U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 1584
Priority Pollutant Phenols in Methanol

This Standard Reference Material (SRM) is intended primarily for use in the calibration of chromatographic instrumentation used in the determination of the phenols. Because of its miscibility with water, this SRM can also be used to fortify aqueous samples with known amounts of phenols.

Certified Concentrations of Phenols in Methanol. Certified values for the concentrations of ten of the eleven phenols on the United States Environmental Protection Agency's List of Priority Pollutants are provided in Table 1. These certified values are based on analytical results by either gas chromatography (GC) or high performance liquid chromatography (HPLC) and on the concentrations calculated from the mass of the phenol, corrected for compound purity, added to the mass of methanol. Because the density of methanol changes with temperature, the concentration values given are certifed for the temperature range between 19 and 27 °C only. An information value is provided for one additional phenolic compound. This value is based on the results of a single analytical technique. The concentration of each phenol as calculated and determined by either HPLC or GC is given in Table 2. The values are reported in μg/mL units and are corrected for compound purity as determined by GC. Nine of the eleven compounds were at least 99 percent pure, while the remaining two were at least 97 percent pure.

NOTICE AND WARNINGS TO USERS

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Use: Samples for analysis should be withdrawn from ampoules immediately after opening and processed without delay for any certified value in Table 1, to be valid within the stated uncertainty.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

Analytical determinations were performed at the NBS Center for Analytical Chemistry, Organic Analytical Research Division by J.M. Brown-Thomas, S.N. Chesler, D.K. Hancock, W.F. Kline, R.M. Parris, and R.E. Rebbert.

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 were performed under the direction of J.M. Brown-Thomas 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.

April 20, 1984
Washington, DC 20234

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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PREPARATION AND ANALYSIS

The methanol solution of the eleven phenols were prepared at the National Bureau of Standards and ampouled cold into two-mL amber glass ampoules. The ampoules were purged with nitrogen just prior to filling and sealed under nitrogen. Samples representing early, middle, and final stages of ampouling were analyzed by HPLC. No significant differences in concentration of the eleven compounds were found.

Randomly selected ampoules were analyzed for four of the eleven phenols by HPLC on a reversed-phase C-8 (5 μm) column using an aqueous acetic acid/acetonitrile/methanol mobile phase and UV (254 nm) detection. Four external standard solutions were used to provide response factors for quantification by external standards. GC on a fused silica OV-1701 capillary column with flame ionization detection was used for the determination of seven of the eleven compounds. Four independent calibration solutions were used to obtain compound responses relative to 6-chloro-m-cresol, the internal standard.

Table 1

Certified and Noncertified Concentrations of Phenols in SRM 1584 at 23 °C ± 4 °C

Certified Values[a]

Compound	Concentration (μg / mL)
2-Chlorophenol	64.4 ± 1.4
Phenol	29.7 ± 0.9
2-Nitrophenol	25.2 ± 0.7
2,4-Dimethylphenol	51.6 ± 0.2
2,4-Dichlorophenol	35.6 ± 1.3
4-Chloro-m-cresol	27.4 ± 0.4
2,4,6-Trichlorophenol	20.4 ± 1.9
4-Nitrophenol	20.7 ± 0.7
4,6-Dinitro-o-cresol	20.1 ± 0.9
Pentachlorophenol	15.4 ± 1.1

Noncertified Value[b]

2.4-Dinitrophenol	(22.4)

[a]For each compound, the certified value is derived as the mean of the calculated value and chromatographic determination. 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 the random error of measurement and the systematic bias between the calculated and chromatographic values.

[b]The information value for 2,4-dinitrophenol represents the weighted average of the results obtained from two independent sets of HPLC determinations, and, in our judgment, is the present best estimate of the true value. This concentration is not certified because the calculated value did not agree with the HPLC determinations.

Table 2

Summary of Results by the Analytical Methods Used in Certification

(Average Concentration, $\mu g / mL$)[a]

	Calculated	GC[b]	HPLC[b]
2-Chlorophenol	63.8	65.0 ± 0.8	
Phenol	29.6	29.8 ± 0.8	
2-Nitrophenol	25.1	25.2 ± 0.7	
2,4-Dimethylphenol	51.6	51.6 ± 0.2	
2,4-Dichlorophenol	35.8	35.5 ± 1.2	
4-Chloro-m-cresol	27.4	27.4 ± 0.4	
2,4,6-Trichlorophenol	20.2	20.5 ± 1.8	
4-Nitrophenol	20.5		20.8 ± 0.6
4,6-Dinitro-o-cresol	19.6		20.6 ± 0.4
Pentachlorophenol	15.7		15.1 ± 0.8
2,4-Dinitrophenol	25.9		21.8 ± 0.9 22.9 ± 0.4[c]

[a] Mass-to-volume conversion accomplished using density of methanol measured at 23 °C, 0.781 g/mL.

[b] Uncertainties are given as 95 percent confidence intervals, considering variation among measurements of eight samples and response factors for four calibration solutions.

[c] Measurement made by HPLC with detection at 290 nm. Results represent triplicate analyses of three ampoules.