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 1639

Halocarbons (in methanol) for Water Analysis

This Standard Reference Material is intended primarily for calibrating chromatographic instrumentation used in the determination of halocarbons. It is also useful in recovery studies for adding accurate amounts of the certified compounds to a sample. Because of its miscibility with water, it is particularly useful in analyzing water samples for these compounds.

Certified Concentrations of the Halocarbons: The certified concentrations and estimated uncertainties of seven halocarbons in methanol are shown in Table 1. Because the density of methanol changes with temperature, these concentrations are certified for the temperature range of 20 to 26 °C.

They are based on the analytical results by gas chromatography (GC) and on the concentrations calculated from the mass of the halocarbon added, corrected for compound purity, to methanol. In addition to GC, high performance liquid chromatography (HPLC) was used to confirm the homogeneity of the entire lot. Table 2 shows the calculated and GC-determined concentrations.

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Use: Samples of the SRM for analysis should be withdrawn from ampoules equilibrated at 23 ± 3 °C. Samples should be withdrawn immediately after opening and used without delay for any certified value in Table 1 to be valid within the stated uncertainty.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. A suggested procedure for preparing and equilibrating water calibration standards is described in a separate section.

Analytical determinations were performed at the Center for Analytical Chemistry, Organic Analytical Research Division, by S.N. Chesler, R.G. Christensen, and F.R. Guenther.

The statistical design and analysis of the experimental work was provided by K. Kafadar and K.R. Eberhardt of the Statistical Engineering Division.

The coordination of the technical measurements leading to certification was performed under the direction of F.R. Guenther, S.N. Chesler, and H.S. Hertz.

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

Washington, DC 20234
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George A. Uriano, Chief
Office of Standard Reference Materials

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Table 1. Certified Concentration of Halocarbons in SRM 1639 at 23 ± 3 °C

Compound	Concentration, ng/μL*
Chloroform	6235 ± 340
Chlorodibromomethane	124.6 ± 1.1
Bromodichloromethane	389.9 ± 7.1
Bromoform	86.5 ± 1.4
Carbon tetrachloride	157.0 ± 4.4
Trichloroethylene	85.8 ± 2.6
Tetrachloroethylene	40.6 ± 0.9

*Estimated uncertainty is given as 95% confidence limits obtained from the GC measurements.

Table 2. Summary of Results

Compound	Concentration, ng/μL	
	Calculated[a]	GC[b]
Chloroform	6311 ± 34	6235 ± 338
Chlorodibromomethane	124.9 ± 0.1	124.6 ± 1.1
Bromodichloromethane	392.0 ± 0.3	389.9 ± 7.1
Bromoform	87.7 ± 0.1	86.5 ± 1.4
Carbon tetrachloride	160.1 ± 0.1	157.0 ± 4.4
Trichloroethylene	87.9 ± 0.1	85.8 ± 2.6
Tetrachloroethylene	40.95 ± 0.03	40.6 ± 0.9

[a]The calculated concentration is based on the total mass of the halocarbon added to the methanol.
[b]Estimated uncertainty is given as 95% confidence limits.

Suggested Procedure for Preparing and Equilibrating Water Calibration Standards

The following procedure provides aqueous halocarbon concentrations in the range normally found in drinking water supplies.

1. Allow ampoule to equilibrate at a temperature of 23 ± 3 °C and shake ampoule for one minute.
2. Open ampoule and complete sampling of contents within five minutes.
3. Insert one-μL disposable micropipet (such as "Microcap"* available from laboratory supply houses) into ampoule and fill micropipet from top of SRM solution.
4. Inject the one-μL volume into a stoppered 100-mL volumetric flask containing 100-mL pure water and rinse the micropipet twice with this solution.
5. Shake the flask for at least one minute and analyze entire sample. However, because the one-minute shaking time may not have achieved a uniform concentration of halocarbons in the water, the analysis of aliquots may require a longer shaking time.

Other concentrations can be obtained by the use of other calibrated micropipets and/or volumetric flasks of suitable volume.

Preparation and Analysis of SRM 1639

The methanol solution of the seven halocarbons was prepared at NBS. It was chilled and ampouled into 2-mL amber glass ampoules. The ampoules were purged with argon immediately before adding the solution. Samples representing early, middle, and final stages of ampouling were analyzed by GC. HPLC was also used to confirm homogeneity of the material for the SRM. No significant differences in concentrations of the seven compounds were found.

GC analyses were done on a 30 m x 0.25 mm inside diameter fused silica capillary column coated with a one-μm thick immobilized SE-52* phase. Bromotrichloromethane was used as an internal standard. A Hall electrolytic conductivity detector was used in the halogen mode for the determination of all constituents except bromoform and chlorodibromomethane. These highly brominated compounds were determined using electron capture detection.

*Note: To describe the procedure adequately, identification of a commercial product by the manufacturer's name was necessary. This identification does not imply endorsement by NBS nor that the particular product is the best available.