



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 1941

#### Organics in Marine Sediment

Standard Reference Material (SRM) 1941 is intended for use in validating analytical methods for the determination of trace levels of selected polycyclic aromatic hydrocarbons (PAHs) and sulfur in marine sediments. Noncertified concentrations of additional PAHs, polychlorinated biphenyls (PCBs), chlorinated pesticides, and inorganic constituents are also provided. A unit of SRM 1941 contains approximately 70 g of sediment.

#### Certified Concentrations

Certified values for the concentrations of 11 PAHs naturally present in the sediment are provided in Table 1. The certified concentration of sulfur in SRM 1941 is shown in Table 2. The values in Table 1 are based on the results obtained from the analyses of this material using three different sample preparation procedures and analytical techniques based on gas chromatography with flame ionization detection, gas chromatography with mass spectrometric detection, and reversed-phase liquid chromatography with fluorescence detection. A summary of the analytical results for the PAHs obtained by using the different analytical techniques is provided in Appendix A. Noncertified concentrations for additional PAHs and for PCBs and pesticides are provided in Appendices B and C, respectively. Noncertified concentrations for trace element constituents are provided in Appendix D.

Table 1. Certified Concentrations of PAHs in SRM 1941

Compound	Concentration (ng/g dry weight) <sup>a,b</sup>
Phenanthrene	577 ± 59
Anthracene	202 ± 42
Fluoranthene	1220 ± 240
Pyrene	1080 ± 200
Benzo[a]anthracene	550 ± 79
Benzo[b]fluoranthene	780 ± 190
Benzo[k]fluoranthene	444 ± 49
Benzo[a]pyrene	670 ± 130
Perylene	422 ± 33
Benzo[ghi]perylene	516 ± 83
Indeno[1,2,3-cd]pyrene	569 ± 40

<sup>a</sup> Concentrations are reported on a dry weight basis; material, as received, contains residual moisture.

<sup>b</sup> The certified values are weighted means of results from two or more analytical techniques. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel [1]. Each uncertainty is obtained from a 95% prediction interval plus an allowance for systematic error among the methods used. The allowance for systematic error is equal to the greatest difference between the weighted mean (certified value) and the component means for the analytical 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 5 g.

Gaithersburg, MD 20899  
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(Revision of certificate dated 10-29-89)

William P. Reed, Acting Chief  
Standard Reference Materials Program

Table 2. Certified Concentration of Sulfur in SRM 1941

Sulfur

$1.717 \pm 0.027$  percent by weight<sup>a,b</sup>

<sup>a</sup> Concentration is reported on a dry weight basis; material as received contains residual moisture.

<sup>b</sup> The stated uncertainty is a 95% prediction interval for the concentration in a randomly chosen bottle of SRM 1941, plus an allowance for systematic error in the chemical analysis. Neglecting systematic error, the uncertainty limits will cover the concentration of approximately 95% of samples of this SRM having a minimum sample size of 100 mg.

## NOTICE AND WARNING TO USERS

**Handling:** This material consists of very fine particles ( $\leq 150 \mu\text{m}$ ) so transfer of the material should be performed with care. Use proper disposal methods.

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

**Storage:** The sediment is packaged in amber glass bottles and should be stored in the dark at temperatures lower than 30 °C.

**Use:** Aliquots of this SRM for analysis should be withdrawn from bottles immediately after opening and analyzed without delay for the certified values listed in Table 1 to be valid within the stated uncertainties. The bottles should be tightly closed immediately after removal of the subsamples to preserve the integrity of the remaining material for later analyses. The concentrations of constituents in SRM 1941 are reported on a dry weight basis. The SRM sediment as received contains approximately 4% moisture; therefore, a separate aliquot of the SRM should be removed from the bottle at the time of analysis and dried by either freeze drying or oven drying (see section on Moisture Determination for experimental procedure) to determine the correction factor to convert the sample weighed to the dry weight basis.

Collection and preparation of the SRM were performed in the NIST Center for Analytical Chemistry in the Organic Analytical Research Division by S. N. Chesler, M. M. Schantz, 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 K. E. Hehn, W. R. Kelly, S. F. Stone, and R. Zeisler.

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

The coordination of the technical measurements leading to certification was under the direction of M. M. Schantz, S. A. Wise, and W. E. May of the Organic Analytical Research Division.

The technical and 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 1941 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 sediment material used for SRM 1941 was collected with the assistance of Sandy Freitas of Battelle New England Research Laboratory, Duxbury, MA.

## PREPARATION AND ANALYSIS

**SRM Preparation.** The marine sediment used to prepare this SRM was collected in the Chesapeake Bay at the mouth of the Baltimore (MD) Harbor near the Francis Scott Key Bridge (39°12.85'N and 76°31.70'W). The sediment was air dried, pulverized, sieved ( $\leq 150 \mu\text{m}$  used for the SRM), homogenized in a cone blender, and subsampled (~70 g) into amber glass bottles with Teflon-lined screw caps. The material had been radiation-sterilized (<sup>60</sup>Co) at an estimated minimum dose of 3.2 megarads.

**Moisture Determination.** The results for both the organic and inorganic constituents in SRM 1941 are reported on a dry weight basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 1941 was determined by measuring the weight loss after freeze drying or oven drying. For the freeze drying studies, duplicate subsamples (~2-3 g) of the sediment from 16 different bottles of SRM 1941 were dried in five batches at different times. The samples were dried for 3-5 days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. For the oven drying studies, duplicate subsamples (~2 g) of the sediment from eight of the same bottles used in the freeze drying study were dried in two different batches in an oven at 90 °C for 18 h and the weight loss determined. Based on these studies, the water content in SRM 1941 at the time of the certification analyses was determined to be  $3.98 \pm 0.57\%$ . Analytical results for the organic and inorganic constituents were determined on an "as received" basis and then converted to a dry weight basis by multiplying by the correction factor for moisture content of 1.041.

**Polycyclic Aromatic Hydrocarbons.** The SRM was analyzed for selected PAHs using gas chromatography with flame ionization detection (GC-FID), gas chromatography with mass spectrometric detection (GC-MS), and liquid chromatography with fluorescence detection (LC-FL). A more detailed discussion of the analysis of SRM 1941 is reported elsewhere [2]. A total of three different sample preparation/cleanup procedures were used in connection with the analyses by GC-FID, GC-MS, or LC-FL.

For GC-FID analyses, 7- to 25-g subsamples of sediment from 12 randomly selected bottles were Soxhlet extracted for 16-20 h using methylene chloride. A silica solid phase extraction column was used to remove the polar interferences from the sediment extract. Finely divided copper was then added to the extract to remove elemental sulfur, which is present at relatively high levels in this marine sediment (see Table 2). The PAH fraction was isolated from the sediment extract by normal-phase liquid chromatography using a semi-preparative aminosilane column [3]. The GC-FID analyses were performed using a 0.25 mm x 60 m fused silica capillary column with a 5% phenyl substituted polysiloxane phase (0.25  $\mu$ m film thickness). A representative chromatogram from the GC-FID analysis of the PAH fraction is shown in Figure 1.

For the GC-MS analyses, approximately 10-g subsamples of sediment from four randomly selected bottles were Soxhlet extracted for 16-20 h using methylene chloride. The extract was filtered through a 0.45- $\mu$ m fluoropolymer filter prior to quantitative analysis by GC-MS using a column identical to that employed for the GC-FID analyses.

For the LC-FL analyses, approximately 11-g subsamples of sediment from three randomly selected bottles were Soxhlet extracted using hexane:acetone (1:1 v/v). An aminosilane solid phase extraction column was used to remove the more polar interferences from the sediment extract. The extracts were then analyzed by reversed-phase LC using a polymeric octadecylsilane (C<sub>18</sub>) column (4.6 mm i.d. x 25 cm, 5- $\mu$ m particle size) with wavelength programmed fluorescence detection [4-6]; these results are designated as LC-FL (Direct) in Appendix A. To quantify several PAHs that have low fluorescence sensitivity or that are subject to interferences from the matrix, a portion of the sediment extract was fractionated on a semi-preparative aminosilane column to isolate isomeric PAH fractions as described previously [4-6]. These isomeric PAH fractions were analyzed by reversed-phase LC-FL on the same octadecylsilane column; these results are designated as LC-FL (Fraction) in Appendix A.

The internal standards used for quantification purposes are listed in Appendix E. For the GC-FID method, two PAHs not significantly present in the sediment were utilized as internal standards. For the GC-MS and LC-FL methods, selected perdeuterated PAHs were utilized as internal standards. The internal standards were added to the sediment samples immediately prior to extraction. Calibration response factors for the analytes relative to the internal standards were determined by analyzing SRM 1491, "Aromatic Hydrocarbons in Hexane/Toluene," in the case of GC-FID and GC-MS analyses or SRM 1647a, "Polycyclic Aromatic Hydrocarbons in Acetonitrile," in the case of LC-FL analyses.

**PCBs and Chlorinated Pesticides.** The SRM was analyzed for selected PCBs and chlorinated pesticides using gas chromatography with electron capture detection (GC-ECD). Subsamples (approximately 11 g) of sediment from four bottles were Soxhlet extracted for 16-20 h using methylene chloride. A silica solid phase extraction column was used to remove the polar interferences from the sediment extract. Additional analytical interferences were removed from this extract on a semi-preparative aminosilane liquid chromatographic column from which the analytes of interest were collected in two fractions according to previously calibrated retention zones. GC-ECD

analyses were performed using the same type of capillary column as used for the PAH measurements. Representative chromatograms from the analyses of the PCB and 4,4'-DDE fraction and the more polar pesticide fraction are shown in Figures 2 and 3, respectively.

Individual PCB congeners and chlorinated pesticides not significantly present in the sediment extract, were added to the sediment prior to extraction for use as internal standards for quantification purposes (see Appendix E). Calibration response factors for the analytes relative to the internal standards were determined by fractionating and analyzing fractions of gravimetrically prepared calibration solutions of the analytes of interest and the internal standards.

**Sulfur Determination.** The general procedure for the determination of sulfur by isotope dilution thermal ionization mass spectrometry (ID-TIMS) has been described in detail previously [7,8]. Subsamples of approximately (0.5 g) each from six bottles of the SRM were allowed to equilibrate with laboratory air in a class 100 hood for 3.5 h. A 100 mg subsample was removed from each of the six different samples and added to Pyrex Carius tubes along with enriched  $^{34}\text{S}$  tracer and NIST high purity nitric acid. The remaining sample was then oven dried at 90 °C for 18 h to establish the dry weight independent of the moisture determination for the organic and other inorganic constituents. The Carius tubes were then sealed and heated at 240 °C for 16 h. This procedure oxidizes all sulfur to sulfate and completely mixes the spike, which is in the sulfate form, with the sulfur in the sample. The sulfate in the samples was reduced to  $\text{H}_2\text{S}$  which was then precipitated as  $\text{As}_2\text{S}_3$ . The  $\text{As}_2\text{S}_3$  was dissolved in aqueous ammonia and a small amount of this solution, equivalent to about  $1.5\ \mu\text{g S}$ , was mixed with silica gel on a rhenium filament and the sulfur isotopic ratios were determined as the  $\text{AsS}^+$  molecular ion.

**Additional Inorganic Constituents.** The SRM was analyzed for selected inorganic constituents using neutron activation analysis (NAA). The NAA followed previously developed procedures for sequential instrumental multi-element determinations in biological materials [9]. Three subsamples, each weighing approximately 300 mg, from each of two bottles of the sediment were pelletized and analyzed using the sequential procedure. Selected elements (B, Na, Al, Si, S, Cl, K, Ti, Mn, Fe, Cd, Sm, and Gd) were first determined using neutron capture prompt gamma ray activation analysis (PGAA). This step was followed by instrumental NAA measurement of the short-lived nuclides for the determination of Na, Al, Cl, Ti, V, and Mn. After decay, instrumental NAA was performed for the determination of Sc, Cr, Fe, Co, Zn, As, Se, Rb, Ag, Sb, Cs, La, Ce, Sm, Eu, Hf, Tb, Ta, Th, and U. In cases where an element was determined by both procedures (e.g., Na, Al, Cl, Ti, Mn, and Sm), the results with more precise counting statistics and/or less spectral interference are reported. The results of the inorganic analyses are summarized in Appendix D and are provided as noncertified values. A more detailed discussion of the analytical procedures used to obtain these results are being published [2,10].

## References

- [1] Paule, R.C.; and Mandel, J. Consensus Values and Weighting Factors, *J. Res. Nat. Bur. Stand. (U.S.)* 87, 377-385 (1982).
- [2] Schantz, M.M.; Benner, B.A. Jr.; Chesler, S.N.; Koster, B.J.; Hehn, K.E.; Stone, S.F.; Kelly, W.R.; Zeisler, R.; and Wise, S.A. Preparation and Analysis of a Marine Sediment Reference Material for the Determination of Trace Organic Constituents, submitted to *Fresenius Z. Anal. Chem.*, in press.
- [3] Wise, S.A.; Chesler, S.N.; Hertz, H.S.; Hilpert, L.R.; and May, W.E. Chemically-Bonded Aminosilane Stationary Phase for the High Performance Liquid Chromatographic Separation of Polynuclear Aromatic Hydrocarbons, *Anal. Chem.* 49, 2306-2310 (1977).
- [4] May, W.E.; and Wise, S.A. Liquid Chromatographic Determination of Polycyclic Aromatic Hydrocarbons in Air Particulate Extracts, *Anal. Chem.* 56, 225-232 (1984).
- [5] Kline, W.F.; Wise, S.A.; and May, W.E. The Application of Perdeuterated Polycyclic Aromatic Hydrocarbons (PAH) as Internal Standards for the Liquid Chromatographic Determination of PAH in Petroleum Crude Oil and Other Complex Mixtures, *J. Liquid Chromatogr.* 8, 223-237 (1985).
- [6] Wise, S.A.; Benner, B.A.; Byrd, G.D.; Chesler, S.N.; Rebbert, R.E.; and Schantz, M.M. Determination of Polycyclic Aromatic Hydrocarbons in a Coal Tar Standard Reference Material, *Anal. Chem.* 60, 887-894 (1988).

[7] Paulsen, P.J.; and Kelly, W.R. Determination of Sulfur as Arsenic Monosulfide Ion by Isotope Dilution Thermal Ionization Mass Spectrometry, *Anal. Chem.* **56**, 708-713 (1984).

[8] Kelly, W.R.; and Paulsen, P.J. Precise and Accurate Determination of High Concentrations of Sulfur by Isotope Dilution Thermal Ionization Mass Spectrometry, *Talanta* **31**, 1063-1068 (1984).

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

[10] Stone, S.F.; Koster, B.J.; Zeisler, R. Instrumental Neutron Activation Analysis of SRM 1941 "Organics in Marine Sediment": Elemental Content and Homogeneity, *Biol. Trace Elem. Res.*, in press.

## SUPPLEMENTAL INFORMATION

### Noncertified Quantitative Values

Appendices A through D contain supplementary analytical results obtained during the course of the certification of SRM 1941. Appendix A contains a summary of the analytical results for the determination of PAHs using the various analytical techniques. Noncertified concentration values are listed in Appendix B for additional PAHs, in Appendix C for 15 PCB congeners and seven chlorinated pesticides, and in Appendix D for 31 inorganic constituents. The values reported are the results obtained using only one analytical technique and may include unrecognized bias; therefore, they are provided for information only. The uncertainties given represent only the precision of the measurement process.

## APPENDIX A

### Summary of Analytical Results for the Determination of PAHs in SRM 1941

Compound	Concentration (ng/g dry weight) <sup>a</sup>			
	GC-FID	GC-MS	LC-FL (Direct)	LC-FL (Fraction)
Phenanthrene	597± 4 <sup>b</sup>	603± 10	531± 12	
Anthracene	202± 6	228± 12	174± 8	
Fluoranthene	1116± 20	1401± 41	1135± 10	
Pyrene	1008± 16	1238± 18	989± 34	
Benz[a]anthracene	538± 12	599± 14	516± 7	521± 11 <sup>d</sup>
Chrysene	577± 12 <sup>c</sup>	702± 16 <sup>c</sup>	425± 42	473± 5 <sup>d</sup>
Triphenylene				192± 3 <sup>d</sup>
Benzo[b]fluoranthene	635± 17	864± 28	839± 14	843
Benzo[j]fluoranthene	351± 14			
Benzo[k]fluoranthene	439± 19	857± 25 <sup>f</sup>	456± 6 <sup>e</sup> 441± 8 <sup>e</sup>	443± 16
Benzo[e]pyrene	472± 25	672± 24		
Benzo[a]pyrene	566± 12	754± 49	674± 12	690± 25
Perylene	415± 8	437± 27	411± 6	426± 5
Benzo[ghi]perylene	478± 14	566± 26		504± 7
Indeno[1,2,3-cd]pyrene	572± 28	559± 19	573± 20	575± 8

<sup>a</sup>Concentrations reported on dry weight basis; material, as received, contains residual moisture.

<sup>b</sup>Uncertainties for GC, LC, and GC-MS measurements are ± one standard deviation of a single measurement; For GC-FID measurements, 12 samples were analyzed in triplicate; for LC measurements, three samples were analyzed in triplicate; for GC-MS measurements, four samples were analyzed in duplicate.

<sup>c</sup>Value is the sum of the chrysene and triphenylene.

<sup>d</sup>Determined using triphenylene-d<sub>12</sub> as internal standard.

<sup>e</sup>Benzo[k]fluoranthene was determined at different times, i.e., during initial analyses of total PAH fraction and during benzo[b]fluoranthene analyses.

<sup>f</sup>Value is the sum of the benzo[k]fluoranthene and benzo[j]fluoranthene.

## APPENDIX B

### Noncertified Concentrations of PAHs in SRM 1941

Note: Although bias has not been evaluated for the procedures used, the noncertified concentrations should be useful for comparison with results obtained using similar procedures (i.e., solvent extraction and GC-MS on a similar column).

Compound <sup>a</sup>	Concentration (ng/g dry weight) <sup>b</sup>
Naphthalene	1322 ± 14
2-Methylnaphthalene	406 ± 36
1-Methylnaphthalene	229 ± 19
Biphenyl	115 ± 15
2,6-Dimethylnaphthalene	198 ± 23
Acenaphthylene	115 ± 10
Acenaphthene	52 ± 2
Fluorene	104 ± 5
3-Methylphenanthrene	150 ± 5
2-Methylphenanthrene	190 ± 6
2-Methylanthracene	66 ± 7
9-Methyl and 4-Methylphenanthrene <sup>c</sup>	145 ± 8
1-Methylphenanthrene	109 ± 6
2,6-Dimethylphenanthrene	68 ± 4
2,7-Dimethylphenanthrene	52 ± 4
1,3-, 2,10-, 3,9-, and 3,10-Dimethylphenanthrene <sup>c</sup>	161 ± 11
1,6- and 2,9-Dimethylphenanthrene <sup>c</sup>	93 ± 6
1,7-Dimethylphenanthrene	62 ± 4
2,3-Dimethylphenanthrene	36 ± 3
Benzo[a]fluoranthene	146 ± 4
Triphenylene <sup>d</sup>	192 ± 3
Chrysene <sup>d</sup>	449
Benzo[j]fluoranthene <sup>c</sup>	351 ± 14
Benzo[e]pyrene <sup>c</sup>	573

<sup>a</sup>Naphthalene through benzo[a]fluoranthene were determined using GC-MS; four sediment extracts were analyzed in duplicate; uncertainties are ± one standard deviation of a single measurement.

<sup>b</sup>Concentrations are reported on dry weight basis; material, as received, contains residual moisture.

<sup>c</sup>Concentration is the sum of two or more compounds.

<sup>d</sup>Triphenylene and chrysene were determined by LC-fluorescence; value for chrysene is the mean value of results obtained by the two LC-fluorescence procedures (see Appendix A).

<sup>e</sup>Benzo[j]fluoranthene and benzo[e]pyrene were determined by GC-FID; value for benzo[e]pyrene is the mean value of the results obtained by GC-FID and GC-MS (see Appendix A).

## APPENDIX C

### Noncertified Concentrations of Selected PCB Congeners and Chlorinated Pesticides in SRM 1941 as Determined by GC-ECD

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

Polychlorinated Biphenyls <sup>a</sup>		Concentration (ng/g dry weight) <sup>b</sup>
PCB 18	(2,2',5-Trichlorobiphenyl)	9.90 ± 0.25
15	(4,4'-Dichlorobiphenyl)	
PCB 28	(2,4,4'-Trichlorobiphenyl)	16.1 ± 0.4
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)	10.4 ± 0.4
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)	22.4 ± 0.7
95	(2,2',3,5',6-Pentachlorobiphenyl)	
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)	22.0 ± 0.7
90	(2,2',3,4',5-Pentachlorobiphenyl)	
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)	5.76 ± 0.23
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)	15.2 ± 0.7
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	24.9 ± 1.8
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)	22.0 ± 1.4
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	7.29 ± 0.26
190	(2,3,3',4,4',5,6-Heptachlorobiphenyl)	
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	14.3 ± 0.3
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)	12.5 ± 0.6
159	(2,3,3',4,5,5'-Hexachlorobiphenyl)	
182	(2,2',3',4,4',5,6'-Heptachlorobiphenyl)	
PCB 195	(2,2',3,3',4,4',5,6'-Octachlorobiphenyl)	1.51 ± 0.10
208	(2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl)	
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	4.81 ± 0.15
PCB 209	(Decachlorobiphenyl)	8.35 ± 0.21
Chlorinated Pesticides		
Heptachlor Epoxide		0.23 ± 0.02
cis-Chlordane (alpha-Chlordane)		2.06 ± 0.05
trans-Nonachlor		0.97 ± 0.03
Dieldrin		0.63 ± 0.03
4,4'-DDE		9.71 ± 0.17
4,4'-DDD		10.3 ± 0.1
4,4'-DDT		1.11 ± 0.05

<sup>a</sup>PCBs are numbered according to K. Ballschmiter and M. Zell, *Fresenius Z. Anal. Chem.* 302, 20 (1980).

<sup>b</sup>Concentrations are reported on dry weight basis; material, as received, contains residual moisture. Four extracts were analyzed in triplicate; uncertainties are ± one standard deviation of a single measurement.

## APPENDIX D

### Noncertified Concentrations of Inorganic Constituents in SRM 1941

Note: These noncertified values were obtained using instrumental neutron activation 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 as required to arrive at a certified value; therefore, unrecognized bias may exist for the determination of some analytes in this matrix.

Element	Concentration ( $\mu\text{g/g}$ dry weight) <sup>a,b</sup>		Element	Concentration ( $\mu\text{g/g}$ dry weight) <sup>a,b</sup>	
B <sup>c</sup>	76	± 2	Se	10.1	± 0.5
Na (%)	1.29	± 0.03	Rb	92	± 1
Al (%)	6.5	± 0.2	Ag	1.2	± 0.5
Si <sup>c</sup> (%)	22.2	± 0.8	Cd <sup>c</sup>	2.3	± 0.3
			Sb	15.2	± 0.4
Cl (%)	1.64	± 0.04	Cs	4.8	± 0.1
K <sup>c</sup> (%)	1.58	± 0.01	La	360	± 10
Sc	34.4	± 0.4	Ce	272	± 4
Ti <sup>c</sup> (%)	1.72	± 0.03	Sm <sup>c</sup>	25.7	± 0.4
V	810	± 30	Eu	2.19	± 0.06
Cr	640	± 10	Tb	2.2	± 0.6
Mn	790	± 10	Gd <sup>c</sup>	15.2	± 0.4
Fe (%)	10.6	± 0.1	Hf	22.4	± 0.3
Co	27.5	± 0.1	Ta	16.4	± 0.5
Zn	1010	± 40	Th	25.6	± 0.3
As	75	± 4	U <sup>d</sup>	22	± 1

<sup>a</sup>Results are reported on a dry weight basis in  $\mu\text{g/g}$ , except where noted in percent; material, as received, contains residual moisture; minimum sample size of 250 mg.

<sup>b</sup>Uncertainties are  $t_{s/\sqrt{n}}$  at the 95% confidence level.

<sup>c</sup>Results were determined by PGAA.

<sup>d</sup>Uranium concentration was obtained by measurement of fission products.



## Appendix E

### Internal Standards Used in the Analysis of SRM 1941 for the Determination of Organic Constituents

Technique <sup>a</sup>	Internal Standard	Analytes Determined
GC-FID	1-Butylpyrene	Phenanthrene, Anthracene, Fluoranthene, Pyrene, Benz[a]anthracene, Chrysene, and Benzo[fluoranthene] isomers
	m-Tetraphenyl	Benzo[e]pyrene, Benzo[a]pyrene, Perylene, Benzo[ghi]perylene, and Indeno[1,2,3-cd]pyrene
GC-ECD	PCB 103	PCB 18, 28, 52, 66, and 101
	PCB 198	PCB 105, 118, 138, 153, 180, 170, 187, 195, 206, 209, and 4,4'-DDE
	Endrin	Heptachlor Epoxide, cis-Chlordane, trans-Nonachlor, Dieldrin, 4,4'-DDD, and 4,4'-DDT
LC-FL (Total)	Phenanthrene-d <sub>10</sub>	Phenanthrene and Anthracene
	Fluoranthene-d <sub>10</sub>	Fluoranthene, Pyrene, Benz[a]anthracene, and Chrysene
	Perylene-d <sub>12</sub>	Perylene, Benzo[k]fluoranthene, Benzo[a]pyrene, Benzo[ghi]perylene, and Indeno[1,2,3-cd]pyrene
LC-FL (Fraction)	Triphenylene-d <sub>12</sub>	Triphenylene, Benz[a]anthracene, and Chrysene
	Perylene-d <sub>12</sub>	Perylene, Benzo[k]fluoranthene, and Benzo[a]pyrene
	Benzo[ghi]perylene-d <sub>12</sub>	Benzo[ghi]perylene and Indeno[1,2,3-cd]pyrene
GC-MS (Method A)	Naphthalene-d <sub>8</sub>	Naphthalene, Methyl/Dimethylnaphthalenes, and Biphenyl
	Acenaphthene-d <sub>10</sub>	Acenaphthylene and Acenaphthene
	Phenanthrene-d <sub>10</sub>	Fluorene
GC-MS (Method B)	Phenanthrene-d <sub>10</sub>	Phenanthrene, Anthracene, and Methyl/Dimethyl Phenanthrenes
	Pyrene-d <sub>10</sub>	Fluoranthene and Pyrene
	Benz[a]anthracene-d <sub>12</sub>	Benz[a]anthracene and Chrysene/Triphenylene
	Benzo[e]pyrene-d <sub>12</sub>	Benzo[fluoranthene] isomers, Benzo[e]pyrene, Benzo[a]pyrene, and Perylene
	Benzo[ghi]perylene-d <sub>12</sub>	Benzo[ghi]perylene and Indeno[1,2,3-cd]pyrene

<sup>a</sup> See main text for description of the techniques used for analysis.

Note: The following figures are provided as descriptive information only and should not be used as a reference for quantitative determination.

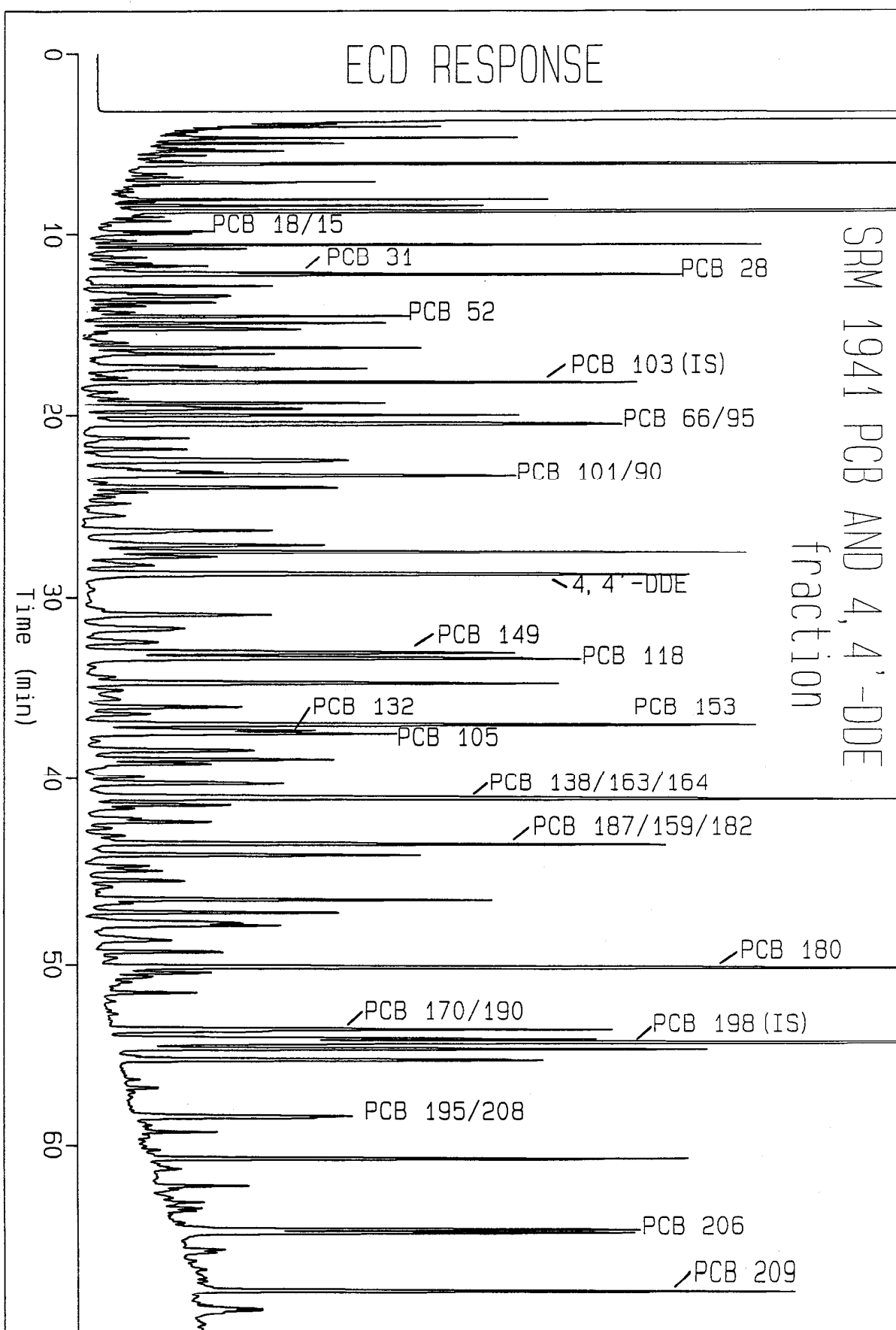
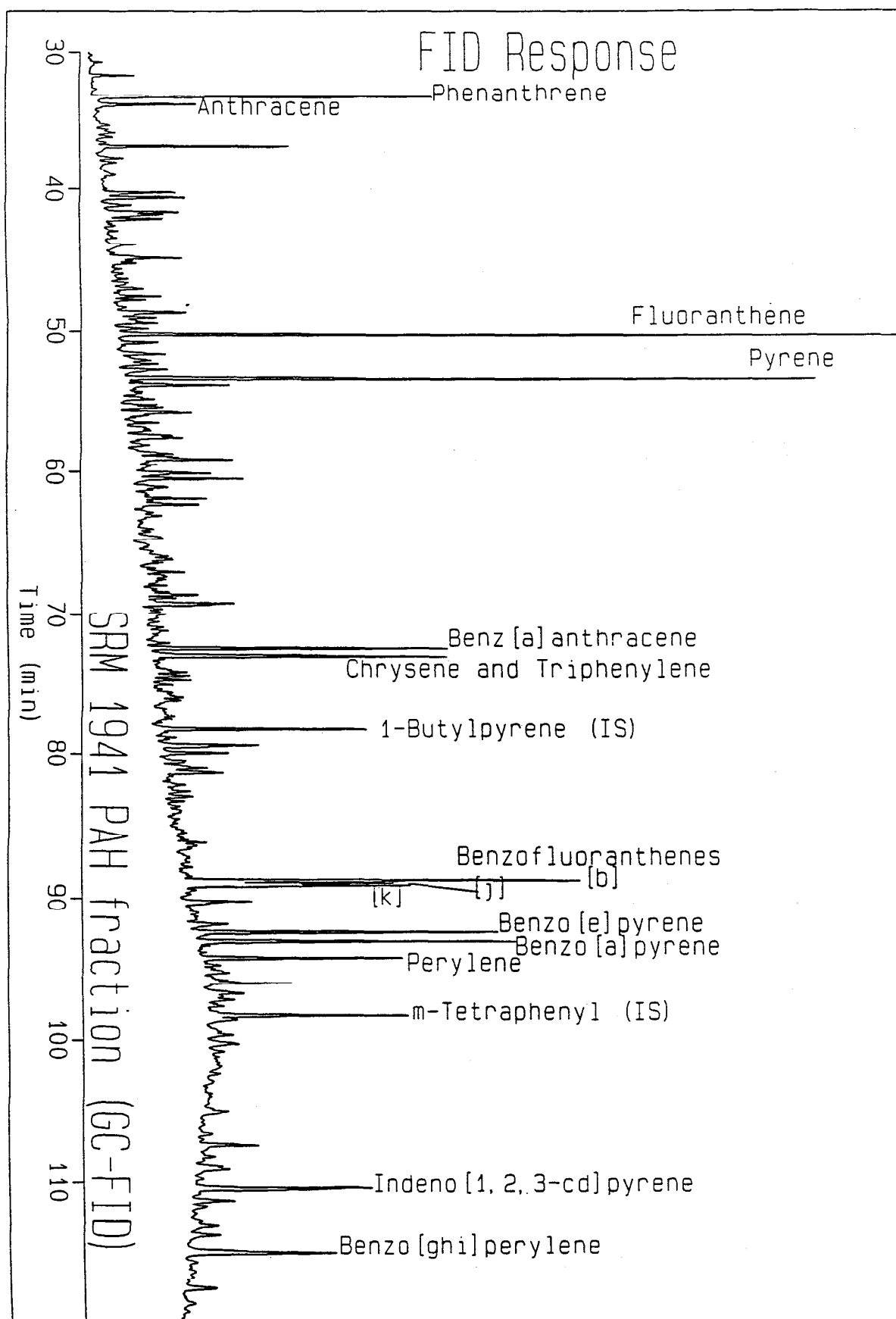


Figure 2. GC-ECD analysis of PCB and 4,4'-DDE fraction isolated from SRM 1941.

Figure 1. GC-FID analysis of the PAH fraction isolated from SRM 1941.



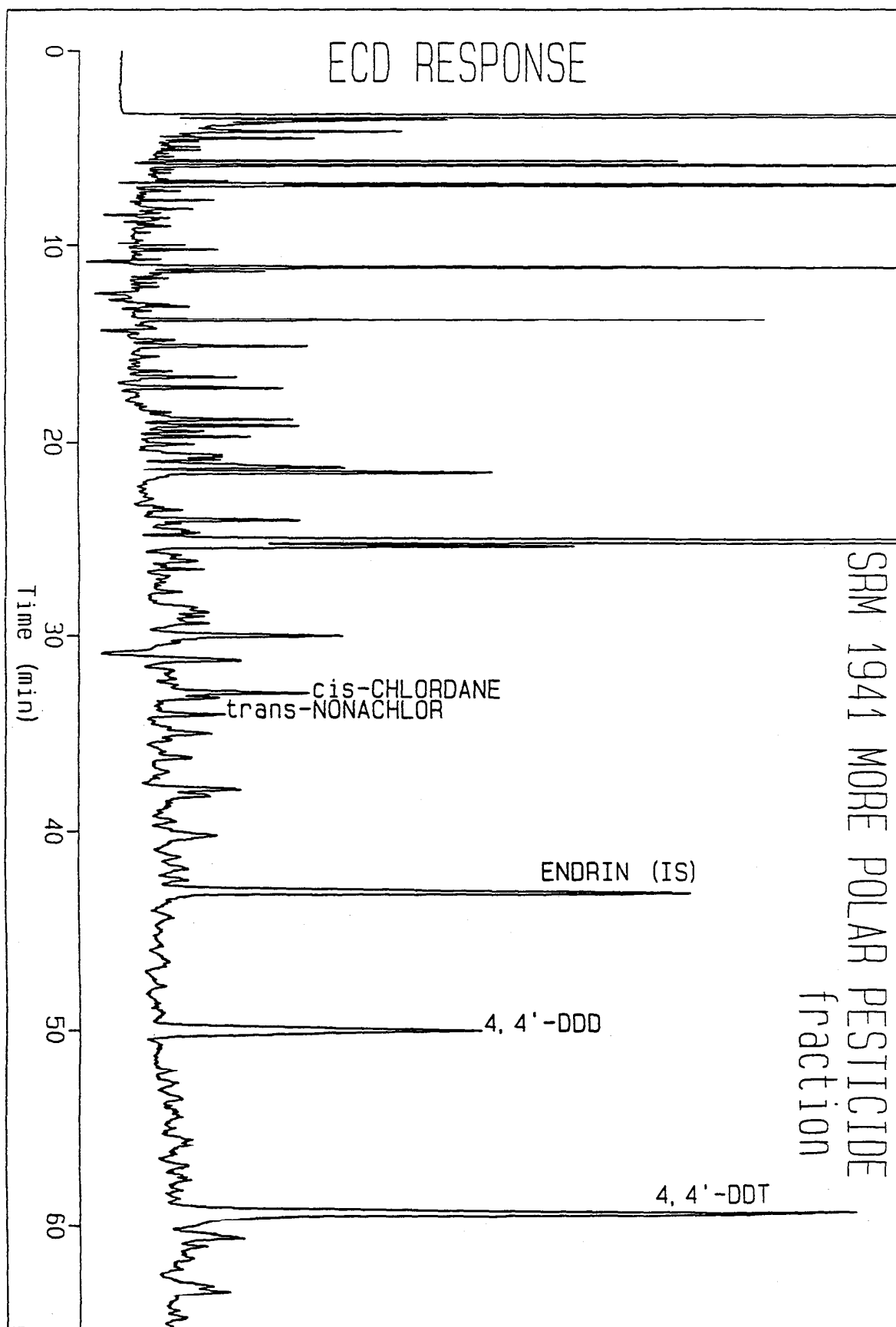


Figure 3. GC-ECD analysis of more polar pesticide fraction isolated from SRM 1941.