National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 912b

Urea

This Standard Reference Material (SRM) is certified as a neat chemical of known purity. It is a primary standard intended for use during calibration procedures and method validations implemented by clinical laboratories. A unit of SRM 912b consists of 25 g of high-purity crystalline urea.

Certified Urea Mass Fraction: 99.95 % ± 0.01%

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 certified value [2] was determined using a mass balance approach for purity assessment. The uncertainty was determined as the 95 % coverage interval (U_{95}) [3,4] of the probability distribution function evaluated using a Bayesian statistical approach implemented via a Markov Chain Monte Carlo method. The measurand is the total concentration of urea. Metrological traceability is to the SI derived unit for mass fraction (expressed as percent). Biuret is the primary impurity component [5], followed by water with approximate mass fractions of 0.025 % and 0.015 %, respectively.

Expiration of Certification: The certification of **SRM 912b** is valid, within the measurement uncertainty specified, until **30 September 2027**, provided the SRM is handled and stored in accordance with the 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, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of the technical activities were under the chairmanship of M.A. Nelson of the NIST Chemical Sciences Division.

Analytical measurements at NIST were performed by B.E. Lang, M.A. Nelson, and J.S. Pritchett of the NIST Chemical Sciences Division.

Statistical analysis was provided by B. Toman of the NIST Statistical Engineering Division.

Support aspects involved in 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: 30 May 2017

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR STORAGE, AND USE

Storage: The SRM should be stored in the original bottle at temperatures between 20 °C and 30 °C. It must be tightly re-capped after use and protected from excessive moisture, heat, and light. Refrigeration is recommended (4 °C), however the material should be brought to room temperature (between 20 °C and 30 °C) before opening the container. Urea at these temperatures is significantly hygroscopic when exposed to high relative humidity conditions (>75 %) [6] and this material should be contained under controlled low humidity.

Use: SRM 912b stored as described above, can be used without preliminary drying. The minimum sample size required is 10 mg.

SOURCE AND ANALYSIS[(1)]

Source of Material: The SRM material was obtained from Spectrum Chemical Manufacturing Corp. (New Brunswick, NJ).

Analytical Approach and Homogeneity Assessment: Analyses for chemical purity and homogeneity were performed by NIST. Evaluations were performed using twenty two units, randomly selected from across the filling run, via differential scanning calorimetry (DSC), ^{1}H-nuclear magnetic resonance (NMR), and a mass balance approach for purity evaluation that included Karl Fischer titration, Liquid Chromatography with Ultraviolet Detection (LC-UV), ion chromatography with electrochemical detection, and thermogravimetric analysis (TGA). No trend in mass fraction purity was observed with respect to filling order and there is no discernible inhomogeneity at the 95 % confidence level. Biuret and water content mass fractions were respectively determined using a targeted LC-UV calibration method and Karl Fischer titration.

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; U.S. Government Printing Office: Washington, DC (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed May 2017).

[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 http://ws680.nist.gov/publication/get_pdf.cfm?pub_id=200349 (accessed May 2017).

[3] 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/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2017); 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/tn1297/index.cfm (accessed May 2017).

[4] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the Guide to Expression of Uncertainty in Measurement, Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed May 2017).

[5] Redemann, C.E; Riesenfeld F.C; La Viola F.S.; *Formation of Biuret from Urea*; Ind. Eng. Chem. Res., Vol. 50, pp 633–636 (1958).

[6] Werner, E.A.; *Urea as a Hygroscopic Substance*; Nature, Vol. 139, pp. 512–512 (1937).

*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 s*rminfo@nist.gov*; or via the Internet at http://www.nist.gov/srm.*

[(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.