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Evaluation of a New Gel Permeation Chromatography (GPC) System for Sample Cleanup Prior to GC or GC/MS

Application Note 16820301

Keywords
GC
GPC AutoPrep 2000
Gel Permeation Chromatography
Pesticides
Semivolatiles
USEPA Method 3640A

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Introduction

Gel Permeation Chromatography (GPC) is a size-exclusion cleanup procedure that uses organic solvents and a hydrophobic gel (primarily a cross-linked divinylbenzene-styrene copolymer) to separate macromolecules. GPC is a highly effective cleanup method for removing high molecular weight interferences from sample extracts. It is recommended for the elimination of lipids, polymers, proteins, natural resins, and cellular components from a sample prior to further analysis (Czuczwa and Alford-Stevens, 1989).

GPC cleanup has been used extensively for numerous environmental analyses, especially for the preparation of samples prior to semivolatile and pesticide analysis by GC and GC/MS. It is an effective way to protect GC columns, improve accuracy, and allow for lower detection limits. Laboratories participating in the USEPA Contract Laboratory Program (CLP) or that are following CLP protocols must use GPC as outlined in USEPA Method 3640A and the USEPA Contract Laboratory Program Statement of Work for Organics Analysis (Document nos. OLM02.1 and OLM04.2).

OI Analytical recently introduced the GPC AutoPrep 2000 system for automated GPC cleanup (Figure 1). The system uses an autosampler for both injecting up to 60 samples and collecting the purified fractions into a variety of collection vessels. The system features a modular design and electronic valve actuation, and it employs a syringe pump for sample pick up and a wash pump to rinse the needle and prevent sample carryover. The system is controlled via a PC using a Windows®-based software program.

Figure 1. GPC AutoPrep 2000 System

The purpose of this study was to evaluate the performance characteristics for USEPA calibration requirements under USEPA Method 3640A using the GPC AutoPrep 2000 system and the three most common types of columns used with this system.

Experimental

Apparatus

Sample cleanup was achieved using an OI Analytical GPC AutoPrep 2000 equipped with either a 700 mm x 25 mm glass column containing 70 g of Envirobead™ S-X3 Resin, an Envirosep-ABC™ column, or an Optima™ PTFE column. A 5-mL sample loop was used for the glass and Envirosep-ABC column. A 2.5-mL loop was used for the Optima column. Cleanup was achieved using a flow rate of 5 mL/min with methylene chloride as the mobile phase. A flow rate of 4 mL/min was used for the Optima column. Elution profiles were recorded using a UV monitor (254 nm) and strip chart recorder. Collected fractions were evaporated to 1 mL using a Labconco RapidVap system.

GC analysis was performed using an Agilent Technologies 6890 Series GC with a J&W Scientific DB 17MS column (20 m x 18 µm I.D.) and an ECD for detection. Hydrogen was used as the carrier gas.

Standards and Reagents

A GPC calibration standard containing corn oil, bis(2-ethylhexyl)phthalate, methoxychlor, perylene, and sulfur was obtained from Accustandard (New Haven, CT).

A semivolatile pesticides stock standard was obtained from Radian International (Austin, TX). A working standard was prepared from the original stock to a concentration of 2.5 µg/mL in acetone/methanol. 200 µL of the working standard was diluted to 10 mL with methylene chloride and injected into the GPC AutoPrep 2000 system.

A pesticides surrogate spiking solution was obtained from Ultra Scientific (N. Kingston, RI). 200 µL of a 5.0 µg/mL standard was diluted in 10 mL of methylene chloride and used as a blank on the GPC system.

All reagents were pesticide grade or higher.

Methods

The GPC column was calibrated using the method outlined in USEPA Method 3640A, Section 7 (1994). The flow rate of the column eluant was verified by collecting the eluant in a graduated cylinder for 10 minutes and measuring the volume. The elution times for the corn oil, phthalate, methoxychlor, perylene, and sulfur were determined for each column. A “dump” time was chosen that would remove greater than 85% of the phthalate but collect greater than 95% of the methoxychlor. Fraction collection was stopped after the elution of perylene but before sulfur eluted. A pesticide standard was prepared as outlined above and injected into the AutoPrep 2000 system (n=5) along with a blank containing a surrogate standard and recoveries determined for each column type.

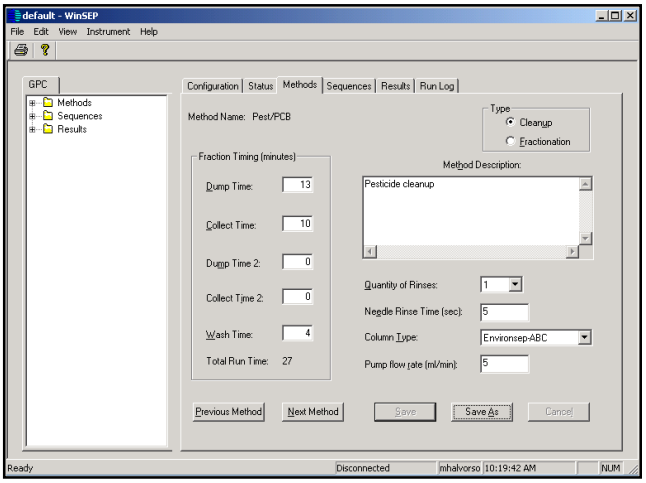

Figure 2. WinSEP Methods Screen Showing a Typical Dump Time, Collect Time, and Wash Time when Using the AutoPrep 2000 to Collect Pesticides and PCBs on an Envirosep-ABC GPC Column with a Flow Rate of 5 mL/min and a Mobile Phase of 100% Methylene Chloride

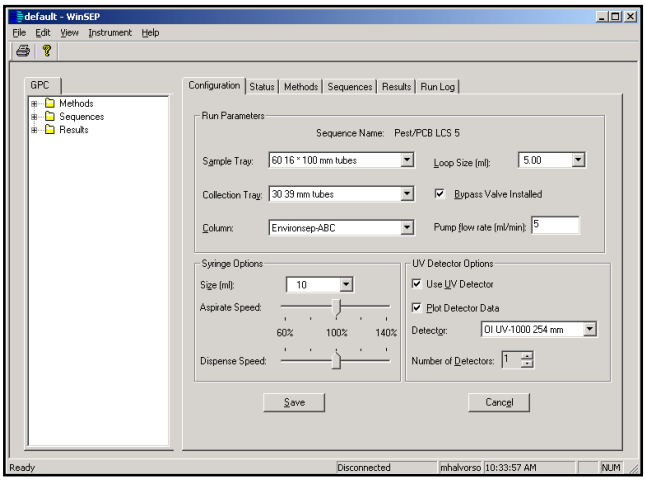

Figure 3. WinSEP Configuration Page Showing GPC Parameters used for Sample Cleanup Using an Envirosep-ABC Column

Results

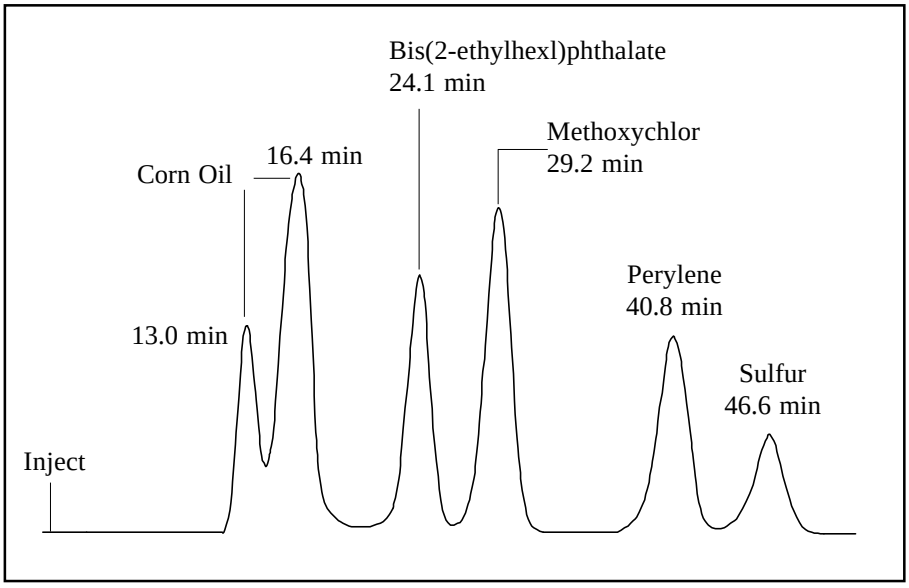

Figure 4. Chromatogram of a USEPA GPC Calibration Standard Using a Glass Column with Envirobead S-X3 Select Resin (Rate: 30.0 cm/hr; Range: 0.5 Au)

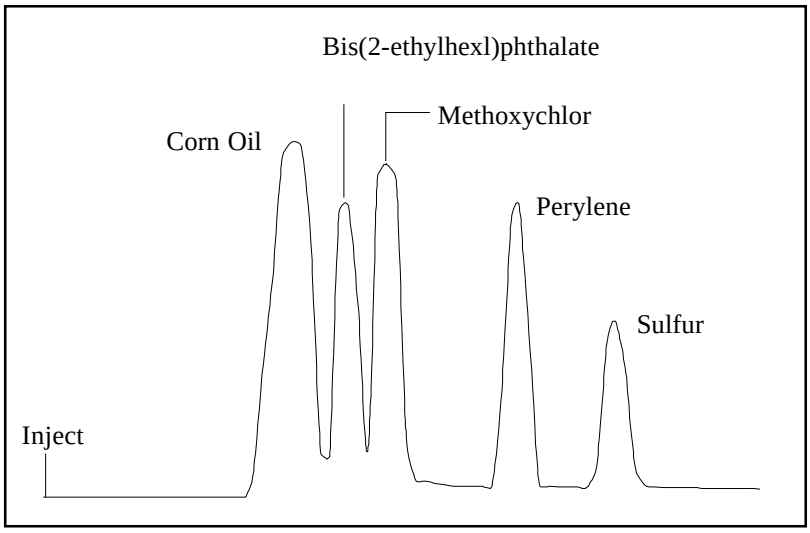

Figure 5. Chromatogram of a USEPA GPC Calibration Standard Using an Envirosep-ABC Column (Rate: 30.0 cm/hr; Range: 0.5 Au)

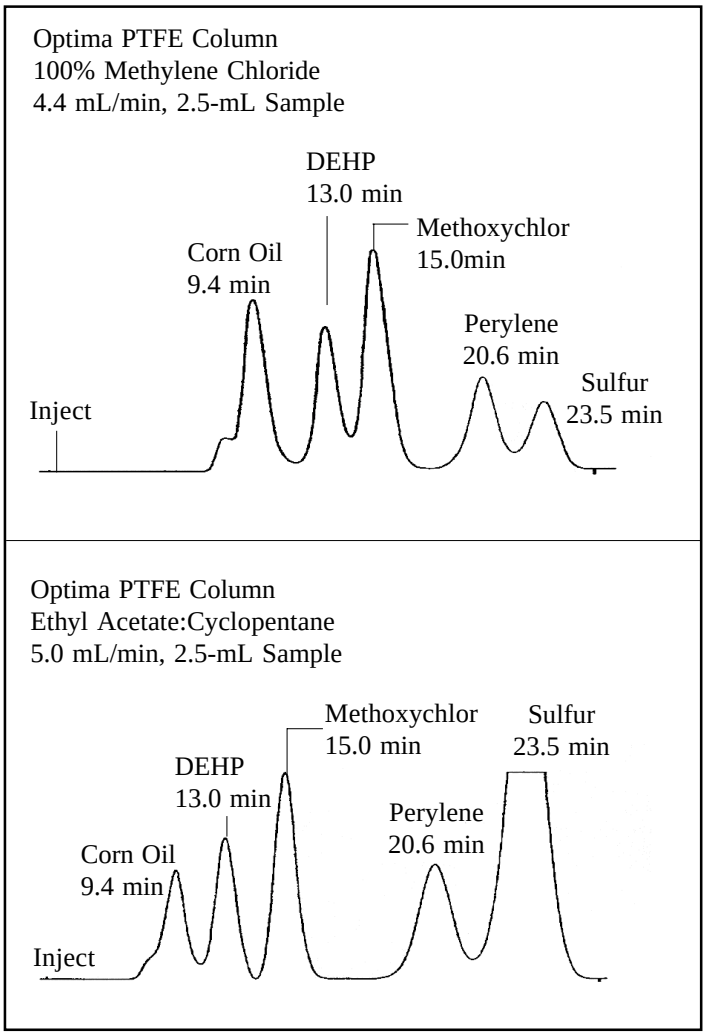

Figure 6. UV Chromatogram for USEPA Calibration Compounds on an Optima Column Packed with Methylene Chloride or 70:30 Ethyl Acetate and Cyclopentane

Table 1. GPC Cleanup Parameters Calculated by Using a USEPA Calibration Standard

GPC Column Parameters for USEPA Calibration Standard	Dump Time	Collect Time	Wash Time	Total Time	Flow Rate
70 g Glass Column with Envirobead S-X3 Select Resin	26 min	17 min	20 min	63 min	5 mL/min
Envirosep-ABC Stainless Steel Column (350 x 21.2 mm) w/ 60 x 21.2 mm Guard Column	13 min	9 min	15 min	37 min	5 mL/min
Optima PTFE Column	11 min	7 min	15 min	33 min	4 mL/min

Table 2. GPC Recoveries of Pesticide Using the AutoPrep 2000 and an EnviroSep-ABC Column

Pesticide	Av.rec.,%	Recovery Range,%	%RSD (n=5)
Aldrin	90.5	83.4–96.4	7.0
α-BHC	90.2	82.0–99.7	8.5
β-BHC	81.9	75.5–87.0	6.0
δ-BHC	80.4	69.1–96.5	5.8
γ-BHC-Lindane	88.8	81.3–94.4	6.1
α-Chlordane	94.4	90.6–97.3	2.7
γ-Chlordane	93.8	90.0–98.9	3.4
p,p'-DDD	98.2	93.9–104.1	4.6
p,p'-DDE	99.4	94.6–106.8	5.6
p,p'-DDT	105.8	99.6–107.2	4.0
Dieldrin	96.1	90.0–100.2	4.0
Endosulfan I	73.0	69.7–75.4	3.4
Endosulfan II	105.5	102.8–108.1	2.2
Endosulfan Sulfate	94.0	90.0–98.4	3.7
Endrin	107.1	101.0–111.2	3.9
Endrin Aldehyde	91.7	89.0–94.4	2.8
Endrin Ketone	93.7	92.3–95.8	1.4
Heptachlor	91.1	84.8–96.5	6.2
Heptachlor Epoxide	90.9	85.2–95.4	4.2
Methoxychlor	101.0	98.7–103.2	1.6

Table 3. GPC Recoveries of Pesticide Using the AutoPrep 2000 and a Glass Column with Envirobead S-X3 Select Resin

Pesticide	**Av.rec.,%**	**Recovery Range,%**	**%RSD (n=5)**
Aldrin	82.2	73.3–87.3	6.6
α-BHC	74.3	66.6–79.3	6.7
β-BHC	77.0	69.3–79.5	6.6
δ-BHC	66.0	58.1–70.9	7.2
γ-BHC-Lindane	75.5	67.1–81.1	7.1
α-Chlordane	81.3	72.5–87.2	6.8
γ-Chlordane	81.8	75.4–86.4	5.0
p,p'-DDD	81.5	73.9–86.5	5.7
p,p'-DDE	86.8	79.6–90.6	4.8
p,p'-DDT	75.1	68.3–79.9	6.5
Dieldrin	86.8	82.0–90.6	3.9
Endosulfan I	62.2	56.7–65.0	5.1
Endosulfan II	69.9	66.8–73.3	3.4
Endosulfan Sulfate	76.7	71.5–81.5	5.1
Endrin	90.8	79.1–98.3	7.8
Endrin Aldehyde	75.8	72.0–80.1	3.4
Endrin Ketone	75.6	73.6–77.4	1.8
Heptachlor	70.9	61.7–77.0	7.9
Heptachlor Epoxide	81.4	72.4–85.4	6.5
Methoxychlor	73.2	70.9–85.2	7.8

Table 4. GPC Recoveries of Pesticide Using the AutoPrep 2000 and an Optima PTFE Column

Pesticide	**Av.rec.,%**	**Recovery Range,%**	**%RSD (n=5)**
Aldrin	96.5	91.2–100.0	4.4
α-BHC	85.4	80.3–87.0	3.5
β-BHC	87.5	85.2–92.4	3.4
δ-BHC	73.8	65.2–86.5	5.1
γ-BHC-Lindane	88.3	83.3–91.8	3.6
α-Chlordane	94.9	91.1–98.8	3.4
γ-Chlordane	95.1	90.3–97.5	3.4
p,p'-DDD	94.0	91.0–98.2	4.9
p,p'-DDE	103.7	96.0–110.2	5.8
p,p'-DDT	84.8	81.3–88.2	3.3
Dieldrin	103.4	99.1–105.8	2.5
Endosulfan I	72.8	69.2–74.4	2.9
Endosulfan II	80.4	78.2–82.6	2.5
Endosulfan Sulfate	84.6	83.2–85.8	1.1
Endrin	102.9	96.6–106.3	4.5
Endrin Aldehyde	82.6	75.5–85.9	4.5
Endrin Ketone	88.0	84.0–90.7	3.5
Heptachlor	85.6	81.2–88.0	3.3
Heptachlor Epoxide	97.6	95.3–100.5	2.1
Methoxychlor	81.9	76.8–86.4	4.5

Summary and Conclusions

The objective of this study was to evaluate the performance characteristics of the OI Analytical GPC AutoPrep 2000 System for meeting USEPA requirements under SW-846 Method 3640A. Method guidelines require greater than 85% resolution between corn oil and phthalate, greater than 85% resolution between phthalate and methoxychlor, and greater than 90% resolution between perylene and sulfur. All three types of columns used on this system were capable of meeting these requirements. Resolution was not adequate for the Optima column using a methylene chloride mobile phase, but it was adequate when 70:30 ethyl acetate:cyclopentane was used as the mobile phase. The glass column packed with Envirobead S-X3 resin provided the best resolution but required the longest run time.

Average recovery data was adequate for both the Optima column and the Envirosep-ABC column, ranging from 72% to 111% with all %RSDs at less than 9%. Recovery data from the glass column packed with Envirobeads S-X3 was lower than the other two columns, ranging from 62% to 91%. Further studies are in progress to evaluate the recovery of pesticides and semivolatile compounds from a variety of matrices using the new OI Analytical GPC AutoPrep 2000 system.

Acknowledgements

We would like to thank Lisa Wool and Meredith Clarge of USEPA, Region 6 for their contributions to this study.

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Post-extraction Gel Permeation Chromatography Cleanup of Animal Liver Prior to HPLC Analysis For Anticoagulant Rodenticides

Application Note 19230603

Keywords
Anticoagulant Standard
Anticoagulant Rodenticide
Brodificoum
Fluorescence Detection
Gel Permeation Chromatography (GPC)
GPC Cleanup
High Performance Liquid Chromatography (HPLC)
Liver Extract
SP 2000 GPC Cleanup System
RapidVap
Warfarin

Introduction

Anticoagulants such as 4-hydroxycoumarins and indandiones have been used for many years as rodenticides. These compounds are toxic to all mammals. Accidental poisoning of nontarget animals such as dogs, cats, wild animals, and occasionally humans can occur. If a fatality occurs, liver tissue analysis for these anticoagulants is usually required to confirm their presence.

High performance liquid chromatography (HPLC) with UV and fluorescence detection appears the most effective analytical method for measuring anticoagulant rodenticides. Cleanup of liver extract is required for adequate detection. Several published methods (Hunter, K. 1983, 1985; Munday et al., 1982) describe the use of GPC to clean liver extracts containing coumarin-based rodenticides. GPC cleanup allows the detection of low (1 ppm or less) anticoagulant levels. Many laboratories only receive samples for analysis infrequently, making it costly and impractical to use specialized automated GPC systems currently on the market (Chalermchaikit et al., 1993; Langseth and Nymoen, 1991).

OI Analytical recently introduced the Model SP 2000™ GPC Cleanup System, OI Analytical part number 320389, as a simple and economical solution for GPC cleanup (Figure 1). The SP 2000 consists of an isocratic HPLC pump, manual sample injector, switching valve, and compact keypad control module (system control unit). The sample injects by syringe into a calibrated 5-mL sample loop (other sizes available). The sample is introduced into the column by switching the valve to the inject position. During the elution (cleanup) process, the sample components are separated using a gel permeation chromatography column packed with Envirobeads™ S-X3 and the fraction containing the desired analytes such as rodenticides are collected at a predetermined time into a prepared collection vessel. Flow rate, total analysis time, start of fraction collection, and duration of fraction collection are all set up on the easy-to-use system control unit.

Figure 1. Model SP 2000 GPC Cleanup System

REGISTERED COMPANY ISO 9001 SGS

Analysis

Dr. Sean Linder, chief chemist with the Arkansas Livestock and Poultry Commission, Veterinary Diagnostic Laboratory, in Little Rock, Arkansas, recently purchased a SP-2000 for liver extract cleanup prior to analysis for rodenticides such as brodificoum, bromodiolone and warfarin. He found that the system is very cost-efficient, effective for cleanup and easy to use. He also used a RapidVap® N_2 Evaporation System, OI Analytical part number 981-019 (Figure 2) to concentrate the liver extract both before and after GPC cleanup.

Figure 2. RapidVap N_2 Evaporation System

After GPC cleanup and evaporation, sample extracts were reconstituted in methanol and analyzed using an Agilent™ Model 1100 HPLC equipped with a Model 1100 diode array detector and a Model 1100 fluorescence detector (excitation = 318 nm; emission = 390 nm). The flow rate was 1.0 mL/min and the mobile phase consisted of a gradient mixture of TEAA buffer (3.85 g ammonium acetate, 2 mL glacial acetic acid, 2 mL triethylamine and 1 L water) and methanol. The gradient conditions follow:

Table 1. Gradient conditions using a Waters C18 analytical column (250 x 4.6 mm) maintained at 30°C.

Time (min)	% TEAA Buffer, pH 5.2	% Methanol
0	38	62
4	18	82
12	18	82
17	38	62
25	38	62

Attached below are some of the chromatographic results from Dr. Linder's work at the Arkansas Poultry and Livestock Commission.

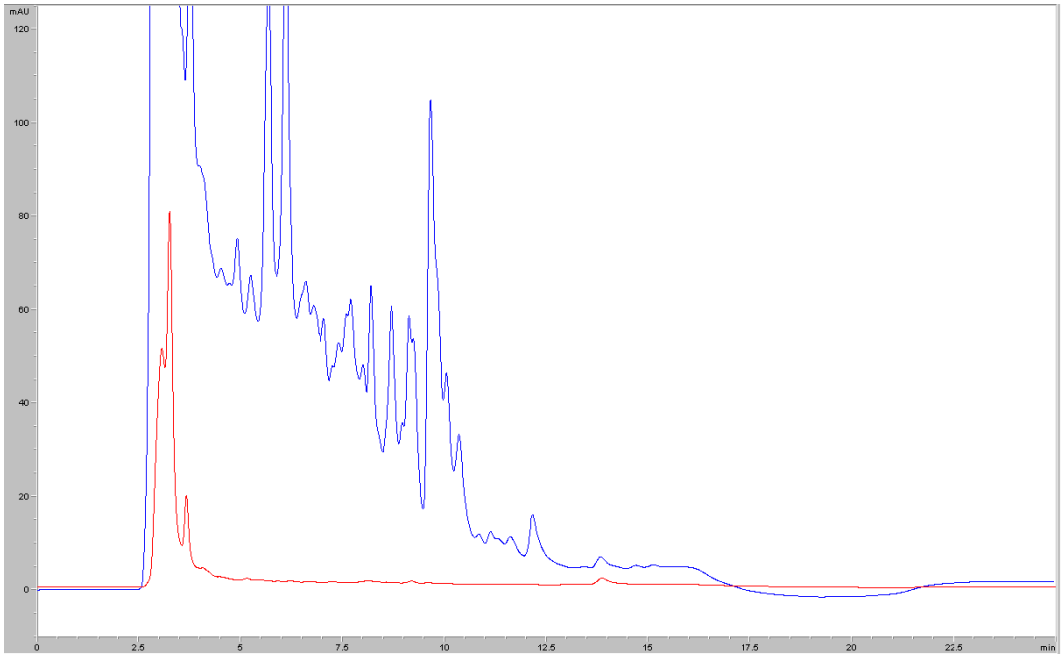

Figure 3. Chromatogram of a blank liver extract with no GPC cleanup. UV detection is at 280 nm and fluorescense at excitation wavelength of 318 nm and emission wavelength at 390 nm.

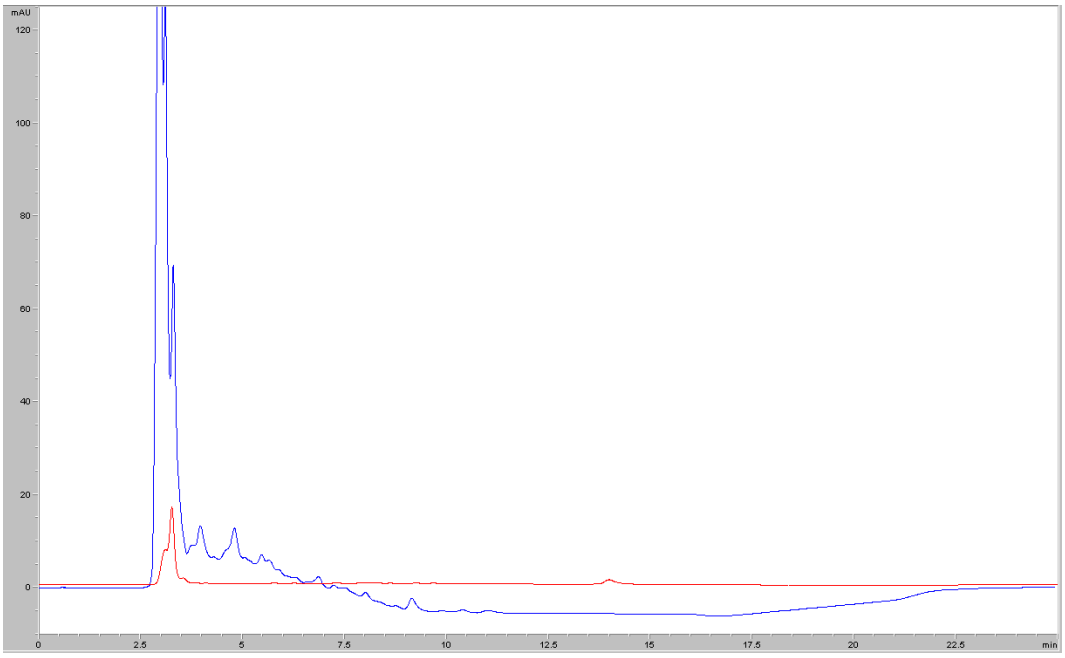

Figure 4. Chromatogram of a blank liver extract with GPC cleanup. The scale is the same as that of Figure 2.

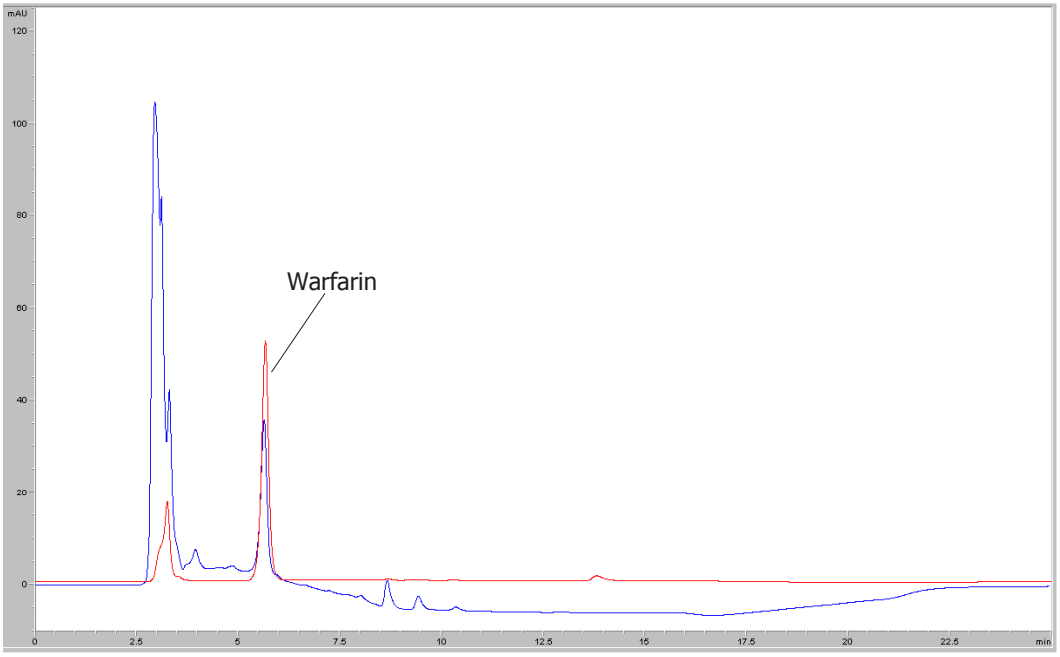

Figure 5. Chromatogram of a liver extract fortified with 6 ppm of warfarin (5.8 min).

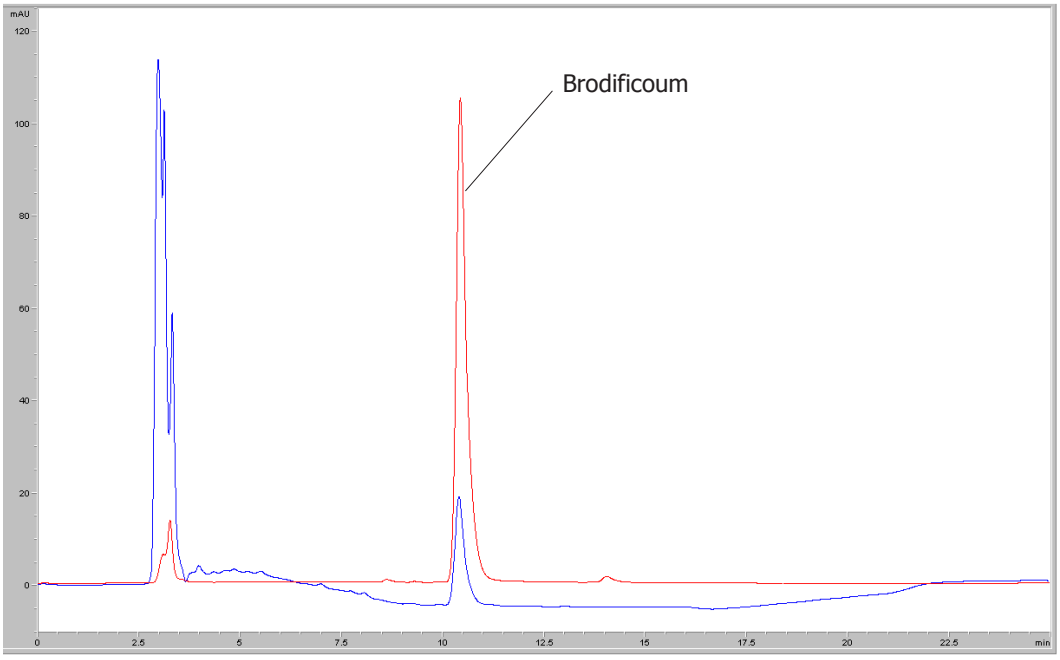

Figure 6. Chromatogram of a liver extract fortified with 3.6 ppm of brodificoum.

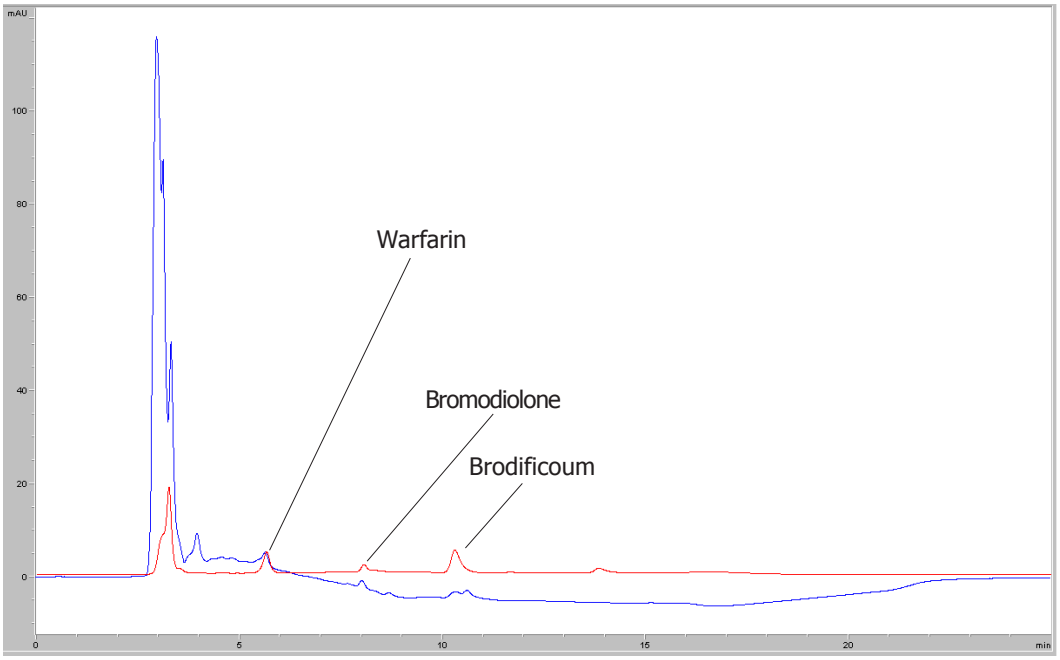

Figure 7. Chromatogram of a liver extract fortified with 500 ppb of warfarin (5.8 min), 100 ppb of bromodiolone, and 150 ppb brodificoum (10.4 min).

Conclusion

Gel permeation chromatography (GPC) efficiently removed lipids and other high molecular weight interfering compounds from liver extracts prior to analysis for hydroxycoumarins. No additional cleanup procedure was needed. Warfarin and brodificoum were readily detected at levels of 500 ppb. The SP 2000 GPC system is an effective and simple system that makes GPC affordable for laboratories with limited budgets and small sample throughput. For information on published methods for GPC cleanup and extraction of anticoagulants from liver and other tissues, see the references listed below.

Acknowledgment

OI Analytical wishes to thank and acknowledge Dr. Sean Linder and Cheryl Fossler of the Arkansas Livestock Commission, Veterinary Diagnostic Laboratory for their contributions to this study.

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Application Note 20540204

Using the GPC AutoPrep 2000 System for Cleanup of Olive Oil Prior to Pesticides Analysis by GC/XSD or GC/PFPD

Abstract

Gel Permeation Chromatography (GPC) using polystyrene divinylbenzene beads is widely employed for removing higher molecular weight coextractives from food and environmental matrices prior to gas chromatography (GC), GC/mass spectrometry (MS), or high-performance liquid chromatography (HPLC) analysis for pesticides and other environmental contaminants. The OI Analytical GPC AutoPrep 2000 (Figure 1) totally automates separation of interfering coextractives from target analytes. This system uses an autosampler for injecting sample extracts and collecting cleaned fractions into a variety of sample collection vessels.

This study evaluates GPC AutoPrep 2000 system performance for the cleanup of olive oil prior to analyzing for pesticides. Methylene chloride:hexanes (50:50 v/v) extracted pesticides from olive oil. This solvent mixture was also used as the mobile phase during the GPC cleanup procedure. After GPC cleanup, a Labconco RapidVap® N2 System (Figure 2) evaporated sample extracts to dryness. The samples were reconstituted in appropriate solvent for analysis. Sample analysis used a GC with a halogen-specific detector (XSD™) for chlorinated pesticides and a pulsed flame photometric detector (PFPD) for the phosphorus- and sulfur-containing pesticides. Recoveries for several pesticides were determined.

Keywords

Envirobeads
EnviroSep-ABC
Gel Permeation Chromatography
GPC
GPC AutoPrep 2000
Olive Oil
Pesticides
RapidVap

Figure 1. OI Analytical GPC AutoPrep 2000

Presented at the 2004 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Chicago, IL March 7–12, 2004

REGISTERED COMPANY ISO 9001 SGS

Introduction

GPC is a size-exclusion cleanup process that uses organic solvents and a hydrophobic gel (primarily a cross-linked divinylbenzene-styrene copolymer) to separate macromolecules. GPC is a highly effective post-extraction cleanup method for removing high-molecular-weight interferences such as lipids, polymers, proteins, pigments, natural resins, and cellular components from sample extracts prior to analysis.

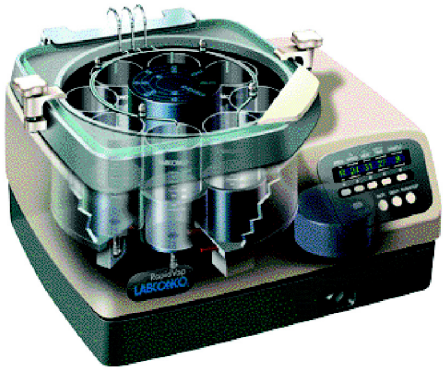


Figure 2. RapidVap N2 Evaporation System

Post-extraction GPC cleanup is used extensively for preparing food and environmental samples prior to GC, GC/MS, or HPLC analysis for pesticides, fungicides, semivolatiles, and other environmental contaminants. The FDA Pesticide Analytical Manual (Volume 1, Section 304) recommends separating fats in oils and fatty foods from analytes by GPC prior to analyzing pesticides and PCBs by GC or GC/MS. If sample extracts with high lipid content are injected onto a GC or HPLC column, the injection port and column head can easily become contaminated, resulting in recovery losses and poor chromatography.

The OI Analytical GPC AutoPrep 2000 automates the GPC cleanup process. The GPC AutoPrep 2000 uses an autosampler for both injecting sample extracts and collecting cleaned fractions into a variety of collection vessels. The system features a modular design and electronic valve actuation. A syringe pump picks up the sample extract, and a wash pump and rinse station eliminate sample carryover. The system is controlled via a PC using WinSEP™ GPC control software, a Windows®-based program, which includes an extensive quality monitoring program.

This study evaluates the GPC AutoPrep 2000 performance for cleanup of olive oil prior to analyzing for pesticides. Sample analysis used a GC with a halogen specific detector (XSD) for chlorinated pesticides and a pulsed flame photometric detector (PFPD) for the phosphorus- and sulfur-containing pesticides. XSD and PFPD detectors were chosen for their high degree of selectivity and sensitivity for analytes of interest.

Experimental

Materials

All solvents were distilled in glass suitable for HPLC, GC, pesticide residue analysis, and spectrophotometry. All chemicals were ACS reagent-grade quality or greater. GPC calibration standards were prepared according to USEPA Method 3640A and contained corn oil, bis(2-ethylhexyl) phthalate, methoxychlor, perylene, and sulfur. Pesticide standards were obtained from OI Analytical (College Station, TX) or Restek (Bellfonte, PA). Extra-virgin olive oil was obtained from Bertolli USA (Englewood Cliffs, NJ). Olive oil working solution was prepared by adding olive oil to a mixture of 50:50 methylene chloride:hexanes to a concentration of 0.1 g/mL of olive oil. The olive oil working solution was fortified with varying concentrations of either FAPAS® Series 9 organophosphate (OP) pesticide mix one, g-BHC (lindane), or OI Analytical Pesticide Standard (part number 234023).

GPC Cleanup

Pesticide cleanup of olive oil by GPC used the GPC AutoPrep 2000 equipped with a 700 mm x 25 mm glass column containing 33 g of Envirobeads® S-X3 resin. The system used a 5-ml sample loop and a flow rate of 5 mL/minute with a 50:50 mixture of methylene chloride:hexanes as the mobile phase. The GPC column was calibrated by either the method of eluting fat and pesticides as outlined in the FDA Pesticide Analytical Manual, Volume1, Section 304 C5, or by using a calibration standard and UV detector as described in USEPA Method 3640A. Fractions were collected every two minutes using the FDA method (Figure 3). These fractions were then evaporated to dryness and either weighed (olive oil blanks) or reconstituted in ethyl acetate and injected on the GC. Calibration using USEPA Method 3640A employed a GPC calibration standard (as described above), an OI Analytical UVD-1000 UV Detector set at 254 nm, and WinSep software (Figure 4). Based on the UV trace, column eluate collection began just before bis(2-ethyhexyl) phthalate elution and after corn oil elution (Figure 5).

Eluate collection stopped after perylene elution. After GPC cleanup, the collected fractions were evaporated using a Labconco RapidVap N2 and reconstituted in appropriate solvent for GC analysis.

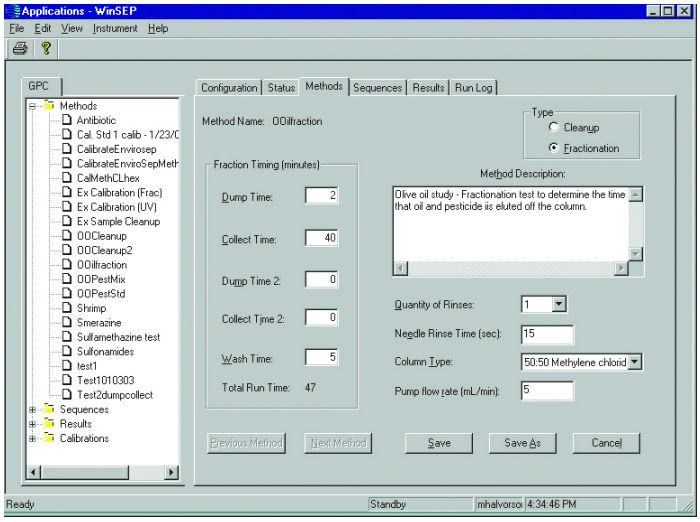


Figure 3. WinSEP Methods page showing fractionation parameters for eluting oil and pesticide

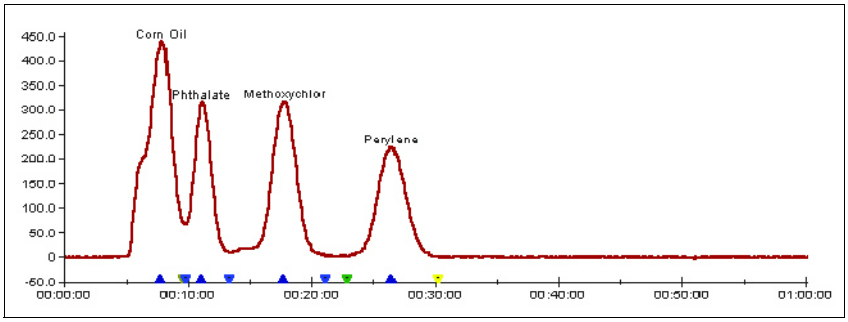


Figure 4. Typical chromatogram of a USEPA calibration standard using an Envirobeads SX-3 column with a 50:50 methylene chloride:hexane mobile phase at 5 mL/minute, WinSEP software, and the UVD-1000 UV detector (254 nm, 1.000 AUFS)

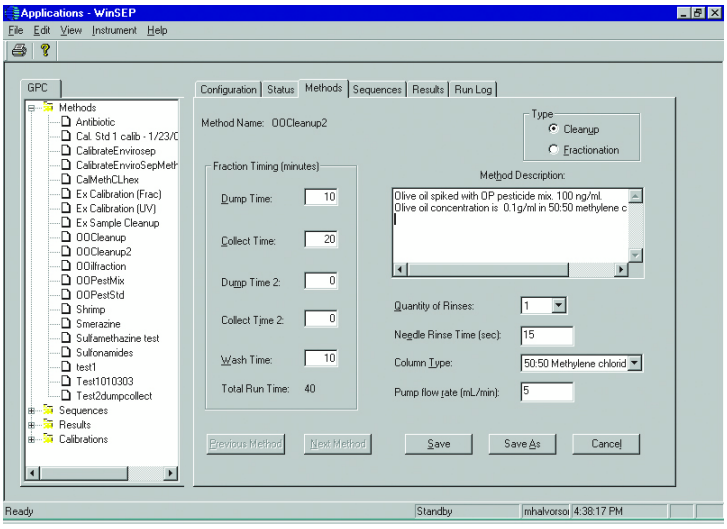


Figure 5. WinSEP Methods page showing fractionation parameters for olive oil cleanup

GC Analysis

Pesticides were analyzed using an Agilent® Series 6850 GC with an OI Analytical Model 5360 Halogen Specific Detector (XSD) and Agilent HP5 column (30 m x 0.320 mm, 0.25-µm phase) and an Agilent Series 6890 GC equipped with two OI Analytical Model 5380 Pulsed Flame Photometric Detectors (PFPD) configured for both phosphorus and sulfur modes and Agilent HP5 columns.

Results

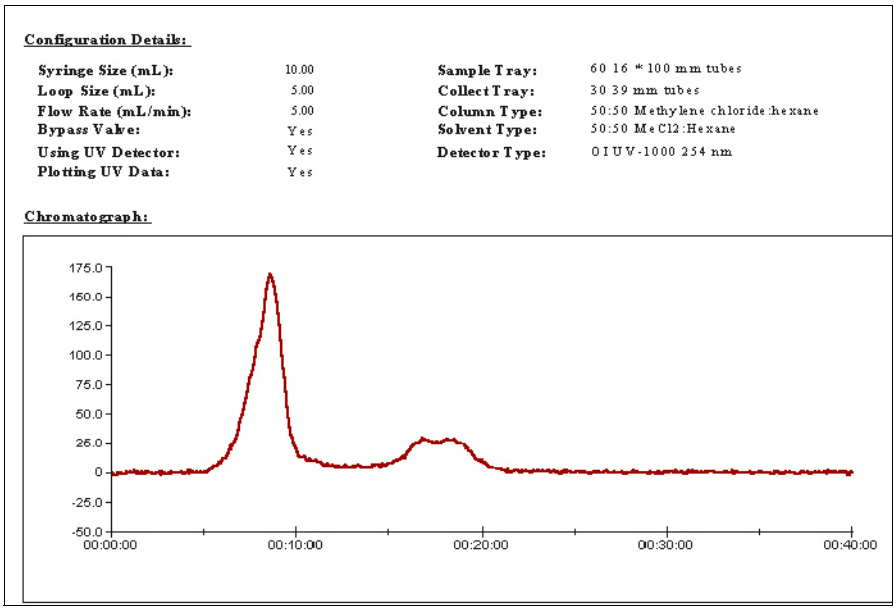


Figure 6. Chromatogram of olive oil using the Envirobeads S-X3 column with a 50:50 methylene chloride:hexane mobile phase at 5 mL/minute, WinSEP software, and the UVD-1000 UV detector (254 nm, 1.000 AUFS). The second, broader peak, which does not affect pesticide recovery, increases with olive oil's age and exposure to light.

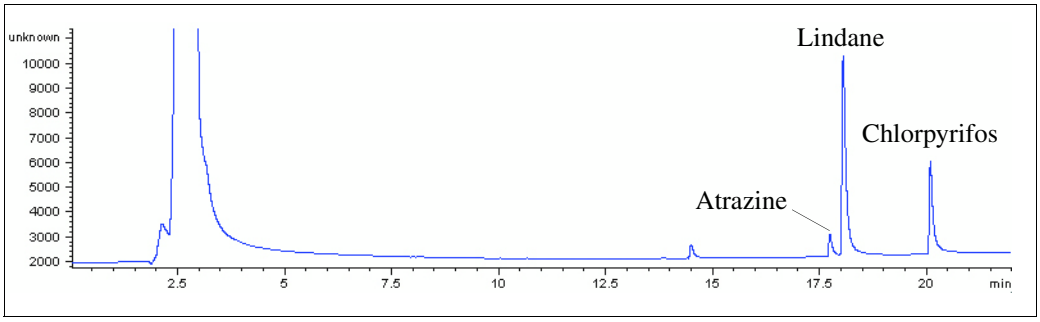


Figure 7. GC/XSD chromatogram of a mixed standard containing azobenzene, thimet (or phorate), atrazine, lindane, diazinon, and chlorpyrifos. The XSD only detects compounds contain halogens such as chlorine.

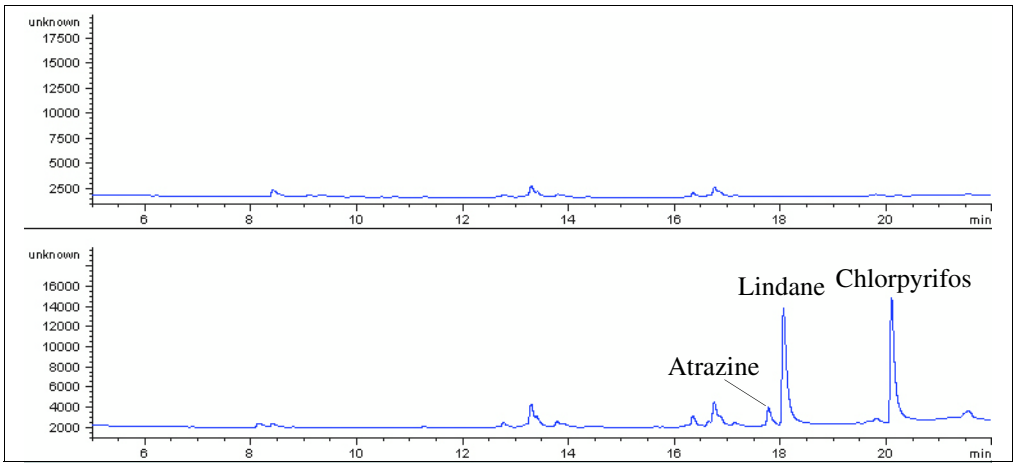
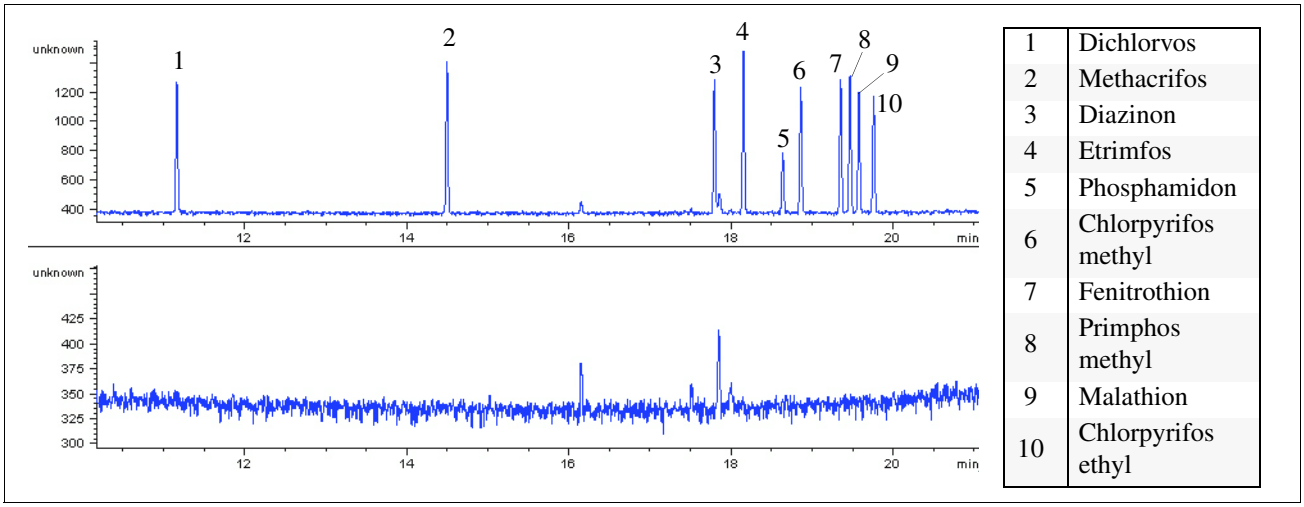


Figure 8. GC/XSD chromatograms of olive oil after GPC cleanup. The top chromatogram shows a sample without pesticide added. The bottom chromatogram shows a sample with a pesticides mixture added (azobenzene, thimet, atrazine, lindane, diazinon, and chlorpyrifos). Note the XSD's specificity for detecting halogen-containing compounds.



1	Dichlorvos
2	Methacrifos
3	Diazinon
4	Etrimfos
5	Phosphamidon
6	Chlorpyrifos methyl
7	Fenitrothion
8	Primphos methyl
9	Malathion
10	Chlorpyrifos ethyl

Figure 9. GC/PFPD chromatograms of olive oil after GPC cleanup. The top chromatogram shows a sample fortified with 50 ng/mL of FAPAS Series 9 OP mix 1. The bottom chromatogram shows an olive oil blank after GPC cleanup.

Table 1. Recovery of lindane (500 ng/mL) from olive oil (n = 3)

Amount Recovered (ng/mL)	**% Recovery**
429	85.8
432	86.4
467	93.4

Conclusion

The GPC AutoPrep 2000 was a highly effective and efficient tool for olive oil cleanup prior to pesticide analysis. No differences were observed in chromatographic results when calibrating the GPC column by fractionation versus using a GPC calibration standard and UV detector at 254 nm. Studies are currently underway to evaluate effectiveness of using nonchlorinated solvents for GPC cleanup of olive oil for pesticide analysis. The XSD provided sensitive and specific detection of chlorinated pesticides. The PFPD yielded excellent detection of organophosphate pesticides.

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Application Note 22410205

Evaluation of Several Solvents for Post-Extraction Gel Permeation Chromatography Cleanup for Pesticides in Animal Fats

Abstract

The Association of Analytical Chemists (AOAC) Official Method Number 984.21 describes a gel permeation chromatography (GPC) procedure for removing lipids from animal fats, followed by identification and measurement of chlorinated pesticides by gas chromatography (GC) with electrochemical (EC) detection. This method uses a chlorinated solvent, methylene chloride, and is only valid for use with chlorinated pesticides. This study investigates alternatives to methylene chloride for GPC cleanup of animal fats, as well as expanding the method's effectiveness to organophosphorus pesticides.

Two grams of either rendered beef, pork, or poultry fat were placed in a volumetric flask, fortified with pesticides, and diluted with appropriate GPC solvent to a 10-mL final volume. GPC cleanup used the OI Analytical GPC AutoPrep 2000 system (Figure 1) with a 5-mL sample loop, a column packed with Envirobeads® S-X3, and a variety of elution solvents. The RapidVap® N2 system (Figure 2) evaporated extracts to dryness before and after GPC cleanup. Sample extracts were analyzed by GC using the Halogen Specific Detector (XSD™) for chlorinated pesticides and Pulsed Flame Photometric Detector (PFPD) for organophosphorus pesticides. Recoveries of pesticides were determined.

Keywords

Fats
Gel Permeation Chromatography
GPC AutoPrep 2000
Pesticides
PFPD
RapidVap N2 System
XSD

Figure 1. GPC AutoPrep 2000 Gel Permeation Chromatography System

Presented at the 2005 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Orlando, FL February 27–March 4, 2005

REGISTERED COMPANY ISO 9001 SGS

Introduction

GPC has been extensively used as an effective post-extraction cleanup procedure for removing high-molecular-weight interferences from sample extracts. GPC cleanup eliminates high-molecular-weight compounds such as lipids, pigments, proteins, and polymers from organic extracts prior to analysis by GC, GC/MS, HPLC, or LC/MS. This sample cleanup method is based on GPC's ability to separate molecules in solution by size.

The Official Methods of Analysis of AOAC International (17th edition) recommends GPC cleanup for removing beef, poultry, or swine fat prior to analysis for organochlorine residues by GC (AOAC Official Method 984.21). Animal fat is dissolved in 1:1 methylene chloride/cyclohexane. A column packed with 60 g of Envirobeads SX-3 separates pesticides with a 1:1 methylene chloride/cyclohexane elution solvent and flow rate of 5 mL/minute. This method is only specified for organochlorine pesticide residues. However, evaluating fats for organophosphorus pesticides is also common. Also, this method uses methylene chloride, a halogenated solvent associated with health risks, as well as higher disposal costs. This study evaluates the effectiveness of different solvent mixtures for separating animal fats for both organochlorine and organophosphorus classes of pesticides.

This study used the OI Analytical GPC AutoPrep 2000 system, which automates the GPC cleanup process. The GPC AutoPrep 2000 system uses an autosampler for both injecting the sample extract and collecting cleaned fractions into a variety of collection vessels. The system features a modular design and electronic valve actuation. A syringe pump picks up the sample extract, and a wash pump and rinse station eliminate sample carryover. A PC controls the system using WinSEP™ GPC control software, a Windows®-based program that includes extensive quality monitoring capabilities.

Figure 2. RapidVap N2 Evaporation System

A RapidVap N2 system evaporated extracts to dryness after GPC cleanup. Analysis was performed by GC with an XSD for chlorinated pesticides and PFPD for phosphorus- and sulfur-containing pesticides. The XSD and PFPD were chosen for their high degrees of selectivity and sensitivity for the analytes of interest.

Experimental

Materials

All solvents were distilled in glass suitable for HPLC, GC, pesticide residue analysis, and spectrophotometry. All chemicals were ACS reagent grade quality or better. GPC calibration standards were prepared according to USEPA Method 3640A and contained corn oil, bis(2-ethylhexyl)phthalate, methoxychlor, perylene, and sulfur. Pesticide standards were obtained from OI Analytical (College Station, TX) or Restek (Bellfonte, PA). Beef, pork, and poultry fat were obtained from Readfield Meats (Bryan, Texas) and rendered before use.

Sample Preparation

Weigh either 2 g or 1.2 g of rendered fat into a 10-mL flask. Fortify with pesticide standards in the appropriate GPC mobile phase and dilute to 10 mL with the appropriate solvent. Mix thoroughly and filter with a Whatman 0.45-µm TF filter if particulate matter is visible.

GPC Cleanup

Pesticide cleanup of the animal fats used the GPC AutoPrep 2000 equipped with a 700 mm x 25 mm glass column containing the following weights of Envirobeads S-X3 resin and mobile phases (Table 1).

Table 1. Envirobeads S-X3 resin weights and mobile phases

Resin Weight	Mobile Phase (Eluent)
70 g	100% Methylene chloride
35 g	1:1 Methylene chloride/hexane
60 g	1:1 Methylene chloride/cyclohexane
60 g	1:1 Cyclohexane/ethyl acetate
50 g	7:3 Ethyl acetate/cyclopentane

The system used a 5-mL sample loop and flow rate of 5 mL/minute. The GPC column was calibrated by either the method of eluting fat and pesticides per the FDA Pesticide Analytical Manual, Volume 1, Section 304 C5, or by using a calibration standard and UV detector as described in USEPA Method 3640A.

- For calibration using the FDA method, fractions were collected every three minutes. These fractions were evaporated to dryness and either weighed or reconstituted in ethyl acetate, and injected onto the GC.
- Calibration with USEPA Method 3640A used a GPC calibration standard (as described above), OI Analytical UVD-1000 UV Detector set at 254 nm, and WinSep software. Based on the UV trace, column eluate collection was started just before bis(2-ethyhexyl)phthalate elution and after corn oil elution. Eluate collection was stopped after perylene elution.

After GPC cleanup, the RapidVap N2 evaporated the collected fractions. The samples were reconstituted in the appropriate solvent for GC analysis.

GC Analysis

Pesticides were analyzed with an Agilent® Series 6890 GC with an OI Analytical Model 5360 XSD and Agilent HP5 column (30 m x 0.320 mm, 0.25-µm phase), or OI Analytical Model 5380 PFPD configured for phosphorus mode and an Agilent HP 5 column or Restek Rtx®-35MS column.

Results

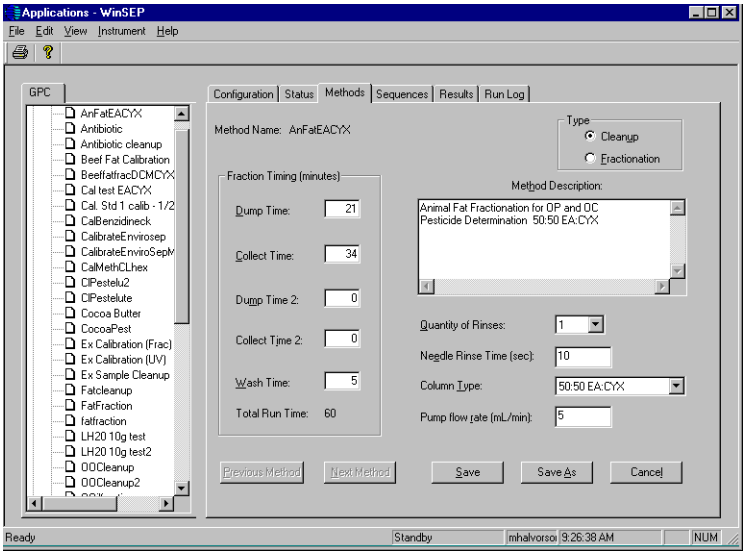


Figure 3. WinSEP Methods page showing typical parameters for GPC cleanup of animal fats

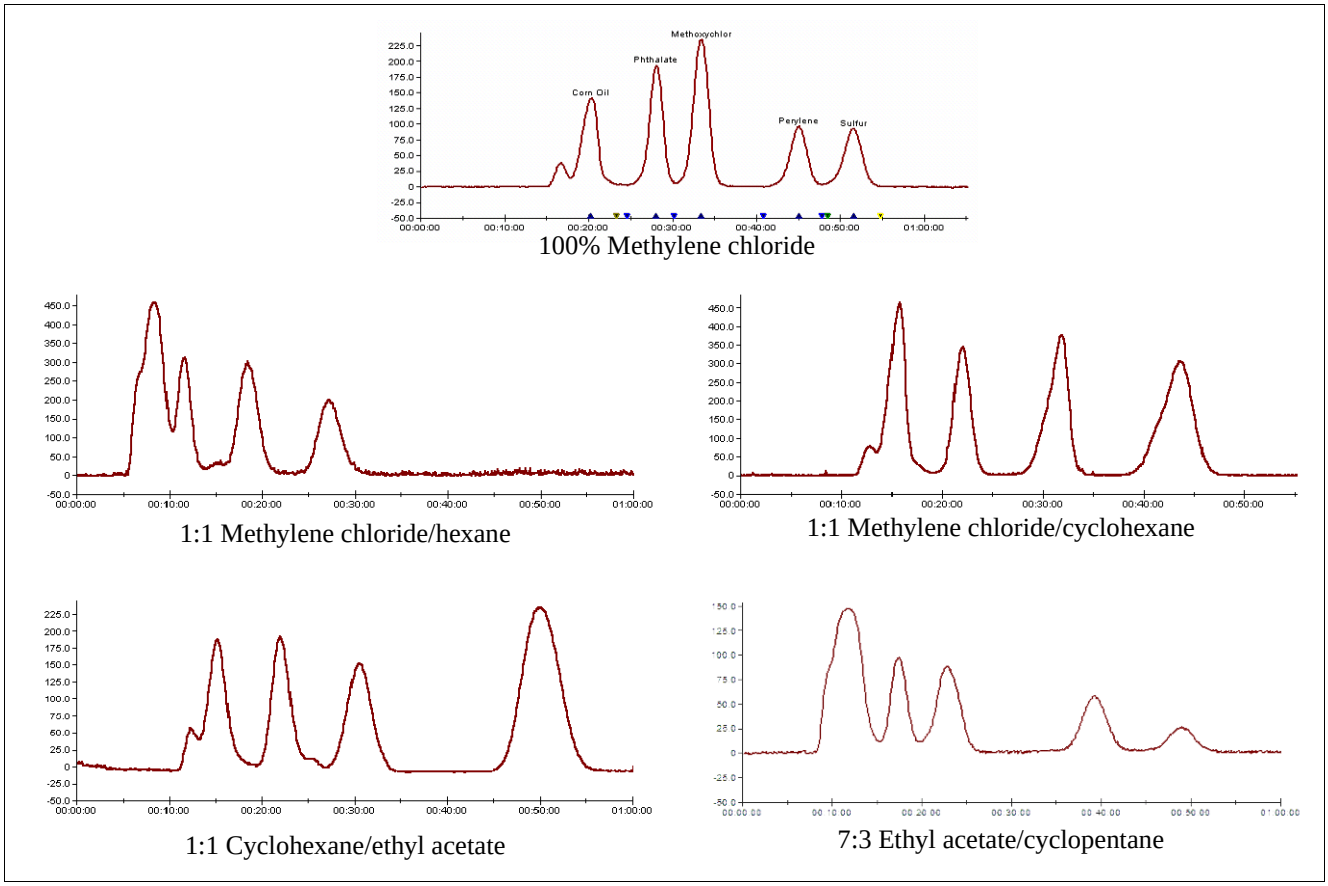


Figure 4. Chromatograms of a USEPA Method 3640A calibration standard using different eluents at a 5 mL/minute flow rate, WinSEP software, and an OI Analytical UV Detector (254 nm, 1.000 AUFS)

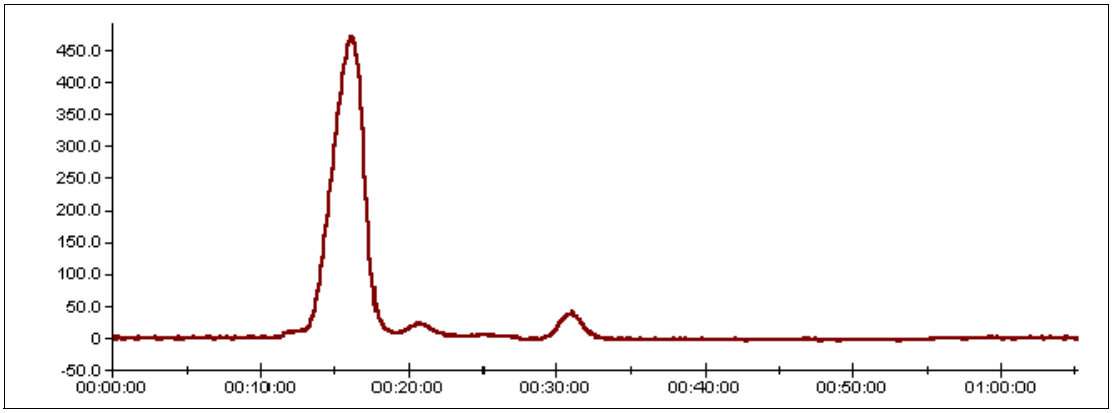


Figure 5. Chromatogram of 0.6 g of beef fat spiked with 500 ng/mL organochlorine pesticide mix during GPC cleanup with UV detection (254 nm). The smaller peak is an organochlorine pesticide.

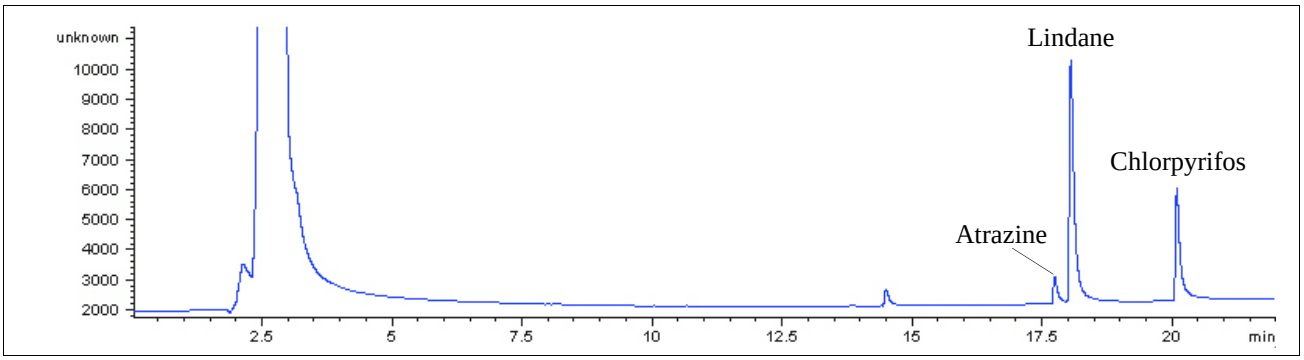
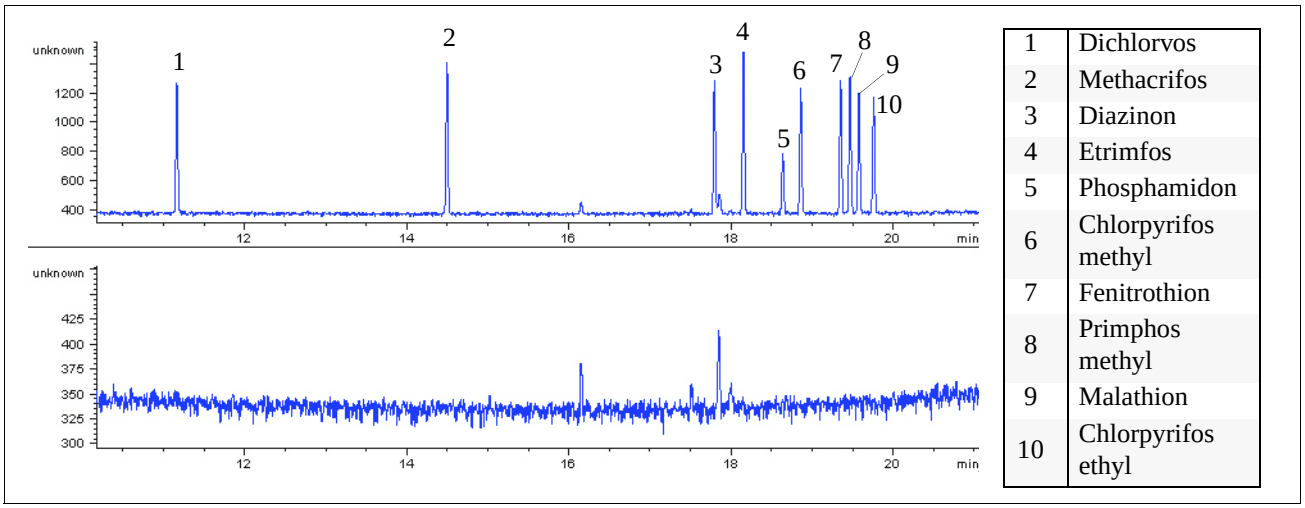


Figure 6. GC/XSD chromatogram of a mixed standard containing azobenzene, thimet (or phorate), atrazine, lindane, diazinon, and chlorpyrifos. The XSD only detects compounds containing halogens such as chlorine.



1	Dichlorvos
2	Methacrifos
3	Diazinon
4	Etrimfos
5	Phosphamidon
6	Chlorpyrifos methyl
7	Fenitrothion
8	Primphos methyl
9	Malathion
10	Chlorpyrifos ethyl

Figure 7. GC/PFPD chromatograms of poultry fat after GPC cleanup. The top chromatogram shows a sample fortified with 50 ng/mL of FAPAS Series 9 OP mix 1. The bottom chromatogram shows a poultry fat blank after GPC cleanup.

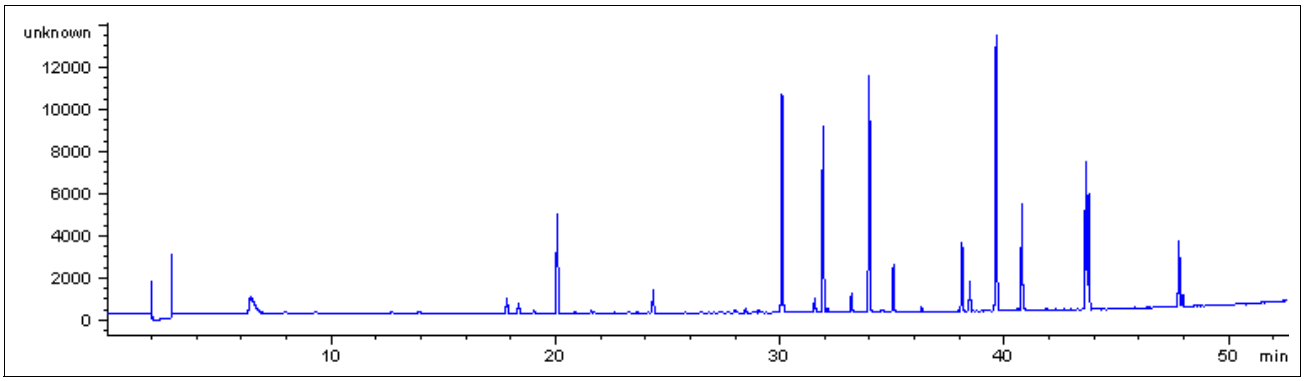


Figure 8. A chromatogram of beef fat fortified with 400 ng/mL of OP pesticide calibration mix A using the PFPD in phosphorus mode

Table 2. Percent recovery results of fortified poultry, pork, and beef lipid samples (n = 3) using 1:1 ethyl acetate/cyclohexane mobile phase

Compound	Poultry	Pork	Beef
Diazinon	92.6	99.9	89.0
Dichlorvos	111.2	83.7	102.0
Malathion	112.5	102.2	96.9

Conclusion

- All eluents tested, except 100% methylene chloride, were effective in removing up to 1g of fat from sample extracts. Methylene chloride was only effective at removing less than 0.6 g of fat.

- 1:1 Methylene chloride/cyclohexane, 1:1 ethyl acetate/cyclohexane, and 7:3 ethyl acetate/cyclopentane were the most effective solvents at removing fats and recovering both organophosphate and organochlorine pesticides.

- Both 1:1 ethyl acetate and 7:3 ethyl acetate/ cyclohexane are safer and effective alternatives to eluents containing methylene chloride.

- 7:3 Ethyl acetate/cyclopentane has advantages over other eluents such as faster evaporation times, high fat solubility, good recoveries of both pesticide classes, safer solvent, and no requirement for solvent exchange prior to analysis by GC/PFPD or GC/XSD.

- Load the column with 0.6 g of fat for the most effective analysis of both organophosphate and organochlorine pesticides.

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Optimization of Evaporation and Concentration Parameters Prior to Analysis for Pesticides by GC/XSD or GC/PFPD

Application Note 22420305

Keywords
Evaporation
Halogen Specific Detector
Kuderna-Danish
Organochlorine
Organophosphorus
Pesticide
PFPD
Pulsed Flame Photometric Detector
RapidVap
TurboVap
XSD

Presented at the 2005 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Orlando, FL February 27–March 4, 2005

Abstract

Analyzing for pesticides usually incorporates several steps, including extracting the analytes from the sample matrix, concentrating the extract, post-extraction cleanup, concentration and solvent exchange, and quantitative determination of the analytes of interest. The type of method used for sample concentration often affects analyte recovery. The purpose of this study was to optimize operating conditions of several commercially-available evaporation products for maximal recovery of pesticides prior to analysis by gas chromatography (GC). Sample extracts and solvents or solvent mixtures were fortified with both chlorinated and organophosphorus pesticides and then placed on a Labconco RapidVap® N2 system, RapidVap Vacuum system, Caliper (formerly Zymark) TurboVap® II system, or rotary evaporator (Figure 1). After concentration, extracts and standards were analyzed by GC with a Pulsed Flame Photometric Detector (PFPD) for organophosphorus pesticides, or with a Halogen Specific Detector (XSD™) for chlorinated pesticides. Recovery was then determined. The advantages and disadvantages of each type of method are also discussed.

Introduction

Evaporation is an important step for extracting pesticides from environmental samples such as soil and sediments, wastewater, animal and plant tissue, and foods. A variety of specific instruments have been developed for sample concentration by evaporation including Kuderna-Danish concentrators, rotary evaporators, nitrogen blowdown evaporators, and vacuum evaporators. Pesticide extraction often requires evaporation of greater than 100 mL of solvent after the extraction step, as well as after many post-extraction cleanup steps such as gel permeation chromatography cleanup. Although Kuderna-Danish concentrators and rotary evaporators are very efficient for evaporation, they are limited to only handling one sample at a time. Several commercial evaporator systems can handle volumes greater than 100 mL and evaporate multiple samples. The Labconco RapidVap N2 system and RapidVap Vacuum system can evaporate up to eight samples with a capacity 450 mL of solvent. The Caliper TurboVap II system can evaporate up to six samples with a volume of 200 mL. This study evaluates the efficiency of these three systems for evaporation and concentration of extracts containing organochlorine and organophosphorus pesticides.

Experimental

Materials

All solvents were distilled in glass that is suitable for HPLC, GC, pesticide residue analysis, and spectrophotometry. Pesticide standards were obtained from OI Analytical (College Station, TX) or Restek Corporation. (Bellfonte, PA). Samples were evaporated in ether 200-mL TurboVap tubes with 1-mL stems, 450-mL RapidVap tubes with 1.5-mL stems or 450-mL RapidVap tubes with flat bottoms.

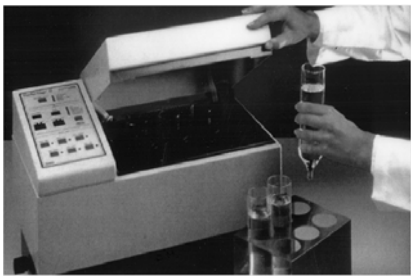
Evaporation and Concentration

Evaporation tubes were filled with 200 mL of solvent spiked with a 500 ng/mL chlorinated pesticide standard or 400 ng/mL organophosphate pesticide standard. Both the TurboVap II and the RapidVap systems were preheated to 40 °C, and the tubes were placed in the instruments for evaporation. Solvents containing methylene chloride were evaporated to dryness and 1 mL of either hexane or ethyl acetate was added prior to GC analysis. Other solvents were concentrated to 1 mL and then injected into the GC. The RapidVap N2 system used a mixing speed of 55% with a nitrogen pressure of 12 psi for all solvents. The RapidVap vacuum system used a of mixing speed of 20–30% and the vacuum was set from 120–400 mbar depending on the solvent. Great care was taken to prevent sample bumping. The temperature for both the RapidVap N2 system and TurboVap II system was set at 40 °C for solvents containing methylene chloride. For solvents containing ethyl acetate, the TurboVap II was set at its maximum temperature of 56 °C and the RapidVap N2 system was set at 75 °C.

GC Analysis

Pesticides were analyzed using an Agilent® 6890 GC with an OI Analytical Model 5360 XSD and Agilent HP 5 column (30 m x 0.320 mm, 0.25-µm phase), and an Agilent 6890 GC equipped with an OI Analytical Model 5380 PFPD configured for phosphorus mode and Agilent HP 5 column or Restek Rtx®-35MS column.

RapidVap Vacuum system



TurboVap II system

RapidVap N2 system

Figure 1. Evaporation systems used for sample concentration

Results

Table 1. Time in minutes to evaporate 200 mL of solvent using three different automated evaporation techniques. Heating temperature was 40 °C for solvents containing methylene chloride, 75 °C for solvents containing ethyl acetate on the RapidVap N2 system, and 56 °C on the TurboVap II system.

Solvent	Time (min) RapidVap N2	Time (min) RapidVap Vacuum	Time (min) TurboVap II
Methylene chloride (Dichloromethane)	48	58	80
1:1 Cyclohexane:ethyl acetate	35	45	57
1:1 Cyclohexane:methylene chloride	60	67	Not determined
7:3 Ethyl acetate:cyclopentane	38	49	60
1:1 Hexane:methylene chloride	41	53	64

Table 2. Estimated sample capacity and relative labor cost comparison per eight-hour shift to evaporate 200 mL of methylene chloride using different evaporation methods

Instrument	Sample Capacity Per Eight-Hour Shift	Estimated Operating Labor Costs
RapidVap N2 system	61	Low
RapidVap Vacuum system	49	Low to moderate
TurboVap II system	36	Low
Rotary evaporator	12	Moderate to high
Kuderna-Danish apparatus	10	Moderate to high

Table 3. Recovery of 500 ppb of lindane from 200 mL of methylene chloride evaporated to dryness and reconstituted in 1 mL of hexane for injection for GC/XSD analysis. The recovery range is for all solvent types tested. Solvents not containing methylene chloride were concentrated to 1 mL prior to injection for GC/XSD analysis.

Instrument	Recovery	Recovery Range for All Solvent Types
RapidVap N2 system	90.4%	75–125%
RapidVap Vacuum system	89.7%	53–112%
TurboVap II system	88.4%	68–20%

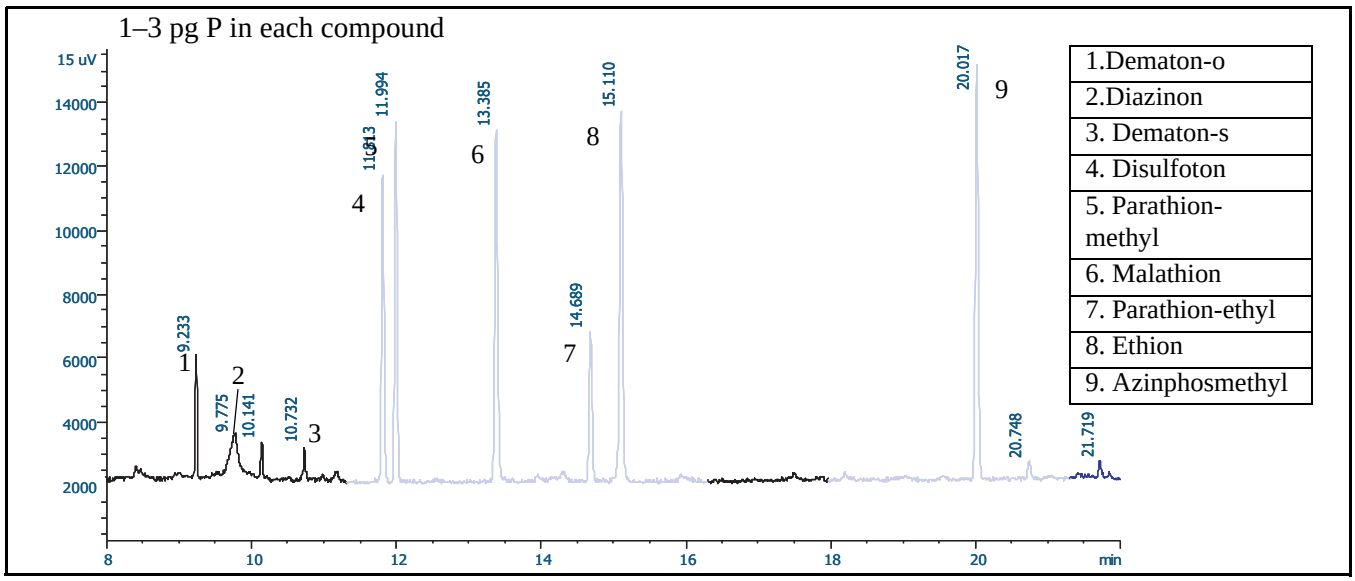


Figure 2. A mixture of nine different organophosphorus pesticides using the PFPD in phosphorus mode

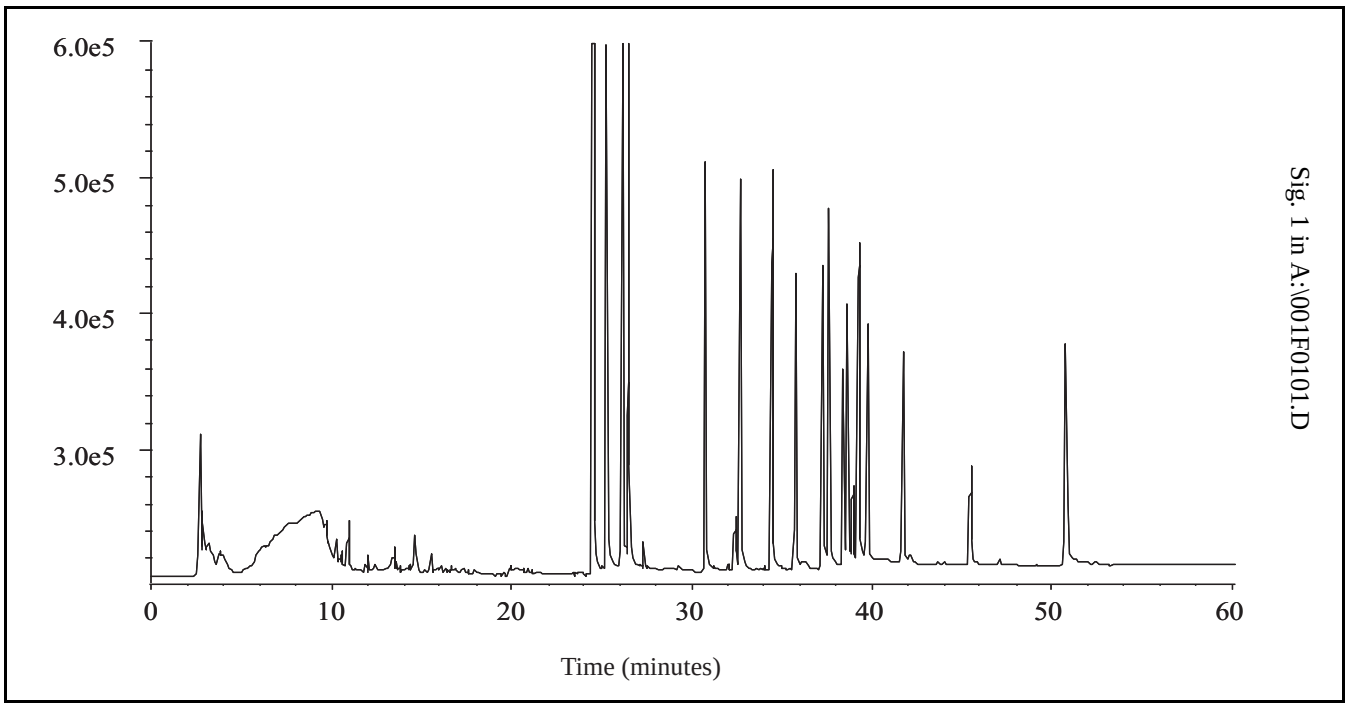
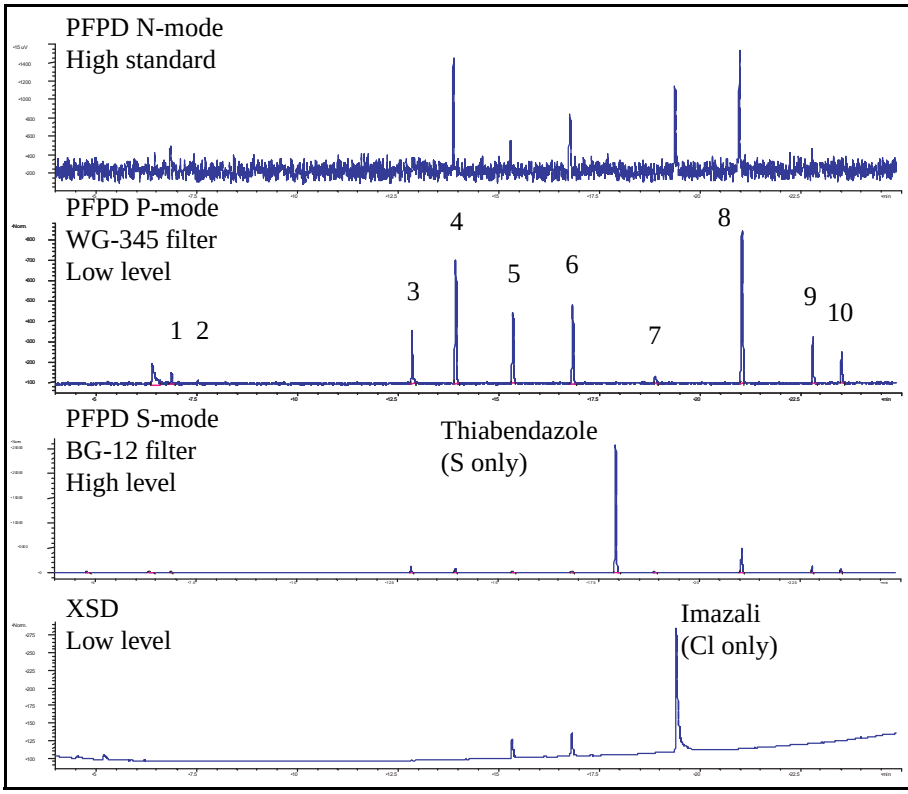


Figure 3. Organochlorine pesticide mix AB on an Agilent 6890 GC with HP-5 column and Model 5360 XSD



Compound	Low Conc. (ppb)
1. Methamidophos	100
2. Acephate	60
3. Dimethoate	60
4. Diazinon	60
5. me-Chlorpyrifos	50
6. Chlorpyrifos	50
7. Disulfoton sulfone	50
8. Ethion	60
9. Phosmet	100
10. Azinphos-me	150

Figure 4. Pesticide mix on both GC/PFPD with different filters for P- or S-containing pesticides and GC/XSD for halogenated pesticides

Conclusions

- Preliminary data indicates all three multisample evaporation systems are effective for recovery of chlorinated pesticides. Work continues to evaluate the effectiveness of these systems for organophosphate pesticides.

- The Labconco RapidVap N2 system was the fastest evaporation system, had a higher sample capacity, and the lowest cost per sample compared to the other two systems.

- The RapidVap Vacuum system requires more monitoring than the other systems to prevent sample bumping. However, its advantages over the other two systems include different sample tubes sizes and an optional cold trap to reduce solvent vapors.

- Preliminary results indicate the best and the most accurate recoveries for both chlorinated and organophosphate pesticides occurred when solvent exchange was not necessary prior to injection for GC/XSD or GC/PFPD analysis.

- All three systems save labor costs compared to traditional Kuderna-Danish concentrators and rotary evaporators.

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Application Note 22420405

Optimization of Evaporation and Concentration Parameters Prior to Analysis for Pesticides by GC/XSD or GC/PFPD

Abstract

Analyzing for pesticides usually incorporates several steps, including extracting the analytes from the sample matrix, concentrating the extract, post-extraction cleanup, concentration and solvent exchange, and quantitative determination of the analytes of interest. The type of method used for sample concentration often affects analyte recovery. The purpose of this study was to optimize operating conditions of several commercially-available evaporation products for maximal recovery of pesticides prior to analysis by gas chromatography (GC). Sample extracts and solvents or solvent mixtures were fortified with both chlorinated and organophosphorus pesticides and then placed on a Labconco RapidVap® N2 system, RapidVap Vacuum system, Caliper (formerly Zymark) TurboVap® II system, or rotary evaporator (Figure 1). After concentration, extracts and standards were analyzed by GC with a Pulsed Flame Photometric Detector (PFPD) for organophosphorus pesticides, or with a Halogen Specific Detector (XSD™) for chlorinated pesticides. Recovery was then determined. The advantages and disadvantages of each type of method are also discussed.

Keywords

Evaporation
Halogen Specific Detector
Kuderna-Danish
Organochlorine
Organophosphorus
Pesticide
PFPD
Pulsed Flame Photometric Detector
RapidVap
TurboVap
XSD

Introduction

Evaporation is an important step for extracting pesticides from environmental samples such as soil and sediments, wastewater, animal and plant tissue, and foods. A variety of specific instruments have been developed for sample concentration by evaporation including Kuderna-Danish concentrators, rotary evaporators, nitrogen blowdown evaporators, and vacuum evaporators. Pesticide extraction often requires evaporation of greater than 100 mL of solvent after the extraction step, as well as after many post-extraction cleanup steps such as gel permeation chromatography cleanup. Although Kuderna-Danish concentrators and rotary evaporators are very efficient for evaporation, they are limited to only handling one sample at a time. Several commercial evaporator systems can handle volumes greater than 100 mL and evaporate multiple samples. The Labconco RapidVap N2 system and RapidVap Vacuum system can evaporate up to eight samples with a capacity 450 mL of solvent. The Caliper TurboVap II system can evaporate up to six samples with a volume of 200 mL. This study evaluates the efficiency of these three systems for evaporation and concentration of extracts containing organochlorine and organophosphorus pesticides.

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REGISTERED COMPANY ISO 9001 SGS

Experimental

Materials

All solvents were distilled in glass that is suitable for HPLC, GC, pesticide residue analysis, and spectrophotometry. Pesticide standards were obtained from OI Analytical (College Station, TX) or Restek Corporation. (Bellfonte, PA). Samples were evaporated in ether 200-mL TurboVap tubes with 1-mL stems, 450-mL RapidVap tubes with 1.5-mL stems or 450-mL RapidVap tubes with flat bottoms.

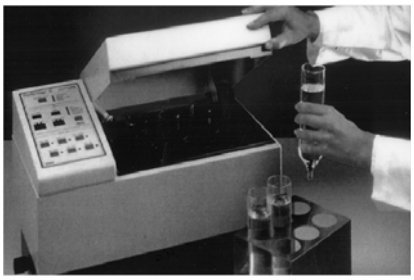
Evaporation and Concentration

Evaporation tubes were filled with 200 mL of solvent spiked with a 500 ng/mL chlorinated pesticide standard or 400 ng/mL organophosphate pesticide standard. Both the TurboVap II and the RapidVap systems were preheated to 40 °C, and the tubes were placed in the instruments for evaporation. Solvents containing methylene chloride were evaporated to dryness and 1 mL of either hexane or ethyl acetate was added prior to GC analysis. Other solvents were concentrated to 1 mL and then injected into the GC. The RapidVap N2 system used a mixing speed of 55% with a nitrogen pressure of 12 psi for all solvents. The RapidVap vacuum system used a mixing speed of 20–30% and the vacuum was set from 120–400 mbar, depending on the solvent. Great care was taken to prevent sample bumping. The temperature for both the RapidVap N2 system and TurboVap II system was set at 40 °C for solvents containing methylene chloride. For solvents containing ethyl acetate, the TurboVap II was set at its maximum temperature of 56 °C and the RapidVap N2 system was set at 75 °C.

GC Analysis

Pesticides were analyzed using an Agilent® 6890 GC with an OI Analytical Model 5360 XSD and Agilent HP 5 column (30 m x 0.320 mm, 0.25-µm phase), and an Agilent 6890 GC equipped with an OI Analytical Model 5380 PFPD configured for phosphorus mode and Agilent HP 5 column or Restek Rtx®-35MS column.

RapidVap Vacuum system



TurboVap II system

RapidVap N2 system

Figure 1. Evaporation systems used for sample concentration

Results

Table 1. Time in minutes to evaporate 200 mL of solvent using three different automated evaporation techniques. Heating temperature was 40 °C for solvents containing methylene chloride, 75 °C for solvents containing ethyl acetate on the RapidVap N2 system, and 56 °C on the TurboVap II system.

Solvent	Time (min) RapidVap N2	Time (min) RapidVap Vacuum	Time (min) TurboVap II
Methylene chloride (Dichloromethane)	48	58	80
1:1 Cyclohexane:ethyl acetate	35	45	57
1:1 Cyclohexane:methylene chloride	60	67	Not determined
7:3 Ethyl acetate:cyclopentane	38	49	60
1:1 Hexane:methylene chloride	41	53	64

Table 2. Estimated sample capacity and relative labor requirements comparison per eight-hour shift to evaporate 200 mL of methylene chloride using different evaporation methods

Instrument	Sample Capacity Per Eight-Hour Shift	Estimated Operating Labor Requirements
RapidVap N2 system	61	Low
RapidVap Vacuum system	49	Low to moderate
TurboVap II system	36	Low
Rotary evaporator	12	Moderate to high
Kuderna-Danish apparatus	10	Moderate to high

Table 3. Recovery of 500 ppb of lindane from 200 mL of methylene chloride evaporated to dryness and reconstituted in 1 mL of hexane for injection for GC/XSD analysis. The recovery range is for all solvent types tested. Solvents not containing methylene chloride were concentrated to 1 mL prior to injection for GC/XSD analysis.

Instrument	Recovery	Recovery Range for All Solvent Types
RapidVap N2 system	90.4%	75–125%
RapidVap Vacuum system	89.7%	53–112%
TurboVap II system	88.4%	68–120%

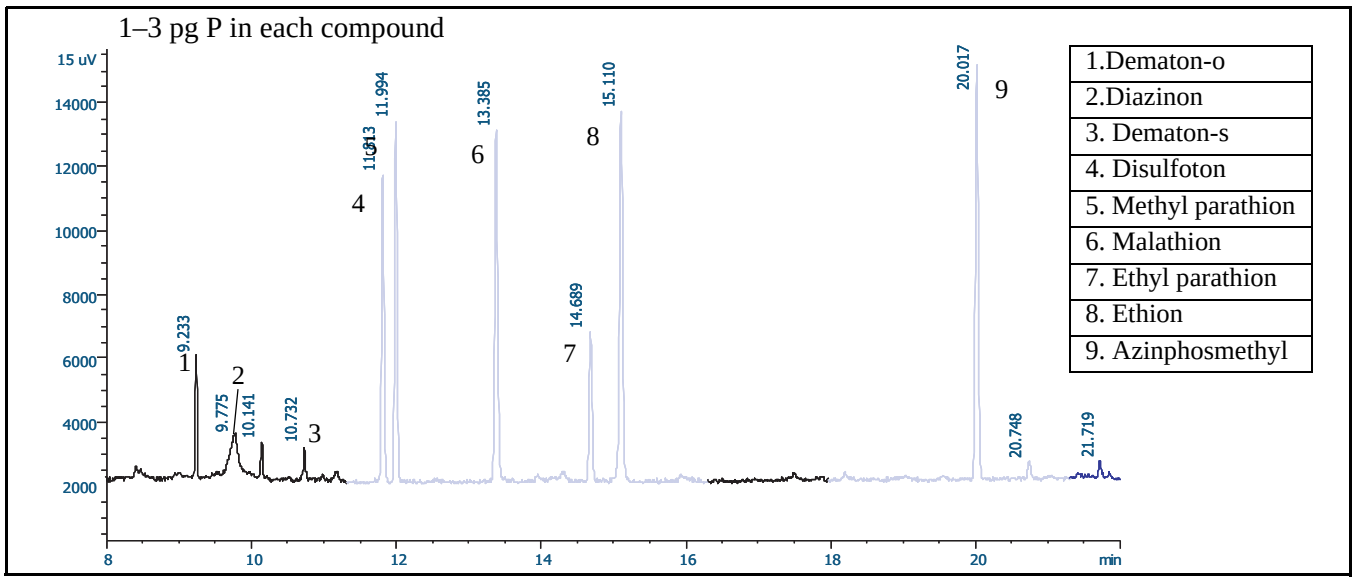


Figure 2. A mixture of nine different organophosphorus pesticides using the PFPD in phosphorus mode

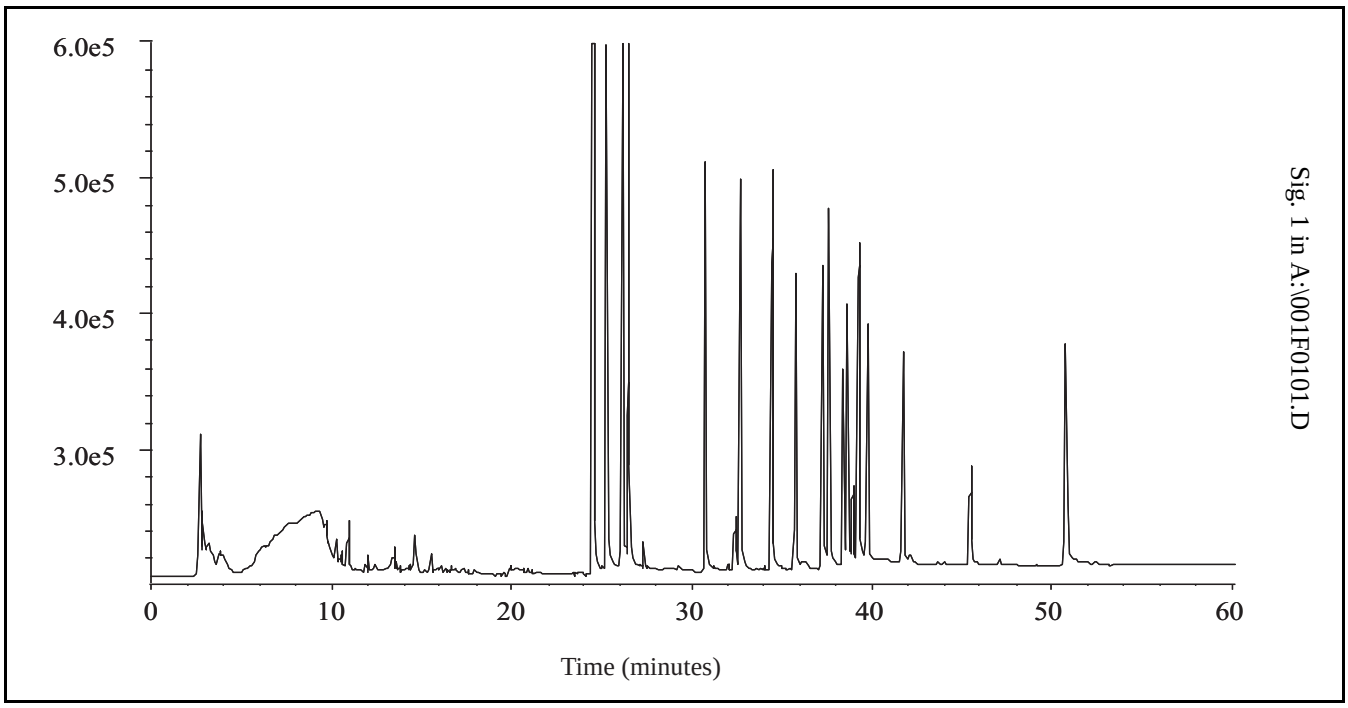
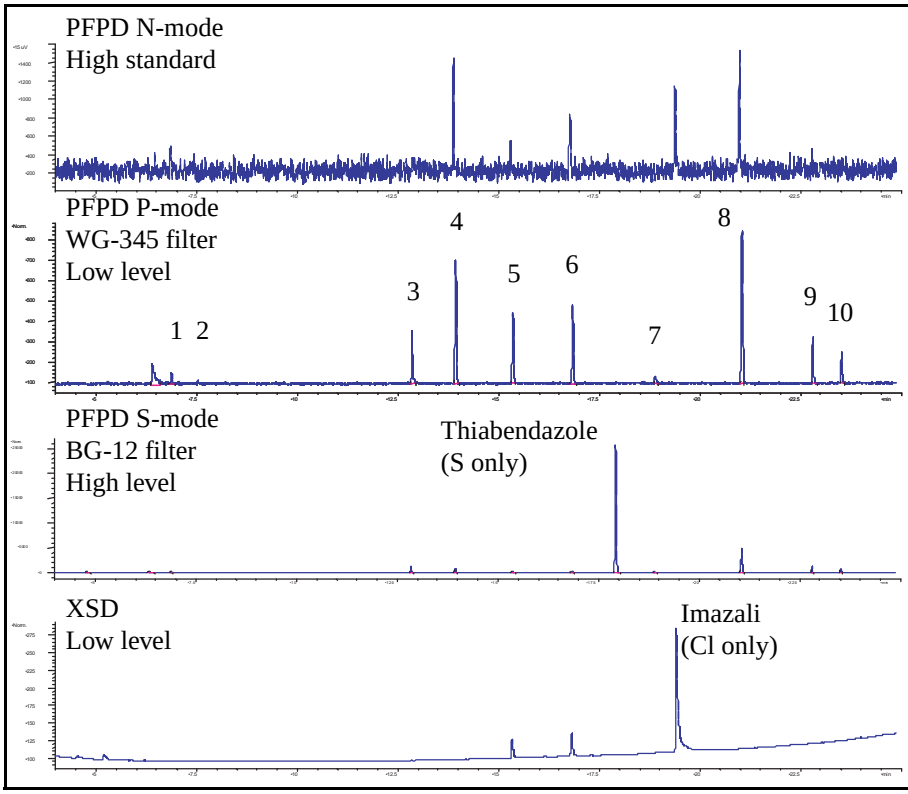


Figure 3. Organochlorine pesticide mix AB on an Agilent 6890 GC with HP-5 column and Model 5360 XSD



Compound	Low Conc. (ppb)
1. Methamidophos	100
2. Acephate	60
3. Dimethoate	60
4. Diazinon	60
5. Chlorpyrifos-methyl	50
6. Chlorpyrifos	50
7. Disulfoton sulfone	50
8. Ethion	60
9. Phosmet	100
10. Azinphosmethyl	150

Figure 4. Pesticide mix on both GC/PFPD with different filters for P- or S-containing pesticides and GC/XSD for halogenated pesticides

Conclusions

- Preliminary data indicates all three multisample evaporation systems are effective for recovery of chlorinated pesticides. Work continues to evaluate the effectiveness of these systems for organophosphate pesticides.

- The Labconco RapidVap N2 system was the fastest evaporation system, had a higher sample capacity, and the lowest cost per sample compared to the other two systems.

- The RapidVap Vacuum system requires more monitoring than the other systems to prevent sample bumping. However, its advantages over the other two systems include different sample tubes sizes and an optional cold trap to reduce solvent vapors.

- Preliminary results indicate the best and the most accurate recoveries for both chlorinated and organophosphate pesticides occurred when solvent exchange was not necessary prior to injection for GC/XSD or GC/PFPD analysis.

- All three systems save labor costs compared to traditional Kuderna-Danish concentrators and rotary evaporators.

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