National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1974

Organics in Mussel Tissue (Mytilus edulis)

This Standard Reference Material (SRM) is intended primarily for use in validating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs) in marine bivalve tissue or materials of similar matrix. Noncertified concentrations of additional PAHs, polychlorinated biphenyls (PCBs), chlorinated pesticides, and inorganic constituents are also provided. A unit of SRM 1974 consists of three bottles, each containing approximately 15-20 g (wet weight) of frozen tissue homogenate.

CERTIFIED CONCENTRATIONS

Certified concentrations of nine PAHs, which are naturally present in the mussel tissue, are provided in Table 1. These values are based on the results obtained from the analysis of this material using two different sample preparation procedures and two different analytical techniques (gas chromatography-mass spectrometry and reversed-phase liquid chromatography with fluorescence detection). A summary of the results obtained from the two independent analytical procedures is provided in Appendix A. Noncertified concentrations for additional PAHs, PCBs, and pesticides are provided in Appendices B, C, and D, respectively. Noncertified concentrations for trace inorganic constituents are provided in Appendix E.

Table 1. Certified Concentrations of PAHs in SRM 1974[a]

Compound	Concentration	
	ng/g wet weight[b]	ng/g dry weight[a]
Phenanthrene	5.6 ± 1.4	45 ± 11
Anthracene	0.75 ± 0.21	6.1 ± 1.7
Fluoranthene	33.6 ± 5.8	272 ± 47
Pyrene	34.1 ± 3.7	276 ± 30
Perylene	1.05 ± 0.29	8.5 ± 2.4
Benzo[b]fluoranthene	6.5 ± 1.2	52.3 ± 9.4
Benzo[a]pyrene	2.29 ± 0.47	18.6 ± 3.8
Benzo[ghi]perylene	2.47 ± 0.28	20.0 ± 2.3
Indeno[1,2,3-cd]pyrene	1.80 ± 0.33	14.6 ± 2.7

[a] Certified values were determined on a wet weight basis; concentrations were converted to a dry weight basis for user convenience.

[b] The certified values are equally weighted means of results from two analytical techniques. The uncertainty is obtained from a 95% prediction interval plus an allowance for systematic error between the methods used. In the absence of systematic error, the resulting uncertainty limits will cover the concentration of approximately 95% of samples of this SRM having a minimum sample size of 15 g (wet weight).

Gaithersburg, MD 20899
May 15, 1991
(Revision of certificate dated 10-15-90)

William P. Reed, Chief
Standard Reference Materials Program

(over)

NOTICE AND WARNING TO USERS

Storage: SRM 1974 is provided as a frozen tissue homogenate in glass bottles. The tissue homogenate should not be allowed to thaw prior to analysis. This material has been stored at NIST at -80 °C (or lower) since it was prepared. SRM 1974 should be stored at temperatures less than -80 °C, if possible, since the validity of the certified values at higher temperatures is unknown.

Expiration of Certification: This certification is valid within the specified uncertainty limits for one year from the date of purchase. In the event that the certification should become invalid before then, purchasers will be notified by NIST. Please return the attached registration form to facilitate notification.

Handling: This material is a frozen tissue homogenate; if allowed to warm, it will not retain its powder-like form. After extended storage at temperatures of -25 °C or higher, the homogenate will lose its powder-like form. For the handling of this material during sample preparation, the following steps are suggested. If weighing relatively large quantities, remove a portion from the bottle and reweigh the bottle to determine the weight of the subsample. Avoid heavy frost buildup on the containers by rapid handling and wiping of the containers prior to weighing. To transfer subsamples to another container for weighing, use a pre-cooled thick-walled glass container rather than a thin-walled plastic container to minimize heat transfer to the sample. If possible use a cold work space, e.g., an insulated container with dry ice or liquid nitrogen coolant on the bottom. Use pre-cooled implements, such as Teflon-coated spatulas, for transferring the powder.

For inorganic analytical techniques that require the use of a dry sample for sample preparation and analysis, the mussel tissue may be freeze-dried by starting at -20 °C and slowing increasing the temperature to 0 or 5 °C until a stable weight is obtained. However, after freeze-drying, the sample is hygroscopic. SRM 1974 should not be dried prior to analysis for organic constituents since drying may alter the certified and noncertified concentrations of the organic constituents. Normal biohazard safety precautions for the handling of biological tissues should be exercised.

Use: Subsamples for analysis should be withdrawn from the bottle immediately after opening and used without delay for the certified values listed in Table 1 to be valid. It is intended that the total contents of a bottle be used immediately after it has been opened. The concentrations of organic and inorganic constituents in SRM 1974 are reported on both a wet weight and a dry weight basis for user convenience. The SRM tissue homogenate, as received, contains approximately 88% moisture. A separate subsample of the SRM should be removed from the bottle at the time of analysis and dried (see section on Conversion to Dry Weight Basis) to determine the concentration based on dry weight.

Collection and preparation of SRM 1974 were performed in the NIST Center for Analytical Chemistry in the Organic Analytical Research Division by B. A. Benner, Jr., R. G. Christensen, B. J. Koster, M. M. Schantz, M. R. Summers, and S. A. Wise.

Analytical measurements were performed in the NIST Center for Analytical Chemistry in the Organic Analytical Research Division by B. A. Benner, Jr., B. J. Koster, M. M. Schantz, and S. A. Wise and in the Inorganic Analytical Research Division by R. Zeisler. Additional measurements of selected inorganic constituents were performed at the Nuclear Research Center, Institute for Applied Physical Chemistry in Jülich, Federal Republic of Germany by K. May, P. Ostapczuk and M. Stoeppler.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by W. F. Guthrie, S. B. Schiller, and K. R. Eberhardt of the Statistical Engineering Division in the NIST Center for Computing and Applied Mathematics.

The overall coordination of the collection, preparation, and technical measurements leading to certification was under the direction of S. A. Wise and W. E. May.

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

The collection, preparation, and certification of SRM 1974 were supported in part by the Ocean Assessments Division, National Oceanic and Atmospheric Administration (NOAA); Office of the Chief of Naval Operations, Department of the Navy; and Minerals Management Service, Department of the Interior. The mussels used for SRM 1974 were collected with the assistance of S. Freitas of Battelle New England Research Laboratory, Duxbury, MA.

PREPARATION AND ANALYSIS

Sample Collection. The mussels (*Mytilus edulis*) used for the preparation of SRM 1974 were collected on December 1, 1987 from Dorchester Bay within Boston Harbor, MA (Position 42°18.25'N, 71°02.31'W). Approximately 2400 individual mussels were collected by hand at low tide. The samples were transported to the Battelle New England Laboratory (Duxbury, MA) where the mussels were rinsed in a tank supplied with pumped sea water; rocks and other debris were removed. The samples were placed in insulated, Teflon-lined wooden containers, frozen and transported to NIST on dry ice. The samples were transferred to Teflon bags and stored in a liquid nitrogen vapor freezer (-120 °C) until they were shucked.

Sample Preparation. The mussel tissue was removed from the shell using the following procedure. The mussels were allowed to warm up to about 0 °C; the tissue was removed from the shell using a titanium knife and placed in Teflon bags (approximately 1 kg per bag) and immediately returned to a liquid nitrogen freezer. Approximately 28 kg of mussel tissue were prepared for use as the SRM. The frozen mussel tissue was pulverized in batches of approximately 150 g each using a cryogenic procedure described previously [1]. The total 28 kg of pulverized material was then combined in an aluminum mixing drum. The mixing drum was designed to fit inside the liquid nitrogen vapor freezer and to rotate in the freezer thereby mixing the frozen tissue powder. After mixing for 2 h, subsamples (15-20 g) of the mussel tissue homogenate were aliquoted into pre-cooled glass bottles. The bottles of SRM 1974 have been stored at -80 °C since preparation.

Conversion to Dry Weight Basis. The moisture content of the mussel homogenate was determined by measuring the weight loss after freeze drying. Twenty bottles of SRM 1974 were selected according to a stratified randomization scheme for the drying study. The entire contents of each bottle were transferred to a Teflon jar and dried for 5 days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. Based on these studies, a 95% prediction interval for the moisture content of a new bottle of SRM 1974 is 87.7 ± 0.2%. This interval will cover the moisture content of approximately 95% of all bottles. Analytical results for the organic constituents were determined on a wet weight basis and then converted to a dry weight basis by dividing by the conversion factor of 0.123. Inorganic constituents were determined in SRM 1974 using freeze-dried material.

Polycyclic Aromatic Hydrocarbons. The SRM was analyzed for the determination of selected PAHs using gas chromatography with mass spectrometric detection (GC-MS) and reversed-phase liquid chromatography with fluorescence detection (LC-FL). A more detailed discussion of the analysis of SRM 1974 will be reported elsewhere [2].

For the GC-MS analyses, a subsample of 13-26 g (wet weight) of the mussel homogenate from 12 randomly selected bottles was mixed with approximately 100 g of sodium sulfate, an internal standard solution (see below) was added to the sodium sulfate-tissue mixture, and then the mixture was Soxhlet extracted for 18 h using 300 mL of methylene chloride. The extract was concentrated, and gel-permeation chromatography on a semipreparative divinylbenzene-polystyrene column (10 μm particle size, 100 Å pore size) was used to remove the majority of the lipid and biogenic materials. The eluant was then passed through a silica solid phase extraction (SPE) cartridge as the final cleanup step prior to GC-MS analysis. GC-MS analyses were performed using a 0.25 mm x 60 m fused silica capillary column with a 5% phenyl substituted polysiloxane phase (0.25μm film thickness).

For the LC-FL analyses, a subsample of 14-18 g (wet weight) of the mussel homogenate from six randomly selected bottles was mixed with approximately 100 g of sodium sulfate, an internal standard solution (see below) was added to the sodium sulfate-tissue mixture, and then the mixture was Soxhlet extracted for 18 h using 250 mL of hexane:acetone (1:1 v/v). The extract was concentrated and then passed through an aminosilane SPE cartridge to remove the lipid and more polar interferences. The eluant from the SPE cartridge was concentrated and the SPE procedure repeated a second and third time. After the third SPE cleanup, the eluant was concentrated and injected onto a semipreparative aminosilane column to isolate the PAH fraction by normal-phase LC [3]. The isolated PAH fraction was then analyzed by reversed-phase LC using a polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. x 25 cm, 5-μm particle size) with wavelength programmed fluorescence detection [4-6].

For both the GC-MS and LC-FL measurements, perdeuterated PAHs were utilized as the internal standards. The internal standard solutions were added to the mussel tissue/sodium sulfate mixture immediately prior to Soxhlet extraction. For GC-MS analyses the following internal standards were used: naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, pyrene-d_{10}, benz[a]anthracene-d_{12}, benzo[e]pyrene-d_{12}, and benzo[ghi]perylene-d_{14}. For the LC-FL analyses the following internal standards were used: phenanthrene-d_{10}, fluoranthene-d_{10}, and perylene-d_{12}. Response factors for the analytes relative to the internal standards were determined by analyzing SRM 1491 "Aromatic Hydrocarbons in Hexane/Toluene" for the GC-MS analyses or SRM 1647a "Polycyclic Aromatic Hydrocarbons (in Acetonitrile)" for the LC-FL analyses.

PCBs and Chlorinated Pesticides. The SRM was analyzed for the determination of selected PCBs and chlorinated pesticides using GC with electron capture detection (GC-ECD). Subsamples from 11 bottles of SRM 1974 were extracted, and the extracts were passed through the gel permeation column as described above for the GC-MS analysis. Following the gel permeation chromatography, normal-phase LC on the semipreparative aminosilane column was used to isolate two fractions containing (1) the PCBs and lower polarity chlorinated pesticides and (2) the more polar chlorinated pesticides. GC-ECD analysis was performed on a column similar to the one used for the GC-MS determination of the PAHs. Representative chromatograms from the analysis of the PCB and lower polarity pesticide fraction and the more polar pesticide fraction are shown in Figures 1 and 2, respectively. The results of the GC-ECD analyses are summarized in Appendices C and D.

Selected PCB congeners and chlorinated pesticides (PCB 103, PCB 198, endrin, 4,4'-DDT-d_8), which are not significantly present in the sample, were added to the mussel/sodium sulfate mixture immediately prior to extraction for use as internal standards for quantification purposes. Response factors for the analytes relative to the internal standards were determined by processing gravimetrically prepared calibration solutions of the analytes of interest and the internal standards through the entire analytical procedure.

Inorganic Constituents. A number of major, minor and trace elements were determined using instrumental neutron activation analysis (INAA), differential pulse anodic stripping voltammetry (DPASV), and cold vapor atomic absorption spectrometry (CVAAS). INAA was performed at NIST following the principles of the previously described procedure for marine bivalves [7]. Samples from six randomly selected bottles of SRM 1974 were freeze-dried and three portions of 250 mg each from each bottle were pelletized and analyzed by INAA. The usual sequential determination via short-lived and long-lived nuclides was carried out on only one pellet, the second pellet was used only with a short irradiation, and the third pellet was used with a long irradiation. Hence, the reported values (Appendix E) are based on 12 determinations (two pellets from each of six bottles) for each of the elements determined by INAA.

Three portions from one bottle of the freeze-dried SRM were analyzed at KFA Jülich by DPASV to determine Co, Ni, Zn, Cu, Cd, and Pb, and by CVAAS to determine Hg. The voltammetric determinations were carried out according to previously published procedures for biological and environmental samples [8], after high pressure ashing digestion with nitric acid [9]. CVAAS was performed after wet digestion in completely closed quartz vessels [10]. The reported values for CVAAS and DPASV are based on three sample dissolutions which were each analyzed in duplicate. The reported values for Co and Zn are the combined results from INAA and DPASV.

REFERENCES

[1] R. Zeisler, J. K. Langland, and S. H. Harrison, Cryogenic Homogenization of Biological Tissues, **Anal. Chem.** 55, 2431-2434 (1983).

[2] S. A. Wise, R. G. Christensen, B. A. Benner, Jr., B. J. Koster, M. M. Schantz, and R. Zeisler, Preparation and Analysis of a Frozen Mussel Tissue Reference Material for the Determination of Trace Organic Constituents, to be submitted for publication.

[3] S. A. Wise, S. N. Chesler, H. S. Hertz, L. R. Hilpert, and W. E. May, Chemically-Bonded Aminosilane Stationary Phase for the High Performance Liquid Chromatographic Separation of Polynuclear Aromatic Hydrocarbons, **Anal. Chem.** 49, 2306-2310 (1977).

[4] W. E. May and S. A. Wise, Liquid Chromatographic Determination of Polycyclic Aromatic Hydrocarbons in Air Particulate Extracts, **Anal. Chem. 56**, 225-232 (1984).

[5] S. A. Wise, B. A. Benner, G. D. Byrd, S.N. Chesler, R. E. Rebbert, and M. M. Schantz, Determination of Polycyclic Aromatic Hydrocarbons in a Coal Tar Standard Reference Material, **Anal. Chem. 60**, 887-894 (1988).

[6] M. M. Schantz, B. A. Benner, Jr., S. N. Chesler, B. J. Koster, S. F. Stone, R. Zeisler, and S. A. Wise, Preparation and Analysis of a Marine Sediment Reference Material for the Determination of Trace Organic Constituents, **Fresenius J. Anal. Chem.**, in press.

[7] R. Zeisler, S. F. Stone, and R. W. Sanders, Sequential Determination of Biological and Pollutant Elements in Marine Bivalves, **Anal. Chem. 60**, 2760-2765 (1988).

[8] P. Ostapczuk, P. Valenta, and H. W. Nürnberg, Square Wave Voltammetry - A Rapid and Reliable Determination Method for Zn, Cd, Pb, Cu, Ni, and Co in Biological and Environmental Samples, **J. Electroanal. Chem. 214**, 51-64 (1986).

[9] P. Ostapczuk, M. Froning, and M. Stoeppler, Voltammetric Determination of Heavy Metals in Environmental Samples after HPA Digestion with Nitric Acid, **Fresenius Z. Anal. Chem. 334**, 661 (1989).

[10] K. May and M. Stoeppler, Complete Wet Digestion in Partly and Completely Closed Quartz Vessels for Subsequent Trace and Ultratrace Mercury Determinations, **Fresenius Z. Anal. Chem. 317**, 248-251 (1984).

[11] K. Ballschmiter and M. Zell, Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography - Composition of Technical Aroclor- and Clophen-PCB Mixtures, **Fresenius Z. Anal. Chem. 302**, 20-31 (1980).

SUPPLEMENTAL INFORMATION

Noncertified Quantitative Values

Appendices A through D contain supplemental analytical results obtained from the analysis of SRM 1974. Appendix A contains a comparison of the analytical results for the PAHs obtained using the two analytical techniques during the certification of SRM 1974. Noncertified concentration values are listed in Appendix B for additional PAHs, in Appendix C for 13 PCB congeners in Appendix D for 12 chlorinated pesticides, and in Appendix E for 34 inorganic constituents. The values reported are the results obtained by the measurement technique indicated and may include unrecognized bias; therefore, they are provided for information only. The uncertainties given represent only the precision of the measurement process. NIST does not recommend that this information be used for calibration, bias evaluation, or similar purposes for which certified values are used.

APPENDIX A

Summary of Analytical Results for the Determination of PAHs in SRM 1974

	Concentration (ng/g dry weight)[a]	
Compound	LC/Fluorescence	GC-MS
Phenanthrene	44.6 ± 2.7	45.3 ± 7.3
Anthracene	5.97 ± 0.52	6.14 ± 0.72
Fluoranthene	289 ± 10	255 ± 21
Pyrene	294 ± 10	259 ± 12
Perylene	8.56 ± 0.35	8.5 ± 1.7
Benzo[b]fluoranthene	55.9 ± 2.2	48.7 ± 5.2
Benzo[a]pyrene	20.1 ± 2.3	17.1 ± 2.2
Benzo[ghi]perylene	19.6 ± 1.4	20.3 ± 2.3
Indeno[1,2,3-cd]pyrene	15.6 ± 1.4	13.6 ± 1.4

[a] Uncertainties are one standard deviation of a single measurement.

APPENDIX B

Noncertified Concentrations of PAHs in SRM 1974

NOTE: Although bias has not been evaluated for the procedures used, these noncertified concentrations should be useful for comparison with results obtained using similar procedures (i.e, solvent extraction and GC-MS or LC-FL on similar columns).

Compound	Concentration ng/g wet weight[a]	ng/g dry weight[a]
2-Methylnaphthalene[b,c]	2.1 ± 0.5	17 ± 4
1-Methylnaphthalene[b,c]	1.1 ± 0.2	9 ± 2
Fluorene[b,c]	1.5 ± 0.2	12 ± 2
9-Methyl- and 4-methylphenanthrene[b,c,d]	2.7 ± 0.6	22 ± 5
1-Methylphenanthrene[b,c]	2.3 ± 0.6	19 ± 5
2- and 9-Ethylphenanthrenes and 3,6-Dimethylphenanthrene[b,d]	4.2 ± 1.0	34 ± 8
2,6-Dimethylphenanthrene[b]	4.6 ± 0.9	37 ± 7
2,7-Dimethylphenanthrene[b]	4.3 ± 1.1	35 ± 9
1,3-,2,10, 3,9-, and 3,10-Dimethyl-phenanthrenes[b,c]	11 ± 2	91 ± 17
1,6- and 2,9-Dimethylphenanthrenes[b,d]	5.8 ± 1.4	47 ± 11
1,7-Dimethylphenanthrene[b]	5.2 ± 1.1	42 ± 9
Benz[a]anthracene[b]	4.6 ± 0.4	37 ± 3
Chrysene/Triphenylene[b,d]	15.3 ± 1.4	124 ± 11
Benzo[a]fluoranthene[b]	0.51 ± 0.15	4.1 ± 1.2
Benzo[j]fluoranthene/Benzo[k]fluoranthene[b,d]	4.3 ± 0.7	35 ± 6
Benzo[k]fluoranthene[e]	3.0 ± 0.1	24 ± 1
Benzo[e]pyrene[b]	10 ± 1	81 ± 6
Indeno[1,2,3-cd]fluoranthene[b]	0.48 ± 0.07	3.9 ± 0.6
Dibenz[a,h]anthracene[e]	0.35 ± 0.01	2.8 ± 0.1

[a] Uncertainties are one standard deviation of a single measurement treating all measurements as statistically independent and identically distributed.
[b] Concentration was determined by GC-MS.
[c] Three bottles were analyzed for these compounds; nine to twelve bottles were analyzed for all other compounds determined by GC-MS; six bottles were analyzed for compounds determined by LC-FL.
[d] Represents the coelution of two or more compounds.
[e] Concentration was determined by LC-FL.

APPENDIX C

Noncertified Concentrations of Selected PCB Congeners in SRM 1974

The noncertified values in Appendix C have been removed and are currently being revised. A new Certificate with the revised values for the PCB congeners will be sent to you as soon as it is available.

Polychlorinated Biphenyls[a]

PCB 18 (2,2',5-Trichlorobiphenyl)
15 (4,4'-Dichlorobiphenyl)

PCB 28 (2,4,4'-Trichlorobiphenyl)

PCB 44 (2,2'3,5'-Tetrachlorobiphenyl)

PCB 52 (2,2',5,5'-Tetrachlorobiphenyl)

PCB 66 (2,3',4,4'-Tetrachlorobiphenyl)
95 (2,2'3,5',6-Pentachlorobiphenyl)

PCB 101 (2,2',4,5,5'-Pentachlorobiphenyl)
90 (2,2',3,4',5-Pentachlorobiphenyl)

PCB 105 (2,3,3',4,4'-Pentachlorobiphenyl)

PCB 118 (2,3',4,4',5-Pentachlorobiphenyl)

PCB 128 (2,2',3,3',4,4'-Hexachlorobiphenyl)

PCB 138 (2,2',3,4,4',5'-Hexachlorobiphenyl)
163 (2,3,3',4',5,6-Hexachlorobiphenyl)
164 (2,3,3',4',5',6-Hexachlorobiphenyl)

PCB 153 (2,2',4,4',5,5'-Hexachlorobiphenyl)

PCB 180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl)

PCB 187 (2,2',3,4',5,5'6-Heptachlorobiphenyl)
159 (2,3,3',4,5,5'-Hexachlorobiphenyl)
182 (2,2,'3',4,4',5,6'-Heptachlorobiphenyl)

[a] PCBs are numbered according to reference [11]; PCB congener listed first is the major component; additional PCB congeners listed may be present as minor components.

APPENDIX D

Noncertified Concentrations of Selected Chlorinated Pesticides in SRM 1974 as Determined by GC-ECD

NOTE: Although bias has not been evaluated for the procedure used, these non-certified concentrations should be useful for comparison with results obtained using similar procedures (i.e, solvent extraction and GC-ECD on a similar column).

Chlorinated Pesticides	Concentration ng/g wet weight[a]	ng/g dry weight[a]
Heptachlor	0.30 ± 0.02	2.4 ± 0 .2
gamma-BHC	0.19 ± 0.01	1.5 ± 0.1
Heptachlor Epoxide	0.25 ± 0.02	2.0 ± 0.2
cis-Chlordane (alpha-Chlordane)	6.3 ± 0.5	51 ± 4
4 trans-Nonachlor	2.3 ± 0.1	19 ± 1
Dieldrin	3.6 ± 0.2	29 ± 2
2,4'-DDE	0.69 ± 0.02	5.6 ± 0.2
4,4'-DDE	4.6 ± 0.2	37 ± 2
2,4'-DDD	4.6 ± 0.5	37 ± 4
4,4'-DDD	10 ± 1	81 ± 6
2,4'-DDT	0.38 ± 0.04	3.1 ± 0.3
4,4'-DDT	0.25 ± 0.02	2.0 ± 0.2

[a] Uncertainties are one standard deviation of a single measurement treating all measurements as statistically independent and identically distributed. Samples from eleven bottles were extracted; each extract was analyzed in triplicate.

APPENDIX E

Noncertified Concentrations of Inorganic Constituents in SRM 1974

NOTE: These noncertified values were obtained using procedures that have been used previously to provide certified values for similar SRM's. However, this SRM was not analyzed using a second analytical procedure; therefore, unrecognized bias may exist for the determination of some analytes in this matrix.

Element[a]	μg/g wet weight[b]	μg/g dry weight[b]
Na (%)	0.406 ± 0.011	3.29 ± 0.09
Mg (%)	0.059 ± 0.004	0.48 ± 0 .03
Al	62.1 ± 5.7	503 ± 46
Cl (%)	0.746 ± 0.021	6.04 ± 0.17
K (%)	0.136 ± 0.040	1.10 ± 0.33
Sc	0.0105 ± 0.0011	0.085 ± 0.009
V	0.191 ± 0.036	1.55 ± 0.29
Cr	0.322 ± 0.026	2.61 ± 0.21
Mn	1.26 ± 0.15	10.2 ± 1.2
Co	0.047 ± 0.001[c]	0.38 ± 0.01[c]
Fe	61.8 ± 3.3	500 ± 27
Ni	0.124 ± 0.010[d]	1.00 ± 0.08[d]
Cu	1.14 ± 0.24[d]	9.2 ± 1.9d
Zn	11.3 ± 0.5	91.6 ± 3.8[c]
As	1.20 ± 0.04	9.72 ± 0.35
Se	0.247 ± 0.007	2.00 ± 0.06
Br	46.1 ± 2.2	373 ± 18
Rb	0.700 ± 0.020	5.67 ± 0.16
Sr	7.4 ± 1.8	60 ± 14
Mo	0.24 ± 0.06	2.0 ± 0.5
Ag	0.105 ± 0.003	0.854 ± 0.021
Cd	0.17 ± 0.05[d]	1.4 ± 0.4[d]
Sb	0.00324 ± 0.00002	0.0262 ± 0.0002
Cs	0.0049 ± 0.0004	0.040 ± 0.003
La	0.043 ± 0.009	0.35 ± 0.08
Ce	0.065 ± 0.017	0.53 ± 0.13
Sm	0.0079 ± 0.0017	0.064 ± 0.014
Eu	0.0015 ± 0.0003	0.012 ± 0.002
Hf	0.006 ± 0.004	0.05 ± 0.03
Ta	0.0022 ± 0.0003	0.018 ± 0.003
Au	0.00589 ± 0.00013	0.0476 ± 0.0010
Hg	24.0 ± 1.7[e]	194 ± 14[e]
Pb	1.20 ± 0.07[d]	9.7 ± 0.6[d]
Th	0.009 ± 0.002	0.07 ± 0.02

[a] Elements listed in order of atomic number.

[b] Uncertainties are one standard deviation of a single measurement assuming all measurements are statistically independent and identically distributed. For INAA results, samples from six bottles were analyzed in duplicate.

[c] Value is the combination of the INAA and voltammetry results.

[d] Value determined by voltammetry at KFA Jülich; three subsamples from one bottle analyzed in duplicate.

[e] Value determined by CVAAS at KFA Jülich; three subsamples from one bottle of SRM 1974 analyzed in duplicate.

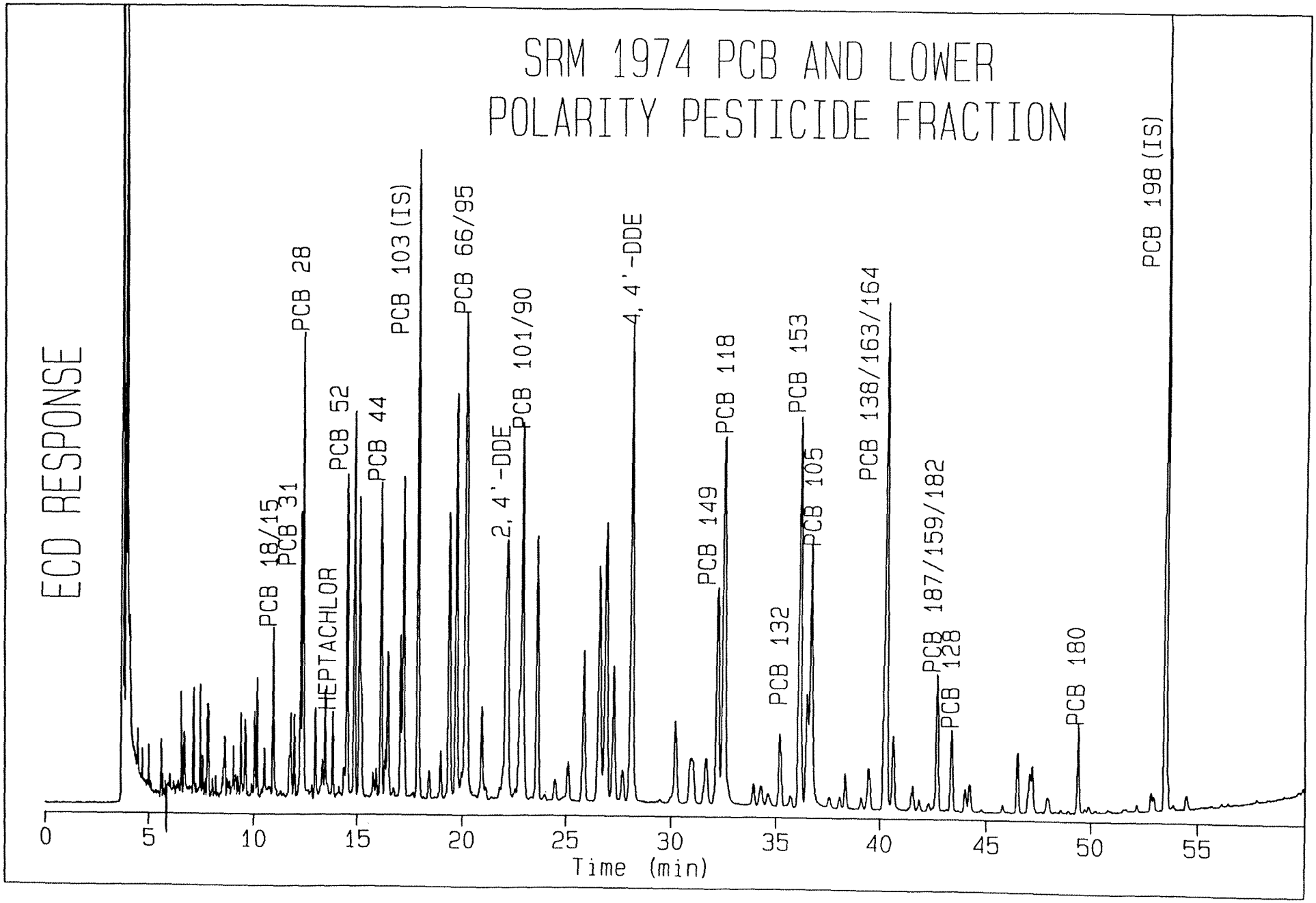

Figure 1. GC-ECD analysis of the PCB and lower polarity chlorinated pesticide fraction isolated from SRM 1974.

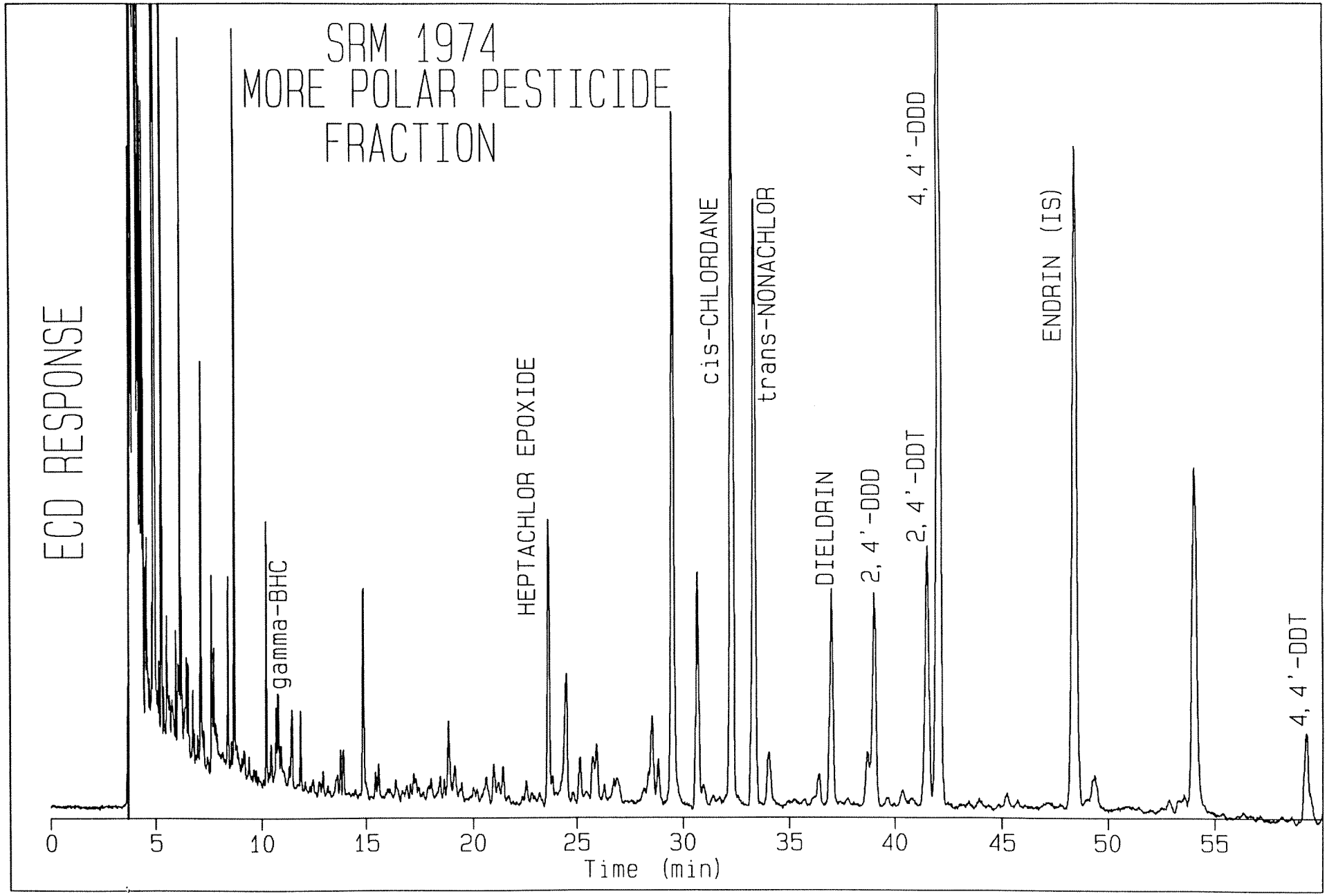

Figure 2. GC-ECD analysis of the more polar chlorinated pesticide fraction isolated from SRM 1974.