



biocomma



Second
Edition

Copure[®] QuEChERS

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

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m EN 15662 Method

" AOAC 2007. 01 Method

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www.biocomma.com



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.

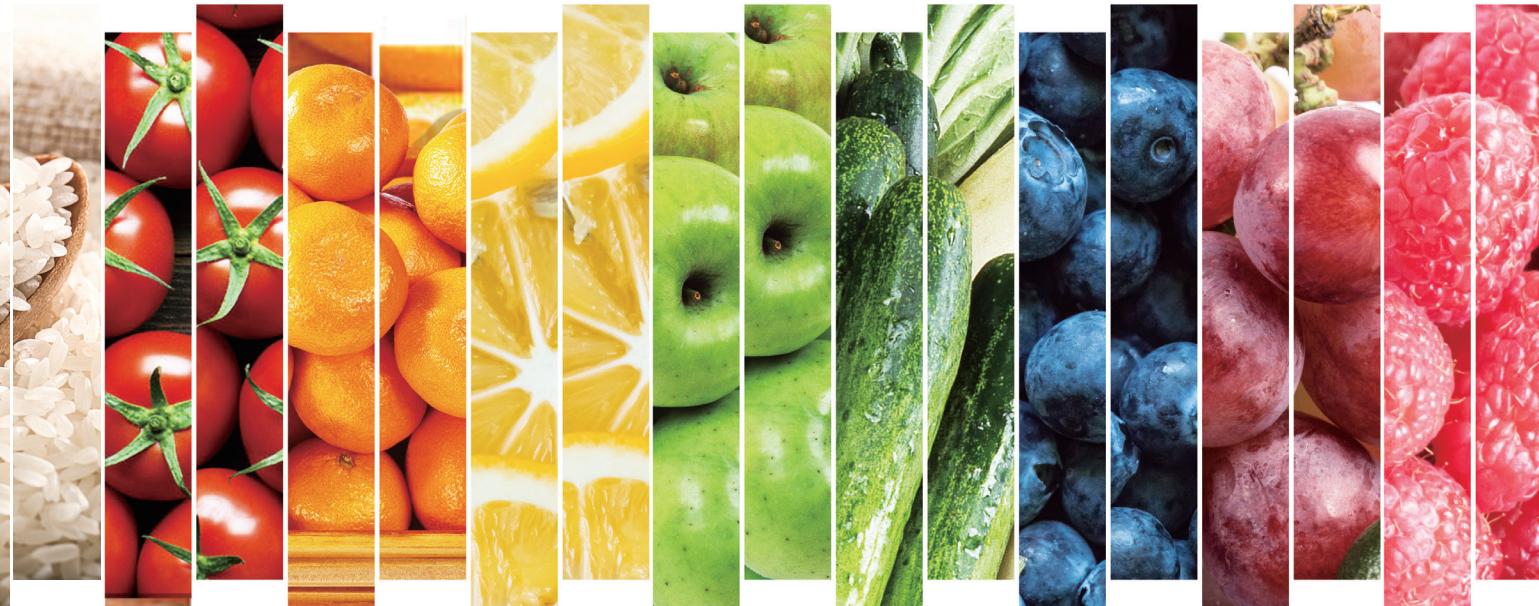


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Overview

In 2003, Michelangelo Anastassiades and Steven J Lehota 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

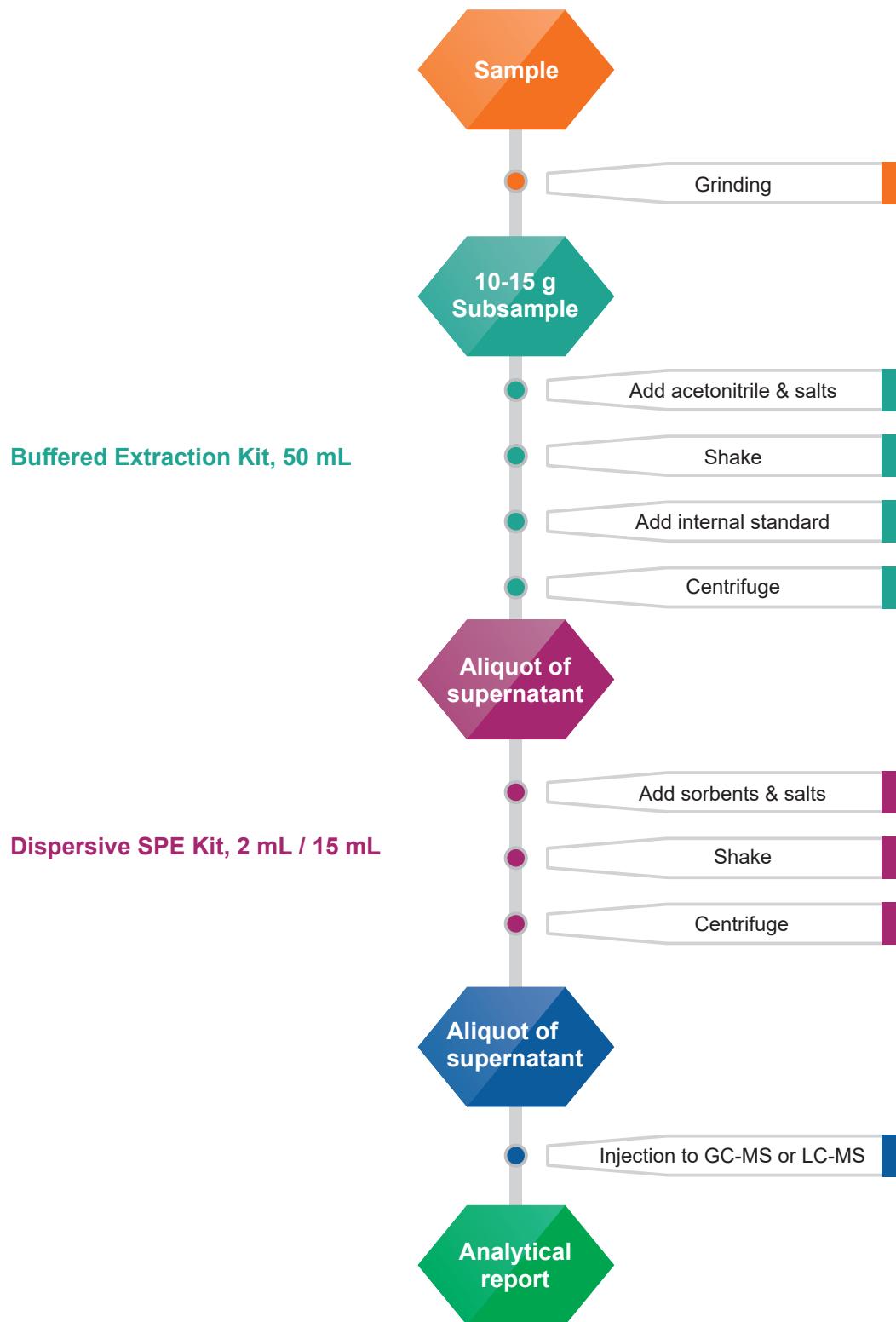
Related Methods

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, MgSO₄ 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 MgSO₄, 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	25 mg PSA, 25 mg C18, 150 mg MgSO ₄	100/Box
COQ015032H	15 mL	and vegetables with fats and waxes	150 mg PSA, 150 mg C18, 900 mg MgSO ₄	50/Box
COQ002020H	2 mL	General fruits	25 mg PSA, 2.5 mg GCB, 150 mg MgSO ₄	100/Box
COQ015020H	15 mL	and vegetables with pigments	150 mg PSA, 15 mg GCB, 900 mg MgSO ₄	50/Box
COQ002024H	2 mL	General fruits	25 mg PSA, 7.5 mg GCB, 150 mg MgSO ₄	100/Box
COQ015024H	15 mL	and vegetables with highly pigments	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	50 mg PSA, 50 mg GCB, 150 mg MgSO ₄	100/Box
COQ015036H	15 mL	and vegetables with pigments	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
MGSO4-1	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

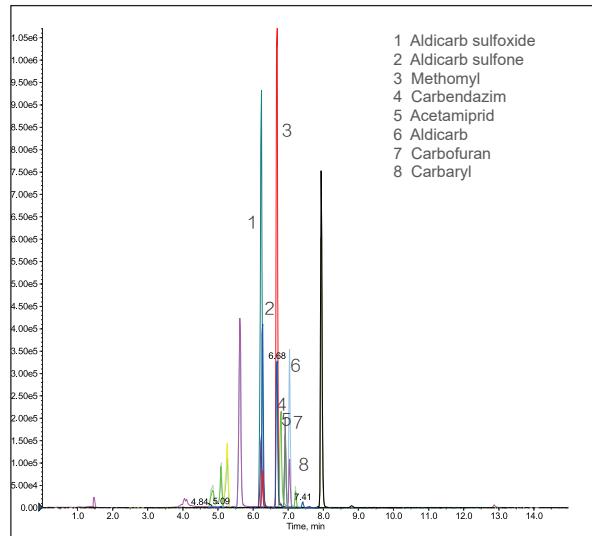
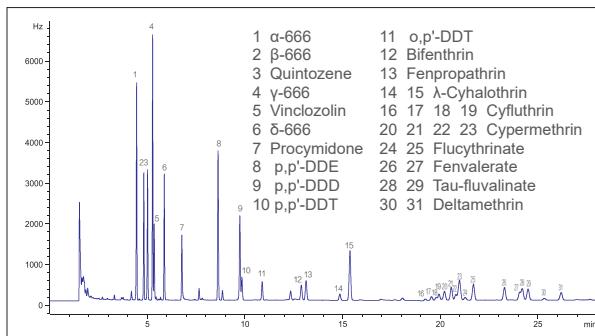
Table 4. Recoveries and relative standard deviations (RSD) of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in cucumber

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
Fenpropothrin	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



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 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

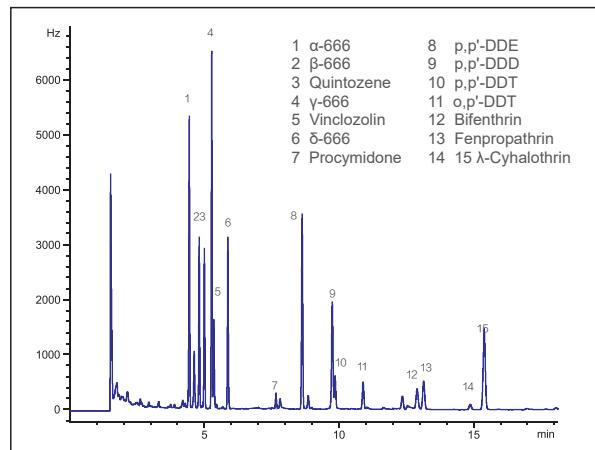


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.26 mg/kg in flowering cabbage

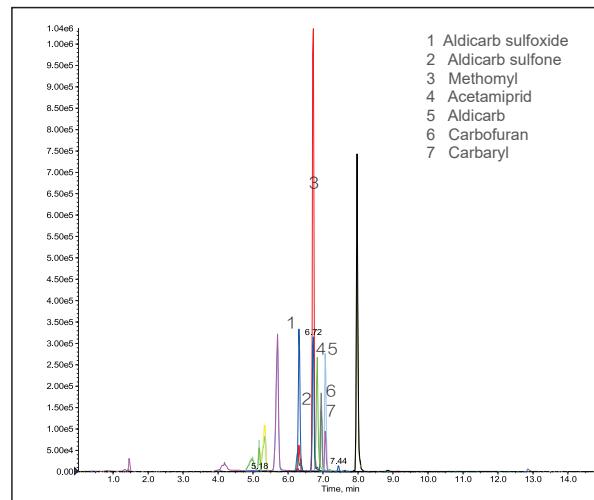


Figure 2. Chromatogram of carbamate pesticides spiked at 0.06 mg/kg in flowering cabbage

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

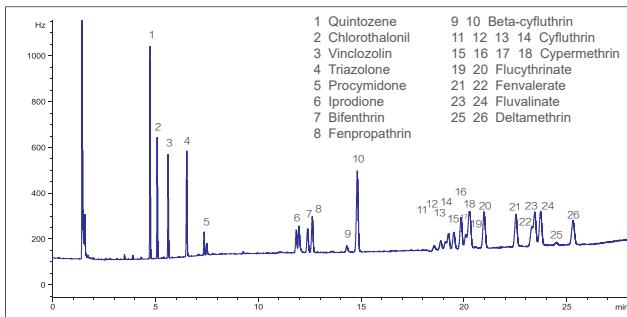


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in rice

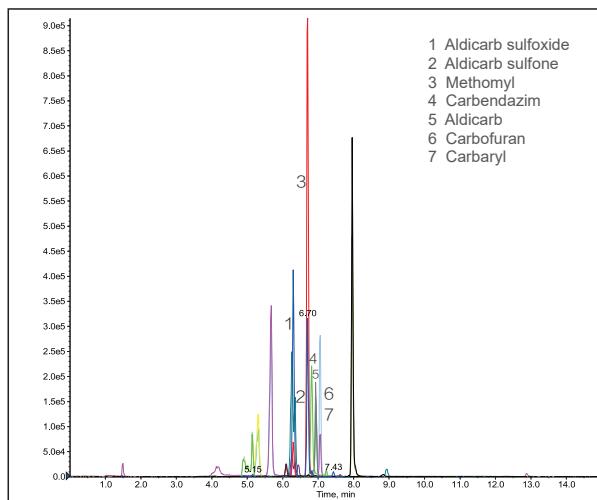


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in rice

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

Analysis of Pesticide Residues in Cucumber Using Copure® QuEChERS AOAC Kits by LC-MS/MS

Application Scope

This method applies to analyse and validate multi-residual pesticides in general 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 general vegetables and fruits(Cat. No. COQ015031H)
 biocomma® multi-tube vortexer (Cat. No. BC-1000)

Procedure

Extraction

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 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 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.

Chromatographic analysis

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
1.50	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 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

Chromatograms of spiked multi-residual pesticides in cucumber

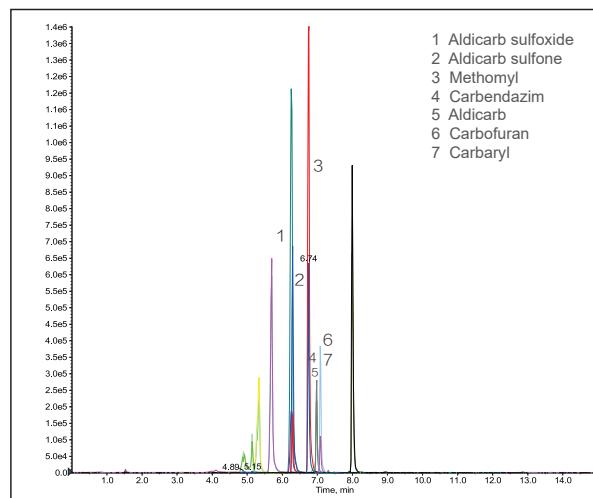


Figure 1. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in cucumber

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)	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

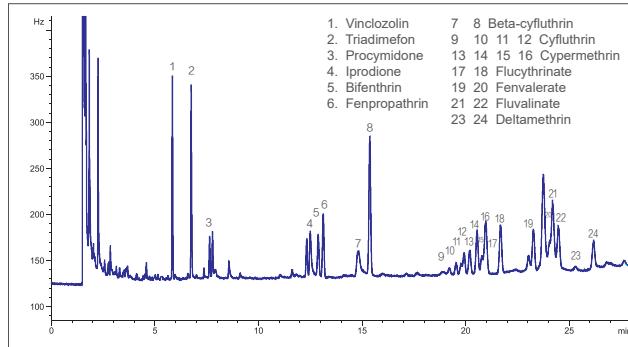


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.1 mg/kg in flowering cabbage

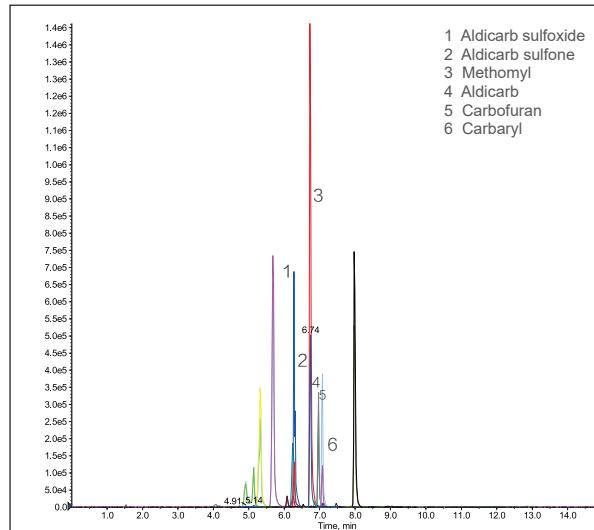


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in flowering cabbage

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

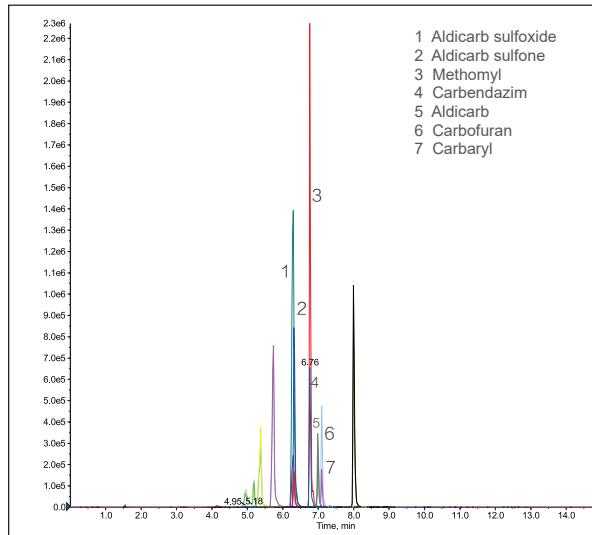
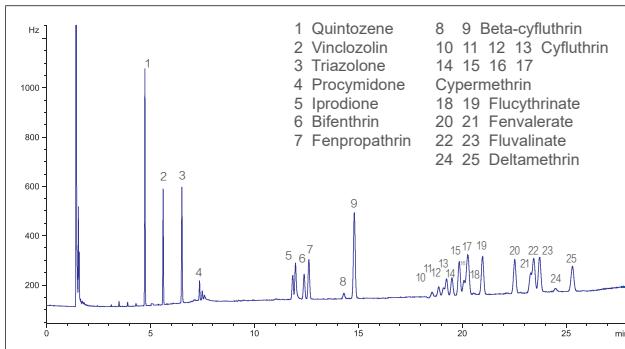
Table 4. Recoveries and relative standard deviations (RSD) of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in rice

Compound	Recoveries(%)			Average Recovery(%)	RSD(%)
	1	2	3		
Quintozone	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



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
Iprodione	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

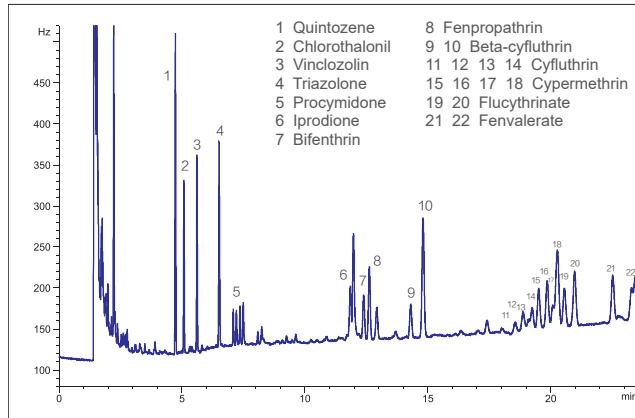


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.1 mg/kg in eggplant

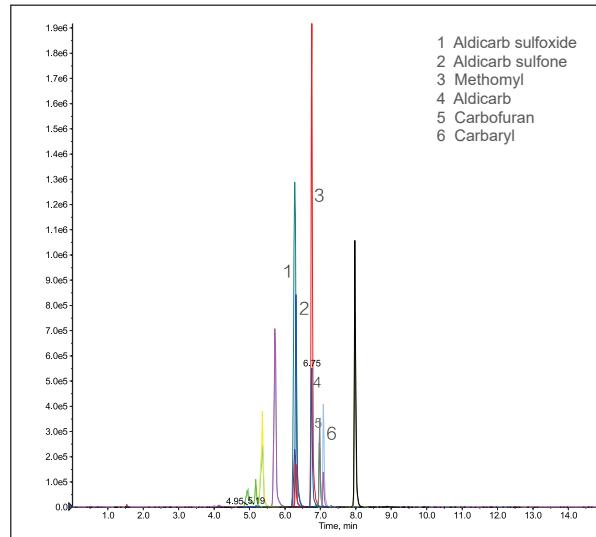


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in eggplant

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

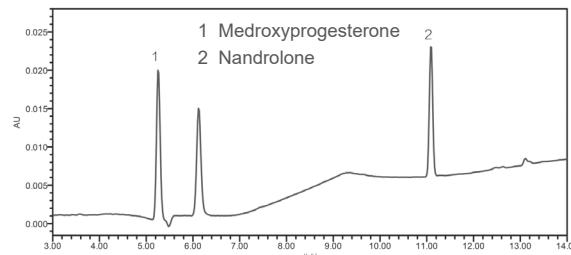


Figure 1. Chromatogram of steroids spiked at 0.5 mg/kg in pork

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

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

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

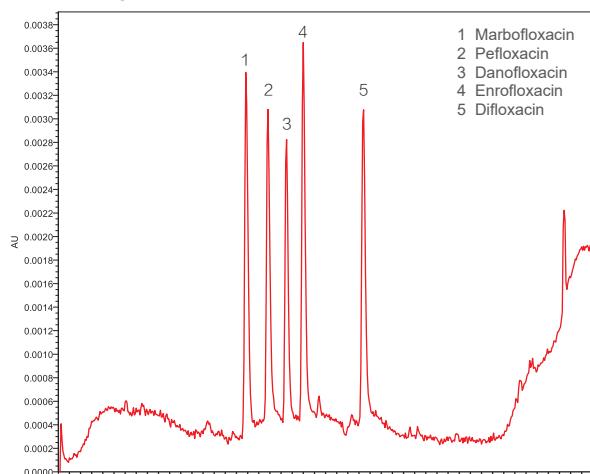


Figure 1. Chromatogram of quinolones spiked at 0.8 mg/kg in pork

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)

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

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 chloramphenicols 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

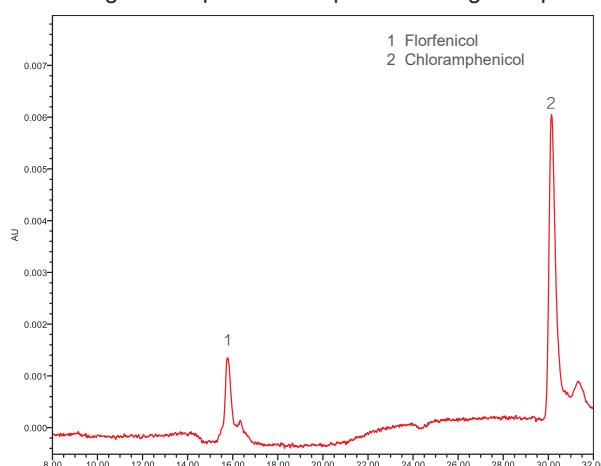


Figure 1. Chromatogram of chloramphenicols analogue spiked at 5.0 mg/kg in pork

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 1min, centrifuge for 5 min at 5000r/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

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

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

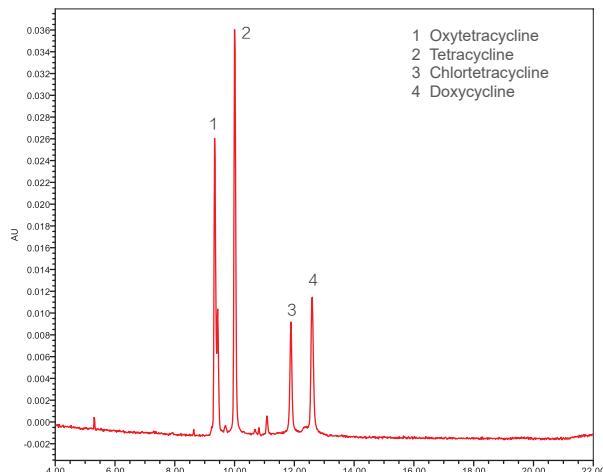


Figure 1. Chromatograms of tetracyclines spiked at 1.0 mg/kg in pork



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