



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 3060

#### Monoester Phthalates in Acetonitrile

This Standard Reference Material (SRM) is intended primarily for use in the calibration of chromatographic instrumentation. A unit of SRM 3060 consists of five 2 mL ampoules, each containing approximately 1.2 mL of an acetonitrile solution of monoester phthalates.

**Certified Mass Fraction Values:** The certified mass fraction values for monoester phthalates listed in Table 1 are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using liquid chromatography (LC) [1,2]. 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.

**Reference Mass Fraction Values:** The reference mass fractions values for monoester phthalates listed in Table 2 are based on results obtained from the gravimetric preparation of the solution and from the analytical results determined by using liquid chromatography. Reference values are values that are the best estimates of the true values; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1].

**Information Mass Fraction Value:** An information mass fraction value for monoester phthalates is listed in Table 3. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [1]. Information values cannot be used to establish metrological traceability.

**Expiration of Certification:** The certification of **SRM 3060** is valid, within the measurement uncertainty specified, until **30 April 2026**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for 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 before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification on this SRM was under the direction of J.R. Kucklick and J.L. Reiner of the NIST Chemical Sciences Division.

Statistical consultation for this SRM was provided by J.H. Yen of the NIST Statistical Engineering Division.

Analytical measurements were performed by B.A. Benner Jr., A.S.P. Boggs, J.R. Kucklick, and J.L. Reiner of the NIST Chemical Sciences 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

## INSTRUCTIONS FOR USE

**Handling:** This material contains monoester phthalates, many of which have been reported to have toxic properties, and should be handled with care. Use proper disposal methods. Please refer to the Safety Data Sheet for additional health and safety information.

**Storage:** Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

**Opening of Ampoule:** Open ampoules carefully to prevent contamination and injury. The ampoules are pre-scored and should **NOT** be opened using a file. Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the values in Tables 1 and 2 to be valid within the stated uncertainties. Certified values are not applicable to material stored in ampoules that have been opened for more than 5 min, even if they are resealed.

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

**SRM Preparation:** The monoester phthalates used in the preparation of this SRM were obtained from CanSyn Chemical Corporation (Toronto, Ontario, Canada). The solution was prepared at NIST by weighing and then dissolving the individual monoester phthalates into acetonitrile. The total mass of this solution was measured, and the mass fractions were determined for the components. This bulk solution was then chilled to approximately –5 °C, and 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules that were then flame sealed.

**Liquid Chromatographic Analysis:** Aliquots from ampoules selected by a stratified random sampling were analyzed by using liquid chromatography tandem mass spectrometry (LC-MS/MS) on a Betasil Phenyl column (150 mm x 2.1 mm x 3.0 µm, Thermo Scientific, Waltham, MA). An internal standard solution containing <sup>13</sup>C-labeled monoester phthalates was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of the monoester phthalates and internal standard compounds in acetonitrile were chromatographically analyzed to determine analyte response factors.

**Certified Mass Fraction Values:** Each certified value, is a mean of average mass fractions, with one average from gravimetric preparation and one average from chromatographic measurements. The measurand is the mass fraction for each monoester phthalate listed in Table 1. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence. The expanded uncertainty is calculated as  $U = ku_c$ , where the combined uncertainty  $u_c$  incorporates the observed difference between the results from the methods and their respective uncertainties, consistently with the ISO/JCGM Guide and its Supplement 1, and  $k = 2$  is a coverage factor corresponding to approximately 95 % confidence [3–5]. Metrological traceability is to the derived SI unit for mass fraction (expressed as milligrams per kilogram).

Table 1. Certified Mass Fractions Values for SRM 3060 Monoester Phthalates in Acetonitrile

Monoester Phthalate	CAS Number	Mass Fraction (mg/kg)
Monobutyl phthalate	131-70-4	32.7 ± 0.4
Monobenzyl phthalate	2528-16-7	15.3 ± 1.3
Mono(7-carboxyheptyl) phthalate	856869-57-3	3.29 ± 0.12
Mono(2-ethyl-5-carboxypentyl) phthalate	40809-41-4	21.7 ± 0.9
Mono(2-ethylhexyl) phthalate	4376-20-9	3.03 ± 0.04
Mono(2-ethyl-5-oxohexyl) phthalate	40321-98-0	13.3 ± 0.9
Monoethyl phthalate	2306-33-4	131 ± 3
Monoisobutyl phthalate	30833-53-5	17.7 ± 0.2
Monomethyl phthalate	4376-18-5	3.30 ± 0.10

<sup>(1)</sup> Certain commercial equipment, instruments, or materials 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 materials or equipment identified are necessarily the best available for the purpose.

**Reference Mass Fraction Values:** Each reference value is a mean of average mass fractions, with one average from gravimetric preparation and one average from chromatographic measurements. The measurand is the mass fraction for each monoester phthalate listed in Table 2 as determined by the methods indicated. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence. The expanded uncertainty is calculated as  $U = ku_c$ , where the combined uncertainty  $u_c$  incorporates the observed difference between the results from the methods and their respective uncertainties, consistently with the ISO/JCGM Guide and its Supplement 1, and  $k = 2$  is a coverage factor corresponding to approximately 95 % confidence [3–5]. Metrological traceability is to the derived SI unit for mass fraction (expressed as milligrams per kilogram).

Table 2. Reference Mass Fractions Values for SRM 3060 Monoester Phthalates in Acetonitrile

Monoester Phthalate	CAS Number	Mass Fraction (mg/kg)
Mono(2-ethyl-5-hydroxyhexyl) phthalate	40321-99-1	17.2 ± 3.0
Mono(3,5,5-trimethylhexyl) phthalate	297182-83-3	3.39 ± 0.38
Monooctyl phthalate	5393-19-1	3.59 ± 0.66

**Information Mass Fraction Value:** The information value was determined by the gravimetric preparation. No uncertainty is provided because there is insufficient information available for its assessment.

Table 3. Information Mass Fraction for SRM 3060 Monoester Phthalates in Acetonitrile

Monoester Phthalate	CAS Number	Mass Fraction (mg/kg)
Mono(3-carboxypropyl) phthalate	66851-46-5	6.23

## REFERENCES

- [1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions 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 July 2016).
- [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 [www.nist.gov/pml/pubs/index.cfm/](http://www.nist.gov/pml/pubs/index.cfm/) (accessed July 2016).
- [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/utis/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf) (accessed July 2016); 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/index.cfm> (accessed July 2016).
- [4] 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*; JCGM (2008); available at [http://www.bipm.org/utis/common/documents/jcgm/JCGM\\_101\\_2008\\_E.pdf](http://www.bipm.org/utis/common/documents/jcgm/JCGM_101_2008_E.pdf) (accessed July 2016).
- [5] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall (1993).

*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>.*