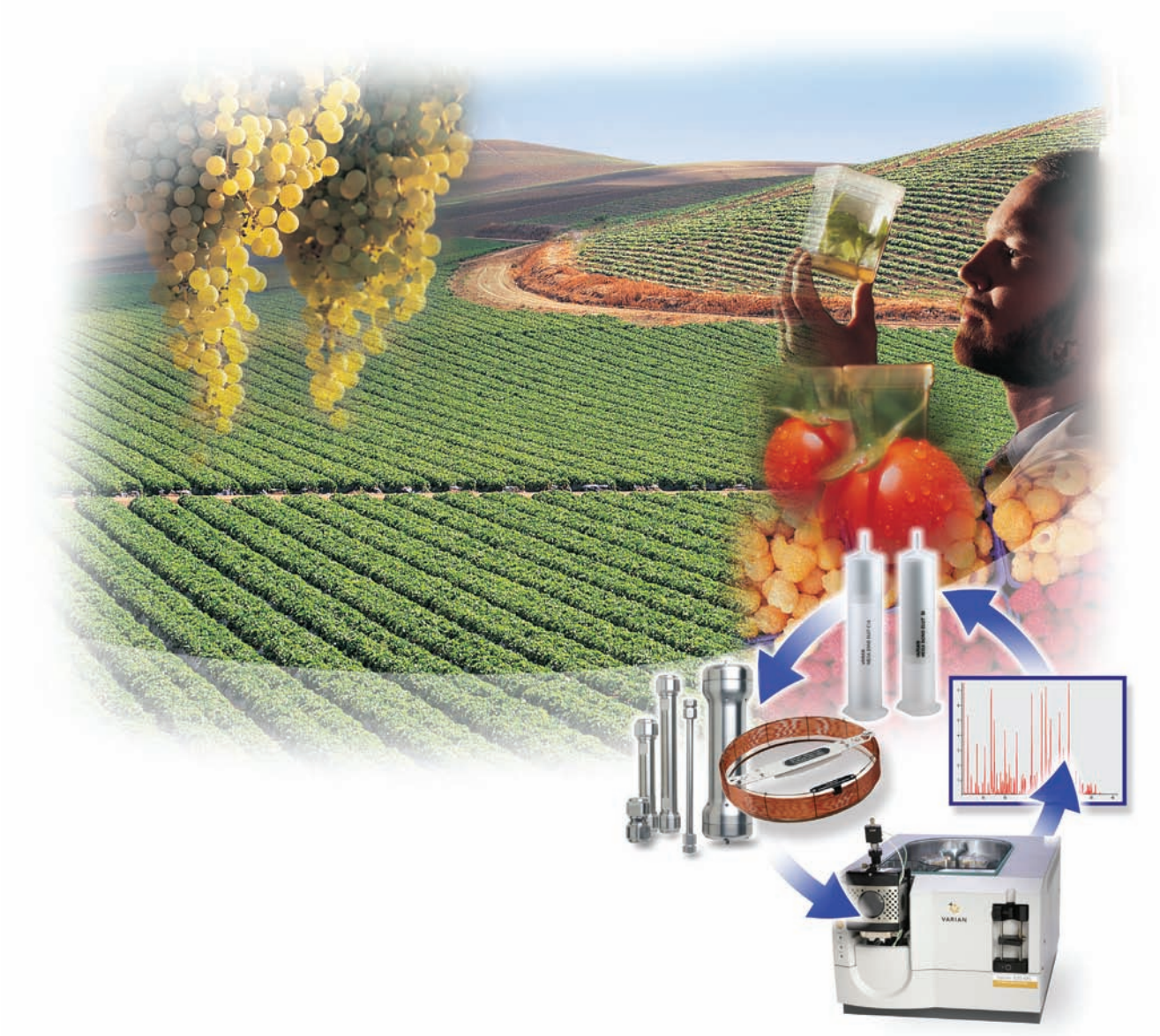
VARIAN, INC.

SAMPLE PREPARATION
LC AND GC COLUMNS
GC/MS/MS
LC/MS/MS

Complete Solutions for Pesticide Residue Analysis in Foods

FROM SAMPLE PREP TO HPLC AND GC ANALYSIS WITH MS/MS DETECTION



VARIAN

www.varianinc.com

Complete Solutions for Pesticide Residue Analysis in Foods

Pesticides are widely used in many areas of modern agriculture as they are considered economically important for high yield production. In today's world of extensive importing and exporting of food goods, the analysis and monitoring of pesticides is essential, although challenging.

The determination of an effective, standardized approach for extracting pesticides from food sources and subsequent identification of compounds can be considerably problematic due to:

- Low and decreasing regulatory levels
- Wide range of compounds and foods
- Pressure to deliver results quickly
- Strict regulations for QA/QC controls

To overcome these challenges, Varian offers a complete support solution for the extraction, isolation, identification and quantitation of pesticide residues, from sample preparation to reporting of results.

Using these superior solutions ensures:

- Increased laboratory productivity
- Outstanding sensitivity and increased selectivity
- Unambiguous identity confirmation
- Rapid method development

Varian also provides comprehensive training programs and support services to help you gain the most from your pesticide analyses. Our EduCare training programs include courses, on-site training, applications assistance, plus web-based and customized training programs designed to help you optimize instrument performance, acquiring faster, more reliable and accurate results.

The Varian Care Program provides flexible on-demand support from a worldwide network of specialists, from installation and start-up guidance to maintenance, advanced theory and application training.

Bond Elut™ SPE Cartridges

Pursuit™, Polaris™ and Pursuit XRs™ HPLC Columns

320-MS Quadrupole MS/MS with LC Interface

FactorFour™ GC Capillary Column

450-GC with 240-MS Ion Trap GC/MS

320-MS Quadrupole MS/MS with GC Interface

Training and Application Support

Contents:

VARIAN, INC.

Sample Preparation

Preparing the Sample for Analysis

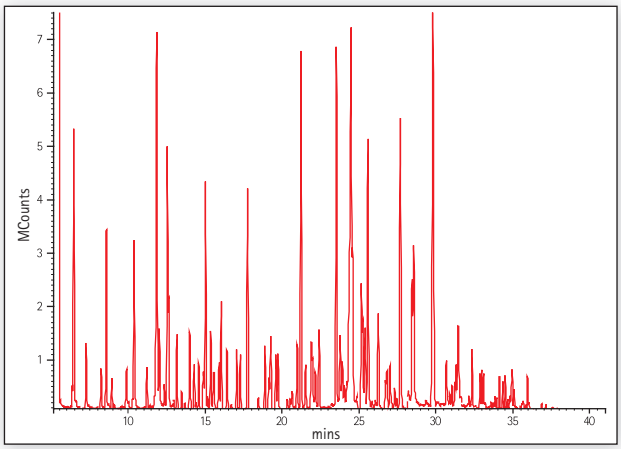
The primary aims of the sample preparation process are removing matrix interferences and increasing the sensitivity of analysis.

Three key extraction methods are often used in sample preparation:

- QuEChERS: Quick, Easy, Cheap, Effective, Rugged, Safe
- SLE: Solid Supported Liquid/Liquid Extraction
- MSPD: Matrix Solid Phase Dispersion

The sample preparation method chosen depends largely upon the food matrix and target list of pesticides, as well as existing successful applications. In addition, many useful modifications for the above sample preparation approaches can be found in the literature.

From an analytical perspective, older pesticides have more temperature stability than those recently developed, and are primarily detected by GC with MS, ECD, NPD, TSD or PFPD detectors. The latest generations of biodegradable pesticides are more thermally labile. These residues are frequently analyzed using LC/MS/MS to avoid hot injection temperatures that can degrade thermally labile compounds when using GC.



This chromatogram shows the successful analysis of 174 pesticide residues from tomatoes. The QuEChERS sample preparation method with bulk SPE PSA sorbent was used (see p. 4), together with analysis by the 320-MS Quadrupole MS/MS with GC interface and FactorFour™ GC columns.

For more information and access to the full Application Note A02405, visit our website: www.varianinc.com/pesticides.

New Graphitized Carbon Black

Varian now offers graphitized carbon black in popular SPE cartridge configurations for analyzing pesticide residues in food. Use Bond Elut™ Carbon for any applications needing a low surface area, non-porous carbon. Strict quality controls ensure that Bond Elut Carbon provides reproducible flow and recoveries for extraction and clean-up applications.

The use of carbon and carbon/NH_2 can be found in a variety of methods for analyzing pesticide residues in foods. Carbon is effective in removing plant pigments and other interfering compounds as presented in the Japanese Positive List System for Agricultural Chemical Residues in Food.

Ordering Information

Test Method	Bed Mass (mg)	Cartridge Size (mL)	Part No.	Quantity (/pk)
Bond Elut Carbon	250	6	12102201	30
Bond Elut Carbon	500	6	12252201	30
Bond Elut Carbon/NH_2	500 ea.	6	12252202	30
Bond Elut Sodium Sulfate Jr. Water Removal Column	3 g	-	12162051B	100

For a copy of the Carbon Datasheet, or for additional information, please contact your local Varian, Inc. sales office or visit us at www.varianinc.com.

For more information regarding pesticides visit our Pesticides link at: www.varianinc.com/pesticides.

QuEChERS Method

The QuEChERS method is designed for extraction of multiple pesticide residues from fruit, vegetables and other low fat foods. It uses Varian bulk primary and secondary amine (PSA) SPE sorbent to remove matrix impurities. PSA removes fatty acids, sugars and other H-bonding matrix co-extractables.

QuEChERS offers significant advantages over traditional homogenization techniques, as outlined below:

Quick:
Extract up to eight samples within 30 minutes.

Easy:
Eliminates the need for additional sample processing, greatly reducing the risk of error.

Cheap:
Solvents and bulk materials can be bought inexpensively and minimal labor is required.

Effective:
Offers high recovery for a wide range of pesticides.

Rugged:
Simple methods allow high reproducibility and ease of use.

Safe:
Significant reduction of organic solvent use minimizes exposure.

Ordering Information for Bondesil™ PSA Bulk Sorbent for use with QuEChERS Method

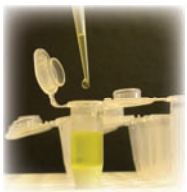
Part No.	Quantity (g/pk)
12213023	10
12213024	100
12213025	1000

Ref. Anastassiades, M., Lehotay, S.J., Stajnbaher, D., and Schenck, F., Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid Phase Extraction" for the Determination of Pesticide Residues in Produce (QuEChERS method). J AOAC Int., 86, No. 2 (2003) 412-431. Method example provided by D. Brown, Bodycote LawLabs, Birmingham, UK. For method specifics and alterations see Applications Bibliography pages 16-19 and review specific literature reference for details.

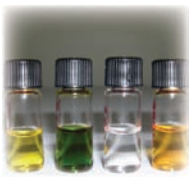
The flow diagram below illustrates the simple stages of the QuEChERS method.

Sample Preparation
Homogenize fruit and vegetables at a low temperature.

Extraction/Partition
Place a 10 g sample into a 50 mL Teflon tube with acetonitrile, NaCl and internal standard. Shake well. Spin for one minute in a centrifuge.



Clean Up
Transfer the supernatant to a 15 mL Teflon tube. Add $MgSO_4$ and Bondesil PSA, shake well for 20 seconds then centrifuge.



GC/MS/MS, LC/MS/MS
Same extract injected into both LC/MS and GC/MS systems. Requires highly selective triple quadrupole detectors for matrix rejection.

VARIAN, INC.

Chem Elut™ Method

Chem Elut contains hydromatrix, a high-purity diatomaceous earth with a high capacity for aqueous adsorption. This is an exceptional medium for Solid Supported Liquid/Liquid Extraction (SLE).

Compared to conventional Liquid/Liquid extraction, Chem Elut offers:

- Simple gravity flow, walk away operation
- Elimination of unwanted emulsions
- Reduced technique dependence
- Improved results and throughput

Chem Elut cartridges come in a variety of sizes to meet a wide range of application requirements.

Ordering Information for Chem Elut 5 mL Cartridge for Use in SLE Method

Test Method	Varian Product	Part No.	Quantity (/pk)
SLE	Chem Elut	12198006	100

For additional Chem Elut products and further information, visit www.varianinc.com.

Chem Elut (SLE) Extraction Method for the Confirmation of More Than 100 Pesticides at 0.01 mg/kg From a Variety of Food Sources

Add water to 5 or 10 g sample (wet or dry weight) to a total of 10 mL.

↓

Homogenize with 20 mL methanol. If necessary, filter or centrifuge.

↓

Add NaCl solution to an aliquot and apply to Chem Elut.

↓

Elution with dichloromethane; evaporate to dryness.

↓

Reconstitute in methanol/water (final sample concentration 1 g/mL). Pass through 0.45 µm syringe filter.

↓

LC/MS/MS in MRM mode.

Ref: Klein J., Alder L., J. AOAC Intern. Vol. 86(5) pages 1015-1037 (2003).

Ordering Information for 0.45 µm PTFE Syringe Filters

Part No.	Diameter (mm)	Quantity (/pk)
A4102	4	100
A4170	17	500
A4106	30	100
A4134	30	500

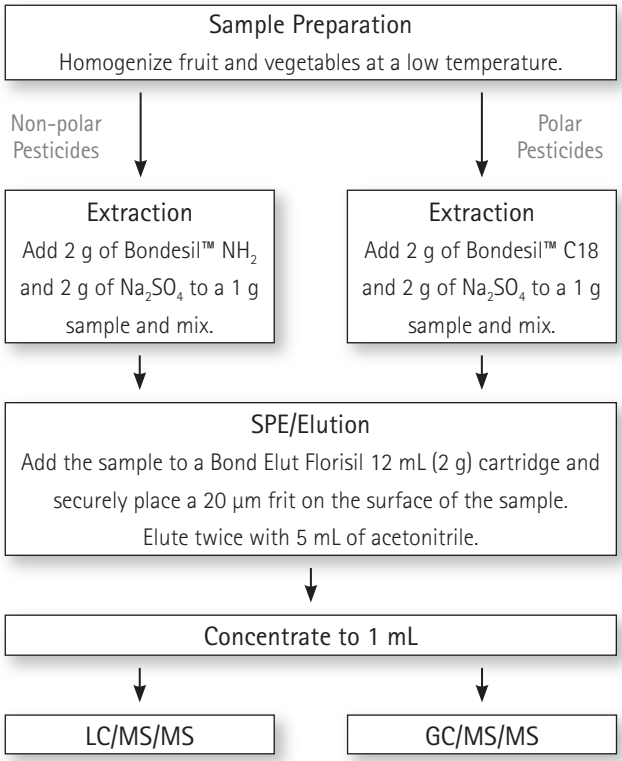
Matrix Solid Phase Dispersion Method

Matrix Solid Phase Dispersion (MSPD) combines a solid or viscous food sample with a specified ratio of bonded silica, often with the use of a mortar and pestle, as shown in the diagram opposite.

The process ensures homogenous tissue disruption and creates a paste as the tissue adsorbs to the silica surface. The paste is packed into a standard SPE cartridge. Appropriate non-polar solvent schemes are then used to extract compounds of interest. Bond Elut™ Florisil is often used to assist in the removal of polar impurities, as can be seen in the diagram opposite.

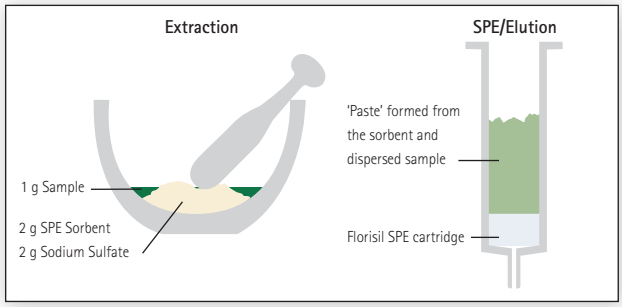
MSPD is effective for both low and high fat food matrices and is preferred over traditional homogenization or classical SPE techniques.

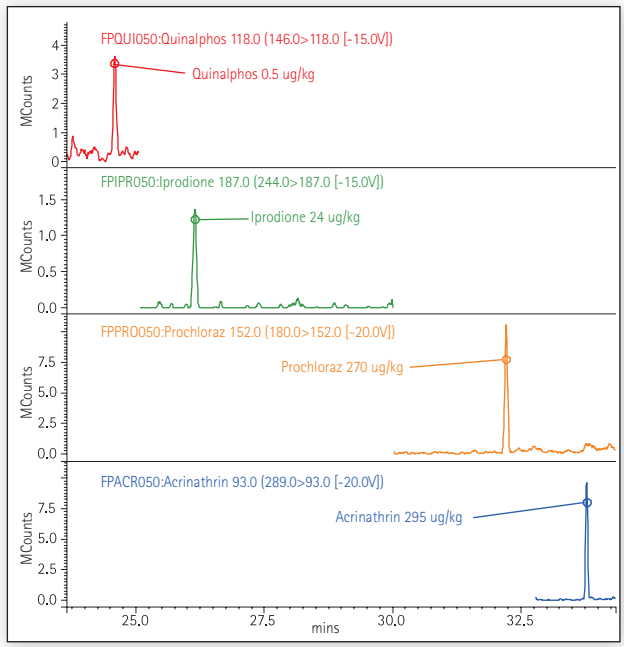
MSPD Extraction Flow Chart



Ref: Brown, D. and Korte, E., Analysis of Multiple Pesticide Residues in Avocados - Comparison of Extraction Methods European Pesticide and Residue Workshop 2006, Poster Presentation.

A Mortar and Pestle Combines the Food Sample With Bonded Silica and Polar Impurities are Removed From the Sample Using Florisil





Residues detected in an avocado by GC/MS/MS. MSPD extraction of 0.6 ppb Quinalphos, 24 ppb Iprodione and 300 ppb of Prochloraz and Acrinathrin.

Ordering Information For Bulk Bondesil For Use with the MSPD Method

Recommended Varian Product	Part No.	Quantity (g/pk)
Bulk Bondesil C18	12213011	10
Bulk Bondesil C18	12213012	100
Bulk Bondesil C18	12213013	1000
Bulk Bondesil NH_2	12213020	10
Bulk Bondesil NH_2	12213021	100
Bulk Bondesil NH_2	12213022	1000
12 mL 2 g Florisil	12256022	20 pcs

VARIAN, INC.

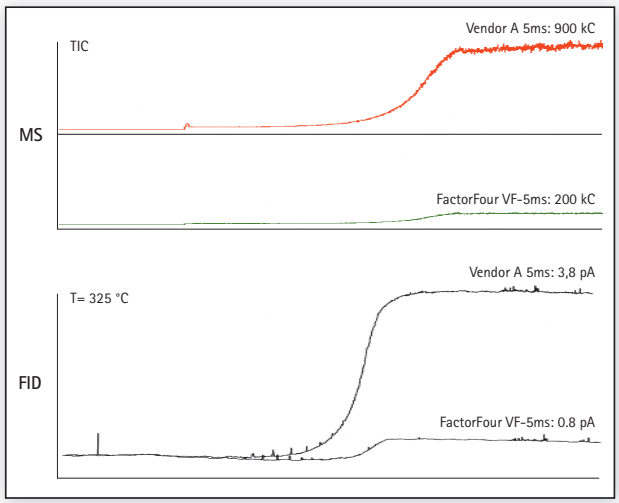
Separation: GC

FactorFour™ GC Columns for Improved Accuracy and Reliability

FactorFour high performance capillary GC columns offer improved results and cost effective throughput for pesticide residue analysis in food. With high quality starting materials, advances in phase stabilization and proprietary techniques in surface deactivation, FactorFour delivers ultra low bleed and high inertness for exceptional quality and reproducible gas chromatography, essential for accurate analysis of trace level pesticides.

FactorFour columns increase confidence in your pesticide analyses. The reduced column background ensures better signal to noise ratios, which result in improved detection levels of pesticides that elute at higher column operating temperatures.

Comparison of Vendor A and FactorFour VF-5ms Columns

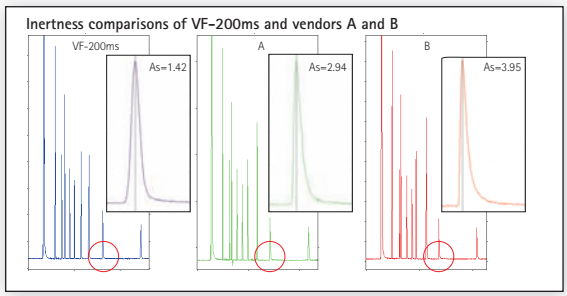


FactorFour bleed rates are up to four times lower than a column from another vendor.

Benefits of Low Bleed FactorFour Technology

- Greater signal to noise ratios result in lower detection levels and greater confidence
- Superior peak shape and high response for difficult pesticides allow simplified, time-saving data review
- Increased column lifetime reduces the cost per analysis
- Reduced detector contamination decreases down time and provides trouble-free analysis
- Faster stabilization time increases throughput
- Better MS spectra allow accurate identification

FactorFour Columns Show Higher Inertness



Comparison of inertness between the VF-200ms and comparable columns from vendors A and B. VF-200ms offers superior peak symmetry for the polar 1-decanol standard.

Other FactorFour Columns Available

VF-1ms	VF-5ms	VF-5ht	VF-5 Pesticides
VF-Xms	VF-624ms	VF-1301ms	VF-1701 Pesticides
VF-1701ms	VF-35ms	VF-17ms	VF-200ms
VF-23ms	VF-WAXms		

To learn more about pesticide analysis, visit: www.varianinc.com/pesticidetips/.

Access more information about our FactorFour GC columns at www.varianinc.com.

Dual Column GC Application

Specialist GC and Guard Column Solutions for Comprehensive Pesticide Analysis

Both FactorFour™ VF-5 Pesticides and VF-1701 Pesticides columns are individually tested with key pesticides prone to degradation due to column activity, such as p,p'-DDT, Endrin and Aldrin, to ensure optimum column performance and reproducibility. The ultra low bleed and high inertness offer improved sensitivity and effective pesticide detection even at low picogram levels.

Combining the VF-5 Pesticides and VF-1701 Pesticides columns can be effective in methods using ECD, where a confirmational analysis on a second column is needed for accurate identification.
The VF-5 Pesticides column is the perfect choice for many GC/MS and GC/MS/MS applications where column inertness is a critical parameter.

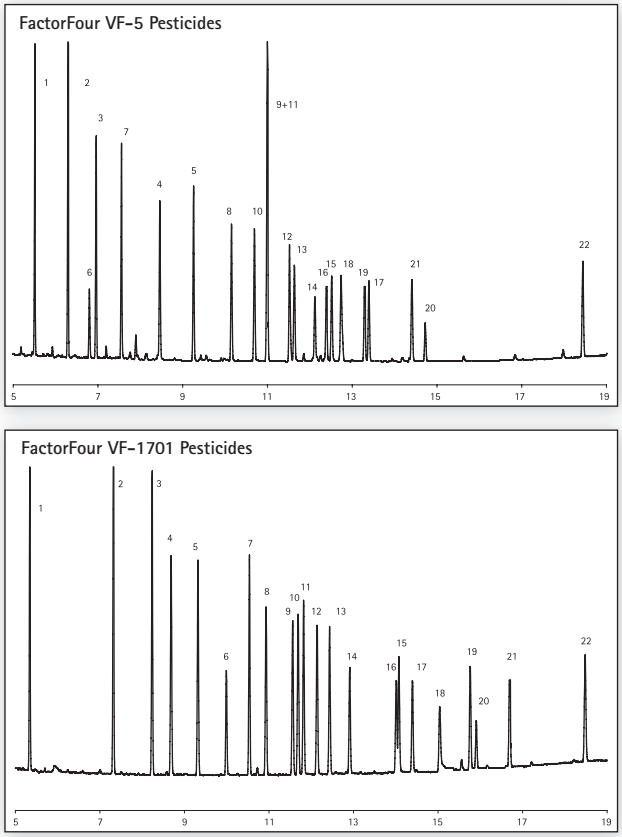
Retention Gaps and EZ-Guard™ Integrated Guard Columns

Guard columns or retention gaps are often added to the front of an analytical column for protection from contamination, or to act as an injection band focusing device when introducing a liquid sample into the capillary (with on-column and splitless techniques). In order to ensure accurate analytical results and prolong column lifetime, columns require a sound maintenance program to protect against the complex and highly contaminating matrices of many food samples.

Retention gaps are available in three deactivation types: low- medium- and high polarity, to match the polarity of the sample solvent. Varian retention gaps come complete with CP-quick-seal connectors for easy installation on all capillary column diameters.

EZ-Guard columns are a combination of a FactorFour column with an integrated 5 or 10 m guard column, eliminating the possibility of leaks by eliminating the need for coupling. EZ-Guard is uncoated and surface deactivated to ensure inertness and quality peak shape. It also features an integrated detector end transfer line, uncoated and deactivated, to assist in faster system stabilization.

Dual Column Application with VF-5 Pesticides and VF-1701 Pesticides Columns. See Application Note SI-00924



Peak Identification:

1. 2,4,5,6-Tetrachloro-m-xylene
2. α-BHC
3. γ-BHC
4. Heptachlor
5. Aldrin
6. β-BHC
7. δ-BHC
8. Heptachlor epoxide
9. Endosulfan I
10. γ-Chlordane
11. α-Chlordane
12. 4,4'-DDE
13. Dieldrin
14. Endrin
15. 4,4'-DDD
16. Endosulfan II
17. 4,4'-DDT
18. Endrin aldehyde
19. Endosulfan sulfate
20. Methoxychlor
21. Endrin ketone
22. Decachlorobiphenyl

Conditions

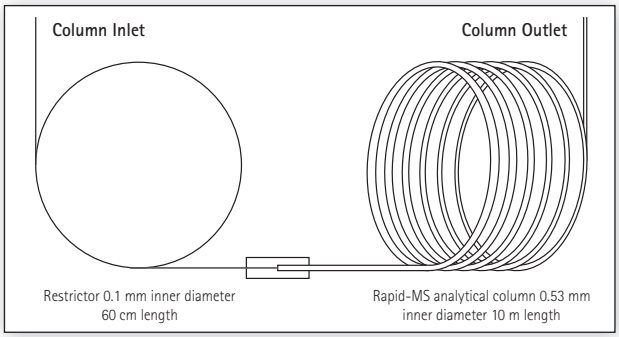
Technique: GC
Instrument: Varian CP-3800
Column: FactorFour VF-1701 Pesticides and VF-5 Pesticides
30 m x 0.25 mm df = 0.25 µm (Part no. CP9070 and CP9074 respectively)
Temperature: 59 °C (hold 30 sec) to 150 °C at 15 °C per min to 275 °C at 4 °C per min
Carrier Gas: Helium, 1.4 mL per min constant flow
Injector: Split/splitless, in splitless mode, T = 250 °C
Detector: ECD, T = 275 °C
Sample Size: 0.5 µL
Sample: Pesticide 8081 Standards Mix, conc.: 200 ng/mL

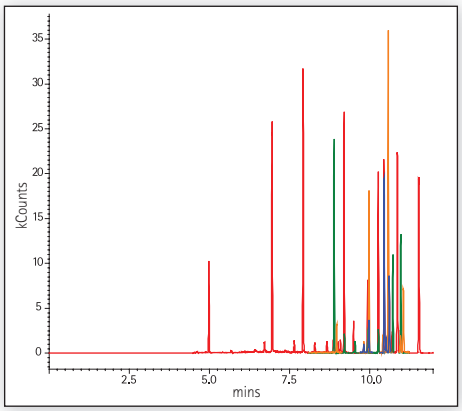
VARIAN, INC.

Rapid-MS

VF Rapid-MS Pesticide Columns Offer Sensitive, High-Throughput Analysis

The VF Rapid-MS Pesticide column works with a pre-column restrictor in front of a wide bore FactorFour™ column (see diagram below). The combined restrictor and MS vacuum allow the analytical column to operate at a very high carrier gas velocity without loss of column efficiency or resolution.





The chromatogram above shows how Rapid-MS and large volume injection techniques combine fast analysis with extremely low detection levels.

Conditions

Column: VF Rapid-MS pesticide column P/N CP8138 with a 2.5 m x 0.53 mm retention gap
Injection Volume: 50 µL large volume injection (LVI)
Sample Conc.: 0.8 ppb of pesticide and PCB standards
Injection Speed: 5 µL/s
Liner: Carbofrit liner
Column Oven: 40 °C (3.00 min) -> 20 °C/min -> 250 °C (0 min)
Injector Temp.: 65 °C (0.50 min) -> 200 °C/min -> 350 °C (5 min)

Time	Split Ratio
Initial	1:25
0.45 m	Off
2.10 m	1:50

Key benefits include:

- Decreased MS analysis time to improve efficiency and throughput
- Increased sensitivity for enhanced detection limits
- Lower elution temperatures reduce bleed contamination and increase signal to noise ratios

Number	Retention Time	Compound Name
1	4.986	Hexachlorobutadiene
2	6.960	Pentachlorobenzene
3	7.928	Hexachlorobenzene
4	8.298	α-HCH
5	8.672	β-HCH
6	8.877	γ-HCH
7	8.898	PCB28
8	8.979	Heptachlor
9	9.024	δ-HCH
10	9.206	PCB52
11	9.223	Aldrin
12	9.508	Telodrin
13	9.554	Isodrin
14	9.942	o,p'-DDE
15	9.953	Cis-heptachlorepoxide
16	9.984	PCB101
17	9.985	Trans-heptachlorepoxide
18	10.276	p,p'-DDE
19	10.282	α-Endosulfan
20	10.457	o,p'-DDD
21	10.619	o,p'-DDT
22	10.517	Dieldrin
23	10.590	PCB118
24	10.714	Endrin
25	10.731	PCB138
26	10.873	p,p'-DDD
27	10.973	β-Endosulfan
28	10.978	PCB153
29	11.061	p,p'-DDT
30	11.544	PCB180

Separation: HPLC

Varian Offers a Comprehensive Line of High Purity Silica HPLC Columns

Varian Pursuit XRs™, Pursuit™ and Polaris™ HPLC columns are referenced in a variety of successful food analysis applications and published papers. Guidance regarding the type of HPLC column required for successful and reproducible separation can be found in the scientific literature.

Analytical columns are available in 3, 5 and 10 µm with 1.0-4.6 mm column ID and 30-250 mm column lengths.

Pursuit (C18, C8, Diphenyl, PFP and PAH)

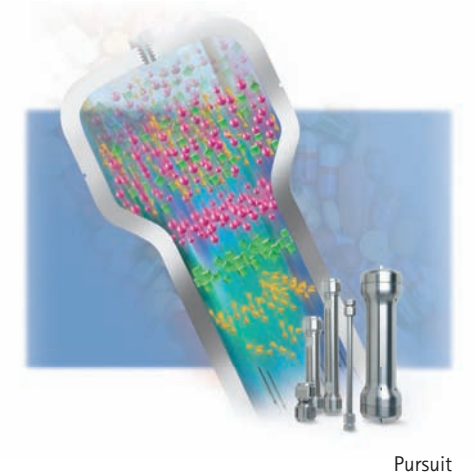
For fast separations with outstanding resolution, while maintaining excellent peak symmetry, choose from the Varian Pursuit line of columns. Pursuit incorporates high phase density bonding and extensive end-capping to a high purity 200 angstrom silica particle. Combined, these features offer reduced hydrophobicity with enhanced compound selectivity.

Pursuit XRs (C18, C8, and Diphenyl)

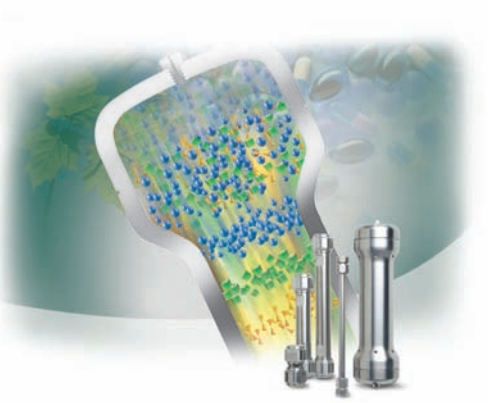
Pursuit XRs is the reliable choice for successfully resolving complex mixtures such as multiple pesticide residues. With a combination of high surface area, advance bonding chemistry and ultra-high purity 100 angstrom silica, Pursuit XRs delivers enhanced resolution, ruggedness, and reliability. It offers excellent first-time resolution for both polar and non-polar analytes at pH conditions between 1.5 –10.

Polaris (C18-A, C8-A, C18-Ether, C8-Ether, Amide C18)

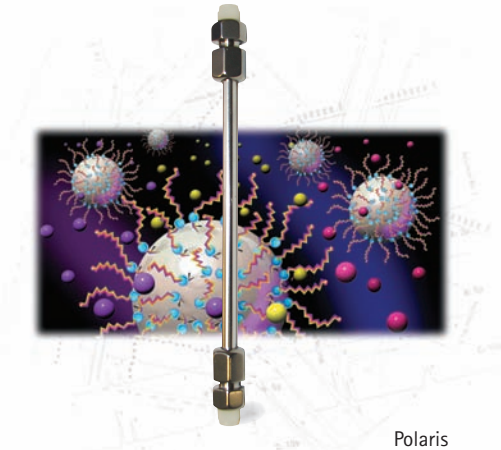
Polaris phases are based on the use of polar modified alkyl chains bonded to a high purity silica particle. Polaris is easily wetted with polar eluents and shows improved retention and selectivity for polar compounds. In addition, the polar embedded groups shield reactive silanols from polar silanophilic compounds, improving peak symmetry.



Pursuit



Pursuit XRs



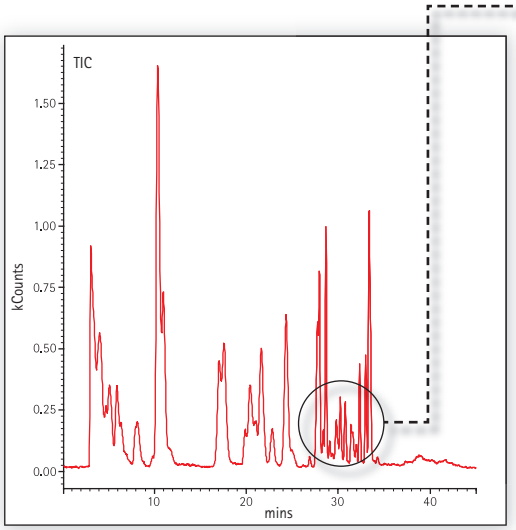
Polaris

A Complete Solution

Multi-class pesticide residues found in food samples can be analyzed using the QuEChERS extraction method, a Polaris™ HPLC column and the 320-MS LC/MS. Targeted Multiple Reaction Monitoring gives the user confidence in compound identity and provides excellent quantification of a wide range of compounds.

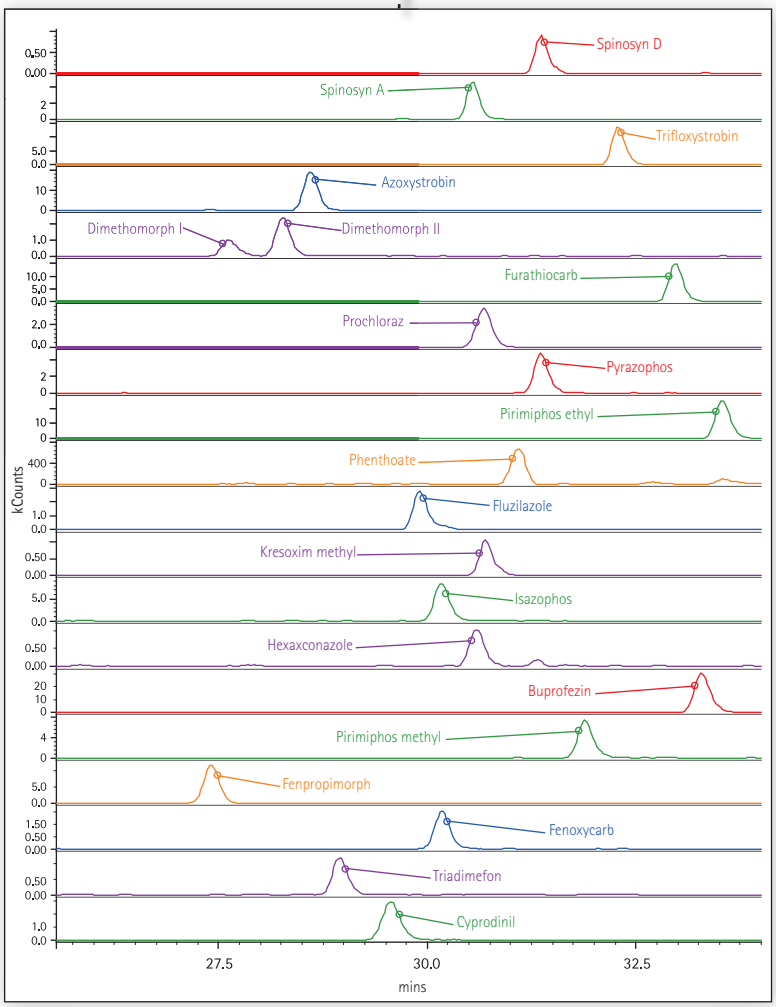
HPLC Mobile Phase Conditions

Run Time (min)	Solvent A (%)	Solvent B (%)
0	75	25
20	60	40
30	0	100
37	0	100
45	75	25



Extract of spinach matrix fortified with 80 pesticides at 100 µg/kg using the QuEChERS method.

LC/MS/MS Conditions:
Detector: Varian 320-MS Triple Quadrupole Mass Spectrometer
Interface: Electrospray Ionization, +/- Ion Mode
Nebulizer Gas: Nitrogen
Collision Gas: Argon
Pumps: Varian 212 Solvent Delivery Module
Injection: Varian ProStar™ 410 Autosampler
Column: Polaris™ 3 µm C18 150 x 2 mm (P/N: A2001150x020) at 40 °C
Flow Rate: 200 µL/minute
Injection Volume: 50 µL Partial Loop Fill
Eluting Solvents:
Solvent A: 5 mM Ammonium Formate - in 0.1% Formic Acid w/v
Solvent B: Acetonitrile: Methanol, 3:1 v/v



Good peak shape and sensitivity are shown. Excellent separation of co-eluting compounds is achieved using the MRM mode of the 320-MS. Data supplied by Don Brown, Bodycote LawLabs, Birmingham, UK.

MS/MS Detection

Increased Sensitivity and Productivity

Liquid or gas chromatography followed by MS/MS detection allows accurate identification and quantitation of pesticides in complex matrices. Varian offers a choice of triple quadrupole and ion trap mass spectrometers for pesticide analysis, depending on your needs.

Triple Quadrupole

The 300 Series triple quadrupole mass spectrometers for both LC and GC offer outstanding sensitivity and selectivity. Both LC and GC quadrupole systems are robust instruments designed to boost productivity and generate high quality data. For routine applications in complex matrices such as food extracts, or for new method development, the Varian 300 Series instrumentation offers a simple yet complete solution, including a detector 180° off-axis to the source and intuitive MS instrument control and data handling software.

Other important features and benefits of the Varian 300 Series include:

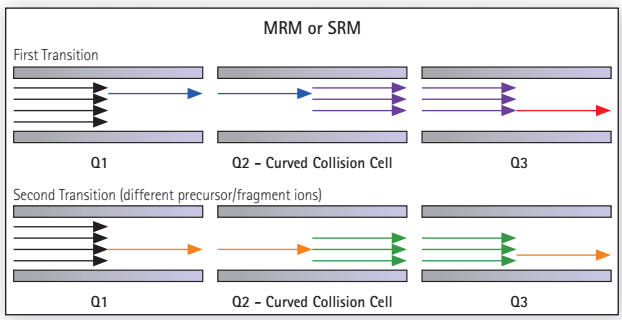
- Fast MS/MS acquisition rate (<5 ms dwell time) for rapid multi-compound screening
- Electronic Gas (API, CI and CID) Flow and Pressure Control for improved precision and ease of use, particularly for thermally labile compounds
- Data reporting to comply with stringent QC checks

The Varian 320 MS/MS Scanning Process

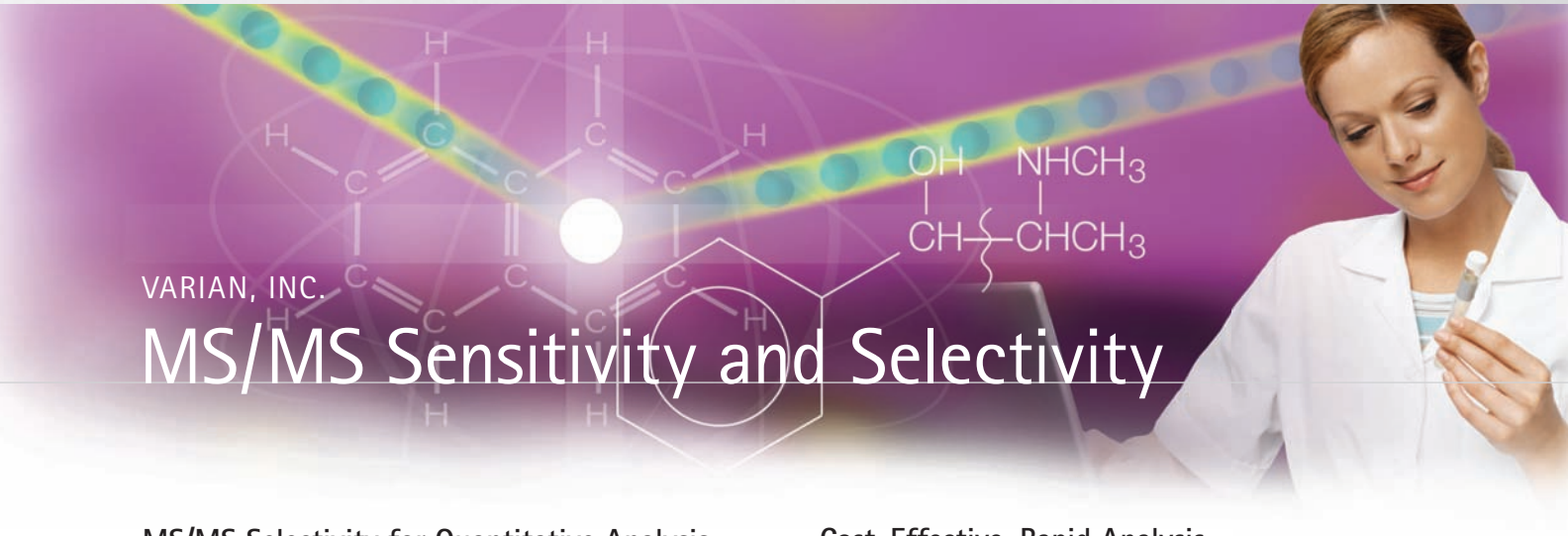
Tandem MS (MS/MS) selectively filters and detects product ions produced only from selected precursor ions. The innovative design of Varian's 300 Series instrumentation, with its 180° curved collision cell, enhances mass filtering and detection process by further reducing noise and so increasing sensitivity. With GC/MS/MS or LC/MS/MS, quantitative results for pesticide residues can be obtained from complex matrices with high reliability and virtually no interference from matrix compounds.

320-MS Triple Quadrupole MS with LC Interface and 320-MS Triple Quadrupole MS with GC Interface

450-GC with
240-MS Ion Trap GC/MS



The MS/MS mode of operation, when applied to pesticide multiresidue analysis, is known as Multiple Reaction Monitoring (MRM) or Selected Reaction Monitoring (SRM). As the diagram above shows, a single mass is selected (filtered) by the first quadrupole (Q1), dissociated by collision induced dissociation (CID) in the collision cell (Q2) and the ions of choice are then selected (filtered) by the last quadrupole (Q3). For each pesticide a second transition is used for confirmatory purposes according to control quality procedures.



VARIAN, INC.

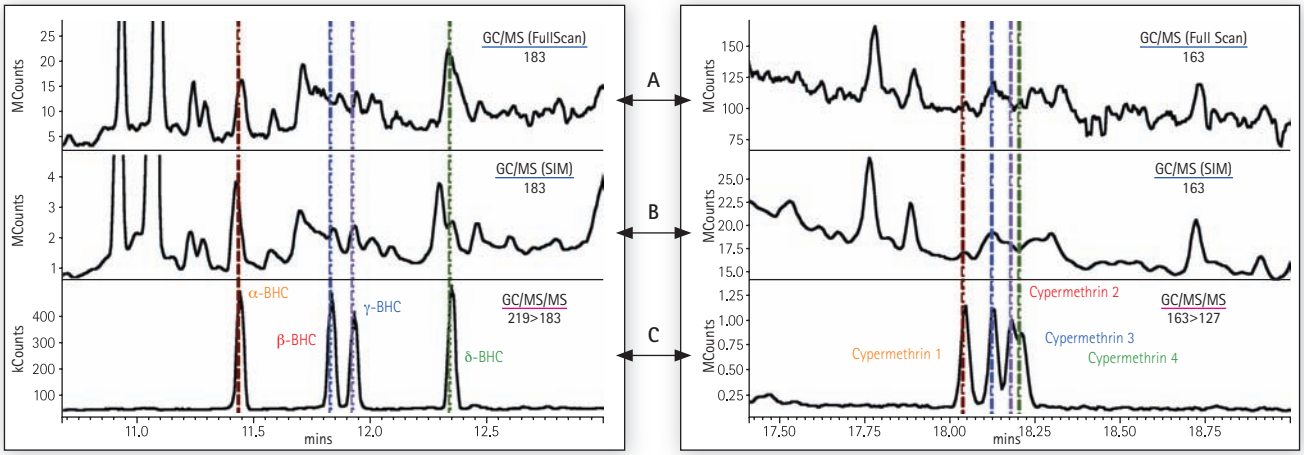
MS/MS Sensitivity and Selectivity

MS/MS Selectivity for Quantitative Analysis

With single quad GC/MS it is difficult to determine α, β, γ, δ-BHC or Cypermethrin 1,2,3 or 4 in complex matrices as illustrated in sections A and B in the chromatograms below. However, with a triple quadrupole system from Varian, many advantages are apparent: excellent matrix rejection, leading to better sensitivity, more accurate quantitation and the ability to screen for (and quantitate) a larger suite of compounds. This gives the user a higher level of accuracy and better explanation of results.

Cost-Effective, Rapid Analysis

The features of the triple quadrupole system translate into greater confidence in the reliability of results, ease of data interpretation, improved rapid routine analysis and a lower cost per sample. When utilizing the 320-MS, effects of co-extracted contaminants are minimized and both chromatography and quantification are greatly improved as shown in section C in the chromatograms below.



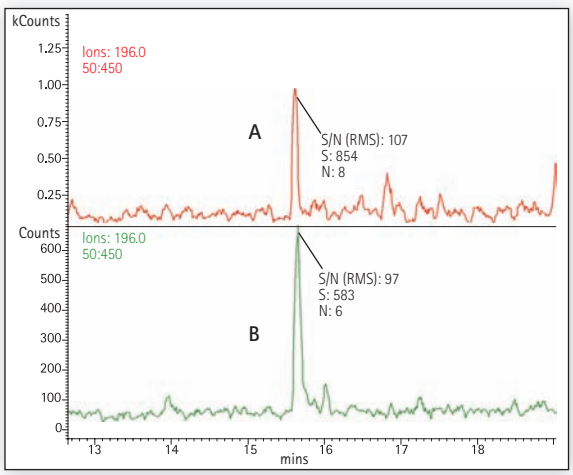
Detection of BCH (10 ppb) with full scan (A) SIM (B) and MRM acquisition mode (C).

Detection of Cypermethrin (10 ppb) full scan (A) SIM (B) and MRM acquisition mode (C).

Ion Trap and MS/MS

The Varian 240-MS Ion Trap GC/MS offers the greatest full scan sensitivity and is an ideal choice for cost conscious laboratories analyzing thermally stable compounds in light to moderate matrices. The rugged design includes a unique pulsed external source which remains clean for a longer period of time, for increased productivity.

For the identification and quantification of target and unknown pesticides in food samples, the 240-MS offers the most selective and lowest level of detection by providing alternating EI and CI ionization, and full scan, SIS, and MS/MS scan modes during the same run. Data acquired from the Varian 240-MS can be matched against searchable libraries to identify unknown compounds in food samples.



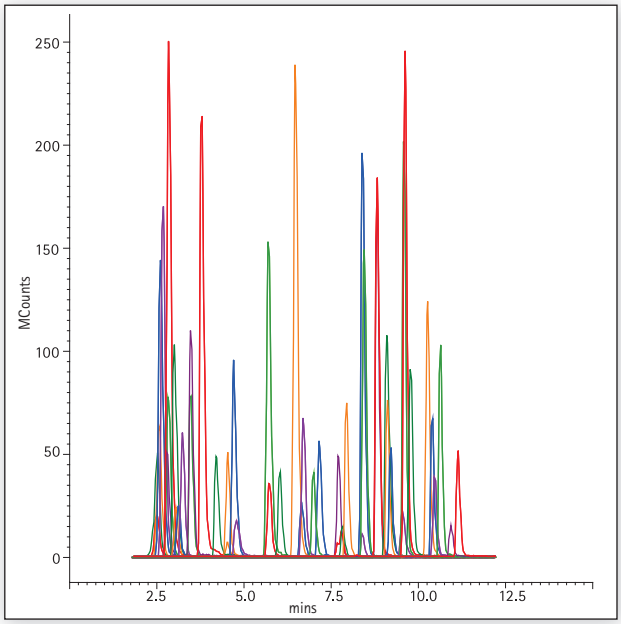
Extracted ion chromatogram of Atraton at m/z 196 at 5 ppb concentration in a crude mixed vegetable extract (A) and pure solvent (B). The 240-MS provides excellent quantitation at low concentrations in the full scan mode, even in complex matrices.

MS/MS Pesticide Solutions

320-MS for Effective, Trouble-Free Pesticide Detection

The following application illustrates the effectiveness of Varian's consumables and instrumentation for low level, multi-residue pesticide analysis. This data summarizes the results of calibration studies with RSD and recovery values from an orange matrix using the QuEChERS extraction method.

Varian 320-MS LC/MS with SRM transitions



Varian 320-MS LC/MS with SRM transitions offers high quality quantification with more than 10 data points per peak and an analysis of 48 pesticides in less than 11 minutes.

HPLC Parameters:
Column: Pursuit™ 3 µm, 50 x 2 mm P/N: A3001 050 x 020
Mobile Phase: Solvent A: Water + 0.2 % Formic Acid + 2 mM Ammonium Formate.
Solvent B: 90 % Methanol/10 % Mobile Phase A
Flow: 200 µL/min (212 LC pumps)10 µL, micro pick-up (410 autosampler)

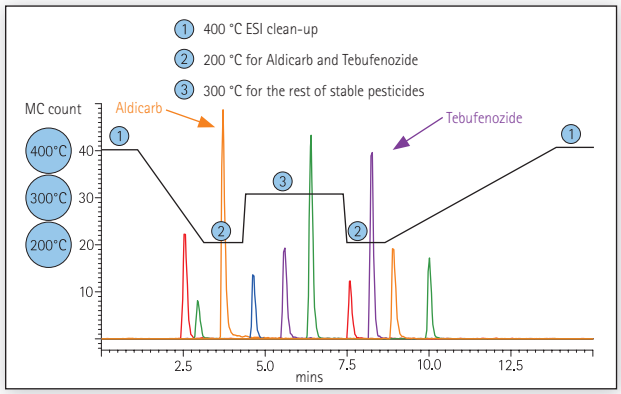
Electrospray parameters:
Ionization: ESI (+/-)
Drying Gas: N_2, 35 psi, SelecTemp™

			Concentration Level 10 ppb	
RT(min)	Peak Name	ESI	% Recovery	% RSD
2.555	Pymetrozine	+	87.5	3.68
2.557	Acephate	+	74.5	4.81
2.599	Omethoate	+	82.9	2.34
2.638	Propamocarb	+	83.3	2.79
2.675	Oxamyl	+	75.7	1.79
2.801	Metomyl	+	69.4	0.59
2.846	Carbendazime	+	86.7	1.59
2.857	Thiamethoxan	+	89.4	5.83
3.009	Thiabendazole	+	67.8	3.98
3.032	Imidacloprid	+	76.3	5.41
3.131	Clothianidin	+	74.9	6.77
3.222	Acetamiprid	+	90.0	0.63
3.467	Cymoxanil	+	67.0	3.85
3.468	Thiacloprid	+	80.7	2.57
3.751	Aldicarb	+	83.5	2.11
4.164	Propoxur	+	67.8	9.56
4.481	Carbaryl	+	81.4	3.25
4.651	Thiodicarb	+	79.0	2.44
4.784	Imazalil	+	85.0	4.18

For full details of this application, please see Varian Application Note SI-01256, available on our website.

SelecTemp™ Control

A patented feature of the 320 Varian API interface, SelecTemp allows the temperature of the drying gas to be controlled on a time segment basis. This enables the analysis of various pesticide groups, including those which are thermally labile, using one method and potentially one injection. As demonstrated in the chromatogram below, by adjusting the temperature of the drying gas, thermal degradation is minimized while desolvation is optimized.



VARIAN, INC.

Accessories

In addition to a full line of quality analytical instrumentation, detectors, GC and LC columns and sample preparation products, Varian also offers a comprehensive line of support accessories to assist in throughput, ease of use and protection of columns and instrumentation.

Protect Your Performance

The Gas Clean Filter System prevents contaminants entering your GC. Harmful impurities in the carrier gas such as oxygen and moisture, even at low ppm level concentrations can result in irreversible damage to the column, loss of sensitivity and unnecessary instrument downtime.

The Gas Clean Filter System consists of a base connecting unit and a filter. Base connecting units come with 1, 2 or 4 filter capacity and are available for 1/4" and 1/8" gas lines. The uniquely designed connection unit allows rapid replacement of saturated filters, while preventing air from entering your system.

The filters contain highly sensitive indicators, alerting you when the filter needs to be replaced. The purity of the carrier gas delivered to the GC (oxygen less than 50 ppb, moisture and hydrocarbons less than 0.1 ppm) is better than 6.0 grade.

Gas Clean Replacement Filters	Part No.
Gas Clean GC/MS Filter	CP17973
Gas Clean Moisture Filter	CP17971
Gas Clean Oxygen Filter	CP17970
Gas Clean Charcoal Filter	CP17972

Gas Clean Base Units	Part No.
1 Filter base connecting unit 1/8"	CP7988
2 Filter base connecting unit 1/8"	CP738407
4 Filter base connecting unit 1/8"	CP736520
1 Filter base connecting unit 1/4"	CP7980
2 Filter base connecting unit 1/4"	CP738406
4 Filter base connecting unit 1/4"	CP7989

GC Accessories

Septa

Varian offers optimized high quality BTO septa for trace level pesticide analyses. BTO septa are ideal for use with low bleed FactorFour™ MS capillary columns. The Centerguide™ guides the needle for easy penetration and reduced tearing.

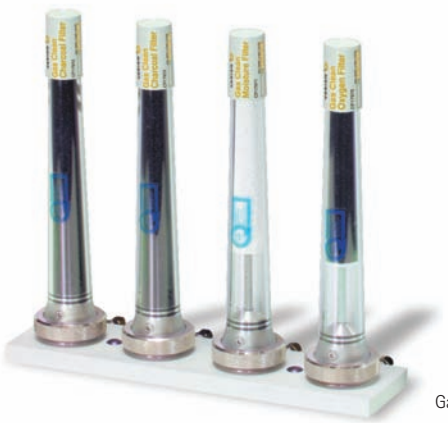
Varian Injector	BTO Septa (mm)	Part No.	Quantity (pcs)
Model 1177	9	CR298713	50
Models 1075/77/78/79 and 1093/94	11.5	CR298777	50

Varian Injector Liners

Varian offers a wide selection of specialized injector liners for the entire range of Varian injector models and injection techniques. Please contact your local Varian office or technical helpdesk for assistance in choosing the appropriate liner.

Ordering Information for Retention Gaps (see page 8 for more details)

ID (mm)	Non-Polar	Medium Polar	Polar	Quantity (/Pk)	Length (M)
0.25	CP8007	CP8017	CP8087	5	2.5
0.32	CP8008	CP8018	CP8088	5	2.5
0.53	CP8009	CP8019	CP8089	5	2.5



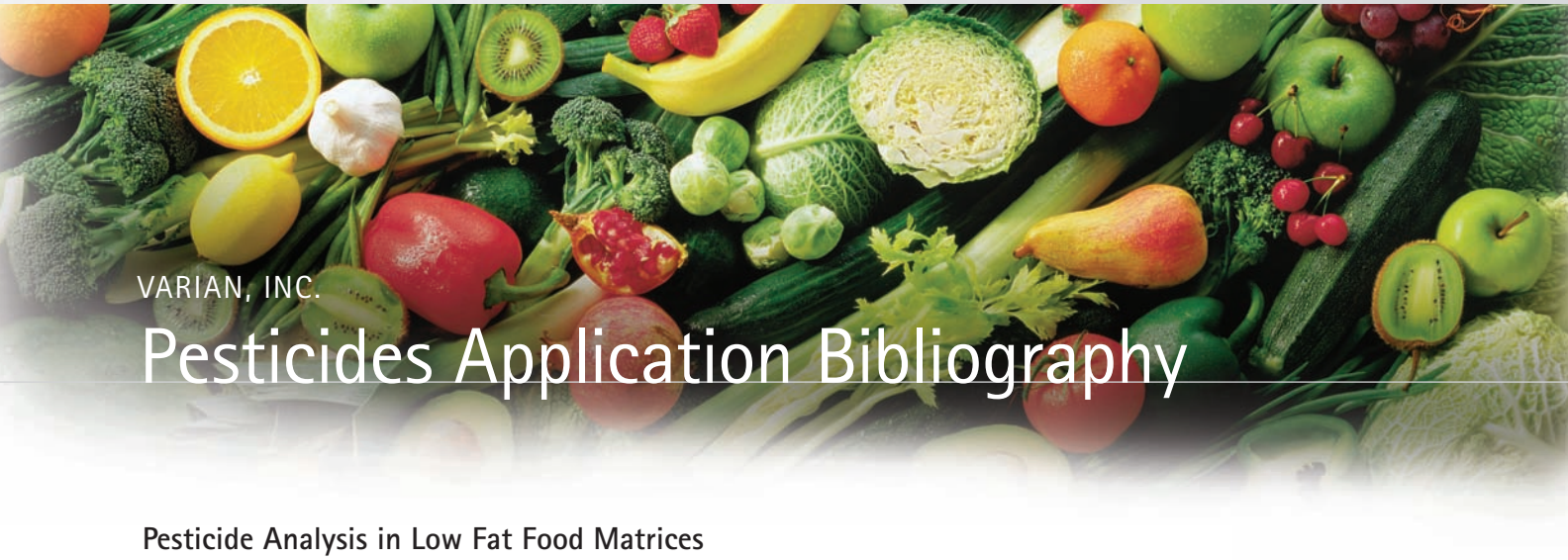
Gas Clean Filter System

Pesticides Application Bibliography

The following tables show many useful applications involving the removal of pesticide residues from various food matrices. Listed by matrix type and method of analysis, they provide a highly valuable reference point when analyzing pesticide residues in foods.

Pesticide Analysis In High Fat Food Matrices

Title of Paper	Key Method Feature	Authors	Source	High Fat Matrix	Sample Preparation	Sorbent	Recommended Column	Analysis	Application Note No.
Determination of Pesticide Residues in Olive Oil by Matrix Solid Phase Dispersion After Preliminary Solvent Extraction		P. Gros J. Tourte S. Couturier	Poster	Olive Oil	MSPD, LLE	NH_2, FI	FactorFour™ VF-5ms and VF-17ms	GC: ECD, NPD	
Isolation and Gas Chromatographic Determination of Chlorosulfuron in Milk		Long , et al.	J. Assoc. Off. Anal. Chem. Vol 72, No.5, 1989, p. 813-815	Milk	MSPD	C18	FactorFour VF-5ms	GC/NPD	M1967
Analysis of Pesticide Residues in Meat Using Matrix Solid Phase Dispersion (MSPD) and GC With NP/EC Detection	High Fat Matrix	H.B. Christensen M.E. Poulsen R.L.L. Bille	Danish Institute for Food and Veterinary Research, Poster EPRW 2004	Meat	MSPD, GPC	C18, FI	FactorFour VF-5 Pesticides and VF-1701 Pesticides	GC: NPD, ECD	
On-line GPC GC Analysis of Organophosphorus Pesticides in Edible Oils		B. Jongenotter HG. Janssen	LCGC Europe, June 2002, p. 1-10	Oils	GPC	Tailor Made PLgel	FactorFour VF-5ms Varian CP-Sil 13 CB, Polymer Labs PLgel	GC/FPD	
Extraction of Trace Chlorinated Pesticides from Chicken Fat Using MSPD		G. Sheng A. Dixon R. Pocci D.R. Nau	Varian, Inc. Application Note	Chicken Fat	MSPD, GPC	C18, FI	FactorFour VF-5ms	GC/ECD	M2821
Pesticide Extraction from Milk, Fruit, Vegetable		J. Meola R.Sheridan	Poster from California Pesticide Residue Workshop in Sacremento.	Food, Vegetables and Milk	Stacked SPE	SAX, PSA Carbon		Extraction only	M2837
Development of a Multi-Residue Screening Method for the Determination of Pesticides in Cereals and Dry Animal Feed Using Gas Chromatography-triple Quadrupole Tandem Mass Spectrometry		S. Walorczyk	J. Chromatog. A 1165 (2007) 200-212	Cereals and dry feed	QuEChERS	PSA, C18	FactorFour VF-5ms	GC/MS/MS	
Analysis of Multiple Pesticide Residues in Avocados - Comparison of Extraction Methods	High Fat Matrix	D. Brown E. Korte	EPRW Poster 2006	Avocado	MSPD, QuEChERS	C18, PSA NH_2	FactorFour VF-5ms, Pursuit™ C18	GC/MS/MS LC/MS/MS	
Buffered QuEChERS Method to Improve Results of Problematic Pesticide Residues in Different Commodities	Effects of Buffering in QuEChERS	S.J. Lehotay K. Mastovska A.R. Lightfield	USDA Poster	Orange, Lettuce, Eggs, Avocado	QuEChERS	PSA, C18	FactorFour VF-5ms Pursuit C18	GC/MS/MS LC/MS/MS	



VARIAN, INC.

Pesticides Application Bibliography

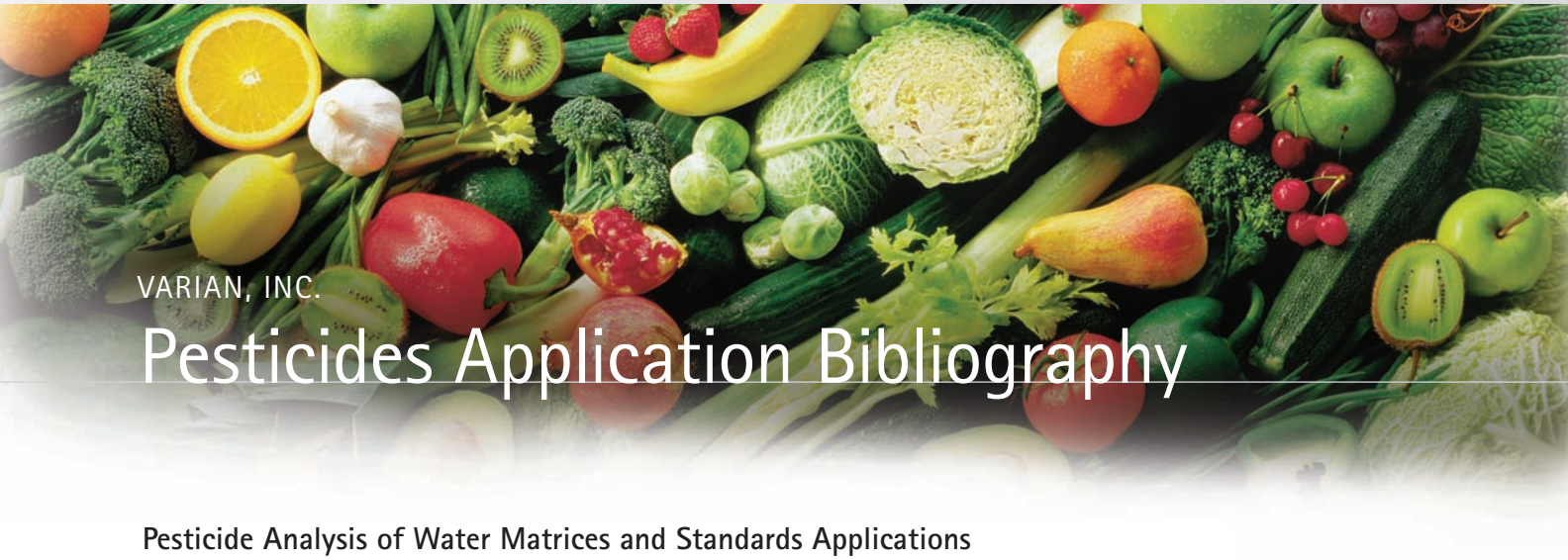
Pesticide Analysis in Low Fat Food Matrices

Title of Paper	Key Method Feature	Authors	Source	Low Fat Matrix	Sample Preparation	Sorbent	Recommended Column	Analysis	Application Note No.
Fast LC/MS/MS Quantitiation of N-Methyl Carbamate Pesticides in Food Using Bond Elut Plexa™ and Pursuit XRs C18		E. Chang R. Suchara G. Li R. Arora	The Application Notebook (LC/GC) Sept, 2007, pg. 52-53	Food	SPE	Plexa	Pursuit XRs™ C18	LC/MS/MS	SI-00946
Comparison of Solid Phase Extraction Sorbents for Clean-up of Pesticide Residue Analysis in Fresh Fruit and Vegetables	Comparison of many established techniques	F. Schenck S. Lehotay V. Vega	J. Sep. Sci 2002, 25, 883-890	Fruit and Vegetables	QuEChERS, Luke, Canadian PMRA	C18, NH_2, PSA, SAX, Carbon, Al,	FactorFour™ VF-1ms VF-5ms	GC: ECD, FPD, MS	
The Determination of Multiple Pesticide Residues in Fruit and Vegetables using Triple Quad GC/MS/MS	Validated for over 150 pesticide residues	D. Brown	Varian, Inc. Application Note	Fruit and Vegetables	QuEChERS	PSA	FactorFour VF-5ms	GC/MS/MS	A02405
Determination of Trace of 151 Pesticides GC/MS in Foods and Vegetables	151 Pesticides in 30 m	A. de Kok M. Kroon	Varian, Inc. Application Note	Food, Vegetables	LLE	None	FactorFour VF-5 Pesticides	GC/MS (Ion Trap)	00578 2406GC
Multi-Residue Analysis of Pesticides in Spices Using GC/MS/MS	52 Pesticides inc. OCs, OPs, ONs, SPYs	M. Hetmanski	Lecture at Varian UK Seminar, July 2006	Spices	QuEChERS	PSA, Carbon	FactorFour VF-17ms	GC/MS/MS	
Miniaturized Automated Matrix Solid Phase Dispersion Extraction for the Preparation of Small Amounts of Solid Samples	On-line MSPD	E.M. Kristenson C.J. Slooten E.G.J. Haverkate R.J.J. Vreuls U.A.Th. Brinkman	Poster, Vrije Universiteit, Dept. of Analytical Chemistry and Applied Spectroscopy	Fruit and Insects	MSPD	C18, C8, Si	FactorFour VF-5ms	GC/MS	
Multi-Residue Analysis of Pesticides in Vegetable and Fruit Extracts by Gas Chromatography Mass Spectrometry Using the Varian 4000 GC/MS	80 Pesticides	H. Wang E. George	Varian, Inc. Application Note	Fruit and Vegetables	Proprietary QuEChERS	None	FactorFour VF-5ms	GC/MS	SI-00699
Determination of Pesticides in Fruit and Vegetables Using GC/MS/MS	EU Validation Method	D. Brown, T. Faye	Varian, Inc. Application Note	Fruit Tomato	QuEChERS	PSA	FactorFour VF-5 Pesticides	GC/MS/MS	SI-00587
The Determination of Multiple Pesticide Residues in Fruit and Vegetables Using Rapid Extraction and LC and GC Triple Quadrupole MS/MS	LC/GC Combined Strategy	D. Brown	Poster at British Mass Spectrometry Society Sept. 2004	Fruit and Vegetables	QuEChERS	PSA	GC: FactorFour VF-5ms, LC: Polaris™ C18 3 µm	GC/MS/MS LC/MS/MS	
Comparison of an Acetonitrile Extraction/Partitioning and "Dispersive Solid Phase Extraction" Method With Classical Multi-Residue Methods for the Extraction of Herbicide Residues in Barley Samples		C. Díez W.A. Traag P. Zommer P. Marinero J. Atienza	J. Chromatog. A 1131 (2006) 11–23	Barley Samples	QuEChERS, LLE. GPC	PSA	Pursuit XRs C18 VF-5 Pesticides VF-1701 Pesticides	GC/MS/MS LC/MS/MS GC/TOF/MS	
Analsyis of Pesticide Residues in Fruit and Vegetables With Ethyl Acetate Extraction Using Gas and Liquid Chromatography With Tandem Mass Spectrometric Detection		T. Pihlstrom G. Blomkvist P. Friman U. Pagard B. Osterdahl	Anal Bioanal Chem DOI 10.1007/ s00216-007-1425-6	Fruits and Vegetables	LLE	None	GC: FactorFour VF-5ms, LC: Pursuit XRs C18	GC/MS/MS LC/MS/MS	

Pesticides Application Bibliography

Pesticide Analysis in Low Fat Food Matrices Continued

Title of Paper	Key Method Feature	Authors	Source	Low Fat Matrix	Sample Preparation	Sorbent	Recommended Column	Analysis	Application Note No.
Efficient Analysis of Pesticide and Herbicide Residues in Food		D. Nau, et al.	European Food and Drink Review Winter pgs: 63-69, 1996	Variety of Foods	MSPD	C18, Fl	FactorFour™ VF-5ms LC: Pursuit™ C18	GC/MS, GC/ECD, LC/UV	M2807
Fast and Easy Multi-Residue Method Employing Acetonitrile Extraction/ Partitioning and "Dispersive Solid Phase Extraction" for the Determination of Pesticide Residues in Produce		M. Anastassiades S. Lehotay D. Stajnbaher F. Schenck	Journal of AOAC International 86(2): 412-431 (2003)	Fruit, Vegetables, Cereals	QuEChERS	PSA, Nexus, Carbon, SAX, NH_2, C18, Al-N, CN	Pursuit C18 FactorFour VF-5ms	LC/MS/MS GC/MS/MS	
New Developments in QuEChERs Methodology	Extending the scope of QuEChERS	M. Anastassiades B. Tasdelen E. Sherbaum	EPRW 2006, Corfu, Presentation	Fruit and Vegetables	QuEChERS	PSA, Carbon,	Pursuit C18 FactorFour VF-5ms	LC/MS/MS GC/MS	
Sample Preparation of Pesticide Residues from Foodstuffs with Chem Elut™	Elimination of Emulsions	L. Adler J. Klein	J. AOAC Intern. Vol. 86(5) p 1015-1037 (2003)	Fruits and Vegetables	SLE	Chem Elut	Pursuit C18	LC/MS/MS	Inspirations No. 2
Determination of Acidic Pesticides Using a Simple Modification of an Existing LC-MS/MS Method	Improved recoveries with Chem Elut pH 4.5	H. Zhang G. H. Pérez L. Alder	Poster presented at EPRW, May 2006	Food	SLE	Chem Elut pH 4.5	Pursuit XRs™ C18	LC/MS/MS	
One Year Routine Application of a New Method Based on Liquid Chromatography-Tandem Mass Spectronomy to the Analysis of 16 Multiclass Pesticides in Vegetable Samples	Robustness	A. Aguera et al.	J.Chrom A, 1045 (2004) 125-135	Vegetables	Ethyl Acetate	None	Polaris™ C18-A	LC/MS/MS	
Determination of Priority Pesticides in Baby Foods by Gas Chromatography Tandem Quadrupole Mass Spectrometry	Large Volume Injection GC	C. C. Leandro et al.	J.Chrom A, 1085 (2005) 207-212	Baby Foods	QuEChERS	PSA, C18	FactorFour VF-17ms	GC/MS/MS	
Fast and Sensitive Determination of Pesticide Residues Using Low-Pressure Gas Chromatography With Triple Quadrupole Mass Spectrometer	Low pressure GC	S. Walorczyk B. Gnusowski	J.Chrom A, 1128 (2006) 236-243	Vegetables	Ethyl acetate	Aminopropyl	FactorFour VF-5ms	GC/MS/MS	
A Mini-Multi-Residue Method for the Analysis of Pesticide Residues in Low-Fat Products	QuEChERS Method Protocol	M. Anastassiades	www.quechers.com	Foods	QuEChERS	PSA, C18, Carbon	No analytical methods referenced		
LC/MS/MS Analysis of Multi-Residue Pesticides in Vegetables Using Liquid Liquid Extraction and Pursuit XRs C18 HPLC Columns		R. Arora E. Chang G. Q. Li L.P. Raman	The Application Notebook (LC/GC), June 2007, p 38-39	Vegetables	LLE	None	Pursuit XRs C18	LC/MS/MS	
Development and Validation of a Multi-Residue Method for Pesticide Determination in Honey Using On-Column Liquid-Liquid Extraction and Liquid Chromatography-Tandem Mass Spectrometry		C. Pirard et al.	J. Chrom A, 1152,116-123 (2007)	Honey	SLE	Chem Elut	Polaris C18-A	LC/MS/MS	SI-01002



VARIAN, INC.

Pesticides Application Bibliography

Pesticide Analysis of Water Matrices and Standards Applications

Title of Paper	Key Method Feature	Authors	Standards, Water Matrix	Recommended Column	Analysis	Application Note No.
Analysis of EPA 625 / CLP Pesticides Using New Generation FactorFour™ VF-5 Pesticides		I. Krijnsen	Standards	FactorFour VF-5 Pesticides	GC/ECD	SI-00538
Analysis of EPA 8081 Pesticides Using New Generation FactorFour VF-5 Pesticides	20 Minute Run Time	I. Krijnsen	Standards	FactorFour VF-5 Pesticides	GC/ECD	SI-00539
Analysis of EPA 8081 Pesticides Using New Generation FactorFour VF-1701 Pesticides	20 Minute Run Time	I. Krijnsen	Standards	FactorFour VF-1701 Pesticides	GC/ECD	SI-00541
Pesticides at 50 ppb Using VF-1701 Pesticides and GC ECD	50 ppb Levels for 32 Pesticides	R. Wagner I. Hotaling	Standards	FactorFour VF-1701 Pesticides	GC/ECD	SI-00613
Chlordane standard at 1000 ppb Using GC ECD and VF-1701 Pesticides		R. Wagner I. Hotaling	Standards	FactorFour VF-1701 Pesticides	GC/ECD	SI-00614
Determination of Glyphosate and Aminomethylphosphonic Acid (AMPA) in Water Using HPLC	Results at 0.1ug/L	Mr. Charreteur, Centre de Genie Industriel	Water	Microsorb MV Amino	HPLC, UV/VIS	A02462
Determination of Triazine Herbicides and Their Degradates in Drinking Water (EPA 536) Using the Varian 320-MS LC/MS/MS Triple Quadrupole Mass Spectrometer	0.25 - 5 ppb levels	T. Payne	Water	Pursuit™ C18 3 µm	LC/MS/MS	00625
Dual Column 8081 Pesticide Analysis by GC ECD		I. Krijnsen	Standards	FactorFour VF-5 Pesticides FactorFour VF-1701 Pesticides	GC/ECD	SI-00924

To view available application notes, tips and other useful materials, visit our website at: www.varianinc.com/pesticides/

VARIAN, INC.

Complete Solutions for Pesticide Residue Analysis in Foods

FROM SAMPLE PREP TO HPLC AND GC ANALYSIS WITH MS/MS DETECTION

Varian, Inc. – Supporting Customers Worldwide

All products are eligible for on-demand support from a worldwide network of specialists who can assist with any need. Varian, Inc. offers:

- **Comprehensive programs** that include a full range of services – from minor repairs, to complex applications development and training.
- **Factory-qualified** Field Support Representatives and Specialists, specifically chosen for each installation.
- **Global support** teams strategically located around the world.

To learn more about Varian's industry leading customer support, visit our Web site at www.varianinc.com/support

Varian, Inc. – Serving Industries Worldwide

Biosciences

Pharmaceuticals

Clinical Research and Forensics

Food and Agriculture

Chemical Analysis

Environmental

Fuels and Energy

Material Sciences

Varian, Inc.
www.varianinc.com

North America: 800.926.3000, 925.939.2400
Europe The Netherlands: 31.118.67.1000
Asia Pacific Australia: 613.9560.7133
Latin America Brazil: 55.11.3238.0400

Other sales offices and dealers throughout the world– check our Web site.

GC • LC • MS • GPC/SEC • AA • ICP • ICP-MS • UV-Vis-NIR • FT-IR • Fluorescence • Dissolution • NMR • MRI • FTMS • Consumables • Data Systems

VARIAN

SI-0985/A/01.08 Printed in the UK.