: weigh approximately 2 g of KCl to the nearest 0.01 mg (previously ignited at 500 °C), dissolve it in approximately 70 ml of water, and then add 3 ml of $HClO_4$. Slowly evaporate the solution to crystallize $KClO_4$, and then fume to remove HCl and excess $HClO_4$. Dissolve the $KClO_4$ in 70 ml of hot water. Slowly evaporate this solution to 10 ml, cool to room temperature, add 0.5 ml of $HClO_4$, and then cool to about 5 °C. Filter the crystalline $KClO_4$ on a 15 ml, medium-porosity glass filter crucible. Wash the crystalline $KClO_4$ with 95 percent ethanol. Dry the crystalline $KClO_4$ at 100 °C, then heat to 300 °C to remove the occluded mother-liquor, and weigh. The potassium remaining in the filtrate was determined by isotope-dilution mass spectrometry. A correction is applied to reflect the $RbClO_4$ contribution to the weight of $KClO_4$.

The total potassium is the sum of the potassium calculated from the weight of crystalline $KClO_4$ and the potassium remaining in the filtrate of this crystallization. The certified value is reported as weight percent total potassium in the material.

Chloride: weigh approximately 2 g of KCl to the nearest 0.01 mg (previously ignited at 500 °C) in a Pt boat. Transfer the sample to a 125 ml Erlenmeyer flask and dissolve in 5 ml of distilled water. Titrate by means of electrogenerated silver ion according to a modification of the method of Marinenko and Taylor [J. Res. NBS 67A, 31 (1963)] using 20 wt. percent $HClO_4$ as the electrolyte. Following the pregeneration of about 99.95% of the Ag^+ ion at 500 mA, transfer the sample into the cell by a syphon arrangement, alloy to equilibrate for 1 hour in the dark, and complete titration using 0.6 mA current to an amperometric end point.