National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 999c

Potassium Chloride Primary Standard

This Standard Reference Material (SRM) is intended for use as an analytical standard of known potassium (K) and chloride (Cl^-) content. This lot of potassium chloride (KCl) was prepared to ensure a material of high purity and homogeneity and has been assayed after drying at 500 °C. A unit of SRM 999c consists of a single glass bottle containing 30 g of the material.

Certified Mass Fraction Values: Table 1 lists the certified values for this SRM, expressed as mass fractions, *w*, of KCl, K, and Cl^-. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. A certified value is the present best estimate of the true value. The expanded uncertainty, *U*, is calculated as $U = ku_c$, where u_c is the combined standard uncertainty calculated according to Supplement 1 to the ISO/JCGM Guide [2]. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of inherent sources of uncertainty of the assay techniques, material heterogeneity, and applicable corrections for interfering trace elements. The respective values of the coverage factor, *k*, for each measurand in Table 1 correspond to an approximately 95 % level of confidence for the effective degrees of freedom, ν_{eff}, stated for the given measurand.

Reference Mass Fraction Values: Table 2 lists a reference value for the mass fraction of bromine (Br) and alkalinity, expressed as potassium hydroxide (KOH). A reference value is a best estimate of the true value; however, the value does not meet NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision and may not include all other sources of uncertainty [1].

Information Mass Fraction Values: Table 3 lists information values for trace elements. Information values are non-certified values that may be of interest and use to the SRM user, but insufficient information is available to provide an uncertainty associated with the value [1]. The information values are given to provide additional characterization of the material only. These values should not be used to monitor or assess analytical performance. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 999c** is valid, within the measurement uncertainty specified, until **01 October 2035**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage and Use"). Accordingly, periodic recalibration or recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM lot over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification of SRM 999c was provided by K.W. Pratt of the NIST Chemical Sciences Division.

Analytical measurements leading to the certification of SRM 999c were made by K.W. Pratt, J.R. Sieber, T.W. Vetter, and J.F. Waters of the NIST Chemical Sciences Division.

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Statistical analyses were performed by W.F. Guthrie of the NIST Statistical Engineering Division.

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

Calculation of Certified Values: The certified value for w_{KCl} is obtained from a combination, using DerSimonian-Laird weighting [3], of the results of independent coulometric and gravimetric analyses, corrected for the aluminum oxide (Al_2O_3), calcium chloride ($CaCl_2$), potassium bromide (KBr), potassium fluoride (KF), potassium hydroxide (KOH), potassium sulfate (K_2SO_4), rubidium chloride (RbCl), silicon dioxide (SiO_2), and sodium chloride (NaCl) impurities. The certified value for w_K is obtained from a combination, using DerSimonian-Laird weighting, of w_K obtained directly from the gravimetric analyses, corrected for the Al_2O_3, $CaCl_2$, NaCl, RbCl, and SiO_2 impurities; and the indirect w_K, which is calculated from the coulometric w_{KCl} and the additional K from the KBr, KF, KOH, and K_2SO_4 impurities. The certified value for w_{Cl} is obtained from a combination, using DerSimonian-Laird weighting, of the direct results of the coulometric analyses, corrected for interfering Br; and the indirect w_{Cl}, which is calculated from the gravimetric w_{KCl} and the additional Cl from the $CaCl_2$, NaCl, and RbCl impurities. The corrections were obtained from the trace element determinations and the appropriate gravimetric factors [4]. A portion of the K is present in SRM 999c as KBr, KF, KOH, and K_2SO_4; and a portion of the chloride is present as $CaCl_2$, NaCl, and RbCl. Hence, the sum of the certified values for w_K and w_{Cl} does not equal the certified value for w_{KCl}. The certified values for the mass fractions of K, Cl, and KCl are metrologically traceable to the SI units for mass, current, and time in the coulometric assay; to the SI unit for mass in the gravimetric assay; and to the derived SI unit kilogram per kilogram for mass fraction for the corrections for trace elements.

Table 1. Certified Values for SRM 999c

Measurand	Mass Fraction (%)	u_c (%)	ν_{eff}	Coverage Factor (k)	Expanded Uncertainty (%)
w_{KCl}	99.987	0.010	115	1.981	0.021
w_K	52.443	0.0052	126	1.979	0.010
w_{Cl}	47.5519	0.0039	25	2.058	0.0081

Calculation of Reference Values: The measurand for bromine (Br) is mass fraction of Br as determined by wavelength-dispersive X-ray fluorescence analysis (WDXRF). Metrological traceability is to the derived SI unit for mass fraction expressed as micrograms per gram. The alkalinity was obtained from pH measurements in a carbon dioxide-free 1.0 mol/L solution of dried SRM 999c. The reference value for KOH alkalinity is metrologically traceable via the quantity pH to the primary measurement of pH and to the SI unit for mass. The expanded uncertainty is calculated using $k = 2$.

Table 2. Reference Values for SRM 999c

Element or Property	Mass Fraction (µg/g)	Expanded Uncertainty (µg/g)
Bromine (Br)	50	27
Alkalinity (as KOH)	1.16	0.35

Information Values: Table 3 lists information values for the mass fractions of trace elements in SRM 999c. No other elements were detected. The information values were obtained by glow discharge mass spectrometry performed by a commercial laboratory. Information values cannot be used to establish metrological traceability.

Table 3. Information Values for SRM 999c

Element	Mass Fraction (µg/g)
Aluminum (Al)	0.5
Calcium (Ca)	<0.5
Fluorine (F)	<1
Sodium (Na)	14
Rubidium (Rb)	8
Sulfur (S)	6
Silicon (Si)	0.2

INSTRUCTIONS FOR STORAGE AND USE

Storage: This SRM should be stored in its original bottle under normal laboratory conditions.

Use: Before it is sampled, the unit should be thoroughly mixed by carefully inverting and rotating the tightly sealed bottle. A minimum test portion mass of 200 mg should be used for analytical determinations. The bottle must be tightly re-capped after use and protected from moisture.

Drying Instructions: Dry the material for 4 h at 500 °C in platinum or fused silica (borosilicate glass is unsatisfactory) vessels. After the SRM has been dried, store it in a desiccator over anhydrous magnesium perchlorate and gently crush any lumps of KCl present before using.

SOURCE, HOMOGENEITY, AND ANALYSIS[(1)]

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

Homogeneity: This SRM is homogeneous within the uncertainty limits for the nominal sample mass, 200 mg, used for the coulometric chloride assays. Samples less than 200 mg are not recommended in order to avoid possible heterogeneity with smaller sample sizes.

Assay Techniques: The coulometric assay value was obtained by automated titration [6] with coulometrically generated Ag^+ using potentiometric detection of the endpoint. The gravimetric assay value was obtained by converting test portions of SRM 999c material to K_2SO_4 (procedure based on reference 7). Corrections for air buoyancy were made using a density of 1.984 g/cm^3 for SRM 999c. Corrections for interfering trace contaminants in each determination were applied as described above.

NOTICE TO USERS

NIST encourages the use of its SRMs to establish metrological traceability for the user's measurement results, and NIST strives to maintain the SRM inventory supply. However, NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of SRMs as primary benchmarks for the quality and accuracy of the user's in-house (working) standards. As such, SRMs should be used to validate or otherwise assign values to the more routinely used standards in a laboratory. When the metrologically traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of the calibration measurements for the in-house standard. Comparisons between NIST SRMs and such working measurement standards should take place at intervals appropriate to the conservation of the SRM primary standard and the stability of relevant in-house standards. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

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 values in Tables 2 and 3 may be useful in this evaluation.

[(1)] Certain commercial equipment, instruments, or materials are identified in this certificate 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.

REFERENCES

[1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at https://www.nist.gov/srm/upload/SP260-136.PDF (accessed Sep 2019).

[2] 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* , Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Sep 2019).

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[4] Wieser, M.E.; et al.; *Atomic Weights of the elements 2011 (IUPAC Technical Report)*; Pure Appl. Chem., Vol. 85, pp. 1047–1078 (2013).

[5] *Reagent Chemicals: American Chemical Society Specifications*; 9th ed.; American Chemical Society: Washington, DC (1999).

[6] Pratt, K.W.; *Automated, High-Precision Coulometric Titrimetry Part II. Strong and Weak Acids and Bases*; Anal. Chim. Acta, Vol. 289, 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 Sep 2019).

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Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.