

Standard Reference Material® 99b

Soda Feldspar

CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for use in the determination of constituent elements in feldspar or material of similar matrix.

Description: SRM 99b is powdered soda feldspar that was sieved to less than 250 mesh (<60 µm) and blended to ensure homogeneity. A unit of SRM 99b consists of one bottle containing approximately 40 grams of fine powder.

Certified Values: Certified values for eight elements in SRM 99b are listed in Table 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 [1]. The measurands in Table 1 are mass fractions for each analyte reported and metrological traceability is to the International System of Units (SI) derived unit for mass fraction expressed as a milligrams per kilogram or percentage [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/JCGM Guide [4,5].

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

Constituent	Value ^(a)	Expanded Uncertainty ^(a)	Units	Coverage Factor, <i>k</i>
Aluminum (Al)	10.36	0.16	%	2
Potassium (K)	3.09	0.18	%	2
Sodium (Na)	5.25	0.18	%	2
Barium (Ba)	1409	50	mg/kg	2
Iron (Fe)	278.7	8.0	mg/kg	2
Lead (Pb)	71.2	4.4	mg/kg	2
Rubidium (Rb)	72.6	3.0	mg/kg	2
Strontium (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/JCGM Guide or its Supplement [4,5].

Non-Certified Values: Non-certified values are provided in Appendix A.

Additional Information: Values of potential interest to users and additional information are provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 99b** are valid within the measurement uncertainty specified until **31 December 2032**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (<https://www.nist.gov/srm>) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

Safety: Please consult the Safety Data Sheet provided with this material.

Storage: The original unopened bottles of SRM 99b should be stored at room temperature ($20\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$). An open bottle can be reused until the material reaches its expiration date, provided that the open bottle is resealed and stored at room temperature ($20\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$).

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.

REFERENCES

- [1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf> (accessed Aug 2024).
- [2] 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 <https://www.nist.gov/pml/special-publication-811> (accessed Aug 2024).
- [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 (GUM 1995 with Minor Corrections)*; Joint Committee for Guides in Metrology (JCGM) (2008); available at <https://www.bipm.org/en/committees/jc/jcgm/publications> (accessed Aug 2024); 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 <https://www.nist.gov/pml/nist-technical-note-1297> (accessed Aug 2024).
- [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: <https://www.bipm.org/en/committees/jc/jcgm/publications> (accessed Aug 2024).

Certificate Revision History: 07 August 2024 (Change of period of validity; updated format; editorial changes); 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).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at <https://www.nist.gov/srm>.

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APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the International System of Units (SI) or other higher-order reference system. Non-certified values for four constituents are reported in Table A1. 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/JCGM Guide [4,5].

Table A1. Non-Certified Mass Fraction Values for SRM 99b Soda Feldspar

Constituent	Value ^(a)	Expanded Uncertainty ^(a)	Units	Coverage Factor, <i>k</i>
Calcium (Ca)	1.18	0.13	%	2
Silicon (Si)	32.07	0.63	%	2.2
Manganese (Mn)	17.47	0.66	mg/kg	2
Phosphorus (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/JCGM Guide or its Supplement [4,5].

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Certificate of Analysis and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

***** End of Appendix A *****

APPENDIX B

Values of Potential Interest to Users: Values of potential interest to users for seven constituents provided in Table B1.

Table B1. Mass Fraction Values of Potential Interest for SRM 99b Soda Feldspar

Constituent	Value (mg/kg)
Beryllium (Be)	2.8
Cerium (Ce)	2.5
Gallium (Ga)	15
Lanthanum (La)	1.9
Lithium (Li)	7.9
Yttrium (Y)	1.6
Zinc (Zn)	2.6

Source, Preparation, and Analysis: 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 B2.

Table B2. 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

***** End of Appendix B *****