



Pesticide Analysis...From Soup to Nuts

Maintaining a safe food supply is critically important for everyone. Monitoring pesticide levels in food is much more crucial than merely complying with federal and state regulations. It is an integral part of protecting the health of our bodies and our environment.

All of us want to minimize our exposure to potentially hazardous chemicals. Monitoring low-level pesticide residues in our food supply, however, is one of the most challenging analytical applications. It involves dealing with diverse, numerous, and often complex sample matrices that can contain many possible interfering compounds. Solving this difficult problem requires the right tools.

Efficiently Removing Interfering Compounds

Gel permeation chromatography (GPC) cleanup is used extensively to remove interfering compounds from food and environmental samples. This cost-effective method ensures good data quality and adequate detection limits while minimizing maintenance and calibration costs of analytical systems. GPC is a size-exclusion process that uses organic solvents and a hydrophobic gel (primarily a cross-linked divinylbenzene-styrene copolymer) to separate macromolecules. GPC

is a highly effective in removing high-molecular-weight interferences such as lipids, polymers, proteins, pigments, natural resins, and cellular components from samples prior to analysis. This cleanup method can be applied to a wide range of samples including plant and animal tissue, soil, sludge, and sediment.

Tools for GPC Cleanup

OI Analytical's GPC AutoPrep 2000 automates the GPC process, making it more efficient and reliable. The GPCAutoPrep 2000 uses an autosampler for both injecting sample 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 and a wash pump rinses the needle, preventing sample carryover. The system is controlled with a PC using WinSEP™ GPC control software, a Windows®-based program, which includes an extensive quality monitoring program.



GPC AutoPrep 2000

A simple and economical solution to GPC cleanup is provided by the Model SP 2000. This manually-operated, highly-affordable instrument uses the same columns as the automated GPC AutoPrep 2000 and achieves the same precise results. The Model SP 2000 includes an isocratic HPLC pump, sample injector, switching valve, and control module.

Regulatory Methods Using GPC Cleanup

USEPA SW-846 Method 3640A

USEPA Contract Laboratory Program, Statement of Work for Organics Analysis, Document OLM01.0

FDA Pesticide Analytical Manual, Volume 1

USGS SOP No. HC521A

USDA and USDI GPC Protocols, Analytical Chemistry Laboratory Guidebook (Residue Chemistry)

AOAC Cleanup Methods for Meat Products



**RapidVap N2
Evaporation System**

Multiple Sample Evaporation Systems

For the most efficient concentration of sample extracts prior to or after GPC cleanup, OI Analytical offers the RapidVap® N2 Evaporation System. Vortexing, heating, and nitrogen blowing on samples combine to speed evaporation, resulting in greater throughput. Microprocessor-controlled vortex motion increases surface area for faster evaporation while continually mixing the sample maximizes recovery. The RapidVap provides an automated alternative to Kuderna-Danish and rotary evaporators. Its dry block heating method supplies a reliable alternative to maintenance-intensive, contamination-prone water baths.

The Right Detector Makes All the Difference

Pesticide residues are usually present in food samples at very low concentrations, and many potentially interfering compounds from sample matrices make identifying these pesticides a complex and difficult application. Low-level pesticide residue analyses, therefore, require detectors with significantly improved sensitivity and selectivity.

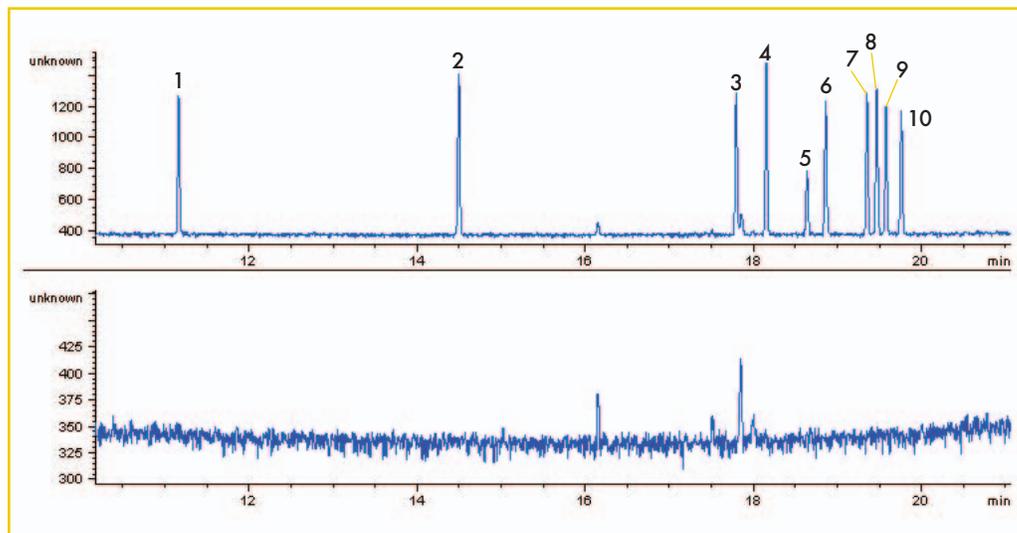
Pulsed Flame Photometric Detector

The Model 5380 Pulsed Flame Photometric Detector (PFPD) is regularly used for analyzing organophosphate pesticides because of its excellent selectivity and extremely low detection limits. It is also well suited for detecting sulfur-containing pesticides because of its essentially infinite selectivity for sulfur in the presence of complex matrices, exceptional sensitivity, low maintenance requirements, and long-term stability. The PFPD's operating conditions can be optimized to selectively analyze for phosphorus, sulfur, or nitrogen and to eliminate matrix interference from other heteroatoms. Alternatively, analyze both sulfur and phosphorus pesticides simultaneously with no selectivity preference toward either sulfur or phosphorus through proper selection of optical filters.



Model 5380 PFPD

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



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

Halogen-Specific Detection

OI Analytical developed the Model 5360A Halogen Specific Detector (XSD™) to address the need for a sensitive and selective detector for halogenated compounds. This highly-selective detector responds to chlorinated pesticides but does not respond to sulfur interference. While the XSD is not quite as sensitive as an electron capture detector, it exhibits virtually no response to matrix interference, resulting in significantly greater selectivity and reducing extensive sample preparation requirements. Compared to the electrolytic conductivity detector, the XSD offers similar sensitivity and selectivity but requires virtually no maintenance or upkeep, and eliminates the use of organic solvents.

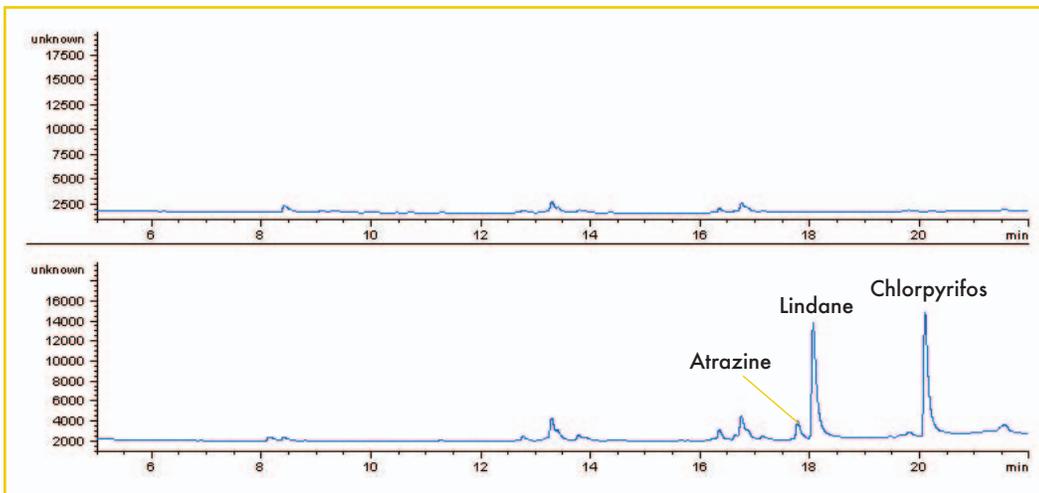


Model 5360A XSD

Challenges and Choices

Providing assurance of a safe and wholesome food supply is a significant responsibility for the food industry. Choosing reliable, efficient, and accurate instruments are key elements to fulfilling this mission. The right tools provide precise results that can be reported with confidence. Their proven technologies streamline workflow and eliminate costly repeat analyses.

For more information on pesticide analysis solutions, download [document 104](#), “Pesticides...From Cleanup to Analysis.”



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.