



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 70b

Potassium Feldspar

This Standard Reference Material (SRM) is intended primarily for the evaluation of methods for analysis of constituent elements in feldspar or material of similar matrix. SRM 70b is powdered potassium feldspar that was sieved to less than 75 μm (<200 mesh) and blended to ensure homogeneity. A unit of SRM 70b consists of one bottle containing approximately 40 grams of powder.

Certified Mass Fraction Values: Certified values for elements of SRM 70b are reported in Table 1 as mass fractions on an as-received basis [1]. 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 [2]. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST.

Reference Mass Fraction Values: Reference values for elements are reported in Table 2 as mass fractions on an as-received basis. A reference value is a non-certified value that is the best estimate of the true value based on available data. These values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2].

Information Mass Fraction Value: An information value for chromium is reported in Table 3 as a mass fraction on an as-received basis. An information value is considered to be a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2].

Expiration of Certification: The certification of **SRM 70b** is valid, within the measurement uncertainty specified, until **01 September 2023**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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

The coordination of technical measurements for certification was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Analyses leading to the certification of this SRM were performed at NIST by A.F. Marlow, D.J. O'Kelly, S.A. Rabb, and J.R. Sieber of the NIST Chemical Sciences Division and P.E. Stutzman of the NIST Materials and Structural Systems Division.

Statistical consultation for this SRM was provided by A. Possolo of the NIST Statistical Engineering Division.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 31 December 2013

Robert L Watters, Jr., Director
Office of Reference Materials

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

To relate analytical determinations to the assigned values on this Certificate of Analysis, a minimum test portion of 100 mg is recommended on the basis of homogeneity testing and quantitative analyses performed at NIST. The powder does not require preparation prior to weighing because the loss on ignition at 950 °C is <0.5 %. The material should be stored in its original container, tightly capped, in a cool, dry location.

PREPARATION AND ANALYSIS⁽¹⁾

The material for SRM 70b was prepared from high-purity, ceramic grade feldspar obtained from pegmatite deposits in the Custer feldspar district in the Black Hills of South Dakota. The material for SRM 70b is a mixture of alkali feldspar, plagioclase feldspar, and quartz with a small amount of mica determined using X-ray diffraction. The material was blended and bottled at NIST. Methods used by NIST for quantitative analyses are provided in Table 4.

Certified Mass Fraction Values: The measurands are the mass fractions of the elements in feldspar. The certified values are metrologically traceable to the SI unit of mass. Each certified value is the DerSimonian-Laird [3,4] estimate of the mean of a random effects model fitted to the data from two methods. The uncertainty listed with each certified value is an expanded uncertainty calculated according to the ISO/JCGM Guide [5,6], and it expresses contributions from all recognized sources of uncertainty, including differences between analytical methods, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration function, assay of primary materials, and balance calibration. The effective coverage factor for each evaluated uncertainty is 2, except for calcium for which it is 2.5.

Table 1. Certified Mass Fraction Values for SRM 70b

Element	Mass Fraction (%)
Aluminum (Al)	7.98 ± 0.34
Calcium (Ca)	0.1770 ± 0.0051
Potassium (K)	6.33 ± 0.74
Sodium (Na)	2.36 ± 0.15

Element	Mass Fraction (mg/kg)
Barium (Ba)	28.2 ± 6.0
Manganese (Mn)	63.0 ± 4.7
Phosphorus (P)	790 ± 53

⁽¹⁾ Certain commercial instruments, materials, or processes are identified in this report 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 instruments, materials, or processes identified are necessarily the best available for the purpose.

Reference Values: The measurands are the mass fractions of elements in feldspar, as determined by the method indicated. The reference values are metrologically traceable to the SI unit of mass. Each reference value is either the mean or the median of the replicated measured values from a single method indicated in Table 4, in some cases after identification and removal of outliers. The uncertainty listed with each reference value is an expanded uncertainty calculated according to the ISO/JCGM Guide [5,6], and it expresses contributions from all recognized sources of uncertainty, including dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration function, assay of primary materials, and balance calibration. The effective coverage factor for each evaluated uncertainty is 2, except for vanadium and zinc for which it is 2.1.

Table 2. Reference Values for SRM 70b

Element	Mass Fraction (%)		
Iron (Fe)	0.13	±	0.02
Silicon (Si)	34.4	±	0.4

Element	Mass Fraction (mg/kg)		
Lead (Pb)	57	±	3
Magnesium (Mg)	298	±	20
Rubidium (Rb)	495	±	32
Strontium (Sr)	27	±	2
Titanium (Ti)	32	±	3
Vanadium (V)	0.93	±	0.01
Zinc (Zn)	7.7	±	0.1

Information Values: The information value reported for chromium is the mean value from one method performed at NIST.

Table 3. Information Value for SRM 70b

Element	Mass Fraction (mg/kg)
Chromium (Cr)	0.7

Table 4. Analytical Methods Performed at NIST

Method	Element
X-ray fluorescence spectrometry after borate fusion	Al, Ba, Ca, K, Fe, Mg, Mn, Na, P, Pb, Rb, Si, Sr, Ti
Inductively coupled plasma optical emission spectrometry	Ba, Cr, P, V, Zn
Instrumental neutron activation analysis	Al, Ca, K, Mn, Na

REFERENCES

- [1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at <http://www.nist.gov/pml/pubs/index.cfm/> (accessed Dec 2013).
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- [3] DerSimonian, R.; Laird, N.; *Meta-analysis in Clinical Trials*; Control Clin. Trials, Vol. 7, pp. 177–188 (1986).
- [4] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46(3), pp. 323–331, 2009.
- [5] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (GUM 1995 with Minor Corrections)*; Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Dec 2013); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <http://www.nist.gov/pml/pubs/index.cfm> (accessed Dec 2013).
- [6] 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*; JCGM (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Dec 2013).

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>.