Application Note 91

Capillary GC Analyses of Chlorinated Pesticides in Dry Pet Food

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 • pet food

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, dry pet food purchased at a local grocery store. Extracts were prepared by weighing out 50 grams of pet food, manually crushing it, and adding 100mL of acetonitrile (1). *The untreated extracts contained no pesticides.*

Table 1. Chlorinated Pesticide Standards Mixture

(TCL Pesticide 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.

In general, recovery of the chlorinated pesticides was poor. The extraction procedure apparently was unable to remove these analytes from the fat in the pet food.* Table 2 lists the recovery values for the pesticides, determined using the SPB-608 column. Values ranged from 5% to 122%. The limits of detection for the standards ranged from 0.1 to 10.0 ppm. These values should improve with a more effective extraction procedure.

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). Pesticide recovery can be affected by the fat content of the sample, thus development of an effective extraction procedure is important.

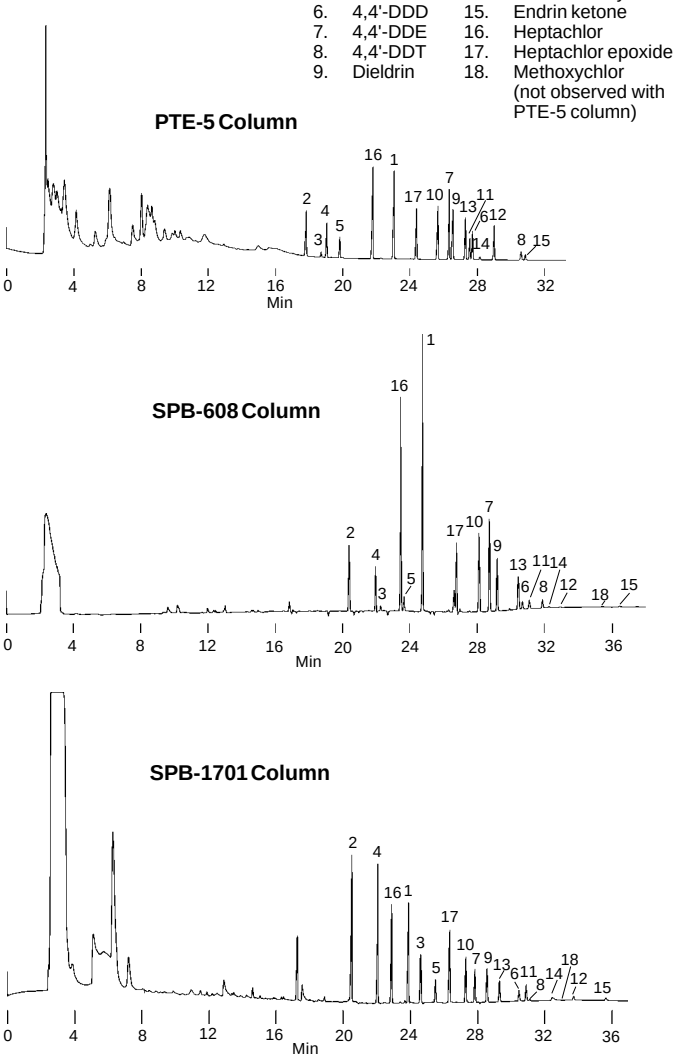
Table 2. Recovery of Chlorinated Pesticides from Dry Pet Food (SPB-608 Column)

Pesticide	Recovery (%)
Aldrin	122
α-BHC	17
β-BHC	14
γ-BHC	5
δ-BHC	84
4,4'-DDD	23
4,4'-DDE	91
4,4'-DDT	61
Dieldrin	51
Endosulfan I	65
Endosulfan II	19
Endosulfan sulfate	—
Endrin	60
Endrin aldehyde	—
Endrin ketone	—
Heptachlor	6
Heptachlor epoxide	41
Methoxychlor	—

Figure A. Chlorinated Pesticides from Dry Pet Food

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



795-0848, 0849, 0850

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.

* 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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