

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*



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.

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 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- μ m 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%

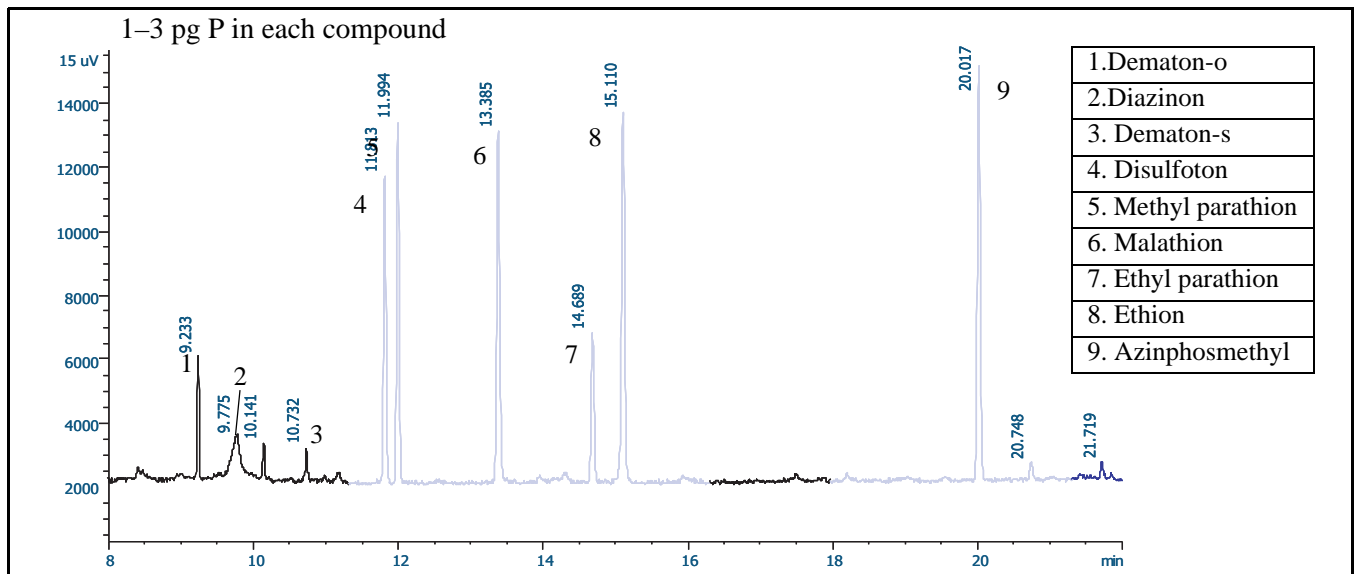


Figure 2. A mixture of nine different organophosphorus pesticides using the PFPD in phosphorus mode

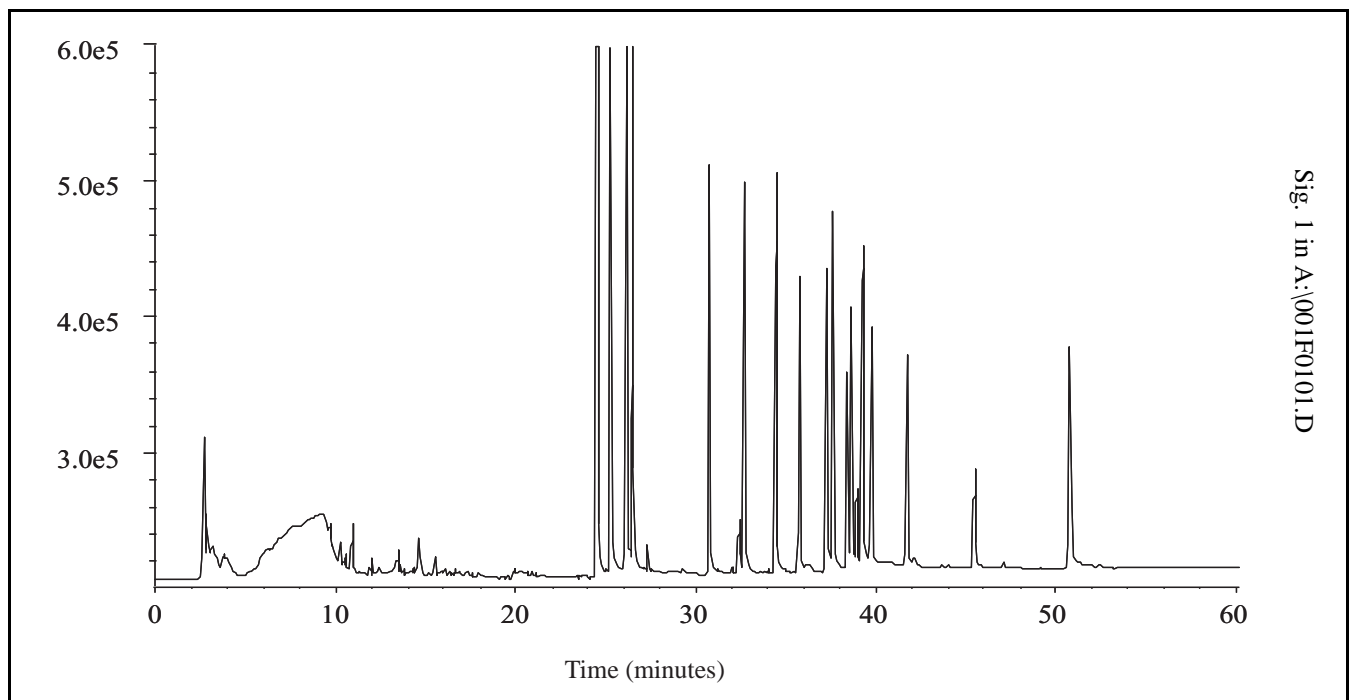


Figure 3. Organochlorine pesticide mix AB on an Agilent 6890 GC with HP-5 column and Model 5360 XSD

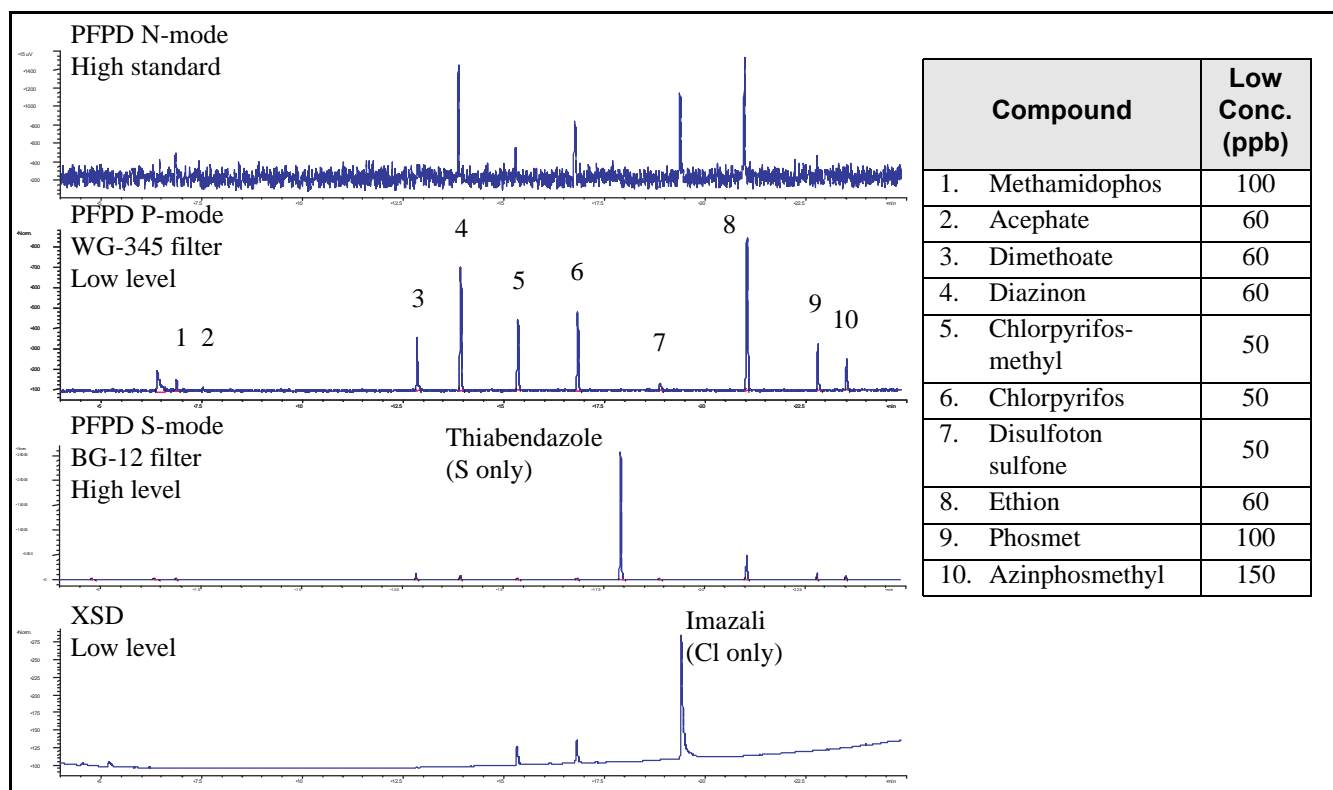


Figure 4. Pesticide mix on both GC/PFPD with different filters for P- or S-containing pesticides and GC/XSD for halogenated pesticides

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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