



# Certificate of Analysis

## Standard Reference Material<sup>®</sup> 141d



This Standard Reference Material (SRM) is intended primarily for use in validating microchemical procedures for the determination of carbon, hydrogen, and nitrogen in organic matter. SRM 141d is supplied in crystalline form in a 2 g unit.

**Certified Value and Uncertainty:** The purity of this material was assessed at NIST by differential scanning calorimetry (DSC), gas chromatography (GC), and direct probe mass spectrometry (DPMS). Based upon these measurements, the certified purity and expanded uncertainty of the acetanilide are given below [1].

Certified Purity as Mass Fraction: 99.92 %  $\pm$  0.01 %

The uncertainty in the certified value is expressed as an expanded uncertainty,  $U$ , at the 95 % level of confidence, and is calculated according to the method described in the ISO Guide [2]. The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  is intended to represent, at the level of one standard deviation, the measurement error. The coverage factor,  $k$ , is determined from the Student's  $t$ -distribution corresponding to the appropriate associated degrees of freedom and 95 % level of confidence.

**Theoretical Chemical Composition:** Pure acetanilide has the following theoretical chemical composition expressed as fractions (in %) of its total relative molecular mass [3].

Element	Fractions (%)
Carbon	71.089
Hydrogen	6.711
Nitrogen	10.363
Oxygen	11.837

*NIST recommends that the theoretical chemical composition be used for this material. The analytical techniques used to generate the supplemental information validated the theoretical composition of this material within their experimental error.*

**Expiration of Certification:** The certification of **SRM 141d** is valid, within the measurement uncertainties specified, until **30 September 2017**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Notice and Warning to Users"). 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.

Analytical measurements at NIST were performed by R.M. Parris, M.P. Urquiza, L.K. Walton, and M.J. Welch of the NIST Analytical Chemistry Division.

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Robert L. Watters, Jr., Chief  
Measurement Services Division

The overall direction and coordination of the technical activities were under the chairmanship of M.J. Welch of the NIST Analytical Chemistry Division.

Statistical analysis was provided by L.M. Gill of the NIST Statistical Engineering Division.

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

## NOTICE AND WARNING TO USERS

**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 141d is not hygroscopic under ordinary conditions of storage as described above, and can be used without preliminary drying.

## SOURCE AND ANALYSIS<sup>(1)</sup>

**Source of Material:** The SRM material was obtained from Fluka Chemical Corporation, Ronkonkoma, NY.

**Analytical Approach:** Analyses for purity were performed by NIST. Ten samples, selected by a stratified random process across the filling run, were analyzed by DSC. All exhibited high purity (>99.9 %) and no trend with respect to filling order was found. A small unidentified impurity was found by DPMS, but its level could not be estimated. Two samples were analyzed by with flame ionization detection (GC-FID) and found to have small impurity levels consistent with the DSC data. No measurable impurities were found by GC/MS.

## SUPPLEMENTAL INFORMATION

To confirm the theoretical chemical composition for carbon, hydrogen, and nitrogen, three collaborating laboratories performed the analyses. Fifty bottles representative of the entire bottled lot were selected by a stratified random sampling. Each laboratory tested three taken from the group of fifty, chosen such that one bottle came from the first third of the bottling run, one from the second third, and one from the final third. Analyses were performed in duplicate from each of the three bottles for each of the three elements: carbon, hydrogen, and nitrogen. The three laboratories were: Atlantic Microlab, Inc., Norcross, GA; Galbraith Laboratories, Inc., Knoxville, TN; and Schwartzkopf Microanalytical Laboratory, Inc., Woodside, NY. The data from the three laboratories fell within the ranges presented in Table 1.

Table 1. Range of Values (Fractions) for Measured<sup>(a)</sup> Relative Atomic Masses for Carbon, Hydrogen, and Nitrogen in SRM 141d

Element	Range Fraction (in %)
Carbon	71.01 – 71.25
Hydrogen	6.65 – 6.80
Nitrogen	10.22 – 10.49
Oxygen <sup>(b)</sup>	11.59 – 11.93

<sup>(a)</sup> These results are provided to demonstrate user experience with this material.

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

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<sup>(1)</sup> 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 2012).
- [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/utls/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed May 2012); 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 2012).
- [3] IUPAC Commission on Atomic Weights and Isotopic Abundances; *Atomic Weights of the Elements 1995*; Pure & Appl. Chem., Vol. 68, p. 2339 (1996).

<b>Certificate Revision History:</b> 08 May 2012 (Extension of certification period; editorial changes); 10 September 2007 (Update of expiration date and editorial changes); 10 April 1998 (Original certificate date).
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*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](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*