Chlorinated Pesticides Analysis by Capillary GC: Alternative Approaches

We manufacture and test SPB-608 capillary columns specifically for analyses of chlorinated pesticides. 30m x 0.25mm ID columns can be used in capillary GC systems; 15m x 0.53mm ID columns can be used in either capillary or packed column systems. A general purpose SPB-5 column can be used in combination with an SPB-608 column – the alternative elution orders and elution times provide confirmational support. Solid phase extraction and solid phase microextraction reduce sample preparation time and costs, relative to liquid-liquid extraction. Because no solvent is introduced onto the column, SPME also enables analysts to use 15m narrowbore columns, rather than 30m columns, for faster analyses. Procedures for using these sample preparation techniques are described here.

Key Words

- chlorinated pesticides
- priority pollutants
- hazardous waste
- solid phase microextraction
- solid phase extraction

Chlorinated pesticides are particularly difficult to quantify by packed column GC. Some of these compounds are incompletely separated, others can break down during analysis. Even small lot-to-lot inconsistencies in packing deactivation cause sample stability to vary from column to column. US Environmental Protection Agency (EPA) Method 608 includes an option for using capillary columns for analyzing 16 chlorinated pesticides in wastewater (1). We manufacture and test SPB™-608 bonded phase capillary columns specifically for analyses of these pesticides. 30m x 0.25mm ID columns can be used in capillary GC systems; 15m x 0.53mm ID columns can be used in either capillary or packed column systems. A general purpose SPB-5 column can be used in combination with an SPB-608 column – the alternative elution orders and elution times provide confirmational support.

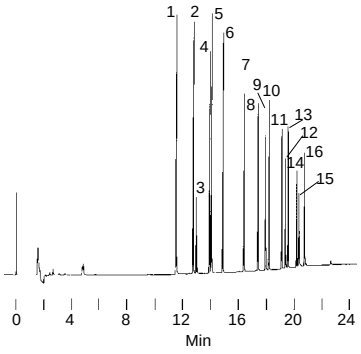
Using 0.25mm ID SPB-608 Columns in Capillary GC Systems

A 30m x 0.25mm ID SPB-608 column is useful for monitoring chlorinated pesticides in sediment, soil, foods, and other matrices, as well as in water. When used with a simple temperature program, a 30m x 0.25mm ID SPB-608 column will separate the 16 pesticides listed in US EPA Method 608 in about 20 minutes (Figure A).[a] Sample breakdown is well within limits specified by the EPA.

Simple, rapid separations make an SPB-608 column the best primary column for chlorinated pesticides analyses. Differences in pesticide elution order and retention times between polar SPB-608 columns and nonpolar SPB-5 columns (Figures A and B, Table

Figure A. 16 Chlorinated Pesticides Separated in 20 Minutes

Column: **SPB-608, 30m x 0.25mm ID, 0.25µm film**
Cat. No.: **24103-U**
Oven: 150°C (4 min) to 290°C at 8°C/min., hold 5 min
Carrier: helium, 25cm/sec, set at 290°C*
(nitrogen make-up gas, 60mL/min)
Det.: ECD
Inj.: 0.8µL decane containing 160ppg each pesticide, splitless (hold 45 sec)



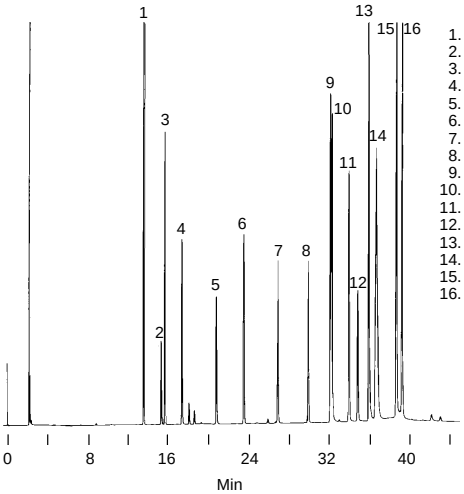
1. α-BHC
2. γ-BHC (Lindane)
3. β-BHC
4. Heptachlor
5. δ-BHC
6. Aldrin
7. Heptachlor epoxide
8. Endosulfan I
9. 4,4'-DDE
10. Dieldrin
11. Endrin
12. 4,4'-DDD
13. Endosulfan II
14. 4,4'-DDT
15. Endrin aldehyde
16. Endosulfan sulfate

796-0056

*A halogenated gas such as chloromethane or chlorotrifluoromethane is used to determine linear velocity by ECD.

Figure B. An Alternative Elution Order

Column: **SPB-5, 30m x 0.25mm ID, 0.25µm film**
Cat. No.: **24034**
Oven: 150°C to 230°C at 2°C/min., hold 5 min
Carrier: helium, 20cm/sec, set at 220°C
Det.: ECD
Inj.: 0.8µL methanol, on-column amounts of each pesticide listed on figure, split (100:1)



1. α-BHC (80pg)
2. β-BHC (80pg)
3. γ-BHC (80pg)
4. δ-BHC (80pg)
5. Heptachlor (80pg)
6. Aldrin (80pg)
7. Heptachlor epoxide (80pg)
8. Endosulfan I (160pg)
9. Dieldrin (160pg)
10. 4,4'-DDE (160pg)
11. Endrin (160pg)
12. Endosulfan II (160pg)
13. 4,4'-DDD (480pg)
14. Endrin aldehyde (480pg)
15. Endosulfan sulfate (480pg)
16. 4,4'-DDT (480pg)

796-0057

[a]We recommend you read about using an electron capture detector with capillary columns before using these columns. Consult the instructions included with your detector, or basic capillary chromatography texts.

1) enable you to use both columns jointly to confirm pesticide identities. Routine analyses can be done on an SPB-608 column, to take advantage of the shorter analysis time. If confirmation is necessary, the sample also can be analyzed on an SPB-5 column. A pesticide usually can be identified with confidence if it elutes from the two columns at the appropriate, complementary retention times (Table 1), and if the two peaks can be shown to represent approximately the same calculated concentration.

Table 1. Elution Orders and Retention Times for Chlorinated Pesticides on SPB-608 and SPB-5 Capillary Columns*

Compound	t_R SPB-608	t_R SPB-5	Compound
α-BHC	11.5	13.5	α-BHC
γ-BHC (Lindane)	12.7	15.1	β-BHC
β-BHC	13.0	15.5	γ-BHC
Heptachlor	13.9	17.2	δ-BHC
δ-BHC	14.0	20.6	Heptachlor
Aldrin	14.9	23.5	Aldrin
Heptachlor epoxide	16.4	26.8	Heptachlor epoxide
Endosulfan I	17.4	29.7	Endosulfan I
4,4'-DDE	17.9	31.9	Dieldrin
Dieldrin	18.2	32.1	4,4'-DDE
Endrin	19.1	33.9	Endrin
4,4'-DDD	19.4	34.9	Endosulfan II
Endosulfan II	19.6	35.9	4,4'-DDD
4,4'-DDT	20.2	36.6	Endrin aldehyde
Endrin aldehyde	20.4	38.5	Endosulfan sulfate
Endosulfan sulfate	20.9	39.1	4,4'-DDT

*Under conditions listed in Figures A and B.

In addition to reducing analysis time, SPB-608 columns improve resolution of some of these pesticides. 4,4'-DDE and dieldrin are separated to baseline (Figure A) and therefore are easier to quantify. This separation is very difficult to obtain from an SPB-5 or equivalent column (Figure B). There is, however, loss of separation between heptachlor and δ-BHC on an SPB-608 column. When used in combination, an SPB-608 column and an SPB-5 column overcome all the resolution problems encountered in this analysis.

You can minimize analyte breakdown and adsorption problems by using cold on-column injection. Precautions must be taken with splitless injections to ensure reproducible analyses and minimize the possibility of analyte adsorption or breakdown. These include:

1. The injection sleeve must be designed for splitless injections. It must be clean and thoroughly deactivated to prevent compounds such as endrin and DDT from being adsorbed at low levels. The sleeve must be properly sealed in the injection port to prevent sample loss in the injector.
2. Injection port temperature must be between 200°C and 250°C, to maximize response and minimize analyte breakdown. Lower temperatures can cause incomplete vaporization, leading to poor response and peak broadening. Higher temperatures can cause breakdown of some pesticides.
3. The column should be installed to specified distances in both the injector and detector. Read and follow your instrument manufacturer's recommendations for column installation.
4. Make-up gas is necessary for the proper operation of an electron capture detector. Nitrogen and methane/argon are the two recommended make-up gases. Use helium or hydrogen as the carrier gas.
5. Column ends should be cut squarely and smoothly, and column or ferrule fragments should not be allowed to enter the column. In addition, we recommend using a guard column to keep nonvolatile residues out of the column. A 1m length of deactivated fused silica tubing can be connected to the column inlet by using a GlasSeal™ capillary column connector (see our catalog).
6. The solvent should dissolve the pesticides well and, for good focusing, should have a boiling point 20-30°C higher than the starting temperature of the analysis.

Using 0.53mm ID SPB-608 Columns in Capillary or Packed Column Systems

0.25mm ID SPB-608 columns can be used only in dedicated capillary systems. 0.53mm ID SPB-608 columns have the same bonded phase as their 0.25mm ID counterparts, resolve the chlorinated pesticides just as effectively (Figure C), meet standards of inertness, low bleed, and reproducibility acceptable to the EPA, and are thoroughly deactivated to prevent sample breakdown. A 0.53mm ID column can be rinsed to remove accumulated contaminants that affect quantification. The advantage to using a 0.53mm column is that it can be installed in a packed column system, by using our injector and detector conversion kits (Figure D). These conversion devices are inexpensive, easy to install or remove, and eliminate the need for a sample splitter, thus eliminating both the expense and the discrimination problems associated with splitters.

When we compared the performance of a 0.53mm ID SPB-608 column and a packed column, using both isothermal and temperature programmed conditions, the capillary column provided greater resolution and faster analysis (Figure C). Temperature programming provided the best results – all 16 pesticides were resolved in 12 minutes. The programmed mode provided sharp peaks and increased sensitivity – especially for later eluting components. Under isothermal conditions, the 16 pesticides were resolved in about 17 minutes, and methoxychlor was eluted in 34 minutes. One of the internal standards, bromobenzene, was not separated from mirex. The isothermal analysis is slower than the programmed analysis, but it can be used with older packed column instruments (Figure E) that do not have temperature programming capability, or adequate flow control in the 3mL/minute range needed for programmed analysis.

To meet EPA requirements for analysis of priority pollutant chlorinated pesticides, a column must meet demanding standards. Inertness is determined by evaluating the breakdown of DDT and endrin, using an electron capture detector (ECD). Under either isothermal or temperature programmed conditions, 0.53mm ID SPB-608 columns meet the criteria of less than 3% breakdown of 4,4'-DDT to 4,4'-DDD and 4,4'-DDE, and less than 10% endrin breakdown to endrin aldehyde. Test results show that in isothermal analyses 4,4'-DDT breakdown is less than 1%, and endrin breakdown is less than 2%. In programmed analyses breakdown is 1% for DDT and 3% for endrin.

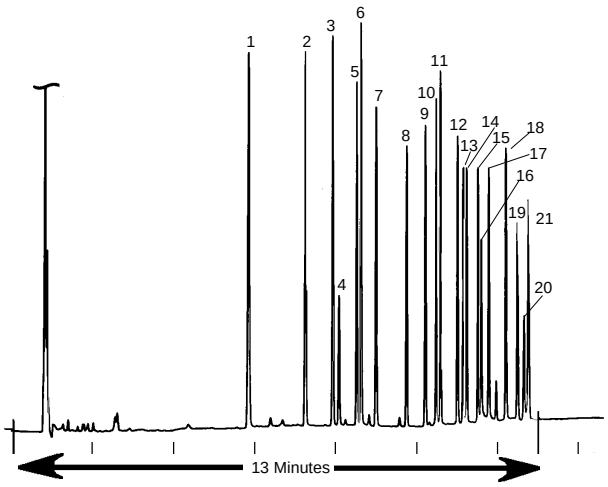
To ensure that each SPB-608 column meets or exceeds the EPA criteria for analyzing picograms of chlorinated pesticides, we measure the relative response for 4,4'-DDT and endrin. 0.53mm ID columns successfully met the EPA criteria for both isothermal and temperature programmed conditions.

Figure C. Faster Analysis and Greater Resolution of EPA Method 608 Pesticides

Column: **SPB-608, 15m x 0.53mm ID, 0.5µm film**
Cat. No.: **25310-U**
Oven: 150°C (4 min) to 280°C at 16°C/min., hold 5 min
Carrier: helium, 38cm/sec (3cc/min)
Det.: ECD
Inj.: 0.6µL decane containing 120pg each pesticide, direct injection

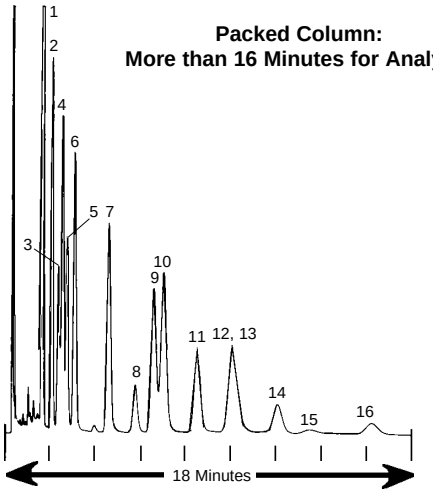
Wide Bore Capillary Column:
Pesticides Resolved in Less Than 13 Minutes

1. Tetrachloro m-xylene (int. std.)
2. α-BHC
3. γ-BHC
4. β-BHC
5. Heptachlor
6. δ-BHC
7. Aldrin
8. Heptachlor epoxide
9. Endosulfan I
10. 4,4'-DDE
11. Dieldrin
12. Endrin
13. 4,4'-DDD
14. Endosulfan II
15. 4,4'-DDT
16. Endrin aldehyde
17. Endosulfan sulfate
18. Dibutylchlorendate (IS)
19. Methoxychlor
20. Hexabromobenzene (IS)
21. Mirex



Packing: **1.5% SP™-2250/1.95% SP-2401 on 100/120 SUPELCOPORT™**
Cat. No.: **11947** (20g)
Column: 2.0m x 4mm ID glass
Oven: 200°C
Carrier: argon:methane, 95:5, 60mL/min
Det.: ECD
Inj.: 0.5µL decane containing 10ng each pesticide

Packed Column:
More than 16 Minutes for Analysis



1. α-BHC
2. γ-BHC
3. β-BHC
4. Heptachlor
5. δ-BHC
6. Aldrin
7. Heptachlor epoxide
8. Endosulfan I
9. 4,4'-DDE
10. Dieldrin
11. Endrin
12. 4,4'-DDD
13. Endosulfan II
14. 4,4'-DDT
15. Endrin aldehyde
16. Endosulfan sulfate

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Figure D. Packed Column Instrument Converted to Wide Bore Capillary Column Use

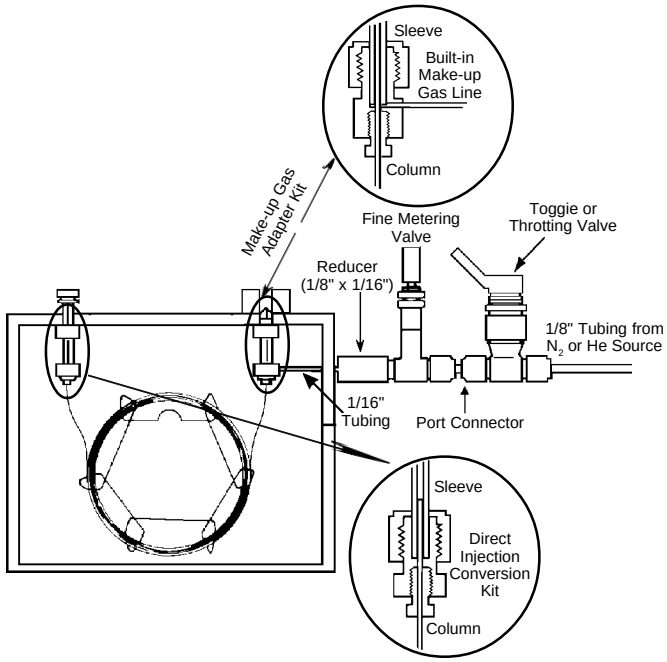

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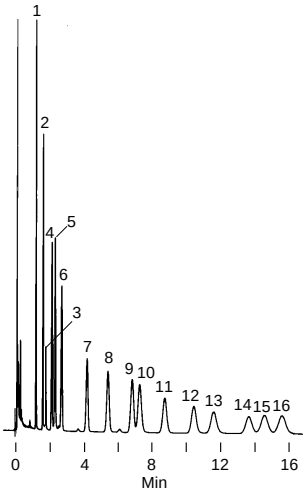
Figure E. Isothermal Analysis, for Older Packed Column Instruments

Column: **SPB-608, 15m x 0.53mm ID, 0.5µm film**
Cat. No.: **25310-U**
Oven: 170°C
Carrier: helium, 20mL/min
Det.: ECD
Inj.: 0.6µL decane containing 120ng each pesticide, direct injection



1. α-BHC
2. γ-BHC
3. β-BHC
4. Heptachlor
5. δ-BHC
6. Aldrin
7. Heptachlor epoxide
8. Endosulfan I
9. Dieldrin
10. 4,4'-DDE
11. Endrin
12. Endosulfan II
13. 4,4'-DDD
14. Endrin aldehyde
15. 4,4'-DDT
16. Endosulfan sulfate

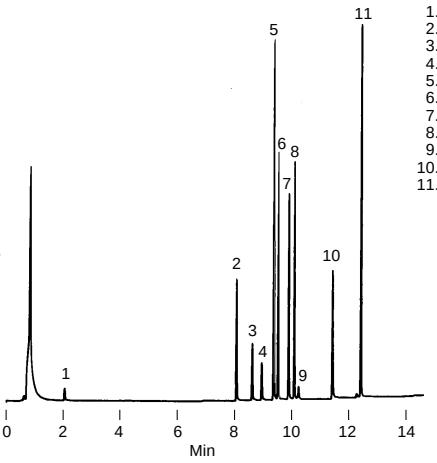
796-0060

In addition to being a suitable replacement for packed columns in EPA Method 608, a 0.53mm ID column can be used to analyze chlorophenoxy herbicide methyl esters in water (US EPA Method 615) or solid waste (EPA Method 8150). In addition to the eight herbicides specified in these methods, Garlon, Picloran, and Blazer® – three compounds whose potential environmental effects are of current interest – also are clearly resolved (Figure F). Procedures differ for isolating the herbicides from water or solid waste samples, but once the herbicides are isolated, they are effectively resolved on a 0.53mm ID SPB-608 column.

Figure F. Chlorophenoxy Herbicides: US EPA Methods 615 and 8150

Column: **SPB-608, 15m x 0.53mm ID, 0.5µm film**
Cat. No.: **25310-U**
Oven: 60°C (1 min) to 280°C at 16°C/min, hold 5 min
Carrier: helium, 5mL/min
Det.: ECD
Inj.: chlorophenoxy methyl esters

1. Dalapon
2. Dicamba
3. Dichlorprop
4. 2,4-D
5. Garlon
6. Silvex®
7. 2,4,5-T
8. Dinoseb
9. 2,4-DB
10. Picloran
11. Blazer®



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Monitoring Chlorinated Pesticides in Hazardous Waste Sites

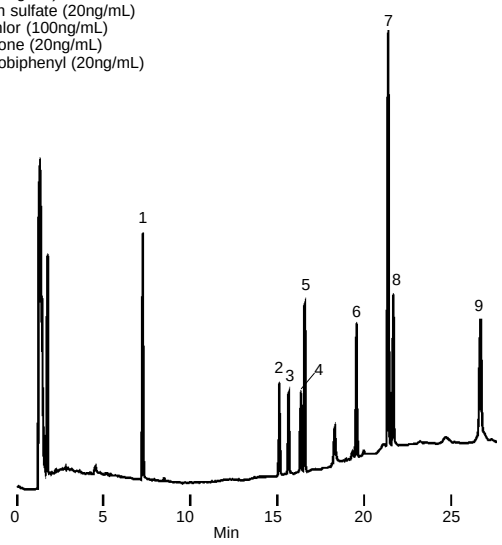
30m x 0.53mm ID SPB-608 capillary columns are suitable for monitoring chlorinated pesticides in samples from abandoned waste sites, according to the US EPA Contract Laboratory Program (CLP) March 1990 Statement of Work (2), and from waste sites currently in use, according to EPA Method 8081. Although the two methods have similar column performance requirements, the CLP method specifies stringent inertness, resolution, and linearity requirements for the wide-bore (0.53mm ID) capillary GC column to be used. Using the CLP method requirements as the measure of SPB-608 column performance ensures that the column also meets Method 8081 requirements.

We evaluated a 0.53mm ID SPB-608 column, using the test mixtures specified in the CLP method, to measure resolution, column inertness, and system linearity. Each mix contains the same two surrogate compounds, tetrachloro-m-xylene and decachlorobiphenyl. Column performance, measured using a resolution check mixture containing seven target pesticides at concentrations of 10-100ng/mL, exceeded the CLP method requirement that resolution between adjacent peaks be greater than 60% of the height of the shorter peak. The analytes in the resolution check mix were resolved to the baseline, including the pairs of analytes cited in the method as having the poorest resolution on a DB-608 column: γ-chlordane/endosulfan I, 4,4'-DDE/dieldrin, and methoxychlor/endrin ketone (Figure G).

Figure G. Baseline Resolution of Target Compounds

Column: **SPB-608, 30m x 0.53mm ID, 0.5µm film**
Cat. No.: **25312**
Oven.: 150°C (0.5min) to 275°C at 6°C/min
Carrier: helium, 5mL/min
Det.: ECD, 300°C
Inj.: 2µL, direct, 200°C, analyte concentrations shown on figure

1. Tetrachloro-m-xylene (20ng/mL)
2. γ-Chlordane (10ng/mL)
3. Endosulfan I (10ng/mL)
4. 4,4'-DDE (20ng/mL)
5. Dieldrin (20ng/mL)
6. Endosulfan sulfate (20ng/mL)
7. Methoxychlor (100ng/mL)
8. Endrin ketone (20ng/mL)
9. Decachlorobiphenyl (20ng/mL)



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Column inertness is evaluated using a mixture of six target pesticides at concentrations of 10-250ng/mL. Decomposition of either endrin or 4,4'-DDT must not exceed 20%, and their combined decomposition must be less than 30%. Endrin recovery is determined by measuring the two by-products, endrin aldehyde and endrin ketone. 4,4'-DDT recovery is determined by measuring the by-products 4,4'-DDD and 4,4'-DDE. The SPB-608 column easily met the inertness criteria in the CLP pesticides method. Endrin decomposition was 8.7%. All decomposition was in the form of endrin ketone; no measurable amounts of endrin aldehyde were evident. 4,4'-DDT decomposition was 0.16%, in the form of 4,4'-DDE. No measurable amounts of 4,4'-DDD were evident. The combined decomposition of 4,4'-DDT and endrin on this column was 8.86%.

The linearity of the instrument, including the column, is determined by calculating the percent relative standard deviation (%RSD) for the calibration factors from a three-point calibration curve for each pesticide and surrogate in individual solutions. Three concentration levels are used to determine linearity: a low concentration mix consisting of the target pesticides at 5-10ng/mL, a mid-range concentration mix (4x low concentration) and a high concentration mix (16x low concentration). The target compounds are analyzed in two different standard mixes, A and B, to avoid coelutions. Absolute retention times are determined by calculating the mean retention time from both mixes. The CLP method allows the RSD for the calibration factors for up to two of the target analytes to be greater than 20% but less than 30%. The RSD for all other analytes must be less than 20%; RSD for the surrogates must be less than 30%.

Linearity for the 0.53mm ID SPE-608 column was well within CLP specifications (Table 2). The mean RSD for all target compounds and surrogates was 9.49%. Absolute retention times for all target analytes and surrogates in the performance evaluation mixture were within the retention time windows established in the calibration runs for the three concentration ranges (Table 2). The target analytes were resolved to the baseline (Figure H), well exceeding the CLP requirement that resolution between adjacent peaks of the individual standard mixes be greater than 90%.

These investigations show that the 0.53mm ID SPB-608 column easily met US EPA requirements for stability, inertness, and linearity, as specified in the CLP Pesticide Statement of Work.

Table 2. SPB-608 Column Easily Meets CLP Retention Time and Linearity Requirements

	Mean t_R (n=3)	Retention Time Window♦ From – To	Calibration Factor %RSD♦♦
Tetrachloro-m-xylene[v]	7.34	7.29 – 7.39	8.78
α-BHC	9.43	9.38 – 9.48	9.90
γ-BHC	10.63	10.58 – 10.68	6.32
β-BHC	10.92	10.87 – 10.97	3.28
Heptachlor	11.84	11.79 – 11.89	2.22
δ-BHC	12.04	11.99 – 12.09	17.16
Aldrin	12.88	12.83 – 12.93	14.61
Heptachlor epoxide	14.06	13.99 – 14.13	9.80
γ-Chlordane	15.17	15.10 – 15.24	5.84
α-Chlordane	15.65	15.58 – 15.72	7.17
Endosulfan I	15.69	15.62 – 15.76	3.33
4,4'-DDE	16.40	16.33 – 16.47	15.66
Dieldrin	16.61	16.54 – 16.68	7.13
Endrin	17.65	17.58 – 17.72	5.00
4,4'-DDD	18.05	17.98 – 18.12	4.47
Endosulfan II	18.22	18.05 – 18.29	27.86
4,4'-DDT	18.98	18.91 – 19.05	6.84
Endrin aldehyde	19.15	19.08 – 19.22	11.93
Endosulfan sulfate	19.62	19.55 – 19.69	10.65
Methoxychlor	21.41	21.34 – 21.48	6.62
Endrin ketone	21.73	21.66 – 21.80	13.62
Decachlorobiphenyl[v]	26.76	26.66 – 26.86	10.52

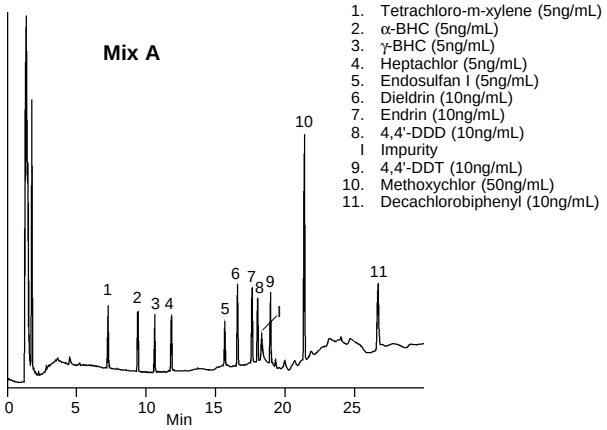
♦±0.05 minutes for compounds eluting before heptachlor epoxide; ±0.07 minutes for heptachlor epoxide and other compounds eluting before decachlorobiphenyl; ±0.10 minutes for decachlorobiphenyl.

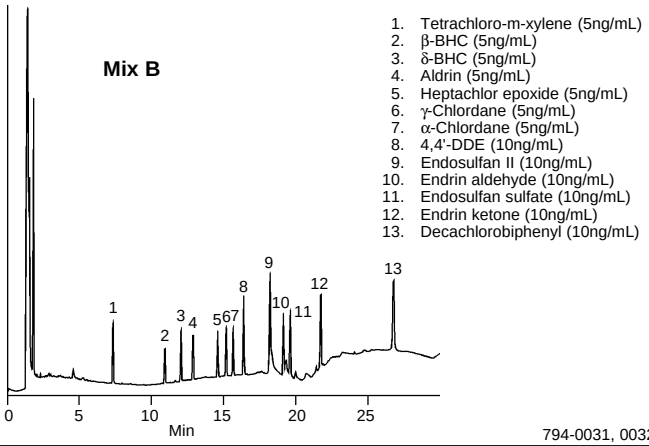
♦♦%RSD for up to two target compounds, not including surrogates, may be >20% but must be ≤30%. %RSD for remainder of target compounds must be ≤20%. %RSD for surrogates must be ≤30%.

[v]Surrogate; retention time measured from analysis of standard mix A.

Figure H. Standard Mixes, Low-Range Concentration

Column: **SPB-608, 30m x 0.53mm ID, 0.5µm film**
Cat. No.: **25312**
Oven.: 150°C (0.5min) to 275°C at 6°C/min
Carrier: helium, 5mL/min
Det.: ECD, 300°C
Inj.: 2µL, direct, 200°C, analyte concentrations shown on figure





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Sample Preparation

Fast Screening for Chlorinated Pesticides, Using Solid-Phase Microextraction (SPME) and Capillary GC

In analyses of chlorinated pesticides in wastewater or other environmental samples, the pesticides usually are extracted by liquid-liquid extraction or with solid phase extraction (SPE) cartridges or disks. These methods are time consuming (4-18 hours by liquid-liquid extraction, 1-2 hours by SPE) and labor intensive (20-45 minutes of handling time per sample), and liquid-liquid extractions require large volumes of sample and solvents. In addition, because pesticides generally are present at ppt levels, mixed with other contaminants at higher concentrations, liquid-liquid extraction and SPE can carry interfering compounds into the final sample, producing extraneous peaks and/or high background. SPE cartridges and, particularly, disks are prone to clogging with samples that contain large amounts of sediment.

In contrast, a recently developed approach to concentrating many analytes, solid phase microextraction (SPME),[b] is faster (15 minutes) and much less labor intensive (about 3 minutes of handling time per sample), and requires only small amounts of sample and no organic solvents. SPME also ensures a low background and less interference, making analyte identification and quantification more reliable.

[b]Technology licensed exclusively to Supelco. US patent pending; European patent #0523092.

SPME is a solventless extraction procedure that does not require complex instrumentation. The technique simply involves immersing a phase-coated fused silica fiber into the liquid sample or the headspace above the sample. Pesticides or other compounds of interest adsorb to the fiber, then are thermally desorbed in the injection port of the GC and are transferred to the capillary column. Selectivity can be altered by changing the phase type or the coating thickness. Salt addition or pH adjustment can improve recovery of difficult-to-extract compounds. Table 3 shows that SPME is consistent in routine use.

Because SPME does not introduce solvent onto the column, a shorter, narrower column and a higher initial temperature can be used, relative to other sample preparation techniques. Chlorinated pesticides can be analyzed on a 15m x 0.20mm ID x 0.20µm phase film SPB-608 or SPB-5 column – either will resolve all of the pesticides listed in Table 3. Columns typically used with other extraction techniques are 30m x 0.25mm ID, to accommodate solvent injection. The short column/high temperature combination reduces analysis time by 10-15 minutes per sample. Figure I shows a dual column analysis of these pesticides following solid phase microextraction from water. All analytes were identified and quantified in less than 15 minutes. The 15m x 0.20mm ID columns were easy to install as a pair and it was not difficult to match flow rates. Figure I also shows an analysis of pesticides from hazardous waste on the SPB-608 column.

The combination of extraction by SPME and analysis on short capillary columns greatly increases the number of samples that

Table 3. SPME Provides Precise Relative Responses for Chlorinated Pesticides at 50ppt

Compound	Relative Response Mean	Std. Dev.	% RSD
α-BHC	0.72	0.07	9.2
β-BHC	0.06	0.01	19.1
γ-BHC	0.53	0.06	10.5
δ-BHC	0.28	0.03	11.9
Heptachlor	1.01	0.10	10.2
Aldrin	1.25	0.06	4.8
Heptachlor epoxide	0.92	0.12	13.2
γ-Chlordane	0.97	0.12	9.9
Endosulfan I	0.87	0.10	11.1
α-Chlordane	0.92	0.11	10.1
4,4'-DDE	0.92	0.07	8.1
Dieldrin	0.83	0.08	9.5
Endrin	0.68	0.06	9.1
Endosulfan II	0.72	0.09	13.0
4,4'-DDD	0.69	0.06	8.9
Endrin aldehyde	0.13	0.04	28.6
Endosulfan sulfate	0.54	0.06	11.9
4,4'-DDT	0.51	0.08	15.2
Endrin ketone	0.57	0.06	10.7
Methoxychlor	0.26	0.04	16.2

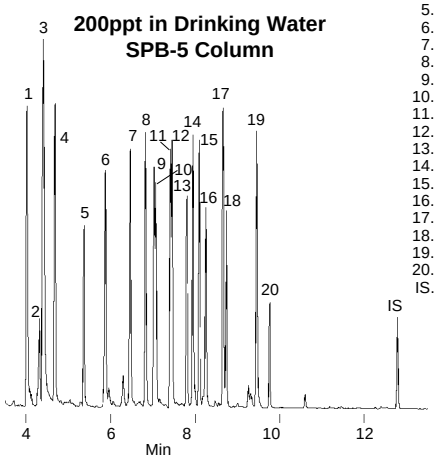
n = 10 extractions

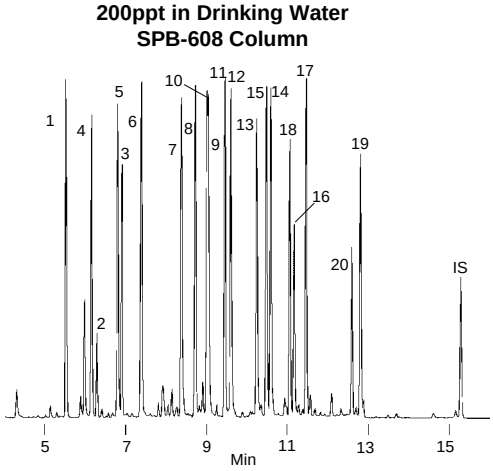
SPME: **100µm PDMS fiber** immersed in 4mL water (15 min, rapid stirring)
Cat. No.: **57300-U** (manual sampling version)
Column: **SPB-5, 15m x 0.20mm ID, 0.20µm film**
Cat. No.: **24165-U**
Oven.: 120°C (1 min) to 180°C at 30°C/min, then to 290°C at 10°C/min
Carrier: helium, 37cm/sec (set at 120°C)
Det.: ECD, 300°C
Inj.: splitless (closed 3 min), 260°C

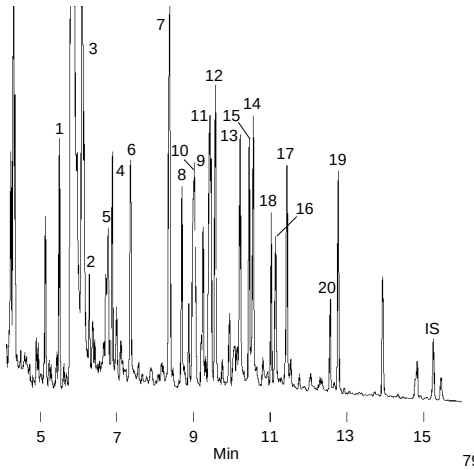
Figure I. Chlorinated Pesticides By SPME/Capillary GC

SPME: **100µm PDMS fiber** immersed in 4mL water (15 min)
Cat. No.: **57300-U** (manual sampling version)
Columns: 15m x 0.20mm ID, 0.20µm film
Cat. Nos.: **SPB-5 - 24165-U; SPB-608 - available on request**
Oven.: 120°C (1 min) to 180°C at 30°C/min, then to 290°C at 10°C/min
Carrier: helium, 37cm/sec (set at 120°C)
Det.: ECD, 300°C
Inj.: splitless (closed 3 min), 260°C

1. Chloromethane
1. α-BHC
2. β-BHC
3. δ-BHC
4. γ-BHC
5. Heptachlor
6. Aldrin
7. Heptachlor epoxide
8. γ-Chlordane
9. Endosulfan I
10. α-Chlordane
11. 4,4'-DDE
12. Dieldrin
13. Endrin
14. Endosulfan II
15. 4,4'-DDD
16. Endrin aldehyde
17. Endosulfan sulfate
18. 4,4'-DDT
19. Endrin ketone
20. Methoxychlor
IS. Decachlorobiphenyl







794-0441, 0442, 0443

can be screened in a day, and improves minimum detection limits while maintaining resolution. SPME can be used for screening samples on site, or prior to GC/MS analysis. Samples found to be highly concentrated can be diluted prior to the GC/MS analysis. SPME is equally compatible with the conditions required by a GC/MS system, however, and thus can be used in the formal analysis, as well as for screening.

SPME is fast, easy, and compatible with short, narrow bore capillary columns that provide fast analyses. Chlorinated pesticides can be extracted with good accuracy, even from wastewater or hazardous waste samples that contain high concentrations of contaminants. Because the apparatus is portable and easy to use, SPME can be employed in the field for quick turnaround methods, yet high precision and accuracy also make SPME effective in formal quantitative analyses.

Solid Phase Extraction of Pesticides from Fruits and Vegetables

Concern over pesticide residues in fruits and vegetables has led to the development of many methods for monitoring these compounds. At the same time, regulatory agencies and concerned analysts are attempting to reduce the amounts of organic solvents used in sample preparation. Solid phase extraction has proven very effective for extracting and concentrating pollutants in analyses of environmental samples. Currently, many SPE methods for extracting pollutants from aqueous environmental samples employ octyl (C8) or octadecyl (C18) phases bonded to a silica support. These materials allow nonpolar analytes to be recovered at high rates and with good reproducibility. Often, however, polar analytes such as carbamate and thiourea pesticides are recovered at low rates under typical reversed phase extraction conditions.

Relative to traditional liquid-liquid extraction or SPE with C8 or C18 silica-based packings, ENVI™-Carb carbon-based packing provides more uniform recovery of a wide variety of nonvolatile analytes. In comparison to values for similar compounds in other studies, SPE on ENVI-Carb carbon produced superior recovery and minimum variability for more polar analytes, such as acids and bases, while maintaining comparable results for less polar compounds (Table 4).

Physical characteristics of ENVI-Carb carbon and C8- or C18-modified silica are listed in Table 5. Because the carbon-based packing is nonporous, samples can be processed rapidly – adsorption does not require dispersion of analytes into porous regions. Furthermore, although the surface area of the nonporous carbon is smaller than that of the porous silica (measured by nitrogen BET), the carbon's capacity for pesticides is not compromised. In fact, the bed weight typically required is only one half that needed with the silica-based packings. The primary surface interaction mechanism is similar for ENVI-Carb packing and silica-based packings, but ENVI-Carb carbon also acts as a weak ion exchanger (3). This expands the range of analytes the material can be used to extract. Samples can be passed through ENVI-Carb tubes under positive pressure or vacuum. We recommend a slow (dropwise) rate.

Investigators at Agriculture and Agri-Food Canada (Ottawa, Ontario) have developed a multiple-residue cleanup and analysis

Table 4. Pesticides Recovery is Highest Using ENVI-Carb SPE Tubes

	Recovery (% ± standard deviation)		
	Solid Phase Extraction		Liquid/Liquid
Analyte	ENVI-Carb* (n=5)	C8/C18 Silica# (n=4)	Extraction# (n=4)
Oxamyl	95 ± 5	53 ± 1	55 ± 16
Methomyl	97 ± 5	43 ± 1	74 ± 8
Aldicarb	96 ± 3	67 ± 8	88 ± 8
Monuron	97 ± 4	90 ± 6	90 ± 4
Carbaryl	98 ± 5	74 ± 15	102 ± 13
Diuron	98 ± 6	90 ± 6	94 ± 3

1 liter water samples, HPLC/UV analyses

*Data from Supelco laboratories.

#Data from B.E. Goodby, *Environmental Laboratory*, June/July 1990, pp19-58.

Table 5. Physical Characteristics of SPE Packings

ENVI-Carb Carbon	C8- & C18-Modified Silica
graphitized carbon black	silane phase-modified silica gel
hydrophobic	hydrophobic
irregular particles, 40-100µm	irregular particles, 40-60µm
nonporous	porous (60-300Å)
100m^2/g surface area	400-600m^2/g surface area

Table 6. Extraction of Pesticides from Fruits and Vegetables

1. Homogenize 50g chopped sample with 100mL acetonitrile (e.g., Omni-mixer, half-speed, 5 min).
2. Add 10g sodium chloride (= 8mL in a graduated cylinder). Homogenize 5 min.
3. Transfer ~13mL of acetonitrile (top) layer to 15mL graduated centrifuge tube.
4. Add ~3g sodium sulfate (liquid level to 15mL mark), cap, shake well to remove water.
5. Centrifuge at high speed for 5 min.
6. Transfer 10mL aliquot (= 5g of sample) to a clean 15mL tube. Evaporate to 0.5mL under clean nitrogen (water bath, 35°C).
7. Transfer to ENVI-Carb SPE tube (6mL tube, 500mg packing).
8. Elute pesticides with 20mL acetonitrile/toluene (3:1).
9. Using a rotary evaporator, concentrate sample to ~2mL. Add 2 x 10mL acetone, concentrating the material to ~2mL after each addition, to make a solvent exchange to acetone.
10. Transfer quantitatively to a clean 15mL tube. Add 50µL internal standard (50ng/µL *cis*-chlordane in acetone), then bring volume to 2.5mL with acetone (final concentrations = 2g/mL extract, 1.0ng/µL *cis*-chlordane).

GC/mass-specific detection (for organochlorine, organophosphorus, nitrogen-containing pesticides)

1. Set aside 0.5mL final extract for GC/MSD analysis. For chromatography, refer to (4).

HPLC/postcolumn derivatization/fluorescence detection (for carbamates)

1. Concentrate remaining 2.0mL final extract to 0.2mL.
2. Add 20µL internal standard (40ng/µL isoprocarb in methanol), then bring volume to 0.8mL with water (pH 3.0 with 36.5-38% HCl/water, 1:4), filter with 0.45µm pore filter (final concentration = 1.0ng/µL isoprocarb). For chromatography, refer to (5).

Process provided by J. Fillion and J.C. Selwyn, Agriculture and Agri-Food Canada, Ottawa, Ontario.

Table 7. Recovery of Pesticides from Fruits and Vegetables, Using ENVI-Carb SPE Tubes

Data for many additional pesticides are listed in Bulletin 900 (available on request)

Analyte	Mean % Recovery	Std. Dev.	C.V. (%)	Commodity / % Recovery						
				Kiwi Fruit	Pine-apple	Beet	Sweet Potato	Squash	Snow Peas	Green Peas
Organochlorine Pesticides										
Aldrin	92.3	5.3	5.7	92	95	92	99	97	86	85
α-BHC	93.3	4.4	4.7	96	94	88	100	96	89	90
p,p' DDE	97.7	7.2	7.4	103	100	98	105	102	91	85
p,p' DDT	96.6	9.1	9.4	102	97	99	106	102	91	79
Dieldrin	96.9	5.1	5.2	97	97	96	104	102	93	89
Endosulfan sulfate	97.4	9.1	9.4	106	98	93	110	101	90	84
Endrin	95.0	5.5	5.8	88	91	98	103	100	94	91
Heptachlor	92.6	5.0	5.4	90	94	92	101	96	89	86
Lindane (γ-BHC)	96.1	5.0	5.2	98	98	90	104	99	92	92
Mirex	96.4	8.9	9.2	102	98	98	104	102	93	78
Methoxychlor	99.1	5.9	5.9	104	98	98	107	103	93	91
Nitrogen-Containing Pesticides / Triazines										
Atrazine	94.7	14.7	15.5	67	83	103	109	104	97	100
Cyanazine*	96.7	6.5	6.7		98	93	108	98	94	89
Cyprazine*	95.7	6.8	7.1		99	87	104	94	89	101
Prometon	89.6	10.4	11.6	75	81	87	104	96	85	99
Simazine	95.1	15.8	16.6	67	80	102	110	107	97	103
Organophosphorus Pesticides										
Azinphos-ethyl	92.6	15.0	16.2	65	80	95	105	103	95	105
Chlorpyrifos	97.9	5.9	6.0	99	99	94	106	104	90	93
Coumaphos	94.9	11.4	12.0	78	96	92	101	85	99	113
Diazinon	96.9	5.6	5.8	90	95	99	107	99	92	96
Dimethoate	99.3	4.6	4.6	105	100	95	104	99	92	100
Disulfoton	71.0	25.4	35.8	24	94	57	92	92	64	74
Ethoprophos	92.0	9.8	10.7	85	107	83	104	91	83	91
Malathion	97.6	6.3	6.5	99	101	92	109	98	91	93
Methamidophos	64.9	8.3	12.7	57	72	59	79	57	65	65
Terbufos	89.0	8.7	9.8	76	93	84	101	97	83	89

n = 7 (n = 6 for *)

Data provided by J. Fillion and J.C. Selwyn, Agriculture and Agri-Food Canada, Ottawa, Ontario.

for monitoring more than 200 organochlorine, organophosphorus, nitrogen-containing, and carbamate pesticides in fruits and vegetables. Their flexible system allows quick screening of "rush" samples as well as thorough cleanup of complex samples. These investigators evaluated the potential value of ENVI-Carb SPE tubes in their procedure, using the process summarized in Table 6. Typical results are summarized in Table 7; data for all 200+ pesticides are summarized in Bulletin 900 (available on request).

Initially, the authors followed a sample extraction procedure that required preparing and using extraction minicolumns containing a mixture of charcoal and Celite® (4), but ENVI-Carb tubes enabled them to recover several pesticides that were not recoverable on charcoal/Celite. By replacing the charcoal/Celite minicolumns with ENVI-Carb SPE tubes, they both improved analyte recovery and eliminated the labor-intensive process of preparing minicolumns.

In the preparation of fruit and vegetable extracts for multiple-residue analysis, additional purification is necessary. Polar interferences from the sample matrix are removed by coupling an aminopropyl-silica-based SPE tube to the ENVI-Carb tube. Under these conditions, pesticide recovery is equally good, except for Folpet. In the Canadian group's sample-screening applications, the analysis for organochlorine, organophosphorus, and nitrogen-containing pesticides (more than 200 compounds) is by capillary gas chromatography with mass-specific detection; residues are identified by retention time and ion ratios. Analytes monitored in each GC run (approximately 115 compounds and 80 compounds, respectively) are listed, along with recovery rates and limits of detection, in (4). Analysis for 10 carbamates is by HPLC with fluorescence detection. Postcolumn derivatization and HPLC columns and conditions are described in (5).

Solid phase extraction of environmental samples on ENVI-Carb tubes offers significant advantages over liquid/liquid extraction or solid phase extraction on silica-based packings. Relative to liquid/liquid extractions, SPE eliminates the need for expensive glassware and large volumes of solvent. The technique also can be easily automated, to allow simultaneous processing of up to 12 or more samples. Relative to silica-based SPE packings, ENVI-Carb tubes offer superior, more consistent recovery for a wide range of organic pollutants.

Low Background Solid Phase Extraction of Chlorinated Pesticides

In the late 1980s, the US Environmental Protection Agency's Office of Solid Waste developed a series of "quick turnaround" (QTM) screening methods, intended to allow decisions to be made quickly about the extent and nature of contamination at potentially hazardous abandoned waste sites. Because the emphasis was on rapid, on-site sample preparation and analysis, solid phase extraction (SPE) was the main focus of sample preparation for monitoring semivolatiles, including pesticides, in aqueous samples. In EPA investigations, an octylsilane-modified, silica-based packing material showed promise for SPE of pesticides. Consequently, Supelco chemists developed a polymerically bonded octylsilane-silica packing, ENVI-8, which effectively extracts pesticides from water. The polymeric bonding allows the

material to resist pH extremes that might be encountered in sampling, and a high carbon loading enhances the packing's capacity for nonpolar analytes.

Any screening method for pesticides requires an extremely inert sample preparation system, because these analytes are of concern at very low concentrations – 10-250ng/100mL – and are monitored by sensitive electron capture detection. To ensure reliable extractions, we packed 6mL glass tubes with 0.5g of ENVI-8 packing and retained the material with Teflon® frits. We extracted pesticide standards at two concentration levels: 250ng each pesticide/mL ("packing capacity level") and 10ng each pesticide/mL ("contamination level"). The higher level was intended to test whether the SPE tubes have adequate capacity for the analytes; the lower level would confirm that background levels in the SPE tube are below the minimum detection levels for the analytes. After monitoring the effects of pH, drying time, and other variables on the extraction, we found the extraction procedure described in Table 8 to be excellent for this purpose. Extractions were performed using a Visiprep™ DL Solid Phase Extraction Vacuum Manifold,▲ fitted with disposable Teflon sample guides and a Visidry™ Drying Attachment.▲▲

Using the procedure in Table 8, we obtained very satisfactory recovery levels for pesticides at both the high concentration and low concentration levels (Table 9). Recovery rates were somewhat variable for endrin and endrin ketone. Overall, the SPE tubes and the extraction procedure provided good results which met the criteria needed for pesticides extractions. Contaminant level (10ng/mL) extractions exhibited few interferences (Figure J), and lot-to-lot variability in recovery rates was acceptable.

We prepare these highly inert ENVI-8 solid phase extraction tubes on request.

Table 8. Solid Phase Extraction of Pesticides for "Quick Turnaround" Screening Method

Extraction Tube:	0.5g ENVI-8 packing in 6mL glass tube, Teflon frits
Conditioning:	3mL methanol, then 2mL 5% methanol in water attach 60mL sample reservoir
Sample Addition:	100mL aqueous sample (pH adjusted to 5.0-7.0) add 5mL methanol, then 10µL of 5.0µg/mL decachlorobiphenyl in methanol (SMC) introduce into 60mL reservoir pass through SPE tube at 5mL/min
Drying:	dry tube under clean nitrogen flow for 2-3 min, using Visidry Drying Attachment
Elution:	add 5mL hexane:acetone (90:10), allow to soak into packing bed allow 2 min static extraction (no vacuum) add 5mL hexane:acetone (90:10), begin dropwise elution (low vacuum) concentrate eluate to 1mL, using clean nitrogen

Table 9. High Recovery of Pesticides by Solid Phase Extraction

	SPE Packing Capacity Level (250ng each pesticide)			Contamination Level (10ng each pesticide)		
Pesticide	Recovery (%)[τ]	Standard Deviation[∇]	Coef. Variation	Recovery (%)[τ]	Standard Deviation[∇]	Coef. Variation
α-BHC	91.4	7.0	7.7	84.6	9.2	10.9
β-BHC	100.1	10.1	10.1	—[ν]	—	—
γ-BHC	94.0	6.1	6.4	88.2	10.8	12.2
δ-BHC	92.9	7.2	7.7	85.1	7.3	8.5
Heptachlor	95.0	5.7	6.0	86.6	6.4	7.3
Aldrin	94.9	4.8	5.1	84.7	13.9	16.4
Heptachlor epoxide	96.8	4.2	4.4	96.3	5.6	5.8
γ-Chlordane	97.4	4.5	4.6	85.8	6.3	7.3
Endosulfan I	94.6	4.7	4.9	84.8	8.5	10.1
α-Chlordane	96.4	4.6	4.7	90.2	7.4	8.2
Dieldrin	98.2	3.8	3.9	88.6	6.0	6.8
4,4'-DDE	99.9	4.2	4.2	80.1	7.7	9.6
Endrin	97.9	13.2	13.5	77.8	20.1	25.9
Endosulfan II	98.2	3.8	3.8	90.0	8.0	8.9
4,4'-DDD	104.4	5.4	5.2	98.6 [o]	10.9	11.1
Endrin aldehyde	101.0	5.5	5.5	98.6 [o]	10.9	11.1
Endosulfan sulfate	104.6	3.9	3.7	96.2	7.9	8.3
4,4'-DDT	107.9	4.9	4.5	95.3	13.4	14.1
Endrin ketone	110.4	14.3	12.9	104.6	29.9	28.6
Methoxychlor	108.4	3.2	2.9	93.4	13.1	14.0
Decachlorobiphenyl (SMC)	101.0	7.0	6.9	72.9	14.0	19.3

[τ]Mean for 5 lots.

[∇]n = 10 extractions.

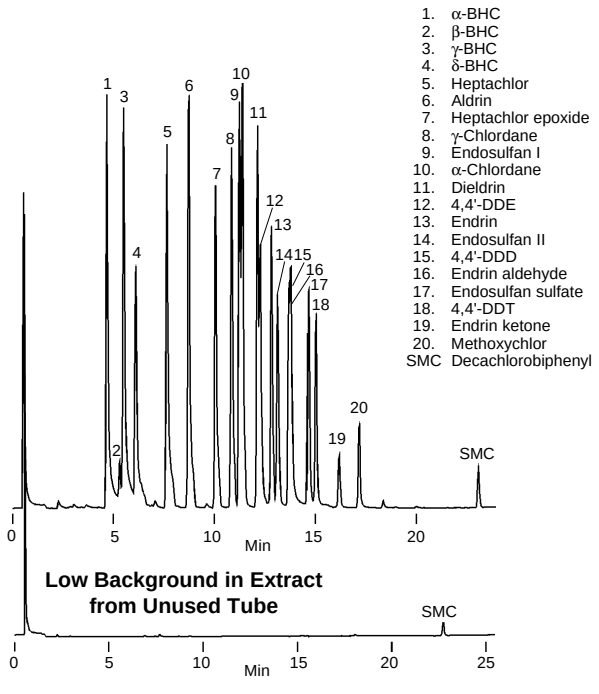
[ν]Could not be monitored under split-splitless, heated injection conditions; cold on-column injection should reduce problem.

[o]Compounds coelute, area counts not separable at 10ng/mL each.

Figure J. Extracted Pesticides

Low Concentration: 10ng each/mL water

Column: **PTE-5 QTM, 15m x 0.53mm ID, 0.5µm film**
Cat. No.: **25355**
Oven.: 150°C (0.5 min) to 275°C at 5°C/min, hold 5 min
Det.: ECD
Inj.: extract from procedure in Table 8, split-splitless injection (hold 45 sec.), 200°C



713-1169, 1168

Column Quality

Bonded phase SPB-608 columns ensure high thermal stability (maximum temperature is 300°C) and low bleed – even when used with sensitive electron capture detectors. When necessary, an SPB-608 column can be rinsed with a nonpolar solvent to remove deposits of nonvolatile material that can affect performance.

To ensure reproducible performance from one column to the next, we test each SPB-608 column with a mixture of the 16 chlorinated pesticides in EPA Method 608. We measure relative responses for endrin and 4,4,-DDT, to ensure minimal breakdown of these unstable components. Our testing criteria also include specifications for overall analysis time and for separation of closely eluting pesticides. To confirm that column performance is satisfactory, we include a chromatogram of the 16 Method 608 pesticides with each column.

Ordering Information:

Description	Cat. No.
Capillary Columns	
SPB-608, 15m x 0.20mm ID, 0.20µm film	**prepared on request**
SPB-608, 30m x 0.25mm ID, 0.25µm film	**24103-U**
SPB-608, 15m x 0.53mm ID, 0.50µm film	**25310-U**
SPB-608, 30m x 0.53mm ID, 0.50µm film	**25312**
SPB-5, 15m x 0.20mm ID, 0.20µm film	**24165-U**
SPB-5, 30m x 0.25mm ID, 0.25µm film	**2-034**
PTE™-5 QTM, 15m x 0.53mm ID, 0.5µm film	**25355**
SPME Holder[c]	
For manual sampling	**57330-U**
For Varian 8100/8200 AutoSampler	**57331**
SPME Fiber Assembly (pk. of 3) 100µm polydimethylsiloxane coating	
For manual sampling	**57300-U**
For Varian 8100/8200 AutoSampler	**57301**
Solid Phase Extraction Tubes	
ENVI-Carb, 3mL, 250mg packing, pk. of 54	**57088**
ENVI-Carb, 6mL, 250mg packing, pk. of 30	**57092**
ENVI-Carb, 6mL, 500mg packing, pk. of 30	**57094**
ENVI-8, 6mL, 0.5g packing, glass tube, Teflon frits	**prepared on request**
AutoTrace SPE Tube Adapters For using ENVI-Carb tubes with Zymark® AutoTrace unit.	
3mL, pk. of 6	**57123**
6mL, pk. of 6	**57126**
Visiprep DL Disposable Liner Solid Phase Extraction Vacuum Manifold	
12-port model	**57044**
24-port model	**57265**
Disposable Teflon Solvent Guides, pk. of 100	**57059**
Visidry Drying Attachment	**57100-U**

For conversion kits for using 0.53mm ID capillary columns in your packed column chromatograph, refer to the Supelco catalog or call our sales department.

[c]Initially you must order both holder and fiber assembly. Holder is reusable indefinitely; fiber lifetime depends on the nature of the analytes and the complexity of the sample matrix. Use of SPME with a Varian 8100/8200 AutoSampler requires an SPME upgrade kit (available from Varian).

Calibration Standards

EPA 608 Pesticides Calibration Mix

20µg/mL each component in hexane:toluene (50:50).

Aldrin	Dieldrin
α-BHC	Endosulfan I
β-BHC	Endosulfan II
γ-BHC (Lindane)	Endosulfan sulfate
δ-BHC	Endrin
4,4'-DDD	Endrin aldehyde
4,4'-DDE	Heptachlor
4,4'-DDT	Heptachlor epoxide (isomer B)

Description	Cat. No.
1mL	**47915-U**

EPA Pesticide Mix

In methanol:methylene chloride, 98:2.

Aldrin, 10µg/mL	Dieldrin, 20µg/mL
α-BHC, 10µg/mL	Endosulfan I, 20µg/mL
β-BHC, 10µg/mL	Endosulfan II, 20µg/mL
γ-BHC (Lindane), 10µg/mL	Endosulfan sulfate, 60µg/mL
δ-BHC, 10µg/mL	Endrin, 20µg/mL
4,4'-DDD, 60µg/mL	Endrin aldehyde, 60µg/mL
4,4'-DDE, 20µg/mL	Heptachlor, 10µg/mL
4,4'-DDT, 60µg/mL	Heptachlor epoxide (isomer B), 10µg/mL

Description	Cat. No.
1mL	**48858-U**

Calibration Standards (contd.)

EPA 608-S Organochlorine Pesticides and PCBs Kit

Individually packaged 1mL solutions in methanol, except as noted.

Aldrin, 20µg/mL
Aroclor 1016, 200µg/mL
Aroclor 1221, 200µg/mL
Aroclor 1232, 200µg/mL
Aroclor 1242, 200µg/mL
Aroclor 1248, 200µg/mL
Aroclor 1254, 200µg/mL
Aroclor 1260, 200µg/mL
α-BHC, 20µg/mL
β-BHC, 20µg/mL*
γ-BHC (Lindane), 20µg/mL
δ-BHC, 20µg/mL*
Chlordane, 20µg/mL
4,4'-DDD, 20µg/
4,4'-DDE, 20µg/mL
4,4'-DDT, 20µg/mL
Dieldrin, 20µg/mL
Endosulfan sulfate, 20µg/mL
Endrin, 20µg/mL
Endrin aldehyde, 20µg/mL
Heptachlor, 20µg/mL
Heptachlor epoxide (isomer B), 20µg/mL
Toxaphene, 20µg/mL

Plus: EPA Pesticide Mix (4-8858)
Aroclor Mix 1 (4-8861)
Aroclor Mix 2 (4-8862)

***** In methanol:methylene chloride, 98:2.

Description	Cat. No.
Kit	**48753-U**

EPA 608-N Organochlorine Pesticides and PCBs Kit

Individually packaged neat compounds in ampuls and vials.

Aldrin, 0.1g
Aroclor 1016, 0.05g
Aroclor 1221, 0.05g
Aroclor 1232, 0.05g
Aroclor 1242, 0.05g
Aroclor 1248, 0.05g
Aroclor 1254, 0.05g
Aroclor 1260, 0.05g
γ-BHC (Lindane), 1g
Chlordane, 1g
4,4'-DDD, 1g
4,4'-DDE, 1g
4,4'-DDT, 1g
Dieldrin, 0.05g
Endosulfan I, 0.025g
Endosulfan II, 0.025g
Endrin, 0.1g
Heptachlor, 1g
Heptachlor epoxide (isomer B), 0.1g
Toxaphene, 1g

Description	Cat. No.
Kit	**48763**

TCL Pesticides Mix

2000µg/mL each component in toluene:hexane (50:50).

Aldrin
α-BHC
β-BHC
δ-BHC
γ-BHC (Lindane)
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan sulfate
Endrin
Endrin aldehyde
Endrin ketone
Heptachlor
Heptachlor epoxide
Methoxychlor

Description	Cat. No.
1mL	**48913**

DDT-Endrin Mix

500µg/mL each component in methanol.

4,4'-DDT
Endrin

Description	Cat. No.
1mL	**48282**

Pesticide Standard Mix A

In hexane:toluene (98:2).

α-BHC, 5µg/mL
γ-BHC, 5µg/mL
4,4'-DDD, 10µg/mL
4,4'-DDT, 10µg/mL
Dieldrin, 10µg/mL
Endosulfan I, 5µg/mL
Endrin, 10µg/mL
Heptachlor, 5µg/mL
Methoxychlor, 50µg/mL

Description	Cat. No.
1mL	**48796**

Pesticide Standard Mix B

In hexane:toluene (99:1).

Aldrin, 5µg/mL
ß-BHC, 5µg/mL
δ-BHC, 5µg/mL
α-Chlordane, 5µg/mL
γ-Chlordane, 5µg/mL
4,4'-DDE, 10µg/mL
Endosulfan II, 10µg/mL
Endosulfan sulfate, 10µg/mL
Endrin aldehyde, 10µg/mL
Endrin ketone, 10µg/mL
Heptachlor epoxide (isomer B), 5µg/mL

Description	Cat. No.
1mL	**48196**

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▲US Pat. No. 4,810,471; other patents pending.

▲▲US Pat. No. D.289,861; 4,810,471; other patents pending.

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Acknowledgment

The extraction process in Table 6 and data in Table 7 were provided by J. Fillion and J.C. Selwyn, Agriculture and Agri-Food Canada, Ottawa, Ontario.

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