

QuEchers - Sample Prep



Ideal for analysing multi-residue pesticides, veterinary drug residues in meat, vegetable and cereal products.

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Q-sep™ innovative chromatography solutions

QuEChERS Products

Fast, Simple Sample Preparation Multi-Residue Pesticide Analysis

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 Use Restek Q-sep in combination with our new QuEChERS method

- up to 100 samples throughput — 4 FID tubes — no modification to method.
- no data solvent usage up to 1000, with no solvent recovery.
- simultaneous multi-residue samples for routine analysis.

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Ideal kits for analyzing multiresidual pesticides, veterinary drug and additives in fruits, vegetables and meat products.

m EN 15662 Method

" AOAC 2007. 01 Method




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biocomma

Copure® QuEChERS

Ideal for analysing multiresidual pesticides, veterinary drug residues in foods, vegetable and animal products.

 <p>HPLC <small>High Performance Liquid Chromatography</small></p>	 <p>GC <small>Gas Chromatography</small></p>	 <p>MS <small>Mass Spectrometry</small></p>
<p>Restek: 800.765.8100 or www.restekcorp.com Email: sales@restekcorp.com Fax: 814.353.2800, ext. 8000</p>		

in DN 1000 Method in QAO 2007.01 Method

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Q-sep® innovative chromatography solutions

QuEChERS Products

Fast, Simple Sample Preparation Multi-Residue Pesticide Analysis

RESTEK EXCLUSIVE
 Over 100 different methods are ready to use for data collection.

- speed up sample throughput—100 times the small volume method.
- no toxic solvent usage up to 100% with no solvent recovery
- simultaneously process multiple samples for accurate analysis.

Chromatography Products



Company Profile

Biocomma Limited is an ISO certified leading manufacturer of sample preparation, sample filtration and sample collection products since 2006 and has formed three technology platforms of porous plastic filters, separation materials and precision injection molding.

The Analysis Business Unit of Biocomma specializes in developing and manufacturing SPE/QuEChERS.

We can offer the following products:

1. Copure® SPE Cartridges, include Polymer-based SPE Cartridges, Silica-based SPE Cartridges, SLE Cartridges, Immunoaffinity Columns, etc.
2. Copure® QuEChERS, include QuEChERS EN kits and AOAC kits, etc.
3. New mode SPE products, include 96/384-well Plates, Rimless SPE Cartridges, Multifunctional clean-up columns, etc.
4. Sample Preparation Equipments, include SPE Vacuum Manifolds, Oil-free Diaphragm Vacuum Pumps, Multi-Tube Vortexer, Positive Pressure-96 Extraction Processor, etc.
5. OEM and ODM services for SPE and QuEChERS products.



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Overview

In 2003, Michelangelo Anastassiades and Steven J Lehotay scientists who developed similar groundbreaking methods to simplify the way labs prepare food samples pesticide analysis. It's called QuEChERS. The "QuEChERS" (Quick, Easy, Cheap, Effective, Rugged, and Safe) method, dispersive SPE (dSPE), is a sample prep technique that has become popular in the area of multi-residue pesticide analysis in food and agricultural products.

Biocomma offers standard EN or AOAC QuEChERS kits, and also offers customized QuEChERS kits for customers, including different specifications of the centrifuge tube, extraction tube, purification tubes and reagents to help you quickly establish a standard detection method.

Features

- ♦ Satisfactory recoveries for a wide variety of

pesticides, veterinary drugs and additives in many food matrices

- ♦ Streamlined procedure with few simple steps, lowering potential errors
- ♦ Minimal organic solvent usage, safer for analysts and environment-friendly
- ♦ Saving time and cost significantly

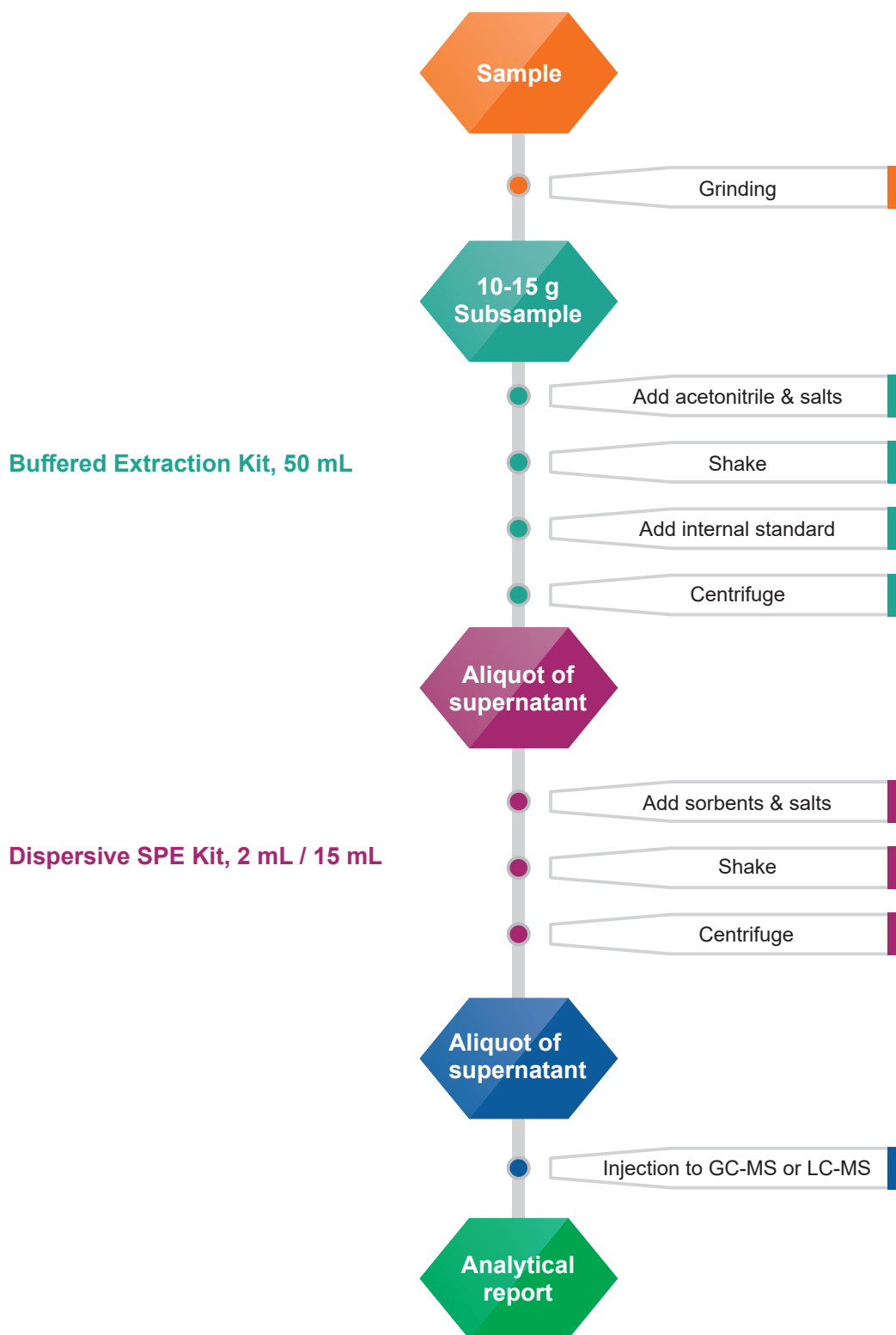
Related Methods

Biocomma provides QuEChERS kits dedicated for most common methods:

- ♦ **BS EN 15662:2018** Foods of plant origin-Multimethod for the determination of pesticide residues using GC- and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE-Modular QuEChERS-method.
- ♦ **AOAC Official Method 2007.01** Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.



Workflow



QuEChERS Extraction Kits

Copure® QuEChERS Kits includes extraction pouches and 50 mL centrifuge tubes, ceramic homogenizers are optional.

The pouches contain anhydrous extraction salts. Among the mixture, MgSO_4 is responsible for removing water from samples, while other components are responsible for maintaining appropriate pH to ensure the recoveries of alkaline-sensitive pesticides.

Directly adding water-abundant samples into tubes containing extraction salts may cause local overheating which compromise the resulting recoveries. To avoid such situations, Biocomma provides separate extraction salt pouches that the operator can add extraction salts after the addition of organic solvents.

Copure® QuEChERS salts are sealed in aluminum foil bags to avoid leakage. The type and amount are printed on the bag for handy choice. The easy-cut mark is very convenient for use. Our automated powder dispensing & packaging assembly line promise the accuracy and repeatability.



Order Information

AOAC 2007.01 Kits

Cat.#	Description	Sorbents	Qty.
COQ050020H	Extraction Salts+50 mL Tube	6 g MgSO_4	50/Box
COQ050020CH	Extraction Salts+50 mL Tube+Ceramic Homogenizers	1.5 g NaOAc	50/Box

BS EN 15662: 2018 Kits

Cat.#	Description	Sorbents	Qty.
COQ050010H	Extraction Salts+50 mL Tube	4 g MgSO_4 , 1 g NaCl	50/Box
COQ050010CH	Extraction Salts+50 mL Tube+Ceramic Homogenizers	1 g Trisodium Citrate, 0.5 g Disodium Citrate	50/Box

Original Method Kits

Cat.#	Description	Sorbents	Qty.
COQ050040H	Extraction Salts+50 mL Tube	4 g MgSO_4	50/Box
COQ050040CH	Extraction Salts+50 mL Tube+Ceramic Homogenizers	1 g NaCl	50/Box

Ceramic Homogenizers

Cat.#	Description	Qty.
009903B	Ceramic Homogenizers, 50 mL	100/Bottle

QuEChERS Premixed Extraction Salts

Copure® QuEChERS Premixed Extraction Salts are suitable for various QuEChERS Standards and used in analysis of multi-residual pesticides.

Features

- ◆ Optimized premixed formula, more flexible operation
- ◆ Two packages optional: easy-cut pouches and bottle package
- ◆ Suitable for AOAC 2007, EN 15662 standards, etc



Order Information

AOAC 2007.01 Kits

Cat.#	Description	Sorbents	Qty.
COQP6150	Extraction Pouches	6 g MgSO ₄	50/Box
COQS6150	Bottled Premixed Extraction Salts	1.5 g NaOAc	1 kg/Bottle

BS EN 15662: 2018 Kits

Cat.#	Description	Sorbents	Qty.
COQP4115	Extraction Pouches	4 g MgSO ₄ , 1 g NaCl	50/Box
COQS4115	Bottled Premixed Extraction Salts	1 g Trisodium Citrate, 0.5 g Disodium Citrate	1 kg/Bottle

Original Method Kits

Cat.#	Description	Sorbents	Qty.
COQP4100	Extraction Pouches	4 g MgSO ₄	50/Box
COQS4100	Bottled Premixed Extraction Salts	1 g NaCl	1 kg/Bottle

Copure® QuEChERS Extraction Salts

Suitable for AOAC 2007, EN15662 standards

Convenient Operation and Low Cost

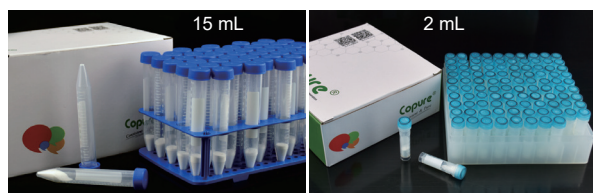
QuEChERS Clean-up Kits

Copure® QuEChERS Clean-up Kits includes sorbents and MgSO_4 , 2 mL and 15 mL centrifuge tubes, ceramic homogenizers are optional as well.

The sorbents include PSA/C18-EC/GCB, etc. PSA is to remove the fatty acids and organic acids from samples. C18-EC is to remove the fats from samples, GCB is to remove the pigments from samples. Choose appropriate sorbent combination with different samples.

Features

- ◆ Supply 2 mL or 15 mL purification tubes
- ◆ Suitable for AOAC 2007, EN 15662 standards, etc



Order Information

BS EN 15662: 2018 Kits

Cat.#	Size	Application	Sorbents	Qty.
COQ002030H	2 mL	General fruits and vegetables	25 mg PSA, 150 mg MgSO_4	100/Box
COQ015022H	15 mL		150 mg PSA, 900 mg MgSO_4	50/Box
COQ002032H	2 mL	General fruits and vegetables with fats and waxes	25 mg PSA, 25 mg C18, 150 mg MgSO_4	100/Box
COQ015032H	15 mL		150 mg PSA, 150 mg C18, 900 mg MgSO_4	50/Box
COQ002020H	2 mL	General fruits and vegetables with pigments	25 mg PSA, 2.5 mg GCB, 150 mg MgSO_4	100/Box
COQ015020H	15 mL		150 mg PSA, 15 mg GCB, 900 mg MgSO_4	50/Box
COQ002024H	2 mL	General fruits and vegetables with highly pigments	25 mg PSA, 7.5 mg GCB, 150 mg MgSO_4	100/Box
COQ015024H	15 mL		150 mg PSA, 45 mg GCB, 900 mg MgSO_4	50/Box

Ceramic Homogenizers

Cat.#	Description	Qty.
009902B	Ceramic Homogenizers, 15 mL	100/Bottle
009901B	Ceramic Homogenizers, 2 mL	200/Bottle

AOAC 2007.01 Kits

Cat.#	Size	Application	Sorbents	Qty.
COQ002031H	2 mL	General fruits and vegetables	50 mg PSA, 150 mg MgSO_4	100/Box
COQ015031H	15 mL		400 mg PSA, 1200 mg MgSO_4	50/Box
COQ002033H	2 mL	General fruits and vegetables with fats and waxes	50 mg PSA, 50 mg C18, 150 mg MgSO_4	100/Box
COQ015033H	15 mL		400 mg PSA, 400 mg C18, 1200 mg MgSO_4	50/Box
COQ002036H	2 mL	General fruits and vegetables with pigments	50 mg PSA, 50 mg GCB, 150 mg MgSO_4	100/Box
COQ015036H	15 mL		400 mg PSA, 400 mg GCB, 1200 mg MgSO_4	50/Box
COQ002040H	2 mL	General fruits and vegetables with pigments and fats	50 mg PSA, 50 mg C18, 50 mg GCB, 150 mg MgSO_4	100/Box
COQ015040H	15 mL		400 mg PSA, 400 mg C18, 400 mg GCB, 1200 mg MgSO_4	50/Box
COQ002025H	2 mL	Other food methods	25 mg C18, 150 mg MgSO_4	100/Box
COQ015025H	15 mL		150 mg C18, 900 mg MgSO_4	50/Box
COQ002035H	2 mL	All food types	50 mg PSA, 50 mg C18, 7.5 mg GCB, 150 mg MgSO_4	100/Box
COQ015035H	15 mL		400 mg PSA, 400 mg C18, 45 mg GCB, 1200 mg MgSO_4	50/Box

QuEChERS Clean-up Pouches

Copure® QuEChERS Clean-up Pouches are used for analysing multiresidual pesticides. Biocomma uses its automatic powder distribution technology to transfer the sorbent into pouches instead of tube, which is very convenient to match with customer's own 15 mL centrifuge tubes.

Features

- ◆ Save 50% of volume, convenient for transportation, saving laboratory space
- ◆ Easy-Cut package to open easily without any cutting tooling
- ◆ Lower cost, suitable for mass quantity testing



Order Information

Cat.#	Type	Sorbents	Qty.
COQ015031P	AOAC 2007	400 mg PSA, 1200 mg MgSO ₄	100/Box
COQ015033P	AOAC 2007	400 mg PSA, 400 mg C18, 1200 mg MgSO ₄	100/Box
COQ015036P	AOAC 2007	400 mg PSA, 400 mg GCB, 1200 mg MgSO ₄	100/Box
COQ015040P	AOAC 2007	400 mg PSA, 400 mg C18, 400 mg GCB, 1200 mg MgSO ₄	100/Box
COQ015025P	AOAC 2007	150 mg C18, 900 mg MgSO ₄	100/Box
COQ015035P	AOAC 2007	400 mg PSA, 400 mg C18, 45 mg GCB, 1200 mg MgSO ₄	100/Box
COQ015022P	EN 15662	150 mg PSA, 900 mg MgSO ₄	100/Box
COQ015032P	EN 15662	150 mg PSA, 150 mg C18, 900 mg MgSO ₄	100/Box
COQ015020P	EN 15662	150 mg PSA, 15 mg GCB, 900 mg MgSO ₄	100/Box
COQ015024P	EN 15662	150 mg PSA, 45 mg GCB, 900 mg MgSO ₄	100/Box



Multi-Tube Vortexer

QuEChERS Good Helper

QuEChERS Bulk Sorbents

Biocomma provides superior quality QuEChERS bulk sorbents which have been verified by our lab.

Order Information

Cat.#	Sorbent	Specification	Qty.
PSA-2-100	PSA	Carbon Content: 8%, Surface area: 480 m ² /g, Particle size: 50-75 µm, Pore size: 70 Å	100 g
C18-1-100	C18	Carbon Content: 17.6%, Surface area: 300 m ² /g, Particle size: 40-75 µm, Pore size: 70 Å	100 g
GCB-1-50	Carb-GCB	Surface area: 100 m ² /g, Particle size: 100-300 mesh	50 g
MGS04-1	Anhydrous MgSO ₄	AR Grade	1 kg
NAOAC-1	NaOAc	AR Grade	1 kg
NaCL-1	NaCl	AR Grade	1 kg
CIT-1	Trisodium Citrate	AR Grade	1 kg
CIT2-1	Disodium Citrate	AR Grade	1 kg

QuEChERS Ceramic Homogenizers

biocomma[®] Ceramic Homogenizers are used for Copure[®] QuEChERS extraction kit and clean-up kit, increase recovery and reproducibility.

Features

- ◆ Inert ceramic material, no impurities dissolution
- ◆ Shorten sample extraction time and reduce labor cost
- ◆ Increase recovery and reproducibility of sample extraction



Order Information

Cat.#	Description	Qty.
009903B	Ceramic Homogenizers, 50 mL	100/Bottle
009902B	Ceramic Homogenizers, 15 mL	100/Bottle
009901B	Ceramic Homogenizers, 2 mL	200/Bottle

biocomma[®] Multi-Tube Vortexer

biocomma[®] BC-1000 is a multi-tube vortexer with various functions and powerful shaking of sample, especially suitable for QuEChERS, as well as general sample extraction. With strong vortex and shearing force, it boosts sample dissolution and blending.

Features

- ◆ 2500 r/min Sufficient extraction of samples
- ◆ Optional intermittent pulse blending mode, suitable for viscous samples
- ◆ Specially designed for QuEChERS extraction and purification, ensures vortex result
- ◆ Matching special centrifuge tube rack, easy observation
- ◆ The extraction efficiency of positive samples meets the requirements



Specifications

Part No.	BC-1000
Speed Range	500-2500 rpm
Accuracy of speed	±1 rpm
Amplitude	3.6 mm
Timer Range	0 s~99 H 59 M
Interval pause timing range	1~99 s
Interval operation timing range	1~999 s
Maximum Loading Capacity	4.5 kg
Input power	AC 100~230 V, 50/60 Hz
Capacity	75 W
Size(L × W × H)	426 × 246 × 474 mm

Order Information

Cat.#	Description	Qty.
BC-1000	biocomma [®] multi-tube Vortexer	1 set/Box

Solid Phase Extraction Products

Copure® SPE Cartridges cover polymer-based, silica-based and adsorption-based SPE Cartridges. They can be assembled into different specifications and widely used in food, medical and industrial field, etc.



Order Information

Polymer-based SPE Cartridges

Sorbent	Cat.#					
	30mg/1mL	60mg/3mL	200mg/3mL	150mg/6mL	500mg/6mL	1000mg/12mL
HLB	COHLB130	COHLB360	COHLB3200	COHLB6150	COHLB6500	COHLB121000
MCX	COMCX130	COMCX360	COMCX3200	COMCX6150	COMCX6500	COMCX121000
MAX	COMAX130	COMAX360	COMAX3200	COMAX6150	COMAX6500	COMAX121000
WCX	COWCX130	COWCX360	COWCX3200	COWCX6150	COWCX6500	COWCX121000
WAX	COWAX130	COWAX360	COWAX3200	COWAX6150	COWAX6500	COWAX121000

Silica-based SPE Cartridges

Sorbent	Cat.#					
	100mg/1mL	200mg/3mL	500mg/3mL	500mg/6mL	1000mg/6mL	1000mg/12mL
C18	COC181100	COC183200	COC183500	COC186500	COC1861000	COC18121000
C8	COC81100	COC83200	COC83500	COC86500	COC861000	COC8121000
Silica	COSIL1100	COSIL3200	COSIL3500	COSIL6500	COSIL61000	COSIL121000
Diol	CODI1100	CODI3200	CODI3500	CODI6500	CODI61000	CODI121000
CN	COCN1100	COCN3200	COCN3500	COCN6500	COCN61000	COCN121000
SCX	COSCX1100	COSCX3200	COSCX3500	COSCX6500	COSCX61000	COSCX121000
SAX	COSAX1100	COSAX3200	COSAX3500	COSAX6500	COSAX61000	COSAX121000
NH ₂	CONH1100	CONH3200	CONH3500	CONH6500	CONH61000	CONH121000
PSA	COPSA1100	COPSA3200	COPSA3500	COPSA6500	COPSA61000	COPSA121000
PRS	COPRS1100	COPRS3200	COPRS3500	COPRS6500	COPRS61000	COPRS121000

Adsorption-based SPE Cartridges

Sorbent	Cat.#					
	100mg/1mL	200mg/3mL	500mg/3mL	500mg/6mL	1000mg/6mL	1000mg/12mL
Carb-GCB	COGCB1100	COGCB3200	COGCB3500	COGCB6500	COGCB61000	COGCB121000
ALA	COALA1100	COALA3200	COALA3500	COALA6500	COALA61000	COALA121000
ALN	COALN1100	COALN3200	COALN3500	COALN6500	COALN61000	COALN121000
ALB	COALB1100	COALB3200	COALB3500	COALB6500	COALB61000	COALB121000
Florisil	COFL1100	COFL3200	COFL3500	COFL6500	COFL61000	COFL121000

Note: For SPE Cartridge of other specs, please contact us.

More Customization



Formulation

Custom Sorbents
Custom Ratios
Application-Specific
Optimization



Packaging

Brand Logos
Custom Brand Packages
Neutral Packages



Applications

Pesticide Residues
Veterinary Drug Residues
Application-Specific
Solutions



OEM 50 mL 提取管



OEM 15 mL 净化管



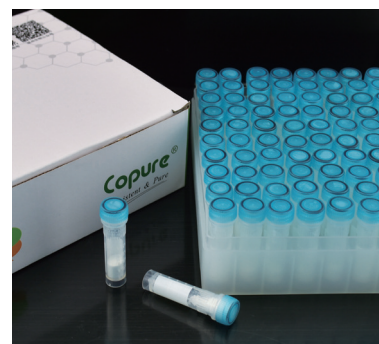
OEM 2 mL 净化管



Copure® 50 mL 提取管



Copure® 15 mL 净化管



Copure® 2 mL 净化管

Analysis of Pesticide Residues in Cucumber Using Copure® QuEChERS EN kits by GC-ECD and LC-MS/MS

Application Scope

This method applies to analyse and validate multi-residual pesticides in general fruits and vegetables.

Reference

BS EN 15662-2008: Foods of plant origin-Determination of pesticide residues using GC-MS and LC-MS/MS

Materials and Equipment

Copure® QuEChERS EN Buffered Extraction kit (Cat. No. COQ050010H)

Copure® QuEChERS EN Dispersive SPE kit for general vegetables and fruits (Cat. No. COQ015022H)

biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Homogenize a cucumber sample that was frozen at -18 °C. Weigh 10.0 g of homogenized cucumber sample into a 50 mL centrifuge tube, add 10 mL acetonitrile solution, and shake for 1 min. Then Add an EN buffered extraction salt pouch containing 4 g anhydrous MgSO₄, 1 g NaCl, 1 g NaCitrate and 0.5 g disodium citrate sesquihydrate (Cat. No. COQ050010H) into the 50 mL centrifuge tube. Vortex for 10 min, then centrifuge for 5 min at 4000 rpm. The upper acetonitrile layer is being cleaned up in the following step.

Dispersive SPE cleanup

Transfer 6 mL of the upper acetonitrile layer into a QuEChERS EN dispersive SPE 15 mL tube containing 900 mg MgSO₄ and 150 mg PSA (Cat. No. COQ015022H). Vortex for 1 min, then centrifuge for 5 min at 4000 rpm. Transfer 1 mL of supernatant, pass through a 0.22 µm membrane, ready for GC-ECD and LC/MS/MS analysis.

Chromatographic analysis

GC-ECD conditions

System: Agilent 7890A

Columns: Agilent J&W HP-5(30 m x 0.32 mm, 0.25 µm) or equivalent

Injection port temperature: 220 °C

Detector temperature: 300 °C

Oven temperature: 180 °C (2 min)

10 °C /min to 230 °C (2 min)

2 °C/min to 260 °C (2 min)

25 °C/min to 270 °C (1.6 min)

Carrier gas: Helium

Flow rate: 1.6 mL/min

Inlet: 220 °C, 1 µL, split 10:1

LC-MS/MS conditions

System: API 4000

Column: Venusil ASB C18 (2.1 mm x 150 mm, 5µm)

Mobile Phase: A: 0.1% HCOOH and 10 mM ammonium acetate in H₂O (Add 1 mL HCOOH and 0.77 g ammonium acetate into 1 L aqueous solution.)

B: MeOH

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	95	5
1.5	95	5
6.0	5	95
11.0	5	95
11.1	95	5
15.0	95	5

Flow rate: 0.35 mL/min

Column temperature: 40 °C

Injection volume: 5 µL

Ion source: ESI

Ionization mode: Positive

Scan mode: MRM

Ion source parameters conditions are listed in Table 2.

Table 2. Ion source parameters conditions

Collision Gas (CAD)	6 psi, N ₂
Curtain Gas (CUR)	12 psi, N ₂
Ion Source Gas 1 (GS1)	50 psi, N ₂
Ion Source Gas 2 (GS2)	50 psi, N ₂
Ion Spray Voltage (IS)	5500 V
Temperature (TEM)	550 °C
Interface Heater (IHE)	ON

Other conditions relating to the analytes are listed in Table 3.

Table 3. Instrument Acquisition Data for the Analysis of Carbamate Pesticides by LC/MS/MS

Compound	RT(min)	MRM channels(m/z)	DP	EP	CE	CXP
Aldicarb	7.06	208.1>89.1	30	10	22	12
		208.1>116.0	30	10	10	12
Carbofuran	7.13	222.3>123.1	48	10	16	12
		222.3>165.2	48	10	31	12
Methomyl	6.51	163.2>88.1	36	10	15	12
		163.2>106.1	36	10	12	12
Aldicarb sulfone	6.25	223.1>86.2	69	10	21	12
		223.1>148.1	69	10	13	12
Aldicarb sulfoxide	6.10	207.1>132.2	60	10	13	12
		207.1>89.1	60	10	22	12
Acetamiprid	6.83	223.4>126.1	70	10	29	12
		223.4>90.0	70	10	46	12
Carbaryl	7.18	202.1>145.2	58	10	12	12
		202.1>127.1	58	10	40	12
Carbendazim	6.82	192.1>160.1	68	10	34	12
		192.1>132.2	68	10	42	12

Results

Results of spiked multi-residual pesticides in cucumber

Table 4. Recoveries and relative standard deviations (RSD) of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in cucumber

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
α -666	97.5	91.0	89.5	92.7	4.59
β -666	101.5	94.0	91.5	95.7	5.44
γ -666	100.5	94.5	91.0	95.3	5.04
δ -666	98.5	96.5	95.4	96.8	1.63
p,p'-DDE	90.0	86.0	83.0	86.3	4.07
p,p'-DDD	100.5	91.2	91.0	94.2	5.76
p,p'-DDT	101.0	100.0	92.0	97.7	5.05
o,p'-DDT	100.0	98.5	89.0	95.8	6.22
Quintozene	104.0	102.5	97.0	101.2	3.64
Vinclozolin	85.2	82.5	86.5	84.7	2.41
Procymidone	115.0	112.0	110.0	112.3	2.24
Bifenthrin	96.5	94.5	89.5	93.5	3.86
Fenpropathrin	105.0	103.5	96.0	101.5	4.75
λ -Cyhalothrin	96.2	94.3	89.8	93.4	3.52
Cyfluthrin	91.1	102.2	89.5	94.3	7.34
Cypermethrin	91.5	92.1	85.9	89.8	3.81
Flucythrinate	100.8	92.1	102.5	98.5	5.67
Fenvalerate	106.1	116.5	112.0	111.5	4.68
Tau-fluvalinate	116.5	107.0	114.0	112.5	4.38
Deltamethrin	102.5	93.4	104.8	100.2	6.01

Table 5. Recoveries and relative standard deviations (RSD) of carbamate pesticides spiked at 0.05 mg/kg in cucumber

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Aldicarb	92.0	100.0	101.2	97.7	5.12
Carbofuran	94.0	95.6	91.4	93.7	2.26
Methomyl	100.0	94.4	89.0	94.5	5.82
Aldicarb sulfone	94.0	94.2	91.4	93.2	1.68
Aldicarb sulfoxide	99.4	95.0	89.5	94.6	5.24
Acetamiprid	103.6	102.6	92.8	99.7	5.99
Carbaryl	95.2	93.8	92.5	93.8	1.44
Carbendazim	97.6	96.4	95.6	96.5	1.46

Chromatograms of spiked multi-residual pesticides in cucumber

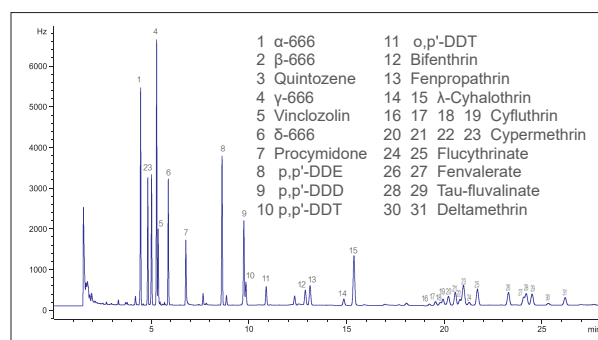


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in cucumber

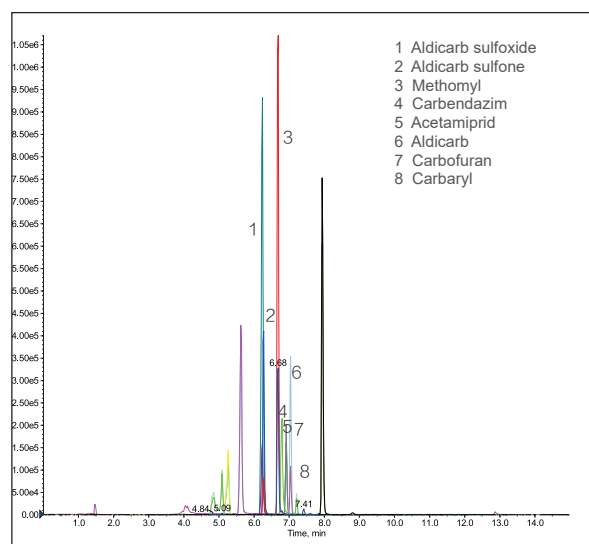


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in cucumber

Order Information

Cat.#	Description	Qty.
COQ050010H	4 g MgSO ₄ , 1 g NaCl, 1 g Trisodium Citrate and 0.5 g Disodium Citrate, 50 mL Tube	50/Box
COQ015022H	900 mg MgSO ₄ , 150 mg PSA, 15 mL Tube	50/Box
SF130-22-PTFE	PTFE / φ13 mm / 0.22 μm / Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-5	2 mL Blue PP Cover with White PTFE / red Silicone Septum, Pre-opening, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

Analysis of Pesticide Residues in Flowering Cabbage Using Copure® QuEChERS EN Kits by GC-ECD and LC-MS/MS

Application Scope

This method applies to analyse and validate multi-residual pesticides in high pigment fruits and vegetables.

Reference

BS EN 15662-2008: Foods of plant origin-Determination of pesticide residues using GC-MS and LC-MS/MS

Materials and Equipment

Copure® QuEChERS EN Buffered Extraction kit (Cat. No. COQ050010H)

Copure® QuEChERS EN Dispersive SPE kit for high pigment fruits and vegetables (Cat. No. COQ015024H)
biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Homogenize a flowering cabbage sample that was frozen at -18 °C. Weigh 10.0 g of homogenized flowering cabbage sample into a 50 mL centrifuge tube, add 10 mL acetonitrile solution, and shake for 1 min. Then Add an EN buffered extraction salt pouch containing 4 g anhydrous MgSO₄, 1 g NaCl, 1 g NaCitrate and 0.5 g disodium citrate sesquihydrate (Cat. No. COQ050010H) into the 50 mL centrifuge tube. Vortex for 10 min, then centrifuge for 5 min at 4000 rpm. The upper acetonitrile layer is being cleaned up in the following step.

Dispersive SPE cleanup

Transfer 2 mL toluene into a QuEChERS EN dispersive SPE 15 mL tube containing 900mg MgSO₄, 150 mg PSA and 45 mg GCB (Cat. No. COQ015024H), and vortex for 30 s. And then transfer 6 mL of the upper acetonitrile layer into the QuEChERS EN dispersive SPE 15 mL tube (Cat. No. COQ015024H). Vortex for 1 min, then centrifuge for 5 min at 4000 rpm. Transfer 1 mL of supernatant, pass through a 0.22 µm membrane, ready for GC-ECD and LC/MS/MS analysis.

Chromatographic analysis

GC-ECD conditions

System: Agilent 7890A

Columns: Agilent J&W HP-5(30 m x 0.32 mm, 0.25 µm)
or equivalent

Injection port temperature: 220 °C

Detector temperature: 300 °C

Oven temperature: 180 °C (2 min)

10 °C /min to 230 °C (2 min)

2 °C/min to 260 °C (2 min)

25 °C/min to 270 °C (1.6 min)

Carrier gas: Helium

Flow rate: 1.6 mL/min

Inlet: 220 °C, 1 µL, split 10:1

LC-MS/MS conditions

System: API 4000

Column: Venusil ASB C18 (2.1 mm x 150 mm, 5µm)

Mobile Phase: A: 0.1% HCOOH and 10 mM ammonium acetate in H₂O (Add 1 mL HCOOH and 0.77 g ammonium acetate into 1 L aqueous solution.)

B: MeOH

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	95	5
1.5	95	5
6.0	5	95
11.0	5	95
11.1	95	5
15.0	95	5

Flow rate: 0.35 mL/min

Column temperature: 40 °C

Injection volume: 5 µL

Ion source: ESI

Ionization mode: Positive

Scan mode: MRM

Ion source parameters and conditions are listed in Table 2.

Table 2. Ion source parameters conditions

Collision Gas (CAD)	6 psi, N ₂
Curtain Gas (CUR)	12 psi, N ₂
Ion Source Gas 1 (GS1)	50 psi, N ₂
Ion Source Gas 2 (GS2)	50 psi, N ₂
Ion Spray Voltage (IS)	5500 V
Temperature (TEM)	550 °C
Interface Heater (IHE)	ON

Other conditions relating to the analytes are listed in Table 3.

Table 3. Instrument Acquisition Data for the Analysis of Carbamate Pesticides by LC/MS/MS

Compound	RT(min)	MRM channels(m/z)	DP	EP	CE	CXP
Aldicarb	7.06	208.1>89.1	30	10	22	12
		208.1>116.0	30	10	10	12
Carbofuran	7.13	222.3>123.1	48	10	16	12
		222.3>165.2	48	10	31	12
Methomyl	6.51	163.2>88.1	36	10	15	12
		163.2>106.1	36	10	12	12
Aldicarb sulfone	6.25	223.1>86.2	69	10	21	12
		223.1>148.1	69	10	13	12
Aldicarb sulfoxide	6.10	207.1>132.2	60	10	13	12
		207.1>89.1	60	10	22	12
Acetamiprid	6.83	223.4>126.1	70	10	29	12
		223.4>90.0	70	10	46	12
Carbaryl	7.18	202.1>145.2	58	10	12	12
		202.1>127.1	58	10	40	12

Results

Results of spiked multi-residual pesticides in flowering cabbage

Table 4. Recoveries and relative standard deviations (RSD) of organochlorine and pyrethroid pesticides spiked at 0.26 mg/kg in flowering cabbage

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
α-666	92.5	95.5	93.5	93.8	1.63
β-666	93.0	98.1	95.5	95.5	2.67
γ-666	96.0	95.5	93.0	94.8	1.69
δ-666	98.0	95.5	95.0	96.2	1.67
p,p'-DDE	87.0	89.5	87.5	88.0	1.50
p,p'-DDD	91.5	98.2	96.5	95.4	3.65
p,p'-DDT	102.5	105.0	98.0	101.8	3.48
o,p'-DDT	99.5	97.5	97.5	98.2	1.18
Quintozene	84.0	87.5	83.6	85.0	2.52
Vinclozolin	85.2	82.5	88.5	85.4	3.52
Procymidone	102.5	99.8	99.0	100.4	1.83
Bifenthrin	91.5	90.5	87.5	89.8	2.32
Fenpropathrin	103.0	100.0	96.0	99.7	3.52
λ-Cyhalothrin	96.2	93.5	95.6	95.1	1.49

Table 5. Recoveries and relative standard deviations (RSD) of carbamate pesticides spiked at 0.06 mg/kg in flowering cabbage

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Aldicarb	91.2	85.7	90.6	89.2	3.38
Carbofuran	99.6	91.6	90.4	93.9	5.33
Methomyl	94.4	90.4	88.4	91.1	3.35
Aldicarb sulfone	96.4	91.0	90.8	92.7	3.43
Aldicarb sulfoxide	94.0	88.0	91.0	91.0	3.30
Acetamiprid	102.0	94.0	92.8	96.3	5.20
Carbaryl Carbendazim	82.0	74.0	80.0	78.7	5.29

Chromatograms of spiked multi-residual pesticides in flowering cabbage

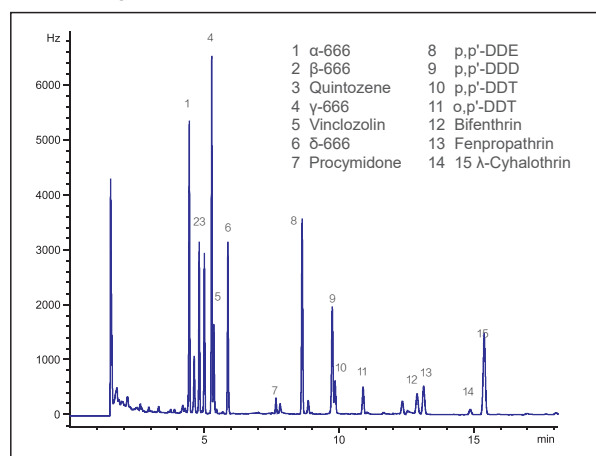


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.26 mg/kg in flowering cabbage

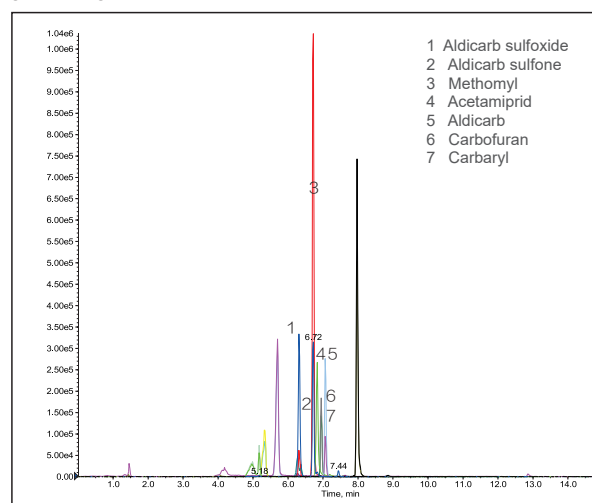


Figure 2. Chromatogram of carbamate pesticides spiked at 0.06 mg/kg in flowering cabbage

Order Information

Cat.#	Description	Qty.
COQ050010H	4 g MgSO ₄ , 1 g NaCl, 1 g Trisodium Citrate and 0.5 g Disodium Citrate, 50 mL Tube	50/Box
COQ015024H	900 mg MgSO ₄ , 150 mg PSA, 45 mg GCB, 15 mL Tube	50/Box
SF130-22-PTFE	PTFE /φ13 mm /0.22 μm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-5	2 mL Blue PP Cover with White PTFE/red Silicone Septum,Pre-opening, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

Analysis of Pesticide Residues in Rice Using Copure® QuEChERS EN Kits by GC-ECD and LC-MS/MS

Application Scope

This method applies to analyse and validate multi-residual pesticides in fruits and vegetables with fatty and waxy.

Reference

BS EN 15662-2008: Foods of plant origin-Determination of pesticide residues using GC-MS and LC-MS/MS

Materials and Equipment

Copure® QuEChERS EN Buffered Extraction kit (Cat. No. COQ050010H)

Copure® QuEChERS EN Dispersive SPE kit for fruits and vegetables with fatty and waxy (Cat. No. COQ015032H)
biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Homogenize a rice sample that was frozen at -18 °C. Weigh 10.0 g of homogenized rice sample into a 50 mL centrifuge tube, add 10 mL acetonitrile solution, and shake for 1 min. Then Add an EN buffered extraction salt pouch containing 4 g anhydrous MgSO₄, 1 g NaCl, 1 g NaCitrate and 0.5 g disodium citrate sesquihydrate (Cat. No. COQ050010H) into the 50 mL centrifuge tube. Vortex for 10 min, then centrifuge for 5 min at 4000 rpm. The upper acetonitrile layer is being cleaned up in the following step.

Dispersive SPE cleanup

Transfer 6 mL of the upper acetonitrile layer into a QuEChERS EN dispersive SPE 15 mL tube containing 900 mg MgSO₄, 150 mg PSA and 150 mg C18 (Cat. No. COQ015032H). Vortex for 1 min, then centrifuge for 5 min at 4000 rpm. Transfer 1 mL of supernatant, pass through a 0.22 µm membrane, ready for GC-ECD and LC/MS/MS analysis.

Chromatographic analysis

GC-ECD conditions

System: Agilent 7890A

Columns: Agilent J&W HP-5(30 m x 0.32 mm, 0.25 µm)

or equivalent

Injection port temperature: 220 °C

Detector temperature: 300 °C

Oven temperature: 180 °C (2 min)

10 °C /min to 230 °C (2 min)

2 °C/min to 260 °C (2 min)

25 °C/min to 270 °C (1.6 min)

Carrier gas: Helium

Flow rate: 1.6 mL/min

Inlet: 220 °C, 1 µL, split 10:1

LC-MS/MS conditions

System: API 4000

Column: Venusil ASB C18 (2.1 mm x 150 mm, 5µm)

Mobile Phase: A: 0.1% HCOOH and 10 mM ammonium acetate in H₂O (Add 1 mL HCOOH and 0.77 g ammonium acetate into 1 L aqueous solution.)

B: MeOH

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	95	5
1.5	95	5
6.0	5	95
11.0	5	95
11.1	95	5
15.0	95	5

Flow rate: 0.35 mL/min

Column temperature: 40 °C

Injection volume: 5 µL

Ion source: ESI

Ionization mode: Positive

Scan mode: MRM

Ion source parameters conditions are listed in Table 2.

Table 2. Ion source parameters conditions

Collision Gas (CAD)	6 psi, N ₂
Curtain Gas (CUR)	12 psi, N ₂
Ion Source Gas 1 (GS1)	50 psi, N ₂
Ion Source Gas 2 (GS2)	50 psi, N ₂
Ion Spray Voltage (IS)	5500 V
Temperature (TEM)	550 °C
Interface Heater (IHE)	ON

Other conditions relating to the analytes are listed in

Table 3.

Table 3. Instrument Acquisition Data for the Analysis of Carbamate Pesticides by LC/MS/MS

Compound RT(min)	RT(min)	MRM channels(m/z)	DP	EP	CE	CXP
Aldicarb	7.06	208.1>89.1	30	10	22	12
		208.1>116.0	30	10	10	12
Carbofuran	7.13	222.3>123.1	48	10	16	12
		222.3>165.2	48	10	31	12
Methomyl	6.51	163.2>88.1	36	10	15	12
		163.2>106.1	36	10	12	12
Aldicarb sulfone	6.25	223.1>86.2	69	10	21	12
		223.1>148.1	69	10	13	12
Aldicarb sulfoxide	6.10	207.1>132.2	60	10	13	12
		207.1>89.1	60	10	22	12
Carbendazim	6.82	192.1>160.1	68	10	34	12
		192.1>132.2	68	10	42	12
Carbaryl	7.18	202.1>145.2	58	10	12	12
		202.1>127.1	58	10	40	12

Results

Results of spiked multi-residual pesticides in rice

Table 4. Recoveries and relative standard deviations (RSD) of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in rice

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Quintozene	82.0	81.0	83.0	82.0	1.22
Chlorothalonil	84.0	86.0	91.5	87.1	4.46
Vinclozolin	84.0	81.5	84.0	83.1	1.74
Triazolone	103.5	99.0	102.5	101.6	2.32
Procymidone	98.4	94.5	97.5	96.8	2.11
Iprodione	103.5	98.0	100.0	100.5	2.77
Bifenthrin	107.5	101.5	107.5	105.5	3.28
Fenpropathrin	86.5	81.5	85.0	84.3	3.04
Beta-cyfluthrin	92.0	87.5	88.4	89.3	2.67
Cyfluthrin	87.6	85.4	85.8	86.2	1.34
Cypermethrin	71.5	76.8	71.2	73.2	4.34
Flucythrinate	131.2	123.2	124.5	126.3	3.40
Fenvalerate	102.5	98.5	88.0	96.3	7.77
Fluvalinate	92.9	90.4	90.7	91.3	1.49
Deltamethrin	117.5	111.5	109.0	112.6	3.88

Table 5. Recoveries and relative standard deviations (RSD) of carbamate pesticides spiked at 0.05 mg/kg in rice

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Aldicarb	94.6	93.6	99.2	95.8	3.12
Carbofuran	89.6	91.6	91.2	90.8	1.17
Methomyl	107.6	114.4	111.2	110.0	3.06
Carbendazim	75.8	82.0	82.6	80.1	4.70
Aldicarb sulfone	97.2	104.4	101.2	100.9	3.57
Aldicarb sulfoxide	93.0	100.0	101.6	98.2	4.66
Carbaryl Carbendazim	86.6	85.8	92.6	88.3	4.21

Chromatograms of spiked multi-residual pesticides in rice

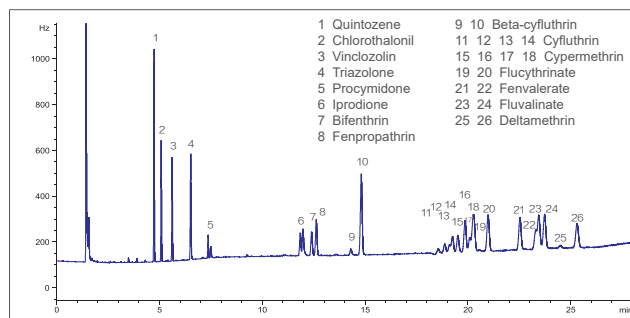


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in rice

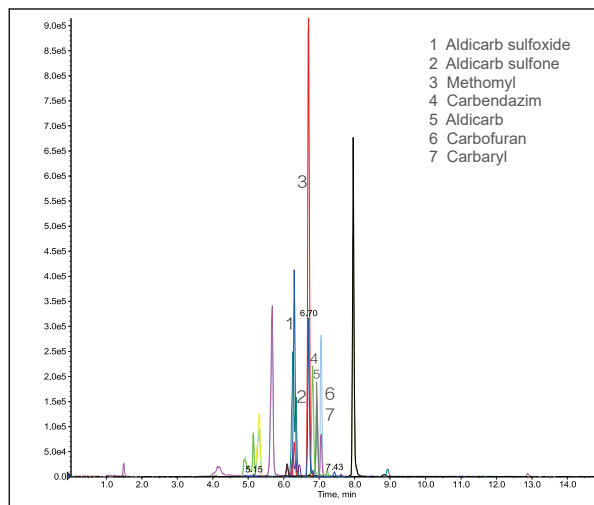


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in rice

Order Information

Cat.#	Description	Qty.
COQ050010H	4 g MgSO ₄ , 1 g NaCl, 1 g Trisodium Citrate and 0.5 g Disodium Citrate, 50 mL Tube	50/Box
COQ015032H	900 mg MgSO ₄ , 150 mg PSA, 150 mg C18, 15 mL Tube	50/Box
SF130-22-PTFE	PTFE /φ13 mm /0.22 μm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-5	2 mL Blue PP Cover with White PTFE/red Silicone Septum,Pre-opening, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

This method applies to analyse and validate multi-residual pesticides in general fruits and vegetables.

AOAC Method 2007.01: Pesticide Residues in Foods by
Acetonitrile Extraction and Partitioning with Magnesium
Sulfate

Copure® QuEChERS AOAC Buffered Extraction kit (Cat. No. COQ050020H)
 Copure® QuEChERS AOAC Dispersive SPE kit for general vegetables and fruits (Cat. No. COQ015031H)
 biocomma® multi-tube vortexer (Cat. No. BC-1000)

Homogenize a cucumber sample that was frozen at -18 °C. Weigh 15.0 g of homogenized cucumber sample into a 50 mL centrifuge tube, add 15 mL of 1% acetic acid in acetonitrile solution. Add an AOAC buffered extraction salt pouch containing 6 g anhydrous $MgSO_4$ and 1.5 g of anhydrous sodium acetate (Cat. No. COQ050020H). Vortex for 10 min, then centrifuge for 5 min at 4000 rpm. The upper acetonitrile layer is being cleaned up in the following step.

Transfer 8 mL of the upper acetonitrile layer into a QuEChERS AOAC dispersive SPE 15 mL tube containing 1.2 g MgSO₄ and 400 mg PSA (Cat. No. COQ015031H). Vortex for 1 min, then centrifuge for 5 min at 4000 rpm. Transfer 1 mL of supernatant, pass through a 0.22 µm membrane, ready for LC/MS/MS analysis.

System: API 4000

Column: Venusil ASB C18 (2.1 mm x 150 mm, 5µm)

Mobile Phase: A: 0.1% HCOOH and 10 mM ammonium acetate in H₂O (Add 1 mL HCOOH and 0.77 g ammonium acetate into 1 L aqueous solution.)

B: MeOH

Elution mode: Gradient elution (as in Table 1)

Time/min	A(%)	B(%)
--	95	5
1.50	95	5
6.0	5	95
11.0	5	95
11.1	95	5
15.0	95	5

Flow rate: 0.35 mL/min

Column temperature: 40 °C

Injection volume: 5 μ L

Ion source: ESI

Ionization mode: Positive

Scan mode: MRM

Ion source parameters conditions are listed in Table 2.

Collision Gas (CAD)	6 psi, N ₂
Curtain Gas (CUR)	12 psi, N ₂
Ion Source Gas 1 (GS1)	50 psi, N ₂
Ion Source Gas 2 (GS2)	50 psi, N ₂
Ion Spray Voltage (IS)	5500 V
Temperature (TEM)	550 °C
Interface Heater (IHE)	ON

Chromatograms of spiked multi-residual pesticides in cucumber

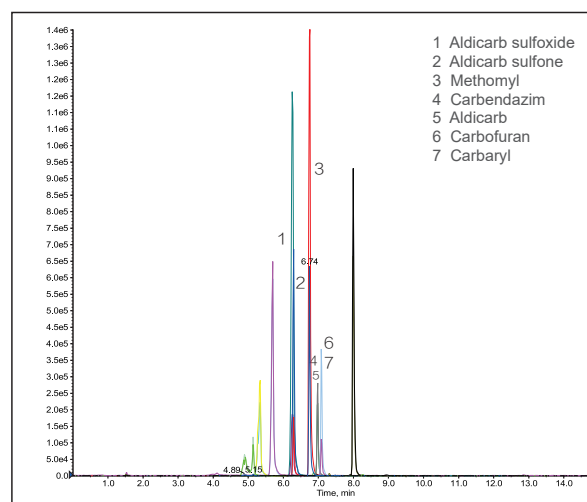


Figure 1. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in cucumber

Other conditions relating to the analytes are listed in Table 3.

Table 3. Instrument Acquisition Data for the Analysis of Carbamate Pesticides by LC/MS/MS

Compound	RT(min)	MRM channels(m/z)	DP	EP	CE	CXP
Aldicarb	7.06	208.1>89.1	30	10	22	12
		208.1>116.0	30	10	10	12
Carbofuran	7.13	222.3>123.1	48	10	16	12
		222.3>165.2	48	10	31	12
Methomyl	6.51	163.2>88.1	36	10	15	12
		163.2>106.1	36	10	12	12
Aldicarb sulfone	6.25	223.1>86.2	69	10	21	12
		223.1>148.1	69	10	13	12
Aldicarb sulfoxide	6.10	207.1>132.2	60	10	13	12
		207.1>89.1	60	10	22	12
Carbendazim	6.82	192.1>160.1	68	10	34	12
		192.1>132.2	68	10	42	12
Carbaryl	7.18	202.1>145.2	58	10	12	12
		202.1>127.1	58	10	40	12

Results

Results of spiked multi-residual pesticides in cucumber

Table 4. Recoveries and relative standard deviations (RSD) of organochlorine and pyrethroid pesticides spiked at 0.05 mg/kg in cucumber

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Aldicarb	101.6	94.2	104.2	100.0	5.19
Carbofuran	110.6	117.0	117.2	114.9	3.27
Methomyl	111.8	104.4	108.4	108.2	3.42
Carbendazim	98.0	92.0	94.6	94.9	3.17
Aldicarb sulfone	118.0	113.0	110.0	113.7	3.56
Aldicarb sulfoxide	100.8	95.2	98.6	98.2	2.87
Carbaryl Carbendazim	110.2	99.4	96.2	101.9	7.20

Order Information

Cat.#	Description	Qty.
COQ050020H	6 g MgSO ₄ , 1.5 g NaOAc, 50 mL Tube	50/Box
COQ015031H	1200 mg MgSO ₄ , 400 mg PSA, 15 mL Tube	50/Box
SF130-22-NL	NL /φ13 mm /0.22 μm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-1	2 mL Blue PP Cover with White PTFE/red Silicone Septum, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

Analysis of Pesticide Residues in Flowering Cabbage Using Copure® QuEChERS AOAC Kits by GC-ECD and LC-MS/MS

Application Scope

This method applies to analyse and validate multi-residual pesticides in pigmented fruits and vegetables.

Reference

AOAC Method 2007.01: Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate

Materials and Equipment

Copure® QuEChERS AOAC Buffered Extraction kit (Cat. No. COQ050020H)

Copure® QuEChERS AOAC Dispersive SPE kit for pigmented fruits and vegetables (Cat. No. COQ015036H) biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Homogenize a flowering cabbage sample that was frozen at -18 °C. Weigh 15.0 g of homogenized flowering cabbage sample into a 50 mL centrifuge tube, add 15 mL of 1% acetic acid in acetonitrile solution. Add an AOAC buffered extraction salt pouch containing 6 g anhydrous MgSO₄ and 1.5 g of anhydrous sodium acetate (Cat. No. COQ050020H). Vortex for 10 min, then centrifuge for 5 min at 4000 rpm. The upper acetonitrile layer is being cleaned up in the following step.

Dispersive SPE cleanup

Transfer 3 mL Toluene into a QuEChERS AOAC dispersive SPE 15 mL tube containing 1.2 g MgSO₄, 400 mg PSA and 400 mg GCB (Cat. No. COQ015036H), vortex for 30 s. And then transfer 8 mL of the upper acetonitrile layer into the QuEChERS AOAC dispersive SPE 15 mL tube (Cat. No. COQ015036H). Vortex for 1 min, then centrifuge for 5 min at 4000 rpm. Transfer 1 mL of supernatant, pass through a 0.22 µm membrane, ready for GC-ECD and LC/MS/MS analysis.

Chromatographic analysis

GC-ECD conditions

System: Agilent 7890A

Columns: Agilent J&W HP-5(30 m x 0.32 mm, 0.25 µm) or equivalent

Injection port temperature: 220 °C

Detector temperature: 300 °C

Oven temperature: 180 °C (2 min)

10 °C/min to 230 °C (2 min)

2 °C/min to 260 °C (2 min)

25 °C/min to 270 °C (1.6 min)

Carrier gas: Helium

Flow rate: 1.6 mL/min

Inlet: 220 °C, 1 µL, split 10:1

LC-MS/MS conditions

System: API 4000

Column: Venusil ASB C18 (2.1 mm x 150 mm, 5µm)

Mobile Phase: A: 0.1% HCOOH and 10 mM ammonium acetate in H₂O (Add 1 mL HCOOH and 0.77 g ammonium acetate into 1 L aqueous solution.)

B: MeOH

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	95	5
1.5	95	5
6.0	5	95
11.0	5	95
11.1	95	5
15.0	95	5

Flow rate: 0.35 mL/min

Column temperature: 40 °C

Injection volume: 5 µL

Ion source: ESI

Ionization mode: Positive

Scan mode: MRM

Ion source parameters conditions are listed in Table 2.

Table 2. Ion source parameters conditions

Collision Gas (CAD)	6 psi, N ₂
Curtain Gas (CUR)	12 psi, N ₂
Ion Source Gas 1 (GS1)	50 psi, N ₂
Ion Source Gas 2 (GS2)	50 psi, N ₂
Ion Spray Voltage (IS)	5500 V
Temperature (TEM)	550 °C
Interface Heater (IHE)	ON

Other conditions relating to the analytes are listed in Table 3.

Table 3. Instrument Acquisition Data for the Analysis of Carbamate Pesticides by LC/MS/MS

Compound RT(min)	RT(min)	MRM channels(m/z)	DP	EP	CE	CXP
Aldicarb	7.06	208.1>89.1	30	10	22	12
		208.1>116.0	30	10	10	12
Carbofuran	7.13	222.3>123.1	48	10	16	12
		222.3>165.2	48	10	31	12
Methomyl	6.51	163.2>88.1	36	10	15	12
		163.2>106.1	36	10	12	12
Aldicarb sulfone	6.25	223.1>86.2	69	10	21	12
		223.1>148.1	69	10	13	12
Aldicarb sulfoxide	6.10	207.1>132.2	60	10	13	12
		207.1>89.1	60	10	22	12
Carbaryl	7.18	202.1>145.2	58	10	12	12
		202.1>127.1	58	10	40	12

Results

Results of spiked multi-residual pesticides in flowering cabbage

Table 4. Recoveries and relative standard deviations (RSD) of organochlorine and pyrethroid pesticides spiked at 0.1 mg/kg in flowering cabbage

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Vinclozolin	101.1	89.1	90.9	93.7	6.91
Triazolone	113.0	114.5	106.2	111.2	3.98
Procymidone	87.6	84.7	82.4	84.9	3.07
Iprodione	119.8	115.1	122.9	119.3	3.29
Bifenthrin	114.9	108.6	108.3	110.6	3.37
Fenpropathrin	91.4	89.4	85.5	88.8	3.38
Beta-cyfluthrin	105.2	114.1	110.9	110.1	4.10
Cyfluthrin	108.1	104.2	101.5	104.6	3.17
Cypermethrin	77.3	78.8	70.2	75.4	6.09
Flucythrinate	93.3	82.2	84.8	86.8	6.69
Fenvalerate	107.8	100.8	104.7	104.4	3.36
Fluvalinate	82.1	80.1	87.4	83.2	4.53
Deltamethrin	113.3	108.2	105.3	108.9	3.72

Table 5. Recoveries and relative standard deviations (RSD) of carbamate pesticides spiked at 0.05 mg/kg in flowering cabbage

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Aldicarb	102.2	110.6	111.2	108.0	4.66
Carbofuran	110.4	116.4	119.8	115.5	4.12
Methomyl	104.6	108.4	110.0	107.7	2.58
Aldicarb sulfone	106.2	107.4	111.6	108.4	2.62
Aldicarb sulfoxide	88.6	81.6	87.0	85.7	4.28
Carbaryl Carbendazim	101.4	102.4	100.6	101.5	0.89

Chromatograms of spiked multi-residual pesticides in flowering cabbage

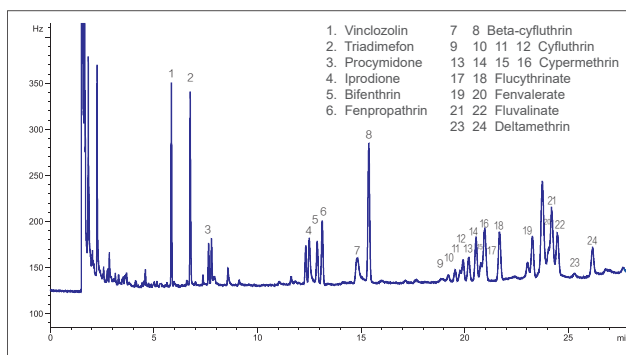


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.1 mg/kg in flowering cabbage

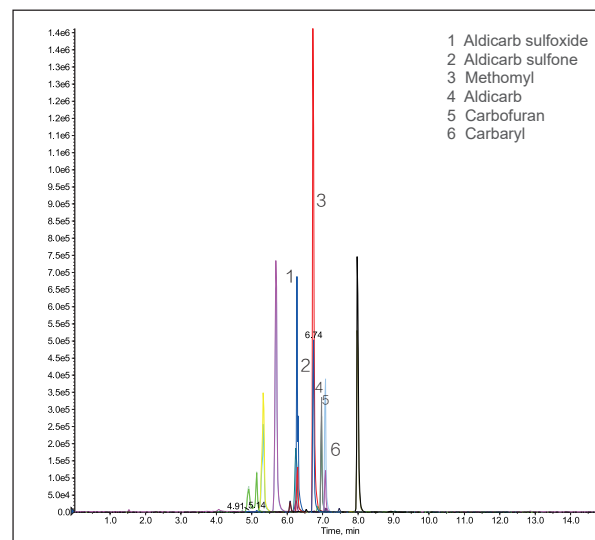


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in flowering cabbage

Order Information

Cat.#	Description	Qty.
COQ050020H	6 g MgSO ₄ , 1.5 g NaOAc, 50 mL Tube	50/Box
COQ015036H	1200 mg MgSO ₄ , 400 mg PSA, 400 mg GCB, 15 mL Tube	50/Box
SF130-22-PTFE	PTFE /φ13 mm /0.22 μm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-1	2 mL Blue PP Cover with White PTFE/red Silicone Septum, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

Analysis of Pesticide Residues in Rice Using Copure® QuEChERS AOAC Kits by GC-ECD and LC-MS/MS

Application Scope

This method applies to analyse and validate multi-residual pesticides in fruits, vegetables and cereal with fatty and waxy.

Reference

AOAC Method 2007.01: Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate

Materials and Equipment

Copure® QuEChERS AOAC Buffered Extraction kit (Cat. No. COQ050020H)

Copure® QuEChERS AOAC Dispersive SPE kit for fruits and vegetables with fatty and waxy (Cat. No. COQ015033H)

biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Homogenize a rice sample that was frozen at -18 °C. Weigh 15.0 g of homogenized rice sample into a 50 mL centrifuge tube, add 15 mL of 1% acetic acid in acetonitrile solution. Add an AOAC buffered extraction salt pouch containing 6 g anhydrous MgSO₄ and 1.5 g of anhydrous sodium acetate (Cat. No. COQ050020H). Vortex for 10 min, then centrifuge for 5 min at 4000 rpm. The upper acetonitrile layer is being cleaned up in the following step.

Dispersive SPE cleanup

Transfer 8 mL of the upper acetonitrile layer into a QuEChERS AOAC dispersive SPE 15 mL tube containing 1.2 g MgSO₄, 400 mg PSA and 400 mg C18 (Cat. No. COQ015033H). Vortex for 1 min, then centrifuge for 5 min at 4000 rpm. Transfer 1 mL of supernatant, pass through a 0.22 µm membrane, ready for GC-ECD and LC/MS/MS analysis.

Chromatographic analysis

GC-ECD conditions

System: Agilent 7890A

Columns: Agilent J&W HP-5(30 m x 0.32 mm, 0.25 µm)

or equivalent

Injection port temperature: 220 °C

Detector temperature: 300 °C

Oven temperature: 180 °C (2 min)

10 °C /min to 230 °C (2 min)

2 °C/min to 260 °C (2 min)

25 °C/min to 270 °C (1.6 min)

Carrier gas: Helium

Flow rate: 1.6 mL/min

Inlet: 220 °C, 1 µL, split 10:1

LC-MS/MS conditions

System: API 4000

Column: Venusil ASB C18 (2.1 mm x 150 mm, 5µm)

Mobile Phase: A: 0.1% HCOOH and 10 mM ammonium acetate in H₂O (Add 1 mL HCOOH and 0.77 g ammonium acetate into 1 L aqueous solution.)

B: MeOH

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	95	5
1.5	95	5
6.0	5	95
11.0	5	95
11.1	95	5
15.0	95	5

Flow rate: 0.35 mL/min

Column temperature: 40 °C

Injection volume: 5 µL

Ion source: ESI

Ionization mode: Positive

Scan mode: MRM

Ion source parameters conditions are listed in Table 2.

Table 2. Ion source parameters conditions

Collision Gas (CAD)	6 psi, N ₂
Curtain Gas (CUR)	12 psi, N ₂
Ion Source Gas 1 (GS1)	50 psi, N ₂
Ion Source Gas 2 (GS2)	50 psi, N ₂
Ion Spray Voltage (IS)	5500 V
Temperature (TEM)	550 °C
Interface Heater (IHE)	ON

Other conditions relating to the analytes are listed in Table 3.

Table 3. Instrument Acquisition Data for the Analysis of Carbamate Pesticides by LC/MS/MS

Compound RT(min)	RT(min)	MRM channels(m/z)	DP	EP	CE	CXP
Aldicarb	7.06	208.1>89.1	30	10	22	12
		208.1>116.0	30	10	10	12
Carbofuran	7.13	222.3>123.1	48	10	16	12
		222.3>165.2	48	10	31	12
Methomyl	6.51	163.2>88.1	36	10	15	12
		163.2>106.1	36	10	12	12
Aldicarb sulfone	6.25	223.1>86.2	69	10	21	12
		223.1>148.1	69	10	13	12
Aldicarb sulfoxide	6.10	207.1>132.2	60	10	13	12
		207.1>89.1	60	10	22	12
Carbendazim	6.82	192.1>160.1	68	10	34	12
		192.1>132.2	68	10	42	12
Carbaryl	7.18	202.1>145.2	58	10	12	12
		202.1>127.1	58	10	40	12

Results

Results of spiked multi-residual pesticides in rice

Table 4. Recoveries and relative standard deviations (RSD) of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in rice

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Quintozene	92.0	101.5	103.3	98.9	6.14
Vinclozolin	93.5	100.4	102.2	98.7	4.65
Triazolone	114.5	122.5	126.5	121.2	5.04
Procymidone	81.5	84.5	86.1	84.0	2.78
Iprodione	113.0	109.0	113.5	111.8	2.21
Bifenthrin	120.5	120.0	122.0	120.8	0.86
Fenpropathrin	100.5	99.3	101.5	100.4	1.10
Beta-cyfluthrin	101.5	100.6	102.5	101.5	0.94
Cyfluthrin	101.7	96.7	96.3	98.2	3.06
Cypermethrin	78.7	70.2	70.9	73.3	6.44
Flucythrinate	126.0	130.0	119.5	125.2	4.23
Fenvalerate	110.0	99.5	111.4	107.0	6.08
Fluvalinate	100.5	100.2	97.4	99.4	1.69
Deltamethrin	122.5	111.6	120.0	118.0	4.84

Table 5. Recoveries and relative standard deviations (RSD) of carbamate pesticides spiked at 0.05 mg/kg in rice

Compound	Recoveries(%)			Average Recoveries(%)	RSD(%)
	1	2	3		
Aldicarb	111.2	118.6	117.6	115.8	3.47
Carbofuran	92.6	85.2	93.4	90.4	5.00
Methomyl	90.0	101.4	98.2	96.5	6.09
Carbendazim	70.6	79.4	70.2	73.4	7.08
Aldicarb sulfone	107.2	114.0	111.6	110.9	3.11
Aldicarb sulfoxide	114.0	119.8	117.8	117.2	2.51
Carbaryl Carbendazim	92.4	98.4	92.0	94.3	3.80

Chromatograms of spiked multi-residual pesticides in rice

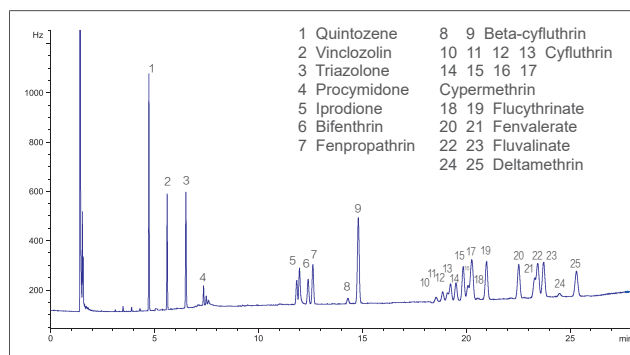


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in rice

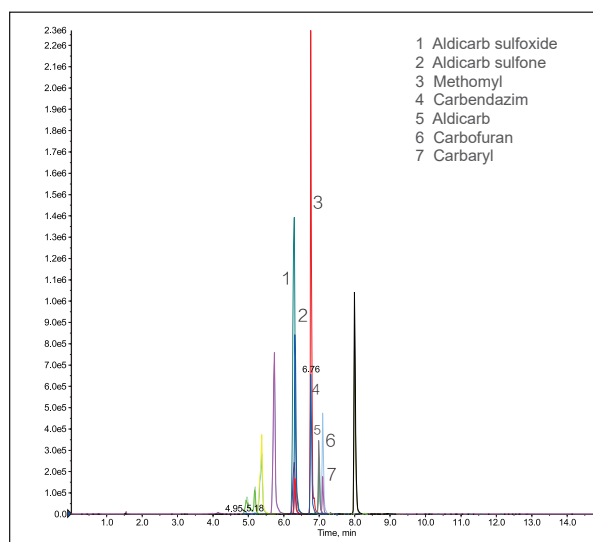


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in rice

Order Information

Cat.#	Description	Qty.
COQ050020H	6 g MgSO ₄ , 1.5 g NaOAc, 50 mL Tube	50/Box
COQ015033H	1200 mg MgSO ₄ , 400 mg PSA, 400 mg C18, 15 mL Tube	50/Box
SF130-22-PTFE	PTFE /φ13 mm /0.22 μm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-5	2 mL Blue PP Cover with White PTFE/red Silicone Septum, Pre-opening, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

Analysis of Pesticide Residues in Eggplant Using Copure® QuEChERS AOAC Kits by GC-ECD and LC-MS/MS

Application Scope

This method applies to analyse and validate multi-residual pesticides in fruits and vegetables with fats and pigment.

Reference

AOAC Method 2007.01: Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate

Materials and Equipment

Copure® QuEChERS AOAC Buffered Extraction kit (Cat. No. COQ050020H)

Copure® QuEChERS AOAC Dispersive SPE kit for fruits and vegetables with fats and pigment (Cat. No. COQ015040H)

biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Homogenize an eggplant sample that was frozen at -18 °C. Weigh 15.0 g of homogenized eggplant sample into a 50 mL centrifuge tube, add 15 mL of 1% acetic acid in acetonitrile solution. Add an AOAC buffered extraction salt pouch containing 6 g anhydrous MgSO₄ and 1.5 g of anhydrous sodium acetate (Cat. No. COQ050020H). Vortex for 10 min, then centrifuge for 5 min at 4000 rpm. The upper acetonitrile layer is being cleaned up in the following step.

Dispersive SPE cleanup

Transfer 3 mL toluene into a QuEChERS AOAC dispersive SPE 15 mL tube containing 1.2 g MgSO₄, 400 mg PSA, 400 mg C18 and 400 mg GCB (Cat. No. COQ015040H), vortex for 30 s. And then transfer 8 mL of the upper acetonitrile layer into the QuEChERS AOAC dispersive SPE 15 mL tube (Cat. No. COQ015040H). Vortex for 1 min, then centrifuge for 5 min at 4000 rpm. Transfer 1 mL of supernatant, pass through a 0.22 µm membrane, ready for GC-ECD and LC/MS/MS analysis.

Chromatographic analysis

GC-ECD conditions

System: Agilent 7890A

Columns: Agilent J&W HP-5(30 m x 0.32 mm, 0.25 µm) or equivalent

Injection port temperature: 220 °C

Detector temperature: 300 °C

Oven temperature: 180 °C (2 min)

10 °C/min to 230 °C (2 min)

2 °C/min to 260 °C (2 min)

25 °C/min to 270 °C (1.6 min)

Carrier gas: Helium

Flow rate: 1.6 mL/min

Inlet: 220 °C, 1 µL, split 10:1

LC-MS/MS conditions

System: API 4000

Column: Venusil ASB C18 (2.1 mm x 150 mm, 5µm)

Mobile Phase: A: 0.1% HCOOH and 10 mM ammonium acetate in H₂O (Add 1 mL HCOOH and 0.77 g ammonium acetate into 1 L aqueous solution.)

B: MeOH

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	95	5
1.5	95	5
6.0	5	95
11.0	5	95
11.1	95	5
15.0	95	5

Flow rate: 0.35 mL/min

Column temperature: 40 °C

Injection volume: 5 µL

Ion source: ESI

Ionization mode: Positive

Scan mode: MRM

Ion source parameters conditions are listed in Table 2.

Table 2. Ion source parameters conditions

Collision Gas (CAD)	6 psi, N ₂
Curtain Gas (CUR)	12 psi, N ₂
Ion Source Gas 1 (GS1)	50 psi, N ₂
Ion Source Gas 2 (GS2)	50 psi, N ₂
Ion Spray Voltage (IS)	5500 V
Temperature (TEM)	550 °C
Interface Heater (IHE)	ON

Other conditions relating to the analytes are listed in Table 3.

Table 3. Instrument Acquisition Data for the Analysis of Carbamate Pesticides by LC/MS/MS

Compound RT(min)	RT(min)	MRM channels(m/z)	DP	EP	CE	CXP
Aldicarb	7.06	208.1>89.1	30	10	22	12
		208.1>116.0	30	10	10	12
Carbofuran	7.13	222.3>123.1	48	10	16	12
		222.3>165.2	48	10	31	12
Methomyl	6.51	163.2>88.1	36	10	15	12
		163.2>106.1	36	10	12	12
Aldicarb sulfone	6.25	223.1>86.2	69	10	21	12
		223.1>148.1	69	10	13	12
Aldicarb sulfoxide	6.10	207.1>132.2	60	10	13	12
		207.1>89.1	60	10	22	12
Carbaryl	7.18	202.1>145.2	58	10	12	12
		202.1>127.1	58	10	40	12

Results

Results of spiked multi-residual pesticides in eggplant

Table 4. Recoveries and relative standard deviations (RSD) of organochlorine and pyrethroid pesticides spiked at 0.1 mg/kg in eggplant

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Quintozene	79.8	73.2	70.4	74.5	6.48
Chlorothalonil	80.3	77.3	75.4	77.7	3.18
Vinclozolin	103.1	94.2	93.9	97.1	5.39
Triazolone	121.9	111.3	111.3	114.8	5.33
Procymidone	113.6	102.4	100.2	105.4	6.82
lprodione	130.6	126.3	128.0	128.3	1.69
Bifenthrin	120.9	107.5	109.6	112.7	6.40
Fenpropathrin	103.9	94.4	96.2	98.2	5.14
Beta-cyfluthrin	100.7	91.6	93.9	95.4	4.96
Cyfluthrin	96.1	87.5	85.6	89.7	6.24
Cypermethrin	80.6	75.8	73.3	76.6	4.85
Flucythrinate	102.4	104.7	112.4	106.5	4.92
Fenvalerate	97.2	87.1	85.0	89.8	7.27

Table 5. Recoveries and relative standard deviations (RSD) of carbamate pesticides spiked at 0.05 mg/kg in eggplant

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Aldicarb	87.2	84.4	93.8	88.5	5.46
Carbofuran	77.6	73.6	72.4	74.5	3.65
Methomyl	75.8	85.6	84.2	81.9	6.47
Aldicarb sulfone	92.2	101.0	104.0	99.1	6.19
Aldicarb sulfoxide	92.0	92.2	88.8	91.0	2.10
Carbaryl Carbendazim	77.0	72.1	73.4	74.2	3.42

Chromatograms of spiked multi-residual pesticides in eggplant

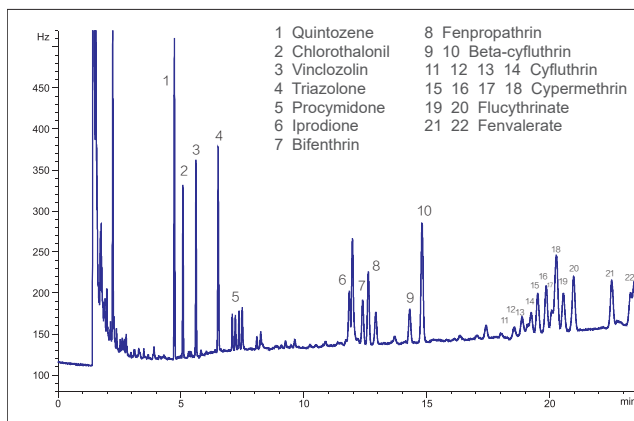


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.1 mg/kg in eggplant

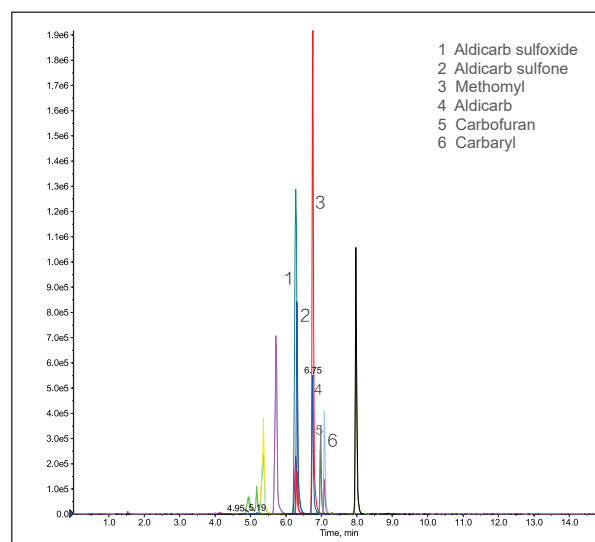


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in eggplant

Order Information

Cat.#	Description	Qty.
COQ050020H	6 g MgSO ₄ , 1.5 g NaOAc, 50 mL Tube	50/Box
COQ015040H	1200 mg MgSO ₄ , 400 mg PSA, 400 mg C18, 400 mg GCB, 15 mL Tube	50/Box
SF130-22-PTFE	PTFE /φ13 mm /0.22 μm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-5	2 mL Blue PP Cover with White PTFE/red Silicone Septum, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

Analysis of Steroids in Pork Using Copure® QuEChERS Kits by HPLC

Application Scope

This method applies to analyse and validate multi-residual steroids in pork.

Materials and Equipment

Copure® QuEChERS extraction kit for veterinary drugs (Cat. NO. COQ050050)

Copure® QuEChERS dispersive SPE kit for veterinary drugs (Cat. NO. COQ015601)

biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Weigh 5.0 g of homogenized pork sample into a 50 mL centrifuge tube, add 10 mL of acetonitrile, and vortex for 1 min. Add a salt packet for veterinary drugs (Cat. No. COQ050050). Vortex for 10 min, and centrifuge for 5 min at 4000 rpm. The upper acetonitrile layer is being cleaned up by the following step.

Dispersive SPE cleanup

Transfer 6 mL of the upper acetonitrile layer into a QuEChERS dispersive SPE 15 mL tube (Cat. No. COQ015601), vortex for 1 min and centrifuge for 5 min at 4000 rpm. Transfer 4 mL of supernatant, and dry by nitrogen at 40 °C. Reconstitute with 1 mL mobile phase solution, and pass through a 0.22 µm membrane. Be ready for HPLC analysis.

Chromatographic analysis

HPLC Conditions

System: Waters Alliance 2695

Column: Phenomenex kinetex®-C18 (250 mm x 4.6 mm, 5µm)

Detector: Waters 2996 DAD

Wave Length: 230 nm

Mobile Phase: A: Water B: Acetonitrile

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	50	50
4.0	50	50
13.0	0	100
15.0	0	100
19.0	50	50
24.0	50	50

Flow rate: 1 mL/min

Injection volume: 20 µL

Results

The results of spike steroids in pork are listed in Table 2.

Table 2. Recoveries of steroids spiked at 0.5 mg/kg in pork

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Medroxyprogesterone	95.0	92.3	93.3	93.5	1.46
Nandrolone	94.8	91.2	87.8	91.2	3.84

Chromatograms of spiked steroids in pork

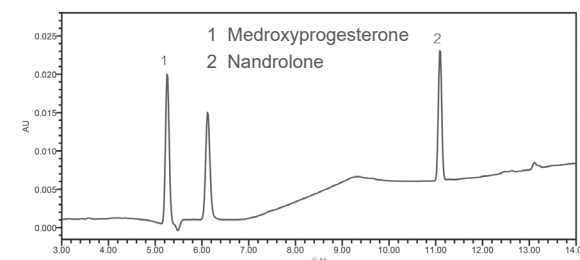


Figure 1. Chromatogram of steroids spiked at 0.5 mg/kg in pork

Order Information

Cat.#	Description	Qty.
COQ050050	Extraction Kit for Veterinary Drugs, 50 mL Tube	50/Box
COQ015601	Dispersive SPE Kit for Veterinary Drugs, 15 mL Tube	50/Box
SF130-22-PTFE	PTFE /φ13 mm /0.22 µm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-5	2 mL Blue PP Cover with White PTFE/red Silicone Septum, Pre-opening, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

Analysis of Quinolones in Pork Using Copure® QuEChERS Kits by HPLC

Application Scope

This method applies to analyse and validate quinolones in general meat.

Materials and Equipment

Copure® QuEChERS extraction kit for veterinary drugs (Cat. NO. COQ050051)

Copure® QuEChERS dispersive SPE kit for veterinary drugs (Cat. NO. COQ015601)

biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Weigh 2.0 g of homogenized pork sample into a 50 mL centrifuge tube. Add a salt packet for veterinary drugs (Cat.No.COQ050051) and 10 mL 1% acetic acid in acetonitrile solution. Vortex for 10 min, and centrifuge for 5 min at 4000 rpm. The upper acetonitrile layer is being cleaned up by the following step.

Dispersive SPE cleanup

Transfer 6 mL of the upper acetonitrile layer into a QuEChERS dispersive SPE 15 mL tube (Cat. No.COQ015601), vortex for 1 min and centrifuge for 5 min at 5000 rpm. Transfer 4 mL of supernatant, and dry by nitrogen at 40 °C. Reconstitute with 1 mL 50% Methanol-Aqueous solution, and pass through a 0.22 µm membrane. Be ready for HPLC analysis.

Chromatographic analysis

HPLC Conditions

System: Waters Alliance 2695

Column: Phenomenex kinetex®-C18 (250 mm x 4.6 mm, 5µm)

Detector: Waters 2996 DAD

Wave Length: 254 nm

Mobile Phase: A: 0.1% formic acid solution B: Acetonitrile

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	90	10
3.0	90	10
8.0	65	35
11.0	35	65
12.0	90	10
17.0	90	10

Flow rate: 1 mL/min

Injection volume: 20 µL

Results

The results of spike quinolones in pork are listed in Table 2

Table 2. Recoveries of quinolones spiked at 0.8 mg/kg in pork

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Marbofloxacin	88.0	81.0	81.6	83.5	4.6
Pefloxacin	93.4	87.7	86.4	89.2	4.2
Danofloxacin	99.1	91.7	91.9	94.2	4.5
Enrofloxacin	104.1	92.1	101.3	99.2	6.3
Difloxacin	102.4	103.5	100.0	102.0	1.8

Chromatograms of spiked quinolones in pork

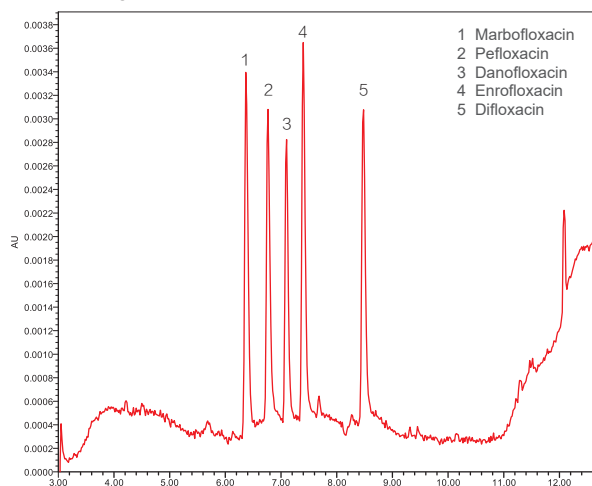


Figure 1. Chromatogram of quinolones spiked at 0.8 mg/kg in pork

Order Information

Cat.#	Description	Qty.
COQ050051	Extraction Kit for Veterinary Drugs, 50 mL Tube	50/Box
COQ015601	Dispersive SPE Kit for Veterinary Drugs, 15 mL Tube	50/Box
SF130-22-PTFE	PTFE /φ13 mm /0.22 µm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-5	2 mL Blue PP Cover with White PTFE/red Silicone Septum, Pre-opening, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

Analysis of Chloramphenicol analogue in Pork Using Copure® QuEChERS Kits by HPLC

Application Scope

This method applies to analyse and validate chloramphenicol analogues in pork.

Materials and Equipment

Copure® QuEChERS extraction kit for veterinary drugs (Cat. NO. COQ050050)

Copure® QuEChERS dispersive SPE kit for veterinary drugs (Cat. NO. COQ015601)

biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Weigh 2.0 g of homogenized meat sample into a 50 mL extraction tube, add 4 mL water, vortex for 1min, add 10 mL of 1% acetic acid in acetonitrile solution, then add a QuEChERS salt pouch (Cat.No.COQ050050). Vortex for 10 min, and centrifuge for 5 min at 5000 r/min. The upper layer acetonitrile is being cleaned up for next step.

Dispersive SPE cleanup

Transfer 6 mL upper layer acetonitrile into 15 mL a QuEChERS dispersive SPE 15 mL tube (Cat.No. COQ015601), vortex for 1 min, centrifuge for 5 min at 5000 r/min. Transfer 4 mL supernatant into another tube, dry at 40 °C under nitrogen, redissolve with 1 mL methanol, then filter over 0.22 µm microporous membrane for HPLC analysis.

Chromatographic analysis

HPLC Conditions

System: Waters Alliance 2695

Column: Phenomenex kinetex®-C18 (250 mm x 4.6 mm, 5 µm)

Detector: Waters 2996 DAD

Wave Length: 268 nm

Mobile Phase: A: 0.1% formic acid solution B: Acetonitrile

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	90	10
6.0	90	10
29.0	70	30
30.0	0	100
36.0	0	100
37.0	90	10
42.0	90	10

Flow rate: 1 mL/min

Injection volume: 20 µL

Results

The results of spike chloramphenicol analogue in pork are listed in Table 2.

Table 2. Recoveries and relative standard deviations (RSD) of chloramphenicol analogues spiked at 5.0 mg/kg in pork

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Chloramphenicol	78.4	80.5	81.6	80.2	2.0
Florfenicol	87.1	82.0	86.7	85.3	3.3

Chromatograms of spiked chloramphenicol analogues in pork

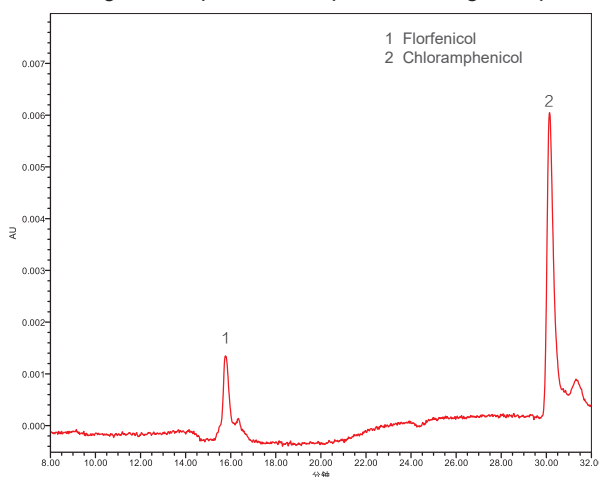


Figure 1. Chromatogram of chloramphenicol analogue spiked at 5.0 mg/kg in pork

Order Information

Cat.#	Description	Qty.
COQ050050	Extraction Kit for Veterinary Drugs, 50 mL Tube	50/Box
COQ015601	Dispersive SPE Kit for Veterinary Drugs, 15 mL Tube	50/Box
SF130-22-NL	NL /φ13 mm /0.22 µm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-5	2 mL Blue PP Cover with White PTFE/red Silicone Septum, Pre-opening, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box

Analysis of Tetracyclines in Pork Using Copure® QuEChERS Kits by HPLC

Application Scope

This method applies to analyse and validate multi-residual tetracyclines and their metabolites in pork.

Materials and Equipment

Copure® QuEChERS extraction kit for veterinary drugs (Cat. NO. COQ050051)

Copure® QuEChERS dispersive SPE kit for veterinary drugs (Cat. NO. COQ015601)

biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

Weigh 2.0 g of homogenized meat sample into a 50 mL extraction tube, add a QuEChERS salt pouch (Cat.No.COQ050051), add 10 mL of 1% acetic acid in acetonitrile solution. Vortex for 10 min, centrifuge for 5 min at 5000 r/min. The upper acetonitrile layer is being cleaned up for next step.

Dispersive SPE cleanup

Transfer 6 mL upper acetonitrile layer into a QuEChERS dispersive SPE 15 mL tube (Cat.No. COQ015601), vortex for 1 min, centrifuge for 5 min at 5000 r/min. Transfer 4 mL supernatant into another tube, dry at 40 °C under nitrogen, redissolve with 1 mL TFA-methanol solution (1:19, v/v), then filter over 0.22 µm microporous membrane for HPLC analysis.

Chromatographic analysis

HPLC Conditions

System: Waters Alliance 2695

Column: Phenomenex kinetex®-C18 (250 mm x 4.6 mm, 5µm)

Detector: Waters 2996 DAD

Wave Length: 350 nm

Mobile Phase: A: 10mM TFA solution B: Acetonitrile

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time/min	A(%)	B(%)
--	96	4
8.0	70	30
18.0	65	35
20.0	96	4
28.0	96	4

Flow rate: 1 mL/min

Injection volume: 20 µL

Results

The results of spike tetracyclines in pork are listed in Table 2.

Table 2. Recoveries and relative standard deviations (RSD) of tetracyclines spiked at 1.0 mg/kg in pork.

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Oxytetracycline	80.9	86.3	81.6	81.6	3.6
Tetracycline	76.0	76.7	75.4	75.4	0.9
Chlortetracycline	89.3	84.4	84.8	84.8	3.2
Doxycycline	86.9	86.9	85.9	85.9	0.7

Chromatograms of spiked tetracyclines in pork

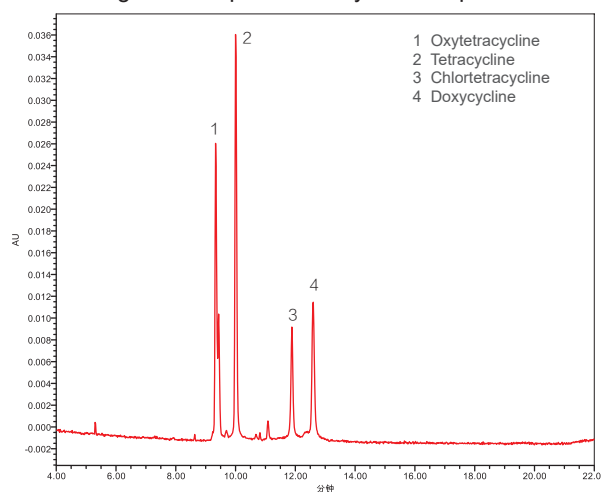


Figure 1. Chromatograms of tetracyclines spiked at 1.0 mg/kg in pork

Order Information

Cat.#	Description	Qty.
COQ050051	Extraction Kit for Veterinary Drugs, 50 mL Tube	50/Box
COQ015601	Dispersive SPE Kit for Veterinary Drugs, 15 mL Tube	50/Box
SF130-22-PTFE	PTFE /φ13 mm /0.22 µm /Hydrophobic	100/Box
V2-AL	2 mL Amber Screw-thread Vials, 9-425	100/Box
SC2-1	2 mL Blue PP Cover with White PTFE/red Silicone Septum, 9-425	100/Box
BC-1000	biocomma® multi-tube Vortexer	1 set/Box



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- Convenient and easy to use.
- Eliminates the need for a second tube to transfer salts.

Supports AOAC (2007.01) & European (EN15662) QuEChERS Methods

Buy 1, get 1 FREE
offer applies to
the following
products:

Description	Material	Methods	qty.	cat#
Q-sep Q110 kit	4g MgSO ₄ , 1g NaCl, 1g TSCD, 0.5g DHS with 50mL Centrifuge Tube	European EN 15662	50 packets & 50 tubes	26235-408
Q-sep Q110 packets	4g MgSO ₄ , 1g NaCl, 1g TSCD, 0.5g DHS	European EN 15662	50 packets	26236-408
Q-sep Q150 kit	6g MgSO ₄ , 1.5g NaOAc, 1g TSCD, 0.5g DHS with 50mL Centrifuge Tube	AOAC 2007.01	50 packets & 50 tubes	26237-408
Q-sep Q150 packets	6g MgSO ₄ , 1.5g NaOAc	AOAC 2007.01	50 packets	26238-408

Offer valid July 1—October 31, 2010.

TSCD = trisodium citrate dihydrate
DHS = disodium hydrogen citrate sesquihydrate
NaOAc = sodium acetate

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QuEChERS Products

Fast, Simple Sample Prep for Multi-Residue Pesticide Analysis



RESTEK EXCLUSIVE:

Save time with new standards—
ready to use, no dilution necessary!

- Speed up sample throughput—4-fold faster than modified Luke methods.
- Reduce solvent usage up to 9-fold, with no chlorinated waste.
- Simultaneously generate samples for GC/MS and LC/MS/MS.

Save Time and Money with QuEChERS

- Ready-to-use extraction and dSPE tubes, no glassware required.
- Preweighed adsorbents for dSPE cleanup.
- Convenient, method-specific internal and QC standards.

Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS (“catchers”) method is a fast, simple, and effective alternative to conventional sample prep for multiresidue pesticide analysis. QuEChERS is based on work done by the US Department of Agriculture Eastern Regional Research Center in Wyndmoor, PA.¹ Researchers there were looking for a simple, effective, and inexpensive way to extract and clean pesticide residues from the many varied sample matrices that they worked with routinely. They had been using the modified Luke extraction method, which is highly effective and rugged, but is solvent, labor, and glassware intensive, leading to a relatively high cost per sample. In contrast, QuEChERS employs a very short shake-extraction step, making it faster and less labor intensive. Solid phase extraction cleanup of extracts from other methods also had been effective, but the complex matrices the investigators were dealing with required multiple individual cartridges to remove the many classes of interferences, which added significant cost and complexity to the process. To reduce costs and speed up sample preparation, they developed a novel dispersive solid phase extraction (dSPE) technique, which effectively removes sugars, lipids, organic acids, sterols, proteins, pigments and excess water, but is far simpler and less expensive than conventional methods (Table I).



Using QuEChERS, samples are prepared in 3 simple steps. As shown on the following page, samples are first homogenized, then extracted and partitioned with an organic solvent and salt solution, with the extracts finally cleaned using the dSPE technique. Using the dSPE approach, the quantity and type of sorbents, can easily be optimized for different matrix interferences and difficult analytes. Results from this approach have been verified and modified at several USDA and Food and Drug Administration labs, and the method now is widely accepted for many types of pesticide residue samples. Validation and proficiency data for the QuEChERS method are available for a wide variety of pesticides in several common food matrices at www.quechers.com.

Restek Q-sep™ products make QuEChERS even simpler. All extraction salts, adsorbents, and sample tubes are included—no specialized equipment or glassware is required. The dSPE centrifuge tube format, available in 2mL and 15mL sizes, contains magnesium sulfate (to partition water from organic solvent) and PSA adsorbent (to remove sugars and fatty acids), with or without graphitized carbon (to remove pigments and sterols) or C18 (to remove nonpolar interferences). Custom products are available by request. If you are frustrated with the time and expense of your current pesticide sample cleanup procedure, we suggest you try this simple, economical new method.

Table I Prepare samples more quickly, easily, and cost-effectively with QuEChERS.

	Mini-Luke or Modified Luke Method	QuEChERS	Savings with QuEChERS
Estimated time to process 6 samples (min.)	120	30	4x faster
Solvent used (mL)	60-90	10	6-9x less solvent
Chlorinated waste (mL)	20-30	0	Safer, cheaper, greener
Glassware/specialized equipment	capacity for 200mL, quartz wool, funnel, water bath or evaporator	none	Ready-to-use



Try QuEChERS risk-free today!

Call 800-356-1688 to request a free sample pack of Q-sep™ QuEChERS tubes.



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Quick and Easy...

Prepare Samples for LC or GC Analysis in 3 Simple Steps

1. Blend

Homogenize the sample.



2. Extract and Dry

Add acetonitrile and internal standard, then shake vigorously for 1 minute.



Add buffering salts and shake, then centrifuge for 5 minutes to separate the phases.



3. Clean Up

Transfer supernatant to dSPE tube.



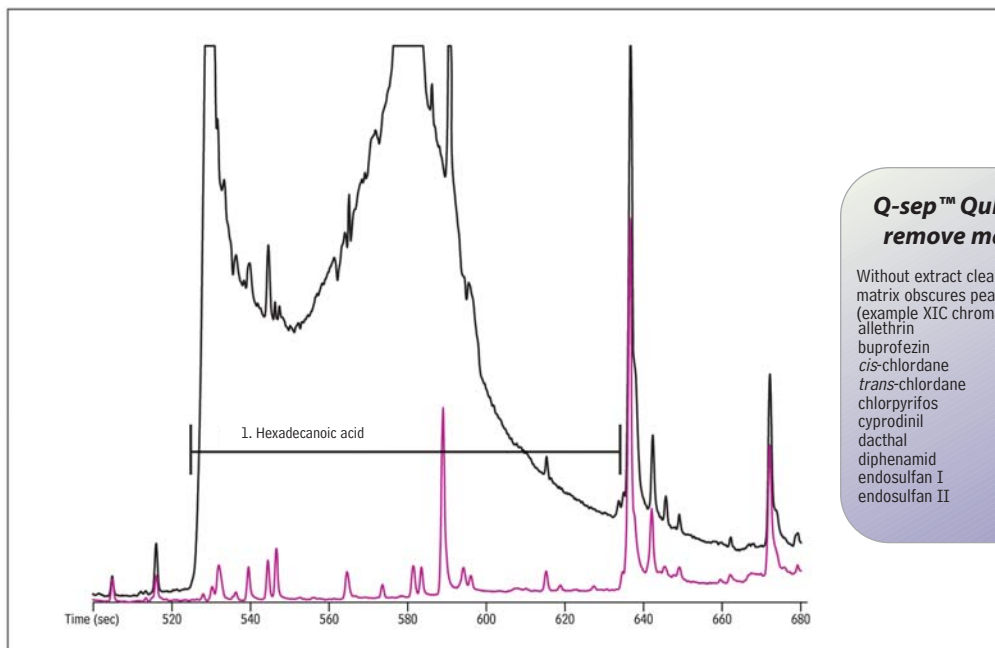
Shake, centrifuge, and transfer to an autosampler vial for analysis by GC or LC.



QuEChERS dSPE Cleanup Assures Optimal Results for Pesticide Analysis

- Improves integration and mass spectral matches.
- Removes matrix interferences that obscure target analytes or cause ion suppression.
- Protects GC inlet, and LC and GC columns from contamination.

Figure 1 QuEChERS dSPE cleanup removes interferences that obscure target pesticides.



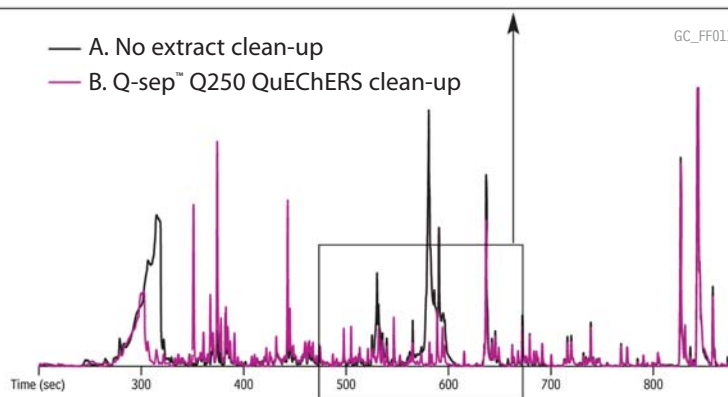
Q-sep™ QuEChERS tubes easily remove matrix interferences.

Without extract clean-up, hexadecanoic acid from the matrix obscures peaks for all the following pesticides. (example XIC chromatogram = endosulfan I)

allethrin	fenthion
buprofezin	metolachlor
cis-chlordane	myclobutanil
trans-chlordane	oxyfluorfen
chlorpyrifos	pendimethalin
cyprodinil	pentachlorothioanisole
dacthal	pirimiphos methyl
diphenamid	triadimefon
endosulfan I	triadimenol
endosulfan II	

— A. No extract clean-up
— B. Q-sep™ Q250 QuEChERS clean-up

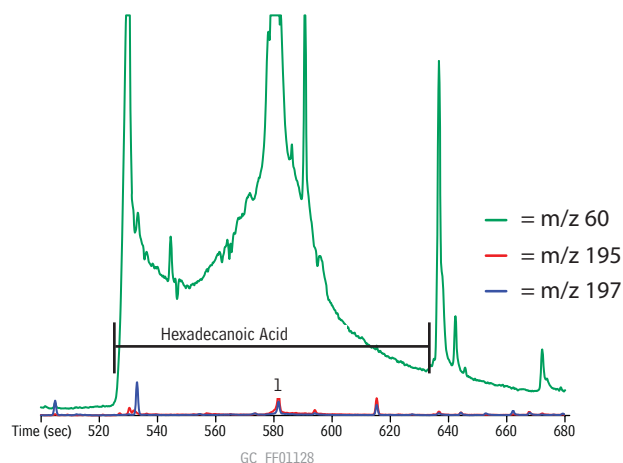
GC_FF01125-27



Column: Rxi®-5Sil MS, 20m, 0.18mm ID, 0.18µm (cat.# 43602)
Sample: sweet potato spiked with pesticide mix, extracted with acetonitrile and Q-sep™ Q110 QuEChERS extraction tube (cat.# 26213)
Inj.: A. extract (without clean-up step) acidified with formic acid to pH 5
Inj. temp.: B. extract with clean-up using Q-sep™ Q250 QuEChERS dSPE clean-up tube (cat.# 26124), acidified with formic acid to pH 5
Carrier gas: 1.0µL splitless (hold 1 min.), 4mm single gooseneck liner with w/wool (cat.# 22405)
Flow rate: 250°C
Oven temp.: helium, constant flow
Det: 1.2mL/min.
Scan range: 72.5°C (hold 1 min.) to 350°C @ 20°C/min.
Ionization: tof MS
Mode: Transfer line temp.: 300°C
Instrument: 45-550amu, m/z 60, 73, 87, 129, 256 plotted
EI
tof
Agilent 6890, LECO Pegasus III

Figure 2 QuEChERS dSPE cleanup significantly improves quantification and identification.

Without cleanup, matrix masks Endosulfan I.

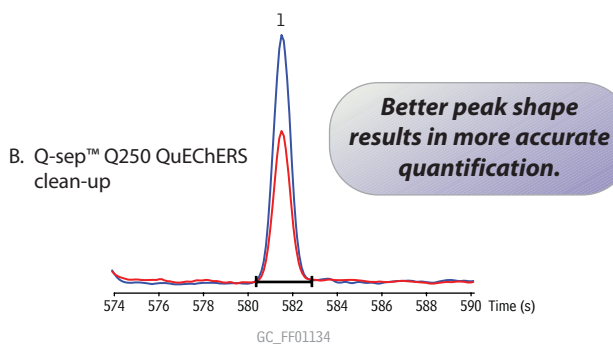
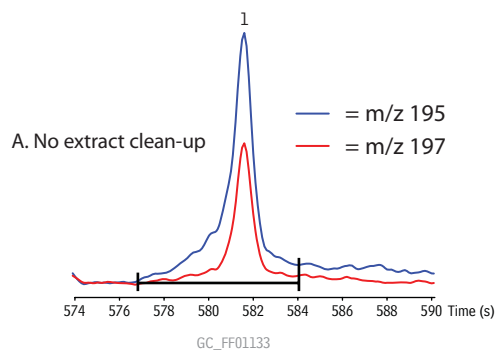


Peak List
1. hexadecanoic acid
2. endosulfan I

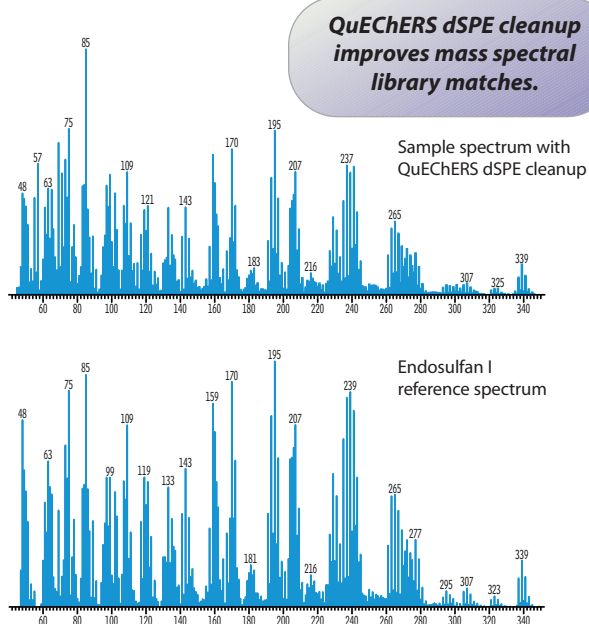
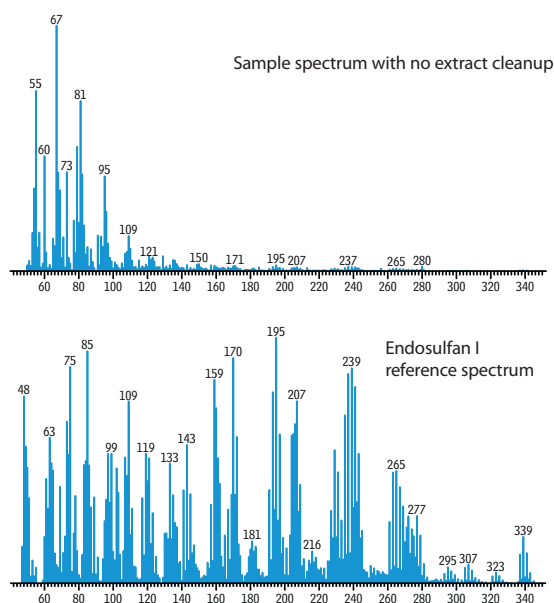
Column: Rxi®-5Sil MS, 20m, 0.18mm ID, 0.18µm (cat.# 43602)
Sample: sweet potato spiked with pesticide mix, extracted with acetonitrile and Q-sep™ Q110 QuEChERS extraction tube (cat.# 26213), then acidified with formic acid to pH 5
Inj.: 1.0µL splitless (hold 1 min.), 4mm single gooseneck liner with w/wool (cat.# 22405)
Inj. temp.: 250°C
Carrier gas: helium, constant flow
Flow rate: 1.2mL/min.
Oven temp.: 72.5°C (hold 1 min.) to 350°C @ 20°C/min.
Det: tof MS
Transfer line temp.: 225°C
Scan range: 45-550amu, m/z 60, 195, 197 plotted
Ionization: EI
Mode: tof
Instrument: Agilent 6890, LECO Pegasus III

QuEChERS dSPE cleanup improves quantification and identification.

Peak Integration (extracted ion chromatograms)



Spectral Identification



Optimize Analysis with Sorbent Choice

Choosing a QuEChERS dSPE Sorbent

Primary and secondary amine exchange material (PSA) is the base sorbent used for QuEChERS dSPE cleanup of fruit and vegetable extracts because it removes many organic acids and sugars that might act as instrumental interferences. In addition, C18 or graphitized carbon black (GCB) may be used to remove lipids or pigments, respectively. Choice of sorbent should be based on matrix composition and target analyte chemistry. Most methods make specific recommendations for acidic, basic, and planar pesticides, which may require additional considerations.

As seen in Table II, GCB can have a negative effect on the recoveries of certain pesticides that can assume planar shapes (e.g. chlorothalonil and thiabendazole). The work shown here was done with 50mg GCB per mL extract, which emphasizes this effect. The EN 15662 QuEChERS method recommends less GCB, which improves recoveries of planar pesticides, but still assures the removal of pigments that can degrade GC/MS performance. To simplify and speed up sample prep, Restek QuEChERS tubes are available in the sorbent combinations and amounts specified by EN 15662 and the AOAC methods.

Table II Select sorbents based on matrix and target analyte chemistry. (Percent recovery using C18 or GCB, relative to PSA alone).

Rt (min.)	pesticide	CAS Number	action/use	classification	C18*	GCB**
9.50	dichlorvos	62-73-7	insecticide	organophosphorus	111	116
9.67	methamidophos	10265-92-6	insecticide	organophosphorus	105	107
11.75	mevinphos	7786-34-7	insecticide	organophosphorus	112	130
12.02	o-phenylphenol	90-43-7	fungicide	unclassified	106	97
12.14	acephate	30560-19-1	insecticide	organophosphorus	128	147
13.89	omethoate	1113-02-6	insecticide	organophosphorus	120	119
14.74	diazinon	333-41-5	insecticide	organophosphorus	108	127
14.98	dimethoate	60-51-5	insecticide	organophosphorus	124	151
15.69	chlorothalonil	1897-45-6	fungicide	organochlorine	125	13
15.86	vinclozolin	50471-44-8	fungicide	organochlorine	102	98
16.21	metalaxyl	57837-19-1	fungicide	organonitrogen	105	117
16.28	carbaryl	63-25-2	insecticide	carbamate	114	111
16.60	malathion	121-75-5	insecticide	organophosphorus	124	160
16.67	dichlofluanid	1085-98-9	fungicide	organohalogen	122	103
17.51	thiabendazole	148-79-8	fungicide	organonitrogen	88	14
17.70	captan	133-06-2	fungicide	organochlorine	88	91
17.76	folpet	133-07-3	fungicide	organochlorine	108	63
18.23	imazalil	35554-44-0	fungicide	organonitrogen	115	95
18.39	endrin	72-20-8	insecticide	organochlorine	104	101
18.62	myclobutanil	88671-89-0	fungicide	organonitrogen	119	114
19.07	4,4-DDT	50-29-3	insecticide	organochlorine	102	95
19.22	fenhexamid	126833-17-8	fungicide	organochlorine	118	77
19.40	propargite 1	2312-35-8	acaricide	organosulfur	110	95
19.43	propargite 2	2312-35-8	acaricide	organosulfur	121	114
19.75	bifenthrin	82657-04-3	insecticide	pyrethroid	106	81
20.04	dicofol	115-32-2	acaricide	organochlorine	98	54
20.05	iprodione	36734-19-7	fungicide	organonitrogen	118	90
20.21	fenpropathrin	39515-41-8	insecticide	pyrethroid	113	96
21.32	cis-permethrin	52645-53-1	insecticide	pyrethroid	106	65
21.47	trans-permethrin	51877-74-8	insecticide	pyrethroid	109	71
23.74	deltamethrin	52918-63-5	insecticide	pyrethroid	97	52

*50mg PSA, 50mg C18, **50mg PSA, 50mg GCB % recovery = $\frac{\text{RRF C18 or GCB}}{\text{RRF PSA}} \times 100$

Strawberry extracts were spiked at 200ng/mL with pesticides and subjected to dSPE with PSA only. Results were used to generate single point calibration curves. Spiked extracts were then subjected to additional dSPE sorbents (either C18 or GCB). Results are shown as percent recoveries relative to PSA alone.

Sorbent Guide

Sorbent Removes

PSA* sugars, fatty acids, organic acids, anthocyanine pigments
 C18 lipids, nonpolar interferences
 GCB** pigments, sterols, nonpolar interferences

*PSA—primary and secondary amine exchange material

**GCB—graphitized carbon black



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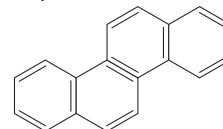
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Example dSPE Cleanup: PAHs in Infant Formula

Analyzing polycyclic aromatic hydrocarbons (PAHs) in infant formula can be difficult as both the target analytes and certain matrix elements are lipophilic in nature and difficult to separate. Proper sorbent choice is critical to removing matrix interferences, while assuring good PAH recoveries. When choosing a sorbent, target analyte and matrix component chemistry must be considered. PAHs are relatively non-polar, planar compounds with no pH-dependent functional groups. Infant formula typically contains significant amount of sugars and can be fortified with fatty acids.

Here, PSA was chosen for dSPE cleanup since both sugars and fatty acids can be removed through hydrogen bonding. Using PSA to remove these matrix compounds is optimal, because it will not bind to the relatively nonpolar PAHs, thus ensuring they remain available for analysis. C18 should not be used here because lipophilic PAHs could also be removed. Similarly, GCB is not recommended, because it also can bind planar PAHs. (Note: GCB is not needed since infant formula does not contain pigments.) Based on the chemical structure of the analytes of interest, as well as the most dominant matrix compounds, PSA is the best choice when analyzing PAHs in infant formula.

Chrysene



Phenanthrene

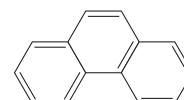
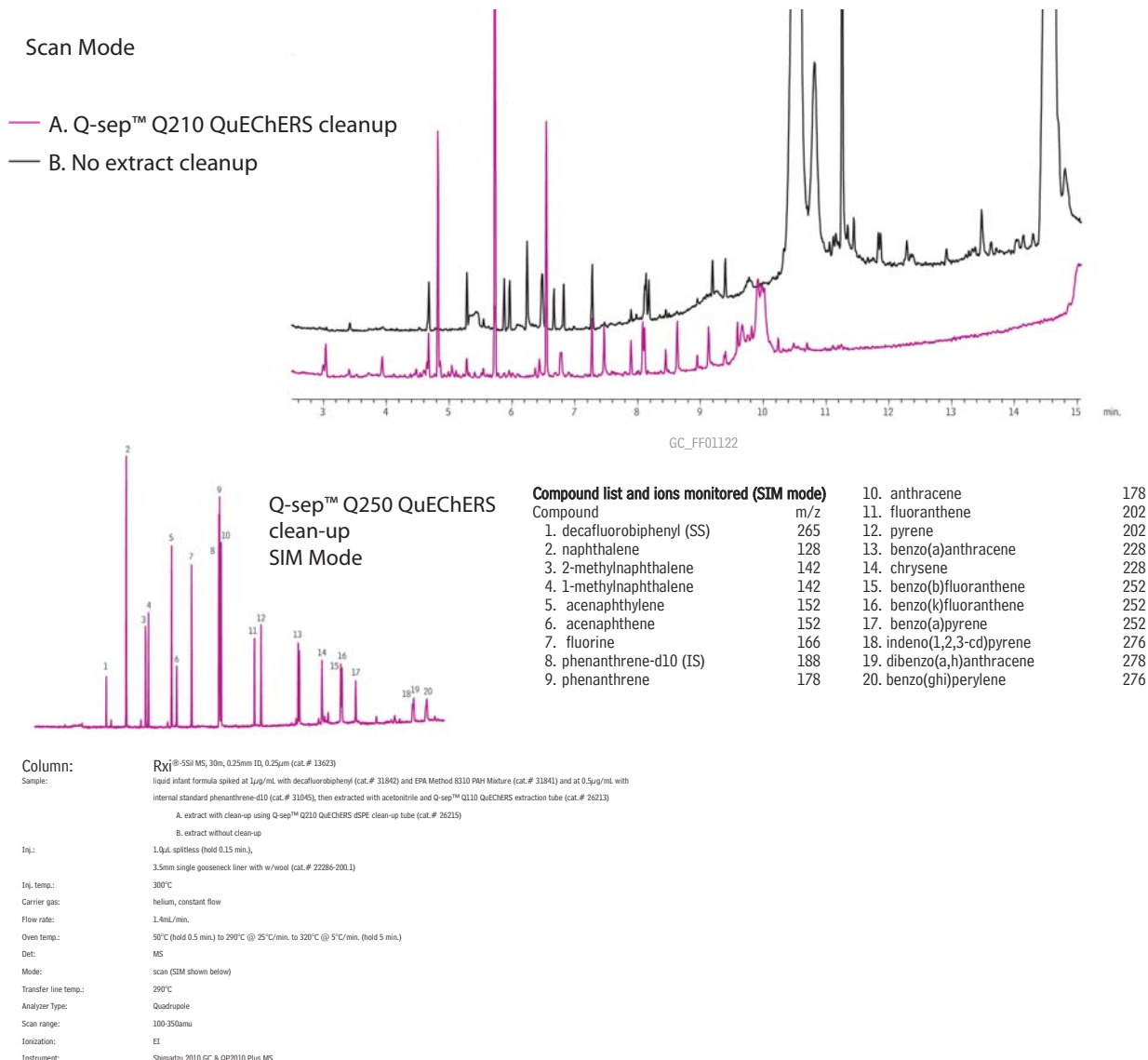


Figure 3 PSA is ideal for removing matrix sugars and fatty acids while leaving PAHs behind for analysis.



QuEChERS Methods for Complex and Varied Matrices

QuEChERS has been successfully applied to many different types of matrices. When developing procedures for your lab, start with these selected references—or visit www.restek.com/quechers for an expanded version that includes hyperlinks. (Note: references not available from Restek.)

General/Original

1. Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce. (M. Anastassiades, S.J. Lehotay, D. Stajnbaher, F.J. Schenck, J. AOAC International 86 (2003) 412.)
2. QuEChERS—A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products. (<http://www.quechers.com> (accessed July 15, 2008).)
3. Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate. (AOAC Official Method 2007.01.)
4. Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and Clean-up by Dispersive SPE (QuEChERS-method). (EN 15662 Version 2008.)
5. Matrix Effects in Pesticide Multi-Residue Analysis by Liquid Chromatography-Mass Spectrometry. (A. Kruve, A. Künnapas, K. Herodes, I. Leito, J. Chromatogr. A 1187 (2008) 58.)
6. Use of Automated Direct Sample Introduction with Analyte Protectants in the GC-MS Analysis of Pesticide Residues. (T. Cajka, K. Mastovská, S.J. Lehotay, J. Hajslová, J. Sep. Sci. 28 (2005) 1048.)

General Fruits and Vegetables

7. Validation of a Fast and Easy Method for the Determination of Residues from 229 Pesticides in Fruits and Vegetables Using Gas and Liquid Chromatography and Mass Spectrometric Detection. (S.J. Lehotay, A. de Kok, M. Hiemstra, P. Van Bodegraven, J. AOAC Int. 88 (2005) 595.)
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24. Determination of Priority Pesticides in Baby Foods by Gas Chromatography Tandem Quadrupole Mass Spectrometry. (C.C. Leandro, R.J. Fussell, B.J. Keely, J. Chromatogr. A 1085 (2005) 207.)

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- Preweighed, ultra-pure sorbents.
- Convenient, method-specific standards.

QuEChERS methods are fast, easy, and cost-effective, and Restek Q-sep™ products make QuEChERS procedures even simpler. All sorbents and sample tubes are included—no specialized equipment or glassware is required. Prepare samples more efficiently with a complete line of QuEChERS supplies from Restek.



Visit www.restek.com/quechers for new products & detailed technical information.

Table I Prepare samples more quickly, easily, and cost-effectively with QuEChERS.

	Mini-Luke or Modified Luke Method	QuEChERS	Savings with QuEChERS
Estimated time to process 6 samples (min.)	120	30	4x faster
Solvent used (mL)	60-90	10	6-9x less solvent
Chlorinated waste (mL)	20-30	0	Safer, cheaper, greener
Glassware/specialized equipment	capacity for 200mL, quartz wool, funnel, water bath or evaporator	none	Ready-to-use

Q-sep™ QuEChERS Sample Prep Packets & Tubes



Description	Material	Methods	qty.	cat#
Extraction Salt Packets and 50mL Centrifuge Tubes				
Q110 kit	4g MgSO ₄ , 1g NaCl, 1g TSCD, 0.5g DHS with 50mL Centrifuge Tube	European EN 15662	50 packets & 50 tubes	26235
Q110 packets	4g MgSO ₄ , 1g NaCl, 1g TSCD, 0.5g DHS	European EN 15662	50 packets	26236
Q150 kit	6g MgSO ₄ , 1.5g NaOAc, with 50mL Centrifuge Tube	AOAC 2007.01	50 packets & 50 tubes	26237
Q150 packets	6g MgSO ₄ , 1.5g NaOAc	AOAC 2007.01	50 packets	26238
Empty 50mL Centrifuge Tube			50-pk.	26239

Sorbent Guide

Sorbent	Removes
PSA*	sugars, fatty acids, organic acids, anthocyanine pigments
C18	lipids, nonpolar interferences
GCB**	pigments, sterols, nonpolar interferences

*PSA—primary and secondary amine exchange material
**GCB—graphitized carbon black

2mL Micro-Centrifuge Tubes for dSPE (clean-up of 1mL extract)

Q210	150mg MgSO ₄ , 25mg PSA	European EN 15662	100-pk.	26215
Q211	150mg MgSO ₄ , 25mg PSA, 25mg C18		100-pk.	26216
Q212	150mg MgSO ₄ , 25mg PSA, 2.5mg GCB	European EN 15662	100-pk.	26217
Q213	150mg MgSO ₄ , 25mg PSA, 7.5mg GCB	European EN 15662	100-pk.	26218
Q250	150mg MgSO ₄ , 50mg PSA	AOAC 2007.01	100-pk.	26124
Q251	150mg MgSO ₄ , 50mg PSA, 50mg C18	AOAC 2007.01	100-pk.	26125
Q253	150mg MgSO ₄ , 50mg PSA, 50mg GCB		100-pk.	26123
Q252	150mg MgSO ₄ , 50mg PSA, 50mg C18, 50mg GCB	AOAC 2007.01	100-pk.	26219

15mL Centrifuge Tubes for dSPE (clean-up of 6mL extract)

Q350	1200mg MgSO ₄ , 400mg PSA	AOAC 2007.01	50-pk.	26220
Q351	1200mg MgSO ₄ , 400mg PSA, 400mg C18	AOAC 2007.01	50-pk.	26221
Q352	1200mg MgSO ₄ , 400mg PSA, 400mg C18, 400mg GCB	AOAC 2007.01	50-pk.	26222
Q370	900mg MgSO ₄ , 150mg PSA	European EN 15662	50-pk.	26223
Q371	900mg MgSO ₄ , 150mg PSA, 15mg GCB	European EN 15662	50-pk.	26224
Q372	900mg MgSO ₄ , 150mg PSA, 45mg GCB	European EN 15662	50-pk.	26225
Q373	900mg MgSO ₄ , 150mg PSA, 150mg C18		50-pk.	26226
Q374	900mg MgSO ₄ , 300mg PSA, 150mg GCB		50-pk.	26126

TSCD = trisodium citrate dihydrate

DHS = disodium hydrogen citrate sesquihydrate

NaOAc = sodium acetate



Q-sep™ 3000 Centrifuge

- Meets requirements of AOAC and European QuEChERS methodology.
- Supports 50mL, 15mL, and 2mL centrifuge tubes.
- Small footprint requires less bench space.
- Safe and reliable—UL, CSA, and CE approved, 1-year warranty.



Centrifuge includes 50mL tube carriers (6), 50mL conical tube inserts (6), 4-place 15mL tube carriers (6), and 2mL tube adaptors (24).

Description	qty.	cat.#
Q-sep 3000 Centrifuge, 110V	ea.	26230
Q-sep 3000 Centrifuge, 220V	ea.	26231
Replacement Accessories		
50mL Tube Carrier for Q-sep 3000 Centrifuge	2-pk.	26232
50mL Conical Tube Insert for Q-sep 3000 Centrifuge	6-pk.	26249
4-Place Tube Carrier for Q-sep 3000 Centrifuge	2-pk.	26233
2mL Tube Adaptors for Q-sep 3000 Centrifuge	4-pk.	26234

GC and HPLC Columns

Rxi®-5Sil MS

(low polarity Crossbond® silarylene phase; selectivity same as DB-5MS)

- Engineered to be a low bleed fused silica GC/MS column.
- Excellent inertness for active compounds.
- Temperature range: -60°C to 350°C.

ID	df (μm)	temp. limits	30-Meter
0.25mm	0.25	-60 to 330/350°C	13623
	0.50	-60 to 330/350°C	13638
ID	df (μm)	temp. limits	20-Meter
0.18mm	0.18	-60 to 330/350°C	43602
	0.36	-60 to 330/350°C	43604

Ultra Aqueous C18 Columns (USP L1)

Physical Characteristics:

particle size: 3μm or 5μm, spherical; pore size: 100Å; carbon load: 15%; endcap: no; pH range: 2.5 to 7.5; temperature limit: 80°C

Chromatographic Properties:

Highly retentive and selective for reversed phase separations of polar analytes. Highly base-deactivated. Compatible with highly aqueous (up to 100%) mobile phases.

	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
Length	cat.#	cat.#	cat.#	cat.#
3μm Columns				
30mm	9178331	9178332	9178333	9178335
50mm	9178351	9178352	9178353	9178355
100mm	9178311	9178312	9178313	9178315
5μm Columns				
30mm	9178531	9178532	9178533	9178535
50mm	9178551	9178552	9178553	9178555
100mm	9178511	9178512	9178513	9178515
150mm	9178561	9178562	9178563	9178565
200mm	9178521	9178522	9178523	9178525
250mm	9178571	9178572	9178573	9178575

QuEChERS Standards

- Ready to use for QuEChERS extractions—no dilutions necessary.
- Support for GC and HPLC with MS, MS/MS, and selective detectors.



QuEChERS Internal Standard Mix for GC/ECD Analysis

PCB 18	tris-(1,3-dichloroisopropyl) phosphate
PCB 28	
PCB 52	
50μg/mL each in acetonitrile, 5mL/ampul	
cat. # 33265 (ea.)	

QuEChERS Internal Standard Mix for GC/MS Analysis

PCB 18	50μg/mL	tris-(1,3-dichloroisopropyl) phosphate	50
PCB 28	50	phosphate	50
PCB 52	50	triphenylmethane	10
triphenyl phosphate	20		
In acetonitrile, 5mL/ampul			
cat. # 33267 (ea.)			

QuEChERS Internal Standard Mix for GC/NPD and LC/MS/MS Analysis

triphenyl phosphate	20μg/mL
tris-(1,3-dichloroisopropyl)phosphate	50μg/mL
In acetonitrile, 5mL/ampul	
cat. # 33266 (ea.)	

QuEChERS Single-Component Reference Standards

Concentration is μg/mL. ACN=acetonitrile

Compound	Solvent	Conc.	cat.# (ea.)
PCB 18 (5mL)	ACN	50	33255
PCB 28 (5mL)	ACN	50	33256
PCB 52 (5mL)	ACN	50	33257
PCB 138 (5mL)	ACN	50	33262
PCB 153 (5mL)	ACN	50	33263
triphenylmethane (5mL)	ACN	10	33260
triphenylphosphate (5mL)	ACN	20	33258
tris(1,3-dichloroisopropyl) phosphate (5mL)	ACN	50	33259

QuEChERS Internal Standard Mix for LC/MS/MS Analysis





nicarbazin
10μg/mL in acetonitrile, 5mL/ampul
cat. # 33261 (ea.)

QuEChERS Quality Control Standards for GC/MS Analysis

PCB 138	PCB 153
50μg/mL each in acetonitrile, 5mL/ampul	
cat. # 33268 (ea.)	

anthracene
100μg/mL in acetonitrile, 5mL/ampul
cat. # 33264 (ea.)

Selection Guide for Q-sep™ Extraction and dSPE Tubes

Commodity types and examples		AOAC 2007.1	EN 15662	Mini-multiresidue	Additional products
	General purpose <ul style="list-style-type: none"> • Celery • Head lettuce • Cucumber • Melon 	Q-sep Q250 2mL, 100-pk. (cat.# 26124) Q-sep Q350 15mL, 50-pk. (cat.# 26220)	Q-sep Q210 2mL, 100-pk. (cat.# 26215) Q-sep Q370 15mL, 50-pk. (cat.# 26223)	Q-sep Q210 2mL, 100-pk. (cat.# 26215)	
	Fatty or waxy fruits & vegetables <ul style="list-style-type: none"> • Cereals • Avocado • Nuts & seeds • Dairy 	Q-sep Q251 2mL, 100-pk. (cat.# 26125) Q-sep Q351 15mL, 50-pk. (cat.# 26221)		Q-sep Q211 2mL, 100-pk. (cat.# 26216)	Q-sep Q373 15mL, 50-pk. (cat.# 26226)
	Pigmented fruits & vegetables <ul style="list-style-type: none"> • Strawberries • Sweet potatoes • Tomatoes 	Q-sep Q352 15mL, 50-pk. (cat.# 26222)	Q-sep Q212 2mL, 100-pk. (cat.# 26217) Q-sep Q371 15mL, 50-pk. (cat.# 26224)	Q-sep Q212 2mL, 100-pk. (cat.# 26217)	Q-sep Q253 2mL, 100-pk. (cat.# 26123)
	Highly pigmented fruits & vegetables <ul style="list-style-type: none"> • Red peppers • Spinach • Blueberries 	Q-sep Q252 2mL, 100-pk. (cat.# 26219)	Q-sep Q213 2mL, 100-pk. (cat.# 26218) Q-sep Q372 15mL, 50-pk. (cat.# 26225)	Q-sep Q213 2mL, 100-pk. (cat.# 26218)	Q-sep Q374 15mL, 50-pk. (cat.# 26126)
Download free instructions at www.restek.com/quechers		Instruction sheet# 805-01 002	Instruction sheet# 805-01 001	Instruction sheet# 805-01 001	Generic dSPE 805-01 003

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Estimated time to process 6 samples (min.)	120	30	4x faster
Solvent used (mL)	60-90	10	6-9x less solvent
Chlorinated waste (mL)	20-30	0	Safer, cheaper, greener
Glassware/specialized equipment	capacity for 200mL, quartz wool, funnel, water bath or evaporator	none	Ready-to-use



Q-sep™ QuEChERS Sample Prep Packets & Tubes

Description	Material	Methods	qty.	cat#
Extraction Salt Packets and 50mL Centrifuge Tubes				
Q110 kit	4g MgSO ₄ , 1g NaCl, 1g TSCD, 0.5g DHS with 50mL Centrifuge Tube	European EN 15662	50 packets & 50 tubes	26235
Q110 packets	4g MgSO ₄ , 1g NaCl, 1g TSCD, 0.5g DHS	European EN 15662	50 packets	26236
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Q150 packets	6g MgSO ₄ , 1.5g NaOAc	AOAC 2007.01	50 packets	26238
Empty 50mL Centrifuge Tube			50-pk.	26239

Sorbent Guide

Sorbent	Removes
PSA*	sugars, fatty acids, organic acids, anthocyanine pigments
C18	lipids, nonpolar interferences
GCB**	pigments, sterols, nonpolar interferences

*PSA—primary and secondary amine exchange material
 **GCB—graphitized carbon black

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Q372	900mg MgSO ₄ , 150mg PSA, 45mg GCB	European EN 15662	50-pk.	26225
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Q374	900mg MgSO ₄ , 300mg PSA, 150mg GCB		50-pk.	26126

TSCD = trisodium citrate dihydrate

DHS = disodium hydrogen citrate sesquihydrate

NaOAc = sodium acetate

Q-sep™ 3000 Centrifuge

- Meets requirements of AOAC and European QuEChERS methodology.
- Supports 50mL, 15mL, and 2mL centrifuge tubes.
- Small footprint requires less bench space.
- Safe and reliable—UL, CSA, and CE approved, 1-year warranty.



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2mL Tube Adaptors for Q-sep 3000 Centrifuge	4-pk.	26234

GC and HPLC Columns

Rxi®-5SIL MS

(low polarity Crossbond® silarylene phase; selectivity same as DB-5MS)

- Engineered to be a low bleed fused silica GC/MS column.
- Excellent inertness for active compounds.
- Temperature range: -60°C to 350°C.

ID	df (μm)	temp. limits	30-Meter
0.25mm	0.25	-60 to 330/350°C	13623
	0.50	-60 to 330/350°C	13638
ID	df (μm)	temp. limits	20-Meter
0.18mm	0.18	-60 to 330/350°C	43602
	0.36	-60 to 330/350°C	43604

Ultra Aqueous C18 Columns (USP L1)

Physical Characteristics:

particle size: 3μm or 5μm, spherical; pore size: 100Å; carbon load: 15%; endcap: no; pH range: 2.5 to 7.5; temperature limit: 80°C

Chromatographic Properties:

Highly retentive and selective for reversed phase separations of polar analytes. Highly base-deactivated. Compatible with highly aqueous (up to 100%) mobile phases.

	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
Length	cat.#	cat.#	cat.#	cat.#
3μm Columns				
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50mm	9178351	9178352	9178353	9178355
100mm	9178311	9178312	9178313	9178315
5μm Columns				
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50mm	9178551	9178552	9178553	9178555
100mm	9178511	9178512	9178513	9178515
150mm	9178561	9178562	9178563	9178565
200mm	9178521	9178522	9178523	9178525
250mm	9178571	9178572	9178573	9178575

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PCB 52	50	triphenylmethane	10
triphenyl phosphate	20		
In acetonitrile, 5mL/ampul			
cat. # 33267 (ea.)			

QuEChERS Internal Standard Mix for GC/NPD and LC/MS/MS Analysis

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tris-(1,3-dichloroisopropyl)phosphate	50μg/mL
In acetonitrile, 5mL/ampul	
cat. # 33266 (ea.)	

QuEChERS Single-Component Reference Standards

Concentration is μg/mL. ACN=acetonitrile

Compound	Solvent	Conc.	cat.# (ea.)
PCB 18 (5mL)	ACN	50	33255
PCB 28 (5mL)	ACN	50	33256
PCB 52 (5mL)	ACN	50	33257
PCB 138 (5mL)	ACN	50	33262
PCB 153 (5mL)	ACN	50	33263
triphenylmethane (5mL)	ACN	10	33260
triphenylphosphate (5mL)	ACN	20	33258
tris(1,3-dichloroisopropyl) phosphate (5mL)	ACN	50	33259

QuEChERS Internal Standard Mix for LC/MS/MS Analysis

nicarbazin
10μg/mL in acetonitrile, 5mL/ampul
cat. # 33261 (ea.)

QuEChERS Quality Control Standards for GC/MS Analysis

PCB 138	PCB 153
50µg/mL each in acetonitrile, 5mL/ampul	
cat. # 33268 (ea.)	
<hr/>	
anthracene	
100µg/mL in acetonitrile, 5mL/ampul	
cat. # 33264 (ea.)	



Lit. Cat.# FFTS1199A

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ISO 9001:2008
cert.# FM80397

QuEChERS Methodology: Mini-Multiresidue Method

Resprep™ Q110, cat.# 26213

Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS (“catchers”) method is based on work done and published by the US Department of Agriculture Eastern Regional Research Center in Wyndmoor, PA.¹ Researchers developed a new extraction method for pesticides in fruits and vegetables, coupled with a clean-up method that removes sugars, lipids, organic acids, sterols, proteins, pigments and excess water. This technique offers a user-friendly alternative to traditional liquid-liquid and solid phase extractions.

The process involves two simple steps. First, the homogenized samples are extracted and partitioned using an organic solvent and salt solution. Then, the supernatant is further extracted and cleaned using a **dispersive solid phase extraction (dSPE) technique**.

Restek products make this approach even simpler. We offer QuEChERS extraction and dSPE products in a variety of standard sizes and formats. The dSPE centrifuge tube format (available in 2mL and 15mL sizes) contains magnesium sulfate (to partition water from organic solvent) and primary secondary amine (PSA) adsorbent (to remove sugars and fatty acids). These tubes are available with or without graphitized carbon (to remove pigments and sterols) and/or C18 packing (to remove nonpolar interferences such as lipids).

Several detailed QuEChERS methods have been published and are listed below. Restek dSPE tubes, listed in Table I (back page), are formulated according to these methods.

- **Mini-Multiresidue Method**

QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products²

- **AOAC Official 2007.01 Method**

Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate³

- **European prEN_15662 Method, Version 2007-10-24**

Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and Clean-up by Dispersive SPE—QuEChERS-method⁴

QuEChERS-based methods have several basic steps in common, which are described below. Specific procedures for sample extraction and dSPE sample clean-up according to *QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*² are given in the following sections.



General Procedures (common to all 3 QuEChERS-based methods listed above)

Step 1: Sample preparation and extraction

Commodities are uniformly ground. Internal standards are also added at this point. Various salts, acids and buffers may then be added to enhance extraction efficiency and protect sensitive analytes.

Step 2: Sample extract cleanup

A subsample of the modified solvent extract from Step 1 is cleaned up using dSPE. Small polypropylene centrifuge tubes are pre-filled with precise weights and proportions of bulk drying salts and SPE adsorbent packings to remove excess water and unwanted contaminants from the sample extracts. After a brief agitation and centrifugation, the cleaned extracts are then prepared for analysis.

Step 3: Sample analysis

Samples may be pH adjusted, solvent-exchanged, or treated with additional agents, to protect sensitive analytes or improve analysis by either GC/MS or LC/MS.

Multiresidue QuEChERS 2007 Procedure

The procedures below are based on *QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*². For complete information, refer to the original source method at www.quechers.de or www.quechers.com.

Sample Extraction

1. Homogenize the frozen commodity to generate a uniform sample representative of the product (Figure 1). For products with more than 5% fat (w/w), see the specialized sample preparation procedure in the source method, *QuEChERS-A Mini Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*.²
2. Weigh 10g of homogenized product into a clean 50mL tube (cat.#26227) as shown in Figure 2.
3. Add 10mL acetonitrile and an appropriate amount of an internal standard solution. See suggestions of internal standards and volume in the source method.²
4. Shake vigorously for 1 minute by hand (Figure 3).



Figure 1



Figure 2



Figure 3 Shake vigorously by hand for 1 minute.

Sample Drying and Buffering

1. Add the contents (listed below) of a Resprep™ Q110 tube (cat.# 26213) to each extracted sample (Figure 4).

4.0g ± 0.2g	magnesium sulfate, anhydrous
1.0g ± 0.05	sodium chloride
1.0g ± 0.05g	trisodium citrate dihydrate
0.5g ± 0.03g	disodium hydrogencitrate sesquihydrate

Note:

If pH <3, sample should be adjusted with addition of 600µL of 5N NaOH (lemons, limes, currants).
If pH >3 and <5, sample should be adjusted with 200µL of 5N NaOH.

2. Shake immediately and vigorously 1 minute (Figure 3).



Figure 4

Phase Separation

Centrifuge for 5 minutes at 3,000 U/min. to separate the solid material (Figure 5). Proceed with dSPE sample clean-up or analyze extract directly without clean-up.

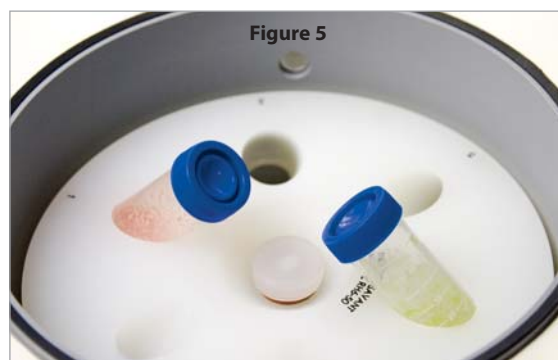


Figure 5

dSPE Sample Clean-up

The sample can be analyzed directly from the raw extract, especially if pesticides with acidic groups (e.g. phenoxyacid herbicides) are of interest. Alternatively, sample clean-up methods can be applied. Specifically, dispersive solid phase extraction is discussed here. Restek dSPE tubes are formulated in accordance with published methods and are listed in Table 1. Select tubes based on the method and sample type; general guidelines for different sample types include:

For samples with co-extracted fats or waxes:

Before or after clean-up, samples are put in freezer (>1hr. to overnight). Cold samples are then re-centrifuged and fats or waxes are removed. If fat remains, clean up with 25mg PSA, 150mg MgSO₄, and 25mg C18 **per mL** of extract. If no fat remains, clean up with 25mg PSA and 150mg MgSO₄ **per mL** of extract.

For samples with remaining fats:

Clean up with 25mg PSA, 150mg MgSO₄, and 25mg C18 **per mL** of extract (see above).

For samples with intensely colored extracts:

Clean up with 25mg PSA, 150mg MgSO₄, and 7.5mg graphitized carbon **per mL** of extract.

For samples with less intensely colored extracts, or high carotinoid or chlorophyll levels:

Clean up with 25mg PSA, 150mg MgSO₄, and 2.5mg graphitized carbon **per mL** of extract.

For all other samples:

Clean up with 25mg PSA and 150mg MgSO₄ **per mL** of extract. Once tubes are selected, dSPE sample clean-up can be accomplished according to the procedure shown below.



1. Using the centrifuged extracts resulting from the **phase separation** stage of sample extract preparation, transfer the supernatant to the dSPE tube as shown in Figure 6. Use Table I to determine the volume of sample that should be transferred.
2. Shake vigorously for 30 seconds or 2 minutes (Figure 7). Use Table I to determine the suggested shake time.
3. Centrifuge for 5 minutes at 3,000U/min. to separate the solid material (Figure 8).
4. Immediately adjust the supernatant pH using a 5% formic acid in acetonitrile solution. Use 10µL **per mL** of supernatant. For sulfonyl urea herbicides, carbosulfan, and benfuracarb, analyze the supernatant without any pH adjustment.
5. Transfer sample to an autosampler vial and test using GC or LC methods (Figure 9).

Note: To determine the amount (mg) of PSA, MgSO₄, and graphitized carbon, use the suggested number of milligrams and multiply by the number of mLs you want to extract.

Table I Restek dSPE tubes (organized by published method).

Cat.#	Name	Centrifuge Tube Size (mL)	Contains	Method	Sample Volume (mL)	Shake Time (min.)	Centrifuge Speed	Centrifuge Time (min.)
26216	Resprep Q211	2	150mg MgSO ₄ , 25mg PSA, 25mg C18	Mini-Multiresidue	1	0.5	3,000 U/min.	5
26215	Resprep Q210	2	150mg MgSO ₄ , 25mg PSA	Mini-Multiresidue, European prEN-15662	1	0.5	3,000 U/min.	5
26217	Resprep Q212	2	150mg MgSO ₄ , 25mg PSA, 2.5mg GCB	Mini-Multiresidue, European prEN-15662	1	2	3,000 U/min.	5
26218	Resprep Q213	2	150mg MgSO ₄ , 25mg PSA, 7.5mg GCB	Mini-Multiresidue, European prEN-15662	1	2	3,000 U/min.	5
26223	Resprep Q370	15	900mg MgSO ₄ , 150mg PSA	European prEN-15662	6	0.5	3,000 U/min.	5
26224	Resprep Q371	15	900mg MgSO ₄ , 150mg PSA, 15mg GCB	European prEN-15662	6	2	3,000 U/min.	5
26225	Resprep Q372	15	900mg MgSO ₄ , 150mg PSA, 45mg GCB	European prEN-15662	6	2	3,000 U/min.	5
26124	Resprep Q250	2	150mg MgSO ₄ , 50mg PSA	AOAC 2007.01	1	0.5	>1,500 rcf	1
26125	Resprep Q251	2	150mg MgSO ₄ , 50mg PSA, 50mg C18	AOAC 2007.01	1	0.5	>1,500 rcf	1
26219	Resprep Q252	2	150mg MgSO ₄ , 50mg PSA, 50mg C18, 50mg GCB	AOAC 2007.01	1	0.5	>1,500 rcf	1
26220	Resprep Q350	15	1,200mg MgSO ₄ , 400mg PSA	AOAC 2007.01	8	0.5	>1,500 rcf	1
26221	Resprep Q351	15	1,200mg MgSO ₄ , 400mg PSA, 400mg C18	AOAC 2007.01	8	0.5	>1,500 rcf	1
26222	Resprep Q352	15	1,200mg MgSO ₄ , 400mg PSA, 400mg C18, 400mg GCB	AOAC 2007.01	8	0.5	>1,500 rcf	1
26123	Resprep Q253	2	150mg MgSO ₄ , 50mg PSA, 50mg GCB	AOAC 2007.01	1	2	>1,500 rcf	1
26226	Resprep Q373	15	900mg MgSO ₄ , 150mg PSA, 150mg C18	similar to European prEN-15662	6	0.5	3,000 U/min.	5
26126	Resprep Q374	15	900mg MgSO ₄ , 300mg PSA, 150mg GCB	NA	6	2	3,000 U/min.	5

PSA = primary and secondary exchange material

GCB = graphitized carbon black

Notes:

U/min.= Undrehungen pro minute and is the German unit

of revolutions per minute (RPM)

rcf = relative centrifugal force and can be converted to RPM using $rcf = 1.12r \left(\frac{RPM}{1000} \right)^2$

r = the radius of the centrifuge rotation.

References

1. M. Anastassiades, S.J. Lehotay, D. Stajnbaher, F.J. Schenck, J. AOAC International 86, p. 412-431 (2003).
2. QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products. [http:// www.quechers.com](http://www.quechers.com) (accessed July 15, 2008).
3. AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.
4. prEN 15662 Version 2007-10-24, Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and Clean-up by Dispersive SPE (QuEChERS-method).



QuEChERS Methodology: AOAC Approach

Resprep™ Q150, cat.# 26214

Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS (“catchers”) method is based on work done and published by the US Department of Agriculture Eastern Regional Research Center in Wyndmoor, PA.¹ Researchers developed a new extraction method for pesticides in fruits and vegetables, coupled with a clean-up method that removes sugars, lipids, organic acids, sterols, proteins, pigments and excess water. This technique offers a user-friendly alternative to traditional liquid-liquid and solid phase extractions.

The process involves two simple steps. First, the homogenized samples are extracted and partitioned using an organic solvent and salt solution. Then, the supernatant is further extracted and cleaned using a **dispersive solid phase extraction (dSPE) technique**.

Restek products make this approach even simpler. We offer QuEChERS extraction and dSPE products in a variety of standard sizes and formats. The dSPE centrifuge tube format (available in 2mL and 15mL sizes) contains magnesium sulfate (to partition water from organic solvent) and primary secondary amine (PSA) adsorbent (to remove sugars and fatty acids). These tubes are available with or without graphitized carbon (to remove pigments and sterols) and/or C18 packing (to remove nonpolar interferences such as lipids).

Several detailed QuEChERS methods have been published and are listed below. Restek dSPE tubes, listed in Table I, are formulated according to these methods.

- **Mini-Multiresidue Method:** QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products²
- **AOAC Official 2007.01 Method:** Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate³
- **European prEN_15662 Method, Version 2007-10-24:** Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and Clean-up by Dispersive SPE—QuEChERS-method⁴

General Procedures

Several common steps to all 3 QuEChERS-based methods above. Specific procedures for sample extraction and dSPE sample clean-up according to *QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*² are given inside this instruction guide.

Step 1: Sample preparation and extraction

Commodities are uniformly ground. Internal standards are also added at this point. Various salts, acids and buffers may then be added to enhance extraction efficiency and protect sensitive analytes.

Step 2: Sample extract cleanup

A subsample of the modified solvent extract from Step 1 is cleaned up using dSPE. Small polypropylene centrifuge tubes are pre-filled with precise weights and proportions of bulk drying salts and SPE adsorbent packings to remove excess water and unwanted contaminants from the sample extracts. After a brief agitation and centrifugation, the cleaned extracts are then prepared for analysis.

Step 3: Sample analysis

Samples may be pH adjusted, solvent-exchanged, or treated with additional agents, to protect sensitive analytes or improve analysis by either GC/MS or LC/MS.

References

1. M. Anastassiades, S.J. Lehotay, D. Stajnbauer, F.J. Schenck, J. AOAC International 86, p. 412-431 (2003).
2. QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products. www.quechers.com (accessed July 15, 2008).
3. AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.
4. prEN 15662 Version 2007-10-24, Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and Clean-up by Dispersive SPE (QuEChERS-method).

Table I Restek dSPE tubes (organized by published method).

Cat.#	Name	Centrifuge Tube		Method	Sample Volume (mL)	Shake Time (min.)	Centrifuge Speed	Centrifuge Time (min.)
		Size (mL)	Contains					
26216	Resprep Q211	2	150mg MgSO ₄ , 25mg PSA, 25mg C18	Mini-Multiresidue	1	0.5	3,000 U/min.	5
26215	Resprep Q210	2	150mg MgSO ₄ , 25mg PSA	Mini-Multiresidue, European prEN-15662	1	0.5	3,000 U/min.	5
26217	Resprep Q212	2	150mg MgSO ₄ , 25mg PSA, 2.5mg GCB	Mini-Multiresidue, European prEN-15662	1	2	3,000 U/min.	5
26218	Resprep Q213	2	150mg MgSO ₄ , 25mg PSA, 7.5mg GCB	Mini-Multiresidue, European prEN-15662	1	2	3,000 U/min.	5
26223	Resprep Q370	15	900mg MgSO ₄ , 150mg PSA	European prEN-15662	6	0.5	3,000 U/min.	5
26224	Resprep Q371	15	900mg MgSO ₄ , 150mg PSA, 15mg GCB	European prEN-15662	6	2	3,000 U/min.	5
26225	Resprep Q372	15	900mg MgSO ₄ , 150mg PSA, 45mg GCB	European prEN-15662	6	2	3,000 U/min.	5
26124	Resprep Q250	2	150mg MgSO ₄ , 50mg PSA	AOAC 2007.01	1	0.5	>1,500 rcf	1
26125	Resprep Q251	2	150mg MgSO ₄ , 50mg PSA, 50mg C18	AOAC 2007.01	1	0.5	>1,500 rcf	1
26219	Resprep Q252	2	150mg MgSO ₄ , 50mg PSA, 50mg C18, 50mg GCB	AOAC 2007.01	1	0.5	>1,500 rcf	1
26220	Resprep Q350	15	1,200mg MgSO ₄ , 400mg PSA	AOAC 2007.01	8	0.5	>1,500 rcf	1
26221	Resprep Q351	15	1,200mg MgSO ₄ , 400mg PSA, 400mg C18	AOAC 2007.01	8	0.5	>1,500 rcf	1
26222	Resprep Q352	15	1,200mg MgSO ₄ , 400mg PSA, 400mg C18, 400mg GCB	AOAC 2007.01	8	0.5	>1,500 rcf	1
26123	Resprep Q253	2	150mg MgSO ₄ , 50mg PSA, 50mg GCB	AOAC 2007.01	1	2	>1,500 rcf	1
26226	Resprep Q373	15	900mg MgSO ₄ , 150mg PSA, 150mg C18	similar to European prEN-15662	6	0.5	3,000 U/min.	5
26126	Resprep Q374	15	900mg MgSO ₄ , 300mg PSA, 150mg GCB	NA	6	2	3,000 U/min.	5

PSA = primary and secondary exchange material
GCB = graphitized carbon black

U/min. = Umdrehungen pro minute and is the German unit
of revolutions per minute (RPM)

rcf = relative centrifugal force and can be converted to RPM using $rcf = 1.12r \left(\frac{RPM}{1000} \right)^2$
r = the radius of the centrifuge rotation.

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AOAC Approach

The procedures below are based on AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.³ For complete information, refer to the original source method.



Sample Extraction

1. Homogenize the frozen commodity to generate a uniform sample representative of the product (Figure 1).
2. Weigh 15g of homogenized product into a clean 50mL tube (cat.# 26227) (Figure 2).
3. Add 15mL of 1% acetic acid in acetonitrile (v/v) and an appropriate amount of an internal standard solution. Add the contents of a Resprep™ Q150 tube (cat.# 26214) to each extracted sample (Table II, Figure 3).
4. Shake vigorously for 1 minute by hand (Figure 4).
5. Centrifuge for 1 minute at >1,500 rcf to separate the solid material (Figure 5). Proceed with sample clean-up.

Table II
Resprep™ Q150 Tube Contents
6.0g magnesium sulfate, anhydrous



dSPE Sample Clean-up

Restek dSPE tubes are formulated in accordance with published methods and are listed in Table I. Select tubes based on the method used and sample type; general guidelines for different sample types include:

For samples with greater than 1% fat:

Clean up with 50mg PSA, 150mg MgSO₄, and 50mg C18 **per mL** of extract.

For samples with colored extracts:

Clean up with 50mg PSA, 150mg MgSO₄, and 50mg graphitized carbon **per mL** of extract.

For samples with colored extracts containing greater than 1% fat:

Clean up with 50mg PSA, 150mg MgSO₄, 50mg C18, and 50mg graphitized carbon **per mL** of extract.

For all other samples:

Clean up with 50mg PSA and 150mg MgSO₄ **per mL** of extract.

Note: To determine the amount (mg) of PSA, MgSO₄, and graphitized carbon, use the suggested number of milligrams and multiply by the number of mLs you want to extract.

Once tubes are selected, dSPE sample clean-up can be accomplished according to the following procedure:

1. Using the centrifuged sample extracts, transfer the supernatant to the dSPE tube as shown in Figures 6A & 6B. Use Table 1 to determine the volume of sample that should be transferred.
2. Shake vigorously for 30 seconds (Figure 7).
3. Centrifuge for 1 minute at >1,500 rcf to separate the solid material (Figure 8).
4. Transfer sample to an autosampler vial and test using GC or LC methods. Additional steps to prepare the sample for specific types of analysis are addressed in AOAC Official Method 2007.01.



dSPE Sample Clean-up

**cat.#s 26215, 26216, 26217, 26218, 26124, 26125, 26123,
26219, 26220, 26221, 26222, 26223, 26224, 26225, 26226, 26126**

Dispersive solid phase extraction (dSPE) is one technique used for sample cleanup. Sample cleanup often is preceded by sample extraction; extraction methods vary from traditional liquid-liquid extraction to cartridge solid phase extraction. This Restek dSPE product can be used as an independent clean-up tool or as part of a QuEChERS method.

Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS (“catchers”) method is based on work done and published by the US Department of Agriculture Eastern Regional Research Center in Wyndmoor, PA.¹ Researchers developed a new extraction method for pesticides in fruits and vegetables, coupled with a clean-up method that removes sugars, lipids, organic acids, sterols, proteins, pigments and excess water. This technique offers a user-friendly alternative to traditional liquid-liquid and solid phase extractions.

The procedure involves two simple steps. First, the homogenized samples are extracted and partitioned using an organic solvent and salt solution. Then, the supernatant is further extracted and cleaned using a **dispersive solid phase extraction (dSPE) technique**.

Restek products make this approach even simpler. We offer QuEChERS extraction and dSPE products in a variety of standard sizes and formats. The dSPE centrifuge tube format (available in 2mL and 15mL sizes) contains magnesium sulfate (to partition water from organic solvent) and primary secondary amine (PSA) adsorbent (to remove sugars and fatty acids). These tubes are available with or without graphitized carbon (to remove pigments and sterols) and/or C18 packing (to remove nonpolar interferences such as lipids).

Several detailed QuEChERS methods have been published and are listed below. Restek dSPE tubes, listed in Table I (back page), are formulated according to these methods.

- **Mini-Multiresidue Method**

QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products²

- **AOAC Official 2007.01 Method**

Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate³

- **European prEN_15662 Method, Version 2007-10-24**

Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and Clean-up by Dispersive SPE—QuEChERS-method⁴



QuEChERS-based methods have several basic steps in common, which are described below. Specific procedures for sample extraction and dSPE sample clean-up according to *QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*² are given in the following sections.

General Procedures (common for all 3 QuEChERS-based methods listed above)

Step 1: Sample preparation and extraction

Commodities are uniformly ground. Internal standards are also added at this point. Various salts, acids and buffers may then be added to enhance extraction efficiency and protect sensitive analytes.

Step 2: Sample extract cleanup

A subsample of the modified solvent extract from Step 1 is cleaned up using dSPE. Small polypropylene centrifuge tubes are pre-filled with precise weights and proportions of bulk drying salts and SPE adsorbent packings to remove excess water and unwanted contaminants from the sample extracts. After a brief agitation and centrifugation, the cleaned extracts are then prepared for analysis.

Step 3: Sample analysis

Samples may be pH adjusted, solvent-exchanged, or treated with additional agents, to protect sensitive analytes or improve analysis by either GC/MS or LC/MS.

dSPE Sample Clean-up

These instructions focus on dSPE sample clean-up procedures. *Sample extraction precedes dSPE clean-up and generalized extraction instructions are provided in the Sample Extraction, Sample Drying and Buffering, and Phase Separation sections.*

The sample can be analyzed directly from the raw extract, especially if pesticides with acidic groups (e.g. phenoxyacid herbicides) are of interest. Alternatively, sample clean-up methods can be applied. Specifically, dispersive solid phase extraction is discussed here. Restek dSPE tubes are formulated in accordance with published methods and are listed in Table I. Select tubes based on the method and sample type; general guidelines for different sample types include:

For samples with co-extracted fats or waxes:

Before or after clean-up, samples are put in freezer (>1hr. to overnight). Cold samples are then re-centrifuged and fats or waxes are removed. If fat remains, clean up PSA, MgSO₄, C18 tubes is recommended. If no fat remains, PSA and MgSO₄ tubes are recommended.

For samples with remaining fats:

Clean up with PSA, MgSO₄, and C18 tubes is recommended (see above).

For samples with colored extracts or high carotinoid or chlorophyll levels:

Clean up with PSA, MgSO₄, and graphitized carbon tubes is recommended.

For samples with colored extracts containing greater than 1% fat:

Clean up with PSA, MgSO₄, C18, and graphitized carbon tubes is recommended.

For all other samples:

Clean up with PSA and MgSO₄ tubes is recommended.



Once tubes are selected, dSPE sample clean-up can be accomplished according to the following procedure, which are based on *QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*². For complete information, refer to the original source method at www.quechers.de or www.quechers.com:

1. Using the centrifuged extracts resulting from the **phase separation stage of sample extract preparation**, transfer the supernatant to the dSPE tube as shown in Figure 1. Use Table I to determine the volume of sample that should be transferred.
2. Shake vigorously for 30 seconds or 2 minutes (Figure 2). Use Table I to determine the suggested shake time.
3. Centrifuge for 5 minutes at 3,000U/min. to separate the solid material (Figure 3).
4. Immediately adjust the supernatant pH using a 5% formic acid in acetonitrile solution. Use 10µL **per mL** of supernatant. For sulfonyl urea herbicides, carbosulfan, and benfuracarb, analyze the supernatant without any pH adjustment.
5. Transfer sample to an autosampler vial and test using GC or LC methods (Figure 4).

Sample Extraction

1. Homogenize the frozen commodity to generate a uniform sample representative of the product (Figure 5). For products with more than 5% fat (w/w), see the specialized sample preparation procedure in the source method, *QuEChERS-A Mini Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*.²
2. Weigh 10g of homogenized product into a clean 50mL tube (cat.#26227) as shown in Figure 6.
3. Add 10mL acetonitrile and an appropriate amount of an internal standard solution. See suggestions of internal standards and volume in the source method.²
4. Shake vigorously for 1 minute by hand (Figure 7).



Figure 5



Figure 6

Sample Drying and Buffering

1. Add the contents (listed below) of a Resprep™ Q110 tube (cat.# 26213) to each extracted sample (Figure 8).

4.0g ± 0.2g	magnesium sulfate, anhydrous
1.0g ± 0.05	sodium chloride
1.0g ± 0.05g	trisodium citrate dihydrate
0.5g ± 0.03g	disodium hydrogencitrate sesquihydrate

Note:

If pH <3, sample should be adjusted with addition of 600µL of 5N NaOH (lemons, limes, currants).
If pH >3 and <5, sample should be adjusted with 200µL of 5N NaOH (raspberries).



Figure 7 Shake vigorously by hand for 1 minute.



Figure 8

Phase Separation

Centrifuge for 5 minutes at 3,000 U/min. to separate the solid material (Figure 9). Proceed with dSPE sample clean-up or analyze extract directly without clean-up.

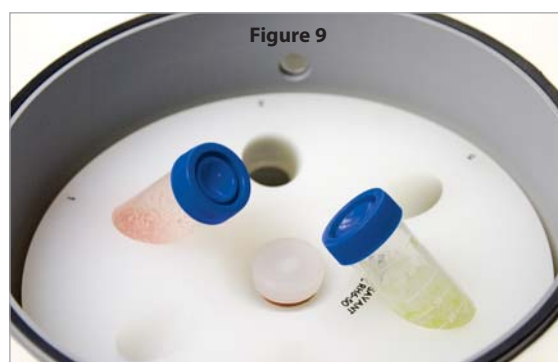


Figure 9

Table I Restek dSPE tubes (organized by published method).

Cat.#	Name	Centrifuge Tube Size (mL)	Contains	Method	Sample Volume (mL)	Shake Time (min.)	Centrifuge Speed	Centrifuge Time (min.)
26216	Resprep Q211	2	150mg MgSO ₄ , 25mg PSA, 25mg C18	Mini-Multiresidue	1	0.5	3,000 U/min.	5
26215	Resprep Q210	2	150mg MgSO ₄ , 25mg PSA	Mini-Multiresidue, European prEN-15662	1	0.5	3,000 U/min.	5
26217	Resprep Q212	2	150mg MgSO ₄ , 25mg PSA, 2.5mg GCB	Mini-Multiresidue, European prEN-15662	1	2	3,000 U/min.	5
26218	Resprep Q213	2	150mg MgSO ₄ , 25mg PSA, 7.5mg GCB	Mini-Multiresidue, European prEN-15662	1	2	3,000 U/min.	5
26223	Resprep Q370	15	900mg MgSO ₄ , 150mg PSA	European prEN-15662	6	0.5	3,000 U/min.	5
26224	Resprep Q371	15	900mg MgSO ₄ , 150mg PSA, 15mg GCB	European prEN-15662	6	2	3,000 U/min.	5
26225	Resprep Q372	15	900mg MgSO ₄ , 150mg PSA, 45mg GCB	European prEN-15662	6	2	3,000 U/min.	5
26124	Resprep Q250	2	150mg MgSO ₄ , 50mg PSA	AOAC 2007.01	1	0.5	>1,500 rcf	1
26125	Resprep Q251	2	150mg MgSO ₄ , 50mg PSA, 50mg C18	AOAC 2007.01	1	0.5	>1,500 rcf	1
26219	Resprep Q252	2	150mg MgSO ₄ , 50mg PSA, 50mg C18, 50mg GCB	AOAC 2007.01	1	0.5	>1,500 rcf	1
26220	Resprep Q350	15	1,200mg MgSO ₄ , 400mg PSA	AOAC 2007.01	8	0.5	>1,500 rcf	1
26221	Resprep Q351	15	1,200mg MgSO ₄ , 400mg PSA, 400mg C18	AOAC 2007.01	8	0.5	>1,500 rcf	1
26222	Resprep Q352	15	1,200mg MgSO ₄ , 400mg PSA, 400mg C18, 400mg GCB	AOAC 2007.01	8	0.5	>1,500 rcf	1
26123	Resprep Q253	2	150mg MgSO ₄ , 50mg PSA, 50mg GCB	AOAC 2007.01	1	2	>1,500 rcf	1
26226	Resprep Q373	15	900mg MgSO ₄ , 150mg PSA, 150mg C18	similar to European prEN-15662	6	0.5	3,000 U/min.	5
26126	Resprep Q374	15	900mg MgSO ₄ , 300mg PSA, 150mg GCB	NA	6	2	3,000 U/min.	5

PSA = primary and secondary exchange material

GCB = graphitized carbon black

Notes:

U/min.= Undrehungen pro minute and is the German unit of revolutions per minute (RPM)

rcf = relative centrifugal force and can be converted to RPM using $rcf = 1.12r \left(\frac{RPM}{1000} \right)^2$
r = the radius of the centrifuge rotation.

References

1. M. Anastassiades, S.J. Lehotay, D. Stajnbaher, F.J. Schenck, J. AOAC International 86, p. 412-431 (2003).
2. QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products. [http:// www.quechers.com](http://www.quechers.com) (accessed July 15, 2008).
3. AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.
4. prEN 15662 Version 2007-10-24, Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and Clean-up by Dispersive SPE (QuEChERS-method).



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- First centrifuge specifically designed for QuEChERS methodology.
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QuEChERS Internal Standards and Quality Control Standards

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- Support for GC and HPLC with MS, MS/MS, and selective detectors.
- Ready to use for QuEChERS extractions—no dilutions necessary.

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Q-sep™ QuEChERS Tubes For Extraction and Clean-Up of Pesticide Residues From Food

Products

- Fast, simple sample extraction and cleanup using dSPE.
- Fourfold increases in sample throughput.
- Fourfold decreases in material cost.
- Convenient, ready to use centrifuge tubes with ultra pure, preweighed adsorbent mixes.

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QuEChERS Approach

Prepare samples more quickly, easily, and cost-effectively with QuEChERS.

	Mini-Luke or Modified Luke Method	QuEChERS	Savings with QuEChERS
Estimated time to process 6 samples (min.)	120	30	4x faster
Solvent used (mL)	60-90	10	6-9x less solvent
Chlorinated waste (mL)	20-30	0	Safer, cheaper, greener
Glassware/Specialized equipment	capacity for 200mL, quartz wool, funnel, water bath or evaporator	none	Ready-to-use

Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS ("catchers") method is based on work done and published by the US Department of Agriculture Eastern Regional Research Center in Wyndmoor, PA.(1) Researchers there were looking for a simple, effective, and inexpensive way



to extract and clean pesticide residues from the many varied sample matrices with which they routinely worked. They had been using the Modified Luke Extraction Method, which is highly effective and rugged, but is both labor and glassware intensive, leading to a relatively high cost per sample. Solid phase extraction also had been effective, but the complex matrices the investigators were dealing with required multiple individual cartridges and packings to remove the many classes of interferences, adding costs and complexity to the process. A new method would have to remove sugars, lipids, organic acids, sterols, proteins, pigments and excess water, any of which often are present, but still be easy to use and inexpensive.



The researchers developed a simple two-step procedure. First, the homogenized samples are extracted and partitioned, using an organic solvent and salt solution. Then, the supernatant is further extracted and cleaned, using a dispersive SPE technique. Multiple adsorbents are placed in a centrifuge tube, along with the 1mL of organic solvent and the extracted residues partitioned from step 1. The contents are thoroughly mixed, then centrifuged, producing a clean extract ready for a variety of GC or HPLC analytical techniques.(2) Validation and proficiency data for the QuEChERS method are available for a wide variety of pesticides in several common food matrices at www.quechers.com

Using the dispersive SPE approach, the quantity and type of adsorbents can be easily adjusted for differing matrix interferences and "difficult" analytes. Results from this approach have been verified and modified at several USDA and Food and Drug Administration labs, and the method now is widely accepted for many types of pesticide residue samples.

Restek Q-sep™ products make the QuEChERS approach even simpler. Extraction salts are preweighed and provided with 50mL centrifuge tubes. The dSPE centrifuge tubes, available in 2mL and 15mL sizes, contain magnesium sulfate (to partition water from organic solvent) and PSA* adsorbent (to remove sugars and fatty acids), with or without graphitized carbon black (to remove pigments and sterols) or C18 (to remove nonpolar interferences). Custom products are available by quote request. If you are frustrated by the time and cost involved with your current approach to pesticide sample extraction and cleanup, we suggest you try this simple and economical new method.



We have products compliant with AOAC, Multi-miniresidue and European methods.

[Click here for our complete Q-sep™ QuEChERS product listing.](#)

Did You Know?

Multiple sorbents are used to remove different types of interferences.

MgSO₄—removes excess water

PSA*—removes sugars, fatty acids, organic acids, and anthocyanine pigments

C18—removes nonpolar interferences

Carbon—removes pigments, sterols, and nonpolar interferences

*PSA—primary and secondary amine exchange material.

References

1. Anastassiades, M., S.J. Lehotay, D. Stajnbaher, F.J. Schenck, *Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce*, J AOAC International, 2003, vol 86 no 22, pp 412-431.
2. Schenck, F.J., *SPE Cleanup and the Analysis of PPB Levels of Pesticides in Fruits and Vegetables*. Florida Pesticide Residue Workshop, 2002.



QuEChERS SPE Tubes for Pesticide Residue Analysis

Laboratory Equipment Magazine's 2009 Readers' Choice Awards Winner!



- Fast, simple sample extraction and cleanup using dSPE.
- Fourfold increases in sample throughput.
- Fourfold decreases in material cost.
- Convenient, ready to use centrifuge tubes with ultra pure, preweighed adsorbent mixes.

Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS ("catchers") method, developed by the USDA Eastern Regional Research Center¹, has become very popular for extraction and clean-up of pesticide residue samples. Our products are available in three centrifuge tube sizes to meet the needs of both extraction and cleanup of a wide variety of sample matrices following various methods.

The researchers developed a simple two-step procedure. First, the homogenized samples are extracted and partitioned, using an organic solvent and salt solution. Then, the supernatant is further extracted and cleaned, using a dispersive SPE technique. Multiple adsorbents are placed in a centrifuge tube, along with the 1mL of organic solvent and the extracted residues partitioned from step 1. The contents are thoroughly mixed, then centrifuged, producing a clean extract ready for a variety of GC or HPLC analytical techniques.² Validation and proficiency data for the QuEChERS method are available for a wide variety of pesticides in several common food matrices at www.quechers.com

Multiple sorbents are used to extract different types of interferences.

MgSO₄ removes excess water

PSA* removes sugars, fatty acids, organic acids, and anthocyanine pigments

C18 removes nonpolar interferences

GCB* removes pigments, sterols, and nonpolar interferences

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Description	Material	Method	qty.	Cat.#
50mL Centrifuge Tubes for Sample Extraction				
Resprep Q110	4g MgSO ₄ , 1g NaCl, 1g trisodium citrate dihydrate, .5g disodium hydrogencitrate sesquihydrate	Mini-Multiresidue, European EN-15662	50-pk.	26213
Resprep Q150	6g MgSO ₄ , 1.5g NaOAc	AOAC 2007.1	50-pk.	26214
Empty 50mL Centrifuge Tube--		Mini-Multiresidue, European EN-15662, AOAC 2007.1	25-pk.	26227
2mL Micro-Centrifuge Tubes for dSPE (clean-up of 1mL extract)				
Resprep Q210	150mg MgSO ₄ , 25mg PSA	Mini-Multiresidue, European EN-15662	100-pk.	26215
Resprep Q211	150mg MgSO ₄ , 25mg PSA, 25mg C18	Mini-Multiresidue	100-pk.	26216
Resprep Q212	150mg MgSO ₄ , 25mg PSA, 2.5mg GCB	Mini-Multiresidue, European EN-15662	100-pk.	26217
Resprep Q213	150mg MgSO ₄ , 25mg PSA, 7.5mg GCB	Mini-Multiresidue, European EN-15662	100-pk.	26218
Resprep Q250	150mg MgSO ₄ , 50mg PSA	AOAC 2007.1	100-pk.	26124
Resprep Q251	150mg MgSO ₄ , 50mg PSA, 50mg C18	AOAC 2007.1	100-pk.	26125
Resprep Q253	150mg MgSO ₄ , 50mg PSA, 50mg GCB	--	100-pk.	26123
Resprep Q252	150mg MgSO ₄ , 50mg PSA, 50mg C18, 50mg GCB	AOAC 2007.1	100-pk.	26219
15mL Centrifuge Tubes for dSPE (clean-up of 6mL extract)				
Resprep Q350	1200mg MgSO ₄ , 400mg PSA	AOAC 2007.1	50-pk.	26220
Resprep Q351	1200mg MgSO ₄ , 400mg PSA, 400mg C18	AOAC 2007.1	50-pk.	26221
Resprep Q352	1200mg MgSO ₄ , 400mg PSA, 400mg C18, 400mg GCB	AOAC 2007.1	50-pk.	26222
Resprep Q370	900mg MgSO ₄ , 150mg PSA	European EN-15662	50-pk.	26223
Resprep Q371	900mg MgSO ₄ , 150mg PSA, 15mg GCB	European EN-15662	50-pk.	26224
Resprep Q372	900mg MgSO ₄ , 150mg PSA, 45mg GCB	European EN-15662	50-pk.	26225
Resprep Q373	900mg MgSO ₄ , 150mg PSA, 150mg C18	--	50-pk.	26226
Resprep Q374	900mg MgSO ₄ , 300mg PSA, 150mg GCB	--	50-pk.	26126

PSA--primary and secondary amine exchange material

GCB--graphitized carbon black

References (not available from Restek)

1. Anastassiades, M., S.J. Lehotay, D. Stajnbaher, F.J. Schenck, Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce, J AOAC International, 2003, vol 86 no 22, pp 412-431.

2. Schenck, F.J., SPE Cleanup and the Analysis of PPB Levels of Pesticides in Fruits and Vegetables. Florida Pesticide Residue Workshop, 2002.

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26214

QuEChERS SPE Resprep™ Q150, 50mL Centrifuge Tube, Contains 6g MgSO₄, 1.5g NaOAc, 50-pk.



26227

QuEChERS SPE Empty 50mL Centrifuge Tube, 25-pk.



26124

QuEChERS SPE Resprep™ Q250, 2mL Centrifuge Tube, Contains 150mg Magnesium Sulfate and 50mg PSA, 100-pk.



26125

QuEChERS SPE Resprep™ Q251, 2mL Centrifuge Tube, Contains 150mg Magnesium Sulfate, 50mg PSA, and 50mg C18, 100-pk.



26123

QuEChERS SPE Resprep™ Q253, 2mL Centrifuge Tube, Contains 150mg Magnesium Sulfate, 50mg PSA, and 50mg Graphitized GCB, 100-pk.



26126

QuEChERS SPE Resprep™ Q374, 15mL Centrifuge Tube, Contains 900mg Magnesium Sulfate, 300mg PSA, and 150mg GCB, 50-pk.