



Certificate of Analysis

Standard Reference Material[®] 3074

Phthalates in Methanol

This Standard Reference Material (SRM) is a solution of one adipate and six phthalates in methanol intended primarily for use in the calibration of chromatographic instrumentation used for the determination of the certified components in the mixture. Because of its miscibility with water, this SRM can also be used to fortify samples with known amounts of the six phthalates. A unit of SRM 3074 consists of five 2 mL ampoules, each containing 1.2 mL of solution.

Certified Values: The certified mass fraction and mass concentration values for the six phthalates are given in Table 1 [1,2]. These values are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography with mass spectrometric detection (GC/MS). 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 accounted for by NIST.

Supplemental Information: Chemical Abstracts Service (CAS) Registry Numbers of the certified components are listed in Table 1. In addition to the six phthalates listed in Table 1, this solution contains *bis*(2-ethylhexyl)adipate (CAS Registry Number 103-23-1). The concentration of *bis*(2-ethylhexyl) adipate in the solution has decreased since the preparation of this solution, so it is not included as a certified value.

Expiration of Certification: The certification of this **SRM 3074** is valid, within the measurement uncertainties specified, until **30 March 2023** 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 before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements leading to the certification was under the direction of M.M. Schantz and S.A. Wise of the NIST Chemical Sciences Division.

Partial support for the preparation and certification of this Standard Reference Material was provided by the U.S. Environmental Protection Agency Office of Water, Office of Enforcement and Compliance Assurance, and Office of Research and Development.

Analytical measurements of the SRM were performed by B.A. Benner, Jr., C.R. Mack, and L.K. Walton of the NIST Chemical Sciences Division.

Preparation of the SRM was performed by B.A. Benner, Jr. and M.P. Cronise of the NIST Office of Reference Materials.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh 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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

CAUTION: This SRM is sealed in glass ampoules with prescored stems. All appropriate safety precautions, including use of gloves during handling, should be taken. Unopened ampoules should be stored upright inside the original container supplied by NIST under normal laboratory conditions.

Handling: This material contains phthalates and an adipate and should be handled with care. Use proper disposal methods.

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

Use: 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 certified values to be valid within the stated uncertainty. Because of the volatility of methanol, certified values are not applicable to material stored in ampoules that have been opened for more than 5 minutes, even if they are resealed.

PREPARATION AND ANALYSIS

Preparation: The six phthalates and one adipate used in the preparation of this SRM were obtained from a commercial source. The weighed components were added to the methanol and mixed until completely dissolved and homogenized. The total mass of this solution was measured and the mass fractions and mass concentrations were calculated from this gravimetric procedure. The gravimetric values were adjusted for the consensus purity estimation of each component, which was determined using capillary gas chromatography with flame ionization detection and differential scanning calorimetry. This bulk solution was then chilled to approximately -5 °C and 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules, which were flame sealed.

Analysis: Aliquots from nine ampoules, selected according to a stratified, random sampling scheme, were analyzed in duplicate using GC/MS employing an immobilized nonpolar (5 % phenylmethylpolysiloxane) stationary phase column (see Figure 1). An internal standard solution containing six ring- d_4 phthalates was added to each sample for quantification purposes. Calibration solutions consisting of weighed amounts of the six phthalates and one adipate and the ring- d_4 phthalate internal standard compounds in methanol were chromatographically analyzed to determine analyte response factors.

Certified Mass Fraction Values: The certified mass fraction values are expressed as the certified value \pm an expanded uncertainty. The certified value is the unweighted average of the mass fractions determined by gravimetry and chromatographic measurements. The expanded uncertainty, at the 95 % level of confidence, is calculated as $U = ku_c$ where u_c is a combined standard uncertainty calculated according to the ISO Guide [3] and $k = 2$ is the coverage factor. The value of u_c includes both a correction for estimated purity and an allowance for differences between the mass fractions determined by gravimetric preparation and chromatographic measurements.

Certified Mass Concentration Values: The certified mass concentration values (in mg/L) were obtained by multiplying the certified mass fraction by the measured density of the SRM solution at 22 °C (0.7914 g/mL). This concentration is for use over the temperature range of 20 °C to 25 °C, and an allowance for the change in density over this temperature range is included in the uncertainty.

Table 1. Certified Values of the Six Phthalates in SRM 3074

Compound	CAS Registry Number ^(a)	Mass Fraction (mg/kg)	Mass Concentration (mg/L)
Dimethylphthalate	131-11-3	55.6 \pm 1.2	44.0 \pm 0.9
Diethylphthalate	84-66-2	51.4 \pm 1.7	40.7 \pm 1.4
Di- <i>n</i> -butylphthalate	84-74-2	51.2 \pm 1.2	40.5 \pm 0.9
Benzylbutylphthalate	85-68-7	52.2 \pm 1.4	41.3 \pm 1.1
<i>Bis</i> (2-ethylhexyl)phthalate	117-81-7	58.6 \pm 1.3	46.4 \pm 1.0
Di- <i>n</i> -octylphthalate	117-84-0	48.2 \pm 1.4	38.2 \pm 1.1

^(a) Chemical Abstracts, Thirteenth Collective Index, Index Guide, American Chemical Society, Columbus, OH (1996).

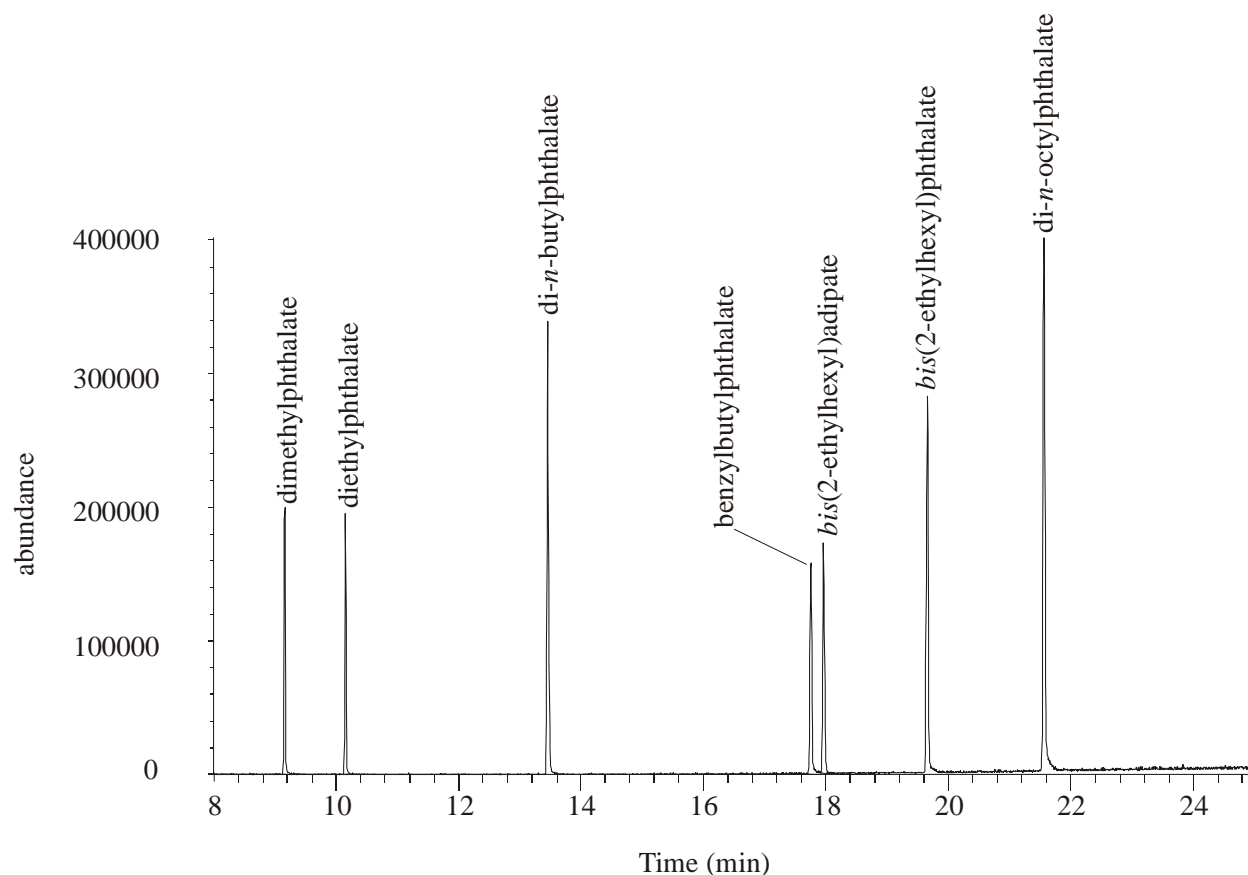


Figure 1. Chromatogram from GC/MS analysis of SRM 3074 using a 0.25 mm i.d. \times 60 m (length) fused-silica capillary column with a 5% phenylmethylpolysiloxane phase (0.25 μ m film thickness). Temperature Program: 100 $^{\circ}$ C (1 min) then 30 $^{\circ}$ C/min to 200 $^{\circ}$ C, then 7.5 $^{\circ}$ C/min to 320 $^{\circ}$ C; MS scan from 50 m/z to 300 m/z (2.4 scans/s), electron multiplier voltage = 2000 V.

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/publications.cfm> (accessed Mar 2013).
- [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://www.nist.gov/pml/pubs/index.cfm/> (accessed Mar 2013).
- [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 (2008); available at http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Feb 2013); 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 Mar 2013).

Certificate Revision History: 05 March 2013 (Extension of certification period; editorial changes); 11 May 2006 (Editorial change); 19 April 2006 (Removal of the adipate component from the list of certified constituents and extension of certification period); 20 February 2003 (Original certificate date).

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>.