

Standard Reference Material[®] 3389

Ginsenosides Calibration Solution

CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for use in producing metrologically traceable calibrants for determining ginsenosides Rb1, Rb2, Rc, Rd, Re, and Rg1 in natural matrix samples. The non-certified value delivered by the SRM is intended for use in harmonizing measurements of ginsenoside Rf in natural matrix samples.

Description: A unit of SRM 3389 consists of two solutions: (1) four ampoules, each containing approximately 1 mL of a six-component mixture of ginsenosides Rb1, Rb2, Rc, Rd, Re, and Rg1 in methanol and (2) one ampule containing approximately 1 mL of a single component ginsenoside Rf in methanol.

Certified Values: The following certified values are metrologically traceable to the International System of Units (SI) [1] through the gravimetric procedures used to prepare the solutions from primary standards provided by the National Research Council (NRC) of Canada.

Constituent	Mass Fraction ^(a) (mg/g)
Rg1	0.524 ± 0.012
Re	1.55 ± 0.03
Rb1	3.83 ± 0.12
Rc	0.754 ± 0.020
Rb2	0.889 ± 0.028
Rd	0.248 ± 0.008

^(a) Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty of the certified value. The true value of the analyte lies within the interval $x \pm U_{95\%}(x)$ with 95 % confidence. For guidance in propagating this uncertainty, see reference 2.

Non-Certified Value: A non-certified value is provided in Appendix A.

Period of Validity: The certified values are valid within the measurement uncertainty specified until **01 January 2030**. The certifications are nullified if the SRM is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM to the end of the period of validity. 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.

Safety: This SRM is sealed in glass ampoules with prescored stems. All appropriate safety precautions, including use of gloves during handling, should be taken.

Storage: Sealed ampoules of SRM 3389 should be stored in the dark at or below $-20\text{ }^{\circ}\text{C}$ until analysis.

Use: Before use, the ampoules should be allowed to warm to room temperature for at least 30 min. Samples for analysis should be withdrawn from ampoules and used without delay. Certified values are not valid for ampoules stored after opening, even if resealed.

Source and Preparation: The methanol solution of the ginsenoside Rb1, Rb2, Rc, Rd, Re, and Rg1 mixture was prepared gravimetrically by Cerilliant/PhytoLab (Round Rock, TX, USA) from individual neat compounds obtained from the National Research Council (NRC) of Canada (Ottawa, ON, Canada). The SRM solution was aliquoted (1 mL) into 2 mL amber glass ampoules and flame sealed.

Analysis: LC-UV and LC-MS measurements for value assignment of SRM 3389 were performed at NIST. Purities of the neat ginsenoside materials were assigned by the Institute for National Measurement Standards (INMS) at NRC Canada. These values were assigned by quantitative proton nuclear magnetic resonance spectroscopy ($q^1\text{H-NMR}$) traceable to NIST PS1 Primary Standard for quantitative NMR (Benzoic Acid).

Additional Information: The development of SRM 3389 was a collaboration between the National Institute of Standards and Technology (NIST), and the National Institutes of Health Office of Dietary Supplements (NIH-ODS). Full details on the production and evaluation of SRM 3389 are provided free of charge in reference 3.

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.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sharpless, K.E.; Sieber, J.R.; 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; U.S. Government Printing Office: Washington, DC (2020); available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2020.pdf> (accessed Dec 2020).
- [2] Possolo AM.; *Evaluating, Expressing, and Propagating Measurement Uncertainty for NIST Reference Materials*; NIST Special Publication (NIST SP) 260-202 (2020) available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-202.pdf> (accessed Dec 2020).
- [3] Wilson, W.B; Sander, L.C; Place, B.J; Yen J.; *Certification of Standard Reference Material® 3389 Ginsenoside Calibration Solution*; NIST Special Publication (NIST SP) 260-196 (2020); available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-196.pdf> (accessed Dec 2020).

If you use this SRM in published work, please reference:

Wilson WB, Sander LC, Place BJ, Yen J (2020) Certification of Standard Reference Material® 3389 Ginsenoside Calibration Solution. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-196. <https://doi.org/10.6028/NIST.SP.260-196>

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, Maryland 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at <https://www.nist.gov/srm>.

***** End of Certificate of Analysis*****

APPENDIX A

Non-Certified Value: 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.

A non-certified value for ginsenoside Rf is given below. This non-certified value is metrologically traceable to the materials and procedures used in its determination.

Constituent	Mass Fraction (mg/g) ^(a)
Rf	1.3 ± 0.1

- ^(a) The value is expressed as $x \pm 2u(x)$, where x is a mean value and $u(x)$ is its associated standard uncertainty. The standard uncertainty combines experimentally determined repeatability imprecision and between-vial heterogeneity. While the best estimate of the analyte mass fraction lies within the interval $x \pm 2u(x)$, this interval may not include the true value. For guidance in propagating this uncertainty, see reference 2.

Period of Validity: The non-certified value is valid within the measurement uncertainty specified until **01 January 2030**. The value-assignment is nullified if this material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Value: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified value during this period, NIST will update this Appendix. Before using this material, users should obtain the most recent version of this documentation, available free of charge through the <https://www.nist.gov/srm> website.

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