U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
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National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1586

Isotopically Labeled and Unlabeled

Priority Pollutants in Methanol

This Standard Reference Material (SRM) is intended primarily for use in the evaluation and calibration of analytical instrumentation used for the determination of priority pollutants as classified by the U.S. Environmental Protection Agency (EPA). In particular this SRM may be used to calibrate and/or test a laboratory's use of EPA Analytical Methods 1624 and 1625 (as well as 624-625 and 524-525). These methods specifically require the use of combined gas chromatography/mass spectrometry (GC/MS) and the use of isotopically labeled internal standards. SRM 1586 is composed of two separate solutions. The ten Priority Pollutants in one solution (SRM 1586-2) contain either deuterium or carbon-13 while the other solution (SRM 1586-1) contains the same compounds with no isotopes except those naturally occurring.

Certified Values of Constituent Organic Compounds: The certified values for the selected organic constituents are shown in Table 1. These certified values are based on results obtained from the gravimetric preparation of these solutions and from the analytical values determined by gas chromatography. Table 2 summarizes the calculated and analytically determined concentrations.

Notice and Warnings to User

Handling: Priority Pollutants are reported to be toxic and should be handled with care. Use proper disposal methods.

Expiration of Certification: This certification is valid, within the limits certified, for one year from the date of purchase. In the event that the certification should become invalid before then, purchasers will be notified by NBS.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10-30 °C.

Use: Samples for analysis should be withdrawn immediately after opening ampoules and should be processed without delay for the certified values in Table 1 to be valid within the stated uncertainty. Certified values are not applicable to material stored in ampoules that have been opened, even if they are resealed.

Preparation and analytical determinations were performed at the Center for Analytical Chemistry, Organic Analytical Research Division, by F.R. Guenther, D.J. Pereles, R.E. Rebbert, M.J. Welch and E. White, V.

Consultation on the statistical design of the experimental work was provided by K.R. Eberhardt of the Statistical Engineering Division.

The coordination of the technical measurements leading to certification was under the direction of S.N. Chesler and W.E.. May.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

October 16, 1984
Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Preparation and Analysis

All chemicals used in the preparation of SRM 1586-1 and 1586-2 were obtained from commercial sources and were deemed the best available at the time. The chemical purities, as determined by gas chromatography, are listed in Table 3 and were used in the determination of the certified values. The isotopic purities of the compounds in SRM 1586-2 as determined by mass spectrometry at NBS are shown inTable 4. Both solutions were prepared at NBS by weighing and mixing the ten individual compounds and the methanol solvent. Each solution was chilled and ampouled into 2 mL-amber glass ampoules. Each ampoule was purged with nitrogen immediately before adding the solution and sealing. Aliquots from randomly selected ampoules were analyzed by gas chromatography with flame ionization detection. A glass column (2 m x 2 mm I.D.), packed with 60/80 Carbopack B and coated with 1% SP-1000 was used for determination of carbon tetrachloride and benzene. The internal standard (IS) for this analysis was 1,2-dichloropropane. A fused silica column (30 m x 0.25 mm I.D. x 0.25 μm film of bonded dimethyl polysiloxane) was used with splitless injection for the determination of the other eight components. For SRM 1586-1 (unlabeled), o-xylene was the IS for chlorobenzene while 6-chloro-m-cresol was the IS for phenol, nitrobenzene, 2-nitrophenol, 2,4-dichlorophenol and naphthalene, and benzo(k)fluoranthene was the IS for bis(2-ethylhexyl) phthalate and benzo(a)pyrene. For SRM 1586-2 (labeled) the same three internal standards were used but in addition bis(2-ethylhexyl) adipate was used as the IS for the bis(2-ethylhexyl) phthalate.

TABLE 1

Certified Concentrations of Priority Pollutants in SRM 1586

Compounds	Concentration (μg/g)[a]
SRM 1586-1	
Carbon tetrachloride	128.5 ± 0.5
Benzene	101.1 ± 0.8
Chlorobenzene	133.0 ± 0.6
Phenol	117.0 ± 1.3
Nitrobenzene	126.0 ± 1.1
2-nitrophenol	103.6 ± 3.2
2,4-dichlorophenol	102.5 ± 0.6
Naphthalene	126.5 ± 1.2
Bis(2-ethylhexyl)phthalate	63.9 ± 1.7
Benzo(a)pyrene	49.2 ± 0.2
SRM 1586-2 (See Table 3 for isotopic purity of these compounds)	
Carbon tetrachloride-^{13}C	124.4 ± 2.1
Benzene-d_6	99.0 ± 0.5
Chlorobenzene-d_5	144.0 ± 1.3
*Phenol-d_5	116.0 ± 0.6
Nitrobenzene-d_5	134.5 ± 1.4
2-nitrophenol-d_4	101.9 ± 2.3
2,4-dichlorophenol-d_3	82.2 ± 1.6
Naphthalene-d_8	126.6 ± 1.0
Bis[2-ethylhexyl]phthalate-d_4	60.4 ± 0.7
Benzo(a)pyrene-d_{12}	44.1 ± 2.1

[a]For each compound, the certified value is the mean of the calculated and chromatographic determinations. The corresponding uncertainty represents the symmetric interval about the certified value which covers the 95% confidence interval from the chromatographic analyses. Thus, the uncertainty reflects both random error of measurement and the systematic bias between the calculated and chromatographic values.

*Weighed as phenol-d_6, but in methanol solution it converts quantitatively to phenol-d_5.

TABLE 2

Summary of Results

Compound	Calculated Values, μg/g	Analytical Values, μg/g
Priority Pollutants SRM 1586-1		
Carbon tetrachloride	128.60	128.4 ± 0.4
Benzene	100.82	101.3 ± 0.6
Chlorobenzene	132.63	133.3 ± 0.3
Phenol	117.30	116.6 ± 1.0
Nitrobenzene	126.01	125.9 ± 1.0
2-nitrophenol	104.39	102.9 ± 2.5
2,4-dichlorophenol	102.42	102.6 ± 0.5
Naphthalene	126.74	126.3 ± 1.0
Bis[2-ethylhexyl]phthalate	64.16	63.6 ± 1.4
Benzo(a)pyrene	49.15	49.2 ± 0.2
Priority Pollutants SRM 1586-2		
Carbon tetrachloride-^{13}C	123.5	125.2 ± 1.3
Benzene-d_6	98.7	99.2 ± 0.3
Chlorobenzene-d_5	143.5	144.4 ± 0.9
*Phenol-d_5	115.9	116.0 ± 0.6
Nitrobenzene-d_5	134.0	135.0 ± 0.9
2-nitrophenol-d_4	102.3	101.4 ± 1.8
2,4-dichlorophenol-d_3	82.4	82.0 ± 1.4
Naphthalene-d_8	126.5	126.7 ± 0.9
Bis[2-ethylhexyl]phthalate-d_4	60.2	60.6 ± 0.5
Benzo(a)pyrene-d_{12}	43.8	44.4 ± 1.8

[a]Uncertainties are given as 95% confidence intervals.

*Weighed as phenol-d_6, but in methanol solution it converts quantitatively to phenol-d_5.

TABLE 3

Chemical Purity of Priority Pollutants in SRM 1586

Determined by Gas Chromatography

Compound	Purity %
SRM 1586-1	
Carbon tetrachloride	99.9
Benzene	99.9
Chlorobenzene	99.9
Phenol	99.9
Nitrobenzene	99.9
2-nitrophenol	99.9
2,4-dichlorophenol	99.9
Naphthalene	99.4
Bis[2-ethylhexyl]phthalate	99.5
Benzo(a)pyrene	99.5
SRM 1586-2	
Carbon tetrachloride-^{13}C	99.6
Benzene-d_6	99.9
Chlorobenzene-d_5	99.9
Phenol-d_6	99.9
Nitrobenzene-d_5	99.9
2-nitrophenol-d_4	99.9
2,4-dichlorophenol-d_3	98.4
Naphthalene-d_8	99.8
Bis[2-ethylhexyl]phthalate-d_4	96.7
Benzo(a)pyrene-d_{12}	98.1

TABLE 4

Isotopic Purity of Priority Pollutants in SRM 1586-2

Determined by Mass Spectrometry

Compound	Isotopic Purity, Percent	Percent of Molecules Totally Labeled
Carbon tetrachloride-^{13}C	99.5	99.5
Benzene-d_6	99.7	97.9
Chlorobenzene-d_5	99.6	97.9
*Phenol-d_6	98.3 (as d_5)	91.4 (as d_5)
Nitrobenzene-d_5	99.6	97.8
2-nitrophenol-d_4	98.9	95.5
2,4-dichlorophenol-d_3	98.7	96.0
Naphthalene-d_8	99.5	95.6
Bis[2-ethylhexyl]phthalate-d_4	98.6 (aromatic ring only)	94.5 (aromatic ring only)
Benzo(a)pyrene-d_{12}	98.8	86.2

*Weighed as phenol-d_6 but in methanol solution it converts quantitatively to phenol-d_5.