



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 99b

Soda Feldspar

This Standard Reference Material (SRM) is intended for use in the determination of constituent elements in feldspar or material of similar matrix. SRM 99b is powdered soda feldspar that was sieved to less than 250 mesh ($<60\ \mu\text{m}$) and blended to ensure homogeneity. A unit of SRM 99b consists of one bottle containing approximately 40 grams of fine powder.

Certified Mass Fraction Values: Certified values for eight elements in SRM 99b are listed in Table 1 as mass fractions [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]. Certified values are based on the weighted means of analyses performed at NIST and collaborating laboratories [3]. The uncertainty listed with each value is an expanded uncertainty based on a 95 % confidence interval and is calculated according to the methods in the ISO Guide [4,5].

Reference Mass Fraction Values: Reference values for four constituents are reported in Table 2. Reference values are provided with uncertainties that may reflect only measurement precision and may not include all sources of uncertainty [2]. Reference values are noncertified values that are best estimates of the true values but do not meet the NIST criteria for certification. The uncertainty listed with each value is an expanded uncertainty based on a 95 % confidence interval and is calculated according to the methods in the ISO Guide [4,5].

Information Mass Fraction Values: The information values for seven constituents provided in Table 3 are noncertified values with no reported uncertainties. An information value is considered to be a value that will 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 99b** is valid, within the measurement uncertainty specified, until **31 December 2020**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for 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 the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements for certification of this SRM was under the direction of R.D. Vocke, Jr. of the NIST Analytical Chemistry Division.

Analytical measurements for certification of this SRM were performed by A.F. Marlow and J.R. Sieber of the NIST Analytical Chemistry Division and S.G. Wilson at the U.S. Geological Survey (USGS), Denver, CO.

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

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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INSTRUCTIONS FOR 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 performed at NIST using X-ray fluorescence spectrometry. The powder does not require preparation prior to weighing. The material should be stored in its original container, tightly capped, in a cool, dry location.

Source, Preparation, and Analysis⁽¹⁾: The material for SRM 99b was prepared from high-purity feldspar from a mine in the Spruce Pine pegmatite district of North Carolina. The material was blended and bottled at USGS. Methods used by NIST and USGS for characterizations are provided in Table 4.

Table 1. Certified Mass Fraction Values for SRM 99b Soda Feldspar

Constituent	Value ^(a)	Expanded Uncertainty ^(a)	Units	Coverage Factor, <i>k</i>
Al	10.36	0.16	%	2
K	3.09	0.18	%	2
Na	5.25	0.18	%	2
Ba	1409	50	mg/kg	2
Fe	278.7	8.0	mg/kg	2
Pb	71.2	4.4	mg/kg	2
Rb	72.6	3.0	mg/kg	2
Sr	444	10	mg/kg	2

^(a) The assigned value is a weighted mean of the results from two sets of measurements from two independent analytical methods using a Gaussian, linear mixed effects model [3]. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor *k*, calculated by Monte–Carlo simulation of uncertainty components using methods from the ISO Guide or its Supplement [4,5].

Table 2. Reference Mass Fraction Values for SRM 99b Soda Feldspar

Constituent	Value ^(a)	Expanded Uncertainty ^(a)	Units	Coverage Factor, <i>k</i>
Ca	1.18	0.13	%	2
Si	32.07	0.63	%	2.2
Mn	17.47	0.66	mg/kg	2
P	44	3	mg/kg	2.2

^(a) The assigned value is a weighted mean of the results from two sets of measurements from two independent analytical methods using a Gaussian, linear mixed effects model [3]. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor *k*, calculated by Monte–Carlo simulation of uncertainty components using methods from the ISO Guide or its Supplement [4,5].

⁽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 National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 3. Information Mass Fraction Values for SRM 99b Soda Feldspar

Constituent	Value (mg/kg)
Be	2.8
Ce	2.5
Ga	15
La	1.9
Li	7.9
Y	1.6
Zn	2.6

Table 4. Analytical Methods

Constituent	Method
Pb, Sr	WDXRF, ICP-OES, ICP-MS
Al, Ba, Ca, Fe, K, Na, Rb,	WDXRF, ICP-OES,
La, Mn, Zn	ICP-OES, ICP-MS
P, Si	WDXRF
Be, Li	ICP-OES
Ce, Ga, Y	ICP-MS

Methods Key:

WDXRF: wavelength-dispersive x-ray fluorescence performed at NIST

ICP-OES: inductively coupled plasma optical emission spectrometry performed at USGS

ICP-MS: inductively coupled plasma mass spectrometry performed at USGS

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://ts.nist.gov/WeightsAndMeasures/Metric/mpo_pubs.cfm (accessed Oct 2011).
- [2] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at <http://www.nist.gov/srm/publications.cfm> (accessed Oct 2011).
- [3] Searle, S.R.; Casella, G.; McCulloch, C.E.; *Variance Components*; John Wiley & Sons, Inc.: New York (1992).
- [4] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO 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 Oct 2011); 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/physlab/pubs/index.cfm> (accessed Oct 2011).
- [5] 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: http://www.bipm.org/utis/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Oct 2011).

Certificate Revision History: 07 October 2011 (Correction of unit content mass; editorial revisions); 06 May 2011 (Correction of Ca units to percentage; editorial revisions); 15 March 2011 (Original certificate).

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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.