

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1589

Polychlorinated Biphenyls (as Aroclor 1260) in Human Serum

This Standard Reference Material (SRM) is intended for use in evaluating the accuracy of analytical methods for the determination of polychlorinated biphenyls (PCB's) as Aroclor 1260 in human serum. It may also be used in calibrating instruments and equipment used in these procedures and in validating working or secondary reference materials.

Certified Values

The certified concentration of the Aroclor 1260 mixture in this serum is given in Table 1 in both ng/g and ng/mL units. The concentration listed in ng/g is based on the calculated value from the gravimetric preparation of the material, the analyses of the reconstituted material, and blank measurements for the original unlyophilized, unfortified human serum. The certified concentrations apply to the solution obtained after the serum has been reconstituted using the procedures described in "Instructions for Use."

Table 1

Certified Concentrations of Aroclor 1260 in Reconstituted SRM 1589

<u>ng/g</u>	<u>ng/mL^a</u>
106.0 ± 1.3	107.9 ± 1.4

^aApplicable for the determination of Aroclor in the temperature range of 20 and 25 °C. An additional 0.1 ng/mL is added to the uncertainty because of the change of density with temperature.

The uncertainties listed represent two standard deviations of the certified values and include the analytical imprecision, the bottle-to-bottle variability, and the specified imprecision in the reconstitution of the SRM samples. (See "Instructions for Use".)

Information Values for Dioxins

The concentrations of 1,2,3,4-tetrachlorodibenzo-p-dioxin (1,2,3,4-TCDD) and 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) in the reconstituted serum reported in Table 2 were calculated based upon gravimetry. Analyses were not performed to verify these concentrations.

Table 2
Informational Values

<u>Compound</u>	<u>ng/g</u>	<u>ng/mL</u>
1,2,3,4-TCDD	0.153	0.156
2,3,7,8-TCDD	0.081	0.083

Notice and Warning to Users

Handle as if capable of transmitting hepatitis! Although this product was tested with licensed third generation reagents and found nonreactive for the presence of hepatitis B surface antigen (HB_sAG), no known test method can offer assurances that products derived from human blood will not transmit hepatitis. Furthermore, PCB's and dioxins are reported to be very toxic and/or carcinogenic and therefore should be handled with care. Use proper disposal methods.

SRM 1589 is intended for "in vitro" diagnostic use only.

Instructions for Use

This Standard Reference Material is supplied as a freeze-dried preparation in a septum sealed vial. It should be stored at refrigerator temperature (approximately 5 °C) and should not be frozen nor exposed to sunlight or ultraviolet radiation. The material is not certified for use after one year from the date of purchase.

For use, it is necessary to reconstitute the lyophilized material with high-purity distilled water. Bring vial to room temperature; remove metal closure; and, lightly tap bottom of vial to dislodge any dried serum particles on stopper. Carefully remove stopper to avoid possible loss of serum particles. Use a type 1 Class A volumetric transfer pipet or other dispenser of known accuracy to add slowly 10.00 ± 0.02 mL of distilled water at 20 to 25 °C to the sides of the vial while continually turning the vial. Replace stopper, swirl vial two or three times, and let stand for about 10 minutes. Mix contents by gently swirling, let stand for approximately 30 minutes, swirl again, let stand 10 minutes and finally invert the vial several times. Do not shake vigorously because this will cause frothing. Total time for reconstitution is approximately 1 hour. After reconstituting, use contents as soon as possible or store at about 5 °C until ready, preferably within 8 hours.

Preparation and Analysis of SRM 1589

The bulk serum solution containing Aroclor 1260 (including 1,2,3,4-TCDD and 2,3,7,8-TCDD) was gravimetrically prepared at NBS. At Whittaker, M.A. Bioproducts, Walkersville, MD., the solution was dispensed into vials and lyophilized under NBS supervision. After reconstitution and the addition of an internal standard (2,2,3,3',4,5,5',6, octachlorobiphenyl, BS # 198[1]), the sample was extracted repeatedly with hexane/diethyl ether, concentrated and purified by passing it through a washed silica cartridge. The eluant was concentrated evaporatively to approximately 200 μ L; and, 3 μ L was injected into a capillary gas chromatograph equipped with an electron capture detector. A fused silica column (30 m x 0.25 mm I.D. x 0.25 μ m film of bonded dimethyl polysiloxane) was used with split injection. A chromatogram representing the determination of Aroclor 1260 in this SRM is shown in Figure 1. "I.S." is the internal standard and the peaks numbered from 1 to 6 are reference peaks for quantification of Aroclor 1260.

Analyses leading to certification were performed in the NBS Center for Analytical Chemistry, Organic Analytical Research Division, by R.E. Rebbert and L.T. Sniegowski.

The statistical analysis of the data was performed by R.C. Paule of the NBS National Measurement Laboratory.

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.

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Stanley D. Rasberry, Chief
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Reference

[1] Ballschmiter, K. and M. Zell, *Fresenius Z. Anal. Chem.* 302, 20 (1980).

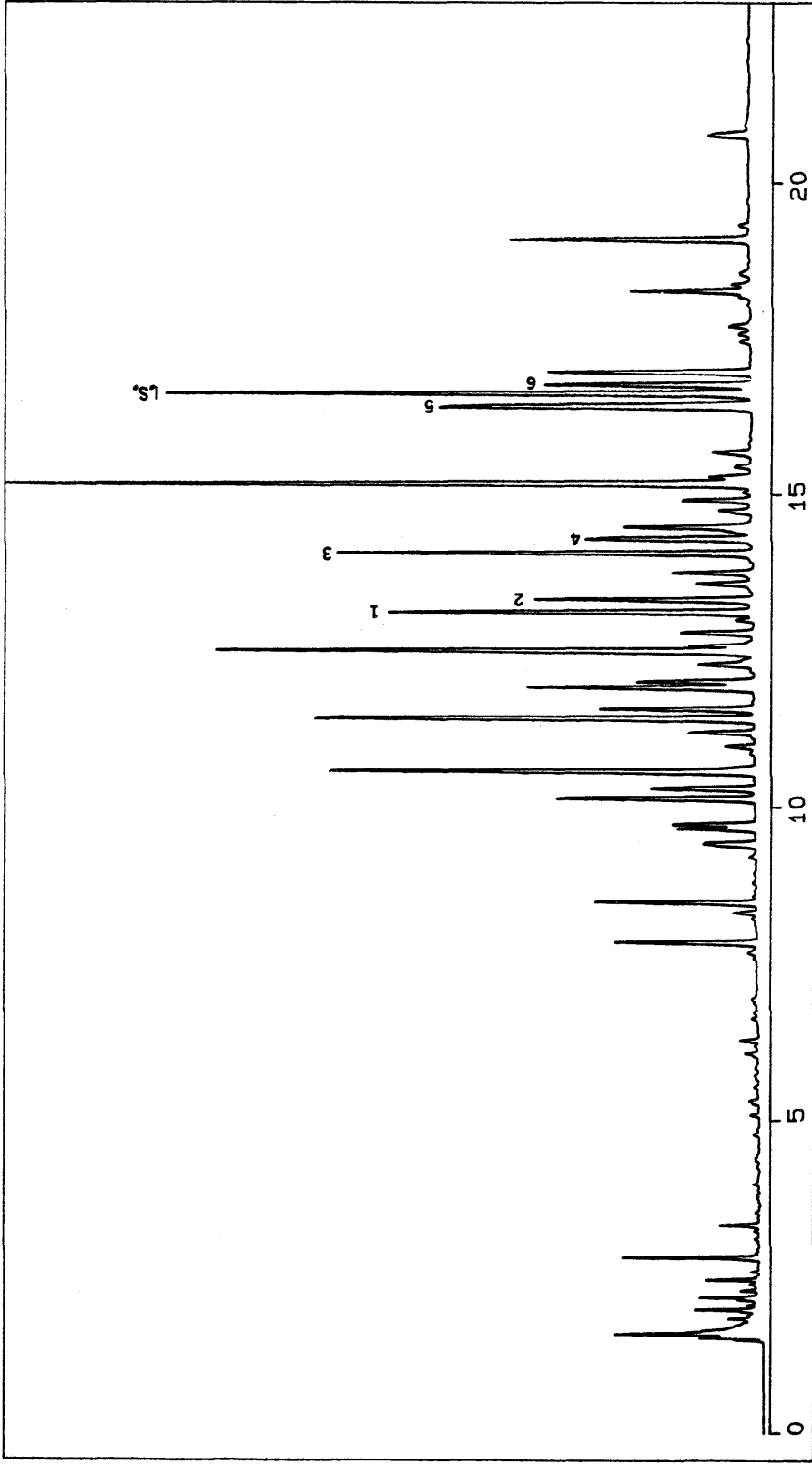


Figure 1

Chromatogram Representing Determination of Aroclor 1260 in SRM 1589