

Capillary GC Analyses of Chlorinated Pesticides in Apples

Based on federal and state regulations for identifying and quantifying low levels of pesticides in food and environmental samples, we selected three capillary columns to screen for chlorinated pesticides. A nonpolar and two low/intermediate polarity phases were chosen to evaluate differences in component elution order and retention times. Low level screening analyses were performed effectively by using split/splitless injection and an electron capture detector (ECD). Each column separated the 18 pesticides in approximately 35 minutes. Example chromatograms are shown.

Key Words:

• pesticides • aldrin • endrin • methoxychlor • fruit

Federal and state regulations require that pesticides in food and environmental samples be identified and quantified at low levels. Based on these regulations, we selected three capillary GC columns to screen for pesticides at low levels. We chose a nonpolar phase, PTE™-5, and two low/intermediate polarity phases, SPB™-608 and SPB-1701, to illustrate the differences in component elution order and retention times for chlorinated pesticides.

Eighteen chlorinated pesticides (Table 1) were spiked into, then extracted from, apples purchased at a local grocery store. Extracts were prepared by weighing out 50 grams of fruit, blending it, and adding 100mL of acetonitrile. The fruit extract was reextracted in hexane, using a partitioning process (1). The untreated extracts contained no pesticides.

Table 1. Chlorinated Pesticide Standards Mixture
(TCL Pesticides Mix, Cat. No. 4-8913, 2000µg/mL each component in toluene:hexane, 50:50)

Aldrin
α-BHC
β-BHC
γ-BHC
δ-BHC
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

Samples of the spiked and unspiked extracts were injected onto each capillary column under the conditions listed in Figure A. Figure A shows chromatograms of the extracted pesticides from each column. The low/intermediate polarity SPB-608 and SPB-1701 columns selectively eluted the analytes, based on dipole-dipole and hydrogen bonding interactions between the solute and the stationary phase. Each column separated all of the analytes. The nonpolar PTE-5 column eluted the analytes by boiling point. One pair of analytes (endosulfan sulfate and 4,4'-DDT) coeluted from the nonpolar column. Analysis time for each column was 31-36 minutes. Elution order differences among the three columns are evident in Figure A. In a two-column analysis, these differences can be used to help confirm the identities of the analytes.

Table 2 lists the recovery values for the pesticides, determined using the SPB-608 column. Recovery of the spiked analytes ranged from 15% to 122%.

Based on these evaluations, we determined that the three stationary phases, PTE-5, SPB-608, and SPB-1701, exhibited differences in retention times, resolution, and elution order for 18 common chlorinated pesticides. Using these columns, screening analyses for low levels of these pesticides can be performed effectively, in approximately 35 minutes, with split/splitless injection and an electron capture detector (ECD).

Table 2. Recovery of Chlorinated Pesticides from Apples (SPB-608 Column)

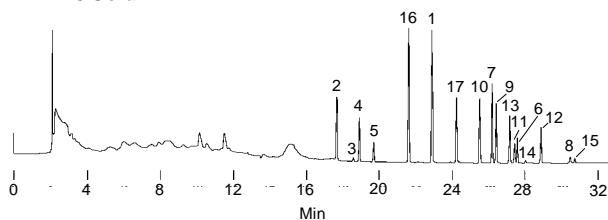
Pesticide	Recovery (%)
Aldrin	122
α-BHC	70
β-BHC	56
γ-BHC	35
δ-BHC	111
4,4'-DDD	53
4,4'-DDE	112
4,4'-DDT	29
Dieldrin	89
Endosulfan I	101
Endosulfan II	71
Endosulfan sulfate	15
Endrin	98
Endrin aldehyde	88
Endrin ketone	—
Heptachlor	47
Heptachlor epoxide	91
Methoxychlor	41

Figure A. Chlorinated Pesticides from Apples

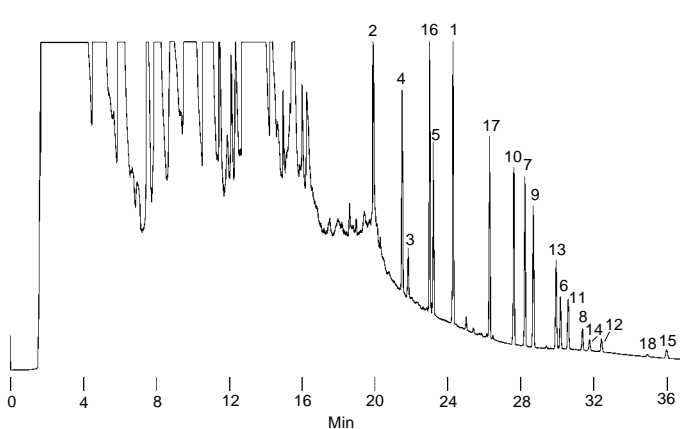
Stationary Phases: PTE-5, SPB-608, SPB-1701
 Column Dimensions: 30m x 0.25mm ID, 0.25µm phase film
 Catalog Nos.: 24135-U (PTE-5), 24103-U (SPB-608), 24113 (SPB-1701)
 Oven: 100°C to 280°C at 6°C/min
 Carrier: helium, 40cm/sec.
 Det.: ECD, 250°C
 Sample: 1µL of 0.1µg/mL extract, split/splitless 45 sec, 250°C

- | | |
|-------------|------------------------|
| 1. Aldrin | 10. Endosulfan I |
| 2. α-BHC | 11. Endosulfan II |
| 3. β-BHC | 12. Endosulfan sulfate |
| 4. γ-BHC | 13. Endrin |
| 5. δ-BHC | 14. Endrin aldehyde |
| 6. 4,4'-DDD | 15. Endrin ketone |
| 7. 4,4'-DDE | 16. Heptachlor |
| 8. 4,4'-DDT | 17. Heptachlor epoxide |
| 9. Dieldrin | 18. Methoxychlor |
- (not observed with PTE-5 column)

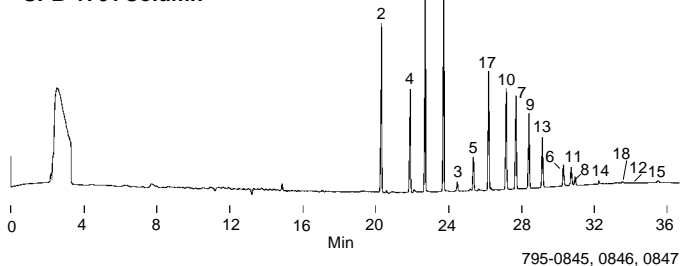
PTE-5 Column



SPB-608 Column



SPB-1701 Column



Ordering Information:

Description	Cat. No.
Fused Silica Capillary Columns	
all 30m x 0.25mm ID, 0.25µm phase film	
PTE-5	24135-U
SPB-608	24103-U
SPB-1701	24113
Chlorinated Pesticides Mixture	
(TCL Pesticides Mix)	
2000µg/mL each analyte in Figure A in toluene:hexane, 50:50	48913

Reference

1. Kaphalia, B.S. *Assoc. Official Analytical Chemists* **73** (4) 1990.

Note: For a suitable extraction procedure, refer to AOAC Methods, 16th edition (Order from AOAC International, 481 North Frederick Avenue, Suite 500, Gaithersburg, Maryland 20877-2504 USA. Tel.: +1-301-924-7077; FAX: +1-301-924-7089.)

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