National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 141e

Acetanilide

This Standard Reference Material (SRM) is intended for use in validating microchemical procedures for the determination of carbon, hydrogen, and nitrogen in organic matter. A unit of SRM 141e contains 2 g of crystalline material.

Certified Chemical Identity: The chemical identity of acetanilide ($CH_3CONHC_6H_5$) is certified with highest confidence. This was sufficiently evinced, with no ambiguity, by nuclear magnetic resonance (NMR) spectroscopy and gas chromatography-mass spectrometry (GC-MS).

NIST recommends the elemental composition of pure acetanilide be used for the validation of microchemical procedures with this material. Pure acetanilide has the following theoretical elemental composition, derived from the molecular formula using corresponding IUPAC atomic weights [4], expressed as fractions (in percent) of total relative molecular mass. Traceability of the composition of this SRM is realized through the unambiguous determination of chemical structure and the derived mass fraction of acetanilide. The theoretical elemental composition of this material was confirmed by the elemental analysis described in the supplemental information section of this certificate.

Element	Fraction (%)
Carbon (C)	71.088
Hydrogen (H)	6.712
Nitrogen (N)	10.363
Oxygen (O)	11.837

Certified Purity Value: The certified acetanilide mass purity was evaluated at NIST using quantitative nuclear magnetic resonance (qNMR) spectroscopy with an internal standard, differential scanning calorimetry (DSC), gas chromatography with flame ionization detection (GC-FID), and GC-MS. The purity value determined from these methods was estimated using a Bayesian consensus model implemented via Markov Chain Monte Carlo analysis. The purity as mass fraction (%) [1] of acetanilide, with associated standard uncertainty (u) and 95% confidence interval (U_{95}) [2,3], is:

Purity as Mass Fraction: 99.88 %, u = 0.10 %, U_{95} = [99.60 %, 100 %]

The U_{95} is determined as the 95 % coverage interval of the probability distribution function evaluated using the Bayesian consensus model. Metrological traceability is to the SI derived unit for mass fraction (expressed as percent).

Expiration of Certification: The certification of **SRM 141e** is valid, within the measurement uncertainty specified, until **30 September 2026**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 08 May 2017

Steven J. Choquette, Director
Office of Reference Materials

Analytical measurements at NIST were performed by 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.

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 or register online) will facilitate notification.

INSTRUCTIONS FOR STORAGE AND USE

Storage: The SRM should be stored in its original bottle at temperatures between approximately 20 °C and 25 °C. It must be tightly re-capped after use and protected from excessive moisture and light.

Use: SRM 141e is not hygroscopic under ordinary conditions of storage as described above, and can be used without preliminary drying.

SOURCE AND ANALYSIS[(1)]

Source of Material: The SRM material was obtained from Sigma Aldrich Corporation (Milwaukee, WI).

Analytical Approach: Analyses for chemical purity were performed by NIST. Ten samples, uniformly selected from across the filling run, were analyzed by qNMR with an internal standard and DSC. No trend in mass fraction purity was observed with respect to filling order.

SUPPLEMENTAL INFORMATION

Confirmatory Laboratory Analyses: To confirm the theoretical chemical composition for carbon, hydrogen, and nitrogen, elemental microanalyses were performed by three independent testing laboratories: Atlantic Microlab, Inc. (Norcross, GA), Galbraith Laboratories, Inc. (Knoxville, TN) and Micro-Analysis, Inc. (Wilmington, DE). Ten bottles were randomly and uniformly sampled from across the entire production lot. Each laboratory tested material from three to five of these ten samples. Duplicate carbon, hydrogen, and nitrogen analyses were performed for each. The results from the three laboratories fell within the ranges presented in Table 1.

Table 1. Ranges of the chemical composition (fractions) of carbon, hydrogen, and nitrogen in SRM 141e Acetanilide determined via elemental microanalysis

Element	Range Fraction (%)
Carbon	70.99 – 71.28
Hydrogen	6.63 – 6.91
Nitrogen	10.25 – 10.42
Oxygen[(a)]	11.44 – 11.89

[(a)] Oxygen content is calculated by assuming that all that is not carbon, hydrogen, or nitrogen must be oxygen [% O = 100 - (% C + % H + % N)].

[(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 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.

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/sp811/indexfull.cfm (accessed May 2017).

[2] 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/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).

[3] 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/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed May 2017).

[4] IUPAC Commission on Isotopic Abundances and Atomic Weights, *Atomic Weights of the Elements 2013 (IUPAC Technical Report)*, Pure & Appl. Chem., Vol. 88 Issue 3, pp. 265—291 (2016).

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.