Determination of Polychlorinated Biphenyls (PCBs) in Soils and Solid Waste by Accelerated Solvent Extraction and GC-MS/MS

Fabrizio Galbiati, Thermo Fisher Scientific (Schweiz) AG, Reinach, Switzerland
Luca Teli, Consulenze Ambientali SpA, Scanzorosciate, Italy

Key Words
Pressurized Fluid Extraction, U.S. EPA Method 8082A, PCB, Inline Clean-Up, Rocket Evaporator, TSQ 8000 Triple Quadrupole GC-MS, Xcalibur, TraceFinder

Goal
To demonstrate an accelerated solvent extraction and GC-MS/MS procedure for polychlorinated biphenyls (PCB) in soils and solid waste.

Introduction
Polychlorinated biphenyls (PCBs) belong to a broad family of synthetic organic chemicals known as chlorinated hydrocarbons. In the United States, PCBs were produced from 1929 until their manufacture was banned in 1979. These compounds have a range of toxicity and vary in consistency from thin, light-colored liquids to yellow or black waxy solids. Due to their non-flammability, chemical stability, high boiling point, and electrical insulating properties, PCBs were used in hundreds of industrial and commercial applications including electrical, heat transfer, and hydraulic equipment; as plasticizers in paints, plastics, and rubber products; in pigments, dyes, and carbonless copy paper; and in many other industrial applications.

Techniques such as Soxhlet (U.S. EPA Method 3540), sonication (U.S. EPA Method 3550), and microwave extraction (U.S. EPA Method 3546) are presently used for the extraction of PCBs from soil prior to their analytical determination. Those techniques are, however, very labor intensive and suffer from high solvent consumption. Accelerated solvent extraction was developed to meet the new requirements for reducing solvent usage in the preparation of solid samples. With accelerated solvent extraction, extractions can be completed in very short periods of time and with minimal amounts of solvent compared to conventional sample extraction techniques such as Soxhlet and sonication. Furthermore, interferences may be extracted along with desired analytes during those conventional extraction processes. These unwanted co-extractables may interfere with analyte detection and need to be eliminated. For example, according to U.S. EPA Method 3660B, the removal of elemental sulfur from soils, sediments, and industrial wastes requires a post-extraction cleanup with copper powder

Figure 1. Thermo Scientific Dionex ASE 350 Accelerated Solvent Extractor and Thermo Scientific Rocket Evaporator.

or tetrabutylammonium sulfite. In U.S. EPA Method 3665A, the removal of the interfering organic compounds is instead achieved with a sequential treatment with concentrated sulfuric acid and 5% acqueous potassium permanganate. Recent advances using accelerated solvent extraction systems, as described in several publications,[1–20] include procedures for selective removal of interferences during sample extraction, thus combining extraction and purification into a single step.

Although all 209 of the PCB congeners can be synthesized, only about 130 individual congeners have been identified in commercial PCB mixtures at concentrations ≥ 0.05%. The method reported here is applicable for the determination of 32 of the 209 possible PCB congeners in soils, including the dioxin-like PCB ("non-*ortho*", "mono-*ortho* PCB", and "di-*ortho* PCB") with concentrations between 5 and 200 µg/kg expressed on a dry matter basis. Following U.S. EPA Method 8082A, tetrachloro-*m*-xylene was used as a surrogate and decachlorobiphenyl as an internal standard.

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Experimental

Equipment

An ED53 oven from Binder™ was used for drying the samples. A Sartorius™ analytical balance was used for weighing the dry soil samples and standards. A Fritsch Pulverisette™ planetary ball mill was used to grind the samples. The extractions were carried out with a Thermo Scientific™ Dionex™ ASE 350™ Accelerated Solvent Extractor (P/N 083114 (120 V) or 083146 (240 V)) equipped with 34 mL stainless steel extraction cells (P/N 068089). The extracts were collected in 60 mL double-ended Thermo Scientific™ Rocket™ Flip-Flop™ tubes (P/N 076360) and directly concentrated in a 2 mL autosampler glass vial (Thermo Scientific™ Chromacol™ VAGK ISP: GC 2-SVW + 9-SCK(B)-ST1) with the Thermo Scientific Rocket Evaporator (P/N 075904 (120 V) or 082766 (240 V)). The samples were analyzed with a Thermo Scientific™ TRACE™ 1310 Gas Chromatograph equipped with a Split/Splitless Injector, Thermo Scientific™ TraceGOLD™ TG-17MS GC Columns (30 m × 0.25 mm × 0.25 µm, P/N 26089-1420) and a Thermo Scientific™ TSQ™ 8000 Triple Quadrupole GC-MS/MS (Figure 2).

Figure 2. Thermo Scientific TSQ 8000 Triple Quadrupole GC-MS/MS and Thermo Scientific TRACE 1310 Gas Chromatograph.

Solvents and Standards

Hexane, Pesticide Grade, (Fluka® P/N 34484) was the extraction solvent for ASE. Toluene, Pesticide Grade, (Fluka P/N 34494) was used as a keeper to carefully control the final volume during concentration. Diatomaceous earth (DE) was Thermo Scientific™ Dionex™ ASE™ Prep DE (P/N 062819). Silica gel (70–230 mesh for column chromatography, P/N 453337) was purchased from Carlo Erba Reagents. PCB calibration mixture at a concentration of 10 µg/mL in isooctane (P/N RPCM-240), decachlorobiphenyl (internal standard, P/N PPS-150) at a concentration of 1000 µg/mL in toluene and tetrachloro-*m*-xilene (surrogate, P/N IST-440) at a concentration of 2000 µg/mL in acetone were purchased from ULTRA Scientific. *n*-Octadecanoic acid octadecyl ester (P/N 46408) and anhydrous sodium sulfate (P/N 71962) were purchased from Fluka.

The internal standard solution was prepared by adding 0.1 mL of decachlorobiphenyl to one liter of hexane. Surrogate solution was prepared by adding 50 µL of tetrachloro-*m*-xilene to one liter of hexane. Both solutions had a final concentration of 0.10 mg/L.

PCB standard solutions with concentrations of 0.20, 0.10, 0.05, 0.01, 0.005, and 0.001 mg/L were prepared by diluting the stock solution. The internal standard solution was added to each and the standards were brought to the final volume of 10 mL with hexane.

The silica gel was activated by heating in a muffle furnace at 550 °C for at least 4 h. The test solution of *n*-octadecanoic acid octadecyl ester ($C_{36}H_{72}O_2$) was prepared by dissolving 100 mg of *n*-octadecanoic acid octadecyl ester in 100 mL n-hexane. The clean-up efficiency of each batch of silica gel was checked by adding 10 mL of the test solution to a clean-up column filled with 2.0 g of silica gel and 2 g of sodium sulfate. After chromatographic analysis of the eluate the percent recovery of the *n*-octadecanoic acid octadecyl ester should not exceed 5%.

Extraction, Concentration, and Measurement

A Thermo Scientific™ Dionex™ cellulose filter (P/N 056780) was placed in the bottom of a 34 mL extraction cell, followed by 5 g of activated silica gel and another cellulose filter (Figure 3). One gram of dried and sieved sample was mixed in a glass beaker with a sufficient amount of ASE Prep DE and the resulting mixture carefully poured into the extraction cell, followed by 1 mL of the surrogate solution. Any empty volume was filled with ASE Prep DE. The accelerated solvent extractor was programmed according to the conditions reported below.

Conditions for Accelerated Solvent Extraction

Solvent	*n*-Hexane
Temperature	130 °C
Static Cycles	2
Static Extraction Time	9 min
Rinse Volume	50%
Purge Time	90 s
Total Extraction Time per Sample	~30 min
Total Solvent Volume per Sample	~40 mL

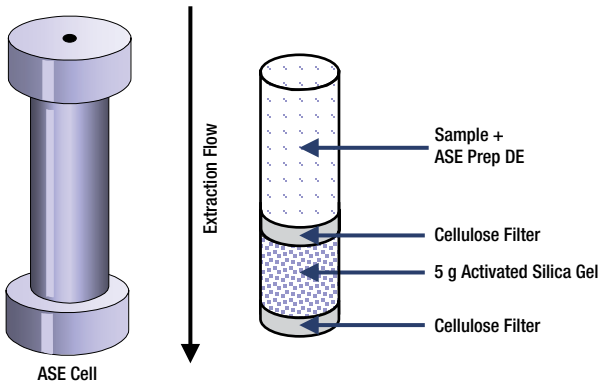

Figure 3. Extraction cell schematic.

The extracts were directly collected into Flip-Flop vials. After the addition of 1.5 mL of toluene and 1 mL of internal standard, they were concentrated on the Rocket Evaporator until the final volume of ~1 mL.

The GC and MS conditions are summarized below.

The chosen selected reaction monitoring (SRM) transitions are given in Table 1.

GC and Injector Conditions	
Split/Splitless Injector	
Injector Temperature	250 °C
Liner	SSL Splitless Liner, Deactivated, 4 mm × 6.3 mm × 78.5 mm, P/N 453A1925
Injected Volume	2 µL
Splitless Time	1.0 min
Split Flow	50 mL/min
Surge Pressure	5.0 kPa
GC Program	
GC Column	TraceGOLD TG-17MS (30 m × 0.25 mm × 0.25 µm)
Carrier Gas	Helium
Flow Rate	1.0 mL/min, constant
Initial Temperature	130 °C for 2 min
	10 °C/min to 220 °C (16 min)
	5 °C/min to 235 °C (8 min)
	25 °C/min to 330 °C (5 min)
Final Temperature	330 °C for 5 min

Mass Spectrometer Parameters	
Source Temperature	280 °C
Ionization	EI
Electron Energy	70 eV
Emission Current	50 µA
Q2 Gas Pressure (Argon)	1.5 mTorr
Collision Energy	26 to 28 eV
Q1 Peak Width FWHM	0.61 Da
Q3 Peak Width FWHM	0.59 Da

Table 1. SRM transitions. The asterisk indicates the "dioxin-like" PCBs. The quantifier ion *m/z* is shown in bold.

PCB #	Compound Name	Molecular Formula	Retention Time	Nominal Mass	Exact Mass	Precursor Ion (*m/z*)	Product Ion (*m/z*)	Collision Energy (eV)
18	2,2',5-Trichlorobiphenyl	$C_{12}H_7Cl_3$	11.21	258	255.9613	256.0	**186.0**	26
						258.0	186.0	26
28	2,4,4'-Trichlorobiphenyl	$C_{12}H_7Cl_3$	12.30	258	255.9613	256.0	**186.0**	26
						258.0	186.0	26
31	2,4',5-Trichlorobiphenyl	$C_{12}H_7Cl_3$	12.30	258	255.9613	256.0	**186.0**	26
						258.0	186.0	26
52	2,2',5,5'-Tetrachlorobiphenyl	$C_{12}H_6Cl_4$	13.19	292	291.9194	289.9	**220.0**	26
						291.9	222.0	26
44	2,2',3,5'-Tetrachlorobiphenyl	$C_{12}H_6Cl_4$	14.22	292	291.9194	289.9	**220.0**	26
						291.9	222.0	26
95	2,2',3,5',6-Pentachlorobiphenyl	$C_{12}H_5Cl_5$	15.91	326	325.8804	323.9	**253.9**	28
						325.9	255.9	26
101	2,2',4,5,5'-Pentachlorobiphenyl	$C_{12}H_5Cl_5$	16.40	326	325.8804	323.9	**253.9**	28
						325.9	255.9	26
99	2,2',4,4',5-Pentachlorobiphenyl	$C_{12}H_5Cl_5$	16.70	326	325.8804	323.9	**253.9**	28
						325.9	255.9	26
81	3,4,4',5-Tetrachlorobiphenyl	$C_{12}H_6Cl_4$	18.71	292	291.9194	289.9	**220.0**	26
						291.9	222.0	26
151	2,2',3,5,5',6-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	19.31	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
110	2,3,3',4',6-Pentachlorobiphenyl	$C_{12}H_5Cl_5$	19.35	326	325.8804	323.9	**253.9**	28
						325.9	255.9	26
77*	3,3',4,4'-Tetrachlorobiphenyl	$C_{12}H_6Cl_4$	19.63	292	291.9194	289.9	**220.0**	26
						291.9	222.0	26
123*	2,3',4,4',5'-Pentachlorobiphenyl	$C_{12}H_5Cl_5$	20.35	326	325.8804	323.9	**253.9**	28
						325.9	255.9	26
149	2,2',3,4',5',6-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	20.50	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
118*	2,3',4,4',5-Pentachlorobiphenyl	$C_{12}H_5Cl_5$	20.65	326	325.8804	323.9	**253.9**	28
						325.9	255.9	26

Table 1 (Continued). SRM transitions. The asterisk indicates the "dioxin-like" PCBs. The quantifier ion *m/z* is shown in bold.

PCB #	Compound Name	Molecular Formula	Retention Time	Nominal Mass	Exact Mass	Precursor Ion (*m/z*)	Product Ion (*m/z*)	Collision Energy (eV)
146	2,2',3,4',5,5'-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	21.07	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
153	2,2',4,4',5,5'-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	21.65	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
114*	2,3,4,4',5-Pentachlorobiphenyl	$C_{12}H_5Cl_5$	22.07	326	325.8804	323.9	**253.9**	28
						325.9	255.9	26
105*	2,3,3',4,4'-Pentachlorobiphenyl	$C_{12}H_5Cl_5$	24.17	326	325.8804	323.9	**253.9**	28
						325.9	255.9	26
138	2,2',3,4,4',5'-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	25.65	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
187	2,2',3,4',5,5',6-Heptachlorobiphenyl	$C_{12}H_3Cl_7$	25.98	395	393.8025	393.8	**323.9**	28
						395.8	325.9	28
183	2,2',3,4,4',5',6-Heptachlorobiphenyl	$C_{12}H_3Cl_7$	26.68	395	393.8025	393.8	**323.9**	28
						395.8	325.9	28
126*	3,3',4,4',5-Pentachlorobiphenyl	$C_{12}H_5Cl_5$	26.94	326	325.8804	323.9	**253.9**	28
						325.9	255.9	26
167*	2,3',4,4',5,5'-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	27.77	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
128	2,2',3,3',4,4'-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	29.95	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
177	2,2',3,3',4,5',6'-Heptachlorobiphenyl	$C_{12}H_3Cl_7$	30.42	395	393.8025	393.8	**323.9**	28
						395.8	325.9	28
156*	2,3,3',4,4',5-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	31.00	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
180*	2,2',3,4,4',5,5'-Heptachlorobiphenyl	$C_{12}H_3Cl_7$	31.50	395	393.8025	393.8	**323.9**	28
						395.8	325.9	28
157*	2,3,3',4,4',5'-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	31.78	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
169*	3,3',4,4',5,5'-Hexachlorobiphenyl	$C_{12}H_4Cl_6$	34.35	361	359.8415	357.8	**287.9**	28
						359.8	289.9	28
170*	2,2',3,3',4,4',5-Heptachlorobiphenyl	$C_{12}H_3Cl_7$	36.22	395	393.8025	393.8	**323.9**	28
						395.8	325.9	28
189*	2,3,3',4,4',5,5'-Heptachlorobiphenyl	$C_{12}H_3Cl_7$	38.63	395	393.8025	393.8	**323.9**	28
						395.8	325.9	28

Results and Discussion

Soils are an extremely complex matrix to analyze, particularly when they originate from contaminated sites. The nature and composition of the soil has a significant impact on the extraction efficiency and method sensitivity. The typical soil consists of approximately 45% mineral, 5% organic matter, 20–30% water, and 20–30% air. In particular, the organic matter contains high molecular weight, non-volatile material that can remain in the GC system and result in poor analytical performance. Many analysts use extensive sample preparation techniques to extract and concentrate the compounds of interest from this unwanted nonvolatile material. However these extraction and concentration techniques can become time consuming and costly.

According to U.S. EPA Method 8082A, the solid sample is extracted with a hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Soxhlet (U.S. EPA Method 3540), automated Soxhlet (U.S. EPA Method 3541), microwave (U.S. EPA Method 3546), ultrasonic extraction (U.S. EPA Method 3550), or supercritical fluid extraction (U.S. EPA Method 3562). The resulting extracts may be subjected to a sequential sulfuric acid/potassium permanganate cleanup to remove the organic matter (U.S. EPA Method 3665) and/or copper powder or tetrabutylammonium sulfite to remove elemental sulfur (U.S. EPA Method 3660). After cleanup, the extract is analyzed by injecting a measured aliquot into a gas chromatograph equipped with either a narrow- or wide-bore fused-silica capillary column and an electron capture detector (GC/ECD). U.S. EPA Method 8082A also mentions pressurized fluid extraction (U.S. EPA Method 3545A) as an alternative extraction procedure; however, it is still followed by a manual extract cleanup. Several recent publications describe procedures for selective removal of interferences during accelerated solvent extraction, thus combining extraction and purification into a single step.[1–20]

PCBs were manufactured as a mixture of various PCB congeners through progressive chlorination of batches of biphenyl until a certain target percentage of chlorine by weight was achieved. The most common commercial mixtures were the Aroclors and each had a distinguishing suffix number that indicated the degree of chlorination: the first two digits referred to the number of carbon atoms in the phenyl rings (for PCBs this was 12), the second two

numbers indicated the percentage of chlorine by mass in the mixture. The quantitation of PCBs as Aroclors is particularly difficult when samples contain more than one Aroclor and when the Aroclors have been weathered by long exposure in the environment or degradation by treatment technologies. This Customer Application Note provides a procedure for the determination of 32 of the 209 possible PCB congeners, including the compounds with a Toxic Equivalency Factor (TEF) between 0.1 and 0.00001 that are classified as "dioxin-like". The 32 PCB congeners were also chosen because many of them represent congeners specific to the common Aroclor formulations.

The test extractions used spiked soils that were previously heated for 4 h at 800 °C in order to completely remove the organic matter. The PCB spike levels were 5, 50, and 200 µg/kg. Twenty samples were extracted for each concentration level. The results showed that spike recovery rates were between 95.0 and 129.6% for the spike at 5 µg/kg, between 82.0 and 107.2% for the spike at 50 µg/kg, and between 87.9 and 112.4% for the spike at 200 µg/kg. These RSD values were compliant with the in-house QC performance criteria. U.S. EPA Method 8082A reports single-laboratory QC data for PCBs extracted by pressurized fluid extraction (U.S. EPA Method 3545) from sewage sludge, a river sediment standard reference material (SRM 1939), and a certified soil reference material (CRM911-050). Certified values are available for five PCB congeners for the sewage sludge and for four congeners in SRM 1939. The soil reference material was certified for Aroclor 1254. All the data in U.S. EPA Method 8082A are provided for guidance purposes only.

The average recovery rates and RSD for the 5, 50, and 200 µg/kg spike levels are reported in Table 2 and Figure 4. The asterisk indicates the "dioxin-like" PCBs: non-*ortho* (77, 126, 169), mono-*ortho* (105, 114, 118, 123, 156, 157, 167, 189) and di-*ortho* (170, 180). Typical SRM chromatograms for a 5 µg/kg spiked sample are shown in Figure 5. Trichlorobiphenyls (18, 28, and 31) are not shown.

Table 2. Average recovery and RSD for the 5, 50, and 200 µg/kg spike levels. The asterisk indicates the "dioxin-like" PCBs.

PCB	Average Recovery %	RSD %	Average Recovery %	RSD %	Average Recovery %	RSD %
	5 µg/kg		50 µg/kg		200 µg/kg	
18	95	14.3	82	13.5	88	18.3
28+31	107	13.6	93	13.5	98	17.9
52	111	14.3	96	13.2	107	18.4
44	118	13.8	100	13.3	108	17.6
95	109	15.3	93	14.5	105	20.4
101	110	12.8	96	13.6	102	19.0
99	105	12.5	94	13.3	99	18.2
81	121	13.0	105	12.8	112	16.2
151	113	11.2	100	12.3	101	17.2
110	106	12.7	91	13.3	99	18.3
77*	124	12.8	105	13.3	107	15.7
123*	119	14.4	106	13.6	112	18.8
149	124	15.0	104	13.0	103	18.0
118*	120	15.2	101	12.5	103	17.7
146	117	13.3	103	12.4	101	16.9
153	129	19.7	105	11.1	102	16.1
114*	115	13.1	101	13.0	100	15.5
105*	114	15.2	97	12.0	100	16.5
138	121	17.1	97	12.1	96	16.4
187	128	17.4	106	11.0	99	14.0
183	128	13.8	107	11.0	101	14.4
126*	117	17.3	102	13.2	104	18.5
167*	108	13.8	96	11.5	96	15.6
128	107	13.0	93	11.4	92	15.6
177	123	13.4	103	10.4	95	13.3
156*	112	13.8	97	11.3	94	15.1
180*	130	18.9	102	10.0	94	13.3
157*	104	13.9	92	11.4	90	15.5
169*	111	14.5	98	11.3	97	16.9
170*	128	16.2	105	10.4	99	13.4
189*	125	13.6	107	10.3	98	12.8

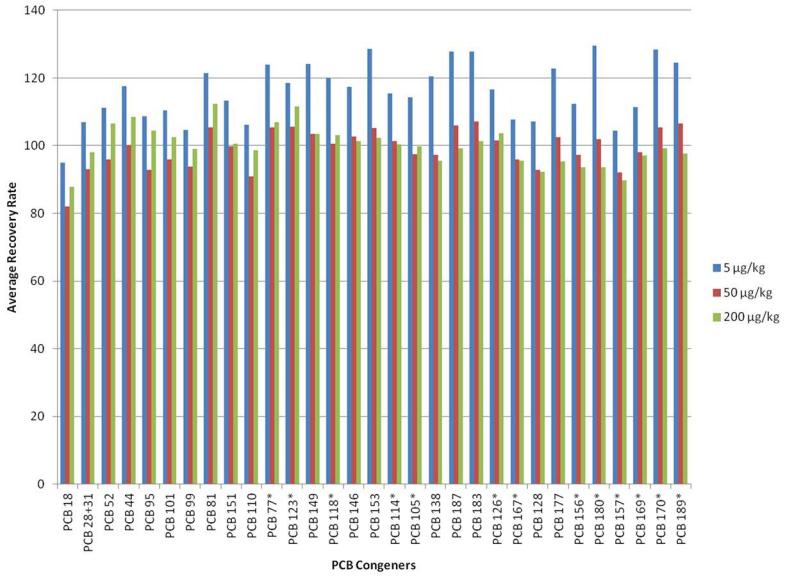

Figure 4. Average recovery rates for the 5, 50, and 200 µg/kg spike levels.

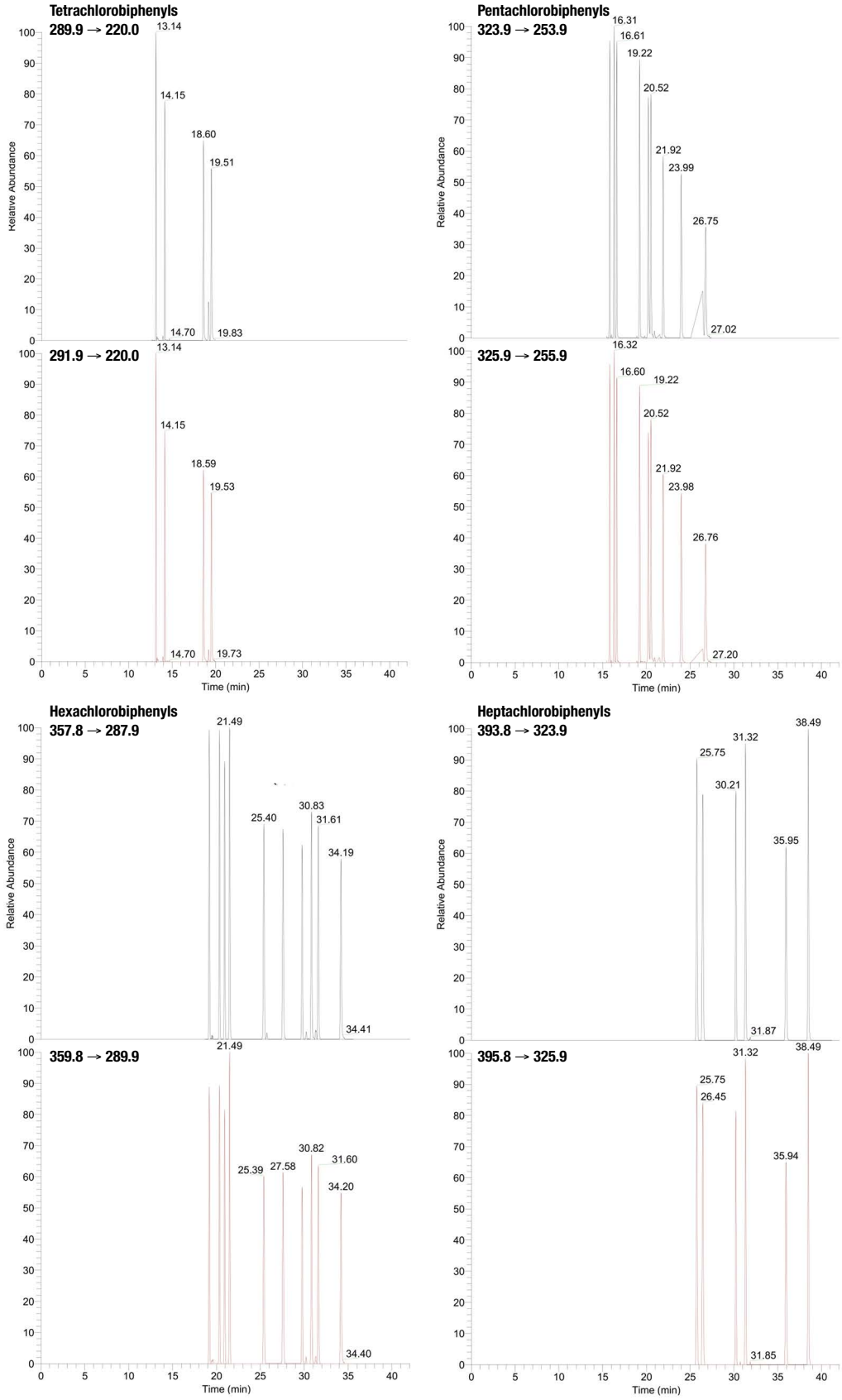

Figure 5. SRM chromatograms for a 5 µg/kg PCB-spiked soil sample after ASE-extraction and concentration.

Two typical chromatograms of a contaminated residential soil sample are shown in Figures 6 and 7. The samples were processed according to the procedure described in the Extraction, Concentration, and Measurement section. Quantitative SRM ions were clearly detected along with the confirmatory SRM ions for all the PCBs tested within the QC ion ratio criteria. With a total concentration of 1.18 mg/kg (Figure 5) and 0.43 mg/kg (Figure 6), both soil samples were over the limits fixed by the Italian Legislation (D.Lgs 152/2006) of 0.06 mg/kg for residential areas (5 mg/kg for industrial areas).

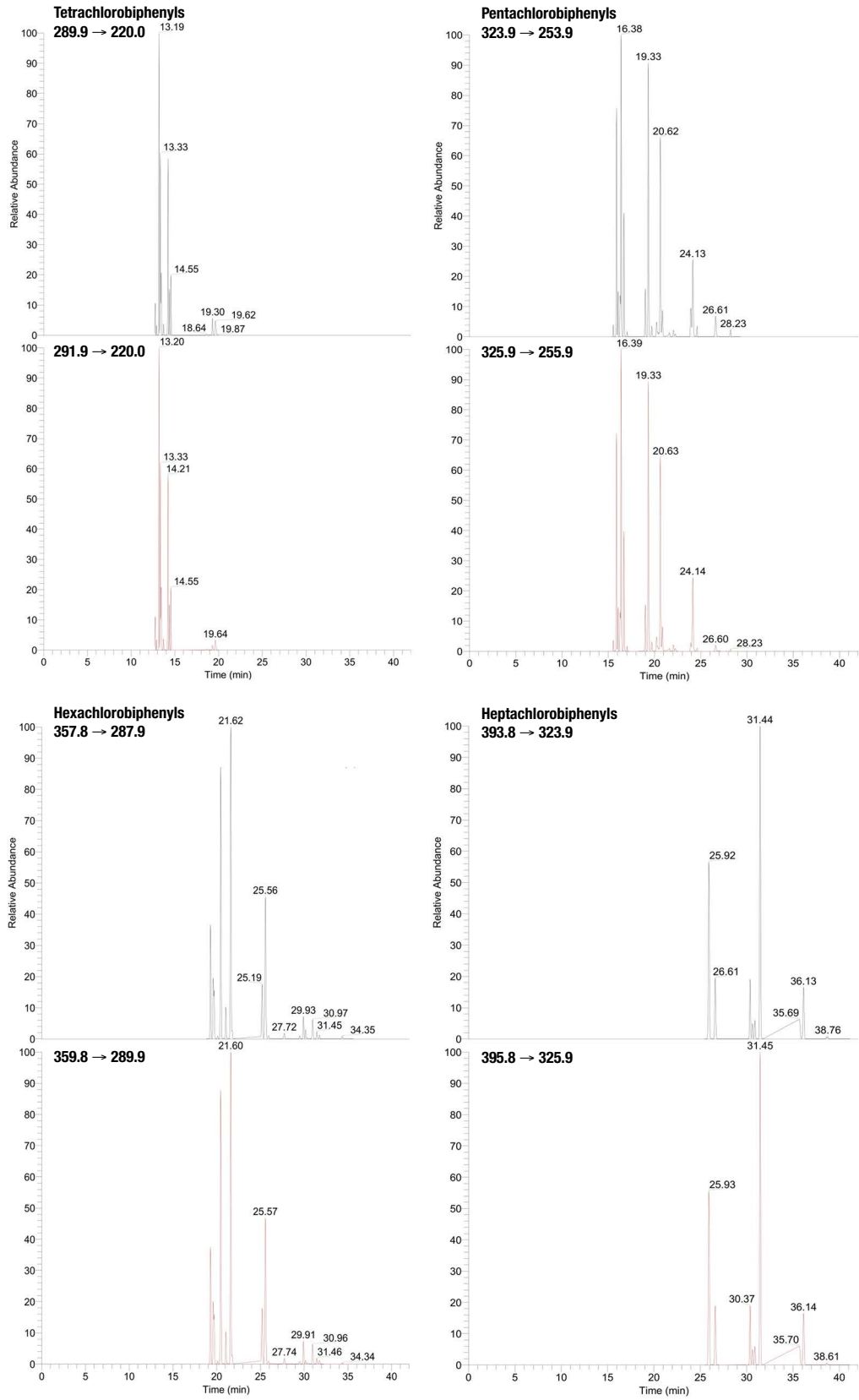

Figure 6. PCB SRM chromatograms for a contaminated soil sample.

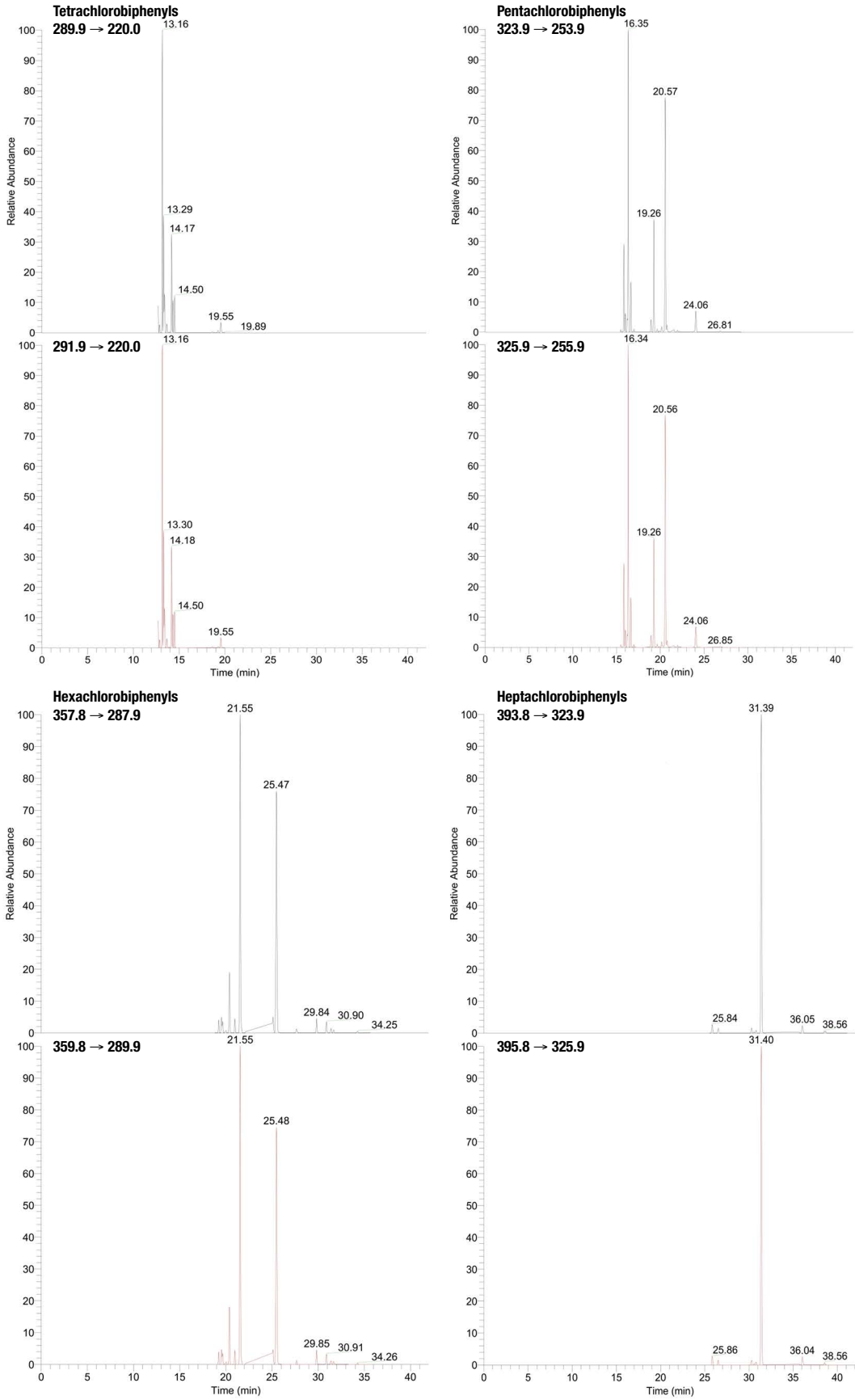

Figure 7. PCB SRM chromatograms for a contaminated soil sample.

Conclusion

When using accelerated solvent extraction with in-cell cleanup to extract polychlorinated biphenyls from contaminated soils, the spike recovery and reproducibility were excellent. The selective removal of interferences with the in-cell cleanup avoids time-consuming and costly post-extraction manual purification procedures. Processing a sample using accelerated solvent extraction requires only 20 min and only 40 mL of solvent. The Rocket Evaporator eliminates the need for cumbersome nitrogen stream evaporation. By using the Flip-Flop system, the samples can be concentrated directly into the GC vial. The high-performance TSQ 8000 triple quadrupole GC-MS system is a reliable, easy-to-use system that enables faster, more precise, error-free analyses, saving time and reducing laboratory costs.

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