biocomma

Better Filter & Better Sample Prep & Better Bioprocess

Chromatography Mass Spectrometry Sample Preparations Application Manual

SPE/SLE/QuEChERS/Immunoaffinity Column/Multi-Functional Purification Column

Food Safety

Textile Inspection

Environmental Monitoring

Company Profile

Biocomma, established in 2006 with its headquarters in Shenzhen, is dedicated to the research, production, and distribution of life science and medical health products. Operating in over 50 countries and regions, the company offers sample preparation solutions for food and clinical testing, including filtration consumables, chromatography consumables, and microbial culture media.

Biocomma also provides products such as filters, swabs, reagent bottles, sterile buffers, and culture media for life science research and manufacturing companies. Our mission is to contribute to a healthier and better world.


Certificate


Certificate


Certificate

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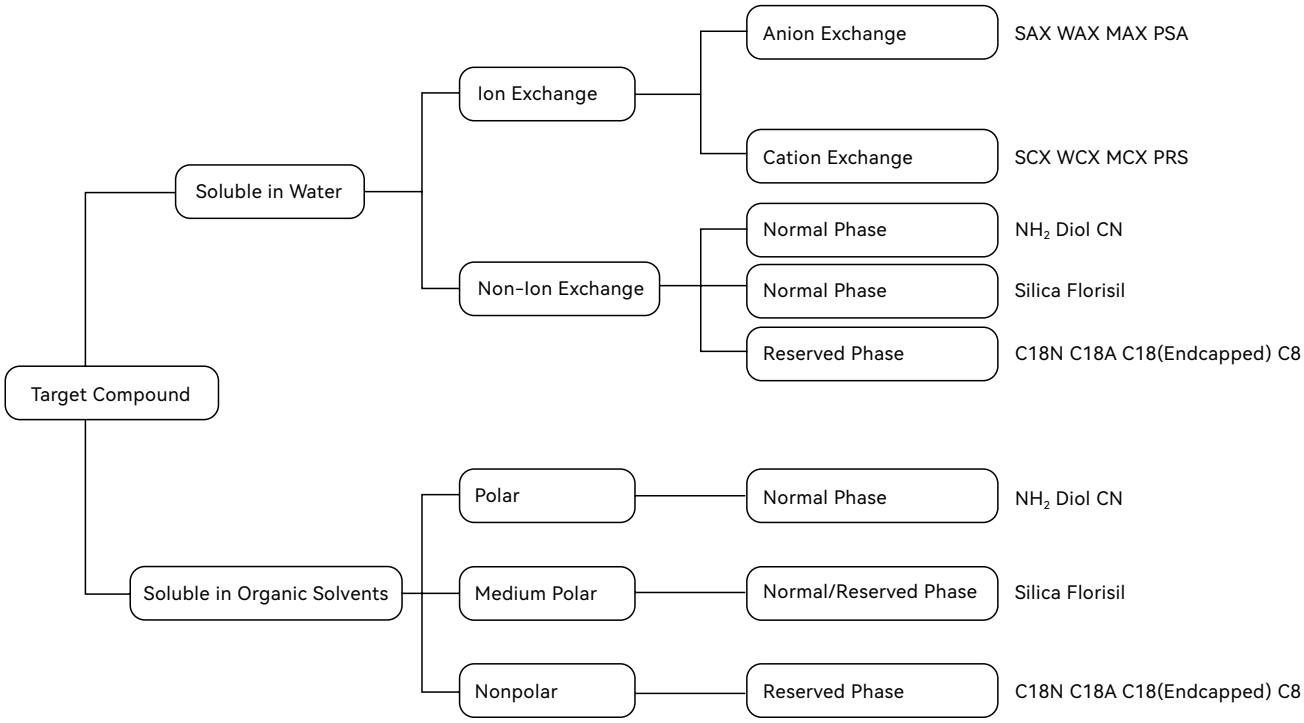
biocomma® Copure® Solid Phase Extraction Cartridges

Overview

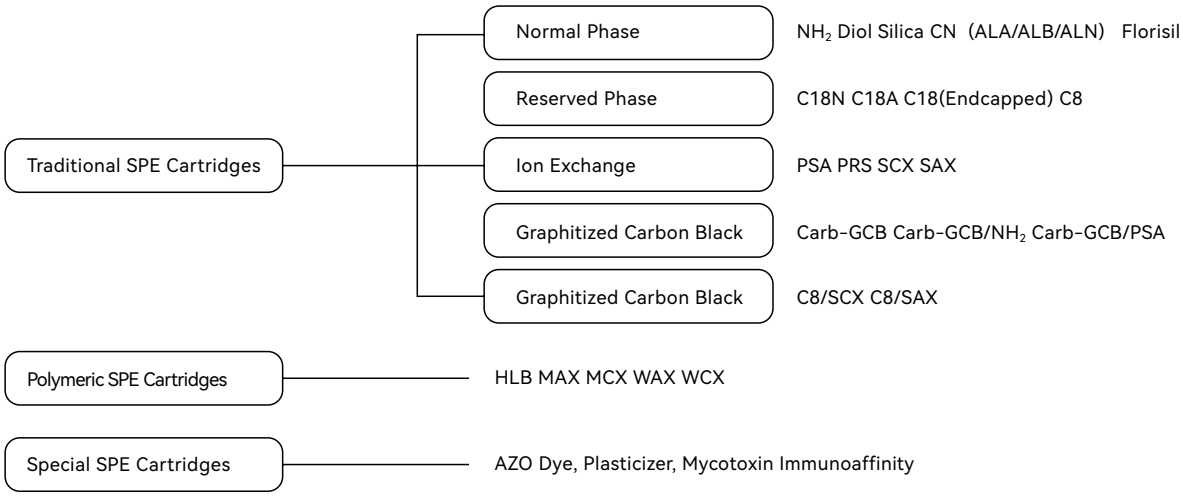
Solid Phase Extraction (SPE) is based on the principles of liquid-solid chromatography. The technology involves the selective adsorption and desorption of analytes, enabling the enrichment, separation, and purification of samples. SPE is a physical extraction process that involves both liquid and solid phases. During the solid-phase extraction process, the adsorbent has a greater affinity for the target compounds than the sample matrix. As the sample passes through the SPE cartridge, the target compounds are adsorbed onto the solid surface, while other components pass through the column with the sample matrix. Subsequently, the target compounds are eluted and collected using an appropriate solvent for subsequent chromatographic analysis. In recent years, SPE has found increasingly widespread applications as a sample preparation technique in laboratories.

SPE Cartridge Sorbent Selection Guide

Choose the appropriate sorbent based on the differences between the target compounds and interferents, such as polarity, molecular weight, and pKa values.



SPE Cartridge Category



Procedures

The procedures may vary slightly depending on retaining target compounds or impurities.

1.Retaining Target Compounds (Figure 1):

1)Activation: remove impurities from the column, activate the sorbent, and create a suitable solvent environment.

2)Loading: dissolve the sample in a specific solvent, transfer it into the column, and retain the components on the sorbent.

3)Washing: remove interfering substances to the maximum extent.

4)Elution: elute the targets by a small volume of solvent and collect them.

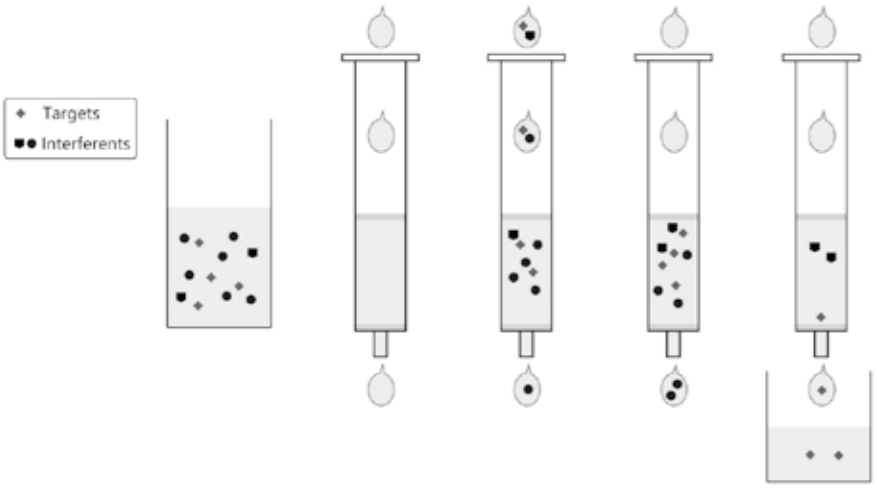


Figure 1. Sample 1.Activation 2. Loading 3.Washing 4.Elution

2.Retaining Impurities (Figure 2):

1)Activation: remove impurities from the column, activate the sorbent, and create a suitable solvent environment.

2)Loading: dissolve the sample in a specific solvent, transfer it into the column, At this stage, most target compounds will flow out, while impurities are retained on the column. Therefore, collection should begin at this step.

3)Washing: remove interfering substances to the maximum extent.。

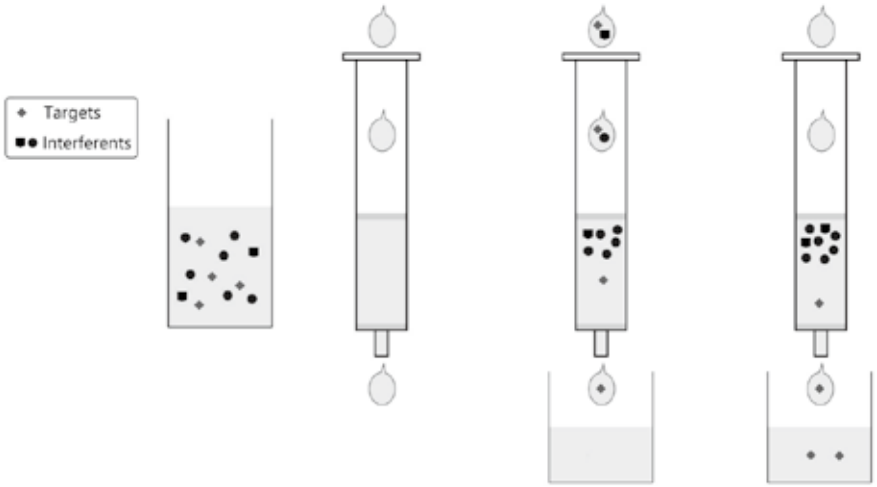


Figure 2. Sample 1.Activation 2. Loading 3.Washing

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 15662-2018: 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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

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 $MgSO_4$, 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 $MgSO_4$ 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 H_2O (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

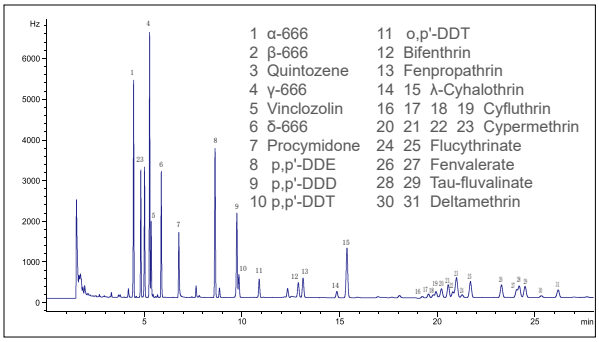


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in cucumber

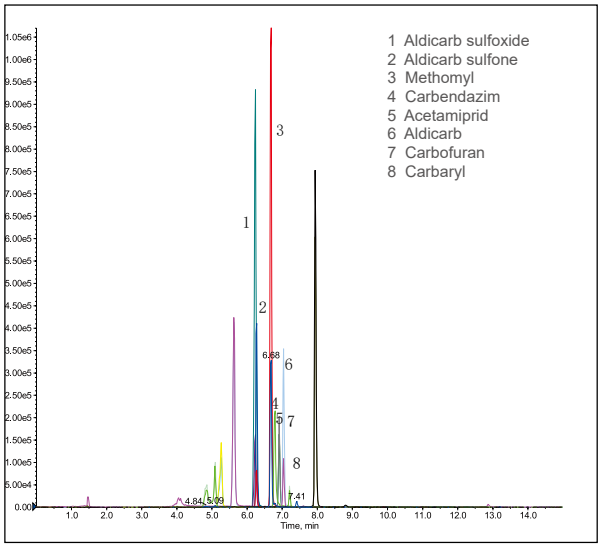


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in cucumber

Order Information

Cat.#	Description	Qty.
COQ050010H	Extraction Salts (4 g $MgSO_4$, 1 g NaCl, 1 g Trisodium Citrate, 0.5 g Disodium Citrate), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ015022H	900 mg $MgSO_4$, 150 mg PSA, 15 mL Tube	50 Pcs/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

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 15662-2018: 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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

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 $MgSO_4$, 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 $MgSO_4$, 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 H_2O (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 onditions 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

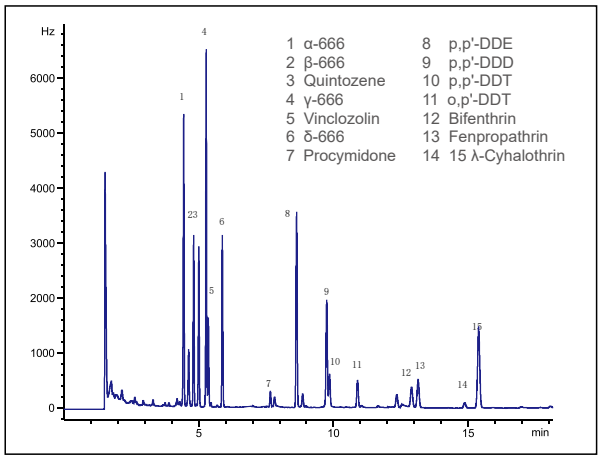


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.26 mg/kg in flowering cabbager

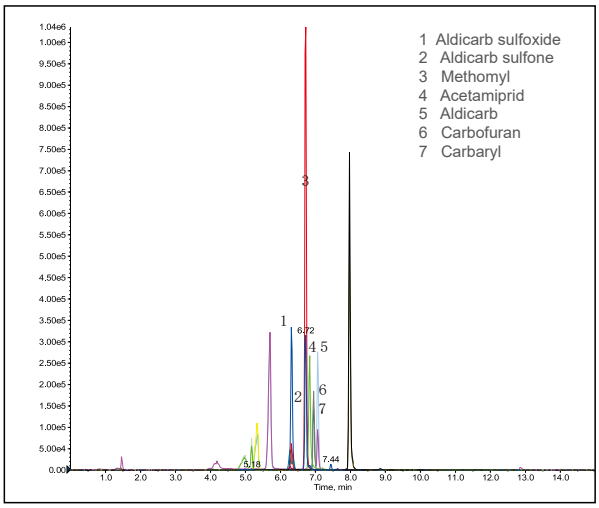


Figure 2. Chromatogram of carbamate pesticides spiked at 0.06 mg/kg in flowering cabbage

Order Information

Cat.#	Description	Qty.
COQ050010H	Extraction Salts (4 g $MgSO_4$, 1 g NaCl, 1 g Trisodium Citrate, 0.5 g Disodium Citrate), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ015024H	900 mg $MgSO_4$, 150 mg PSA, 45 mg GCB, 15 mL Tube	50 Pcs/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

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 15662-2018: 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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

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 $MgSO_4$, 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 $MgSO_4$, 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 H_2O (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

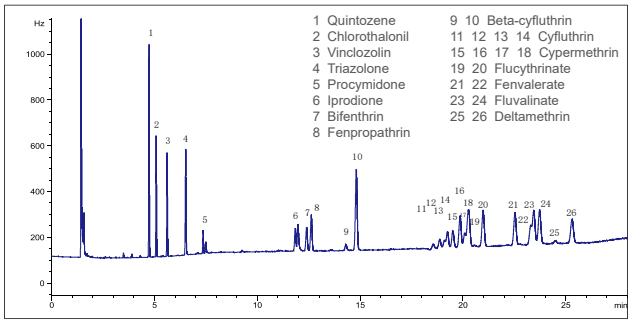


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in rice

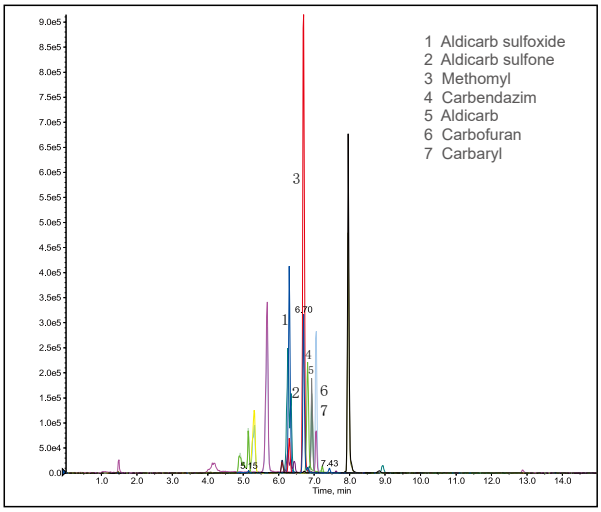


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in rice

Order Information

Cat.#	Description	Qty.
COQ050010H	Extraction Salts (4 g $MgSO_4$, 1 g NaCl, 1 g Trisodium Citrate, 0.5 g Disodium Citrate), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ015032H	900 mg $MgSO_4$, 150 mg PSA, 150 mg C18, 15 mL Tube	50 Pcs/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

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 $MgSO_4$ 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.

Dispersive SPE cleanup

Transfer 8 mL of the upper acetonitrile layer into a QuEChERS AOAC dispersive SPE 15 mL tube containing 1.2 g $MgSO_4$ 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 H_2O (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

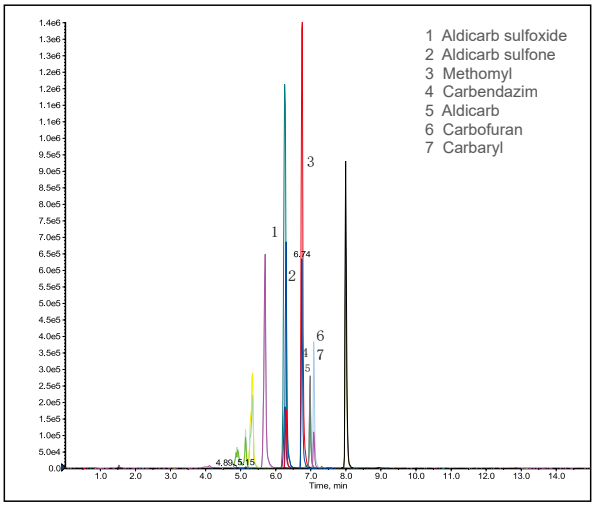


Figure 1. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in cucumber

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	Extraction Salts (6 g $MgSO_4$, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
COQ015031H	1200 mg $MgSO_4$, 400 mg PSA, 15 mL Tube	50/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	Set/Carton

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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

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 $MgSO_4$ 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.

Dispersive SPE cleanup

Transfer 3 mL Toluene into a QuEChERS AOAC dispersive SPE 15 mL tube containing 1.2 g $MgSO_4$, 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 H_2O (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

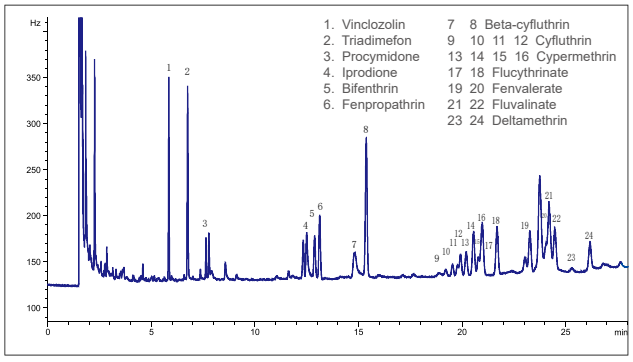


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.1 mg/kg in flowering cabbage

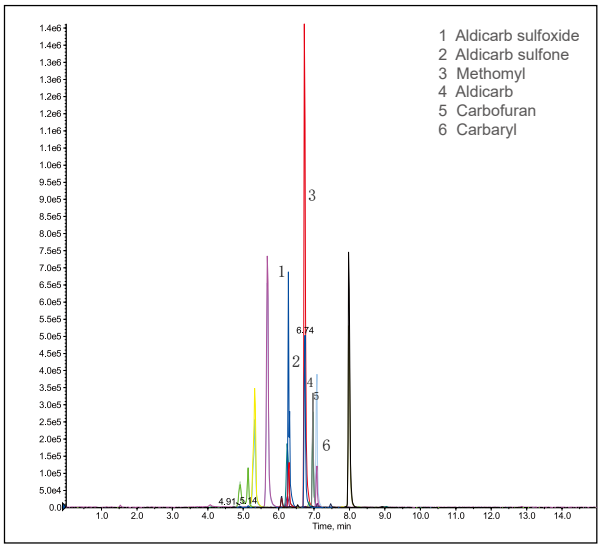


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in flowering cabbage

Order Information

Cat.#	Description	Qty.
COQ050020H	Extraction Salts (6 g $MgSO_4$, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
COQ015036H	1200 mg $MgSO_4$, 400 mg PSA, 400 mg GCB, 15 mL Tube	50 Pcs/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

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 $MgSO_4$ 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.

Dispersive SPE cleanup

Transfer 8 mL of the upper acetonitrile layer into a QuEChERS AOAC dispersive SPE 15 mL tube containing 1.2 g $MgSO_4$,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 H_2O (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

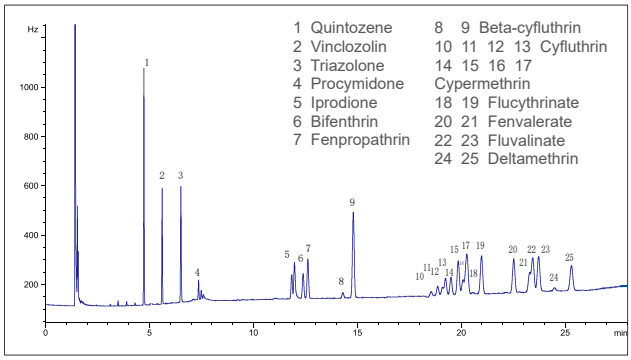


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.2 mg/kg in rice

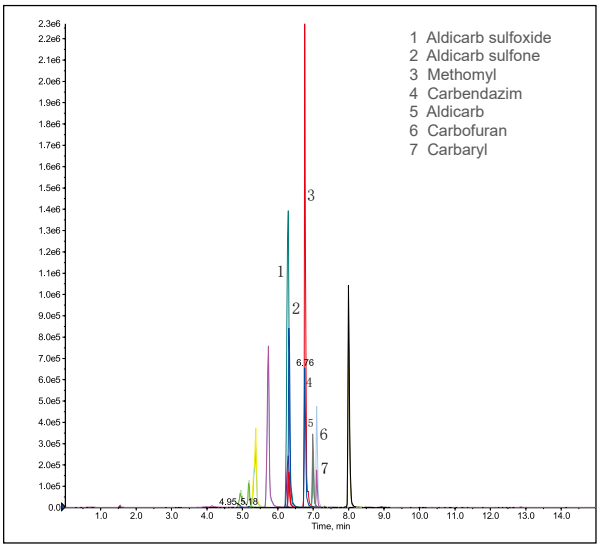


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in rice

Order Information

Cat.#	Description	Qty.
COQ050020H	Extraction Salts (6 g $MgSO_4$, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
COQ015031H	1200 mg $MgSO_4$, 400 mg PSA, 15 mL Tube	50 Pcs/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

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 forfruits and vegetables with fats and pigment(Cat. No. COQ015040H)

biocomma® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

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 $MgSO_4$ 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.

Dispersive SPE cleanup

Transfer 3 mL toluene into a QuEChERS AOAC dispersive SPE 15 mL tube containing 1.2 g $MgSO_4$, 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 dipersive 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 H_2O (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

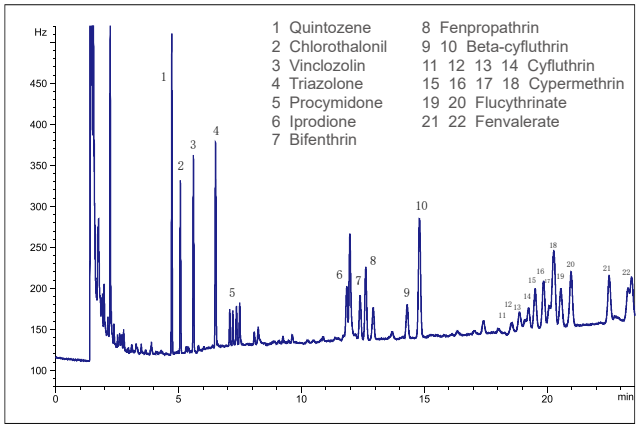


Figure 1. Chromatogram of organochlorine and pyrethroid pesticides spiked at 0.1 mg/kg in eggplant

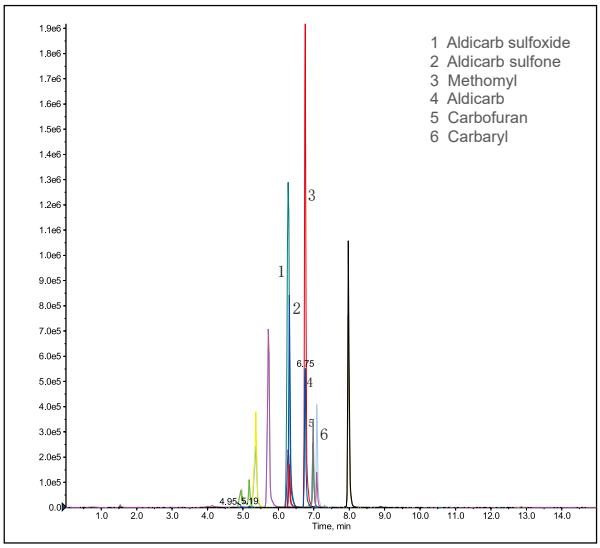


Figure 2. Chromatogram of carbamate pesticides spiked at 0.05 mg/kg in eggplant

Order Information

Cat.#	Description	Qty.
COQ050020H	Extraction Salts (6 g $MgSO_4$, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
COQ015040H	1200 mg $MgSO_4$, 400 mg PSA, 400 mg C18, 400 mg GCB, 15 mL Tube	50 Pcs/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

Multi-Residue of Pesticides in Dark-Colored Fruits, Vegetables, and Tea Using EN and AOAC Kits by LC-MS/MS (Copure® QuEChERS)

Experiment

Extraction and purification of dark-colored fruits and vegetables

Weigh 10.00 g of the sample into a 50 ml centrifuge tube. Add 10 ml of acetonitrile and a ceramic homogenizer, and shake vigorously for 1 min. Add 4 g of $MgSO_4$, 1 g of NaCl, 1 g of trisodium citrate, and 0.5 g of disodium citrate(Cat.No.COQ050010CH). After vigorous shaking for 1 min, centrifuge at 5000 r/min for 5 min. Quantitatively transfer 6 mL of the supernatant to a centrifuge tube containing drying agents and purification materials (900 mg anhydrous $MgSO_4$, 150 mg PSA, and 15 mg GCB)(Cat.No.COQ015020H) . Vortex for 1 min. Centrifuge at 7500 r/min for 2 min. Filter the supernatant through a microporous membrane for further analysis.

Extraction and purification of tea

Weigh 2.00 g of tea into a 50 mL centrifuge tube. Add 10 mL of water. Vortex to mix well, and let it stand for 30 min. Add 15 mL of acetonitrile-acetic acid solution and a ceramic homogenizer, and shake vigorously for 1 min. Add 6 g of $MgSO_4$ and 1.5 g of sodium acetate. (Cat. No.COQ050020CH)After vigorous shaking for 1 min, centrifuge at 7500 r/min for 5 min. Quantitatively transfer 8 mL of the supernatant to a centrifuge tube containing drying agents and purification materials (1200 mg of $MgSO_4$, 400 mg of C18, 400 mg of PSA, and 200 mg of GCB) (Cat.No.COQ015047H). Vortex for 1 min. Centrifuge at 7500 r/min for 2 min. Filter the supernatant through a microporous membrane for further analysis. Filter the supernatant through a microporous membrane for further analysis.

Extraction of Standard Curve Solution

Prepare blank sample in the same procedures. Add mixed standard to dilute to the concentration of 0.002 mg/L, 0.005 mg/L, 0.01 mg/L, 0.02 mg/L, 0.05 mg/L and 0.1 mg/L. Based on the instrument's performance and testing requirements, select at least 5 concentrations for LC-MS determination. Use the peak area of the mass chromatogram for pesticide quantification as the vertical axis and the corresponding matrix-matched standard working solution's mass concentration as the horizontal axis to plot the matrix-matched standard working curve.

1.Chromatographic conditions

Equipment: Tandem Quadrupole Linear Ion Trap Mass Spectrometer AB SCIEXQTRAP4500

Chromatographic column: ACQUITY UPLC BEH C18 Column 2.150mm, 1.7m

Column temperature: 40 ℃

Injection volume: 1 μL

Table 1. Gradient Elution Program

Time(min)	Flow rate (mL/min)	5 mmol/L Ammonium acetate solution (0.01% formic acid)	Methanol (0.01%formic acid)
Initial	0.300	90.0	10.0
1.50	0.300	50.0	50.0
3.00	0.300	10.0	90.0
5.00	0.300	10.0	90.0
5.10	0.300	90.0	10.0
6.00	0.300	90.0	10.0

2.Mass Spectrometry Conditions

Detection mode: MRM

Table 2. Ion Source Control Conditions

Ionization Mode	ESI+
Curtain Gas (Cur)	40.0 Psi
Collision Gas (Cad)	9 Psi
Ionspray Voltage (IS)	5500.0 V
Temperature (TEM)	400.0 ℃
Ion Source Gas 1(Gs1)	50 Psi
Ion Source Gas 1(Gs2)	40 Psi

Table 3. Targes Characteristic Ions (*Quantifier Ion)

Targets	Parent Ion (m/z)	Daughter Ion (m/z)	Retention Time (min)	DP (V)	CE (V)
Acetamiprid	223	126*	1.75	65	28
	223	99	1.75	65	60
Aldicarb	208	116*	2.02	20	11
	208	89	2.02	20	25
Aldicarb Sulfone	240.1	148*	1.29	30	17
	240.1	166	1.29	30	16
Aldicarb Sulfoxide	207	132*	1.19	55	9
	207	89	1.19	55	20
Arazine	228.1	186.2*	2.68	90	25
	228.1	96	2.68	90	35
Saponin	368	199*	3.03	80	19
	368	125	3.03	80	45

Instrumental Conditions

Avermectin	890.5	305*	3.82	60	33
	890.5	567.3	3.82	60	20
Acytophos	318	132*	2.59	58	22
	318	125	2.58	58	33
Azoxystrobin	404.1	372*	2.59	80	20
	404.1	344.1	2.59	80	34
Herbicide	272	198*	2.65	65	20
	272	170	2.65	65	33
Benzocarb	224.1	167*	2.21	64	12
	224.1	109	2.21	64	23
Benefate	364.1	199*	3.1	20	12
	364.1	105	3.1	20	36
Boscalid	343	307*	2.71	100	28
	343	140	2.71	100	27
Ethyrimol Sulfonate	317	166*	2.95	90	31
	317	210	2.95	90	33
Buprofezin	306.2	201.1*	3.34	60	17
	306.2	116.2	3.34	60	22
Carbaryl	202.1	145*	2.3	56	15
	202.1	127	2.3	56	40
Carbendazim	192	160*	1.88	80	25
	192	132	1.87	80	41
Kebowei	222.1	165*	2.2	70	16
	222.1	123.1	2.2	70	29
3-Hydroxy Carbofuran	238	181*	1.73	70	16
	238	163	1.73	70	18
Chlorantran-iliprole	483.9	452.9*	2.54	45	25
	483.9	285.9	2.54	45	19
Chlorfluazuron	540	383*	3.59	70	30
	542	385	3.59	70	30
Cyclomin	222	104*	1.8	90	28
	222	92	1.8	90	35
Green Myron	213.1	72.1*	2.44	60	36
	213.1	46.1	2.44	60	33
Cyclofenozide	395.2	175*	2.82	80	18
	395.2	339	2.82	80	9
Tetramethazine	303	138*	3.14	51	21
	303	102	3.14	51	47
Clomazone	240.1	125*	2.6	73	28
	240.1	89.1	2.61	73	65
Clothianidin	250	169.1*	1.68	35	17
	250	132	1.68	35	21
Cyanazine	241.1	214.1*	2.1	60	24
	241.1	104.1	2.09	60	40
Cyantraniliprole	475	286*	2.29	30	18
	475	444	2.29	30	27
Cyflumetate	465.2	173*	3.19	40	30
	465.2	249.1	3.19	40	19

Cymoxanil	199.1	128*	1.85	53	12
	199.1	111	1.85	53	24
Cyprodinil	226.1	93*	3.07	96	50
	226.1	108.1	3.07	96	35
Diflubenzuron	311	158*	2.98	45	20
	311	141.2	2.99	45	49
Diflufenamide	395.1	266*	3.18	30	34
	395.1	246	3.18	30	46
Refined Dimethenamid	276.1	244.1*	2.7	60	20
	276.1	168.1	2.7	60	33
Kysastrobin	327.2	205*	3	64	14
	327.2	238.1	2.98	64	15
Dinotefuran	203.1	129*	1.23	35	16
	203.1	157	1.22	35	11
Epoxiconazole	330	121*	2.89	85	55
	330	101	2.88	85	70
Fenoxazone	392	331*	3.04	45	12
	392	238	3.04	45	24
Enoxastrobin	434.1	171*	3.21	60	36
	434.1	212	3.2	60	21
Fenamiphos	304.2	217.1*	2.93	90	31
	304.2	202	2.93	90	47
Fenamifos Sulfone	336	308*	2.22	90	21
	336	266	2.22	90	29
Fenamiphos Sulfoxide	320.1	233*	2.17	100	32
	320.1	171	2.17	100	30
Fenazazafen	307.2	161*	3.79	30	22
	307.2	131.1	3.79	30	60
Forchlorfenuron	248.1	129*	2.54	50	23
	248.1	93	2.54	50	47
Anthion	258	199*	2.08	20	12
	258	125	2.08	20	32
Thiazophos	284	228*	2.35	65	14
	284	104	2.35	65	30
Furosecarb	383.2	195*	3.28	85	25
	383.2	252	3.28	85	17
Heptenylphos	251	127*	2.52	65	20
	251	125	2.52	65	18
Hexythiazox	353	228*	3.43	56	21
	353	168	3.43	56	33
Imidacloprid	256.1	175*	1.64	45	27
	256.1	209	1.64	45	22
Chlorothialine	262.1	181.1*	1.71	57	19
	262.1	122	1.71	57	36
Indoxacarb	528	293*	3.13	50	18
	528	249	3.14	50	23
Chlorazophos	316	164*	2.8	70	23
	316	122	2.8	70	35

Isoprocarb	194	95*	2.46	57	19
	194	137	2.45	57	12
Methiocarb	226.1	169.2*	2.72	61	13
	226.1	121.1	2.72	61	23
Methiocarb Sulfoxide	242	185*	1.65	37	18
	242	122	1.65	37	38
Methomyl	163	88*	1.41	38	12
	163	106	1.41	38	14
Methoprene	279	191*	3.89	80	11
	279	237	3.89	80	9
Methoxy-fenozide	369.2	149.1*	2.74	70	21
	369.2	133	2.74	70	15
Metolachlor	284.1	251.9*	2.92	60	19
	284.1	176	2.92	60	33
Methiocarb	166.1	109*	2.11	50	18
	166.1	94.2	2.11	50	40
Metrafenone	409	209.1*	3.14	71	21
	409	227.1	3.14	71	27
Azimidone	215.1	187.1*	2.23	60	24
	215.1	84.1	2.23	60	28
Memethaphos	225	127*	1.89	55	21
	225	193	1.89	55	9
Picoxystrobin	368.1	205.1*	2.92	54	13
	368.1	145.1	2.92	54	27
Prochloraz	376.2	308*	3.11	20	15
	376.2	266	3.11	20	22
Prochloraz-Deaminoimi-dazole	325	282*	3.12	90	21
	327	284	3.12	90	23
Prochloraz-Deimidazole Carboxamide	353	308*	3.1	80	19
	355	310	3.1	80	20
Pyraclostrobin	388.1	194*	3.06	50	18
	388.1	163	3.06	50	36
Metazachlor	413	339*	3	40	28
	413	253	3	40	45
Pyraclostrobin	382.1	194.1*	2.93	30	16
	382.1	163	2.93	30	34
Pyrazosulfuron-Methyl	415.1	182*	2.79	30	25
	415.1	139	2.79	30	59
Pyrethrin I	329.2	161.1*	3.52	75	13
	329.2	133.1	3.53	75	22
Saflufenacil	610.2	413.1*	3.24	30	17
	610.2	180.1	3.24	30	48
Pyrimethanil	200	183*	2.71	30	33
	200	168	2.72	30	40

Spinosad D	746.4	142*	3.14	110	34
	746.4	98	3.14	110	101
Spirofen	411.2	313.1*	3.56	83	15
	411.2	71.1	3.56	83	30
Spiromethin	388	273*	3.43	65	23
	388	255	3.44	65	38
Spirotetramat	374	302*	2.81	87	24
	374	330	2.8	87	20
Tebufenozide	353.1	133.1*	2.93	35	24
	353.1	297.1	2.93	35	11
Buthiuron	229.1	172*	2.25	30	23
	229.1	116.1	2.25	30	35
Flufenuron	381.1	158.2*	3.41	71	23
	381.1	141.2	3.41	71	53
Terbuphos	289.1	103.1*	3.32	53	13
	289.1	233.2	3.33	53	9
Terbufos Sulfone	321	97*	2.62	75	57
	321	115	2.63	75	39
Terbuthion Sulfoxide	305	187*	2.62	57	20
	305	131	2.62	57	38
Thiamethoxam	292	211*	1.47	30	16
	292	181	1.47	30	30
Benzamid	336	186.9*	3.1	90	31
	336	204	3.1	90	23
Fenflux	422	366.1*	3.54	90	23
	422	135.1	3.54	90	43
Fludioxonil	266.1	229*	2.73	30	17
	266.1	158	2.74	30	46
Flufenacet	364	194*	2.84	70	16
	364	152	2.84	70	27
Flubenzuron	489	158.1*	3.45	71	27
	489	141.2	3.45	71	65
Fluopyram	382.9	172.9*	2.72	50	35
	382.9	364.9	2.73	50	23
Fluopyram	397	207.9*	2.8	60	30
	397	172.9	2.8	60	40
Acifluorfen	465.1	344*	3.16	40	19
	465.1	223	3.17	40	43
Furutrione	334.1	247.1*	2.64	50	30
	334.1	303	2.64	50	20
Flusilazole	316	247.1*	2.93	50	26
	316	165	2.92	50	37
Etoproxil	394.2	177.1*	4.13	30	19
	394.2	107.1	4.13	30	59
Fenazole	302.1	123*	2.42	69	39
	302.1	109	2.42	69	43

Flufenapy-ramide	382.1	362*	2.73	90	20
	382.1	342.1	2.73	90	28

Results

Table 4. Spiked Multi-Residue of Pesticide in Bok Choy at 0.0200 mg/kg

Targets	Recovery Rate (%)			Average Recovery (%)	RSD (%)
	1	2	3		
Acetamiprid	89.5	88.0	86.0	87.8	2.00
Aldicarb	85.5	87.0	80.0	84.2	4.38
Aldicarb Sulfone	92.5	90.5	89.0	90.7	1.94
Aldicarb Sulfoxide	91.0	88.0	83.5	87.5	4.31
Arazine	90.0	83.0	86.5	86.5	4.05
Saponin	94.0	84.5	79.5	86.0	8.56
Avermectin	93.5	80.5	75.5	83.2	11.2
Acytophos	95.0	88.5	88.0	90.5	4.32
Azoxystrobin	92.0	88.5	86.5	89.0	3.13
Herbicide	86.0	83.0	86.5	85.2	2.22
Benzocarb	90.0	89.0	84.5	87.8	3.34
Benefate	93.5	92.5	82.0	89.3	7.13
Boscalid	92.5	90.0	85.0	89.2	4.28
Ethyrimol Sulfonate	97.0	95.5	81.5	91.3	9.36
Buprofezin	88.0	87.5	83.0	86.2	3.19
Carbaryl	90.0	88.5	87.5	88.7	1.42
Carbendazim	92.0	90.0	93.5	91.8	1.91
Kebowei	116	118	110	115	3.62
3-Hydroxy Carbofuran	87.5	88.0	87.5	87.7	0.329
Chlorantraniliprole	98.0	95.5	98.5	97.3	1.65
Chlorfluazuron	97.5	85.5	87.5	90.2	7.13
Cyclomin	88.0	85.0	86.5	86.5	1.73
Green Myron	89.0	90.0	92.0	90.3	1.69
Cyclofenozide	91.0	90.0	81.0	87.3	6.31
Tetramethazine	93.5	80.0	91.5	88.3	8.25
Clomazone	91.0	91.0	88.5	90.2	1.60
Clothianidin	75.0	81.5	78.0	78.2	4.16
Cyanazine	98.0	92.0	96.5	95.5	3.27
Cyantraniliprole	86.5	79.5	87.5	84.5	5.16
Cyflumetate	79.5	83.0	81.0	81.2	2.16
Cymoxanil	88.5	87.0	88.5	88.0	0.984
Cyprodinil	88.0	75.0	81.5	81.5	7.98
Diflubenzuron	82.0	93.5	83.5	86.3	7.24
Diflufenamide	90.5	91.0	91.0	90.8	0.318
Refined Dimethenamid	99.5	91.5	95.5	95.5	4.19
Kysastrobin	92.0	84.0	87.5	87.8	4.57
Dinotefuran	88.0	89.5	85.5	87.7	2.30
Epoxiconazole	88.0	87.0	84.0	86.3	2.41
Fenoxazone	96.0	88.5	98.5	94.3	5.52
Enoxastrobin	86.5	86.5	76.5	83.2	6.94
Fenamiphos	87.5	83.5	82.0	84.3	3.37
Fenamifos Sulfone	100	96.0	90.5	95.5	4.99
Fenamiphos Sulfoxide	93.0	89.0	90.0	90.7	2.30
Fenazazafen	82.0	79.5	78.5	80.0	2.25
Forchlorfenuron	85.0	85.0	80.5	83.5	3.11
Anthion	73.0	71.5	80.5	75.0	6.43
Thiazophos	89.5	85.5	85.0	86.7	2.84
Furosecarb	90.0	80.5	92.0	87.5	7.02
Heptenylphos	89.5	92.0	86.5	89.3	3.08
Hexythiazox	79.0	78.5	82.5	80.0	2.72
Imidacloprid	83.0	93.5	82.0	86.2	7.39
Chlorothialine	79.0	83.0	80.0	80.7	2.58
Indoxacarb	116	98.5	112	109	8.41
Chlorazophos	95.0	91.5	82.0	89.5	7.52
Isoprocarb	94.0	88.0	89.5	90.5	3.45
Methiocarb	103	98.0	100	100	2.52
Methiocarb Sulfoxide	85.0	88.5	80.0	84.5	5.06
Methomyl	89.5	87.5	88.0	88.3	1.18
Methoprene	86.0	81.5	77.5	81.7	5.20
Methoxyfenozide	89.0	106	93.0	96.0	9.26
Metolachlor	87.5	87.0	80.0	84.8	4.94
Methiocarb	91.5	89.5	89.5	90.2	1.28
Metrafenone	83.0	87.5	90.5	87.0	4.34
Azimidone	93.0	94.5	98.5	95.3	2.98
Memethaphos	89.5	88.5	90.0	89.3	0.855
Picoxystrobin	96.5	102	92.0	96.8	5.17
Prochloraz	82.0	78.0	76.5	78.8	3.61
Prochloraz-Deaminoimidazole	87.5	76.5	83.5	82.5	6.75
Prochloraz-Deimidazole Carboxamide	91.0	100	99.5	96.8	5.23
Pyraclostrobin	99.5	93.5	96.0	96.3	3.13
Metazachlor	97.5	94.5	92.0	94.7	2.91
Pyraclostrobin	97.0	102	92.0	97.0	5.15
Pyrazosulfuron-Methyl	69.0	63.5	71.0	67.8	5.73
Pyrethrin I	77.5	84.0	76.5	79.3	5.14
Saflufenacil	83.5	90.5	78.5	84.2	7.16
Pyrimethanil	92.5	90.0	100	94.2	5.52
Spinosad D	79.0	82.0	77.5	79.5	2.88
Spirofen	80.5	73.5	74.5	76.2	4.97
Spiromethin	89.0	83.0	78.0	83.3	6.61
Spirotetramat	78.0	81.0	83.0	80.7	3.12
Tebufenozide	96.5	86.0	83.0	88.5	8.01
Buthiuron	94.5	97.5	88.0	93.3	5.21
Flufenuron	79.0	90.5	88.5	86.0	7.14
Terbuphos	94.5	94.5	86.5	91.8	5.03
Terbufos Sulfone	90.5	93.5	87.5	90.5	3.31
Terbuthion Sulfoxide	86.0	87.0	91.0	88.0	3.01
Thiamethoxam	95.5	90.0	82.5	89.3	7.31
Benzamid	91.5	98.0	81.5	90.3	9.20
Fenflux	79.5	83.0	81.5	81.3	2.16
Fludioxonil	103	112	99.0	105	6.34
Flufenacet	79.5	89.5	77.0	82.0	8.07
Flubenzuron	90.5	82.5	77.0	83.3	8.15
Fluopyram	91.0	100	92.0	94.3	5.23
Fluopyram	77.5	92.0	93.0	87.5	9.91
Acifluorfen	88.0	89.5	91.0	89.5	1.68

Furutrione	85.5	93.5	76.5	85.2	9.98
Flusilazole	104	110	96.5	104	6.50
Etoproxil	81.5	78.0	75.5	78.3	3.85
Fenazole	95.0	89.0	89.5	91.2	3.65
Flufenapyramide	100	93.5	98.5	97.3	3.50

Table 5. Spiked Multi-Residue of Pesticide in Tea at 0.375mg/kg

Targets	Recovery Rate (%)			Average Recovery (%)	RSD (%)
	1	2	3		
Acetamiprid	91.2	93.3	93.9	92.8	1.53
Aldicarb	92.0	94.7	91.2	92.6	1.98
Aldicarb Sulfone	92.0	92.0	90.1	91.4	1.20
Aldicarb Sulfoxide	84.3	84.5	84.5	84.4	0.137
Arazine	93.6	78.9	87.7	86.7	8.53
Saponin	96.0	81.3	91.7	89.7	8.43
Avermectin	87.7	76.5	86.9	83.7	7.46
Acytophos	89.3	77.1	83.7	83.4	7.32
Azoxystrobin	88.8	81.1	96.0	88.6	8.41
Herbicide	92.5	87.2	98.1	92.6	5.89
Benzocarb	89.3	83.7	85.9	86.3	3.27
Benefate	90.9	84.3	93.6	89.6	5.34
Boscalid	80.5	84.5	86.9	84.0	3.85
Ethyrimol Sulfonate	93.9	103	101	99.3	4.82
Buprofezin	81.3	81.3	84.3	82.3	2.10
Carbaryl	91.5	86.9	89.1	89.2	2.58
Carbendazim	75.7	62.9	76.3	71.6	10.6
Kebowei	90.9	91.5	88.5	90.3	1.76
3-Hydroxy Carbofuran	94.4	99.5	93.9	95.9	3.23
Chlorantraniliprole	91.7	79.2	86.7	85.9	7.32
Chlorfluazuron	90.9	79.5	89.3	86.6	7.13
Cyclomin	83.7	89.3	85.3	86.1	3.35
Green Myron	86.1	81.3	91.2	86.2	5.74
Cyclofenozide	95.2	88.0	93.3	92.2	4.05
Tetramethazine	82.9	76.8	77.3	79.0	4.29
Clomazone	87.7	80.5	90.1	86.1	5.80
Clothianidin	86.9	90.9	87.7	88.5	2.39
Cyanazine	93.3	88.3	93.1	91.6	3.09
Cyantraniliprole	88.8	77.1	84.0	83.3	7.06
Cyflumetate	101	104	89.1	98.0	8.04
Cymoxanil	93.1	100	92.5	95.2	4.38
Cyprodinil	86.7	69.6	90.9	82.4	13.7
Diflubenzuron	83.5	71.7	79.7	78.3	7.69
Diflufenamide	93.6	85.9	93.9	91.1	4.98
Refined Dimethenamid	89.3	88.5	92.5	90.1	2.35
Kysastrobin	89.3	80.8	91.5	87.2	6.48
Dinotefuran	88.3	85.3	86.1	86.6	1.79
Epoxiconazole	77.6	82.9	89.1	83.2	6.92
Fenoxazone	96.5	104	87.7	96.1	8.49
Enoxastrobin	82.1	84.3	91.7	86.0	5.85
Fenamiphos	91.7	86.1	85.3	87.7	3.98
Fenamifos Sulfone	89.9	90.7	89.3	90.0	0.780
Fenamiphos Sulfoxide	93.3	85.9	87.7	89.0	4.34
Fenazazafen	79.7	74.1	76.3	76.7	3.68
Forchlorfenuron	72.0	57.1	67.2	65.4	11.6
Anthion	86.7	78.1	80.5	81.8	5.43
Thiazophos	89.6	86.1	88.8	88.2	2.08
Furosecarb	84.5	82.1	82.4	83.0	1.58
Heptenylphos	93.9	92.0	91.5	92.5	1.37
Hexythiazox	81.3	82.9	91.7	85.3	6.57
Imidacloprid	91.7	99.2	94.4	95.1	3.99
Chlorothialine	92.5	91.2	94.9	92.9	2.02
Indoxacarb	106	84.5	83.7	91.4	13.8
Chlorazophos	86.9	78.4	88.3	84.5	6.34
Isoprocarb	94.1	90.9	90.1	91.7	2.31
Methiocarb	89.3	84.5	91.7	88.5	4.14
Methiocarb Sulfoxide	82.9	88.5	84.5	85.3	3.38
Methomyl	90.1	97.1	92.8	93.3	3.78
Methoprene	88.3	82.4	82.4	84.4	4.04
Methoxyfenozide	93.1	86.9	95.7	91.9	4.92
Metolachlor	92.0	82.1	91.7	88.6	6.36
Methiocarb	88.5	85.3	89.3	87.7	2.41
Metrafenone	98.4	101	103	101	2.28
Azimidone	88.5	96.5	95.7	93.6	4.71
Memethaphos	89.3	93.3	88.3	90.3	2.93
Picoxystrobin	94.1	79.7	85.9	86.6	8.34
Prochloraz	89.6	102	84.8	92.1	9.64
Prochloraz-Deaminoimidazole	88.5	78.9	86.7	84.7	6.02
Prochloraz-Deimidazole Carboxamide	86.9	78.9	75.2	80.3	7.45
Pyraclostrobin	77.9	74.7	82.4	78.3	4.94
Metazachlor	99.7	94.9	102	98.9	3.66
Pyraclostrobin	94.1	80.5	94.1	89.6	8.76
Pyrazosulfuron-Methyl	81.6	72.8	80.5	78.3	6.12
Pyrethrin I	91.7	85.1	85.3	87.4	4.30
Saflufenacil	91.2	83.7	82.9	85.9	5.33
Pyrimethanil	80.5	70.9	78.9	76.8	6.70
Spinosad D	48.5	44.0	41.3	44.6	8.16
Spirofen	94.1	89.9	88.5	90.8	3.21
Spiromethin	92.5	84.5	88.8	88.6	4.52
Spirotetramat	88.8	72.0	90.9	83.9	12.4
Tebufenozide	82.1	79.2	82.7	81.3	2.30
Buthiuron	92.5	85.1	88.3	88.6	4.19
Flufenuron	80.5	75.5	82.9	79.6	4.74
Terbuphos	76.5	89.3	85.6	83.8	7.86
Terbufos Sulfone	95.7	84.5	89.9	90.0	6.22
Terbuthion Sulfoxide	93.6	86.1	83.7	87.8	5.88
Thiamethoxam	92.3	85.6	86.1	88.0	4.24
Benzamid	91.2	92.5	99.5	94.4	4.73
Fenflux	90.9	82.9	87.7	87.2	4.62
Fludioxonil	95.5	94.1	95.7	95.1	0.917
Flufenacet	94.7	86.1	88.5	89.8	4.94
Flubenzuron	94.9	87.7	93.3	92.0	4.11
Fluopyram	100	93.9	102	98.6	4.28
Fluopyram	95.7	87.7	95.7	93.0	4.97
Acifluorfen	86.1	82.1	88.0	85.4	3.53
Furutrione	95.7	85.6	96.3	92.5	6.50
Flusilazole	88.3	85.3	110	94.5	14.3
Etoproxil	91.7	91.2	88.0	90.3	2.22
Fenazole	86.9	84.0	90.1	87.0	3.51
Flufenapyramide	94.7	89.3	97.3	93.8	4.35

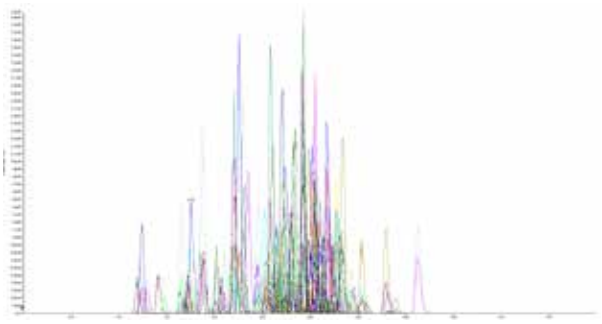


Figure 1. TIC of Multiple Residues of Pesticides in Bok Choy

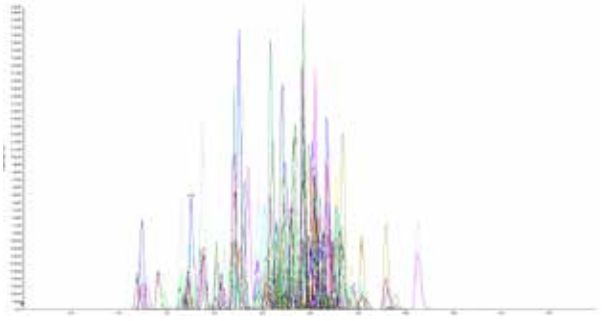
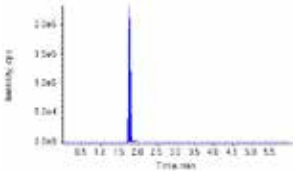
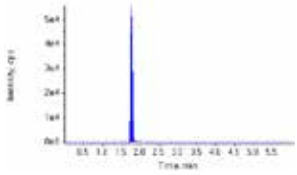


Figure 2. T TIC of Multiple Residues of Pesticides inTea

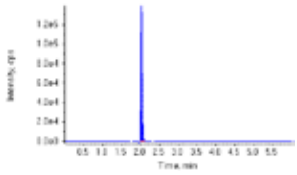
Acetamiprid
223.000/126.000 Da



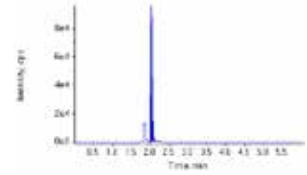
Acetamiprid (qualitative)
223.000/99.000 Da



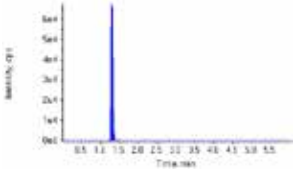
Aldicarb
208.000/116.000 Da



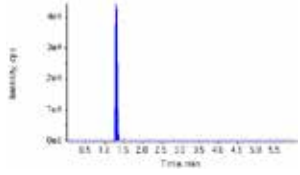
Aldicarb (qualitative)
208.000/89.000 Da



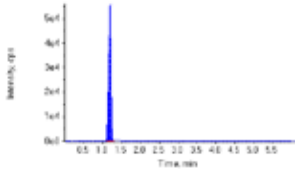
Aldicarb sulfone
240.100/148.000 Da



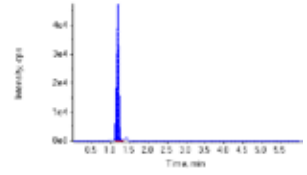
Aldicarb sulfone (qualitative)
240.100/166.000 Da



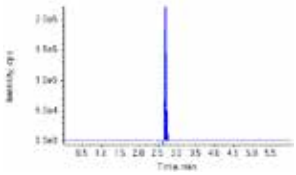
Aldicarb sulfoxide
207.000/132.000 Da



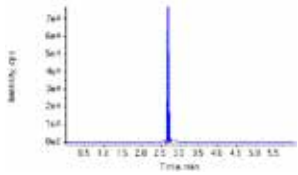
Aldicarb sulfoxide (qualitative)
207.000/89.000 Da



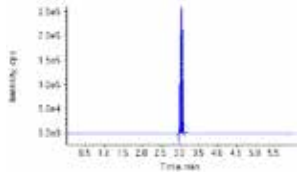
Arazine
228.100/186.200 Da



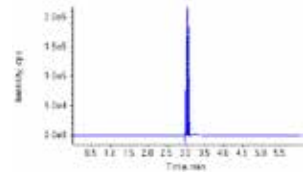
Arazine (qualitative)
228.100/96.000 Da



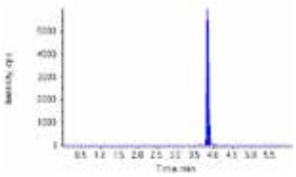
Saponin
368.000/199.000 Da



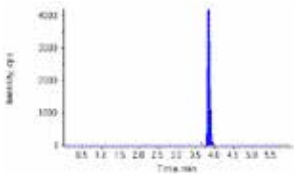
Saponin (qualitative)
368.000/125.000 Da



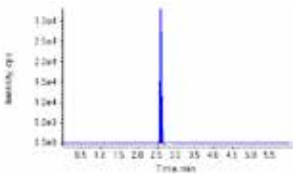
Avermectin
890.500/305.000 Da



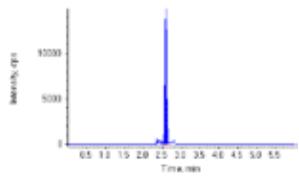
Avermectin (qualitative)
890.500/567.300 Da



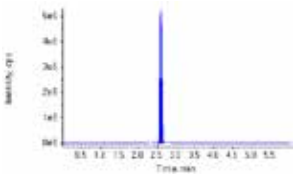
Acytophos
318.000/132.000 Da



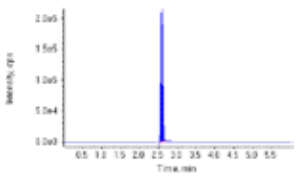
Acytophos (qualitative)
318.000/125.000 Da



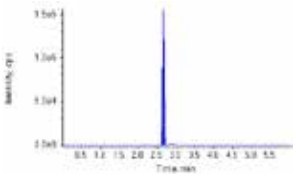
Azoxystrobin
404.100/372.000 Da



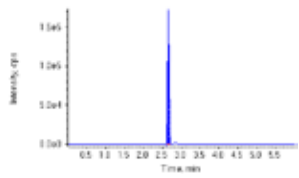
Azoxystrobin (qualitative)
404.100/344.100 Da



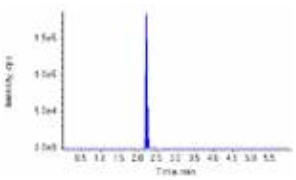
Herbicide
272.000/198.000 Da



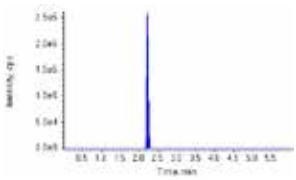
Herbicide (qualitative)
272.000/170.000 Da



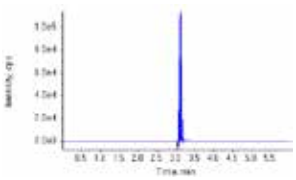
Benzocarb
224.100/167.000 Da



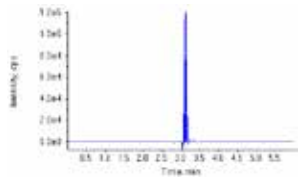
Benzocarb (qualitative)
224.100/109.000 Da



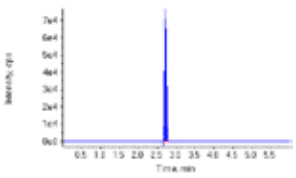
Benzidine
364.100/199.000 Da



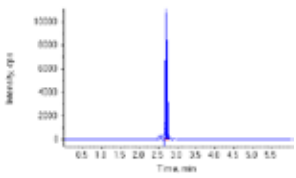
Benzidine (qualitative)
364.100/105.000 Da



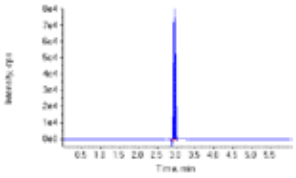
Boscalid
343.000/307.000 Da



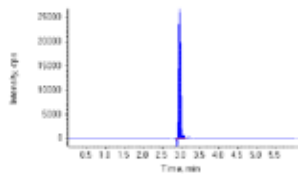
Boscalid (qualitative)
343.000/140.000 Da



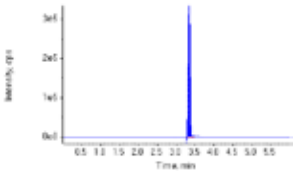
Ethyrimol sulfonate
317.000/166.000 Da



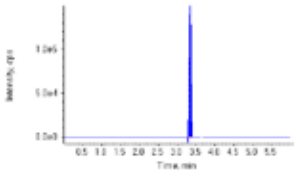
Ethyrimol sulfonate (qualitative)
317.000/210.000 Da



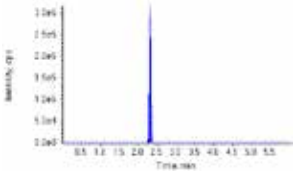
Buprofezin
306.200/201.100 Da



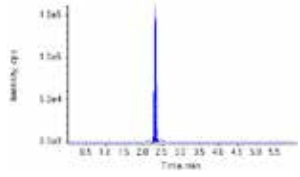
Buprofezin (qualitative)
306.200/116.200 Da



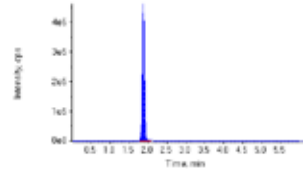
Carbaryl
202.100/145.000 Da



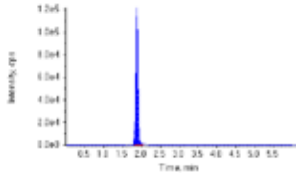
Carbaryl (qualitative)
202.100/127.000 Da



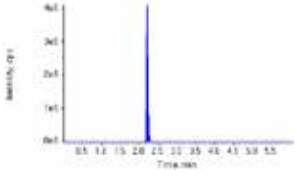
Carbendazim
192.000/160.000 Da



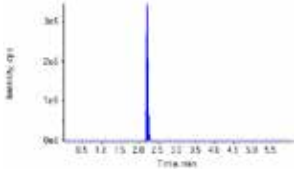
Carbendazim (qualitative)
192.000/132.000 Da



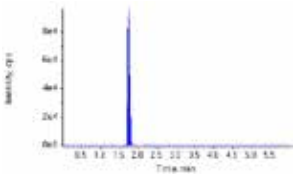
Kebowei
222.100/165.000 Da



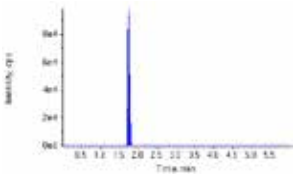
Kebowei (qualitative)
222.100/123.100 Da



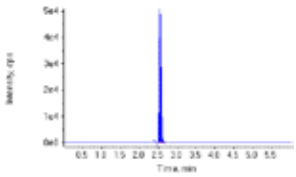
3-Hydroxy carbofuran
238.000/181.000 Da



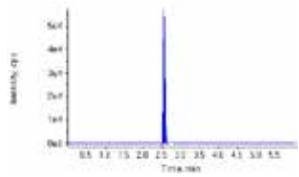
3-Hydroxy carbofuran (qualitative)
238.000/163.000 Da



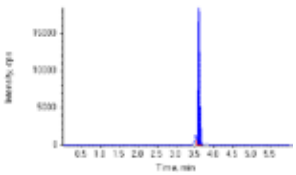
Chlorantraniliprole
483.900/452.900 Da



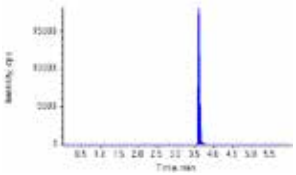
Chlorantraniliprole (qualitative)
483.900/285.900 Da



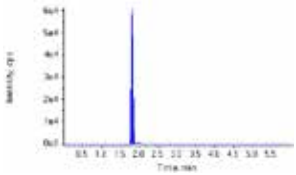
Chlorfluazuron
540.000/383.000 Da



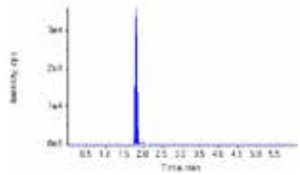
Chlorfluazuron (qualitative)
542.000/385.000 Da



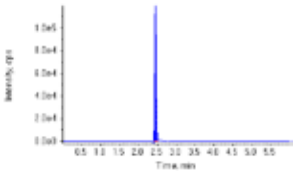
Cyclomin
222.000/104.000 Da



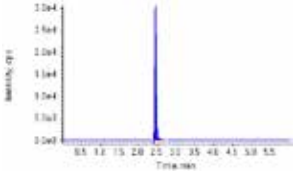
Cyclomin (qualitative)
222.000/92.000 Da



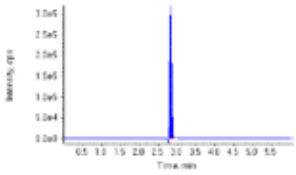
Green Myron
213.100/72.100 Da



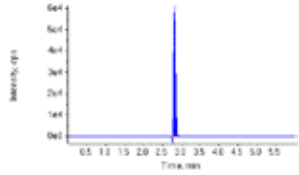
Green Myron (qualitative)
213.100/46.100 Da



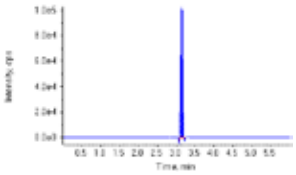
Cyclofenozide
395.200/175.000 Da



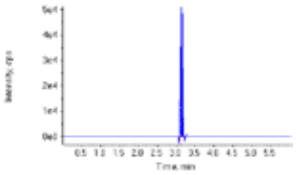
Cyclofenozide (qualitative)
395.200/339.000 Da



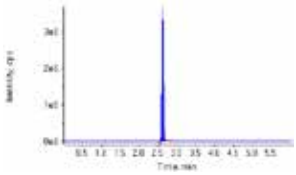
Tetramethazine
303.000/138.000 Da



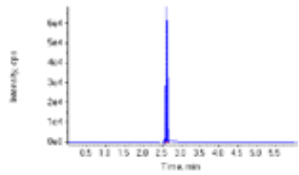
Tetramethazine (qualitative)
303.000/102.000 Da



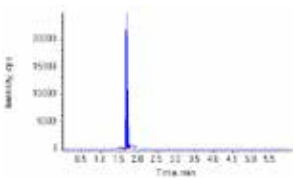
Clomazone
240.100/125.000 Da



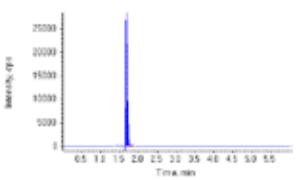
Clomazone (qualitative)
240.100/89.100 Da



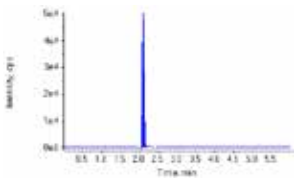
Clothianidin
250.000/169.100 Da



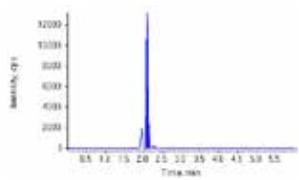
Clothianidin (qualitative)
250.000/132.000 Da



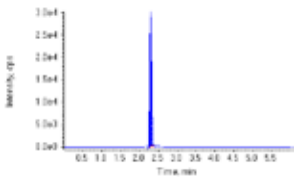
Kusatsu
241.100/214.100 Da



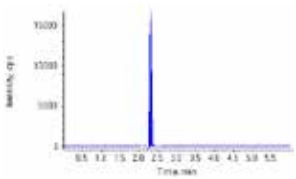
Kusatsu (qualitative)
241.100/104.100 Da



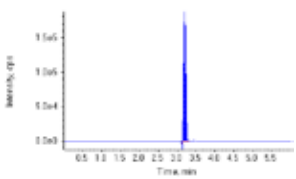
Cyantraniliprole
475.000/286.000 Da



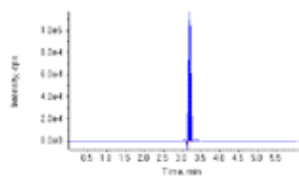
Cyantraniliprole (qualitative)
475.000/444.000 Da



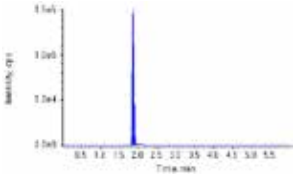
Cyflumetate
465.200/173.000 Da



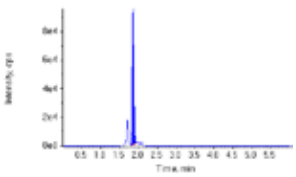
Cyflumetate (qualitative)
465.200/249.100 Da



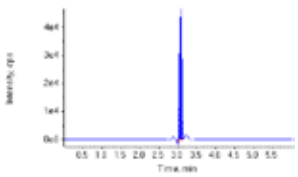
Cymoxanil
199.100/128.000 Da



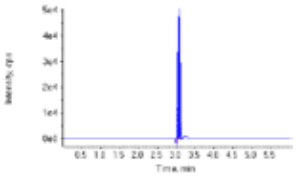
Cymoxanil (qualitative)
199.100/111.000 Da



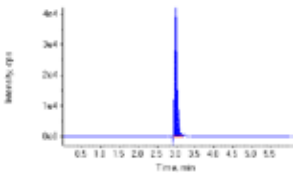
Cyprodinil
226.100/93.000 Da



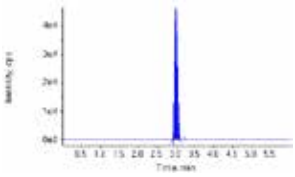
Cyprodinil (qualitative)
226.100/108.100 Da



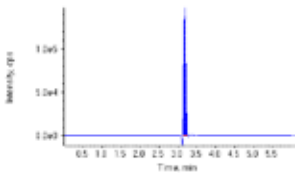
diflubenzuron
311.000/158.000 Da



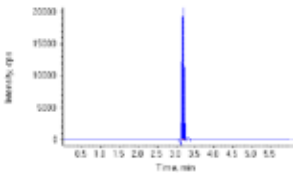
diflubenzuron (qualitative)
311.000/141.200 Da



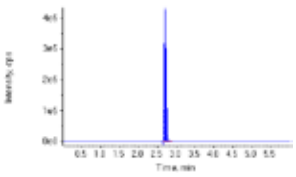
Diflufenamide
395.100/266.000 Da



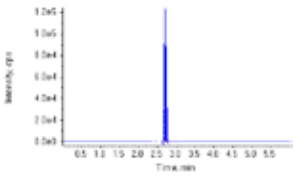
Diflufenamide (qualitative)
395.100/246.000 Da



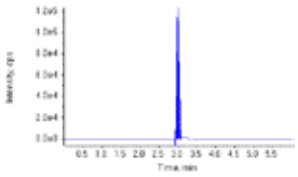
Refined Dimethenamid
276.100/244.100 Da



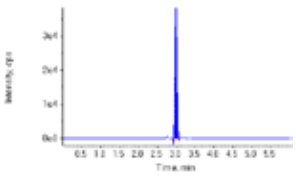
Refined Dimethenamid (qualitative)
276.100/168.100 Da



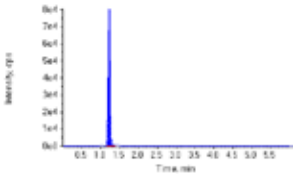
Kresastrobin
327.200/205.000 Da



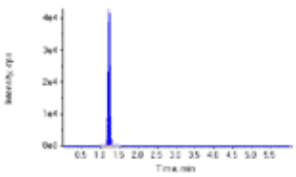
Kresastrobin (qualitative)
327.200/238.100 Da



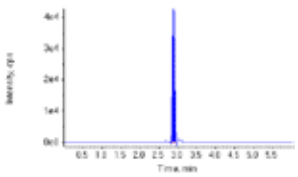
Dinotefuran
203.100/129.000 Da



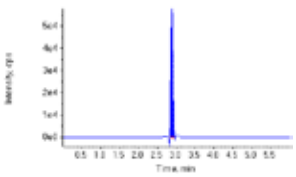
Dinotefuran (qualitative)
203.100/157.000 Da



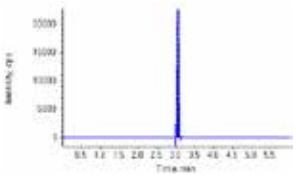
Epoxiconazole
330.000/121.000 Da



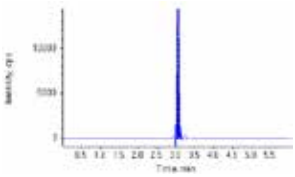
Epoxiconazole (qualitative)
330.000/101.000 Da



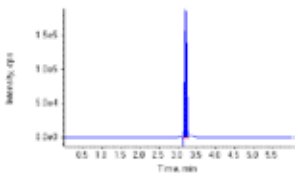
Fenoxazone
392.000/331.000 Da



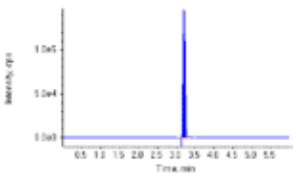
Fenoxazone (qualitative)
392.000/238.000 Da



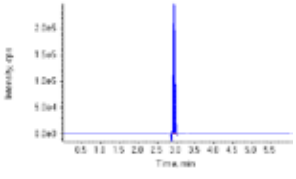
Enoxastrobin
434.100/171.000 Da



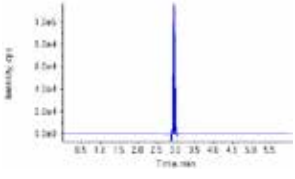
Enoxastrobin (qualitative)
434.100/212.000 Da



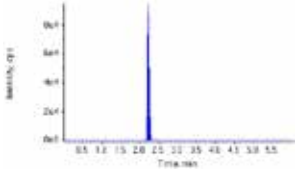
Fenamiphos
304.200/217.100 Da



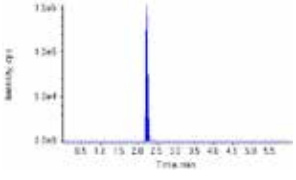
Fenamiphos (qualitative)
304.200/202.000 Da



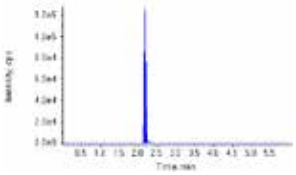
Fenamifos sulfone
336.000/308.000 Da



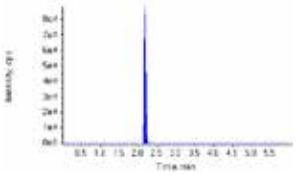
Fenamifos sulfone (qualitative)
336.000/266.000 Da



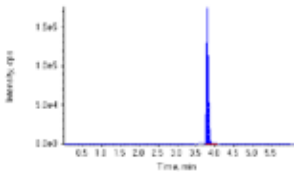
Fenamiphos sulfoxide
320.100/233.000 Da



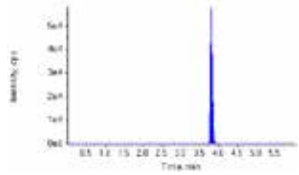
Fenamiphos sulfoxide(qualitative)
320.100/171.000 Da



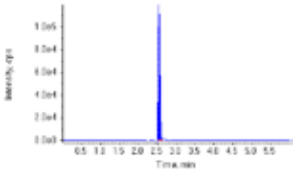
Fenazazafen
307.200/161.000 Da



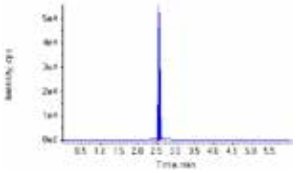
Fenazazafen(qualitative)
307.200/131.100 Da



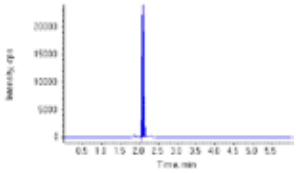
Forchlorfenuron
248.100/129.000 Da



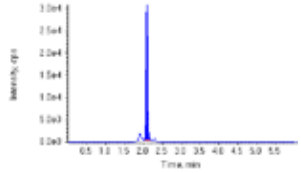
Forchlorfenuron (qualitative)
248.100/93.000 Da



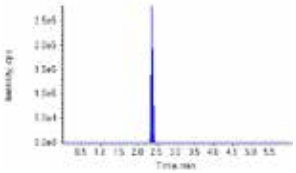
Anthion
258.000/199.000 Da



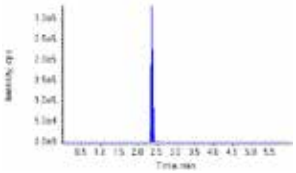
Anthion (qualitative)
258.000/125.000 Da



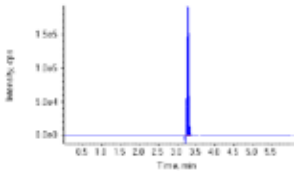
Thiazophos
284.000/228.000 Da



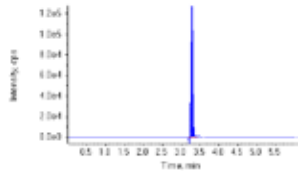
Thiazophos (qualitative)
284.000/104.000 Da



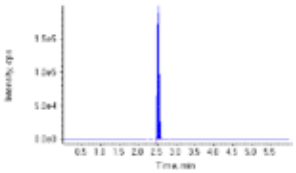
Furosecarb
383.200/195.000 Da



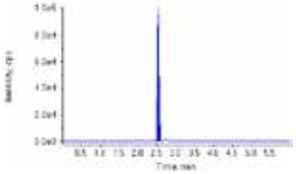
Furosecarb (qualitative)
383.200/252.000 Da



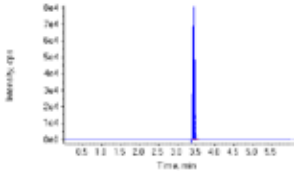
Heptenylphos
251.000/127.000 Da



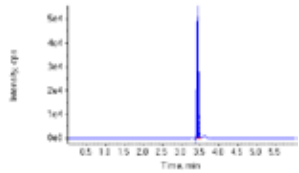
Heptenylphos (qualitative)
251.000/125.000 Da



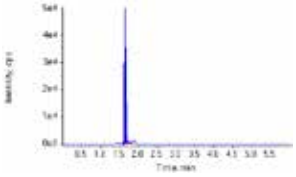
Hexythiazox
353.000/228.000 Da



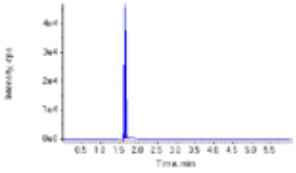
Hexythiazox (qualitative)
353.000/168.000 Da



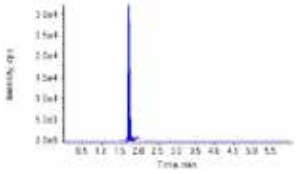
Imidacloprid
256.100/175.000 Da



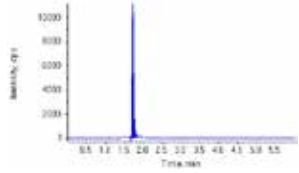
Imidacloprid (qualitative)
256.100/209.000 Da



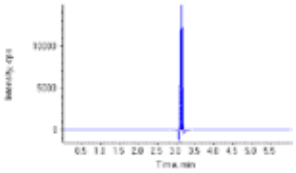
Chlorothialine
262.100/181.100 Da



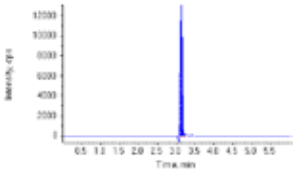
Chlorothialine (qualitative)
262.100/122.000 Da



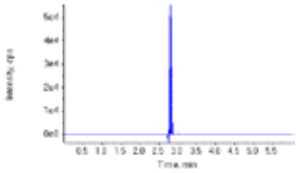
Indoxacarb
528.000/293.000 Da



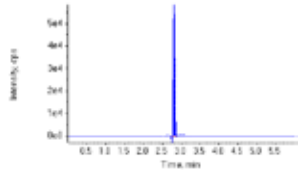
Indoxacarb (qualitative)
528.000/249.000 Da



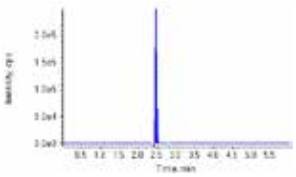
Chlorazophos
316.000/164.000 Da



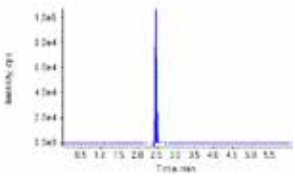
Chlorazophos (qualitative)
316.000/122.000 Da



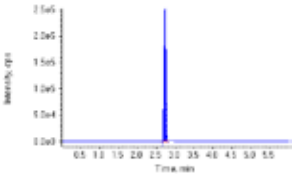
Isoprocarb
194.000/95.000 Da



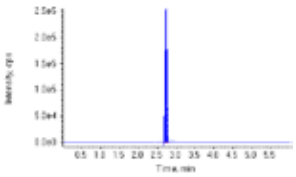
Isoprocarb (qualitative)
194.000/137.000 Da



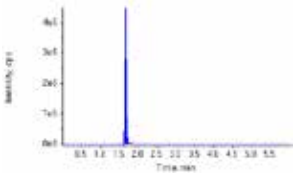
Methiocarb
226.100/169.200 Da



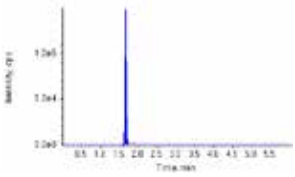
Methiocarb (qualitative)
226.100/121.100 Da



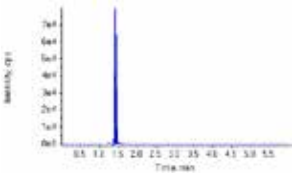
Methiocarb sulfoxide
242.000/185.000 Da



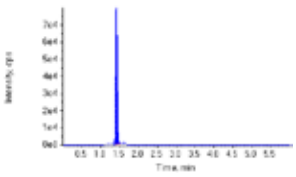
Methiocarb sulfoxide(qualitative)
242.000/122.000 Da



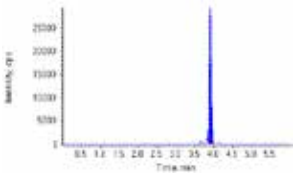
Methomyl
163.000/88.000 Da



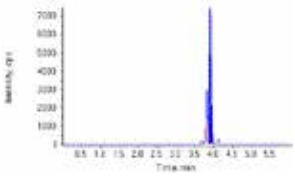
Methomyl (qualitative)
163.000/106.000 Da



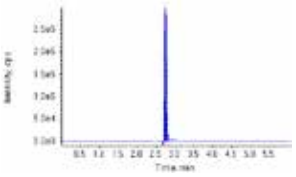
Methoprene
279.000/191.000 Da



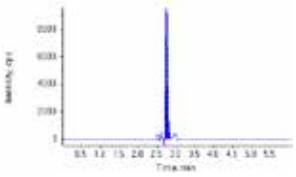
Methoprene (qualitative)
279.000/237.000 Da



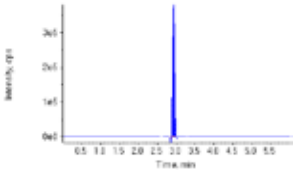
Methoxyfenozide
369.200/149.100 Da



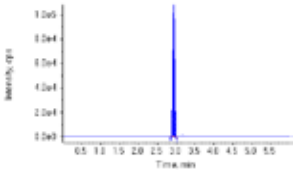
Methoxyfenozide (qualitative)
369.200/133.000 Da



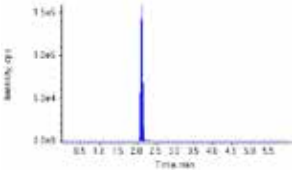
Metolachlor
284.100/251.900 Da



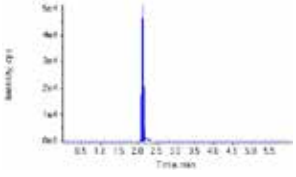
Metolachlor (qualitative)
284.100/176.000 Da



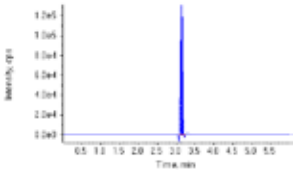
Methiocarb
166.100/109.000 Da



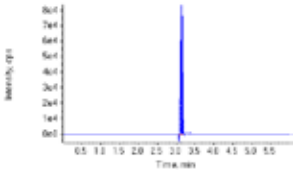
Methiocarb (qualitative)
166.100/94.200 Da



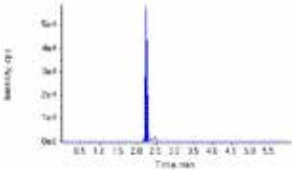
Metrafenone
409.000/209.100 Da



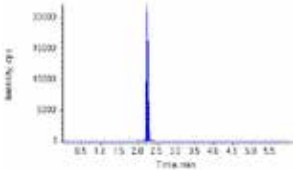
Metrafenone (qualitative)
409.000/227.100 Da



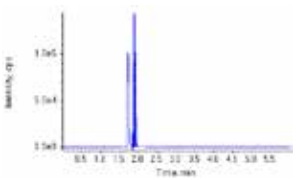
Azimidone
215.100/187.100 Da



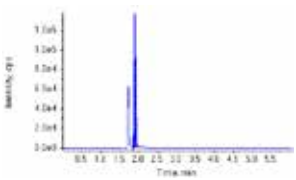
Azimidone (qualitative)
215.100/84.100 Da



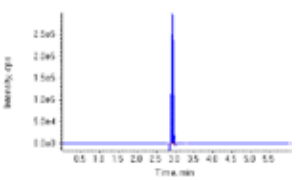
Memethaphos
225.000/127.000 Da



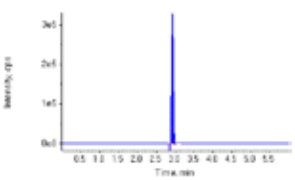
Memethaphos (qualitative)
225.000/193.000 Da



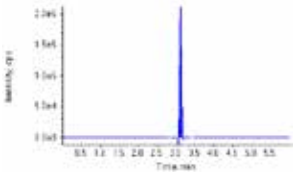
Picoxystrobin
368.100/205.100 Da



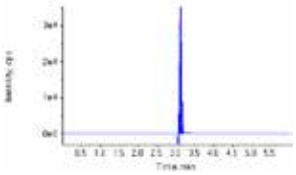
Picoxystrobin (qualitative)
368.100/145.100 Da



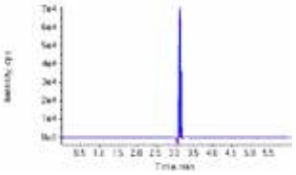
Prochloraz
376.200/308.000 Da



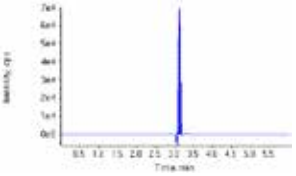
Prochloraz (qualitative)
376.200/266.000 Da



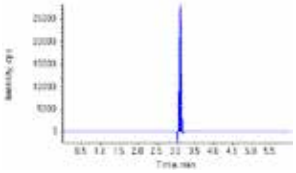
Prochloraz-deaminoimidazole
325.000/282.000 Da



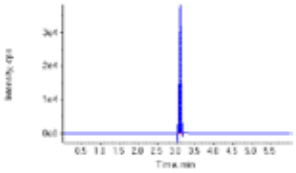
Prochloraz-deaminoimidazole (qualitative)
327.000/284.000 Da



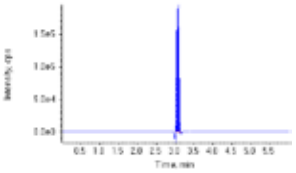
Prochloraz-deimidazole carboxamide
353.000/308.000 Da



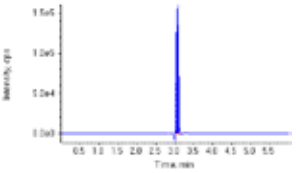
Prochloraz-deimidazole carboxamide
(qualitative)355.000/310.000 Da



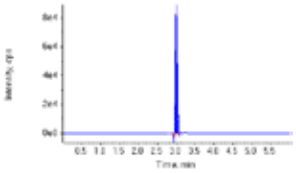
Pyraclostrobin
388.100/194.000 Da



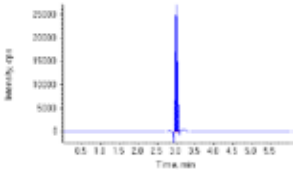
Pyraclostrobin (qualitative)
388.100/163.000 Da



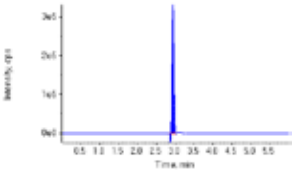
Metazachlor
413.000/339.000 Da



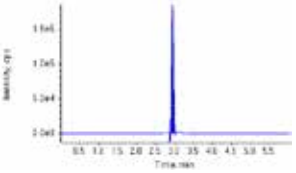
Metazachlor (qualitative)
413.000/253.000 Da



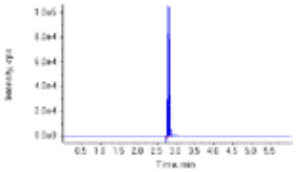
Pyraclostrobin
382.100/194.100 Da



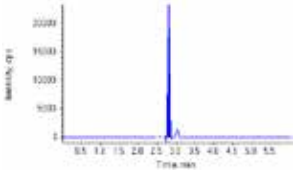
Pyraclostrobin (qualitative)
382.100/163.000 Da



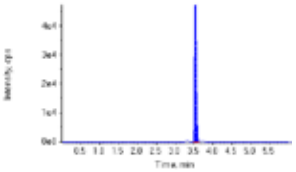
Pyrazosulfuron-methyl
415.100/182.000 Da



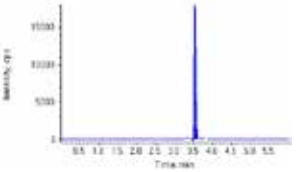
Pyrazosulfuron-methyl (qualitative)
415.100/139.000 Da



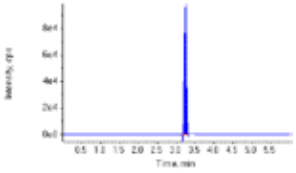
Pyrethrin I
329.200/161.100 Da



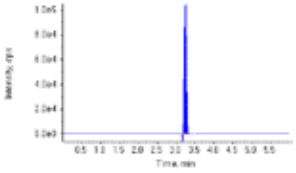
Pyrethrin I (qualitative)
329.200/133.100 Da



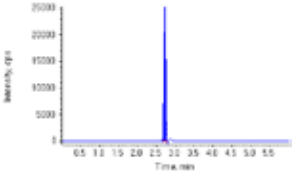
Saflufenacil
610.200/413.100 Da



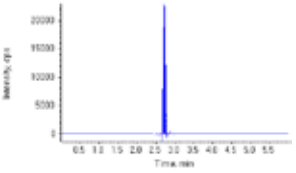
Saflufenacil (qualitative)
610.200/180.100 Da



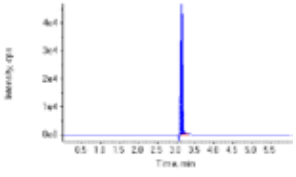
Pyrimethanil
200.000/183.000 Da



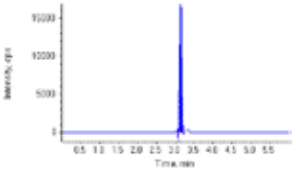
Pyrimethanil (qualitative)
200.000/168.000 Da



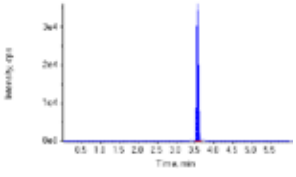
Spinosad D
746.400/142.000 Da



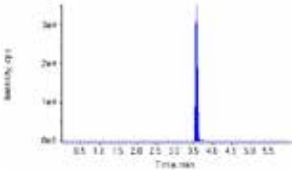
Spinosad D (qualitative)
746.400/98.000 Da



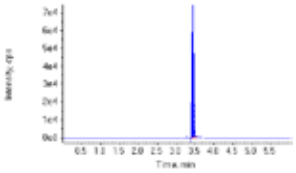
Spirofen
411.200/313.100 Da



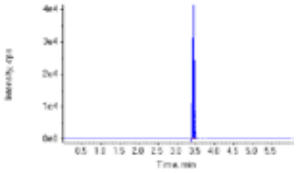
Spirofen (qualitative)
411.200/71.100 Da



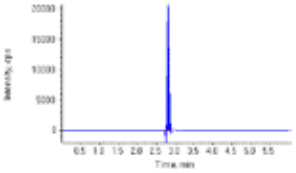
Spiromethin
388.000/273.000 Da



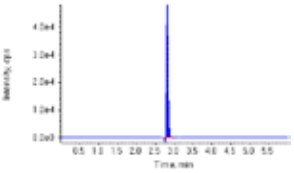
Spiromethin (qualitative)
388.000/255.000 Da



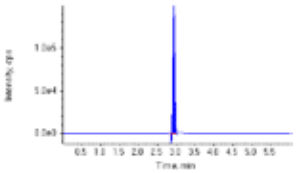
Spirotetramat
374.000/302.000 Da



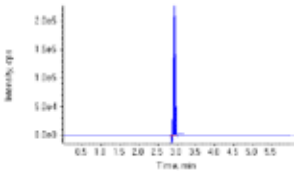
Spirotetramat (qualitative)
374.000/330.000 Da



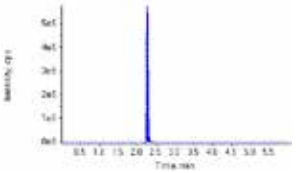
Tebufenozide
353.100/133.100 Da



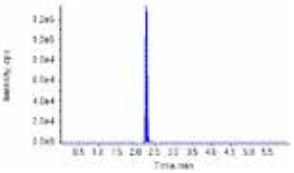
Tebufenozide (qualitative)
353.100/297.100 Da



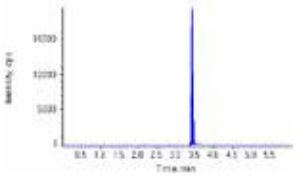
Buthiuron
229.100/172.000 Da



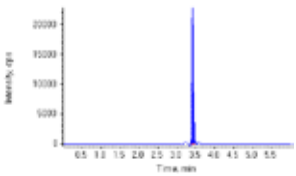
Buthiuron (qualitative)
229.100/116.100 Da



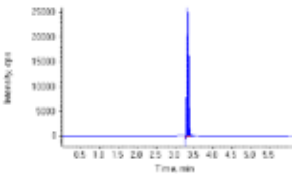
Flufenuron
381.100/158.200 Da



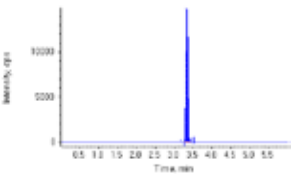
Flufenuron (qualitative)
381.100/141.200 Da



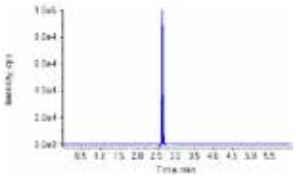
Terbuphos
289.100/103.100 Da



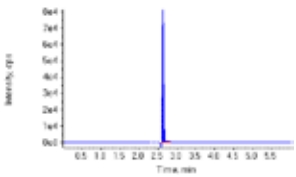
Terbuphos (qualitative)
289.100/233.200 Da



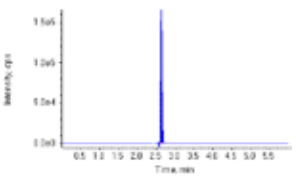
Terbufos sulfone
321.000/97.000 Da



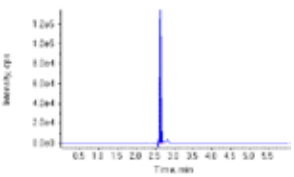
Terbufos sulfone((qualitative)
321.000/115.000 Da



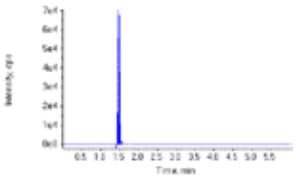
Terbuthion sulfoxide
305.000/187.000 Da



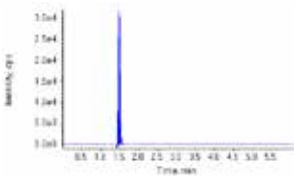
Terbuthion sulfoxide (qualitative)
305.000/131.000 Da



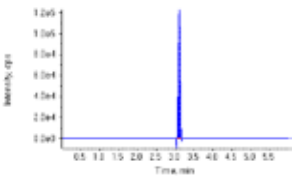
Thiamethoxam
292.000/211.000 Da



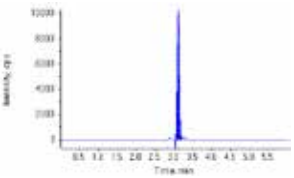
Thiamethoxam (qualitative)
292.000/181.000 Da



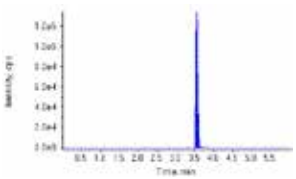
Benzamid
336.000/186.900 Da



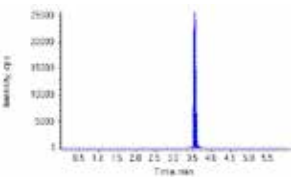
Benzamid (qualitative)
336.000/204.000 Da



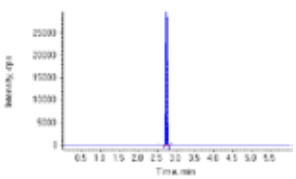
Fenflux
422.000/366.100 Da



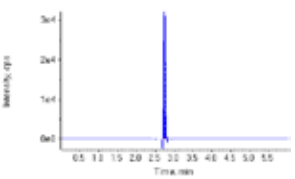
Fenflux (qualitative)
422.000/135.100 Da



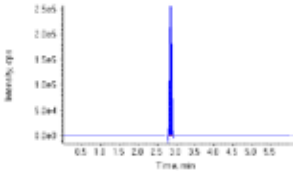
Fludioxonil
266.100/229.000 Da



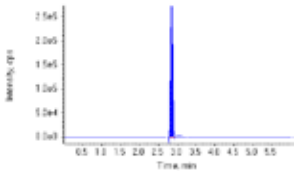
Fludioxonil (qualitative)
266.100/158.000 Da



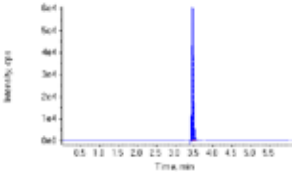
Flufenacet
364.000/194.000 Da



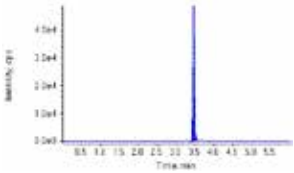
Flufenacet (qualitative)
364.000/152.000 Da



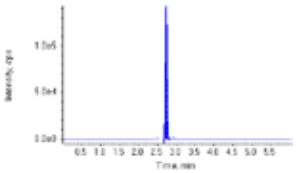
Flubenzuron
489.000/158.100 Da



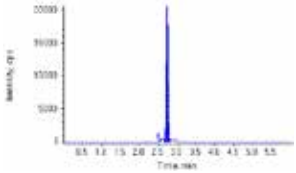
Flubenzuron (qualitative)
489.000/141.200 Da



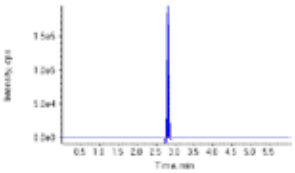
Fluopyram
382.900/172.900 Da



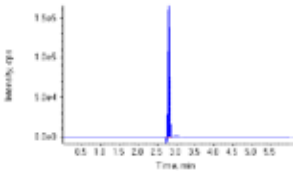
Fluopyram (qualitative)
382.900/364.900 Da



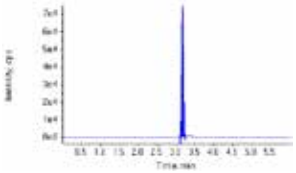
Fluopyram
397.000/207.900 Da



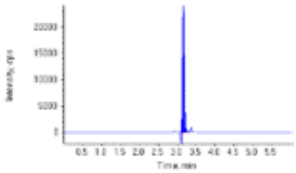
Fluopyram (qualitative)
397.000/172.900 Da



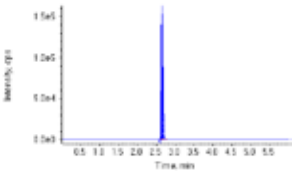
Acifluorfen
465.100/344.000 Da



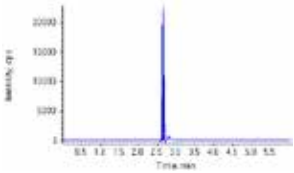
Acifluorfen (qualitative)
465.100/223.000 Da



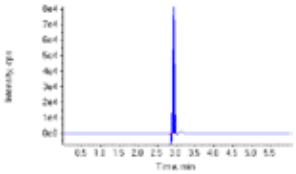
Furutrione
334.100/247.100 Da



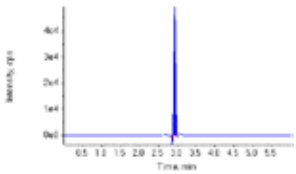
Furutrione (qualitative)
334.100/303.000 Da



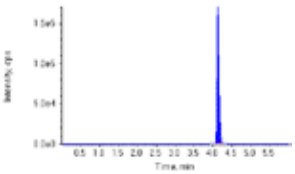
Flusilazole
316.000/247.100 Da



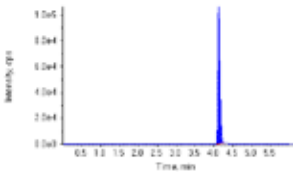
Flusilazole (qualitative)
316.000/165.000 Da



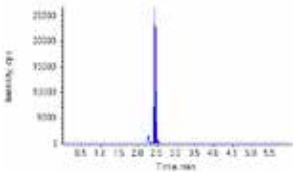
Etoproxil
394.200/177.100 Da



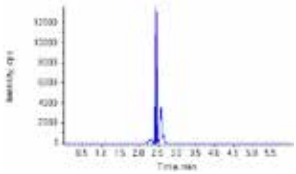
Etoproxil (qualitative)
394.200/107.100 Da



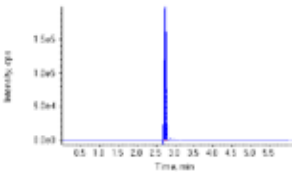
Fenazole
302.100/123.000 Da



Fenazole(qualitative)
302.100/109.000 Da



Flufenapyramide
382.100/362.000 Da



Flufenapyramide (qualitative)
382.100/342.100 Da

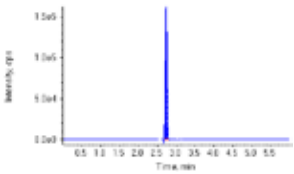


Figure 3. Characteristic Ion Mass Chromatogram of Multi-Residue of Pesticide Standard Solution (0.0200mg/L)

Ordering Information

Cat. #	Description	Qty.
COQ050010CH	Extraction Salts (4 g $MgSO_4$, 1 g NaCl, 1 g Trisodium Citrate, 0.5 g Disodium Citrate), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ050020CH	Extraction Salts (6 g $MgSO_4$, 1.5 g NaOAc), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ015020H	150 mg PSA, 15 mg GCB, 900 mg $MgSO_4$, 15 ml Tube	50 Pcs/Box
COQ050020H	Extraction Salts (6 g $MgSO_4$, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
COQ015047H	400 mg PSA, 400 mg C18, 1200 mg $MgSO_4$, 200 mg GCB,15 ml Tube	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box

Comparison of QuEChERS for Multi-Residue of Pesticides in Vegetable & Grain

Introduction

Pesticide residues are one of the most common threats to food safety. QuEChERS effectively separate trace pesticide residues in vegetables, fruits, grains and other matrices. QuEChERS is capable of effectively separating trace pesticide residues in various matrices such as vegetables and grains.

This experiment compared the purification efficiency and recovery rate of different matrices (simple matrix: cabbage, complex matrix: chives, grain: corn flour) between Copure® QuEChERS and well-known Brand A's QuEChERS kits. As result, regardless of the matrix type, Copure® QuEChERS achieves equivalent to the Brand A.

Experiment

Extraction and purification of vegetable

Weigh 10.000 g of the ground sample (simple matrix: cabbage, complex matrix: chives) into a 50 mL centrifuge tube. Add 10 mL of acetonitrile and vortex for 1 min. Then add QuEChERS extraction salt (Copure®: COQ050010H, Brand A: #1). Shake vigorously for 1 min, then vortex for 5 min. Centrifuge at 4000 r/min for 5 min. Transfer 6 mL of the upper acetonitrile layer to 15 mL tubes (cabbage: Copure®: COQ015022H, Brand A: #2; chives: Copure®: COQ015020H, Brand A: #3). Vortex for 1 min and centrifuge at 4000 r/min for 5 min. Precisely pipette 2 mL of the supernatant to a clean tube and evaporate to nearly dryness at 40 °C in a water bath. Dilute to 1 mL with the initial mobile phase and vortex to mix well. After filter by 0.22 μm nylon membrane, the sample is ready for analysis.

Extraction and purification of grain

Weigh 5.00 g of corn flour into a 50 mL centrifuge tube. Add 10 mL of water, and vortex to mix well. Let it stand for 30 min. Add 15 mL of acetonitrile-acetic acid solution (99+1), and vortex for 5 min. Extract by ultrasonic treatment for 10 min. Then add QuEChERS extraction salt (Copure®: COQ050020H, Brand A: #4). Shake vigorously for 1 min, and vortex for 5 min. Centrifuge at 4000 r/min for 5 min. Transfer 6 mL of the upper acetonitrile layer to a 15 mL tube (Copure®: COQ015033H, Brand A: #5). Vortex for 5 min and centrifuge at 4000 r/min for 5 min. Precisely pipette 2 mL of the supernatant to a clean tube and evaporate to nearly dryness at 40 °C. Dilute to 1 mL with the initial mobile phase and vortex to mix well. After filter by 0.22 μm nylon membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Prepare blank sample in the same procedures. Add mixed standard solution and add dilute to the concentration for LC-MS determination.

Instrumental Conditions

1.Chromatographic conditions

Instrument: Thermo Fisher TSQ Endura

Chromatographic column: Hypersil GOLD C18 (2.1 mm×100 mm, 1.9 μm)

Mobile phase: A: 0.1 % Formic acid water, B: Methanol

Flow rate: 0.4 mL/min

Column temperature: 35 ℃

Injection volume: 5 μL

Table 1. Gradient Elution Program

Time(min)	A(%)	B(%)
0.0	98	2
2.0	95	5
8.0	70	30
16.5	30	70
17.5	30	70
19	2	98
21	2	98
21.5	98	2
22	98	2

2.Mass Spectrometry Conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure: 30 arb

Auxiliary gas pressure: 2 arb

Ion exchange tube: 380 ℃

Auxiliary gas temperature: 350 ℃

Table 2. Targes Characteristic Ions (*Quantifier Ion)

Targets	Parent Ion (m/z)	Daughter Ion (m/z)
Methamidophos	142.1	94.1*,125.1
Oxidized Fruit	214.1	182.9*,125.0
Aldicarb Sulfone	233.2	166.0,72.0*
Monocrotophos	224.1	192.9*,109.1
Imidacloprid	247.0	209.0*,175.1
Dimethoate	230.0	198.9*,125.1
3-Hydroxy Carbofuran	238.2	181.0*,163.0

Aldicarb	213.1	89.1*,98.1
Thiophos	312.0	139.9*,227.9
Phosphamide	300.0	174.0*,127.0
Promox	210.0	111.1*,168.0
Kebowei	222.3	165.0*,123.1
Fenamiphos Sulfoxide	320.1	233.0*,292.0
Fenamifos Sulfone	336.1	266.0*,308.1
Phorate Sulfoxide	277.1	125.1*,259.0
Isoprocarb	194.0	95.1*,137.1
Phorate	293.1	247.0*,251.9
Isocarbophos	312.0	269.9*,235.9
Demeton	259.1	89.1*,61.2
Terbuphos	321.1	274.9*,171.0
Terbuthion Sulfoxide	305.0	248.9*,187.2
Methiocarb	226.0	169.1*,121.1
Violent	208.0	109.0*,151.1
Malathion	331.0	127.1*,199.0
Diphenphos	243.1	172.9*,214.9
Pherophos	323.0	170.9*,294.9
Tefenthion	247.0	137.0*,109.8

Results

Table 3. Spiked Recovery of Cabbage (Light color) at 0.05 mg/kg

No.	Targets	Recovery (%)	
		Copure®	Brand A
1	Methamidophos	80.3	87.7
2	Oxidized Fruit	73.6	84.3
4	Aldicarb Sulfone	103.6	99.0
5	Monocrotophos	83.5	88.4
6	Imidacloprid	83.6	98.9
7	Dimethoate	97.9	119.9
8	3-Hydroxy Carbofuran	96.3	93.8
9	Aldicarb	114.7	108.9
10	Thiophos	88.5	87.4
11	Phosphamide	87.0	85.8
12	Promox	91.7	88.1
13	Kebowei	89.8	84.1
14	Fenamiphos Sulfoxide	83.0	82.9
15	Fenamifos Sulfone	87.8	99.5
16	Phorate Sulfoxide	95.9	95.6
17	Isoprocarb	89.3	89.6
18	Phorate	95.5	98.7
19	Isocarbophos	91.3	99.6
20	Demeton	82.9	78.6
21	Terbuphos	90.0	84.5
22	Terbuthion Sulfoxide	74.5	99.6
23	Methiocarb	68.2	64.8
24	Violent	78.6	75.3
25	Malathion	81.0	80.9
26	Diphenphos	79.9	77.8
27	Pherophos	53.0	46.1
28	Tefenthion	51.2	44.5

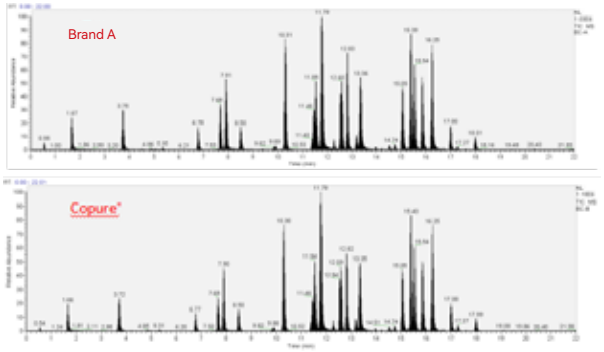


Figure.1 Total Reaction Monitoring Diagram of Cabbage Spiked Samples

Table 4. Spiked Recovery of Chives (Dark Color) at 0.05 mg/kg

No.	Targets	Recovery (%)	
		Copure®	Brand A
1	Methamidophos	86.5	88.2
2	Oxidized Fruit	67.1	67.5
3	Aldicarb Sulfone	101.9	119.9
4	Monocrotophos	67.9	77.6
5	Imidacloprid	88.5	99.9
6	Dimethoate	97.4	112.5
7	3-Hydroxy Carbofuran	89.4	94.3
8	Aldicarb	115.6	101.6
9	Thiophos	94.8	93.6
10	Phosphamide	93.4	95.0
11	Promox	95.1	94.7
12	Kebowei	91.5	94.4
13	Fenamiphos Sulfoxide	86.4	84.6
14	Fenamifos Sulfone	82.5	85.8
15	Phorate Sulfoxide	91.4	93.6
16	Isoprocarb	77.7	75.5
17	Phorate	85.3	86.1
18	Isocarbophos	88.3	85.7
19	Demeton	71.1	70.0
20	Terbuphos	64.3	64.9
21	Terbuthion Sulfoxide	81.5	85.0
22	Tefenthion	52.0	55.1
23	Diphenphos	54.3	54.5
24	Malathion	49.5	52.8

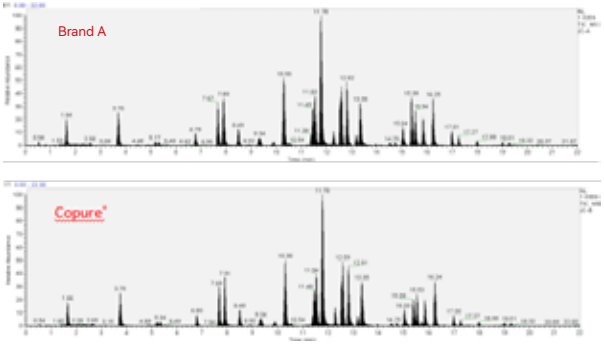


Figure 2. Total Reaction Monitoring Chart of Spiked Chives Samples

Table 4. Spiked Recovery of Grains (Corn Flour) at 0.3 mg/kg)

No.	Targets	Recovery (%)	
		Copure®	Brand A
1	Methamidophos	69.6	67.6
2	Oxidized Fruit	90.6	83.3
3	Aldicarb Sulfone	93.6	93.4
4	Monocrotophos	77.3	77.3
5	Imidacloprid	82.4	80.7
6	Dimethoate	104.9	109.1
7	3-Hydroxy Carbofuran	98.6	98.0
8	Aldicarb	101.9	102.0
9	Thiophos	81.2	85.1
10	Phosphamide	85.4	85.8
11	Promox	93.6	80.5
12	Kebowei	86.4	86.1
13	Fenamiphos Sulfoxide	83.5	81.6
14	Fenamifos Sulfone	103.5	109.9
15	Phorate Sulfoxide	90.4	90.3
16	Isoprocarb	67.5	61.9
17	Phorate	80.8	85.2
18	Isocarbophos	82.1	96.1
19	Demeton	76.9	80.9
20	Terbuphos	69.8	70.9
21	Terbuthion Sulfoxide	75.8	71.8
22	Violent	65.0	62.8
23	Diphenphos	62.1	59.9
24	Ethametsulfuron	57.9	58.4

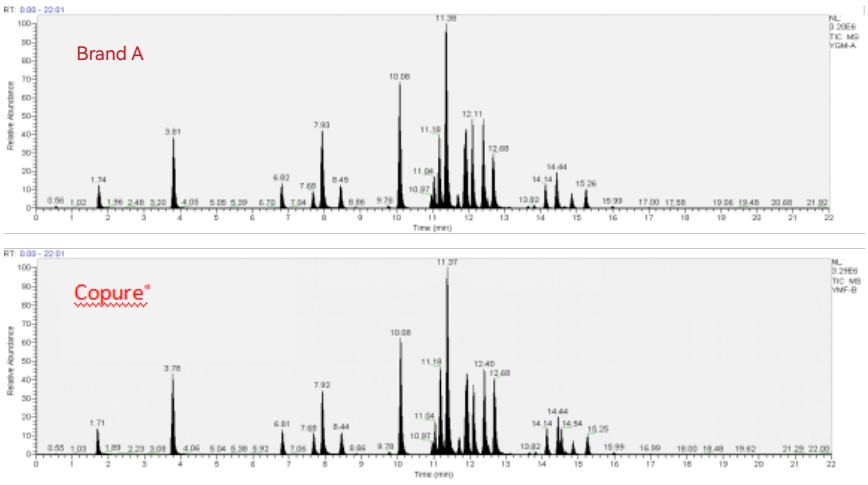


Figure 3. Total Reaction Monitoring Chart of Corn Flour Spiked Samples

Ordering Information

Cat. #	Description	Qty.
COQ050010H	Extraction Salts (4 g $MgSO_4$, 1 g NaCl, 1 g Trisodium Citrate, 0.5 g Disodium Citrate), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ015022H	150 mg PSA, 900 mg$MgSO_4$, 15 ml Tube	50 Pcs/Box
COQ015020H	150 mg PSA, 15 mg GCB, 900 mg$MgSO_4$, 15 ml Tube	50 Pcs/Box
COQ050020H	Extraction Salts (6 g $MgSO_4$, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
COQ015033H	400 mg PSA, 400 mg C18, 1200 mg$MgSO_4$	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box

Fluvalinate in Honey (Copure® Florisil SPE Cartridge)

Experiment

Extraction

Weigh 5 g of honey into a 50 mL centrifuge tube, add 10 mL of water. Vortex for 1 min. Add 20 mL of n-hexane-acetone solution (1:1), and vortex for 3 min. Centrifuge at 9500 r/min for 5 min, and transfer the upper layer to a 150 mL flask. Repeat the extraction once with 20 mL of n-hexane-acetone solution (1:1). Combine the extracted solution, and concentrate in a water bath at 40 °C under reduced pressure to nearly dryness. Add 3 mL of n-hexane to dissolve and set aside.

Purification

(Copure® Florisil SPE Cartridge, 1000 mg/ 6 mL)

Activate the Copure® Florisil SPE Cartridge by 5 mL of n-hexane-acetone solution (95:5), then 5 mL of n-hexane. Pipette the prepared solution to the cartridge. Wash the flask with 3 mL of n-hexane, and transfer to the cartridge. Elute with 10 mL of n-hexane-acetone solution (95:5).

Collect the eluate and evaporate to nearly dryness at 40 ℃. Reconstitute with 1.0 mL of n-hexane. The sample is ready for analysis.

Instrumental Conditions

Instrument: Gas Chromatograph (Agilent 7890B)

Chromatographic column: Agilent Technologies DB-35MS UI (30 m×0.250 mm, 0.25 μm)

Carrier gas: nitrogen (purity ≥ 99.999%)

Injection port temperature: 280 ℃

Detector temperature: 300 °C

Column temperature: The initial column temperature is 80 °C, and maintain 1 min. Raise to 220 °C at a rate of 40 °C/min, and maintain 1 min. Then, raise to 300 °C at a rate of 10 °C/min, and maintain 1.5 min.

Carrier gas flow:1.2 mL/min

Injection volume: 1 μL

Injection method: splitless injection

Results

Table 1. Fluvalinate Spiked Recovery in Honey at 10 μg/kg

Target	Recovery (%)			Average Recovery (%)	RSD (%)
	1	2	3		
Fluvalinate	84.3	79.3	78.1	80.6	4.08

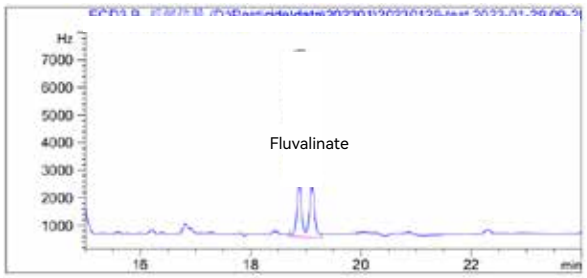


Figure 1. Gas Chromatogram for Fluvalinate in Honey at 10 μg/kg

Ordering Information

Cat. #	Description	Qty.
COFL61000	Copure® Florisil SPE Cartridges, 1000mg / 6mL	30 Pcs/Box
SF130-45-NL	Syringe Filters / NL / Φ13 mm / 0.45 μm / Hydrophilic	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box

High-Throughput of 23 Pesticide Residues in Cabbage (Copure® 24-Well Pesticide Residue Purification Plates)

Introduction

QuEChERS is widely used to detect pesticide residues in plant-based foods. Biocomma introduces the Copure® 24-Well Pesticide Residue Purification Plates enabling rapid, high-throughput analysis with Biocomma® Positive Pressure 24 Processor system and sample vial tray. No syringe filtration is required prior to LC-MS/MS analysis.

Biocomma established a high-throughput LC-MS/MS method with simplified operation and better result stability for detecting 23 pesticide residues in cabbage. The recoveries were 60-120% with CV values below 10% at both 1 ng/g and 5 ng/g concentrations, which is close to traditional QuEChERS methods for your reference.

Experiment

Extraction (Copure® QuEChERS, Cat. #: COQ050010H)

Chop the samples and grind evenly. Weigh 10.0 g of sample into a 50 mL centrifuge tube. Add 10 mL of acetonitrile and vortex mix for 5 min. Add a pack of QuEChERS extraction salt and shake vigorously for 1 min. Vortex for 5 min, then centrifuge at 5000 r/min for 5 min. The upper acetonitrile layer is ready for purification.

Purification (Copure® 24-Well Pesticide Residue Purification Plates, Cat. #: NC24002)

Place the 24-well purification plate on a Biocomma® 24-Well Vial Tray. Add 2 mL of prepared sample. Place the purification plate and vial tray on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the sample vials. Screw on the caps and the sample is ready for analysis.

Instrument Conditions

1.Chromatography Conditions

Equipment: UPLC-MS/MS (Thermo Scientific TSQ Endura)

Chromatographic column: Commasil® BEH T-C18 (100 mm×2.1mm, 3μm)

Mobile phase: A: Water (0.1% formic acid), B: Methanol

Flow rate: 0.4 mL/min

Injection volume: 5 μL

Column temperature: 30℃

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.00	98	2
1.00	95	5
4.00	70	30
8.00	30	70
9.00	30	70
10.00	2	98
13.50	2	98
14.00	98	2
15.00	98	2

2.Mass Spectrometry Conditions

Source of ion: HESI

Electrospray voltage: 3500V

Sheath gas pressure: 40 Arb

Auxiliary air pressure: 1 Arb

Ion transfer tube: 380 ℃

Atomization temperature: 350 ℃

Results

Table 2. Experimental results of spiked recovery of various prohibited pesticide residues

Targets	Spike (ng/g)	Sample			
		Cabbage			
		24-Well Purification Plate (n=8)		Original QuEChERS Method(n=8)	
		Average Recovery (%)	CV(%)	Average Recovery (%)	CV(%)
Clothianidin	1	67.8	9.78	69.2	14.8
	5	106	8.41	102	4.52
Pyraclostrobin	1	98.9	9.40	99.8	16.5
	5	93.4	2.28	84.7	6.89
Phoxim	1	75.1	9.76	59.8	15.5
	5	85.1	5.91	68.4	9.46
Acetamiprid	1	94.5	9.43	74.3	15.8
	5	100	8.46	94.9	10.5
Carbendazim	1	65.4	8.22	56.6	14.1
	5	86.4	5.63	85.8	4.36
Flusilazole	1	78.5	8.15	76.3	7.65
	5	101	7.56	89.6	7.81
Forchlorfenuron	1	75.8	7.61	82.6	10.6
	5	108	2.42	105	4.92
Prochloraz	1	106	9.16	105	15.5
	5	112	6.49	113	3.65
Azoxystrobin	1	99.6	9.41	119	5.09
	5	108	8.11	108	4.16
Toxenpyrad	1	94.1	9.76	55.0	12.9
	5	87.8	1.87	77.1	15.7
Aldicarb	1	93.8	9.77	96.7	12.3
	5	94.4	6.05	106	6.13
Aldicarb Sulfone	1	82.5	7.85	86.5	11.3
	5	107	7.28	93.7	10.6

Fipronil Sulfone	1	90.1	9.80	102	12.6
	5	116	5.68	105	9.36
Fipronil Sulfide	1	99.6	9.69	103	12.3
	5	104	9.64	98.2	7.47
Fluoronitrile	1	98.2	9.11	108	18.1
	5	105	2.40	103	4.29
Fipronil	1	114	9.51	101	11.2
	5	106	9.85	95.9	6.35
Kebowei	1	96.7	6.18	97.5	6.67
	5	106	3.27	102	2.53
3-Hydroxy-carbofuran	1	106	7.17	101	7.78
	5	89.9	4.32	90.0	4.69
Imidacloprid	1	96.1	9.13	75.0	7.41
	5	78.2	8.49	92.7	4.22
Propargite	1	82.3	9.18	53.1	18.1
	5	78.6	4.39	54.1	16.5
Fenflux	1	78.5	9.91	64.1	6.05
	5	101	4.28	94.5	1.67
Prochloraz-Deaminoimidazole	1	98.6	9.85	86.1	15.1
	5	101	6.86	104	4.86
Prochloraz-Deimidazole Carboxamide	1	96.4	9.65	102	15.3
	5	102	4.19	98.5	9.87

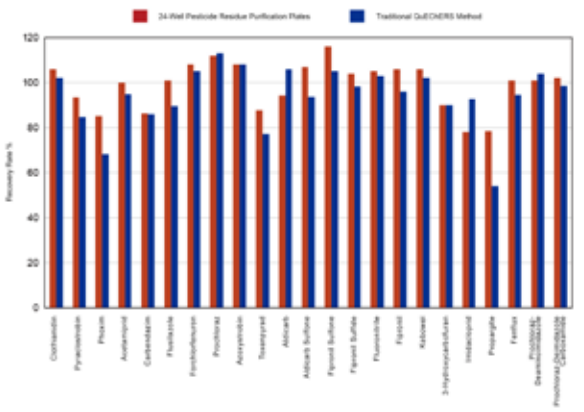


Figure 1. Comparison of Recovery of Multi-Pesticide Residues in Cabbage (5 ng/g)

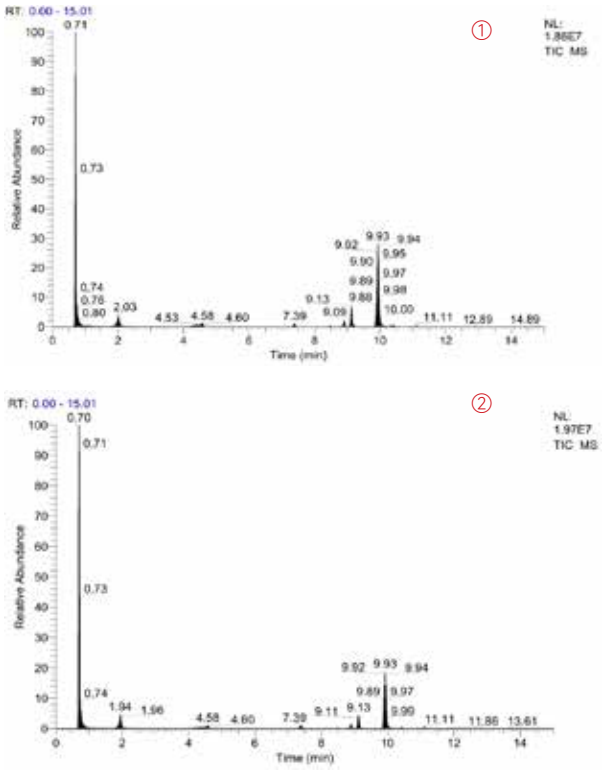


Figure 3. TIC of Multi-Pesticide Residues in Cabbage (5 ng/g)
① Purified Original QuEChERS Method
② Purified by Copure® 24-Well Pesticide Residue Purification Plates

Ordering Information

Cat. #	Description	Qty.
NC24001	Copure® 24-Well Pesticide Residue Purification Plates(suitable for vegetables, fruits and edible fungi with dark pigment)	1 Pc/Box
NC24002	Copure® 24-Well Pesticide Residue Purification Plates(suitable for vegetables, fruits and edible fungi with light pigment)	1 Pc/Box
NC24003	Copure® 24-Well Pesticide Residue Purification Plates (suitable for grains, oil and nuts)	1 Pc/Box
NC24004	Copure® 24-Well Pesticide Residue Purification Plates (suitable for tea and spices)	1 Pc/Box
COQ050010H	Extraction Salts (4 g MgSO4, 1 g NaCl, 1 g Trisodium Citrate, 0.5 g Disodium Citrate), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ050020H	Extraction Salts (6 g MgSO4, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
NC24DZ	24-Well Vial Tray	1 Set/Ctn.
BCY2402	Positive Pressure 24 Processor	1 Set/Ctn.
BCN2403	24 Well Intelligent Nitrogen Evaporator, Flat Bottom Plate	1 Set/Ctn.

High-Throughput of 28 Pesticide Residues in Strawberry and Lettuce (Copure® 24-Well Pesticide Residue Purification Plates)

Introduction

QuEChERS is widely used to detect pesticide residues in plant-based foods. Biocomma introduces the Copure® 24-Well Pesticide Residue Purification Plates enabling rapid, high-throughput analysis with Biocomma® Positive Pressure 24 Processor system and sample vial tray. No syringe filtration is required prior to LC-MS/MS analysis. Biocomma® established a high-throughput LC-MS/MS method with simplified operation and better result stability for detecting 28 pesticide residues in strawberries and lettuce. The recoveries were 60-110% with CV values below 10% at both 5 ng/g and 10 ng/g concentrations, which is close to traditional QuEChERS methods for your reference.

Experiment

Extraction (Copure® QuEChERS, Cat. #: COQ050010H)
Chop the samples and grind evenly. Weigh 10.0 g of sample into a 50 mL centrifuge tube. Add 10 mL of acetonitrile and vortex mix for 5 min. Add a pack of QuEChERS extraction salt and shake vigorously for 1 min. Vortex for 5 min, then centrifuge at 5000 r/min for 5 min. The upper acetonitrile layer is ready for purification.

Purification (Copure® 24-Well Pesticide Residue Purification Plates, Cat. #: NC24002)
Place the 24-well purification plate on a Biocomma® 24-Well Vial Tray. Add 2 mL of prepared sample. Place the purification plate and vial tray on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the sample vials. Screw on the caps and the sample is ready for analysis.

Instrument Conditions

1.Chromatography Conditions
Equipment: UPLC-MS/MS (Thermo Scientific TSQ Endura)
Chromatographic column: Commasil® BEH T-C18 (100 mm×2.1mm, 3μm)
Mobile phase: A: Water (0.1% formic acid), B: Methanol
Flow rate: 0.3 mL/min
Injection volume: 5 μL
Column temperature: 30 ℃

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.00	98	2
2.00	95	5
8.00	70	30
16.50	30	70
17.50	30	70
19.00	2	98
21.00	2	98
21.50	98	2
22.00	98	2

2.Mass Spectrometry Conditions

Source of ion: HESI
Electrospray voltage: 3500V
Sheath gas pressure: 40 Arb
Auxiliary air pressure: 1 Arb
Ion transfer tube: 380 ℃
Atomization temperature: 350 ℃

Results

Table 2. Experimental Results of Spiked Recovery

Targets	Spike (ng/g)	Sample							
		Lettuce				Strawberry			
		24-Well Purification Plate (n=8)		Original QuEChERS Method (n=8)		24-Well Purification Plate (n=8)		Original QuEChERS Method (n=8)	
		Average Recovery (%)	CV/%	Average Recovery (%)	CV/%	Average Recovery (%)	CV/%	Average Recovery (%)	CV/%
Methamidophos	5	94.5	5.74	96.3	10.8	91.3	5.74	95.1	8.68
	10	95.3	5.93	91.3	9.86	92.5	5.93	91.3	8.96
Monocrotophos	5	95.7	5.12	94.7	10.3	95.5	6.35	97.7	9.63
	10	96.1	5.31	93.1	10.1	95.1	6.21	99.1	9.51
Phosphamide	5	94.6	6.50	95.1	9.48	94.4	5.15	95.5	9.48
	10	95.5	6.69	96.5	9.46	95.2	5.59	99.5	9.46
Fenamiphos	5	97.2	8.16	96.2	9.27	96.2	9.16	95.2	9.27
	10	98.5	9.05	93.5	9.22	92.5	9.05	91.5	9.22
Fenamifos Sulfone	5	94.3	6.94	92.3	9.16	95.3	8.94	92.5	11.6
	10	99.5	6.83	93.1	9.11	99.1	8.83	93.1	10.5
Fenamiphos Sulfoxide	5	94.0	7.72	94.5	11.5	94.2	8.72	95.1	12.5
	10	91.8	7.61	89.0	12.0	95.2	8.61	95.0	12.5
Tefenthion	5	98.3	8.50	89.6	8.94	94.3	8.74	96.3	11.8
	10	91.5	8.39	90.7	8.89	90.3	8.93	90.3	9.96
Thiamiphos	5	96.1	6.28	92.8	9.83	95.7	9.12	94.7	10.8
	10	93.3	6.17	95.9	10.5	90.1	9.31	91.1	11.7
Amophos	5	90.2	7.06	97.7	7.61	95.6	9.50	92.1	12.8
	10	98.5	7.95	94.3	8.67	95.0	9.69	91.5	11.4
Pherophos	5	94.7	6.84	92.5	10.9	93.1	9.88	94.1	9.58
	10	92.5	6.73	90.2	11.6	95.1	9.27	95.1	9.43
Terbuphos	5	94.0	7.12	92.6	12.4	96.2	9.16	96.2	9.67
	10	93.3	7.41	94.3	11.9	97.5	9.05	92.5	9.25
Terbufos Sulfone	5	92.8	7.40	97.5	11.9	95.3	8.94	94.3	10.6
	10	96.3	7.51	90.3	10.5	97.1	8.83	93.1	11.1
Chlorsulfuron	5	65.6	7.39	60.7	8.89	64.3	8.74	61.3	9.68
	10	60.1	7.28	61.8	8.83	60.3	8.93	62.3	8.96
Metsulfuron-Methyl	5	60.3	7.17	62.9	12.5	64.7	9.12	64.5	9.63
	10	62.2	7.06	62.7	10.1	60.1	9.31	65.1	9.51
Phorate	5	98.5	6.95	89.3	10.7	95.6	9.50	94.5	12.5
	10	94.7	6.84	90.5	9.89	94.5	9.69	99.5	11.9
Phorate	5	92.5	7.73	91.2	12.4	93.1	9.88	94.1	11.5
	10	94.2	7.62	92.6	13.5	91.1	9.27	95.1	12.3
Phorate Sulfoxide	5	89.3	7.51	94.3	9.45	96.2	6.16	95.1	10.7
	10	89.8	7.40	97.5	9.39	92.5	6.05	92.5	11.2
Isofenphos-Methyl	5	90.3	8.51	91.3	10.5	95.3	4.94	91.3	9.66
	10	90.8	8.40	92.5	11.9	99.1	4.83	93.1	9.81
Demeton	5	91.4	8.50	97.6	10.4	94.0	5.72	92.0	11.5
	10	92.5	8.39	95.7	9.89	91.5	5.61	95.0	12.0
Kebowei	5	92.1	8.28	91.8	8.83	92.3	774	91.3	12.8
	10	94.3	8.17	94.9	9.75	91.3	7.93	92.3	11.6
3-Hydroxy Carbofuran	5	95.2	8.06	93.7	11.5	92.7	6.12	91.7	9.23
	10	98.5	6.95	94.3	12.4	94.1	7.31	95.1	9.31
Aldicarb	5	94.7	6.84	91.5	7.19	92.6	6.50	96.1	12.8
	10	92.5	6.73	91.2	8.56	95.5	7.69	99.5	11.6
Aldicarb Sulfone	5	75.4	6.62	72.6	9.45	73.1	8.08	69.2	12.5
	10	69.3	7.51	74.3	9.15	70.1	9.27	68.1	10.3
Aldicarb Sulfoxide	5	89.8	7.40	97.5	8.75	94.2	7.16	96.2	9.27
	10	96.3	7.51	90.3	8.62	92.5	7.05	90.5	9.52
Diphenphos	5	92.8	8.40	92.5	8.59	99.3	8.94	94.3	9.26
	10	94.7	8.54	91.5	8.31	99.1	8.83	93.5	9.51
Chlorazophos	5	89.5	5.73	90.2	9.42	94.0	8.72	95.4	9.65
	10	88.4	5.62	91.6	9.69	91.2	8.61	91.0	9.50
Isocarbophos	5	87.3	6.51	94.3	10.2	93.5	7.05	92.5	9.72
	10	92.8	7.40	97.5	9.39	94.3	6.94	94.3	9.76
Thiophos	5	91.3	7.51	94.3	7.45	99.1	6.83	93.1	11.1
	10	94.8	5.40	95.5	10.9	94.1	6.72	94.0	12.5

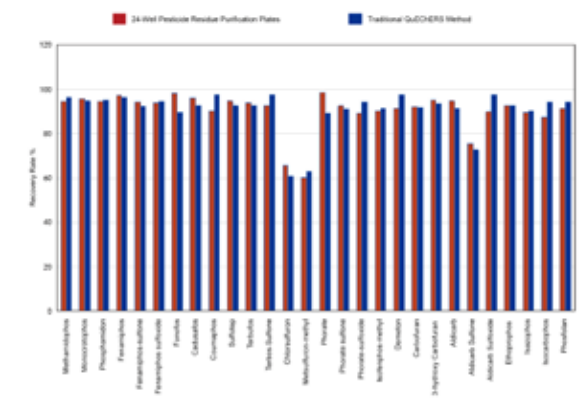


Figure 1. Comparison of Recovery of Multi-Pesticide Residues in Lettuce (5 ng/g)

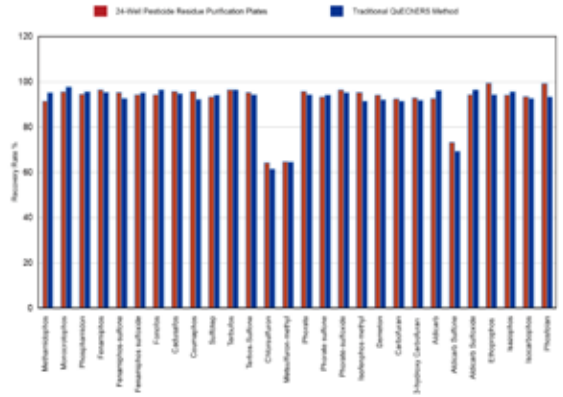


Figure 2. Comparison of Recovery of Multi-Pesticide Residues in Strawberry (5 ng/g)

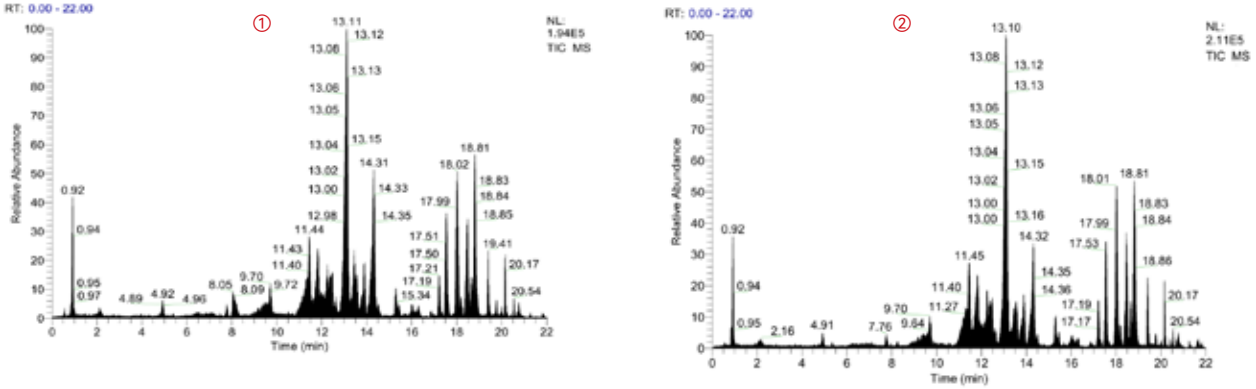


Figure 3. TIC of Multi-Pesticide Residues in Strawberry (5 ng/g)

① Purified Original QuEChERS Method

② Purified by Copure® 24-Well Pesticide Residue Purification Plates

Ordering Information

Cat. #	Description	Qty.
NC24001	Copure® 24-Well Pesticide Residue Purification Plates (suitable for vegetables, fruits and edible fungi with dark pigment)	1 Pc/Box
NC24002	Copure® 24-Well Pesticide Residue Purification Plates (suitable for vegetables, fruits and edible fungi with light pigment)	1 Pc/Box
NC24003	Copure® 24-Well Pesticide Residue Purification Plates (suitable for grains, oil and nuts)	1 Pc/Box
NC24004	Copure® 24-Well Pesticide Residue Purification Plates (suitable for tea and spices)	1 Pc/Box
COQ050010H	Extraction Salts (4 g $MgSO_4$, 1 g NaCl, 1 g Trisodium Citrate, 0.5 g Disodium Citrate), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ050020H	Extraction Salts (6 g $MgSO_4$, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
NC24DZ	24-Well Vial Tray	1 Set/Ctn.
BCY2402	Positive Pressure 24 Processor	1 Set/Ctn.
BCN2403	24 Well Intelligent Nitrogen Evaporator, Flat Bottom Plate	1 Set/Ctn.

High-Throughput of 26 Pesticide Residues in Corn (Copure® 24-Well Pesticide Residue Purification Plates)

Introduction

QuEChERS is widely used to detect pesticide residues in plant-based foods. Biocomma introduces the Copure® 24-Well Pesticide Residue Purification Plates enabling rapid, high-throughput analysis with Biocomma® Positive Pressure 24 Processor system and sample vial tray. No syringe filtration is required prior to LC-MS/MS analysis. Biocomma® established a high-throughput LC-MS/MS method with simplified operation and better result stability for detecting 26 pesticide residues in corn. The recoveries were 60-120% with CV values below 10% at both 1 ng/g and 5 ng/g concentrations, which is close to traditional QuEChERS methods for your reference.

Experiment

Extraction (Copure® QuEChERS, Cat. #: COQ050020H)
Chop the samples and grind evenly. Weigh 5.0 g of sample into a 50 mL centrifuge tube. Add 15 mL of acetonitrile (1% acetic acid) and vortex mix for 5 min. Add a pack of QuEChERS extraction salt and shake vigorously for 1 min. Vortex for 5 min, then centrifuge at 5000 r/min for 5 min. The upper acetonitrile layer is ready for purification.

Purification (Copure® 24-Well Pesticide Residue Purification Plates, Cat. #: NC24003)
Place the 24-well purification plate on a Biocomma® 24-Well Vial Tray. Add 2 mL of prepared sample. Place the purification plate and vial tray on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the sample vials. Screw on the caps and the sample is ready for analysis.

Instrument Conditions

1.Chromatography Conditions

Equipment: UPLC-MS/MS (Thermo Scientific TSQ Endura)

Chromatographic column: Commasil® BEH T-C18 (100 mm×2.1mm, 3μm)

Mobile phase: A: Water (0.1% formic acid), B: Methanol

Flow rate: 0.4 mL/min

Injection volume: 5 μL

Column temperature: 30 ℃

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.00	98	2
1.00	95	5
4.00	70	30
8.00	30	70
9.00	30	70
10.00	2	98
13.50	2	98
14.00	98	2
15.00	98	2

2.Mass Spectrometry Conditions

source of ion: HESI

Electrospray voltage: 3500V

Sheath gas pressure: 40 Arb

Auxiliary air pressure: 1 Arb

Ion transfer tube: 380 ℃

Atomization temperature: 350 ℃

Results

Table 2. Experimental Results of Spiked Recovery

Targets	Spike (ng/g)	Sample			
		Corn			
		24-Well Purification Plate (n=8)		Original QuEChERS Method (n=8)	
		Average Recovery (%)	CV/%	Average Recovery (%)	CV/%
Clothianidin	1	94.3	1.10	101	2.84
	5	98.9	1.62	98.2	3.32
Pyraclostrobin	1	98.7	1.60	86.0	4.28
	5	93.2	3.89	82.6	3.87
Thiamethoxam	1	85.2	7.51	84.7	8.65
	5	106	8.87	97.1	6.67
Phoxim	1	103	2.36	74.7	3.68
	5	92.4	3.60	78.5	15.1
Acetamiprid	1	75.7	6.48	87.5	5.77
	5	105	4.76	101	4.12
Dimethomorph	1	95.4	6.69	54.7	7.02
	5	98.4	3.58	78.5	4.46
Carbendazim	1	78.2	9.85	70.5	11.5
	5	93.9	5.36	98.2	12.4
Flusilazole	1	98.2	5.84	88.2	10.7
	5	95.0	2.68	97.1	4.11
Forchlorfenuron	1	88.6	4.62	90.1	7.11
	5	103	3.53	103	1.47
Prochloraz	1	99.6	5.20	66.0	16.3
	5	83.9	5.90	81.6	5.80
Thiophanate-Methyl	1	112	3.04	124	4.93
	5	117	1.45	128	2.27
Azoxystrobin	1	99.6	4.97	100	6.15
	5	95.1	6.26	94.8	7.29
Toxenpyrad	1	68.5	8.76	70.4	11.2
	5	92.5	1.66	77.1	16.5

Aldicarb	1	67.1	8.69	79.8	13.4
	5	100	6.70	102	8.84
Aldicarb Sulfone	1	103	2.13	99.5	6.79
	5	115	3.02	105	5.35
Fipronil Sulfone	1	98.1	4.61	103	16.1
	5	99.8	2.10	97.9	6.49
Fipronil Sulfide	1	72.5	6.41	63.2	16.3
	5	98.5	3.85	84.7	7.50
Fluoronitrile	1	87.9	3.28	121	7.13
	5	99.9	1.75	97.1	6.34
Fipronil	1	99.1	3.02	80.6	10.5
	5	106	1.75	98.4	11.1
Kebowei	1	80.4	9.92	75.7	7.98
	5	102	3.25	96.8	2.57
3-Hydroxycarbofuran	1	93.1	7.44	82.5	16.9
	5	97.8	4.93	103	2.65
Imidacloprid	1	87.7	9.91	56.5	15.1
	5	80.1	7.73	88.8	14.8
Propargite	1	85.3	9.72	50.5	14.7
	5	88.4	5.02	61.5	15.6
Fenflux	1	68.9	6.05	51.6	17.1
	5	87.6	3.61	77.2	4.75
Prochloraz-Deaminoimidazole	1	114	3.12	98.7	4.45
	5	98.2	9.85	84.3	9.01
Prochloraz-Deimidazole Carboxamide	1	105	3.30	107	2.10
	5	92.6	3.36	98.4	1.49

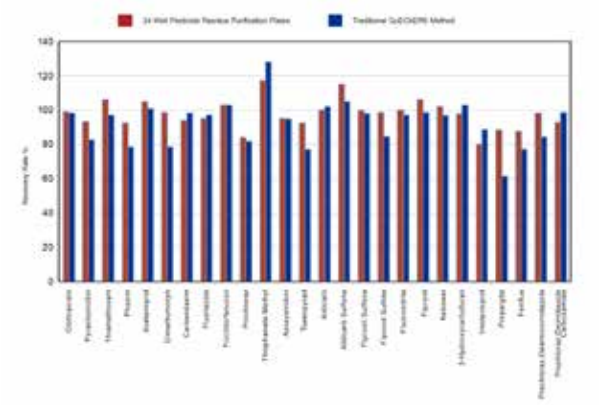


Figure 1. Comparison of Recovery of Multi-Pesticide Residues in Corn (5 ng/g)

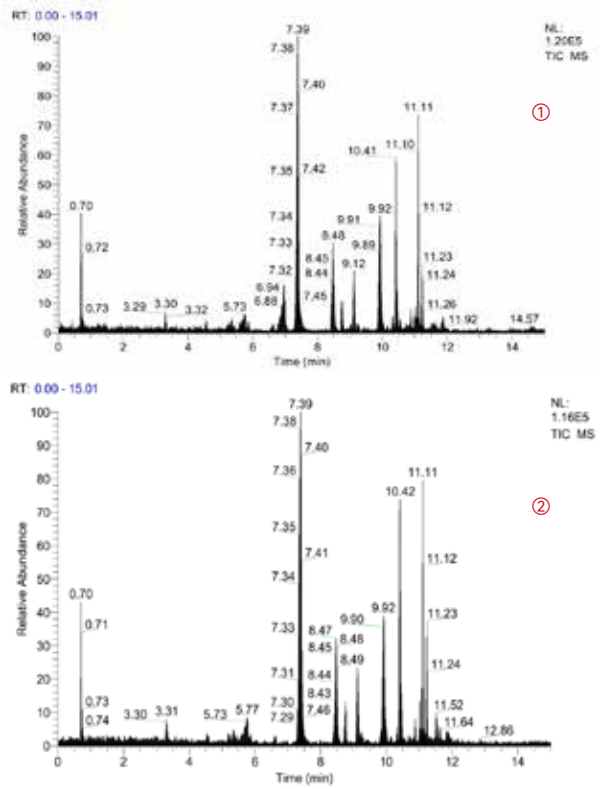


Figure 3. TIC of Multi-Pesticide Residues in Corn (5 ng/g)
① Purified Original QuEChERS Method
② Purified by Copure® 24-Well Pesticide Residue Purification Plates

Ordering Information

Cat. #	Description	Qty.
NC24001	Copure® 24-Well Pesticide Residue Purification Plates (suitable for vegetables, fruits and ediblefungi with dark pigment)	1 Pc/Box
NC24002	Copure® 24-Well Pesticide Residue Purification Plates (suitable for vegetables, fruits and edible fungi with light pigment)	1 Pc/Box
NC24003	Copure® 24-Well Pesticide Residue Purification Plates (suitable for grains, oil and nuts)	1 Pc/Box
NC24004	Copure® 24-Well Pesticide Residue Purification Plates (suitable for tea and spices)	1 Pc/Box
COQ050010H	Extraction Salts (4 g MgSO4, 1 g NaCl, 1 g Trisodium Citrate, 0.5 g Disodium Citrate), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ050020H	Extraction Salts (6 g MgSO4, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
NC24DZ	24-Well Vial Tray	1 Set/Ctn.
BCY2402	Positive Pressure 24 Processor	1 Set/Ctn.
BCN2403	24 Well Intelligent Nitrogen Evaporator, Flat Bottom Plate	1 Set/Ctn.

High-Throughput of 25 Pesticide Residues in Tea (Copure® 24-Well Pesticide Residue Purification Plates)

Introduction

QuEChERS is widely used to detect pesticide residues in plant-based foods. Biocomma introduces the Copure® 24-Well Pesticide Residue Purification Plates enabling rapid, high-throughput analysis with Biocomma® Positive Pressure 24 Processor system and sample vial tray. No syringe filtration is required prior to LC-MS/MS analysis. Biocomma established a high-throughput LC-MS/MS method with simplified operation and better result stability for detecting 25 pesticide residues in tea. The recoveries were 50-120% with CV values below 10% at both 1 ng/g and 5 ng/g concentrations, which is close to traditional QuEChERS methods for your reference.

Experiment

Extraction (Copure® QuEChERS, Cat. #:COQ050020H) Weigh 2.0 g of ground sample into a 50 mL centrifuge tube. Add 10 mL of water, vortex mix, and let stand for 30 min. Add 15 mL of acetonitrile (1% acetic acid) and vortex for 5 min. Then add a pack of QuEChERS extraction salt and shake vigorously for 1 min. Vortex for 5 min, then centrifuge at 5000 r/min for 5 min. The upper acetonitrile layer is ready for purification.

Purification (Copure® 24-Well Pesticide Residue Purification Plates, Cat. #: NC24004)

Place the 24-well purification plate on a Biocomma® 24-Well Vial Tray. Add 2 mL of prepared sample. Place the purification plate and vial tray on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the sample vials. Screw on the caps and the sample is ready for analysis.

Instrument Conditions

1.Chromatography Conditions

Equipment: UPLC-MS/MS (Thermo Scientific TSQ Endura)

Chromatographic column: Commasil® BEH T-C18 (100*2.1 mm,3 μm)

Mobile phase: A: Water (0.1% formic acid), B: Methanol

Flow rate: 0.4 mL/min

Injection volume: 5 μL

Column temperature: 30 ℃

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.00	98	2
1.00	95	5
4.00	70	30
8.00	30	70
9.00	30	70
10.00	2	98
13.50	2	98
14.00	98	2
15.00	98	2

2.Mass Spectrometry Conditions

source of ion: HESI

Electrospray voltage: 3500V

Sheath gas pressure: 40 Arb

Auxiliary air pressure: 1 Arb

Ion transfer tube: 380 ℃

Atomization temperature: 350 ℃

Results

Table 2. Experimental Results of Spiked Recovery

Targets	Spike (ng/g)	Sample			
		Tea			
		24-Well Purification Plate (n=8)		Original QuEChERS Method (n=8)	
		Average Recovery (%)	CV/%	Average Recovery (%)	CV/%
Clothianidin	1	----	----	----	----
	5	60.1	6.27	49.2	2.41
Pyraclostrobin	1	82.8	8.47	93.7	11.5
	5	73.1	9.88	84.4	5.22
Thiamethoxam	1	110	9.15	111	14.1
	5	109	8.25	93.0	13.5
Phoxim	1	93.3	1.44	88.4	12.4
	5	83.6	5.58	85.9	2.72
Acetamiprid	1	81.3	6.75	82.4	14.6
	5	98.1	9.64	85.8	12.3
Dimethomorph	1	86.3	9.22	86.8	10.4
	5	104	4.36	101	4.97
Carbendazim	1	74.6	8.35	76.1	7.16
	5	85.1	9.75	79.4	8.84
Flusilazole	1	89.2	7.48	86.1	4.45
	5	104	3.89	103	1.51
Forchlorfenuron	1	----	----	----	----
	5	61.0	9.95	50.2	2.02

Prochloraz	1	82.6	4.77	85.5	13.1
	5	76.1	5.61	76.9	4.18
Thiophanate-Methyl	1	65.3	8.75	65.2	8.34
	5	76.5	9.49	77.1	5.41
Azoxystrobin	1	98.5	9.11	87.2	11.9
	5	88.4	8.61	87.1	8.13
Toxenpyrad	1	80.1	9.72	86.4	6.65
	5	85.1	7.41	78.5	7.13
Aldicarb	1	95.9	9.91	96.1	11.2
	5	102	7.56	95.3	3.57
Fipronil Sulfone	1	107	8.83	92.3	12.3
	5	113	9.24	112	2.49
Fipronil Sulfide	1	117	7.34	122	11.8
	5	101	5.35	107	8.28
Fluoronitrile	1	96.9	9.85	103	10.7
	5	93.2	8.75	97.3	3.71
Fipronil	1	114	9.17	116	6.00
	5	90.1	8.86	93.3	3.21
Kebowei	1	64.5	8.75	57.5	12.8
	5	89.2	8.22	84.3	3.63
3-Hydroxycar-bofuran	1	64.8	6.00	52.1	13.7
	5	61.1	9.59	51.1	11.5
Imidacloprid	1	90.4	7.65	85.6	7.65
	5	113	8.96	96.7	11.1
Propargite	1	72.9	9.37	51.3	10.8
	5	80.1	6.67	56.1	13.6
Fenflux	1	----	----	----	----
	5	51.8	9.69	51.9	3.64
Prochloraz-Deaminoimidazole	1	96.6	9.52	105	7.89
	5	92.1	3.77	91.9	7.27
Prochloraz-Deimidazole Carboxamide	1	82.1	8.92	76.7	7.14
	5	91.2	6.94	96.7	5.01

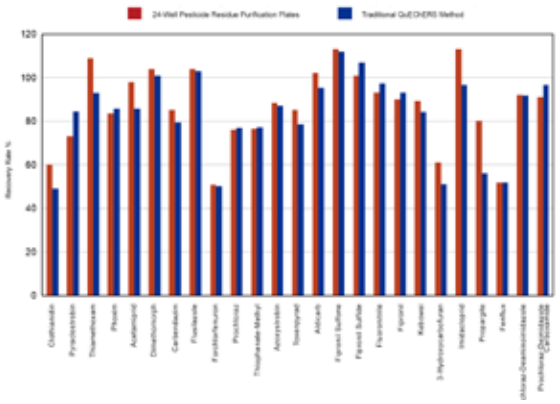


Figure 1. Comparison of Recovery of Multi-Pesticide Residues in Tea (5 ng/g)

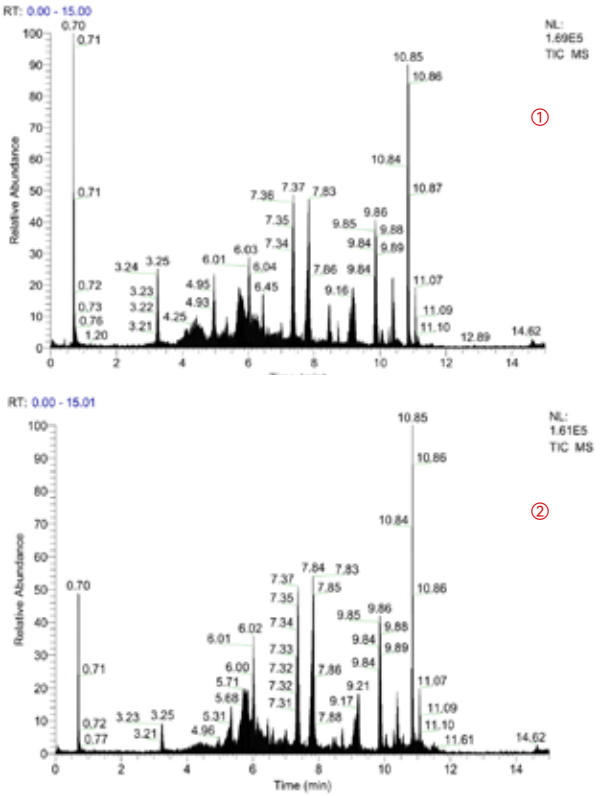


Figure 2. TIC of Multi-Pesticide Residues in Tea (5 ng/g)
① Purified Original QuEChERS Method
② Purified by Copure® 24-Well Pesticide Residue Purification Plates

Ordering Information

Cat. #	Description	Qty.
NC24001	Copure® 24-Well Pesticide Residue Purification Plates (suitable for vegetables, fruits and edible fungi with dark pigment)	1 Pc/Box
NC24002	Copure® 24-Well Pesticide Residue Purification Plates (suitable for vegetables, fruits and edible fungi with light pigment)	1 Pc/Box
NC24003	Copure® 24-Well Pesticide Residue Purification Plates (suitable for grains, oil and nuts)	1 Pc/Box
NC24004	Copure® 24-Well Pesticide Residue Purification Plates (suitable for tea and spices)	1 Pc/Box
COQ050010H	Extraction Salts (4 g MgSO4, 1 g NaCl, 1 g Trisodium Citrate, 0.5 g Disodium Citrate), 50 mL Tube, Ceramic Homogenizers	50 Pcs/Box
COQ050020H	Extraction Salts (6 g MgSO4, 1.5 g NaOAc), 50 mL Tube	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
NC24DZ	24-Well Vial Tray	1 Set/Ctn.
BCY2402	Positive Pressure 24 Processor	1 Set/Ctn.
BCN2403	24 Well Intelligent Nitrogen Evaporator, Flat Bottom Plate	1 Set/Ctn.

Solid Phase Extraction (SPE) of Aldicarb and Its Metabolites in Cabbage

Introduction

Aldicarb and its metabolites are kinds of carbamate pesticides. Carbamate pesticides, as well as Organophosphorus pesticides, can inhibit the cholinesterase of human beings, thus affecting the body's nerve impulse transmission. These pesticides affecting human health seriously. This method was applied to aldicarb and its metabolites using Copure NH_2 cartridge and suitable for LC-MS/MS analysis.

Materials

Biocomma®Copure®NH_2 SPE cartridge, 500 mg/3 mL.(Cat. No.CONH3500).

Experiment

Sample Extraction

Weigh 5 g cabbage sample (accurate to 0.01g) into a 50 mL centrifuge tube. Add 20 mL acetonitrile.

Vortex for 2 minutes and shake well for 20 minutes. Pass the supernatant through anhydrous sodium sulfate and collect in a separatory funnel. Add 20 mL acetonitrile, repeat the same extraction operation one more time. Evaporate the acetonitrile to dryness below 40℃ , redissolve with 2 mL 1:99 methanol/dichloromethane.

SPE Cleanup

Condition: Condition the NH_2 cartridge with 5 mL 1:99 methanol/dichloromethane.

Load: Load 2 mL sample into cartridge, control the flow rate within 1 mL/min, and collect the effluent;

Elute: Elute with 5 mL 1:99 methanol/dichloromethane. Combine the effluent from load and elute steps;

Reconstitute: Evaporate the effluent to dryness under nitrogen and reconstitute with 1 mL mobile phase.

LC-MS/MS Condition

Chromatographic column: Venusil ASB C18(2.1 mm×150 mm, 5μm)

Mobile phase: A: Acetonitrile; B: 0.05% Formic acid aqueous

Table 1. Mobile phase gradient elution procedure

Time (min)	A(%)	B(%)
0.00	10	90
1.50	10	90
4.00	100	0
8.00	100	0
8.01	10	90
13.00	10	90

Flow Rate: 0.3 mL/min

Column Temperature: 30 ℃

Mass Spectrometer: API 4000

Ion source: ESI

Scan mode: Positive ion

Detection method: MRM

Results and Discussion

During this study, the average recoveries were approximately 90% for cabbage. The RSD(Relative Standard Deviation) were less than 5%,which means a excellent reproducibility. The recovery results were shown in Table 2, and the chromatogram were shown in Figure 2 - Figure 4.

Table 2. Recoveries of cabbage(10μg/kg)

Name	Recoveries(%)			Recovery(%)	RSD(%)
	1	2	3		
Aldicarb	89.23	96.74	92.86	92.94	4.05
Aldicarb sulfone	94.65	86.96	92.35	91.32	4.33
Aldicarb sulfoxide	94.63	93.26	89.98	92.62	2.58

Chromatograms of Aldicarb and its metabolites

Figure 1. Chromatogram of Aldicarb and its metabolites

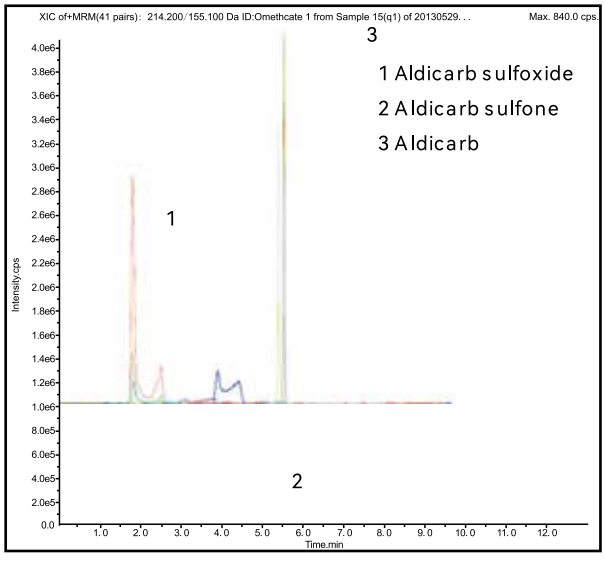


Figure 2. Chromatogram of Aldicarb

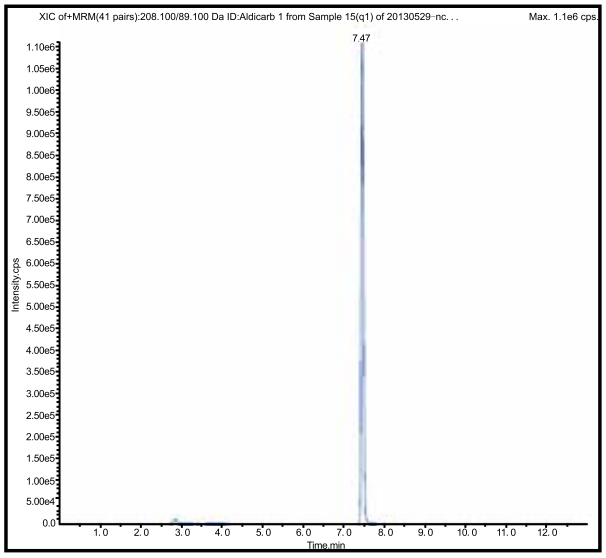


Figure 3. Chromatogramof Aldicarb sulfone

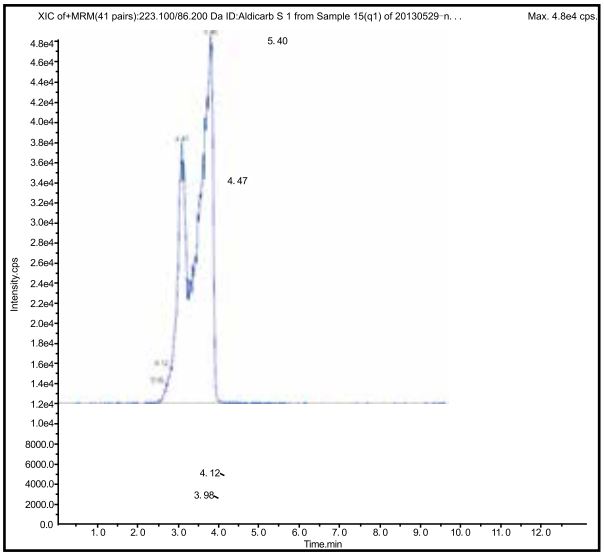
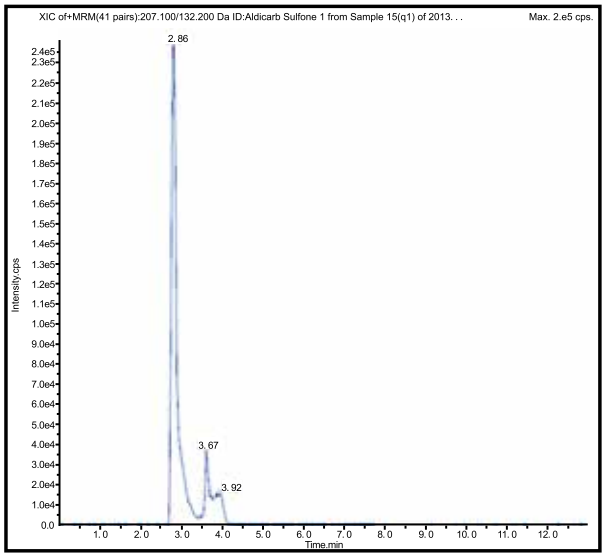


Figure 4. Chromatogram of Aldicarb sulfoxide



Conclusion

There is a need for a rapid, sensitive method for the analysis of carbamate pesticides in fruits and vegetables. This work describes a method that employs a specific solid phase extraction of aldicarb and its metabolites, using Copure NH2. This method can simplify the sample preparation procedure, and save more solvent.

Ordering Information

Cat. #	Description	Qty.
CONH3500	Copure® NH_2 Cartridge, 500 mg/ 3 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Solid Phase Extraction (SPE) of Pyrethroid pesticides in lettuce

Introduction

Pyrethroid pesticides are widely used insecticides, although they are less toxic, but when used for a long-term, they will make a variety of pests resistant. This method was applied to Pyrethroid pesticides use Copure Florisil cartridge and suitable for GC-ECD analysis

Materials

Biocomma® Copure® Florisil SPE 1g/6mL

Experiment

Sample Extraction

Weigh 10 g sample (accurate to 0.01g) into a 50 mL centrifuge tube. Add 20 mL acetonitrile. Vortex for 2 minutes. Add 5 - 7 g NaCl, then close the lid and shake well for 5 minutes. Stand for 10 min at room temperature, then centrifuge for 4 minutes at 5000 r/m. After separation, take the upper acetonitrile layer to be clean up.

SPE Cleanup

Condition: Condition the Florisil SPE cartridge with 5 mL 1:99 acetone/hexane;

Load & Elute: Pass 4 mL of the extraction through the cartridge and collect the effluent, wash the cartridge with 10 mL 1:99 acetone/hexane, combine the effluent together;

Reconstitute: Evaporate the elution to dryness under nitrogen and reconstitute with 1 mL hexane.

GC-ECD Condition

Chromatographic column: HP-5 (30 m×0.32 mm×0.25 μm)

Inlet Temperature: 220 ℃

Detector Temperature: 300 ℃

Column Temperature:

100 ℃ (1 min), 20 ℃ /min to 160 ℃ (3 min)

25 ℃ /min to 200 ℃ (4 min), 8 ℃ /min to 240 ℃ (4 min)

25 ℃ /min to 280 ℃ (3 min)

Carry Gas: N2

Flow Rate: 1mL/min;

Injection volume: 1 μL

Split 10:1

Results and Discussion

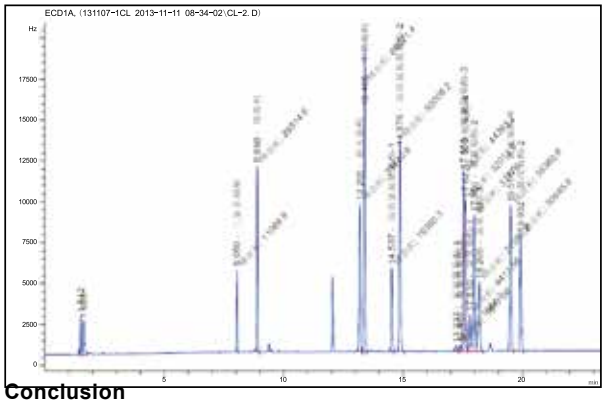
Took lettuce as the sample, eight kinds of pyrethroid pesticides were tested in this study, and the spiked level is 0.5 mg/kg. All compounds were recovered at greater than 90%. The recovery results were shown in Table 2, and the chromatogram was shown in Figure 1.

Table 1. Recoveries of pyrethroid pesticides spiked at 0.5 mg/kg in lettuce

Name	Spiked content(0.5 mg/kg) recovery (%)	RSD(%)
Dicofol	0.5	93.40
Procymidone	0.5	89.35
Bifenthrin	0.5	101.25
Cypermethrin	0.5	90.37
Cyhalothrin	0.5	98.74
Cyfluthrin	0.5	92.38
Cypermethrin	0.5	105.52
Fenvalerate	0.5	103

Chromatogram of pyrethroid pesticides

Figure 1. Chromatogram of pyrethroid pesticides



Conclusion

biocomma® Copure® Florisil is an efficient and accurate determination of pyrethroid pesticide residues in fruits and vegetables. This method can simplify the sample preparation procedure, and save more time and solvent. in Figure 2 - Figure 4.

Ordering Information

Cat. #	Description	Qty.
COFL61000	Copure® Florisil Cartridge, 1000 mg/ 6 mL	30 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Solid Phase Extraction (SPE) of Carbamate Pesticides in Chives

Introduction

Carbamate pesticides have been used in vegetables and fruits cultivation. Unfortunately, some carbamates, such as aldicarb, are highly toxic. Many countries and organization pay much attention to the test of carbamate Pesticides. This application note describes a reliable method to determine the residue levels of carbamates in chives.

Materials

Biocomma® Copure® NH_2 SPE cartridge 1g/6mL (Cat. No. CONH61000).

Experiment

Sample Extraction

Accurately weigh 10 g chives sample into a 50 mL centrifuge tube, add 20mL acetonitrile and vortex for 2 minutes. Add 5-7g sodium chloride into the 50 mL centrifuge tube, then shake vigorously for 5 minutes. Centrifuge at 5000 rpm for 4 minutes, then stand for 10 minutes at room temperature. After phase separation, take 2 mL supernatant of CH3CN layer for SPE cleanup.

SPE Cleanup

Condition: Condition the biocomma Copure NH_2 SPE cartridge with 5 mL methanol/dichloromethane (1:99, v/v).

Load: Load 2 mL upper CH_3CN phase into cartridge and control the flow rate within 1 mL/min, and collect the effluent.

Elute: Elute with 5 mL methanol/dichloromethane (1:99, v/v). Collect the elution and combine the load and elute effluent.

Reconstitute: Evaporate the effluent to dryness under nitrogen at 40 ℃ and reconstitute with 1 mL of 0.1% formic acid in water/ acetonitrile (90:10). The solution is filtered through a syringe filter (PTFE, 0.22 μm, 2.5 mm) and transfer it into a 2 mL vial for LC-MS/MS analysis.

LC-MS/MS CONDITION

Mass spectrometer: AB API4000

Column: C18 (2.1 mm×150 mm, 5μm) or equivalent

Mobile phase: A: Acetonitrile; B: 0.1% Formic acid aqueous solution

Elution mode: Gradient elution (as in Table 1)

Table 1. Gradient program

Time(min)	A (%)	B (%)
0.00	10	90
6.00	90	10
8.00	90	10
8.01	10	90
15.00	10	90

Flow rate: 0.3 mL/min

Column temperature: 30 °C

Injection volume: 20 μL

Ion source: ESI

Ionization mode: Positive

Scan mode: MRM

Ion source parameters conditions are listed as in Table 2.

Source/Gas	
Collision Gas(CAD)	10
Curtain Gas(CUR)	30
Ion Source Gas 1(GS1)	50
Ion Source Gas 2(GS2)	50
Ion Spray Voltage(IS)	5500
Temperature(TEM)	550
Interface Heater(ihe)	On

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

Compound	RT (min)	MRM channels (m/z)	DP (V)	EP (V)	CE (V)	CXP (V)
Carbofuran	3.89	222.3>123.1	48	10	16	12
		222.3>165.2	48	10	31	12
Methomyl	2.83	163.2>88.1	36	10	15	12
		163.2>106.1	36	10	12	12
Propoxur	3.86	210.2>186.3	60	10	23	12
Carbendazim	2.61	192.1>160.1	68	10	34	12
		192.1>132.2	68	10	42	12

Results and Discussion

The recovery results are shown in Table 4, and the chromatogram of spiked chives extracts (0.5 mg/kg) are shown in Figure 1-Figure 5.

Name	Recoveries(%)			Average Recovery (%)
	1	2	3	
Carbofuran	95.26	95.77	91.72	94.25
Methomyl	93.58	96.58	92.98	94.38
Propoxur	105.12	102.25	104.21	103.86
Carbendazim	100.26	104.25	100.86	101.79

Chromatogram of four carbamate pesticides spiked at 0.5 mg/kg in chives

Figure 1. Chromatogram of four carbamate pesticides spiked at 0.5 mg/kg in chives

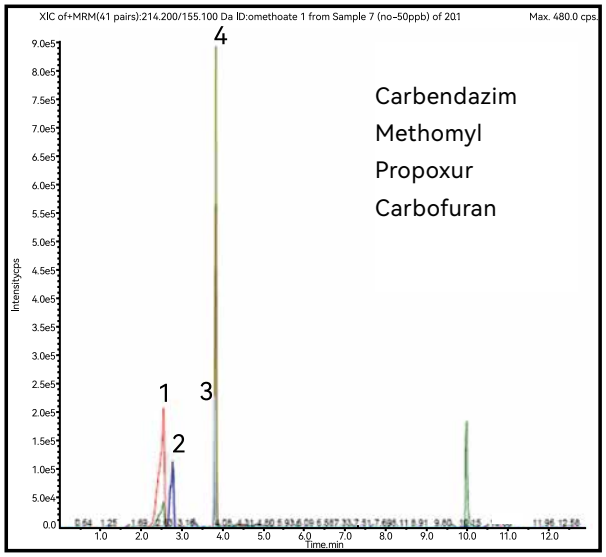


Figure 2. Chromatogram of Methomyl spiked at 0.5 mg/kg in chives

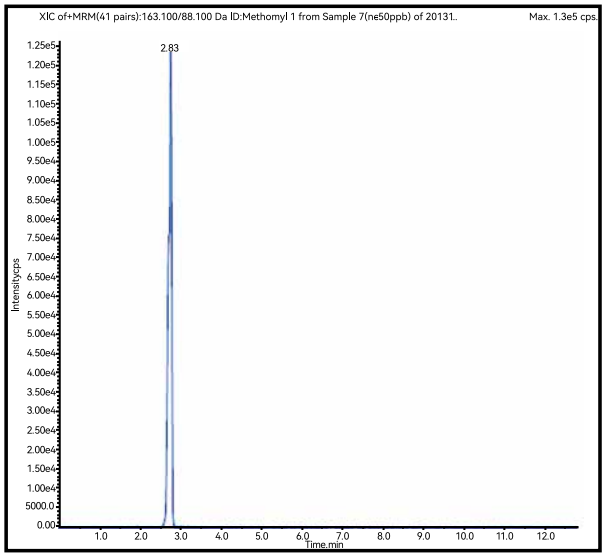


Figure 3. Chromatogram of Carbendazim spiked at 0.5 mg/kg in chives

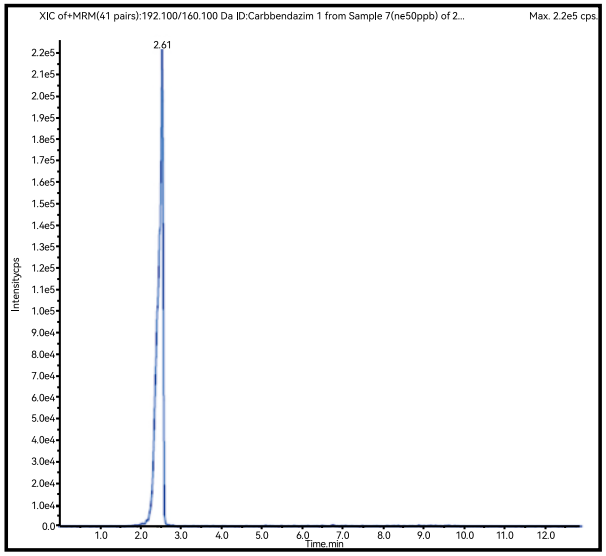


Figure 4. Chromatogram of Carbofuran spiked at 0.5 mg/kg in chives

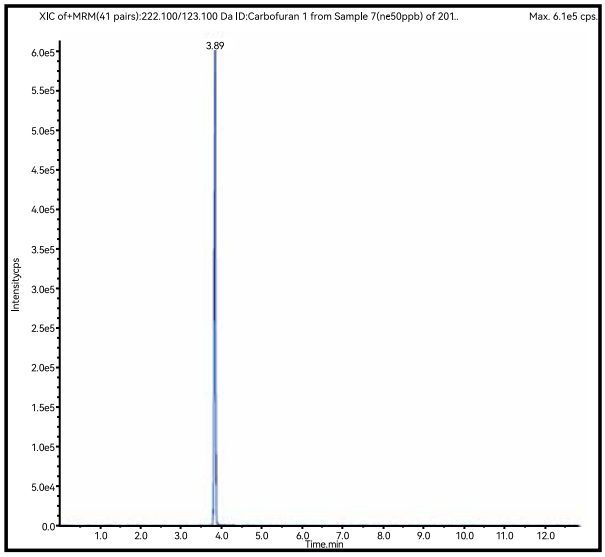
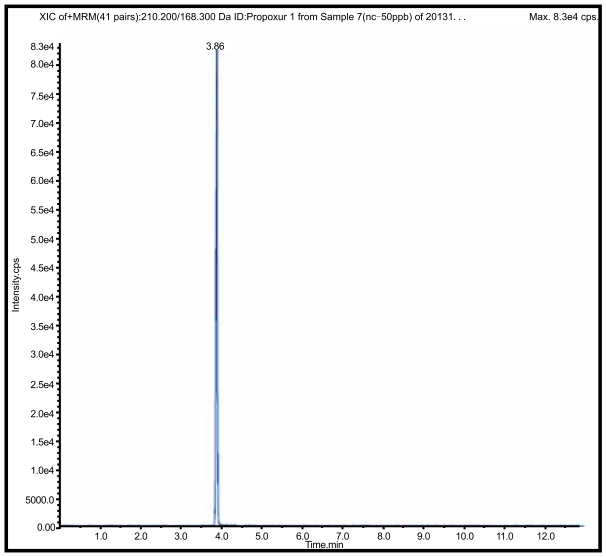


Figure 5. Chromatogram of Propoxur spiked at 0.5 mg/kg in chives



Conclusion

This application note describes a method that employs a specific solid phase extraction of carbamate pesticides using biocomma Copure NH2 SPE cartridge. The recovery results based on matrix spiked standards are excellent for carbamate pesticides determination in chives.

Ordering Information

Cat. #	Description	Qty.
CONH61000	Copure® NH_2 Cartridge, 1000 mg/ 6 mL	30 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Solid Supported Liquid Liquid Extraction(SLE) of Certain Aromatic Amines Derived from Azo Dyes in Textiles

Experiment purpose

Certain aromatic amines derived from banned AZO dyes have been shown to be carcinogenic to humans. For this reason, specific regulations have been introduced in order to monitor their presence in textiles and leathers. This application note uses biocomma Copure AZO Dye SLE cartridge to extract certain aromatic amines derived from banned azo dyes and analyze by GC-MS.

Materials

Biocomma® Copure® AZO Dye SLE cartridge (Cat. No. COAZO060).

Experiment

Sample Extraction

Textile sample is cut in an appropriate manner. Accurately weigh 1 g (accurate to 0.01g) into a 50 mL reaction vessel. Add 17 mL citrate buffer solution preheated to 70 ℃ to the reaction vessel , and then close the vessel tightly. After brief and vigorously shaking, the reaction solution is kept for 30 minutes at (70±2)℃ . All fibers should be wetted.

Subsequently, 3.0 mL aqueous sodium dithionite solution was added to the reaction solution for reductive cleavage of the AZO groups, shake vigorously and immediately kept at (70±2)℃ for another (30±1) minutes, where upon it is cooled to room temperature (20℃ to 25℃) within 2 minutes.

SPE Cleanup

Load: Using a glass pestle, the reaction solution is squeezed out of the fibers, decanted on the biocomma® Copure AZO Dye SLE cartridge, and allowed to be absorbed for 15 minutes.

Elute: Elute with 80 mL ethyl ether. Collect the solvent.

Reconstitute: The ethyl ether extract is concentrated to less than 1 mL (not to dryness) in a rotary vacuum evaporator in a slight vacuum at 35 ℃ . Remove the remainder of the solvent very carefully without vacuum by means of a weak flow of inert gas. Exchange the solvent to 1.0 mL toluene. The solution was filtered through a syringe filter (PTFE, 0.45 µm, 0.25 mm) and transferred into a 2 mL vial for analysis by GC-MS.

GC-MS CONDITION

System: Agilent 7890A

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

Inlet Temperature: 220 ℃

Detector Temperature: 300 ℃

Column Temperature: 40 ℃ (0.5 min),

20 ℃ /min to 150 ℃ (1.5 min),

15 ℃ /min to 210 ℃ (0.5 min),

10 ℃ /min to 250 ℃ (3.0min),

Carry Gas: He

Flow Rate: 0.9 mL/min;

Table 1. Five aromatic amines, retention time and ion for quantitation and quantification

Compound	Time (min)	Ion for quantitation	Ion for quantification
O-toluidine	5.492	106.1	107.1,77,79.1
4-chloroaniline	6.508	127	129.65.1,92
2,4-Diaminotoluene	8.367	112.1	121.1,105,94
4-methoxy-m-phenylenediamine	9.307	123.1	138,95.1,95.8
3,3 ' -dimethylbenzidine	17.110	212	196,106

Results and Discussion

The recovery results were shown in Table 2, and the chromatograms of spiked textile extracts (10 mg/kg) were shown in Figure 1-Figure 6.

Table 2. Recoveries of five aromatic amines spiked at 10 mg/kg in textile

Compound	Time (min)	Ion for quantitation
O-toluidine	5.492	80.85
4-chloroaniline	6.508	83.77
2,4-Diaminotoluene	8.367	62.25
4-methoxy-m-phenylenediamine	9.307	53.00
3,3 ' -dimethylbenzidine	17.110	89.04

Figure 1. Chromatogram of five aromatic amines spiked at 10 mg/kg in aromatic amines

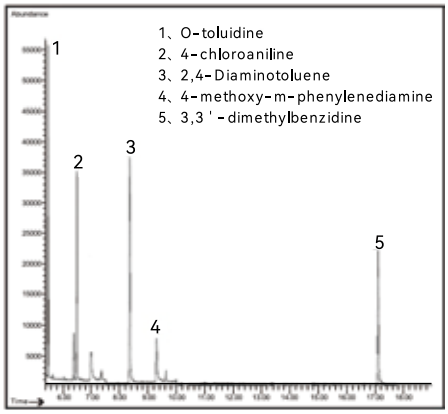


Figure 2. Chromatogram of 3,3'-dimethylbenzidine spiked at 10 mg/kg

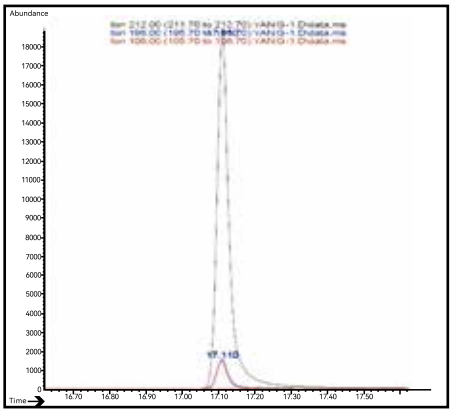


Figure 3. Chromatogram of O-toluidine spiked at 10 mg/kg

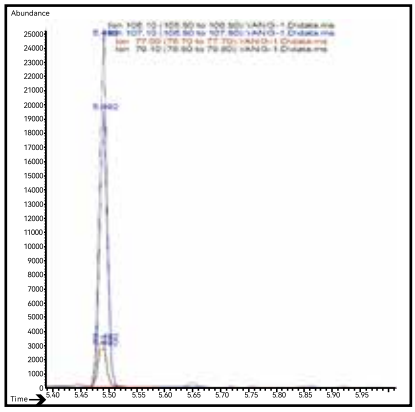


Figure 4. Chromatogram of 4-chloroaniline spiked at 10 mg/kg

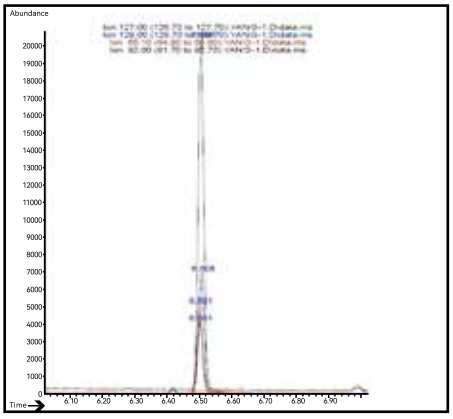


Figure 5. Chromatogram of 2,4-diaminotoluene spiked at 10 mg/kg

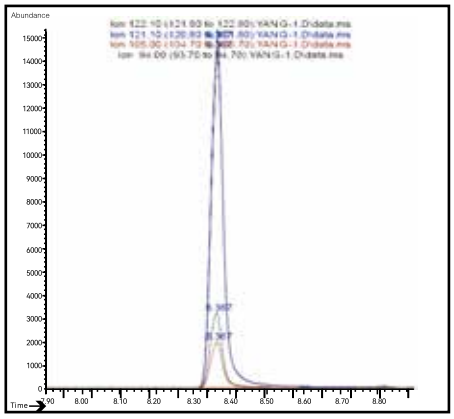
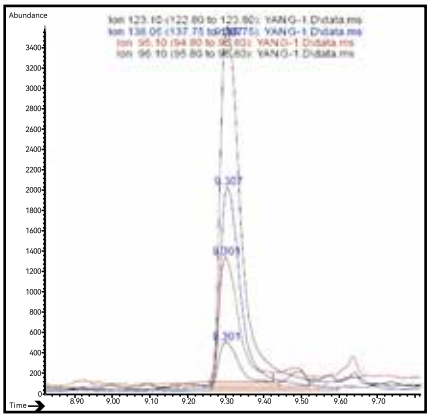


Figure 6. Chromatogram of 4-methoxy-m-phenylen ediamine spiked at 10 mg/kg



Conclusion

The results of this study shown that biocomma Copure AZO Dye SLE cartridge can be used as an effective method for purification of aromatic amines in textile.The recovery results based on matrix spiked standard were good for certain aromatic amines determination in textile.

Ordering Information

Cat. #	Description	Qty.
COAZO060	Biocomma® Copure® AZO Dye SLE cartridge.	4 Pcs/Bag
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Solid Phase Extaraction (SPE) of Pesticide Residue in Tea

Introduction

In order to enhance the yield, many kinds of pesticides were used in tea growing. It will be harmful for human beings when drinking the contaminated tea. So it is necessary to determinate the pesticide residues in tea. While pigment must be removed, because of the influence to analysis.

Materials

Biocomma® Copure®GCB/NH2 SPE cartridge 500mg/500mg/6mL (Cat. No. CONHGC655).

Experiment

Sample Extraction

Weigh 2 g sample (accurate to 0.001 g) into a 50 mL centrifuge tube. Add 10 mL acetonitrile, shake well for 1 minute and stand for 30 minutes, centrifuge for 5 minutes in 4000 r/min, take the upper acetonitrile layer to be clean up.

SPE Cleanup

Condition: Add 2 cm height of Na2SO4 into the GCB/NH2 cartridge and condition with 10 mL 3:1 acetonitrile/toluene.

Load: Load 4 mL extract sample into the cartridge, and collect the effluent;

Elute: Elute with 25 mL 3:1 acetonitrile/toluene. Collect all effluent and combine with the previous step.

Reconstitute: Evaporate to dryness under nitrogen and reconstitute with 1 mL acetonitrile. Filtered through a syringe filter (Nylon, 0.22μm, 25 mm) and transfer into a 2 mL vial for GC-ECD and LC MS/MS analysis.

System Conditions

GC-ECD Conditions

GAS chromatograph: Agilent 7890A

Chromatographic column: Agilent J & W HP-5, (30m×0.32mm, 0.25μm) or equivalent

Inlet temperature: 220 ℃

Detector temperature: 300 ℃

Temperature program: 180 ℃ for 2 min.

10 ℃ / min to 230 ℃ and hold for 2 min.

2 ℃ / min to 260 ℃ and hold for 2 min.

25 ℃ / min to 270 ℃ and hold for 1.6 min.

Flow rate: 1.6 mL/min, Helium

Split: 10: 1

LC-MS/MS Conditions

Chromatographic column: Venusil ASB C18 (2.1 mm×150 mm, 5μm)

System: API 4000

Mobile phase: A: 0.1 % HCOOH+10mM ammonium acetate (1 mL HCOOH & 0.77 g ammonium acetate to 1 L water);

B: methanol solution

Elution mode:Gradient elution

Table 1 Mobile phase gradient elution procedure

Time (min)	A(%)	B(%)
--	95	5
1.50	95	5
6	5	95
11	5	95
11.01	95	5
15	95	5

Flow rate: 0.35 mL/min

Column Temperature: 40 ℃

Injection volume:5 μL

Ion source:ESI

Ionization mode:Positive

Scan mode:MRM

Table 2. Ion source parameters

Source/Gas	
Collision Gas(CAD)	6
Curtain Gas(CUR)	12
Ion Source Gas 1(GS1)	50
Ion Source Gas 2(GS2)	50
Ion Spray Voltage(IS)	5500
Temperature(TEM)	550
Interface Heater(ihe)	ON

Table 3.Instrument acquisition data for the analysis of carbamate pesticides

Compound	RT (min)	MRM	DP (V)	EP (V)	CE (V)	CXP (V)
Aldicarb	7.06	208.1>89.1	30	10	22	12
		208.1>116	30	10	10	12
Carbofuran	7.13	222.3>123.1	48	10	16	12
		222.3>165.2	48	10	31	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	70	10	46	12

Results and Discussion

During this study, the average recoveries of organochlorine and pyrethroid pesticides were 74.70 to 118.20 %, and the average recoveries of carbamate pesticides were 73.1 to 90.90 %, the recovery results were shown in Table 4 and Table 5, and the chromatogram were shown in Figure 1 and Figure 2.

Table 4 Recovery of organochlorine and pyrethroid pesticides in Tea (0.25 mg/kg)

Name	Recoveries(%)			Average Recovery (%)	RSD(%)
	1	2	3		
Vinclozolin	84.5	76.0	80.0	80.2	5.30
Procymidone	110.5	102.0	105.0	105.8	4.07
Iprodione	112.0	107.5	119.0	112.8	5.14
Bifenthrin	94.5	87.5	90.5	90.8	3.87
Fenpropathrin	109.5	100.0	106.5	105.3	4.61
Beta-cyfluthrin	84.0	79.5	82.5	82.0	2.79
Cyfluthrin	86.5	86.8	94.1	89.1	4.83
Flucythrinate	120.5	114.0	120	118.2	3.06
Fenvalerate	95.5	85.0	92.9	91.1	6.00
Fluvalinate	70.4	72.75	81.0	74.7	7.45

Table 5 Recovery of carbamate pesticides in Tea (0.05 mg/kg)

Name	Recoveries(%)			Average Recovery (%)	RSD(%)
	1	2	3		
Aldicarb	95.6	87.2	90.0	90.9	4.70
Carbofuran	84.4	78.0	82.2	81.5	3.99
Aldicarb sulfone	77.4	83.0	81.4	80.6	3.58
Aldicarb sulfoxide	70.0	74.4	75.0	73.1	3.73
Acetamiprid	82.4	94.0	88.4	88.3	6.57

1. Chromatography of pesticides multi-residues in tea.

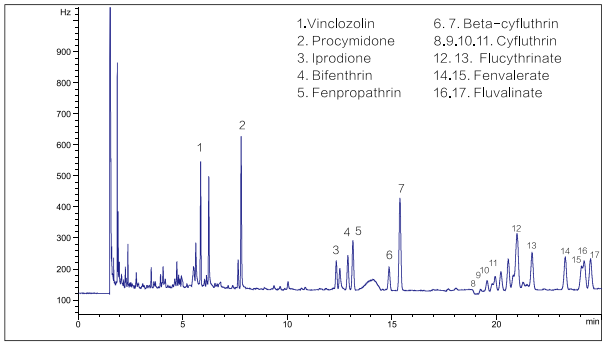


Figure 1 Chromatogram of organochlorine and pyrethroid pesticides in Tea（0.25 mg/kg）

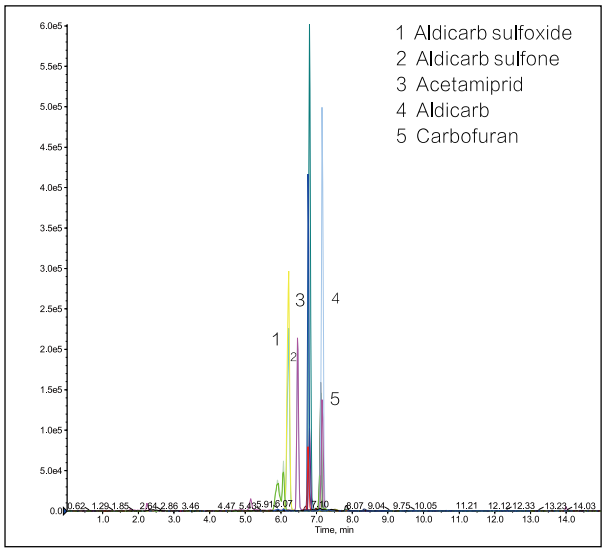


Figure 2 Chromatogram of carbamate pesticides in Tea

Conclusion

This is a rapid, sensitive method for the analysis of pesticides in tea. For samples such as tea, pigments will affect the results. Using biocomma® Copure® GCB/NH2 SPE, this color interference can be removed while obtaining excellent recovery. This method can simplify the sample preparation procedure, and save more time and solvent.

Ordering Information

Cat. #	Description	Qty.
CONHGC655	Copure® GCB/NH_2 Cartridge, 500 mg/500 mg / 6 mL	30 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Solid Phase Extraction (SPE) of Chromium (VI) in Leather

Introduction

Cr(Ⅲ) compounds were used as tanning agent at leather manufacture, but they were easy to be oxidized to Cr(VI) compounds which were genotoxic carcinogens. The European Union also published a regulation to ban Chromium (VI) in leather articles. This application note using biocomma copure PA SPE cartridge can easily extract chromium (VI) from leather.

Materials

Biocomma® Copure® PA SPE cartridge 500 mg/6 mL(Cat. No. COCR6500).

Experiment

Sample Extraction

Weigh 2 g ± 0,01 g of ground leather （accurate to0.001 g ）. Pipette 100 ml of degassed solution (Dissolve 22.8 g K2HPO4·3H2O in 1000 mL water, adjusted to pH 8.0±0.1 with phosphoric acid.

Degas this solution with nitrogen.) into a 250 mL conical flask. Displace oxygen by passing oxygen free nitrogen into the flask for 5 minutes (50 mL/min±10 mL/min). Add the ground leather and close the conical flask with a stopper immediately.

Shake the leather ground leather suspension 3 h ± 5 minutes on a mechanical shaker to extract the chromium(Ⅵ). Immediately after completing 3 hours of extraction, filter the content of conical flask through a membrane filter into a glass bottle with screw cap. Check the pH of the solution. The pH of the solution shall be between 7.5 and 8.0. If the pH of the solution is not within this range, start the complete procedure again.

SPE Cleanup

Condition and Equilibrate: Condition the biocomma copure PA SPE cartridge with 5mL methanol, and equilibrate with 5 mL water, 10 mL of extraction solution. Do not dry the cartridges during or after the pre-treatment.

Load: Transfer 10 mL extraction solution through the cartridge on an SPE system with vacuum device.

Collect the effluent in a 25 mL volumetric flask.

Elute: Elute the cartridge with 10 mL extraction solution into the above 25 mL volumetric flask.

Constant volume with extraction solution. Mark this solution as S1.

Content determination of chromium (VI)

Pipette 10 mL of solution S1 into a 25 mL volumetric flask. Dilute the solution to 3/4 of the flask's volume with extraction solution. Add 0.5 mL of phosphoric acid solution and afterwards 0.5 mL of diphenylcarbazide solution. Make up the flask to volume with extraction solution and mix well. Allow to stand for 15 min±5 min. Measure the absorbance of the solution at 540 nm in a 4 cm UV cell against the blank solution and record the absorbance.

For each run, pipette another 10 mL of solution S1 into a 25 mL volumetric flask and treat it as described above, but without the addition of the diphenylcarbazide solution. Measure the absorbance of this solution in the same way as before and record it.

Fill a 25 mL volumetric flask three quarters full with extraction solution, add 0.5 mL of phosphoric acid and 0.5 mL of diphenylcarbazide solution and make up to the mark with extraction solution and mix well. Prepare this solution daily and store it in the dark. Treat the blank solution in the same way as the analytical solution, excluding the solid phase extraction.

Experiment

The recovery and repeatability results are shown in Table 1. And the decolorization effect by the biocomma® copure® PA SPE cartridge were shown in Figure 1.

Table 1. Recoveries of chromium (VI) spiked at 0.4 ppm in leather.

Name	Recoveries(%)			Average Recovery (%)	RSD(%)
	1	2	3		
chromium(Ⅵ)	87.90	87.15	87.34	87.46	0.45

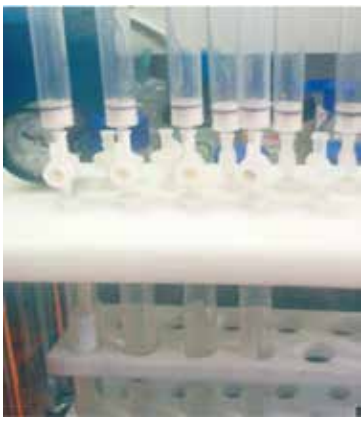


Figure 1.The decolorization performed by biocomma copure PA SPE cartridge

Conclusion

The results of this study show that biocomma® copure® PA SPE cartridge provide an effective method for decolorization of leather as well as good recoveries and reproducibility.

Ordering Information

Cat. #	Description	Qty.
COPACR66	Copure® Cartridge, 500 mg / 6 mL	30 Pcs/Box
SPEMF12G	12-Port SPE Vacuum Manifold	1 Set/Carton

Solid Phase Extraction (SPE) of Organochlorine Pesticides in Milk

Experiment Purpose

Organochlorine pesticides have been widely used insecticides to the world. Humans can ingest them by eating contaminated foods, such as fish, dairy products and other foods with higher fat contents. Long-term exposure in humans can have serious health effects, including damage to the liver, kidney, thyroid gland, bladder and central nervous system as well as serious reproductive problems. This application note used biocomma CopureFlorisil SPE cartridge to extract organochlorine pesticides from milk and analysis by GC-MS.

Materials

Biocomma® Copure® Florisil SPE cartridge 1g/6 mL(Cat. No. COFL61000).

Experiment

Sample Extraction

Weigh 10 g milk sample (accurately to 0.01 g) into a 50 mL centrifuge tube. Add 10mL acetone/hexane (1:9, v/v), vortex for 1 minute. Add 10mL acetone/hexane (1:9, v/v), 5 - 7 g NaCl, 2 mL 200 g/L lead acetate solution, close the lid and vortex for 5 minutes. Centrifuge at 4000 rpm for 5 minutes, Take 10 mL of the upper CH3CN layer for SPE cleanup.

SPE Cleanup

Condition: Condition the biocomma Copure Florisil SPE cartridge with 5mL acetone/hexane (1:9, v/v).

Load: Load 10 mL extraction into cartridge, control the flow rate within 1 mL/min, and collect the effluent.

Elute: Elute with 5 mL acetone/hexane (1:9, v/v). Collect the elution and combine all the effluent.

Reconstitute: Evaporate to dryness under nitrogen at 40 ℃ and reconstitute with 1 mL hexane. The solution was filtered through a syringe filter (Nylon, 0.45μm, 25 mm) and transfer into a 2 mL vial for GC-MS analysis.

GC-MS Conditions

System: Agilent 7890A

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

Inlet Temperature: 260 ℃

Detector Temperature: 300 ℃

Column Temperature: 100 ℃ (2 min),
10 ℃ /min to 240 ℃ (2 min),

Carry Gas: He

Flow Rate: 0.8 mL/min;

Inlet Volume: 1 μL

splitless

Table 1. Eight organochlorine pesticides, retention time and ion for quantitation and quantification

Compound	Time (min)	Ion for quantitation	Ion for quantification
α-666	9.473	217	183,254
β-666	10.071	217	183,254
γ-666	10.693	217	183,254
δ-666	11.573	217	187,254
p,p'-DDE	14.296	235	165,318
p,p'-DDD	15.095	235	165
p,p'-DDT	15.144	235	165
o,p'-DDT	15.378	235	165

Results and Discussion

The recovery and repeatability results were shown in Table 2, and the chromatogram of spiked milk extracts (0.5 mg/kg) was shown in Figure 1.

Table 2. Recoveries and reproducibility of eight organochlorine pesticides spiked at 0.5 mg/kg in milk

Name	Recoveries(%)			Average Recovery (%)	RSD(%)
	1	2	3		
α-666	86.0	88.0	90.0	88.0	2.27
β-666	90.0	90.0	92.0	90.7	1.27
γ-666	84.0	88.0	92.0	88.0	4.55
δ-666	90.0	96.0	98.0	94.7	4.40
p,p'-DDE	94.0	88.0	94.0	92.0	3.77
p,p'-DDD	90.0	98.0	94.0	94.0	4.26
p,p'-DDT	92.0	90.0	94.0	92.0	2.17
o,p'-DDT	98.0	100.0	94.0	97.3	3.14

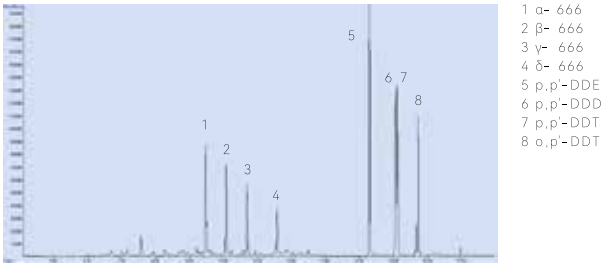


Figure 1.Chromatogram of eight organochlorine pesticides spiked at 0.5 mg/kg in milk

Conclusion

There is a need for a rapid, sensitive method for the analysis of organochlorine pesticides in milk. This work describes a method that employs a specific solid phase extraction of organochlorine pesticides using biocomma Copure Florisil.

Ordering Information

Cat. #	Description	Qty.
COFL61000	Copure® Florisil Cartridge, 1000 mg / 6 mL	30 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Muti-Residues of Veterinary Drugs in Meats by LC-ms/ms (Copure® HLB Cartridges)

Introduction

For the detection methods of tetracyclines, sulfonamides, and quinolones in beef, lamb, Beef, and chickens, Biocomma conducted experiments and optimized some parameters to establish an SPE-HPLC-MS/MS method with good recovery and stability for your reference.

Experiment

Preparation

Na_2EDTA-McIlvaine buffer: weigh 12.9 g of citric acid, 10.9 g of disodium hydrogen phosphate, and 39.2 g of disodium ethylenediaminetetraacetate, and dissolve by 900mL of water. Adjust the pH to 5.0±0.2 with 1 mol/L NaOH. Dilute to 1000 mL with water.

phosphate buffer: dilute 190 mL of 0.05 mol/L sodium dihydrogen phosphate solution to 1000 mL with 0.05 mol/L disodium hydrogen phosphate solution.

Extraction

Weigh 1.00 g of ground meat sample into a clean centrifuge tube. Add 8 mL of Na2EDTA-McIlvaine buffer, and vortex to mix well. Extract by ultrasonic treatment for 20 min. After centrifuge at 120,000 r/min, -5 ℃ for 5 min, transfer the supernatant to another clean centrifuge tube. Add 8 mL of phosphate buffer to the sample residue and repeat the extraction procedures twice. Combine all the extracted solutions. Centrifuge at 120,000 r/min, -5℃ for 5 min. The supernatant is ready for purification.

Purification (Copure® HLB Cartridge, 200 mg/ 6 mL)

Activation: activate the Copure® HLB Cartridge by 5 mL of methanol, then 5 mL of water.

Loading: add the prepared solution to the activated cartridge.

Washing: wash the cartridge by 5 mL of water, then 5 mL of 5% methanol-water solution. Drain the cartridge.

Elution: use 8 mL of methanol: ethyl acetate: ammonia water = 50:50:2 solution for elution.

Collect all the eluate, blow it with nitrogen to about 100 μL at 45°C. Evaporate sample to 100 μL at 45 ℃ . Dilute to 1 mL by 0.1% formic acid. After filter by PTFE hydrophilic membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Prepare 6 blank sample in the same procedures. Add internal standard to prepare the concentrations of 2 μg/L, 10 μg/L, 50 μg/L, 100 μg/L, 200 μg/L, and 500 μg/L for standard curve.

Instrumental Conditions

1.Chromatographic conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)

Chromatographic column: Hypersil GOLD C18 (2.1 mm×100 mm, 1.9 μm)

Mobile phase: A: water (0.1% formic acid), B: methanol: acetonitrile = 2:8 (0.1% formic acid)

Flow rate: 0.3 mL/min

Column temperature: 35 °C

Injection volume: 10 μL

Table 1. Gradient Elution Program

Time (min)	A(%)	B(%)
0.00	95	5
3.00	85	15
6.00	65	35
8.50	5	95
9.00	95	5
10.00	95	5

2.Mass spectrometry conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure: 40 arb

Auxiliary air pressure: 2 arb

Ion exchange tube: 380 ℃

Auxiliary air temperature: 350 ℃

Table 2. Targets, Retention Times and Characteristic Ions (*Quantitative Ion)

No.	Targets	Retention Time (min)	Parent Ion	Daughter Ion
1	Acesulfame	3.06	215.0	108.0、155.9*
2	Sulfapyridine	4.10	250.1	155.8*、183.9
3	Sulfadiazine	3.52	251.1	92.1、155.9*
4	Sulfamethoxazole	6.46	254.0	108.1、155.9*
5	Sulphathiazole	4.04	256.0	155.9*、92.1
6	Sulfamethazine	4.36	265.1	155.8*、171.8
7	Sulfamethoxazole	6.79	268.0	113.0、155.8*
8	Sulfamethiadiazole	5.32	271.0	92.1、155.9*
9	Sulfamethazine	3.50	279.1	124.0*、185.8
10	Sulfamethazine	5.07	279.1	155.8、185.8*
11	Sulfamethoxypyridazine	5.29	281.0	155.8*、126.0
12	Sulfamethoxine	5.38	281.0	155.9*、214.9
13	Sulfamethoxine	5.96	281.0	155.9*、214.9
14	Sulfachloropyridazine	6.04	285.0	92.1、155.9*
15	Sulfadimethoxine	6.41	311.1	155.8*、244.9
16	Sulfadimethoxine	7.47	311.1	155.8*、244.8
17	Pefloxacin	5.23	334.2	290.1*、316.1
18	Dafloxacin	5.48	358.2	314.1、340.0*
19	Marbofloxacin	4.93	363.1	320.0*、342.0
20	Difloxacin	6.19	400.2	299.0、356.1*
21	Doxycycline	7.20	445.2	321.0、428.0*
22	Tetracycline	5.52	445.2	410.0*、427.0
23	Oxytetracycline	5.18	461.2	426.0*、443.0
24	Chlortetracycline	6.00	479.1	444.0*、462.0

Results

Table 3. Results of Muti-Residue of Veterinary Drugs Recovery Experiments

Targes	Beef						Chicken					
	10.0 μg/kg		50.0 μg/kg		100.0 μg/kg		10.0 μg/kg		50.0 μg/kg		100.0 μg/kg	
	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3
Acesulfame	109	1.88	106	3.48	104	2.05	92.1	1.55	82.1	6.56	80.8	6.28
Sulfapyridine	94.5	3.54	98.7	10.0	98.3	4.32	84.2	3.79	96.2	8.97	99.4	3.71
Sulfadiazine	77.4	3.48	98.1	6.25	99.0	3.42	82.5	5.51	91.3	5.97	87.8	0.37
Sulfamethoxazole	101	6.88	100	9.07	104	1.33	105	4.21	97.1	4.95	96.7	3.35
Sulphathiazole	95.2	3.65	89.8	7.24	85.6	3.89	83.1	5.76	85.2	7.93	94.7	6.25
Sulfamethazine	92.5	3.14	93.8	6.68	92.8	4.88	89.9	1.77	98.5	6.67	93.2	2.63
Sulfamethoxazole	90.7	11.9	93.2	12.7	95.1	9.88	101	4.83	98.2	0.77	99.2	6.11
Sulfamethiadiazole	106	3.71	89.6	8.41	85.9	0.57	88.0	4.34	89.2	6.96	94.6	7.85
Sulfamethazine	82.5	1.63	103	4.15	107	0.99	77.6	0.24	88.6	7.07	93.8	0.35
Sulfamethazine	80.6	1.16	104	4.15	102	0.77	99.3	1.34	88.4	3.01	93.1	1.13
Sulfamethoxypyridazine	87.1	3.74	99.6	5.95	97.5	0.22	90.2	5.31	85.9	7.29	93.6	4.31
Sulfamethoxine	82.8	0.22	101	4.38	97.3	2.62	90.4	4.22	89.1	5.45	94.4	4.94
Sulfamethoxine	94.1	6.02	103	7.43	108	1.41	101	4.51	92.8	1.54	100	4.58
Sulfachloropyridazine	87.9	1.96	95.3	6.54	101	3.15	101	6.56	86.2	0.96	92.5	3.75
Sulfadimethoxine	79.1	3.59	99.8	7.87	98.9	0.99	91.8	0.96	94.7	2.05	94.3	2.21
Sulfadimethoxine	67.4	7.02	75.7	5.69	78.6	5.78	102	3.97	107	7.81	106	4.86
Pefloxacin	78.7	3.48	90.9	0.51	95.7	4.99	87.1	3.77	79.8	5.92	85.9	4.08
Dafloxacin	97.5	4.26	78.8	2.96	82.6	4.85	76.3	5.99	72.9	1.78	82.4	4.78
Marbofloxacin	83.9	2.12	95.2	1.73	97.5	8.28	80.8	3.61	93.4	4.17	89.3	3.85
Difloxacin	64.4	3.42	92.8	0.99	95.8	5.45	87.7	0.81	85.9	0.23	87.6	2.81
Doxycycline	103	2.18	74.9	0.36	78.6	0.55	85.2	3.97	82.3	1.53	84.2	5.54
Tetracycline	67.7	2.55	69.3	1.08	77.1	4.27	76.6	1.42	75.8	4.96	90.4	5.30
Oxytetracycline	78.7	4.57	70.3	0.26	85.3	4.71	74.3	2.39	80.6	7.95	86.2	0.69
Chlortetracycline	68.3	3.61	75.4	1.84	82.7	3.95	83.5	7.13	84.3	6.12	98.8	3.71

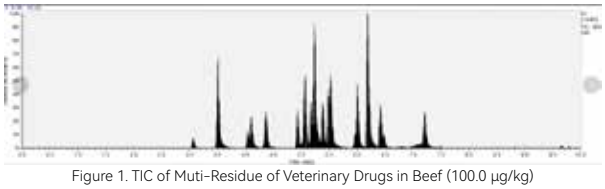


Figure 1. TIC of Muti-Residue of Veterinary Drugs in Beef (100.0 μg/kg)

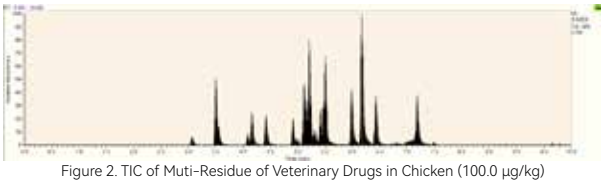


Figure 2. TIC of Muti-Residue of Veterinary Drugs in Chicken (100.0 μg/kg)

Optimization Tips:

1.Lowering the centrifuge temperature to -5°C during extraction inhibit the dispersion of the fat in the sample on the surface of the aqueous solution better, which is beneficial to the extraction and transfer of the sample.

2.After ultrasonic treatment, centrifuging the combined sample again improved removal of matrix effects and facilitates loading.

3.Eluting with 5 mL of 5% methanol-water solution reduces the loss of some target substances during elution.

4.Although the experiment requires to evaporate sample to about 100 μL, minimizing this process reduces the loss of some target substances.

5.Diluting the evaporated sample to 1 mL by 0.1% formic acid effectively improves the peak shape and recovery rate of some targets.

6.In order to improve the loading flow rate, employ Copure® HLB Cartridge, 200 mg/ 6 mL (Cat. No.: COHLB6200-M).

Ordering Information

Cat. #	Description	Qty.
COHLB6200-M	Copure® HLB Cartridge, 200 mg / 6 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF250-22-PTFE-HL	Syringe Filter PTFE/Φ25 mm/0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

β-Agonist Residues in Beef (Copure® MCX Cartridges)

Experiment

Extraction

Weigh 2 g of sample into a 50 mL centrifuge tube. Add 6 mL of 0.2 mol/L ammonium acetate buffer solution and 40 μL of β-glucuronidase/aryl sulfatase. Vortex to mix well. Oscillate in a water bath at 37 ℃ , and keep away from light exposure for 16 h. Leave it reach room temperature. Add 125 μL of 20 ng/mL internal standard solution. Vortex to mix. Centrifuged at 9500 r/min for 5min. Transfer the supernatant into another 50 mL centrifuge tube. Add 5 mL of 0.1 mol/L perchloric acid vortex to mix well. Adjust the pH with perchloric acid to 1.0±0.2. Centrifuged at 9500 r/min for 5min. Transfer the supernatant a clean 50 mL centrifuge tube, and adjust the pH to 10±0.5 with 10 mol/L NaOH solution. Add 15 mL of ethyl acetate, shaking for 5min. Centrifuged at 9500 r/min for 5min. Pipette the upper organic phase to a new tube. Add 10 mL of tert-butyl methyl to residues, shaking for 5min. Centrifuged at 9500 r/min for 5min. Pipette the upper organic phase to the same tube. Evaporate to nearly dryness at 40℃ . Dissolve with 5 mL of 2% formic acid. The sample is ready for purification.

Purification (Copure® MCX Cartridge, 60 mg/ 3 mL)

Activation: activate the Copure® MCX Cartridge by 3 mL of methanol, then 3 mL of 2% formic acid.

Loading: add prepared sample.

Washing: add 3 mL of 2% formic acid, then 3 mL methanol. Drain the cartridge.

Elution: add 5 mL of 5% ammoniated methanol and collect the eluate. Evaporate to near dryness at 40 °C. Add to 0.5 mL of methanol with 0.1% formic acid (10+90, V/V). After filter by 0.22 μm membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Accurately measure the standard solution and internal standard solution. Dilute with methanol with 0.1% formic acid (10+90, V/V) to prepare series of standard concentrations of 0.500 ng/mL, 1.00ng/mL, 2.00 ng/mL, 5.00 ng/mL, 10.0 ng/mL, and 20.0 ng/mL. The internal standard is 5.00 ng/mL.

Instrumental Conditions

1.Chromatographic conditions

Instrument: LC-MS/MS (Triple Quad 5500+)

Chromatographic column: ZORBAX RRHD Eclipse Plus 95Å C18, 2.1 x 100 mm, 1.8 μm

Mobile phase: A: 0.1% formic acid in water, B: acetonitrile

Mobile phase gradient: initial 98% A, 98% A (0 min~0.3 min), 10% A (0.3 min~3.0 min), 10% A (3.0 min~4.0 min), 98% A (4.0 min~4.2 min), 98% A (4.2 min~6.0 min)

Flow rate: 0.3 mL/min

Column temperature: room temperature

Injection volume: 4.0 μL

2.Mass Spectrometry Conditions

Detection method: multi-reactive ion monitoring (MRM)

Table 1. Ion Source Control Conditions

Ionization Mode	ESI+
Curtain Gas(CUR)	40.0 psi
Collision Gas(CAD)	8 psi
IonSpray Voltage(IS)	5500.0 V
Temperature(TEM)	500.0℃
Ion Source Gas 1(GS1)	50.0 psi
Ion Source Gas 1(GS2)	50.0 psi

Table 2. Targes Characteristic Ions (*Quantifier Ion)

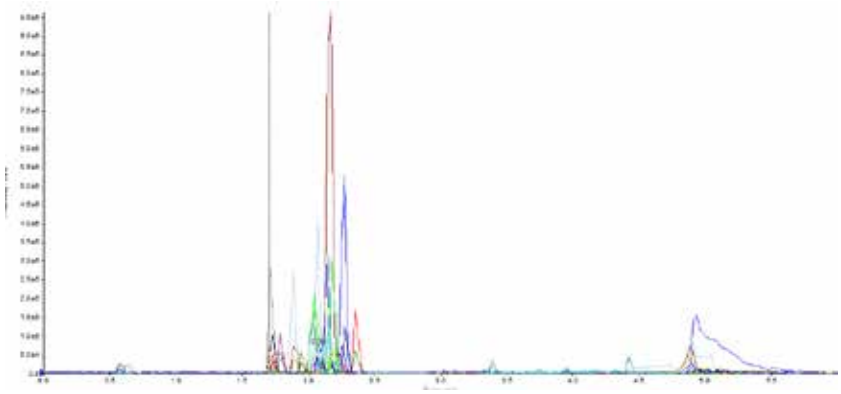
Targets	Parent Ion (m/z)	Daughter Ion (m/z)	Retention Time (min)	DP (V)	CE(V)
Clenbuterol	277.0	203.0 *	2.20	40	21
		168.1			38
Clenbuterol-D9	286.0	204.0	2.18	40	23
Ractopamine	302.2	164.1 *	2.09	40	23
		107.1			51
Ractopamine-D6	308.1	168.1	2.07	40	23
Salbutamol	240.2	148.1 *	1.75	40	24
		222.1			15
Salbutamol-D3	243.1	151.0	1.74	40	25
Cimaterol	220.0	160.0*	1.81	40	22
		202.0			13
Cimaterol-D7	227.2	161.1	1.80	40	23
Zilpaterol	262.1	185.0*	1.75	40	32
		202.1			25
Zilpaterol-D7	269.1	185.0	1.74	40	32
Clorprenaline	214.0	154.1 *	2.10	40	23
		118.0			34
Clorprenaline-D7	221.0	155.0	2.09	40	23
Terbutaline	226.2	152.0*	1.76	40	21
		107.1			36
Terbutaline-D9	235.2	153.1	1.75	40	20
Cibutrol	234.0	160.1 *	1.92	40	21
		143.0			34
Cibutrol-D9	243.2	161.1	1.90	40	21
Mabuterol	311.1	237.2 *	2.30	40	24
		202.1			40
Mabutrol-D9	320.1	238.0	2.28	40	24
Bambuterol	368.2	294.1*	2.21	40	26
		72.2			37

Bambuterol-D9	377.2	295.1	2.20	40	26
Clenbuterol	263.1	203.0 *	2.11	40	24
		245.1			15
Clenbuterol-D7	270.0	204.0	2.10	40	25
Tobutrol	228.0	154.0 *	2.17	40	21
		118.0			35
Tolbuterol-D9	237.0	155.0	2.16	40	21
Clenbuterol Hydroxymethyl	293.0	203.0*	2.06	40	23
		132.1			38
Clenbuterol-D6	299.0	203.0	2.05	40	25
Clenbuterol	367.0	293.0 *	2.25	40	24
		349.2			17
Clenpantel	291.0	203.0*	2.28	40	21
		132.1			38
Clenbuterol	319.1	203.0 *	2.01	40	26
		132.0			39
Ritodrine	288.1	121.1 *	1.97	40	29
		150.1			25
Mapentrol	325.0	237.0 *	2.38	40	24
		217.0			37

Results

Table 3. Spiked β-Agonist in Beef Recovery at 1.50 μg/kg

Targets	Recovery Rate (%)			Average Recovery (%)	RSD (%)
	1	2	3		
Clenbuterol	90.2	95.2	96.5	94.0	3.54
Ractopamine	99.5	98.8	100	99.4	0.608
Salbutamol	92.7	97.2	100	96.6	3.81
Cimaterol	95.2	100	101	98.7	3.15
Zilpaterol	95.7	93.0	99.2	96.0	3.24
Clorprenaline	97.3	96.8	102	98.7	2.91
Terbutaline	95.0	101	94.0	96.7	3.92
Cibutrol	100	92.5	93.3	95.3	4.32
Mabuterol	96.0	89.0	93.8	92.9	3.85
Bambuterol	96.3	97.5	97.3	97.0	0.664
Clenbuterol	97.5	93.7	98.7	96.6	2.70
Tolbuterol	89.2	97.7	93.5	93.5	4.55
Clenbuterol	93.0	98.7	101	97.6	4.22
Brombuterol	68.8	73.0	65.7	69.2	5.29
Ritodrine	65.2	63.5	62.3	63.7	2.29
Clenbuterol	79.8	81.5	78.5	79.9	1.88
Mapentrol	113	118	114	115	2.30
Clenpantel	101	106	99.8	102	3.24



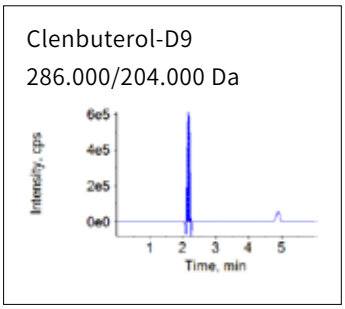
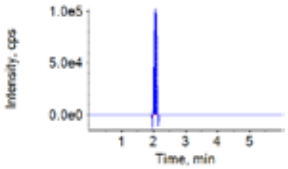
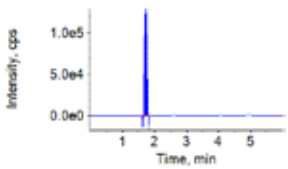


Figure 1. Total Ion Flow Diagram of β-Agonist Drugs

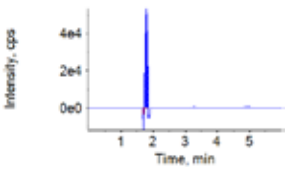
Ractopamine-D6
308.100/168.100 Da



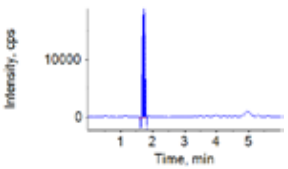
Salbutamol-D3
243.100/151.000 Da



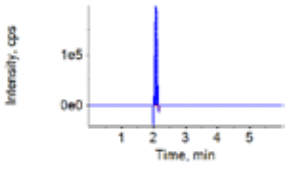
Cimatron-D7
227.200/161.100 Da



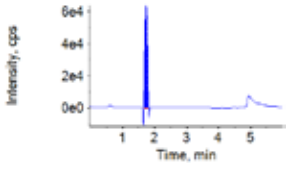
Zipatero D7
269.100/185.000 Da



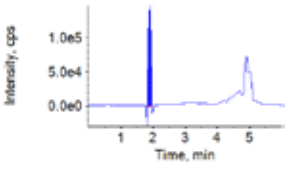
Clorprenaline-D7
221.000/155.000 Da



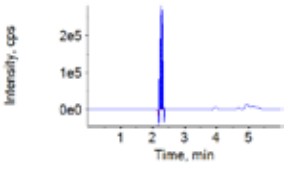
Terbutaline-D9
235.200/153.100 Da



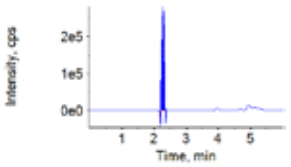
Cibutrol-D9
243.200/161.100 Da



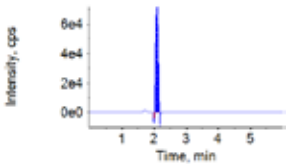
Mabutro D9
320.100/238.000 Da



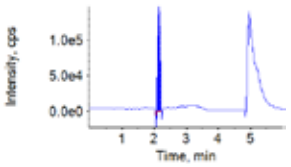
Mabutro D9
320.100/238.000 Da



Clenbuterol-D7
270.000/204.000 Da



Tobutrol-D9
237.000/155.000 Da



Tobutrol-D9
237.000/155.000 Da

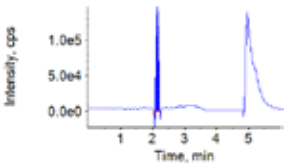
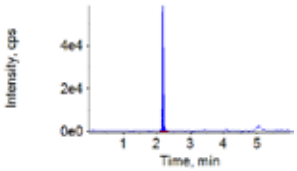
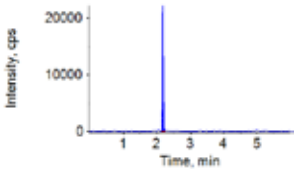


Figure 2. Characteristic Ion Mass Chromatogram of β-Agonist Internal Standard Solution (5.00ng/mL)

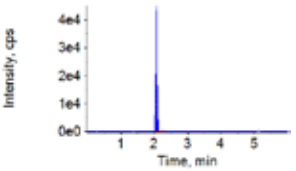
Clenbuterol
277.000/203.000 Da



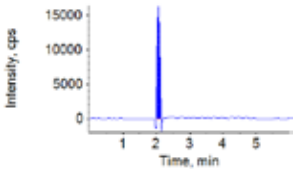
Clenbuterol (qualitative)
277.000/168.100 Da



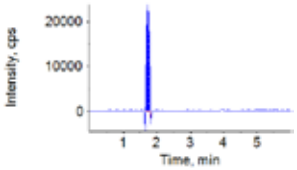
Ractopamine
302.200/164.100 Da



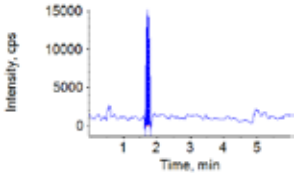
Ractopamine (qualitative)
302.200/107.100 Da



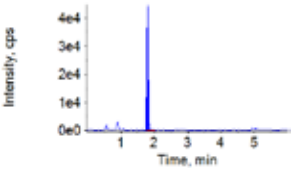
Salbutamol
240.200/148.100 Da



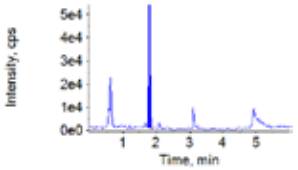
Salbutamol (qualitative)
240.200/222.100 Da



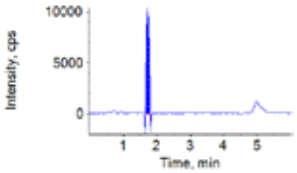
Cimatron
220.000/160.000 Da



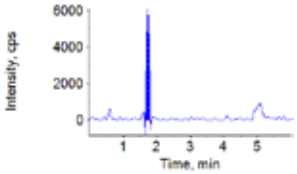
Cimatron (qualitative)
220.000/202.000 Da



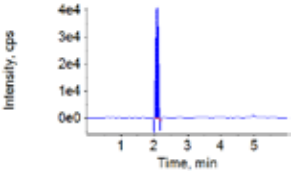
Zipatero
262.100/185.000 Da



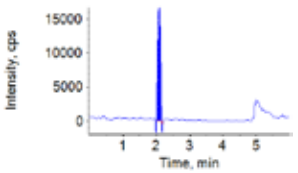
Zipatero (qualitative)
262.100/202.100 Da



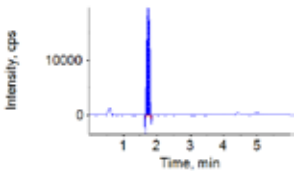
Clorprenaline
214.000/154.100 Da



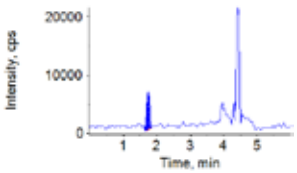
Clorprenaline (qualitative)
214.000/118.000 Da



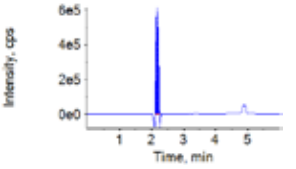
Terbutaline
226.200/152.000 Da



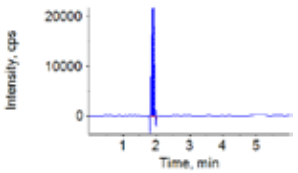
Terbutaline (qualitative)
226.200/107.100 Da



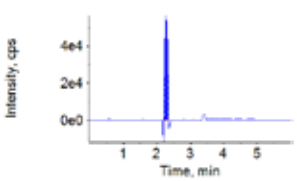
Albuterol
286.000/204.000 Da



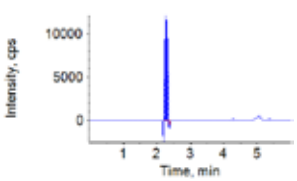
Albutero l(qualitative)
234.000/143.000 Da



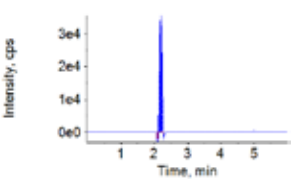
Maputo
311.100/237.200 Da



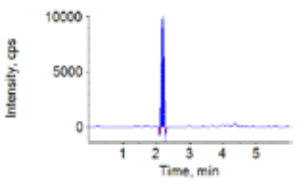
Maputo (qualitative)
311.100/202.100 Da



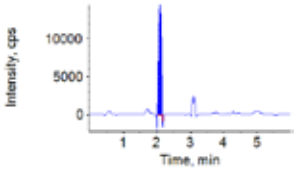
Bambuterol
368.200/294.100 Da



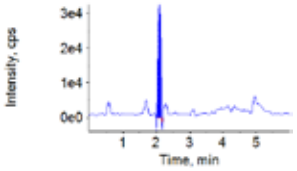
Bambuterol (qualitative)
368.200/72.200 Da



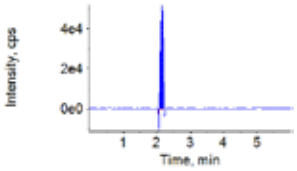
Clenbuterol
263.100/203.000 Da



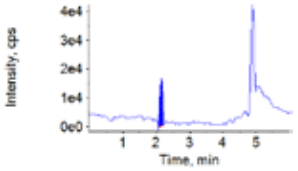
Clenbuterol (qualitative)
263.100/245.100 Da



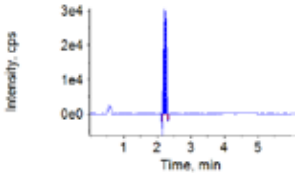
Albuterol
228.000/154.000 Da



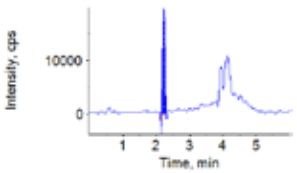
Albuterol (qualitative)
228.000/118.000 Da



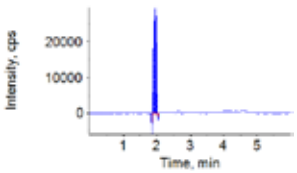
Brombuterol
367.000/293.000 Da



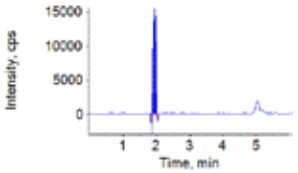
Brombuterol(qualitative)
367.000/349.200 Da



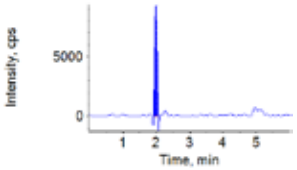
Litogun
288.100/121.100 Da



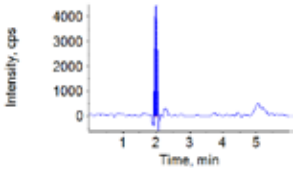
Litogun(qualitative)
288.100/150.100 Da



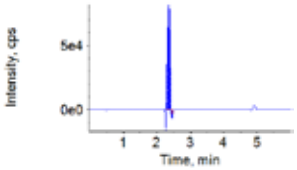
Clenbuterol
319.100/203.000 Da



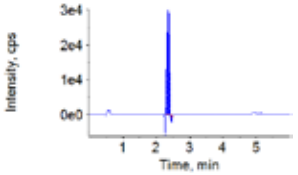
Clenbuterol(qualitative)
319.100/132.000 Da



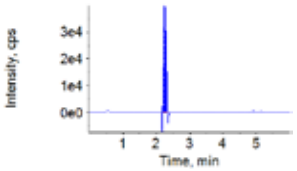
Mapuntero
325.000/237.000 Da



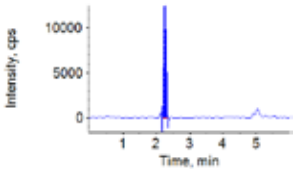
Mapuntero (qualitative)
325.000/217.000 Da



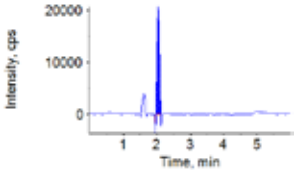
Clenbuterol
291.000/203.000 Da



Clenbuterol (qualitative)
291.000/132.100 Da



Clenbuterol
293.000/203.000 Da



Clenbuterol(qualitative)
293.000/132.100 Da

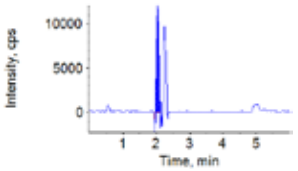


Figure 3. Characteristic Ion Mass Chromatogram of β-Agonist Standard Solution (0.500ng/mL)

Ordering Information

Cat. #	Description	Qty.
COMCX360	Copure® MCX Cartridge, 60 mg / 3 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Oxytetracycline, Tetracycline, Chlortetracycline, and Doxycycline Residues in Seafood (Copure® HLB Cartridges)

Introduction

For the LC-MS detection method of oxytetracycline, tetracycline, chlortetracycline, and doxycycline residues in seafood such as fish, shrimp, crab, and sea cucumber, Biocomma conducted experiments and optimized some parameters to establish an SPE-HPLC-MS/MS method with good recovery and stability for your reference.

Experiment

Preparation

Prepare Na2EDTA-McIlvaine buffer (0.1 mol/L): weigh 12.9 g of citric acid, 10.9 g of disodium hydrogen phosphate, and 37.2 g of disodium ethylenediaminetetraacetate, and dissolve by water. Dilute with water to 1000 mL, and adjust the pH to 4.0±0.05 with 0.1 mol/L HCl or 0.1 mol/L NaOH.

Prepare lead acetate solution (20.0 g/L): dissolve 20.0 g of lead acetate in water and dilute to 1000 mL.

Extraction

Weigh 2 g (accurate to 0.02 g) of ground aquatic samples such as fish and shrimp into a clean centrifuge tube. Add 6 mL of Na2EDTA-McIlvaine (pH=4±0.05) and 2 mL lead acetate. Vortex for 1 min, and extract by ultrasonic treatment for 10 min. After centrifuge at 8000 r/min, 4 ℃ for 5 min, transfer the supernatant to another clean centrifuge tube. Add 6 mL of Na2EDTA-McIlvaine (pH=4±0.05) and repeat the extraction procedures twice. Combine all the extracted solutions. Add 10 ml of n-hexane. Vortex for 1 min, and then centrifuge at 8000 r/min for 5 min. Discard the n-hexane layer and take 10 mL of the lower layer for purification.

Purification (Copure® HLB Cartridge, 60 mg/ 3 mL)

Activation: activate the Copure® HLB Cartridge by 5 mL of methanol, then 5 mL of water.

Loading: add the prepared solution to the activated cartridge.

Washing: wash the cartridge by 5 mL of water, then 5 mL of 5% methanol-water solution. Drain the cartridge.

Elution: add 5 mL of methanol. Collect all the eluate. Evaporate sample to 100 µL at 45 ℃ . Dilute to 1 mL by 0.1% formic acid. After filter by nylon membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Prepare blank sample in the same procedures. Add internal standard to prepare the concentrations of 5 µg/L, 10 µg/L, 50 µg/L, 100 µg/L, and 200 µg/L for standard curve.

Instrumental Conditions

1.Chromatographic conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)

Chromatographic column: Hypersil GOLD C18 (2.1 mm×100 mm, 1.9 µm)

Mobile phase: A: water (containing 0.1% formic acid) B: methanol (containing 0.1% formic acid)

Flow rates: 0.3 mL/min

Column temperature: 30 ℃

Injection volume: 5 µL

Table 1. Gradient Elution Program

Time (min)	A(%)	B(%)
0.00	95	5
2.00	70	30
4.00	70	30
4.20	30	70
6.00	30	70
7.00	95	5
8.00	95	5

2.Mass spectrometry conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure: 40 arb

Auxiliary gas pressure: 2 arb

Ion transfer tube: 380 ℃

Auxiliary air temperature: 350 ℃

Table 2. Targets, Retention Times and Characteristic Ions (Quantitative Ion)

NO.	Targets	Retention Time (min)	Parent Ion	Daughter Ion
1	Doxycycline	5.77	445.2	321.0, 428.0*
2	Tetracycline	4.00	445.2	410.0*, 427.0
3	Oxytetracycline	4.10	461.2	426.0*, 443.0
4	Chlortetracycline	5.63	479.1	444.0*, 462.0

Results

Table 3. Results of Chloramphenicol Spiking Recovery

Targes	Fish						Shrimp					
	10.0 µg/kg		50.0 µg/kg		100.0 µg/kg		10.0 µg/kg		50.0 µg/kg		100.0 µg/kg	
	Recovery rate (%)	RSD (%) n=3	ecovery rate (%)	RSD (%) n=3	ecovery rate (%)	RSD (%) n=3	ecovery rate (%)	RSD (%) n=3	ecovery rate (%))	RSD (%) n=3	ecovery rate (%)	RSD (%) n=3
Doxycycline	73.7	3.91	83.7	3.70	88.9	1.65	76.1	3.97	88.5	3.21	91.6	1.42
Tetracycline	78.2	1.53	85.4	4.53	89.3	3.16	79.6	5.04	84.2	1.98	91.4	2.59
Oxytetracycline	86.4	2.29	94.5	2.47	101	1.39	88.9	6.38	92.4	5.92	103	1.96
Chlortetracycline	75.3	3.15	84.3	4.49	90.1	2.94	79.3	2.04	89.8	1.92	92.1	1.27

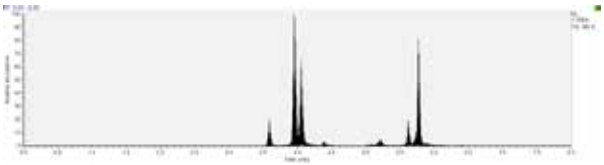


Figure 1. TIC of Tetracyclines in Fish (100.0 µg/kg)

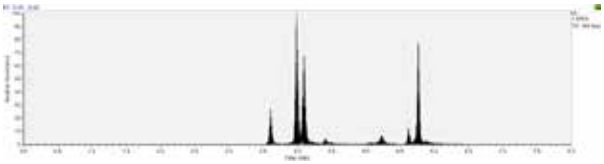


Figure 2. TIC of Tetracycline in Shrimp (100.0 µg/kg)

Ordering Information

Cat. #	Description	Qty.
COHLB360	Copure® HLB Cartridge, 60 mg / 3 mL	50 Pcs/Box
SDC-3000-D	biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 µm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Chloramphenicol Residue of Veterinary Drugs in Beef, Chicken, Fish and Shrimp (Copure® C18 SPE Cartridges)

Introduction

For the detection of chloramphenicol residues in Beef, chicken, fish and shrimp, Biocomma conducted experiments and optimized parameters to establish a SPE-HPLC-MS/MS method with good recovery and stability for your reference.

Experiment

Extraction

Weigh 5 g (accurate to 0.02 g) of ground sample in a clean centrifuge tube. Add 100 μL of internal standard, 10 mL of acetonitrile and 10 mL of 4% NaCl solution. Vortex for 10 min, then centrifuge for 10 min at 8000 r/min. Pipette the supernatant to a clean centrifuge tube. Add 10 mL of n-hexane. Vortex for 1 min, then centrifuge for 2 min at 8000 r/min. After discard the supernatant, add 8 mL of water-saturated ethyl acetate. Vortex for 2 min, then centrifuge for 2 min at 8000 r/min. Concentrate the supernatant by nitrogen evaporator and redissolve by 5 mL of 5% acetonitrile-water solution. The sample is ready for purification.

Purification (Copure® C18 SPE Cartridges, 500 mg /3 mL)

Activation: activate the SPE cartridge by 5 mL of methanol, then 5 mL of water.

Loading: add the prepared solution to the SPE cartridge.

Washing: wash the cartridge by 6 ml of water twice.

Elution: add 8 ml of methanol and evaporate to dryness. Reconstitute with 1 ml of 50% aqueous methanol solution. After filter by nylon membrane, the sample is ready for LC-MS/MS analysis.

Preparation of Standard Curve Solution

Table 1. Standard Solution Preparation Method

Internal standard CAP-D5 solution: 50 ng/mL, chloramphenicol solution 10 ng/mL and 50 ng/mL.

Standard Solution	0.5 ng/mL	1.0 ng/mL	2.0 ng/mL	5.0 ng/mL	10.0 ng/mL
10 ng/mLCAP	50 μL	100 μL	-	-	-
50 ng/mLCAP	-	-	40 μL	100 μL	200 μL
50 ng/mLCAP-D5	100 μL	100 μL	100 μL	100 μL	100 μL

Instrumental Conditions

1.Chromatographic Conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)

Chromatographic column: Hypersil GOLD C18 (2.1 mm×100 mm, 1.9 μm)

Mobile phase: A: water, B: methanol

Elution mode: Table 2.

Flow rate: 0.3 mL/min

Column temperature: 30 ℃

Injection volume: 20 μL

Table 2. Gradient Elution Program

Time (min)	A(%)	B(%)
0.00	95	5
1.00	90	10
4.00	50	50
5.00	50	50
7.00	10	90
8.00	10	10
9.00	95	5
10.00	95	5

2.Mass Spectrometry Conditions

Ion source: HESI Electrospray voltage: 3500 V

Sheath gas pressure: 40 arb Auxiliary gas pressure: 10 arb

Ion exchange tube: 380 ℃ Auxiliary air temperature: 350 ℃

Table 3. Targets, Retention Times and Characteristic Ions (*Quantitative Ions)

Item	Targets	Retention Time (min)	Parent Ion	Daughter Ion (CE/V)
1	Chloromycetin	4.45	320.8	152.0 (15)、256.9* (10)
2	Chloramphenicol-D5	4.43	326.0	157.0* (15)

Results

Table 4. Spiked Chloramphenicol Recovery

Samples	0.1 μg/kg		0.2 μg/kg		1.0 μg/kg	
	Recovery rate (%)	RSD (%) (n=3)	Recovery rate (%)	RSD (%) (n=3)	Recovery rate (%)	RSD (%) (n=3)
Beef	74.4	7.93	80.9	2.26	78.9	4.35
Chicken	90.8	4.62	94.1	5.75	86.6	7.69
Shrimp	88.2	4.49	82.8	3.59	79.4	2.81

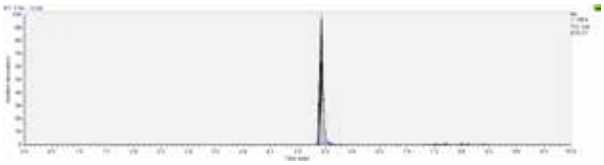


Figure 1. TIC of Chloramphenicol in Beef At 0.1 μg/kg

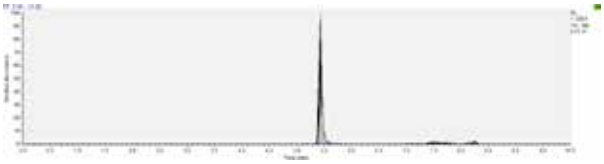


Figure 2. TIC of Chloramphenicol in Chicken at 0.1 μg/kg

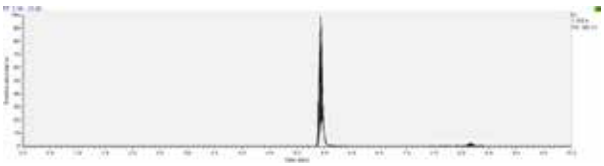


Figure 3. TIC of Chloramphenicol in Shrimp at 0.1 μg/kg

Ordering Information

Cat. #	Description	Qty.
COC183500	Copure®C18 SPE Cartridges, 500 mg / 3mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

Pentachlorophenol Residues in Animal Source Foods (Copure® MAX Cartridges)

Introduction

Pentachlorophenol (PCP), used as a cost-effective chlorinated hydrocarbon insecticide and fungicide, finds extensive applications in agriculture as a pesticide, herbicide, and wood preservative. It exhibits chemical stability, prolonged residual presence, resistance to degradation, and high water solubility. This experiment has optimized sample preparation and established a stable, reliable, and high-recovery SPE-UPLC-MS/MS method suitable for quantifying PCP residues in animal source foods.

Experiment

Extraction

Weigh 2.0 g (accurate to 0.01 g) of the sample into a 50 mL centrifuge tube. Add 5 mL of 8% triethylamine in acetonitrile-water solution (8:2, v/v). Vortex for 5 min, then extract by ultrasonic treatment for 5 min. Centrifuge at 10,000 r/min for 5 min. Transfer the supernatant into another clean tube. Repeat the extraction procedures and combine the extracted solution for purification. (Note: If the combined supernatant is cloudy, centrifuge the sample again)

Purification (Copure® MAX Cartridge, 60 mg/ 3 mL)

Activation: activate the Copure® MAX Cartridge by 5 mL of methanol, then 5 mL of water.

Loading: add the prepared solution to the activated cartridge.

Washing: wash the cartridge by 5 mL 5% ammonia solution, 5 mL methanol and 5 mL 2% formic acid methanol solution sequentially.

Elution: add 4 mL of 8% formic acid methanol solution and collect the eluate. Evaporate sample to about 1 mL at 40 ℃ . Dilute to 2 mL by water. After vortex and filter by PTFE hydrophilic membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Prepare blank sample in the same procedures. After collecting the eluent, add an appropriate amount of standard solution. Evaporate and dilute the concentration of 0.5 ng/mL, 1 ng/mL, 2 ng/mL, 5 ng/mL, and 10 ng/mL.

Instrumental Conditions

1.Chromatographic Conditions

Chromatographic column: Commasil® Coreshell C18 (2.1 mm×100 mm, 1.7 μm)

Mobile phase A: 5 mmol/L ammonium acetate solution (containing 0.1% formic acid)

Mobile phase B: Methanol

Flow rate: 0.35 mL/min

Column temperature: 35 ℃

Injection volume: 5 μL

Table 1. Gradient Elution Program

Time (min)	A/%	B/%
0.0	60	40
0.2	60	40
1.0	10	90
3.0	10	90
3.1	60	40
5.0	60	40

2.Mass spectrometry conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure: 40 arb

Auxiliary air pressure: 10arb

Ion exchange tube: 380 ℃

Auxiliary air temperature: 350 ℃

Table 2. Targets, Retention Times and Characteristic Ions (Quantitative Ion)

Target	Quantitative Transition (M/Z)	Qualifier Transition (M/Z)
Pentachlorophenol	262.7>262.7*	264.7>264.7、266.7>266.7、268.7>268.7

Result

Table 3. Pentachlorophenol Spiking Recovery

Target	Spike (μg/kg)	Beef		Chicken	
		Recovery rate(%)	RSD(%) (n=3)	Recovery rate(%)	RSD(%) (n=3)
Pentachlorophenol	1.00	88.6	3.33	89.2	4.58
	2.00	92.9	2.46	93.8	2.31

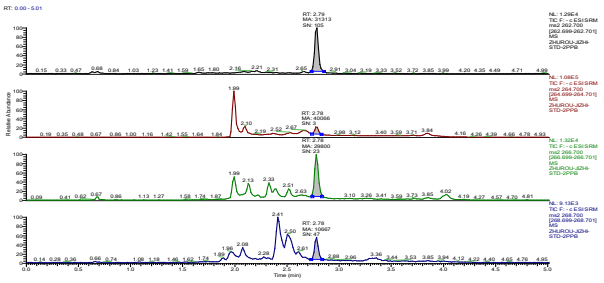


Figure 1. Total Ion Flow Diagram of Beef Spiked at 2.00 μg/kg

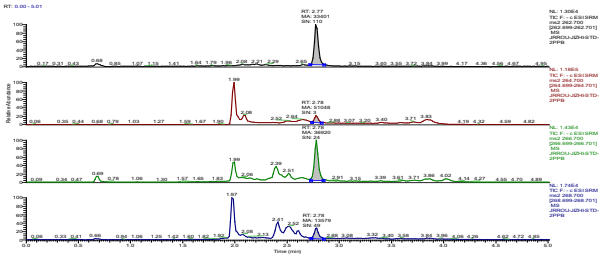


Figure 2. Total Ion Flow Diagram of Chicken Spiked at 2.00 μg/kg

Ordering Information

Cat. #	Description	Qty.
COMAX360	Copure® MAX Cartridge, 60 mg / 3 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-PTFE-HL	Syringe Filters PTFE/Φ25 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Cyromazine Residue in Chicken (Copure® MCX Cartridges)

Experiment

Extraction

Weigh 5 g of ground chicken into a 50 mL centrifuge tube. Add 15mL 1% trichloroacetic acid/acetonitrile (15+85, V/V), homogenize at high speed for 5min. Centrifuge at 9500r/min for 5min, and then pipette the supernatant into a 100 mL separatory funnel.

Add 10 mL of 1% trichloroacetic acid/acetonitrile (15+85, V/V) to the residue. Repeat step 1. Combine the extracts. Add 30 mL of n-hexane and shake for 2 min. After settle into layers, collect the lower layer into a flask. Evaporate to approximately 1 mL at 40 °C in a water bath. Transfer to a 10 mL centrifuge tube. Rinse the flask with 2 mL of 1% trichloroacetic acid/acetonitrile (15+85, V/V), then 2 mL of 0.1 mol/L hydrochloric acid. Centrifuge the combined liquids at 9500 r/min for 5 minutes. The supernatant is ready for purification.

Purification (Copure® MCX Cartridge, 60 mg/ 3 mL)

Activation: activate the Copure® HLB Cartridge by 3mL methanol, then 3 mL of 0.1 mol/L hydrochloric acid.

Loading: add the prepared solution to the activated cartridge.

Washing: wash the cartridge by3 mL of 0.1 mol/ L hydrochloric acid, then 3 mL of methanol. Drain the cartridge.

Elution: add 5 mL of 5% ammoniated methanol. Collect all the eluate. Evaporate sample to near dryness at 40 ℃ . Add 1 mL of mobile phase. After filter by 0.22 μm membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Dilute cyromazine standard working solution with mobile phase to prepare series of standard concentrations of 0.05 μg/mL, 0.1 μg/mL, 0.2 μg/mL, 0.4 μg/mL, 0.8 μg/mL, 1.0 μg/mL, 2.0 μg/mL.

Instrumental Conditions

Instrument: Liquid Chromatograph (1260 Infinity II, with UV detector)

Chromatographic column: Infinity Lab Poroshell 120 HILIC, 2.1 x 100 mm, 2.7 μm

Mobile phase: 25 mmol/L ammonium acetate solution-acetonitrile (40+960, V/V)

Flow rate: 0.300 mL/min

Detection wavelength: 214 nm

Column temperature: 30 ℃

Injection volume: 40.0 μL

Results

Table 1. Cyromazine Spiked Recovery in Chicken at 0.080 mg/kg

Targets	Recovery rate (%)			The average recovery rate (%)	RSD (%)
	1	2	3		
Cyromazine	82.5	88.5	88.0	86.3	3.86

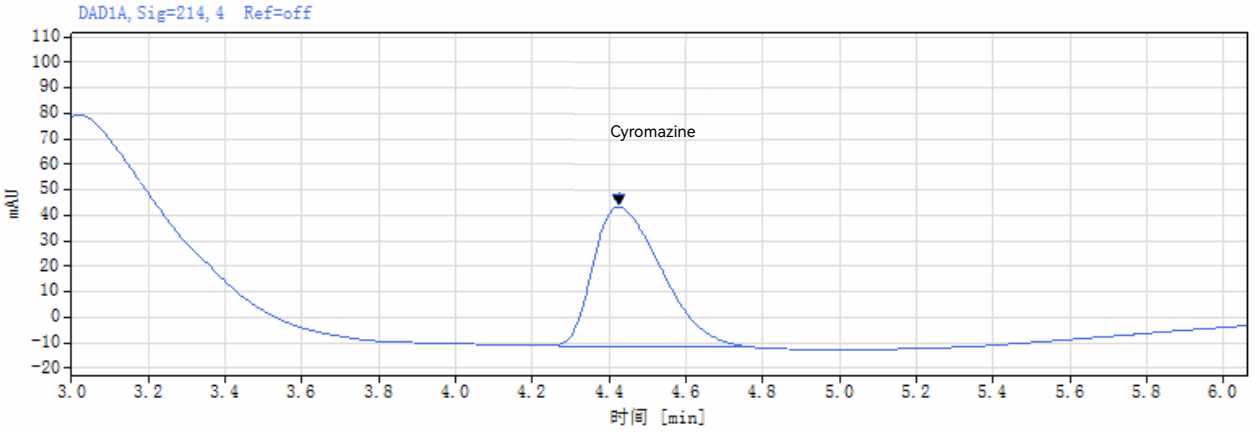


Figure 1. Chromatogram of Cyromazine Standard Solution (0.500 μg/mL)

Ordering Information

Cat. #	Description	Qty.
COMCX360	Copure® MCX Cartridge, 60 mg / 3 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Doxycycline Residues in Eggs (Copure® HLB Cartridges)

Experiment

Extraction

Weigh 2 g of egg into a 50 mL centrifuge tube. Add 8 mL of Na2EDTA-McIlvaine buffer. Vortex for 10 min, centrifuge at 14000 r/min at 4 °C for 10 min. Transfer the supernatant to another clean centrifuge tube. Add 8 mL of Na2EDTA-McIlvaine buffer and repeat the extraction procedures twice. Combine all the extracted solutions. Then, centrifuge at 14000 r/min at 4 °C for 10 min. Filter the supernatant for purification.

Purification (Copure® HLB Cartridge, 60 mg/ 3 mL)

Activation: activate the Copure® HLB Cartridge by 3mL methanol, then 5 mL of water.

Loading: add the prepared solution to the activated cartridge.

Washing: wash the cartridge by 3 mL of water, then 3 mL of 5% methanol-water solution. Drain the cartridge.

Elution: add 5 mL of methanol. Collect all the eluate. Evaporate sample to near dryness (remain few drops) at 40 ℃ . Dilute to 1 mL by 20% acetonitrile solution. Vortex for 30 s to dissolve the residue. After filter by 0.22 μm membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Prepare blank sample in the same procedures. After collecting the eluent, add an appropriate amount of standard solution. Evaporate and dilute the concentration of 1.00 ng/mL, 2.00 ng/mL, 5.00 ng/mL, 10.0 ng/mL, 20.0 ng/mL.

Instrumental Conditions

1.Chromatographic conditions

Instrument: Liquid Chromatography-Tandem Mass Spectrometry (Triple Quad 5500)

Chromatographic column: ACQUITY UPLC BEH C18 1.7m 2.150mm Column

Mobile phase: A: 0.1% formic acid in water, B: Acetonitrile

Mobile phase gradient: initial 95%A, 60%A (0 min~2.50min), 10%A (2.50 min~3.00min),

10%A (3.00min~4.00min), 95%A (4.00min~5.00min)

Flow rate: 0.300 mL/min

Column temperature: 35 ℃

Injection volume: 4.0 μL

2.Mass spectrometry conditions

Detection method: multiple reaction ion monitoring (MRM)

Table 1. Ion Source Control Conditions

Ionization Method	ESI+
Curtain Gas(CUR)	40.0 Psi
Collision Gas(CAD)	8Psi
IonSpray Voltage(IS)	5500.0 V
Temperature(TEM)	450℃
Ion Source Gas 1(GS1)	50 Psi
Ion Source Gas 1(GS2)	50 Psi

Table 2. Targets, Retention Times, Characteristic Ions, Declustering Voltage and Collision Energy (Quantitative Ion)

Targets	Parent Ion (M/Z)	Daughter Ion (M/Z)	Retention Time (Min)	DP (V)	CE (V)
Doxycycline	445.2	154.0*	1.84	130	40
		321.0			40

Results

Table 3. Results of Doxycycline Spiking Recovery Experiments at 5.00 μg/kg

Targes	Recovery Rate (%)			Average Recovery Rate (%)	RSD (%)
	1	2	3		
Doxycycline	77.6	82.8	82.3	80.9	3.55

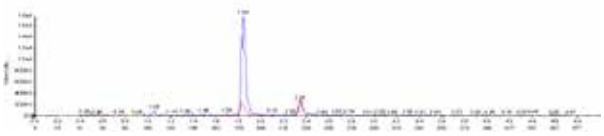
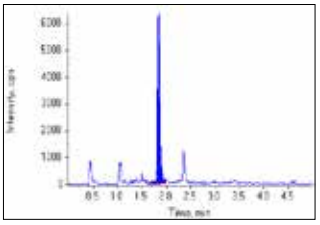
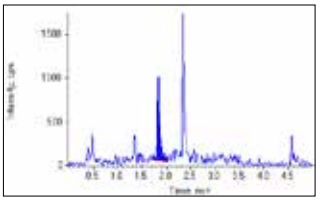


Figure 2. Characteristic Ion Mass Chromatogram of Egg Matrix Matched Standard Solution (2.00ng/mL)



Doxycycline
445.200/154.200 Da



Doxycycline (qualitative)
445.200/321.000 Da

Ordering Information

Cat. #	Description	Qty.
COHLB360	Copure® HLB Cartridge, 60 mg / 3 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Malachite Green & Crystal Violet Residues in Shrimp (Copure® ALN Neutral Alumina Cartridges)

Malachite green and crystal violet, once widely used in aquaculture for their low cost and antibacterial properties, can pose health risks due to their toxicity and accumulation in organisms.

Biocomma established a UPLC-MS/MS method for detection of malachite green and crystal violet in shrimp by Copure® ALN Neutral Alumina Cartridges with good recovery and precision for your reference. The recoveries were 90-110% with RSD less than 5%.

Experiment

Extraction

Weigh 5.00 g of ground shrimp into a clean centrifuge tube. Add 400 µL of mixed internal standard (malachite green-d5 and leuco-malachite green-d6) and 11 mL of acetonitrile. Extract by ultrasonic treatment for 2.0 min, then vortex at 2500 rpm for 5.0 min. Centrifugation at 8000 r/min for 5.0 min. Transfer the supernatant to another 50 mL centrifuge tube. Repeat the extraction procedures. Combine the supernatant and dilute to 25 mL by acetonitrile. The sample is ready for purification.

Purification

(Copure® ALN Neutral Alumina Cartridges, 1 g/ 3 mL)

Activation: activate the cartridge by 5.0 mL of acetonitrile.

Loading: add the prepared solution to the SPE cartridge.

Washing: wash the cartridge by 4.0 mL of acetonitrile.

Elution: evaporate to dryness at 45 °C. Add 1.0 mL of acetonitrile, and sonicate for 5.0 min. then, add 1.0 mL of 5 mmol/L ammonium acetate, and sonicate for 1.0 min. After filter by 0.22 µm filter membrane, the sample is ready for LC-MS/MS analysis.

Preparation of Matrix Blank

Prepare blank sample in the same procedures.

Instrument Conditions

1.Chromatographic Conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)

Chromatographic column: Commasil® Coreshell C18 (2.1 mm×100 mm, 1.7 µm)

Mobile phase: A: water (0.1% formic acid), B: acetonitrile (0.1% formic acid)

Flow rate: 0.3 mL/ min

Column temperature: 30 ℃

Injection volume: 10 µL

Table 1. Gradient Elution Program

Time (min)	B (%)	Curve
0.0	5	5
1.5	60	5
3.0	100	5
10.0	100	5
10.5	5	5
15	5	5

2.Mass Spectrometry Conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure:15 arb

Auxiliary gas pressure: 1 arb

Ion exchange tube: 360 ℃

Auxiliary air temperature: 380 ℃

Table 2. Targets, Retention Times and Characteristic Ions (*Quantifier Ion)

Targets	Precursor (m/z)	Product (m/z)	Collision Energy (V)	RF Lens (V)	RT/min
Malachite Green	329	208	33	168	4.27
		313*	34		
Leuco-Malachite Green	331	239*	28	151	4.27
		316	31		
Malachite Green-D5	334	318*	37	157	4.25
Leuco-Malachite Green-D6	337	240	29.8	139	4.25
		322.2*	19.9		
Crystal Violet	372	251	41	134	4.21
		356*	43		
Leuco-Crystal Violet	374	238	36	157	4.22
		358*	35		

Results

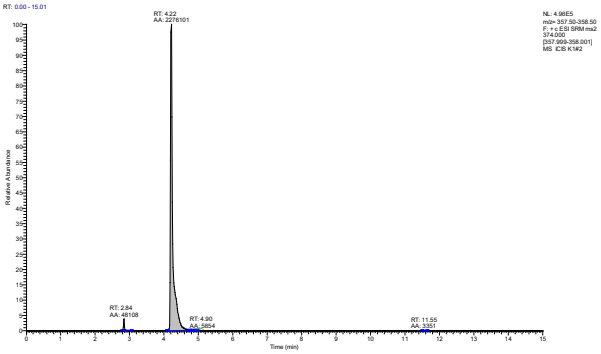


Figure 1. Leuco-Crystal Violet in Shrimp Spiked (8.0 MG/Kg)

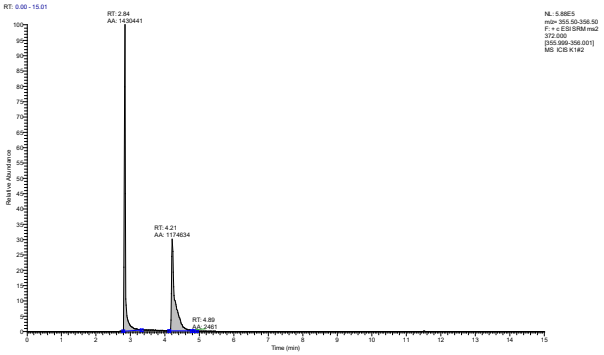


Figure 2. Crystal Violet in Shrimp Spiked (8.0 MG/Kg)

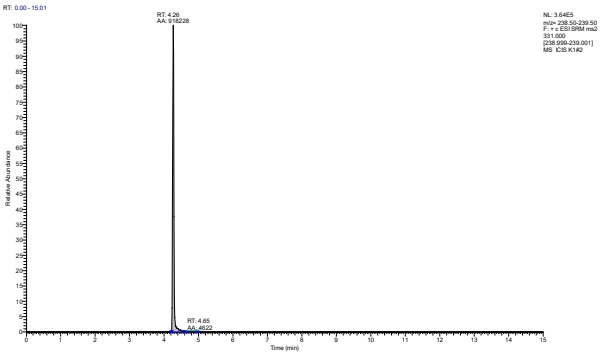


Figure 3. Leuco-Malachite Green in Shrimp Spiked (8.00 µg/kg)

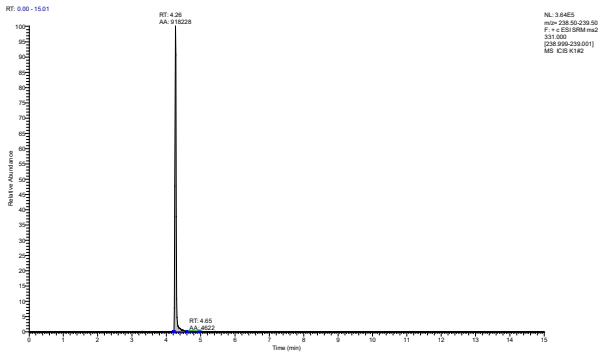


Figure 4. Malachite Green in Shrimp Spiked (8.00 µg/kg)

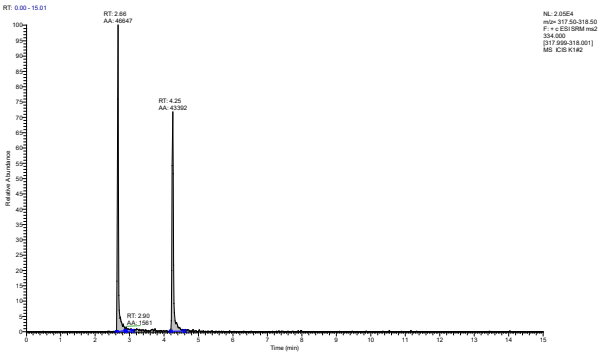


Figure 5. Malachite Green-D5 in Shrimp Spiked (8.00 µg/kg)

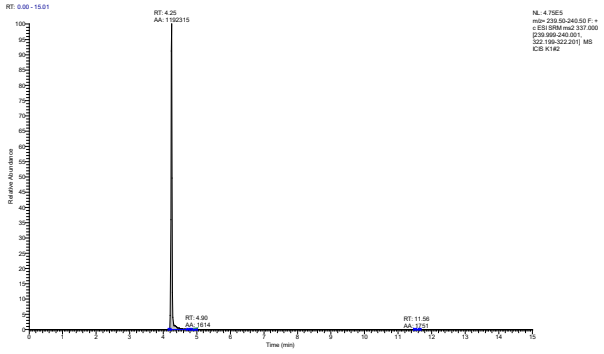


Figure 6. Leuco-Malachite Green-D6 in Shrimp Spiked (8.00 µg/kg)

Table 3 Results of Spiked Recoveries of in Shrimp

Sample	Targets	Spiked Concentration (MG/Kg)	Average Recovery Rate (%) (N=3)	RSD (%) (N=3)
Shrimp	Malachite Green	8.00	98.4	4.74
	Leuco-Malachite Green	8.00	94.5	3.06
	Crystal Violet	8.00	93.1	2.97
	Leuco-Crystal Violet	8.00	106	3.29

Ordering Information

Cat. #	Description	Qty.
COALN31000	Copure® Neutral Alumina Cartridge, 1 g / 3 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
BN24	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 µm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

Olaquindox Metabolites in Beef (Copure® MAX Cartridges)

Quinol, a growth-promoting pharmaceutical additive, is banned as a feed supplement due to its cumulative toxicity, teratogenicity in animals, and potential health risks. It quickly metabolizes in the body, leaving behind a stable metabolite, 3-methyl-quinoxaline-2-carboxylic acid (MQCA), recognized as a residual marker by the International Codex Alimentarius Commission.

Biocomma developed an LC-MS/MS method for MQCA in Beef with good recovery and stability for your reference.

Experiment

Extraction

Weigh 5.00 g of ground Beef into a 50 mL centrifuge tube. Add 10 mL of 0.6% formic acid and mix well. Place in a 47 ℃ shaking water bath and shake for 1 h. Add 3 mL of 1.0 mol/L Tris solution and mix well, then add 0.3 mL of 0.01 g/mL protease solution and mix well. Place in the 47 ℃ shaking water bath for 16-18 h for enzymatic hydrolysis. Cool to room temperature, then add 20 mL of 0.3 mol/L hydrochloric acid solution. After shaking for 5 min, centrifuge at 10,000 r/min for 5 min at 10 ℃ . Filter the supernatant for purification.

Purification (Copure® MAX Cartridge, 60 mg/ 3 mL)

Activate the Copure® MAX Cartridge by 3.0 mL of methanol, then 3.0 mL of water. Add the prepared supernatant to the cartridge. Add 30 mL of 0.05 mol/L sodium acetate - methanol (19+1) to the centrifuge tube, then pipette to the cartridge. Drain the cartridge. Wash the cartridge sequentially by 30 mL of 0.05 mol/L sodium acetate - methanol (19+1), 3×3.0 mL methanol, 3.0 mL water, 3×3.0 mL 0.1 mol/L hydrochloric acid solution, 2×3.0 mL methanol-water solution (1+4), and 2.0 mL ethyl acetate. Drain the cartridge. Add 3.0 mL of 2% formic acid in ethyl acetate and collect the eluate. Evaporate sample to about dryness at 45 ℃ . Reconstitute with 1.0 mL of methanol with 0.1% formic acid (19+1). After vortex and filter by 0.22 μm filter membrane, the sample is ready for analysis.

Instrumental Conditions

1.Chromatographic conditions

Instrument: UPLC-MS/MS （Thermo Fisher TSQ Endura）
Chromatographic column: SuPersil AQ-C18 (2.1 mm×100 mm, 3 μm)
Mobile phase: A: 0.1 % formic acid in water, B: methanol
Flow rate: 0.3 mL/min
Column temperature: 30 ℃
Injection volume: 10 μL

Table 1. Gradient Elution Program

Time (min)	A(%)	B(%)
0	80	20
1.0	80	20
1.1	60	40
3.0	40	60
3.1	20	80
4.0	20	80
5.5	80	20
7.0	80	20

2.Mass spectrometry conditions

Ion source: HESI
Electrospray voltage: 3500 V
Sheath gas pressure: 40 arb
Auxiliary gas pressure: 2 arb
Ion exchange tube: 380 ℃
Auxiliary air temperature: 350 ℃

Table 2. Targets, Retention Times and Characteristic Ions (Quantitative Ion)

Targets	Retention Time (min)	Parent Ion	Daughter Ion
Quinoxaline-2-Carboxylic Acid-D4	3.83	179.1	133.1*, 161.0
3-Methyl-Quinoxaline-2-Carboxylic Acid	4.21	189.1	145.1*, 171.0

Results

Table 3. Results of MQCA Spiking Recovery

Targets	Concentration Levels (μg/kg)	Recovery Rate (%)	Average Recovery Rate (%)	RSD (%)
3-Methyl-quinoxaline-2-carboxylic acid	2.0	74.1	77.5	3.78
		78.9		
		79.4		

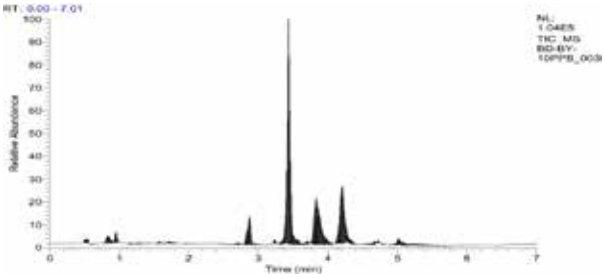


Figure 1. TIC of MQAC in Beef Spiked at 2.00 μg/kg

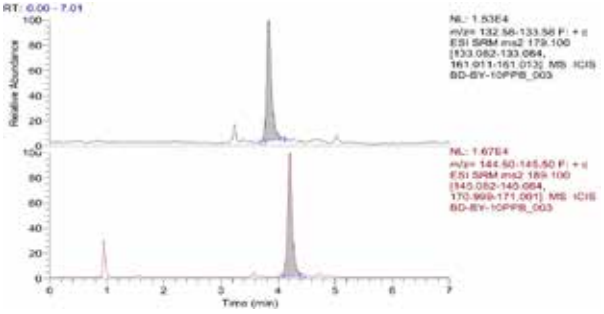


Figure 2. EIC of MQAC in Beef Spiked at 2.00 μg/kg

Ordering Information

Cat. #	Description	Qty.
COMAX360	Copure® MAX Cartridge, 60 mg / 3 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Streptomycin and Dihydrostreptomycin Residues in Honey (Copure® HLB Cartridges)

Introduction

Streptomycin and dihydrostreptomycin belong to aminoglycoside antibiotics, which have antibacterial activity against Gram-negative bacteria to prevent from various animal diseases. In the beekeeping industry, streptomycin and dihydrostreptomycin effectively treat bee rot and maggot disease, but due to unscientific management and use, they often cause residues in bee products. Biocomma optimized sample preparation of streptomycin and dihydrostreptomycin residues in honey to establish a SPE-UPLC-MS/MS method with good recovery and stability for your reference.

Experiment

Extraction

Accurately weigh 5 g of the sample into a 50 mL centrifuge tube. Add 10 mL of buffer solution (2% trichloroacetic acid in phosphate buffer). Vortex for 1 min. Then, extract by ultrasonic treatment for 10 min. Add 10 mL of buffer solution and dilute to 25 mL. Vortex for 1 min and extract by ultrasonic treatment for 5 min. Centrifuge at 10,000 r/min for 5 min. The sample is ready for purification.

Purification (Copure® HLB Cartridge, 60 mg/ 3 mL)

Activation: activate the Copure® HLB Cartridge by 3 mL of methanol, then 3 mL of water.

Loading: add 5 mL of the prepared solution to the activated cartridge.

Washing: wash the cartridge twice by 2 mL of water, and drain the cartridge.

Elution: add 1 mL of formic acid-acetonitrile-water (2:10:88, v:v:v). Collect the eluate and dilute to 2 mL by 1 mL of 2% ammoniated acetonitrile. After vortex and filter by membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Add an appropriate amount of mixed standard intermediate solution to blank sample in turn. Follow the steps of sample extraction and purification to prepare the standard curves with concentrations of 2.5 ng/mL, 5 ng/mL, 10 ng/mL, 20 ng/mL, 50 ng/mL, and 100 ng/mL.

Instrumental Conditions

1.Chromatographic conditions

Chromatographic column: Hilic (2.1 mm×100 mm, 2.7 μm)

Mobile phase A: 5 mmol/L ammonium acetate (containing 0.3% formic acid)

Mobile phase B: acetonitrile (containing 0.3% formic acid)

Flow rates: 0.3 mL/min

Column temperature: 35℃

Injection volume: 10 μL

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%))
0.0	10	90
0.5	10	90
2.5	90	10
4.0	90	10
4.1	10	90
5.0	10	90

2.Mass spectrometry conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure: 40 arb

Auxiliary gas pressure: 10 arb

Ion transfer tube: 350 ℃

Auxiliary gas temperature: 375 ℃

Table 2. Targes Characteristic Ions (*Quantifier Ion)

Targes	Parent Ion	Daughter Ion
Streptomycin	582.2	246.2, 263.2*
Dihydrostreptomycin	584.2	246.0, 263.0*

Results

Table 3. Spike Recovery Results

Targes	5.00 μg/kg		10.0 μg/kg		20.0 μg/kg	
	Recovery rate%	RSD% (n=3)	Recovery rate%	RSD% (n=3)	Recovery rate%	RSD% (n=3)
Streptomycin	81.6	6.12	86.1	5.25	93.8	4.41
Dihydrostreptomycin	83.5	5.94	90.8	5.11	101	3.89

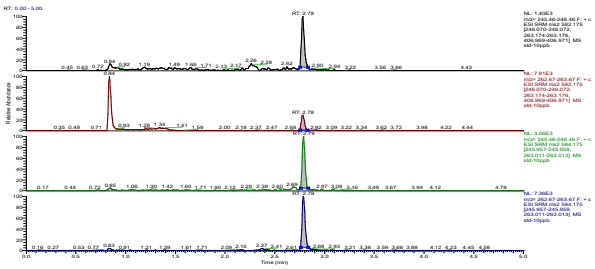


Figure 1. TIC of Honey Spiked Sample (20.0 μg/kg)

Ordering Information

Cat. #	Description	Qty.
COHLB360	Copure® HLB Cartridge, 60 mg / 3 mL	50Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

4-Methylimidazole in Foods (Copure® MCX Cartridges)

Introduction

4-Methylimidazole (4-MI) is a contaminant of carcinogenic risk that is commonly found in food as a by-product of the production of caramel coloring. Biocomma established a LC-MS/MS method for detection of 4-methylimidazole in passion fruit juice and honey with good recovery and precision for your reference.

Experiment

Extraction

Weigh 2.000 g of the sample into a 50 mL centrifuge tube. Add 2 % formic acid aqueous solution to 20 mL and mix well. Centrifuge at 10000 r/min for 5 min. The supernatant is ready for purification.

Purification (Copure® MCX Cartridge, 200 mg/ 6 mL)

Activation: activate the Copure® MCX Cartridge by 5 mL of methanol, then 5 mL of water.

Loading: add 10 mL of prepared sample.

Washing: add 2 mL of 2 % formic acid aqueous solution, 5 mL of water and 5 mL of methanol in sequence.

Elution: add 8 mL of 5 % ammonia methanol and collect the eluate. Evaporate sample to near dryness at 45℃ . Add 1 mL of acetonitrile-5 mmol/L ammonium acetate (9+1). Vortex for 30 s to dissolve the residue. After filter by 0.22 μm membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Dilute the internal standard solution with acetonitrile-5 mmol/L ammonium acetate (9+1) solution to prepare series of standard concentrations of 10 μg/L, 20 μg/L, 50 μg/L, 100 μg/L, 200 μg/L and 400 μg/L.

Instrumental Conditions

1.Chromatographic conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)

Chromatographic column: GOWON HILIC (2.1 mm×100 mm, 2.7 μm)

Mobile phase: A: 5 mmol/L ammonium acetate, B: acetonitrile

Flow rate: 0.6 mL/min

Column temperature: 35 °C

Injection volume: 5 μL

Table 1. Gradient Elution Program

Time (min)	A/%	B/%
0.00	5	95
3.00	5	95
3.50	40	60
4.50	40	60
5.00	5	95
6.00	5	95

2.Mass Spectrometry Conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure: 40 arb

Auxiliary air pressure: 5arb

Ion exchange tube: 380 ℃

Auxiliary air temperature: 350 ℃

Table 2. Targes Characteristic Ions (*Quantifier Ion)

Targets	Retention Time (min)	Parent Ion	Daughter Ion
4-Methylimidazole	2.24	83.1	42.1,56.2*

Result

Table 3. 4-Methylimidazole Spiked Recovery

Sample	10.0 μg/kg		50.0 μg/kg		100.0 μg/kg	
	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3
Passion Fruit Juice	91.6	6.95	102	4.02	93.4	4.21
Honey	104	4.41	92.2	4.23	88.9	2.25

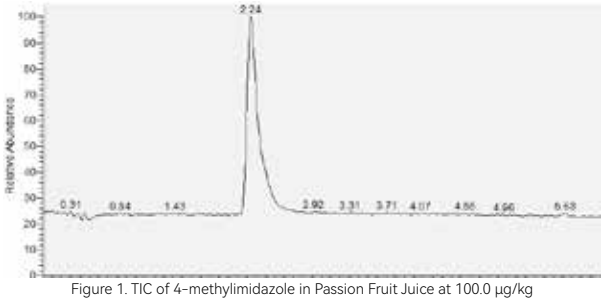


Figure 1. TIC of 4-methylimidazole in Passion Fruit Juice at 100.0 μg/kg

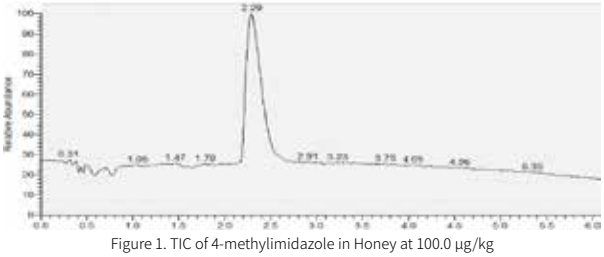


Figure 1. TIC of 4-methylimidazole in Honey at 100.0 μg/kg

Ordering Information

Cat. #	Description	Qty.
COMCX6200	Copure® MCX Cartridge, 200 mg / 6 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-PTFE	Syringe Filters PTFE/Φ25 mm/0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

Nitroimidazole Residues in Beef (Copure® MCX Cartridges)

Experiment

Extraction

Weigh 2 g of ground Beef into a 50 mL centrifuge tube. Add 100 µL of 0.100 µg/mL internal standard solution. Vortex for 30 s, and let stand for 10 min. Then add 15 mL of ethyl acetate. Oscillate for 10 min, and centrifuge at 9500 r/min for 5 min. Pipette the supernatant into another 50 mL centrifuge tube. Repeat the extraction with 15 mL of ethyl acetate, combine the extract. Evaporate sample to near dryness at 40 ℃ with water bath. Add 5 mL of 0.1 mol/L hydrochloric acid. Vortex for 1 min to dissolve the residue. Add 5 mL of n-hexane. Shake for 1 min, and centrifuge at 9500 r/min for 5 min. Discard the n-hexane layer. Add 5 mL of n-hexane to the lower layer and repeat degreasing once. The lower aqueous phase is ready for purification.

Purification (Copure® MCX Cartridge, 60 mg/ 3 mL)

Activation: activate the Copure® MCX Cartridge by 3 mL of methanol, then 3 mL of 0.1 mol/L hydrochloric acid.

Loading: add prepared sample to the activated cartridge.

Washing: add 3 mL of 0.1 mol/L hydrochloric acid, then 3 mL methanol. Drain the cartridge.

Elution: add 5 mL of 5% ammoniated methanol and collect the eluate. Evaporate to near dryness at 40°C. Dilute to 0.5 mL by 0.1% formic acid aqueous solution. Vortex for 30 s to dissolve the residue. After filter by 0.22 µm membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Accurately measure the standard solution and internal standard solution. Dilute with 0.1% formic acid aqueous solution to prepare series of standard concentrations of 0.500 µg/L, 1.00 µg/L, 2.00 µg/L, 5.00 µg/L, 10.0 µg/L, 25.0 µg/L. The internal standard is 20.0 µg/L.

Instrumental Conditions

1.Chromatographic conditions

Instrument: Triple Quadrupole LC-MS/MS System (Triple Quad 5500)

Chromatographic column: ACQUITY UPLC BEH C18, 1.7 m, 2.150 mm column

Mobile phase: A: 0.1% formic acid in water, B: acetonitrile

Mobile phase gradients: initial 95% A, 95% A (0 min~1.00 min), 10% A (1.00 min~3.00 min), 10% A (3.00 min~4.20 min), 95% A (4.20 min~4.50 min), 95% A (4.50 min~5.00 min)

Flow rate: 0.300 mL/min

Column temperature: 40 ℃

Injection volume: 4.0 L

2.Mass Spectrometry Conditions

Detection method: multi-reactive ion monitoring (MRM)

Ionization Mode	ESI+
Curtain Gas(CUR)	45.0Psi
Collision Gas(CAD)	8Psi
IonSpray Voltage(IS)	5000.0V
Temperature(TEM)	500.0℃
Ion Source Gas 1(GS1)	55Psi
Ion Source Gas 1(GS2)	55Psi

Table 2. Targes Characteristic Ions (*Quantifier Ion)

Targets	Parent Ion (m/z)	Daughter Ion (m/z)	Retention Time (min)	DP (V)	CE (V)
Metronidazole	172.1	128.2*	2.51	30	20
		82.1			34
Metronidazole-D3	175.0	131.0	2.49	30	20
Hydroxymetronidazole	188.2	123.2*	2.38	60	19
		126.2			34
Metronidazole-D2	190.0	125.2	2.37	60	13
Dexmethylnidazole	142.0	96.0*	2.64	30	24
		81.2			40
Dimezole-D3	145.0	99.0	2.62	60	15
Hydroxydimetridazole	158.0	140.3*	2.53	60	16
		55.2			25
Hydroxydimetridazole-D3	161.0	143.0	2.52	60	22

Results

Table 3. Nitroimidazoles in Beef at 2.50 µg/kg

Targets	Recovery Rate (%)			Average Recovery (%)	RSD (%)
	1	2	3		
Metronidazole	98.4	94.8	100	97.7	2.73
Hydroxymetronidazole	102	96.4	93.4	97.3	4.49
Dexmethylnidazole	97.2	93.4	100	96.9	3.42
Hydroxymetronidazole	112	98.6	103	105	6.53

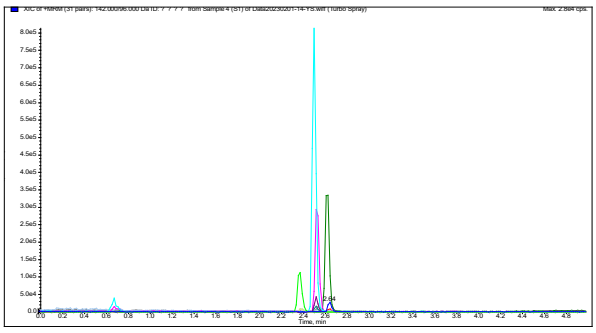
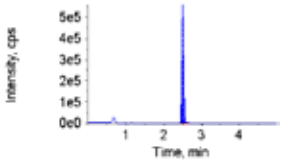
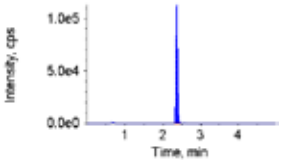


Figure 1. Total Ion Flow Diagram of Nitroimidazole

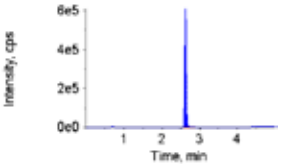
Metronidazole-D3
175.000/131.000 Da



Hydroxymetronidazole-D2
190.000/125.200 Da



Demetrizole-D3
145.000/99.000 Da



Hydroxydimetridazole-D3
161.000/143.000 Da

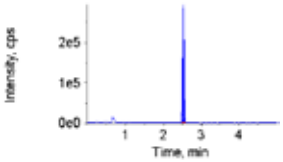
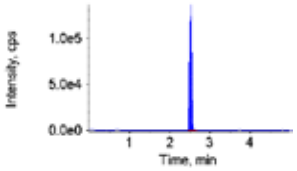
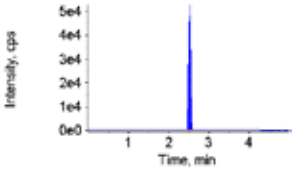


Figure 2. Characteristic Ion Mass Chromatogram of Nitroimidazoles Internal Standard Solution (20.0μg/L)

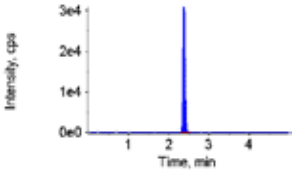
Flagyl
172.100/128.200 Da



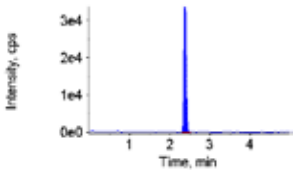
Flagyl (Qualitative)
172.100/82.100 Da



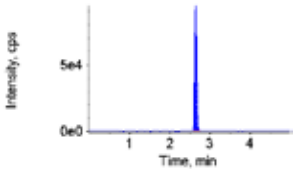
Metronidazole Hydroxylate
188.200/123.200 Da



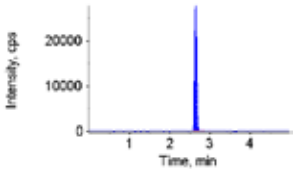
Metronidazole Hydroxylate
188.200/126.200 Da



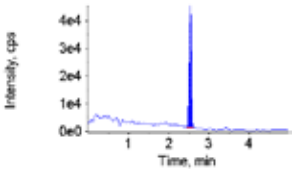
Dimenidazole
142.000/96.000 Da



Dimenidazole (Qualitative)
142.000/81.200 Da



Hydroxydimenidazole
158.000/140.300 Da



Hydroxydimenidazole (Qualitative)
158.000/55.200 Da

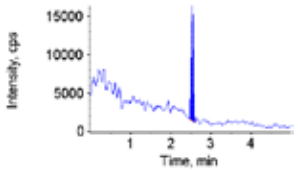


Figure 3. Characteristic Ion Mass Chromatogram of Nitroimidazoles Standard Solution (2.00μg/L)

Ordering Information

Cat. #	Description	Qty.
COMCX360	Copure® MCX Cartridge, 60 mg / 3 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6mm hole	100 Pcs/Box

36 Veterinary Drugs Resides in Beef and Shrimp (Copure® HLB Lim Cartridges)

Introduction

Comparing to traditional SPE cartridges, Copure® HLB Lim Cartridges for Multi-Residue Analysis of Veterinary Drug remove various interferers such as fats, phospholipids, and pigments more rapidly to reduce the matrix effect. The preparation procedures have been simplified to save cost and time by skipping activation and equilibration.

Through low, medium, and high level spiked experiments, the target compounds of 36 veterinary drug residues such as tetracyclines, sulfonamides and quinolones in Beef and shrimp have been determined. Biocomma established a UPLC-MS/MS method with good recovery and precision for your reference.

Experiment

Extraction

Accurately weigh 1 g of the ground sample into a clean centrifuge tube. Add 5 mL of 0.2% formic acid in acetonitrile:water (80:20, v/v) and vortex to mix well. Then, extract by ultrasonic treatment for 20 min, and centrifuge at 8000 r/min for 5 min. The sample is ready for purification.

Purification (Copure® HLB Lim, 200 mg/ 3 mL)

Pipette 2.5 ml supernatant of extracted sample to a Copure®HLB Lim Cartridge. Collect the filtrate and evaporate to dryness. Redissolve to 1 mL by 0.1% formic acid in water-methanol (9:1). After filter by membrane, the sample is ready for UPLC-MS/MS analysis.

Preparation of Standard Curve Solution

Prepare blank sample in the same procedures. After collecting the eluent, add internal standard solution. Evaporate and dilute the concentration of 2 μg/L, 10 μg/L, 50 μg/L, 100μ g/L, 200 μg/L, and 500 μg/L.

Instrument Conditions

1.Chromatographic Conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)
HPLC column: Commasil® Specialized Column for Veterinary Drug Residues (2.1 mm×100 mm, 3μm)
Mobile phase A: water (0.1% formic acid)
Mobile phase B: 0.1% formic acid solution in methanol-acetonitrile (2:8)
Flow rate: 0.3 mL/min
Column temperature: 35 ℃
Injection volume: 10 μL

Table 1. Gradient Elution Program

Time (min)	A(%)	B(%)
0	98	2
3.0	90	10
8.0	65	35
10.0	20	80
11.0	5	95
12.0	98	2

2.Mass Spectrometry Conditions

Ion source: HESI
Electrospray voltage: 3500 V
Sheath gas pressure: 40 arb
Auxiliary gas pressure: 2 arb
Ion transfer tube: 380 ℃
Auxiliary gas temperature: 350 ℃

Table 2. Targets, Retention Times and Characteristic Ions (*Quantifier Ion)

NO.	Targets	Retention Time (min)	Parent Ion	Daughter Ion
1	Acesulfame	3.18	215.0	108.0、155.9*
2	Sulfapyridine	4.67	250.1	155.8*、183.9
3	Sulfadiazine	3.79	251.1	92.1、155.9*
4	Sulfamethoxazole	7.67	254.0	108.1、155.9*
5	Sulphathiazole	4.62	256.0	155.9*、92.1
6	Flumequine	8.59	262.0	201.9、244.1*
7	Oxolinic Acid	10.07	262.0	215.9、244.1*
8	Sulfamerazine	5.03	265.1	155.8*、171.8
9	Sulfamethoxazole	8.12	268.0	113.0、155.8*
10	Sulfamethiadiazole	6.26	271.0	92.1、155.9*
11	Benzoylsulfonamide	8.59	277.0	107.9、155.9*
12	Sulfadimetine	3.22	279.1	124.0*、185.8
13	Sulfadimidine	5.91	279.1	155.8、185.8*
14	Sulfamethoxypyridazine	6.21	281.0	155.8*、126.0
15	Sulfametoxydiazine	6.36	281.0	155.9*、214.9
16	Sulfamonomethoxine	7.10	281.0	155.9*、214.9
17	Sulfachloropyridazine	7.19	285.0	92.1、155.9*
18	Sulfadoxine	7.61	311.1	155.8*、244.9
19	Sulfadimethoxypyrimidine	9.05	311.1	155.8*、244.8
20	Sulfaphenpyrazole	9.10	315.0	157.9*、159.9
21	Norfloxacin	5.34	320.1	233.0、276.0*
22	Enoxacin	5.15	321.1	234.0、303.0*
23	Ciprofloxacin	5.49	332.0	230.9、288.0*
24	Pefloxacin	5.40	334.2	290.1*、316.1
25	Lomefloxacin	5.67	352.0	265.0*、308.0
26	Dafloxacin	5.76	358.2	314.1、340.0*
27	Enrofloxacin	5.85	360.2	245.0、316.0*
28	Ofloxacin	5.31	362.1	261.1、318.1*
29	Marbofloxacin	4.96	363.1	320.0*、342.0
30	Sarafloxacin	6.28	386.2	299.1、342.1*
31	Difloxacin	6.30	400.2	299.0、356.1*
32	Phthalylsulfathiazole	8.11	404.0	148.9、255.8
33	Doxycycline	7.45	445.2	321.0、428.0*
34	Tetracycline	5.55	445.2	410.0*、427.0
35	Oxytetracycline	5.33	461.2	426.0*、443.0
36	Chlortetracycline	6.92	479.1	444.0*、462.0

Results

Table 3. Spiked 36 Veterinary Drugs Resides Recovery

Targes	Beef						Shrimp					
	10.0 μg/kg		50.0 μg/kg		100.0 μg/kg		10.0 μg/kg		50.0 μg/kg		100.0 μg/kg	
	Recovery (%)	RSD (%) n=3	Recovery (%)	RSD (%) n=3	Recovery (%)	RSD (%) n=3	Recovery (%)	RSD (%) n=3	Recovery (%)	RSD (%) n=3	Recovery (%)	RSD (%) n=3
Acesulfame	102	2.62	80.6	3.46	84.5	1.19	87.3	2.02	84.9	5.67	83.8	4.15
Sulfapyridine	82.2	4.48	74.8	7.76	79.2	4.42	90.6	4.06	89.8	3.26	89.7	4.02
Sulfadiazine	86.9	4.83	78.4	6.89	80.8	5.08	87.1	3.87	86.9	5.28	80.7	4.31
Sulfamethoxazole	89.3	3.25	79.8	2.57	83.6	2.03	101	2.25	89.7	3.34	89.4	2.11
sulphathiazole	80.5	3.46	78.7	2.59	84.6	4.33	87.9	4.59	92.3	5.82	85.5	3.98
Flumequine	84.8	7.69	82.3	10.3	76.7	6.79	89.1	7.75	89.4	6.69	84.1	6.71
Oxolinic acid	70.4	11.5	74.8	12.1	78.6	7.94	89.2	6.98	87.7	7.52	88.2	5.97
Sulfamerazine	89.6	1.12	80.3	1.53	80.7	1.81	86.4	3.25	93.0	2.26	85.1	1.90
Sulfamethoxazole	110	6.67	82.8	3.38	89.5	3.86	105	2.65	93.0	3.51	93.1	1.31
Sulfamethiadiazole	85.3	3.32	79.7	4.41	80.4	4.65	82.5	6.01	85.7	2.92	90.7	2.27
Benzoylsulfonamide	93.9	7.85	79.8	8.86	82.2	5.69	85.2	8.86	83.5	6.91	78.9	4.96
sulfadimetine	98.6	3.66	83.8	4.25	78.9	3.32	83.7	5.45	80.9	5.46	78.5	4.84
sulfadimidine	79.7	2.86	81.5	2.27	74.8	2.28	93.5	4.33	91.6	2.33	82.5	5.01
Sulfamethoxypyridazine	76.5	6.03	78.6	5.59	83.3	3.39	94.3	4.53	90.4	4.64	85.7	5.64
Sulfametoxydiazine	78.9	5.82	79.5	5.66	83.4	4.01	96.2	4.92	91.2	5.54	88.6	4.69
Sulfamonomethoxine	95.6	5.51	76.4	3.52	78.4	3.38	86.0	3.66	90.3	3.77	83.8	5.29
Sulfachloropyridazine	70.8	6.35	66.9	7.58	74.2	4.56	94.5	4.57	85.9	3.59	101	4.36
Sulfadoxine	77.9	4.89	80.1	3.92	84.6	4.82	86.5	5.69	93.4	3.35	86.2	2.35
Sulfadimethoxypyrimidine	68.4	6.76	79.2	4.05	75.8	4.53	97.3	5.56	82.2	4.84	79.2	2.96
Sulfaphenpyrazole	66.8	8.22	69.8	6.97	71.5	4.75	77.1	7.71	76.7	6.82	76.4	6.15
Norfloxacin	74.4	4.85	80.9	4.52	78.5	2.83	91.1	1.92	89.6	0.97	86.7	1.34
Enoxacin	78.6	3.39	79.6	4.59	82.3	2.29	84.8	2.55	88.0	1.15	92.9	0.88
Ciprofloxacin	75.4	5.62	81.7	3.95	81.3	4.07	94.1	3.16	90.7	3.02	91.7	2.06
Pefloxacin	79.6	6.23	84.0	4.16	79.4	3.86	83.3	6.53	90.4	5.83	85.0	3.78
Lomefloxacin	76.5	5.54	81.6	5.81	78.9	3.16	91.6	4.52	93.2	2.24	89.1	2.58
Dafloxacin	81.8	5.96	82.2	5.57	78.9	4.87	87.7	5.69	90.5	5.57	86.5	4.43
Enrofloxacin	77.5	5.83	78.8	4.36	80.3	3.82	86.0	3.35	94.3	2.67	86.8	2.17
Ofloxacin	83.3	4.25	76.5	2.28	78.6	2.98	91.1	4.29	93.4	1.01	88.0	1.16
Marbofloxacin	78.3	4.53	75.4	3.85	80.9	3.73	90.3	4.14	91.7	5.04	86.8	2.30
Sarafloxacin	67.6	7.59	78.4	5.82	77.6	5.69	79.4	7.85	92.8	5.85	84.4	4.48
Difloxacin	76.5	6.08	79.9	5.64	82.4	4.56	83.2	5.17	93.3	6.06	84.2	5.45
phthalylsulfathiazole	-	-	88.7	4.96	83.6	3.38	77.8	10.8	69.6	7.58	72.2	6.54
Doxycycline	66.3	6.76	72.8	7.75	79.5	5.85	72.2	9.98	88.1	6.36	86.3	5.66
Tetracycline	78.3	8.57	73.9	7.26	75.8	6.96	73.6	10.1	93.6	6.97	98.0	7.72
Oxytetracycline	65.4	9.29	63.8	8.02	72.6	7.64	63.6	8.86	76.6	5.94	76.9	6.19
Chlortetracycline	67.5	9.06	72.2	6.87	73.9	7.05	84.8	5.78	95.5	4.85	97.8	4.51

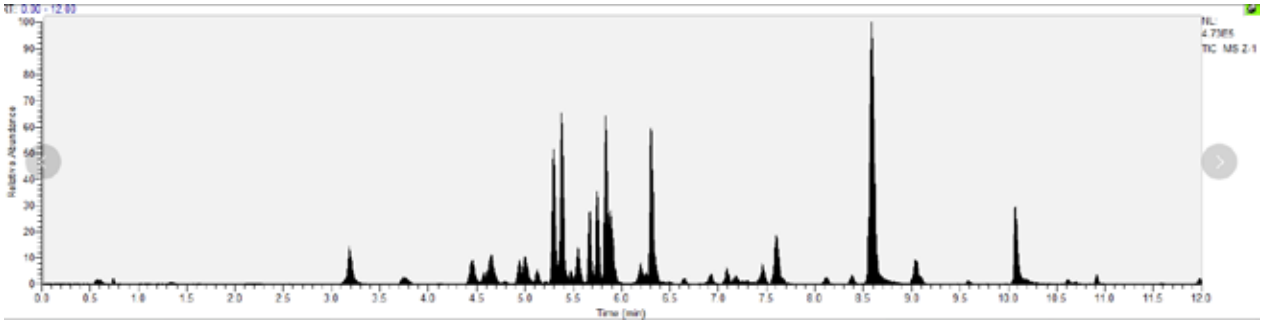


Figure 1. Total Ion Chromatogram of 36 Veterinary Drug Residues in Beef at 10 µg/kg

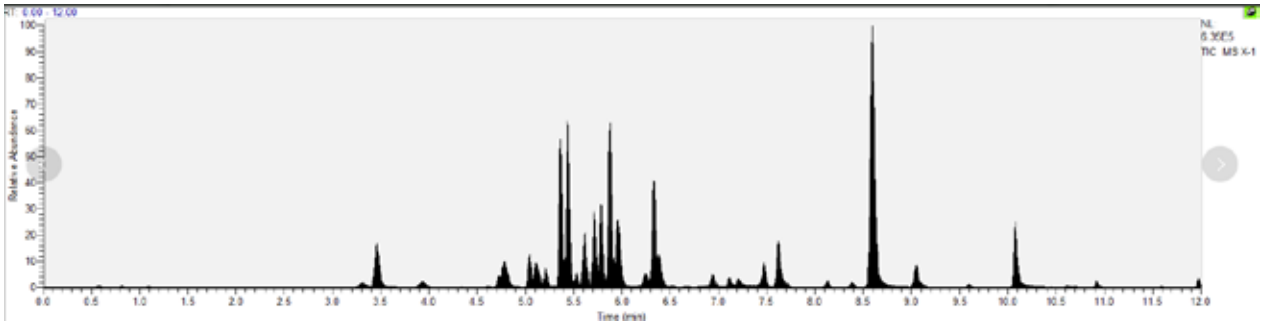


Figure 2. Total Ion Chromatogram of 36 Veterinary Drug Residues in Shrimp 10µg/kg

Ordering Information

Cat.	Description	Qty.
COHLB Lim-3200	Copure® HLB Lim Cartridges, 200 mg / 3mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
SF250-22-NL	Syringe Filters NL / Φ25 mm / 0.22 µm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

Dexamethasone, Betamethasone & Atropine Resides in Beef (Copure® HLB-Lim Cartridges)

Introduction

Comparing to traditional SPE cartridges, Copure® HLB Lim Cartridges for Multi-Residue Analysis of Veterinary Drug remove various interferers such as fats, phospholipids, and pigments more rapidly to reduce the matrix effect. The preparation procedures have been simplified to save cost and time by skipping activation and equilibration.

Biocomma established a UPLC-MS/MS method for detection of dexamethasone, betamethasone and atropine in Beef with good recovery and precision for your reference. The recoveries were 85-110% with RSD less than 10% at low, medium, and high spiked concentration levels.

Experiment

Extraction

Accurately weigh 2 g of ground Beef into a clean centrifuge tube. Add 10 mL of 0.2% formic acid in acetonitrile:water (80:20, v/v) and vortex to mix well. Then, extract by ultrasonic treatment for 15 min, and centrifuged at 8000 r/min for 5 min. The sample is ready for purification.

Purification (Copure® HLB Lim, 200 mg/ 3 mL)

Pipette 1.5 ml supernatant of extracted sample to a Copure® HLB Lim Cartridge. Collect the filtrate and evaporate to dryness. Redissolve to 1 mL by 0.1% formic acid in water-methanol (9:1). After filter by nylon membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Prepare blank sample in the same procedures. After collecting the eluent, add internal standard solution. Evaporate and dilute the concentration of 2 μg/L, 10 μg/L, 20 μg/L, 50 μg/L, 100 μg/L and 200 μg/L.

Instrument Conditions

1.Chromatographic Conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)

Chromatographic column: Commasil® Specialized Columns for Veterinary Residues (2.1 mm×100 mm, 3 μm)

Mobile phase A: water (0.1 % formic acid)

Mobile phase B: methanol: acetonitrile = 2:8 (0.1 % formic acid)

Flow rate: 0.3 mL/min

Column temperature: 35 ℃

Injection volume: 10 μL

Table 1. Gradient Elution Program

Time (min)	A(%)	B(%)
0	98	2
3.0	85	15
6.0	65	35
8.5	5	95
9.0	98	2
10.0	98	2

2.Mass Spectrometry Conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure:40 arb

Auxiliary gas pressure: 10 arb

Ion exchange tube: 380 ℃

Auxiliary air temperature: 350 ℃

Table 2. Targets, Retention Times and Characteristic Ions (*Quantifier Ion)

NO.	Targets	Retention Time (min)	Parent Ion	Daughter Ion
1	Atropine	4.15	290.2	93.1, 124.1*
2	Betamethasone	8.18	393.1	355.1*, 373.1
3	Dexamethasone	8.15	393.2	355.1, 373.1*

Results

Table 3. Spiked Recovery Results

Targets	Beef					
	10.0 μg/kg		50.0 μg/kg		100.0 μg/kg	
	Recovery (%)	RSD (%) n=3	Recovery (%)	RSD (%) n=3	Recovery (%)	RSD (%) n=3
Atropine	108	4.29	98.2	2.26	92.2	1.33
Betamethasone	104	6.68	96.4	3.76	91.3	2.42
Dexamethasone	105	7.20	96.5	3.45	91.4	2.08

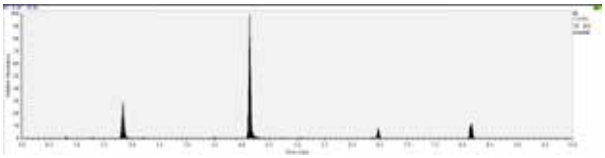


Figure 1. TIC of Dexamethasone, Betamethasone and Atropine Residues in Beef at 50 μg/kg.

Ordering Information

Cat.	Description	Qty.
COHLB Lim-3200	Copure® HLB Lim Cartridges, 200 mg / 3mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
SF250-22-NL	Syringe Filters NL / Φ25 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

Multi- Residues of Veterinary Drug in Meats (Copure® Veterinary Drug Residue Specialty QuEChERS)

Introduction

Sulfonamide, quinolone and tetracycline antibiotics are common human-animal antibiotics, used in both humans and animals. Due to their cost-effectiveness and effective antimicrobial properties, they are widely employed in aquaculture. Biocomma optimized the QuEChERS method with recovery rate of 60%~110% and RSD less than 10% for your reference.

Experiment

Extraction (Copure® QuEChERS, Cat. #: COQ050051H)

Weigh 2.0 g of pulverized meat sample in a 50 mL centrifuge tube. Add 2 mL of Na_2EDTA-McIlvaine buffer (pH=4), then vortex for 1 min. Add 10 mL of 1% acetonitrile acetate solution. Vortex for 1 min, then add the QuEChERS extraction salt. Vortex to mix well. Extract by ultrasonic treatment for 10 min, and centrifuge at 5000 r/min for 2 min. The sample is ready for purification.

Purification (Copure® QuEChERS, Cat. #: COQ015605H)

Add 6 mL of prepared supernatant and the QuEChERS purification salt into a clean tube. Vortex at 2500 r/min for 2 min, then centrifuge at 8000 r/min for 5 min. Evaporate 5 mL of supernatant to nearly dryness. Dilute to 1 mL by methanol solution with 0.1% formic acid (water: methanol = 9:1). The sample is ready for analysis.

Preparation of Matrix Standard Curve Solutions

Prepare blank sample in the same procedures. Add internal standard to prepare the concentrations of 2 μg/L, 10 μg/L, 50 μg/L, 100 μg/L, 200 μg/L, and 500 μg/L for standard curve.

Instrumental Conditions

1.Chromatographic Conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)
Chromatographic column: Commasil® Veterinary Residues Column BEH T-C18 (2.1 mm×100 mm, 3 μm)
Mobile phase A: water (containing 0.1 % formic acid)
Mobile phase B: methanol: acetonitrile = 2:8 (containing 0.1 % formic acid)
Flow rate: 0.3 mL/min
Column temperature:35℃
Injection volume: 10 μL
Table 1. Gradient Elution Program

Time (min)	A/%	B/%
0	98	2
3.0	90	10
8.0	65	35
10.0	20	80
11.0	5	95
12.0	98	2
14.0	98	2

2.Mass Spectrometry Conditions

Ion source: HESI
Electrospray voltage: 3500 V
Sheath gas pressure: 40 arb
Auxiliary gas pressure: 2 arb
Ion exchange tube: 380 ℃
Auxiliary air temperature: 350 ℃
Table 2. Targets, Retention Times and Characteristic Ions (Quantitative Ion)

No.	Targets	Retention Time (min)	Parent Ion	Daughter Ion
1	Acesulfame	3.43	215.0	108.0、155.9*
2	Sulfapyridine	5.01	250.1	155.8*、183.9
3	Sulfadiazine	4.10	251.1	92.1、155.9*
4	Sulfamethoxazole	7.77	254.0	108.1、155.9*
5	Sulfathiazole	4.92	256.0	155.9*、92.1
6	Sulfamethazine	5.32	265.1	155.8*、171.8
7	Sulfamethoxazole	8.20	268.0	113.0、155.8*
8	Sulfamethiadiazole	6.41	271.0	92.1、155.9*
9	Benzosulfonamide	8.70	277.0	107.9、155.9*
10	Sulfamethazine	3.56	279.1	124.0*、185.8
11	Sulfamethazine	6.16	279.1	155.8、185.8*
12	Sulfamethoxypyridazine	6.51	281.0	155.8*、126.0
13	Sulfamethoxine	6.36	281.0	155.9*、214.9
14	Sulfamethoxine	7.22	281.0	155.9*、214.9
15	Sulfachloropyridazine	7.30	285.0	92.1、155.9*
16	Sulfadimethoxine	7.71	311.1	155.8*、244.9
17	Sulfadimethoxine	9.11	311.1	155.8*、244.8
18	Sulfaphenpyrazole	9.17	315.0	157.9*、159.9
19	Phthalsulfathiazole	8.16	404.0	148.9、255.8*
20	Flumequine	10.09	262.0	201.9、244.1*
21	Oxolinic Acid	8.64	262.0	215.9、244.1*
22	Norfloxacin	5.15	320.1	233.0、276.0*
23	Enoxacin	4.95	321.1	234.0、303.0*
24	Ciprofloxacin	5.30	332.0	230.9、288.0*
25	Pefloxacin	5.21	334.2	290.1*、316.1
26	Lomefloxacin	5.49	352.0	265.0*、308.0
27	Dafloxacin	5.57	358.2	314.1、340.0*
28	Enrofloxacin	5.67	360.2	245.0、316.0*
29	Ofloxacin	5.12	362.1	261.1、318.1*
30	Marbofloxacin	4.75	363.1	320.0*、342.0
31	Sarafloxacin	6.08	386.2	299.1、342.1*
32	Difloxacin	6.11	400.2	299.0、356.1*
33	Doxycycline	7.33	445.2	321.0、428.0*
34	Tetracycline	5.55	445.2	410.0*、427.0
35	Oxytetracycline	5.38	461.2	426.0*、443.0
36	Chlortetracycline	6.88	479.1	444.0*、462.0

Results

Table 3. Results of Spiked Recovery of Multi- Residues of Veterinary Drug

Targets	Beef						Shrimp					
	10.0 μg/kg		50.0 μg/kg		100.0 μg/kg		10.0 μg/kg		50.0 μg/kg		100.0 μg/kg	
	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3
Acesulfame	101	3.98	88.8	2.86	80.6	3.95	98.2	3.09	93.6	2.82	82.5	2.71
Sulfapyridine	96.5	1.64	85.9	2.25	86.9	1.73	106	1.64	94.7	1.39	87.5	2.72
Sulfadiazine	92.5	4.15	81.1	3.27	78.6	4.19	96.9	4.08	91.1	2.23	84.3	2.37
Sulfamethoxazole	101	4.03	87.1	3.02	77.4	3.65	91.7	4.82	101	2.27	89.5	3.11
Sulfathiazole	89.2	5.30	82.3	4.27	80.7	2.64	99.7	1.03	98.8	1.15	87.3	2.13
Sulfamethazine	97.9	2.77	91.5	1.81	85.6	2.06	94.9	4.35	99.7	3.58	90.4	2.43
Sulfamethoxazole	104	1.61	88.5	1.32	87.8	1.64	85.8	1.93	91.2	2.12	87.9	1.94
Sulfamethiadiazole	100	2.00	80.6	2.71	78.6	2.44	108	1.45	103	1.65	93.9	1.92
Benzosulfonamide	106	1.85	96.1	2.11	93.1	1.24	97.7	7.30	92.4	4.24	96.9	2.15
Sulfamethazine	75.4	2.85	82.1	2.31	78.6	2.26	106	3.89	92.6	2.46	83.1	2.77
Sulfamethazine	97.9	1.91	93.2	2.04	83.8	2.45	105	6.41	103	4.37	89.9	3.39
Sulfamethoxypyridazine	100	2.43	84.0	2.72	87.4	2.15	108	4.96	102	3.29	90.2	3.34
Sulfamethoxine	107	5.02	80.4	4.25	89.6	1.78	110	3.97	101	2.17	90.6	1.68
Sulfamethoxine	94.9	5.22	87.2	2.86	88.9	2.41	95.2	5.66	102	3.68	98.3	2.59
Sulfachloropyridazine	102	1.96	87.5	2.36	85.2	2.07	101	2.37	95.2	2.35	90.9	2.14
Sulfadimethoxine	110	3.84	79.8	2.75	87.5	2.35	105	3.22	101	2.79	85.8	2.71
Sulfadimethoxine	108	4.24	94.5	2.78	87.6	2.32	83.8	8.43	107	7.58	98.	3.94
Sulfaphenpyrazole	105	8.38	96.7	4.94	92.4	3.25	83.6	3.64	110	6.63	106	6.34
Phthalsulfathiazole	98.3	5.77	97.7	4.92	98.1	3.69	89.5	4.94	90.0	3.62	108	5.27
Flumequine	100	4.89	106	3.82	91.8	4.27	85.5	4.14	95.8	2.82	83.7	2.16
Oxolinic Acid	108	2.94	100	2.25	87.4	3.32	87.6	4.82	104	2.43	89.5	2.36
Norfloxacin	79.3	8.19	69.6	5.89	78.3	4.67	69.8	7.32	82.5	4.38	71.8	2.45
Enoxacin	78.9	4.74	78.6	3.22	75.5	2.87	80.2	5.73	94.4	2.42	94.5	3.49
Ciprofloxacin	101	6.55	81.2	4.84	77.8	2.59	74.4	6.12	84.7	2.87	76.9	2.51
Pefloxacin	95.6	1.66	90.4	2.08	81.9	2.85	73.2	6.87	91.8	2.78	78.5	3.82
Lomefloxacin	96.6	5.63	85.3	3.89	77.5	4.82	77.5	6.65	96.9	4.83	82.9	2.87
Dafloxacin	99.2	1.87	85.5	2.32	81.6	2.36	76.2	4.89	86.7	2.86	79.7	3.89
Enrofloxacin	108	2.66	96.2	3.94	82.7	3.83	77.4	8.22	100	4.72	78.5	5.93
Ofloxacin	105	4.66	91.7	2.92	84.9	2.88	82.8	4.16	97.4	2.97	78.7	3.74
Marbofloxacin	86.8	2.81	84.9	1.87	78.4	3.63	76.9	5.21	86.4	2.20	82.5	2.36
Sarafloxacin	95.1	7.51	88.9	2.86	86.7	2.73	80.4	5.72	91.2	2.76	78.2	3.85
Difloxacin	98.2	4.76	93.4	2.48	84.9	2.35	86.3	4.22	103	4.16	78.5	3.35
Doxycycline	81.6	6.06	90.9	4.46	84.8	3.28	68.4	7.91	74.1	4.92	78.9	3.95
Tetracycline	85.8	2.78	72.7	3.66	74.1	3.49	93.2	2.43	78.4	2.39	84.2	3.21
Oxytetracycline	72.4	3.41	66.7	4.22	67.4	3.86	81.9	4.58	73.6	2.23	79.8	2.72
Chlortetracycline	94.9	3.74	90.6	3.28	85.5	2.77	68.5	5.11	83.3	2.26	88.8	2.18

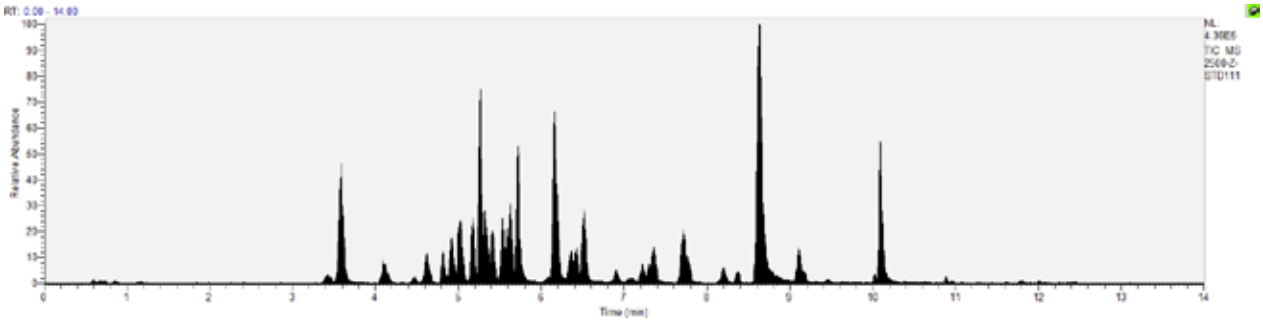


Figure 1. TIC of Sulfonamides, Quinolones and Tetracyclines in Beef at 10 µg/kg

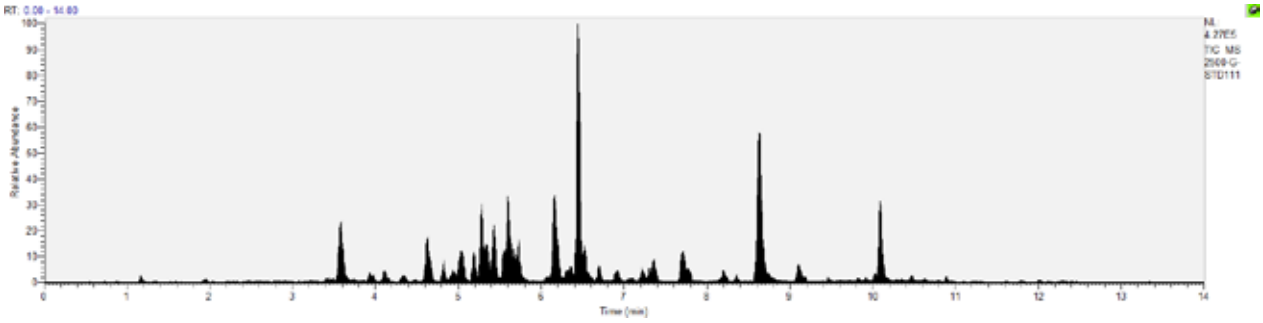


Figure 2. TIC of Sulfonamides, Quinolones and Tetracyclines in Shrimp at 10 µg/kg

Ordering Information

Cat. #	Description	Qty.
COQ050051H	Copure® Veterinary Drug Residue Specialty QuEChERS Extraction Kit	50 Pcs/Box
COQ015605H	Copure® Veterinary Drug Residue Specialty QuEChERS Clean-Up Kit	50 Pcs/Box
SDC-3000-D	biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
BN24	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 µm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

Comparison of QuEChERS for 19 Residues of Sulfonamide in Beef

Introduction

Sulfonamides, cost-effective and widely used in pig farming, can accumulate in Beef, posing health risks. High fat and phospholipid in Beef complicate residue detection. The accumulated phospholipid on chromatographic column not only reduces lifespan, but also causes ion suppression in mass spectrometry, resulting in low recovery rates and impacting the sensitivity and reproducibility of instrumental analysis.

Biocomma has developed a QuEChERS method with simplified operation and good recovery to effectively remove phospholipids, adsorb lipids, and detect 19 residues of sulfonamide veterinary drugs in Beef. The spiked recoveries at both 5 ng/g and 10 ng/g were 60-120 % with RSD less than 10 % for your reference.

Experiment

Extraction (Copure® QuEChERS, Cat. #: COQ050050H)

Weight 2.0 g of ground Beef into a 50 mL centrifuge tube. Added 4 mL of water, and vortex for 2 min. Add 10 mL of 1% acetic acid acetonitrile, and vortex for 10 min. Add QuEChERS extraction salt. Vortex for 2 min, and centrifuged at 5000 r/min and 4 ℃ for 5 min. Let stand for 15 minutes, then the upper acetonitrile layer is ready for purification.

Purification (Copure® QuEChERS, Cat. #: COQ015427H)

Add 6 mL of prepared supernatant and the QuEChERS purification salt into a clean tube. Vortex for 2 min, then centrifuge at 5000 r/min for 5 min. Evaporate 4 mL of supernatant to nearly dryness at 40 ℃ . Dilute to 1 mL by methanol solution with 0.1% formic acid (water: methanol = 9:1). Filter by a 0.22 μm membrane. The sample is ready for analysis.

Instrumental Conditions

1.Chromatographic Conditions

Instrument：UPLC-MS/MS (Thermo Fisher TSQ Endura)

Chromatographic column：Commasil® veterinary drug residues special columns (2.1 mm×100 mm, 3 μm)

Mobile phase A: water containing 0.1 % formic acid

Mobile phase B: methanol-acetonitrile solution containing 0.1% formic acid (methanol: acetonitrile=2:8)

Flow rate: 0.3 mL/min

Column temperature: 35 °C

Injection volume: 10 μL

Table 1. Gradient Elution Program

Time (min)	A(%)	B(%)
0	98	2
3.0	90	10
8.0	65	35
10.0	20	80
11.0	5	95
12.0	98	2
14.0	98	2

2.Mass Spectrometry Conditions

Ion source：HESI

Electrospray voltage：3500 V

Sheath gas pressure：40 arb

Auxiliary air pressure：2 arb

Ion exchange tube：380 ℃

Auxiliary air temperature：350 ℃

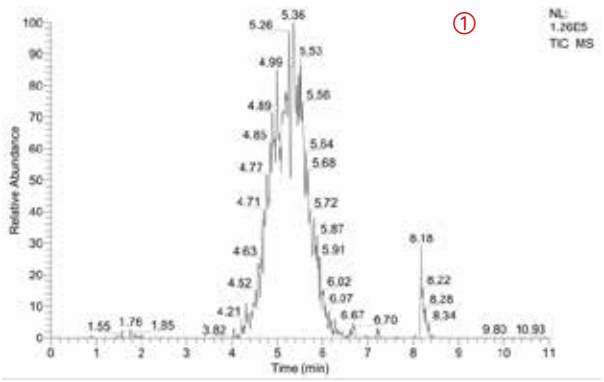
Table 2. Targets, Retention Times and Characteristic Ions (* is the Quantitative Ion)

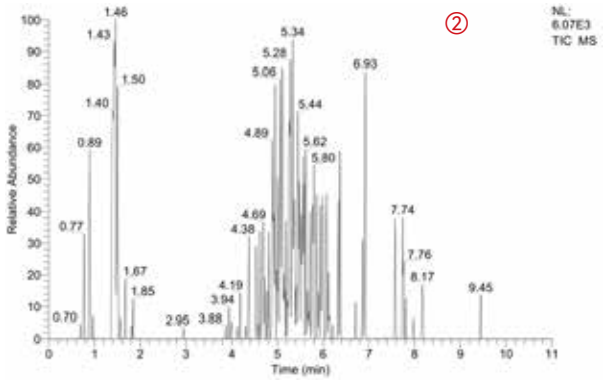
No.	Targets	Retention Time (min)	Parent Ion	Daughter Ion
1	Acesulfonamide	3.41	215.0	108.0、155.9*
2	Sulfapyridine	4.97	250.1	155.8*、183.9
3	Sulfadiazine	4.06	251.1	92.1、155.9*
4	Sulfamethoxazole	7.75	254.0	108.1、155.9*
5	Sulfathiazole	4.88	256.0	155.9*、92.1
6	Sulfamethylpyrimidine	5.27	265.1	155.8*、171.8
7	Sulfamethoxazole	8.18	268.0	113.0、155.8*
8	Sulfamethadiazole	6.38	271.0	92.1、155.9*
9	Phenylsulfonamide	8.65	277.0	107.9、155.9*
10	Sulfamethoxazine	3.56	279.1	124.0*、185.8
11	Sulfamethazine	6.11	279.1	155.8、185.8*
12	Sulfamethoxypyridazine	6.33	281.0	155.8*、126.0
13	Sulfonamide	6.47	281.0	155.9*、214.9
14	Sulfamethoxine	7.19	281.0	155.9*、214.9
15	Sulfachlorpyridazine	7.28	285.0	92.1、155.9*
16	Sulfamethoxazine	7.68	311.1	155.8*、244.9
17	Sulfamethoxine	9.08	311.1	155.8*、244.8
18	Sulfaphenazole	9.14	315.0	157.9*、159.9
19	Phthalosulfathiazole	8.12	404.0	148.9、255.8

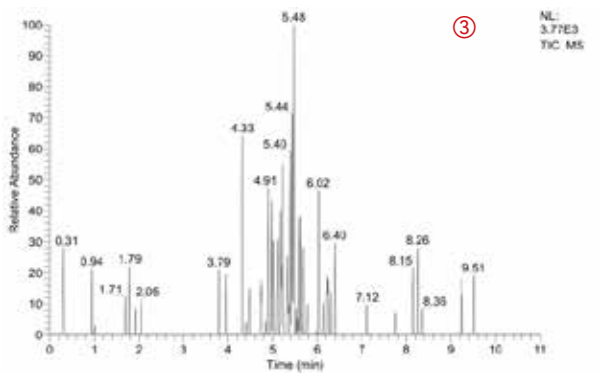
Results

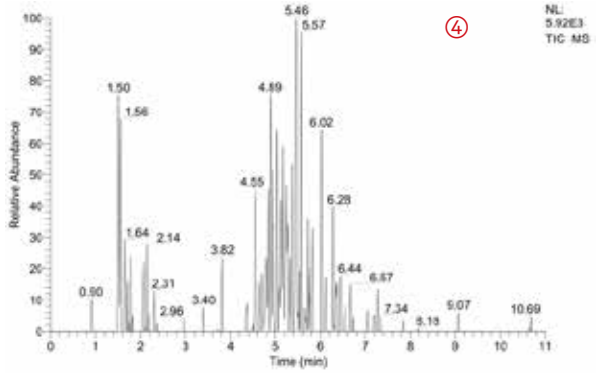
Table 3. Results of Spiked Recovery of 19 Residues of Sulfonamide Veterinary Drugs

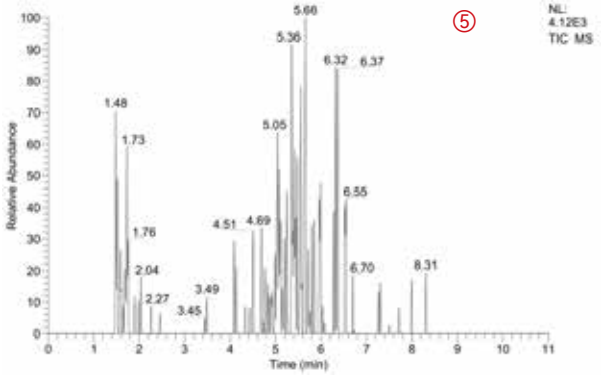
Targets	Spike level (ng/g)	Copure®		Brand A		Brand S		Brand B	
		Recovery rate (%)	RSD (%)	Recovery rate (%)	RSD (%)	Recovery rate (%)	RSD (%)	Recovery rate (%)	RSD (%)
Acesulfame	5.0	82.1	5.11	72.1	5.11	76.5	5.65	56.5	5.75
	10.0	72.3	4.52	51.5	4.25	64.1	6.25	51.5	4.75
Sulfapyridine	5.0	74.6	5.67	86.4	5.16	76.1	5.65	71.2	5.25
	10.0	74.0	5.05	96.5	4.29	95.8	5.12	96.9	6.15
Sulfadiazine	5.0	72.8	5.15	68.9	6.32	71.5	7.36	56.7	6.45
	10.0	77.7	5.12	54.9	5.96	73.6	7.96	61.0	7.52
Sulfamethoxazole	5.0	80.2	4.53	36.9	5.61	56.9	4.15	52.2	6.86
	10.0	71.9	5.12	35.5	6.25	75.5	5.35	66.7	5.12
Sulfathiazole	5.0	69.5	4.53	83.1	6.90	78.5	4.11	68.1	5.08
	10.0	76.9	5.68	73.9	6.54	88.6	6.02	81.8	5.19
Sulfamethazine	5.0	76.1	5.13	64.1	7.39	66.5	5.11	56.9	4.75
	10.0	76.4	4.54	53.1	6.89	79.1	5.03	72.1	5.16
Sulfamethoxazole	5.0	70.2	5.69	54.7	6.15	60.2	4.75	56.4	4.19
	10.0	73.9	4.07	42.6	6.41	62.3	5.15	50.8	6.26
Sulfamethiadiazole	5.0	70.1	5.32	51.5	6.17	50.5	4.95	49.5	6.51
	10.0	71.6	5.03	50.8	5.92	70.4	5.02	65.8	6.29
Benzosulfonamide	5.0	60.5	7.55	----	6.70	40.5	5.17	23.9	5.85
	10.0	59.5	5.14	28.9	6.25	42.8	4.16	33.5	6.38
Sulfamethazine	5.0	80.6	4.55	50.6	6.61	55.1	5.05	40.6	5.62
	10.0	81.5	5.70	76.6	6.27	61.5	5.96	49.5	6.75
Sulfamethazine	5.0	70.5	6.08	45.1	6.13	40.4	5.16	40.2	6.84
	10.0	74.6	6.35	30.1	6.52	45.6	4.82	30.8	6.95
Sulfamethoxypyridazine	5.0	80.1	5.65	50.5	6.34	58.1	4.94	51.0	6.94
	10.0	80.9	4.56	50.0	6.61	73.1	5.01	80.6	5.15
Sulfamethoxine	5.0	76.5	5.15	54.5	7.58	58.9	4.31	51.5	6.28
	10.0	75.4	6.56	55.8	7.91	73.3	5.19	70.3	7.56
Sulfamethoxine	5.0	74.1	6.71	34.8	5.67	42.1	4.31	28.9	7.19
	10.0	60.9	6.09	23.8	5.13	41.1	5.45	27.5	6.65
Sulfachloropyridazine	5.0	76.9	6.92	41.5	5.95	48.8	5.94	41.4	6.69
	10.0	65.9	7.12	30.9	6.54	56.4	6.15	44.6	7.15
Sulfadimethoxine	5.0	68.4	6.57	29.8	5.25	44.6	5.85	30.4	6.68
	10.0	72.2	6.16	32.9	6.01	62.4	6.13	59.1	5.81
Sulfadimethoxine	5.0	65.5	6.57	20.1	2.16	40.1	6.21	29.1	6.16
	10.0	68.6	6.72	22.5	5.31	55.8	5.31	42.1	6.62
Sulfaphenpyrazole	5.0	65.3	5.10	----	5.25	46.7	5.63	----	5.75
	10.0	61.2	5.15	----	4.55	47.2	4.25	25.9	6.75
Phthalsulfathiazole	5.0	68.5	7.31	56.6	4.16	50.2	6.15	48.2	5.25
	10.0	67.4	7.75	50.1	5.29	52.8	5.61	54.8	6.15











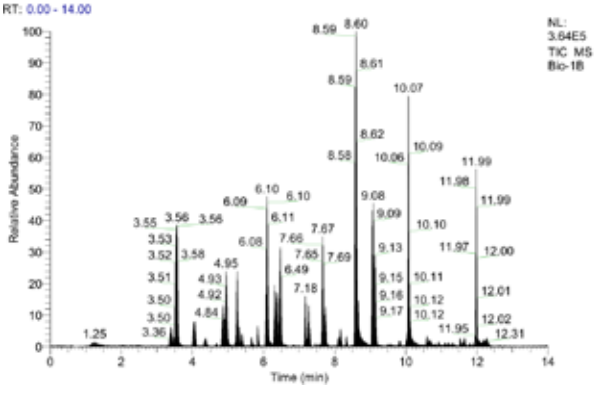


Figure 1. TIC of 19 Residues of Sulfonamide Veterinary Drugs in Beef (Product Ion Scan m/z=184)
① Unpurified Sample
② Purified by Brand A
③ Purified by Brand S
④ Purified by Brand B
⑤ Purified by Copure® QuEChERS

Figure 2. TIC of 19 Residues of Sulfonamide Veterinary Drug in Beef at 5ng/g

Ordering Information

Cat. #	Description	Qty.
COQ050050H	Copure® Veterinary Drug Residue Specialty QuEChERS Extraction Kit	50 Pcs/Box
COQ015427H	Copure® QuEChERS Sulfonamide Purification Kit	50 Pcs/Box
SDC-3000-D	biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
MF047-45-MCE	MCE/Φ47 mm / 0.45 μm	200 Pcs /Box
MF047-45-PTFE	PTFE / Φ47 mm / 0.45 μm	200 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs /Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs /Box

19 Sulfonamide Residues in Beef (Copure® QuEChERS)

Introduction

Sulfonamides, cost-effective and widely used in pig farming, can accumulate in Beef, posing health risks. While SPE cartridges have been the conventional method for purifying sulfonamide, the increasing use of QuEChERS in pesticide residue analysis has prompted customers to seek its application in veterinary drug residue analysis.

Biocomma has developed a QuEChERS method to detect 19 residues of sulfonamide veterinary drugs in Beef. The recovery rate is 76-110% with RSD less than 10% for your reference.

Experiment

Extraction (Copure® QuEChERS, Cat. #: COQ050050H)

Weight 2.0 g of ground Beef into a 50 mL centrifuge tube. Added 4 mL of water, and vortex for 1 min. Add 10 mL of 1% acetic acid acetonitrile, and vortex for 1 min. Add QuEChERS extraction salt. Vortex to mix well. Extract by ultrasonic treatment for 10 min, and centrifuged at 5000 r/min for 2 min. The sample is ready for purification.

Purification (Copure® QuEChERS, Cat. #: COQ015427H)

Add 5 mL of prepared supernatant and the QuEChERS purification salt into a clean tube. Vortex at 2500 r/min for 2 min, then centrifuge at 8000 r/min for 5 min. Evaporate 2 mL of supernatant to nearly dryness. Dilute to 1 mL by methanol solution with 0.1% formic acid (water: methanol = 9:1). The sample is ready for analysis.

Preparation of Standard Curve Solution

Prepare blank sample in the same procedures. Add internal standard to prepare the concentrations of 2 μg/L, 5 μg/L, 10 μg/L, 20 μg/L, 50 μg/L and 100 μg/L.

Instrumental Conditions

1.Chromatographic conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)

Chromatographic column: SinoPak BEH T-C18 (2.1 mm×100 mm, 3 μm)

Mobile phase A: water containing 0.1 % formic acid

Mobile phase B: methanol-acetonitrile solution containing 0.1% formic acid (methanol: acetonitrile=2:8)

Flow rate: 0.3 mL/min

Column temperature: 35 ℃

Injection volume: 10 μL

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.00	98	2
3.00	85	15
6.00	65	35
8.50	5	95
9.00	98	2
10.00	98	2
14.0	98	2

1.Mass Spectrometry Conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure: 40 arb

Auxiliary gas pressure: 2 arb

Ion exchange tube: 380 ℃

Auxiliary air temperature: 350 ℃

Table 2. Targets, Retention Times and Characteristic Ions (* is Quantitative Ions)

No.	Targets	Retention Time (min)	Parent Ion	Daughter Ion
1	Sulfanilamide Acetate	3.06	215.0	108.0, 155.9*
2	Sulfapyridine	4.10	250.1	155.8*, 183.9
3	Sulfadiazine	3.52	251.1	92.1, 155.9*
4	Sulfamethoxazole	6.46	254.0	108.1, 155.9*
5	Sulfathiazole	4.04	256.0	155.9*, 92.1
6	Sulfamethazine	4.36	265.1	155.8*, 171.8
7	Sulfamethizole	6.79	268.0	113.0, 155.8*
8	Sulfamethizole	5.32	271.0	92.1, 155.9*
9	Benzoylsulfonamide	7.22	277.0	107.9, 155.9*
10	Sulfadimethoxine	5.07	279.1	155.8, 185.8*
11	Sulfadimethoxine	5.29	281.0	155.8*, 126.0
12	Sulfamethoxypyridazine	5.38	281.0	155.9*, 214.9
13	Sulfamethoxypyrimidine	5.96	281.0	155.9*, 214.9
14	Sulfamethoxypyrimidine	6.04	285.0	92.1, 155.9*
15	Sulfachloropyridazine	6.41	311.1	155.8*, 244.9
16	Sulfadimethoxypyrimidine	7.47	311.1	155.8*, 244.8
17	Sulfamethoxypyrimidine	5.23	334.2	290.1*, 316.1
18	Sulfamethazine	7.54	315.0	157.9*, 159.9
19	Sulfathiazole	6.72	404.0	148.9, 255.8*

Results

Table 3. Spiked Recovery of 19 Residues of Sulfonamide in Beef

Targets	Beef					
	10.0 μg/kg		50.0 μg/kg		100.0 μg/kg	
	Recovery rate (%)	RSD (%)	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3
Sulfanilamide Acetate	86.8	2.11	90.9	2.80	88.6	1.71
Sulfapyridine	92.5	3.16	96.1	2.68	95.8	2.03
Sulfadiazine	93.3	3.42	96.6	2.73	96.7	2.12
Sulfamethoxazole	88.3	1.77	108	2.39	94.9	1.52
Sulfathiazole	89.5	4.67	91.8	4.24	96.1	2.69
Sulfamethazine	87.6	3.65	100	3.48	96.9	2.38
Sulfamethizole	91.0	4.97	92.5	3.95	95.7	3.26
Sulfamethizole	96.1	3.78	90.2	2.47	103	1.02
Benzoylsulfonamide	88.4	0.83	106	2.15	97.6	1.10
Sulfadimethoxine	83.7	3.46	91.4	2.45	86.7	1.72
Sulfadimethoxine	87.1	2.66	106	2.35	101	1.98
Sulfamethoxypyridazine	81.6	3.06	93.4	4.29	105	3.72
Sulfamethoxypyrimidine	86.2	3.42	88.7	1.74	105	2.44
Sulfamethoxypyrimidine	76.5	3.96	89.6	1.96	90.5	2.01
Sulfachloropyridazine	93.4	0.82	94.5	1.18	93.7	1.87
Sulfadimethoxypyrimidine	87.4	5.16	110	4.89	95.0	2.77
Sulfamethoxypyrimidine	86.2	2.44	110	2.61	102	2.09
Sulfamethazine	78.6	3.13	108	3.46	103	3.32
Sulfathiazole	77.8	2.38	76.8	3.52	91.9	2.06
Sulfamethoxypyrimidine	86.2	2.44	110	2.61	102	2.09
Sulfamethazine	78.6	3.13	108	3.46	103	3.32
Sulfathiazole	77.8	2.38	76.8	3.52	91.9	2.06

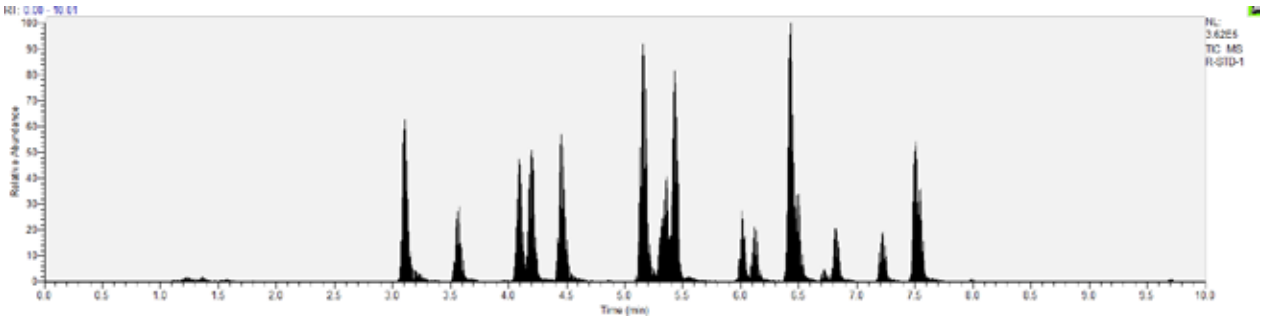


Figure 1. TIC of 19 Sulfonamides Residues in Beef at 50 μg/kg

Ordering Information

Cat. #	Description	Qty.
COQ050050H	Copure® Veterinary Drug Residue Specialty QuEChERS Extraction Kit	50 Pcs/Box
COQ015427H	Copure® QuEChERS Sulfonamide Purification Kit	50 Pcs/Box
SDC-3000-D	biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
BN24	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs /Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs /Box

Tetracycline Residue in Beef & Shrimp (Copure® QuEChERS)

Introduction

Tetracyclines are widely used antibiotics in animal husbandry due to their cost-effectiveness and strong antibacterial activity. Although HLB cartridges have been the conventional method for purification sulfonamide, the need of QuEChERS application in veterinary drug residue analysis increases recently. Biocomma has developed a QuEChERS method to detect tetracycline residues in Beef and shrimp. The recovery rate is greater than 65% with RSD less than 10 % for your reference.

Experiment

Preparation

Na_2EDTA-McIlvaine buffer: weigh 12.9 g of citric acid, 10.9 g of disodium hydrogen phosphate, and 39.2 g of disodium ethylenediaminetetraacetate, and dissolve by 900mL of water. Adjust the pH to 5.0±0.2 with 1 mol/L NaOH. Dilute to 1000 mL with water.

Extraction (Copure® QuEChERS, Cat. #: COQ050051H)

Weight 2.0 g of ground Beef into a 50 mL centrifuge tube. Add 2 mL of Na2EDTA-McIlvaine buffer (pH=4), then vortex for 1 min. Add 10 mL of 1% acetonitrile acetate solution. Vortex for 1 min, then add the QuEChERS extraction salt. Vortex to mix well. Extract by ultrasonic treatment for 10 min, and centrifuge at 5000 r/min for 2 min. The sample is ready for purification.

Purification (Copure® QuEChERS, Cat. #: COQ015601H)

Add 6 mL of prepared supernatant and the QuEChERS purification salt into a clean tube. Vortex at 2500 r/min for 2 min, then centrifuge at 8000 r/min for 5 min. Evaporate 5 mL of supernatant to nearly dryness. Dilute to 1 mL by methanol solution with 0.1% formic acid (water: methanol = 9:1). The sample is ready for analysis.

Preparation of Standard Curve Solution

Prepare blank sample in the same procedures. Add internal standard to prepare the concentrations of 2 μg/L, 10 μg/L, 50 μg/L, 100 μg/L, 200 μg/L and 500 μg/L.

Instrumental Conditions

1.Chromatographic Conditions

Instrument: UPLC-MS/MS (Thermo Fisher TSQ Endura)

Chromatographic column: Yilit SinoPak BEH T-C18 (2.1 mm×100 mm, 3 μm)

Mobile phase A: water containing 0.1 % formic acid

Mobile phase B: methanol-acetonitrile solution containing 0.1% formic acid (methanol: acetonitrile=2:8)

Flow rate: 0.3 mL/min

Column temperature: 35 ℃

Injection volume: 10 μL

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.00	95	5
2.00	70	30
4.00	70	30
4.20	30	70
6.00	30	70
7.00	95	5
8.00	95	5

2.Mass Spectrometry Conditions

Ion source: HESI

Electrospray voltage: 3500 V

Sheath gas pressure: 40 arb

Auxiliary gas pressure: 2 arb

Ion exchange tube: 380 ℃

Auxiliary air temperature: 350 ℃

Table 2. Targets, Retention Times and Characteristic Ions (*is Quantitative Ions)

No.	Targets	Retention Time (min)	Parent Ion	Daughter Ion
1	Doxycycline	3.38	445.2	321.0, 428.0*
2	Tetracycline	2.76	445.2	410.0*, 427.0
3	Oxytetracycline	2.69	461.2	426.0*, 443.0
4	Chlortetracycline	3.23	479.1	444.0*, 462.0

Results

Table 3. Spiked Recovery of Residue in Beef & Shrimp

Targets	Beef						Shrimp					
	10.0 μg/kg		50.0 μg/kg		100.0 μg/kg							
	Recovery rate (%)	RSD (%)	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%)	Recovery rate (%)	RSD (%) n=3	Recovery rate (%)	RSD (%) n=3
Doxycycline	69.3	4.21	84.4	3.74	88.7	2.85	68.1	2.87	76.8	3.36	78.5	2.21
Tetracycline	69.4	2.11	72.6	3.46	74.8	3.26	91.9	2.66	78.6	4.11	88.4	2.27
oxytetracycline	80.3	4.24	74.9	3.62	81.4	2.96	80.4	3.47	79.5	4.19	85.6	3.01
Chlortetracycline	68.7	3.23	84.8	3.27	90.6	3.02	69.8	2.11	74.4	3.25	82.9	2.07

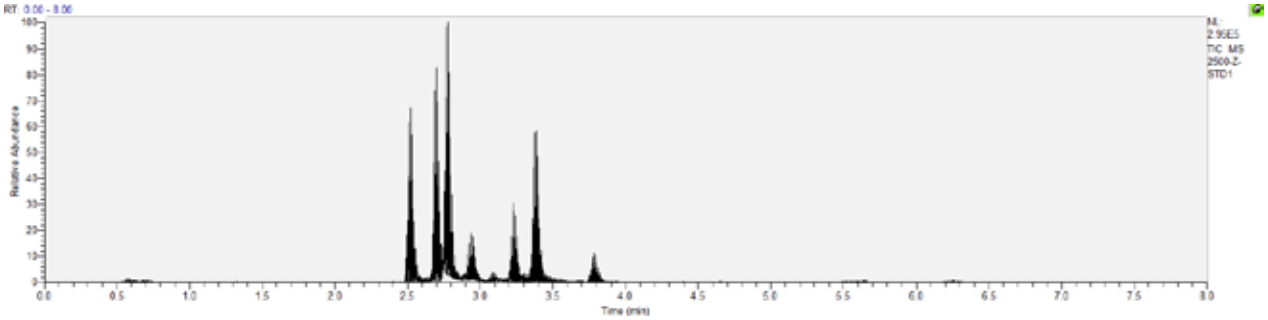


Figure 1. TIC of Tetracyclines in Beef at 10 μg/kg

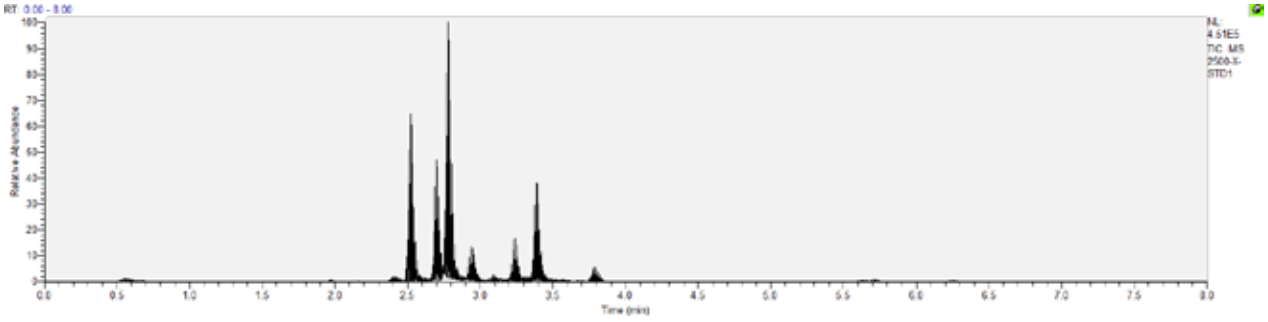


Figure 2. TIC of Tetracyclines in Shrimp at 10 μg/kg

Ordering Information

Cat. #	Description	Qty.
COQ050051H	Copure® Veterinary Drug Residue Specialty QuEChERS Extraction Kit	50 Pcs/Box
COQ015601H	Copure® QuEChERS Sulfonamide Purification Kit	50 Pcs/Box
SDC-3000-D	biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
BN24	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs /Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs /Box

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

Application Scope

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

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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

Procedure

Extraction

Weigh 5.0 g of homogenized Beef 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 dipersive 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 Beef are listed in Table 2.

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

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 Beef

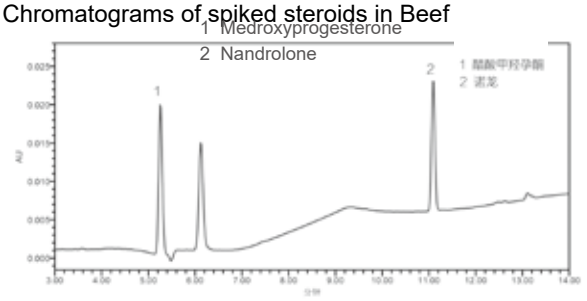


Figure 1. Chromatogram of steroids spiked at 0.5 mg/kg in Beef

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	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

Analysis of Quinolones in Beef 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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

Procedure

Extraction

Weigh 2.0 g of homogenized Beef 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 dipersive 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 Beef are listed in Table 2

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

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 Beef

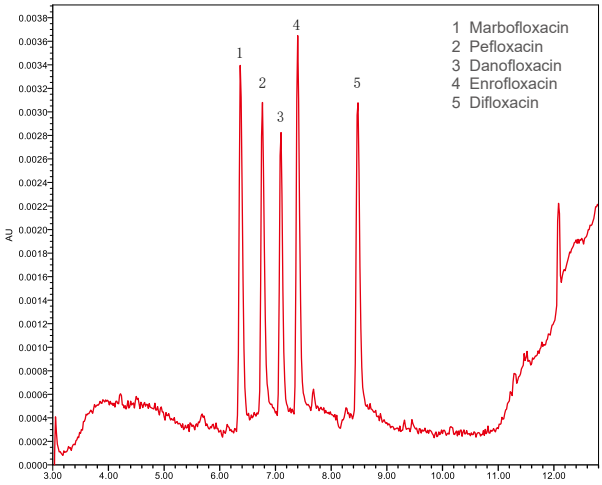


Figure 1. Chromatogram of quinolones spiked at 0.8 mg/kg in Beef

Order Information

Cat.#	Description	Qty.
COQ050051	Extraction Kit forVeterinary Drugs, 50 mL Tube	50/Box
COQ015601	Dispersive SPE Kit for Veterinary Drugs, 15 mL Tube	50/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

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

Application Scope

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

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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

Procedure

Extraction

Weigh 2.0 g of homogenized meet 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 dipersive 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 chloramphenicols analogue in Beef are listed in Table 2.

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

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 Beef

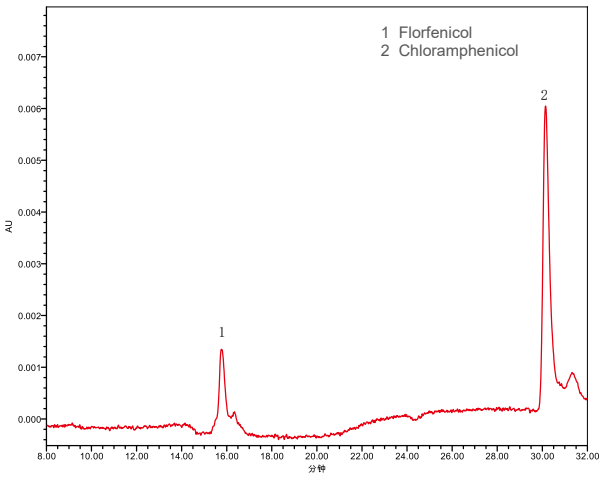


Figure 1. Chromatogram of chloramphenicols analogue spiked at 5.0 mg/kg in Beef

Order Information

Cat.#	Description	Qty.
COQ050050	Extraction Kit forVeterinary Drugs, 50 mL Tube	50/Box
COQ015601	Dispersive SPE Kit for Veterinary Drugs, 15 mL Tube	50/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

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

Application Scope

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

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® Multifunctional Vortex Mixer (Cat. No. BCM2500-E)

Procedure

Extraction

Weigh 2.0 g of homogenized meet 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 dipersive 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

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 Beef are listed in Table 2.

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

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 Beef

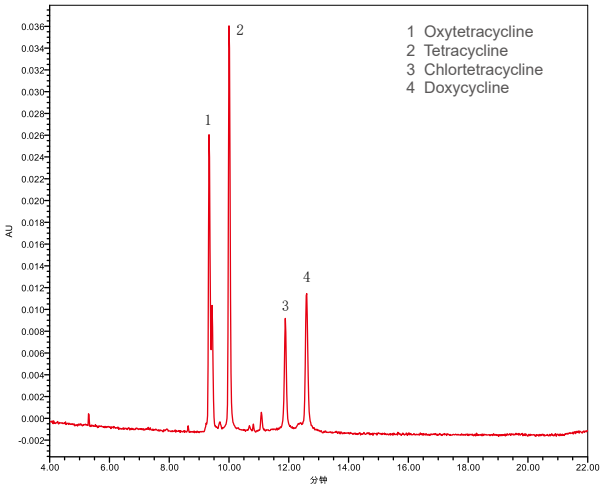


Figure 1. Chromatograms of tetracyclines spiked at 1.0 mg/kg in Beef

Order Information

Cat.#	Description	Qty.
COQ050051	Extraction Kit forVeterinary Drugs, 50 mL Tube	50/Box
COQ015601	Dispersive SPE Kit for Veterinary Drugs, 15 mL Tube	50/Box
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-5	Blue polypropylene screw caps with pre-slit white PTFE/red silicone septa, 6mm hole	100 Pcs/Box
BCM2500-E	Multifunctional Vortex Mixer	1 Set/Carton

7 Synthetic Colorants in Foods (Copure® Polyamide SPE Cartridges)

Introduction

Synthetic colorants often rely on chemicals like benzene, toluene, and naphthalene as key raw materials. They're favored for their low cost, vibrant colors, strong pigmentation, and wide range of shades. However, excessive consumption can pose health risks. Biocomma utilizes the polyamide SPE cartridges, developing a HPLC method with simplified operation and good recovery to detect tartrazine, sunset yellow, new red, carmine cochineal, amaranthus red, allura red, and brilliant blue in foods for your reference.

Experiment

Preparation

Prepare ethanol-ammonia solution: add 700 mL of ethanol and 4 mL of ammonia. Dilute to 1 L by water.

Extraction of juice and beverage sample

Weigh 2.0 g of the sample (accurate to 0.001 g) into a 50 mL centrifuge tube. Add 25 mL of ethanol-ammonia solution, and vortex for 5 min. Extract by ultrasonic treatment for 15 min at 50 °C, and centrifuge at 8000 r/min for 5 min. Transfer the supernatant into a clean 50 mL centrifuge tube. Add 15 mL of the ethanol-ammonia solution to the remaining sample. Repeat the extraction procedures. Combine the supernatants and dilute to 50 mL. Pipette 10 mL of supernatant to a clean tube and concentrate to 2 mL at 50°C. Add 10 mL of 5% aqueous methanol solution and mix well. Adjust the pH to 3~4 by 20% citric acid. The sample is ready for purification.

Extraction of ham, potato chips, bread and chicken wings sample

Weigh 2 g of the sample into a 50 mL centrifuge tube. Add 25 mL of ethanol-ammonia solution, and vortex for 5 min. Extract by ultrasonic treatment for 15 min at 50 °C, and centrifuge at 8000 r/min for 5 min. Transfer the supernatant into a clean 50 mL centrifuge tube. Add 15 mL of the ethanol-ammonia solution to the remaining sample. Repeat the extraction procedures. Combine the supernatants and dilute to 50 mL. Pipette 10 mL of supernatant to a clean tube and concentrate to 2 mL at 50°C. Add 10 mL of 5% aqueous methanol solution and mix well. Adjust the pH to 3~4 by 20% citric acid. The sample is ready for purification.

Purification (Copure® Polyamide (PA) SPE Cartridges, 500 mg/ 6 mL)

Activation: activated the cartridge by 6 mL of methanol, then 6 mL of water.

Loading: add prepared sample to the activated cartridge.

Washing: add 5 mL of water, then 5 mL of methanol. Drain the cartridge.

Elution: add 7 mL of 5% ammoniated methanol solution and collect the eluate. Evaporate to about 0.3 mL at 45 °C. Dilute to 2 mL with 0.02 mmol/L ammonium acetate (pH=9.0). Vortex to mix well. After filter by PTFE membrane, the sample is ready for LC-MS/MS analysis.

Instrumental Conditions

Instrument: liquid chromatograph, ThermoFisher U3000

Chromatographic column: Agilent ZORBAX SB-C18 (4.6 mm×250 mm, 5 μm)

Mobile phase: A: 0.02 mol/L ammonium acetate solution, B: methanol

Flow rate: 1.0 mL/min

Column temperature: 30 ℃

Injection volume: 10 μL

Detector: UV detector

Detector wavelength range: 400~800 nm. (Tartrazine: 415 nm. New red, carmine cochineal, amaranthus red, sunset yellow, allura red and erythrosine: 520 nm. Brilliant blue: 630 nm.)

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.01	90	10
12.0	65	35
19.0	55	45
22.5	50	50
23.0	45	55
24.0	35	65
34.0	35	65
35.0	90	10
42.0	90	10

Results

Table 2. Spiked Recovery Rate

Targets	Recovery rate (%)									
	Beverages		Ham		Bread		chicken wings		Chips	
	3.0mg/kg	5.0mg/kg	3.0mg/kg	5.0mg/kg	3.0mg/kg	5.0mg/kg	3.0mg/kg	5.0mg/kg	3.0mg/kg	5.0mg/kg
Tartrazine	103	100	87.5	96.4	96.7	98.7	89.3	94.6	87.6	93.1
New Red	101	102	92.9	97.9	94.3	102	98.4	92.1	92.4	95.3
Amaranthus Red	93.5	101	98.1	103	87.3	93.5	86.4	90.2	88.4	94.8
Carmine Cochineal	97.5	99.0	89.5	92.1	96.1	92.3	89.9	96.7	81.1	93.9
Sunset Yellow	101	98.4	89.1	93.1	86.4	91.2	85.5	94.3	87.6	92.8
Allura Red	102	101	90.5	95.6	95.9	92.1	87.7	93.2	95.2	93.2
Brilliant Blue	92.9	91.7	95.2	90.7	92.6	97.4	90.7	96.4	94.1	97.5

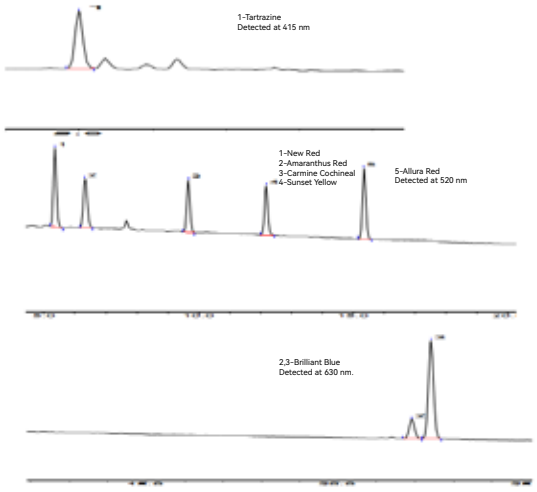


Figure 1. Chromatograms of 7 Synthetic Colorants in Beverage (5.0 mg/kg)

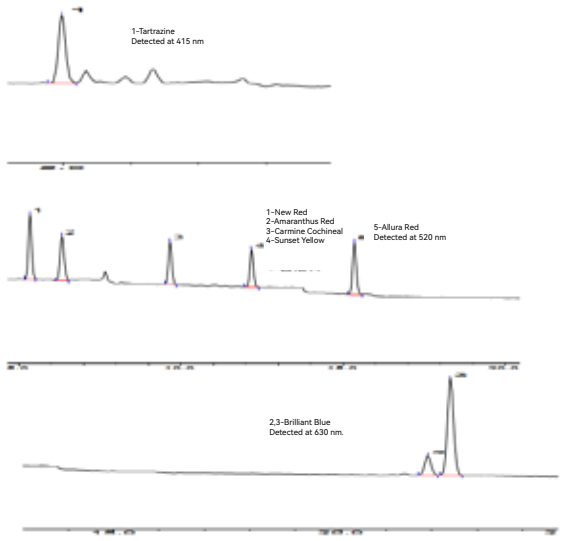


Figure 2. Chromatograms of the 7 Synthetic Colorants in Bread (5.0 mg/kg)

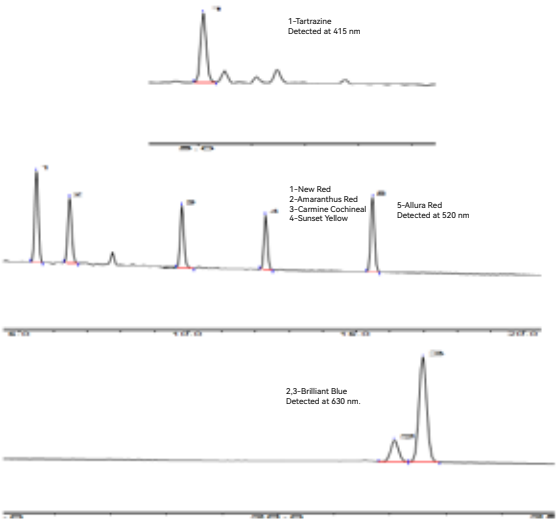


Figure 3. Chromatograms of 7 Synthetic Colorants in Chicken Wings (5.0 mg/kg)

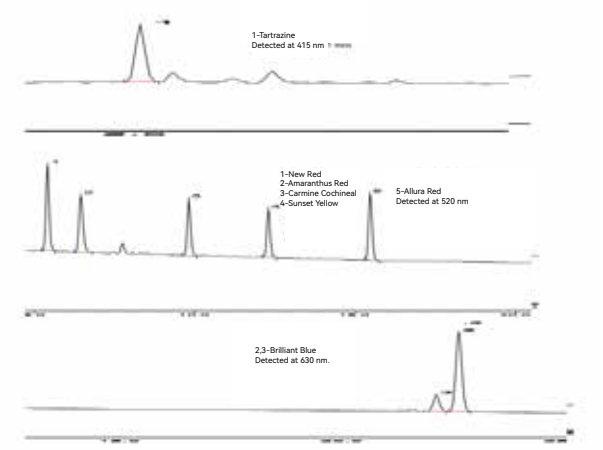


Figure 4. Chromatograms of the 7 Synthetic Colorants in Ham (5.0 mg/kg)

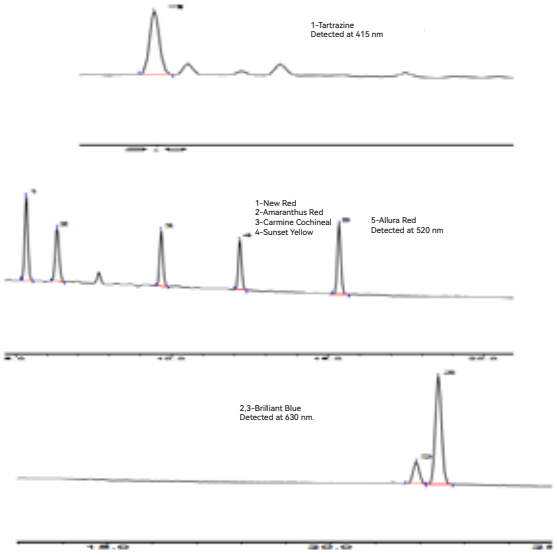


Figure 5. Chromatograms of 7 Synthetic Colorants in Chips (5.0 mg/kg)

Ordering Information

Cat. #	Description	Qty.
COPACR66	Copure® Polyamide (PA) SPE Cartridges, 500 mg/ 6 mL	30 Pcs/Box
SDC-3000-D	biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-PTFE-HL	PTFE-HL / Φ13 mm/0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

11 Synthetic Colorants in Foods (Copure® PWAX Cartridges)

Introduction

Synthetic colorants often rely on chemicals like benzene, toluene, and naphthalene as key raw materials. They're favored for their low cost, vibrant colors, strong pigmentation, and wide range of shades. However, excessive consumption can pose health risks. Biocomma established a HPLC method with simplified operation and good recovery to detect synthetic colorants in foods for your reference.

Experiment

Preparation

Prepare ethanol-ammonia solution: add 700 mL of ethanol and 4 mL of ammonia. Dilute to 1 L by water.

Extraction of liquid and partial solid samples (e.g., beverages, juice, jellies, etc.)

Weigh 2 g of the sample into a 50 mL centrifuge tube. Add 25 mL of ethanol-ammonia solution, and vortex for 5 min. Extract by ultrasonic treatment for 15 min at 50 °C, and centrifuge at 8000 rpm for 5 min. Transfer the supernatant into a clean 50 mL centrifuge tube. Add 15 mL of the ethanol-ammonia solution to the remaining sample. Repeat the extraction procedures. Combine the supernatants and dilute to 50 mL. Pipette 10 mL of supernatant to a clean tube and concentrate to 2 mL at 50°C. Add 10 mL of 5% aqueous methanol solution and mix well. The sample is ready for purification.

Extraction of oily samples (e.g., dessert, chicken wings, potato chips, etc.)

Weigh 2 g of the sample into a 50 mL centrifuge tube. Add 20 mL of petroleum ether, and shake to mix well. Vortex for 10 min. Centrifuge at 8000 rpm for 5 min. Discard the supernatant to remove the petroleum ether solvent. Add 25 mL of ethanol-ammonia solution and vortex for 1 min. ultrasonicate at 50 ℃ for 15 min, and then centrifuge at 8000 rpm for 5 min. Extract by ultrasonic treatment for 15 min at 50 °C, and centrifuge at 8000 rpm for 5 min. Transfer the supernatant into a clean 50 mL centrifuge tube. Add 15 mL of the ethanol-ammonia solution to the remaining sample. Repeat the extraction procedures. Combine the supernatants and dilute to 50 mL. Pipette 10 mL of supernatant to a clean tube and concentrate to 2 mL at 50°C. Add 10 mL of 5% aqueous methanol solution and mix well. The sample is ready for purification.

Purification (Copure® PWAX Cartridges, 150 mg/ 6 mL)

Activation: activated the cartridge by 6 mL of methanol, then 6 mL of water.

Loading: add prepared sample to the activated cartridge.

Washing: add 6 mL of water, then 6 mL of methanol. Drain the cartridge.

Elution: add 6 mL of 5% ammoniated methanol solution and collect the eluate. Evaporate to about 0.3 mL at 45 °C. Dilute to 2 mL with 0.02 mmol/L ammonium acetate (pH=9.0). Vortex to mix well. After filter by PTFE membrane, the sample is ready for LC-MS/MS analysis.

Instrumental Conditions

Instrument: liquid chromatograph, Thermo Fisher U3000

Chromatographic column: Agilent ZORBAX SB-C18 (4.6 mm×250 mm, 5 μm)

Mobile phase: A: 0.02mol/L ammonium acetate solution, B: methanol

Flow rate: 1.0 mL/min

Column temperature: 30 ℃

Injection volume: 10 μL

Detector: UV detector

Detector wavelength range: 400~800 nm. (Tartrazine: 415 nm. New red, carmine cochineal, amaranthus red, sunset yellow, allura red and erythrosine: 520 nm. Brilliant blue: 630 nm.)

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.01	90	10
12.0	65	35
19.0	55	45
22.5	50	50
23.0	45	55
24.0	35	65
34.0	35	65
35.0	90	10
42.0	90	10

Results

Table 2. Spiked Recovery Rates

Targets	Recovery rate (%)									
	Jelly		Coke		Cake		Chicken wings		Potato Chips	
	3.0 mg/kg	5.0 mg/kg	3.0 mg/kg	5.0 mg/kg	3.0 mg/kg	5.0 mg/kg	3.0 mg/kg	5.0 mg/kg	3.0 mg/kg	5.0 mg/kg
Tartrazine	99.6	96.6	94.9	96.4	91.1	98.9	85.1	92.4	95.5	92.1
New Red	99.6	101	92.9	97.9	93.7	97.2	88.8	96.1	82.7	90.6
Amaranthus Red	93.5	102	98.1	103	98.4	100	86.1	97.9	88.4	86.5
Indigotine	92.3	94.1	84.6	86.1	91.1	88.5	87.2	91.7	84.7	89.8
Quinoline Yellow1	101	97.1	97.7	102	96.1	94.8	87.3	94.6	89.9	84.9
Carmine Cochineal	98.5	104	97.5	101	98.4	101	93	100	84.1	87.1
Sunset Yellow	92.3	96.1	99.1	98.1	95.3	103	94.0	98.0	87.0	99.9
Quinoline Yellow2	86.9	92.4	96.6	99.4	97.6	94.2	86.0	97.2	82.9	97.7
Allura Red	92.3	96.7	92.5	99.6	92.8	96.9	89.7	96.9	95.1	100
Carmoisine	94.8	96.5	94	97.4	89.5	92.7	95.4	97.7	90.9	96.9
Brilliant Blue	86.9	92.3	84.6	86.1	89.6	93.5	92.3	96.1	84.8	91.2
Quinoline Yellow 3	87.2	96.8	95.5	103	75.2	77.8	82.5	94.9	84.5	88.5
Quinoline Yellow 4	85.9	94.3	98.4	100	80.6	83.3	87.4	95.6	87.0	99.9
Erythrosine	78.3	82.6	80.9	83.2	80.1	85.2	78.3	86.5	82.9	84.4

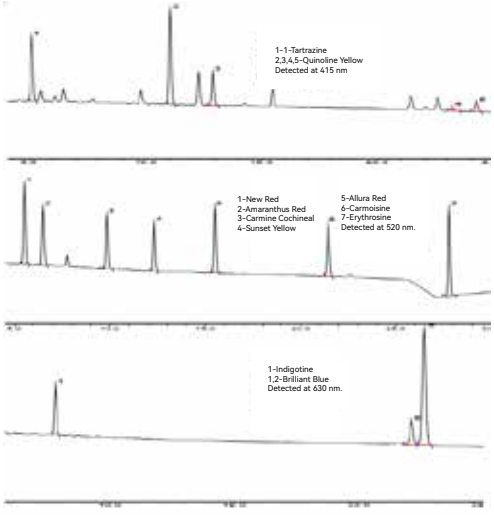


Figure 1. Chromatograms of 7 Synthetic Colorants Standard Solutions (5.0 mg/kg)

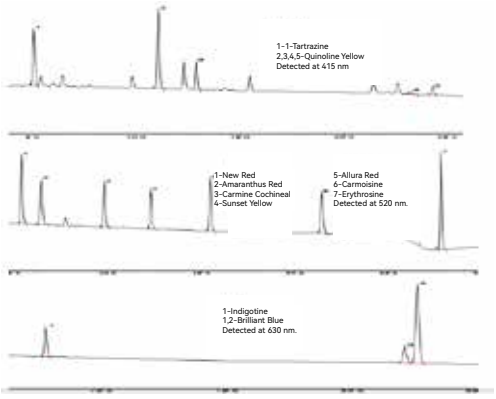


Figure 2. Chromatograms of 11 Synthetic Colorants in Jelly (5.0 mg/kg)

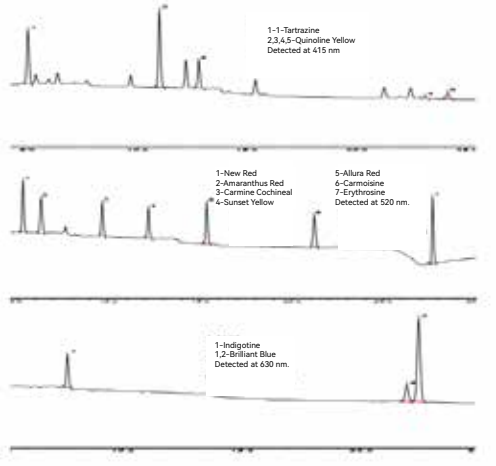


Figure 3. Chromatograms of 11 Synthetic Colorants in Coke (5.0 mg/kg)

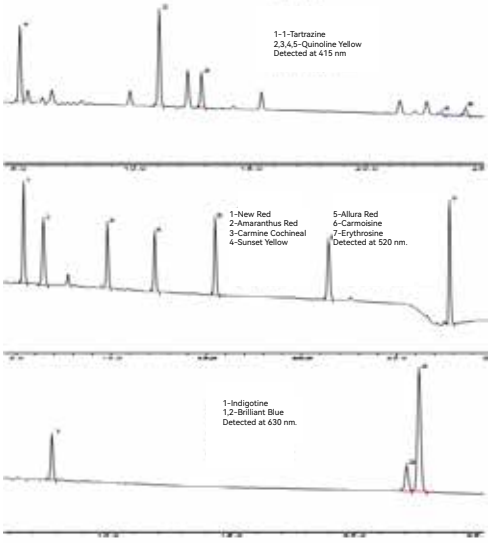


Figure 4. Chromatograms of 11 Synthetic Colorants in Cake (5.0 mg/kg)

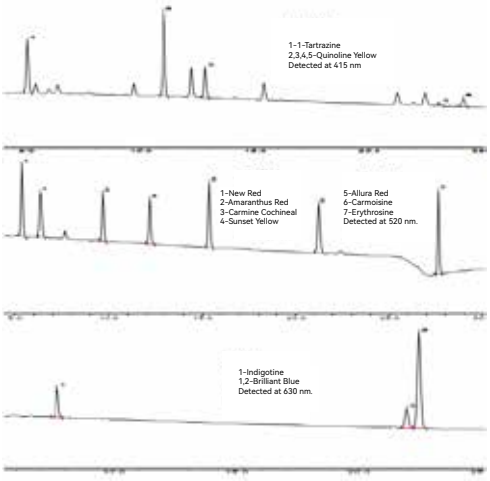


Figure 5. Chromatograms of 11 Synthetic Colorants in Chicken Wing (5.0 mg/kg)

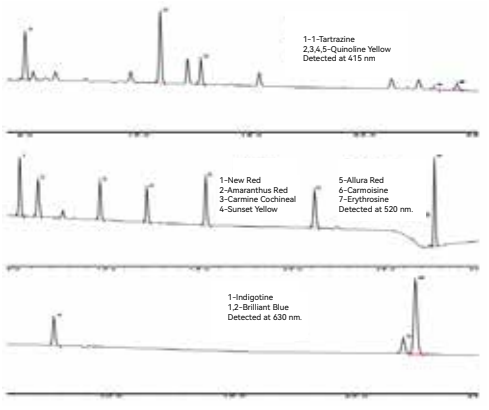


Figure 5. Chromatograms of 11 Synthetic Colorants in Potato Chips (5.0 mg/kg)

Ordering Information

Cat. #	Description	Qty.
COPWAX6150	Copure® PWAX Cartridges, 150 mg/ 6 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF250-22-PTFE-HL	Syringe Filters PTFE-HL / Φ25 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

8 Synthetic Food Colorants in Milk Tea (Copure® PWAX Cartridges)

Introduction

Synthetic food colorants are widely used in the food industry for enhancing color and sensory appeal. However, their excessive use can be harmful. Milk tea, rich in proteins, fats, and sugars, poses challenges for colorant detection. Biocomma addresses these challenges by adding a zinc sulfate solution to the extraction solvent to reduce interference, and develops a HPLC method with simplified operation and good recovery to detect 8 synthetic food colorants of tartrazine, sunset yellow, new red, carmine cochineal, amaranthus red, allura red, brilliant blue and erythrosine in milk tea for routine testing.

Experiment

Preparation

Prepare ethanol-ammonia solution: add 700 mL of ethanol and 4 mL of ammonia. Dilute to 1 L by water.

Extraction

Weigh 2.0 g of the sample in a 50 mL centrifuge tube. Add 5 mL of zinc sulfate solution (120 g/L) and mix well. Add 25 mL of ethanol-ammonia solution and vortex for 1 min. Extract by ultrasonic treatment for 15 min, and centrifuge at 8000 r/min for 5 min. Transfer the supernatant into a clean 50 mL centrifuge tube. Add 15 mL of the ethanol-ammonia solution to the remaining sample. Repeat the extraction procedures. Combine the supernatants and dilute to 50 mL. Centrifuge again if the solution is cloudy. Pipette 10 mL of supernatant to a clean tube and concentrate to 2 mL at 50°C. Add 10 mL of 5% aqueous methanol solution and mix well. The sample is ready for purification.

Purification (Copure® PWAX Cartridge, 150 mg/ 6 mL)

Activation: activated the cartridge by 6 mL of methanol, then 6 mL of water.
Loading: add prepared sample to the activated cartridge.
Washing: add 5 mL of water, then 5 mL of methanol. Drain the cartridge.
Elution: add 8 mL of 2% ammoniated methanol solution and collect the eluate. Evaporate to about 0.3 mL at 45 °C. Dilute to 2 mL with 0.02 mmol/L ammonium acetate solution (pH=9.0). Vortex to mix well. After filter by PTFE membrane, the sample is ready for LC-MS/MS analysis.

Instrumental Conditions

Instrument: Liquid Chromatograph, ThermoFisher U3000
Chromatographic column: Agilent ZORBAX SB-C18 (4.6 mm×250 mm, 5 μm)
Mobile phase: A: 0.02mol/L ammonium acetate solution, B: methanol
Flow rate: 1.0 mL/min
Column temperature: 30 ℃
Injection volume: 10 μL
Detector: UV detector
Detector wavelength range: 400~800 nm. (Tartrazine: 415 nm. New red, carmine cochineal, amaranthus red, sunset yellow, allura red and erythrosine: 520 nm. Brilliant blue: 630 nm.)

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.01	90	10
12.0	65	35
19.0	55	45
22.5	50	50
23.0	45	55
24.0	35	65
34.0	35	65
35.0	90	10
42.0	90	10

Results

Table 2. Spiked Recovery at 5 mg/kg

Targets	Recovery rate (%)		Average recovery rate %
	1	2	
Tartrazine	96.5	94.7	95.6
New red	97.4	101.3	99.4
Amaranthus red	94.1	95.3	94.7
Carmine cochineal	101.6	98.7	100.2
Sunset yellow	98.5	102.6	100.6
Allura red	93.8	92.4	93.1
Brilliant blue	94.3	96.5	95.4
Erythrosine	89.3	85.3	87.3

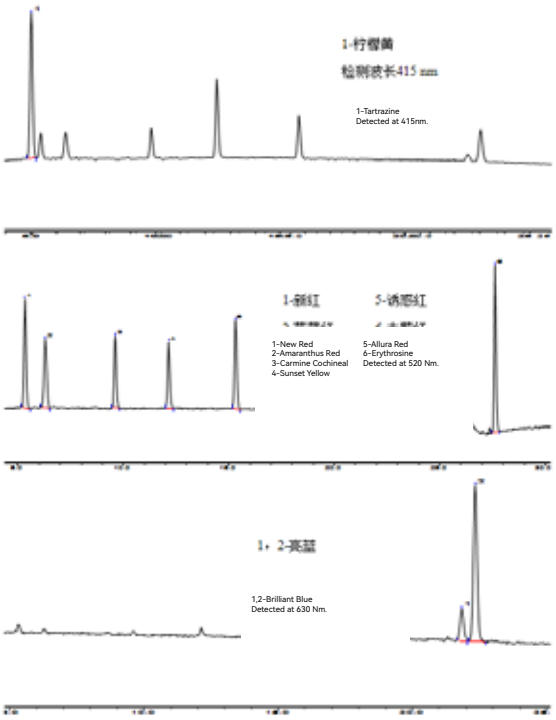


Figure1. Chromatograms of 8 Colorants in Milk Tea (5.0 mg/kg)

Ordering Information

Cat. #	Description	Qty.
COPWAX6150	Copure® PWAX Cartridge, 150 mg/ 6 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF250-22-PTFE-HL	Syringe Filters PTFE-HL / Φ25 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

Propionic Acid in Foods (Copure® HLB Cartridges)

Introduction

Propionic acid and its sodium and calcium salts have bacteriostatic effects, effectively preventing mold growth. It's frequently used as a preservative and antifungal agent in food products. Biocomma has established a simplified method that significantly reduces interference to ensure good recovery for the detection of propionic acid in various samples.

Experiment

Preparation

Weigh 5.0 g sample into a 50 mL centrifuge tube. Add 20 mL of water and 0.5 mL of 1.0 mol/L phosphoric acid solution. Vortex for 2 min and extract by ultrasonic treatment for 10 min. Adjust the pH to 2.8~3.1 with 1.0 mol/L phosphoric acid. Dilute to 50 mL with water. Shake to mix well and centrifuge at 8000 r/min for 5 min. Pipette 5 mL of supernatant to a clean tube. The sample is ready for purification.

Purification (Copure® HLB Cartridges, 500 mg/ 6 mL)

Activation: activated the cartridge by 5 mL of methanol, then 5 mL of water.

Loading: add prepared sample to the activated cartridge.

Washing: add 5 mL of 10% methanol-water solution, then drain the cartridge.

Elution: add 5 mL of 50 % methanol-water solution and collect the eluate. After filter by 0.22 μm nylon membrane, the sample is ready for LC-MS/MS analysis.

Instrumental Conditions

Instrument: liquid chromatograph, ThermoFisher U3000

Chromatographic column: Agilent ZORBAX SB-C18 (4.6 mm×250 mm, 5 μm)

Mobile phase A: 1.5 g of diammonium hydrogen phosphate solution (adjust pH to about 3.0 by 1.0 mol/L phosphoric acid solution)

Mobile phase B: 50 % methanol-water solution

Flow rate: 1.0 mL/min

Column temperature: 25 ℃

Injection volume: 20 μL

Detector: UV detector

Detection wavelength: 214 nm

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.0	95	5
14.0	95	5
18.0	5	95
21.0	5	95
22.0	95	5
30.0	95	5

Results

Table 2. Spiked Recovery Rates

Samples	Spiked Concentration (g/kg)	Recovery Rate (%)			Average Recovery Rate (%)	RSD (%, n=3)
Bread	0.10	96.00	95.83	97.92	96.58	1.20
	1.00	92.32	91.49	90.55	91.45	0.97

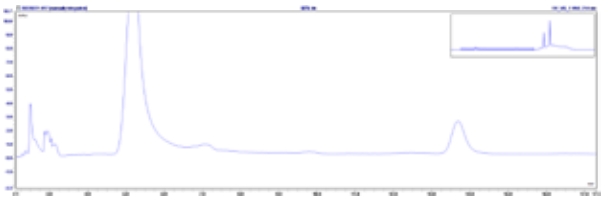


Figure 1. Liquid Chromatogram of Propionic Acid in Bread (Blank)

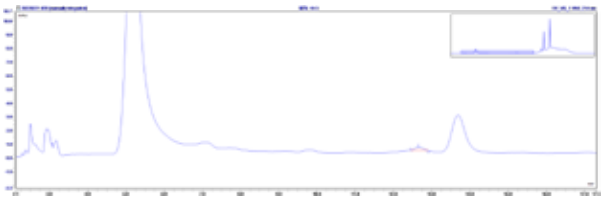


Figure 2. Liquid Chromatogram of Propionic Acid in Bread (0.10 g/kg)

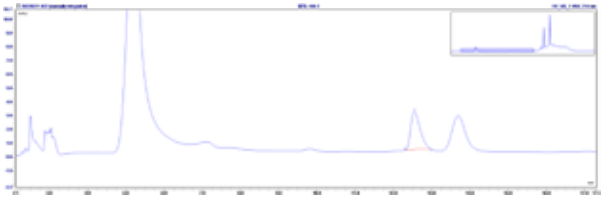


Figure 3. Liquid Chromatogram of Propionic Acid in Bread (1.00 g/kg)

Ordering Information

Cat. #	Description	Qty.
COHLB6500	Copure® HLB Cartridge, 500 mg/6mL	50 Pcs/Box
SDC-3000-D	biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
BN24-E	Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box

Aflatoxin in Peanut (Copure® Aflatoxin B1 Immunoaffinity Columns)

Introduction

Peanuts are susceptible to mold contamination, particularly the presence of aflatoxins like Aflatoxin B1 (AFB1), which significantly diminish nutritional and commercial value. Furthermore, AFB1 contamination poses a substantial threat to the edibility and safety of peanuts and peanut products. The immunoaffinity method offers distinct advantages, including precise results, high recovery rates, and excellent precision. Biocomma established an immunoaffinity UPLC-MS/MS method for detection of AFB1 in peanut by Copure® Aflatoxin B1 Immunoaffinity Columns with good recovery and precision for your reference. The recoveries were 90-110% with RSD less than 5%.

Experiment

Extraction

Weigh 5.00 g of ground peanuts into a 50 mL centrifuge tube. Add 100 µL of internal standard, then 20 mL of acetonitrile-water solution (84+16) and vortex to mix well. Extract by ultrasonic treatment for 20 min. Centrifuge at 8000 r/min for 5 min. Pipette 4.0 mL a clean tube, and add 20 mL of PBS solution. After mixed well, the sample is ready for purification.

Purification (Copure® Aflatoxin B1 Immunoaffinity Columns, 3 mL)

Set the immunoaffinity column on the biocomma® SPE Vacuum Manifold. Add the prepared solution at flow rate of 1~2 drop/s. Then add 5.0 mL PBS buffer and 5.0 mL water. Drain the column. Add 1.0 mL of methanol. Close the valve and soak for 30 s, then elute into a sample vial for LC-MS/MS analysis.

Preparation of Procedural Blank

Carry out the experiment according to the above steps without sample.

Instrumental Conditions

Chromatographic column: Commasil® T-C18 (2.1 mm×100 mm, 3 µm)
Mobile phase A: 5 mmol/L ammonium acetate
Mobile phase B: Methanol (containing 0.1% formic acid)
Flow rate: 0.3 mL/min
Column temperature: 30 ℃
Injection volume: 5 µL

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.0	90	10
1.2	40	60
2.1	10	90
4.8	10	90
5.0	90	10
6.0	90	10

Ion Source: HESI
Scan method: positive ion mode (ESI+)
Sheath gas pressure: 30 arb
Auxiliary gas pressure: 8 arb
Ion exchange tube: 300 ℃
Auxiliary air temperature: 350 ℃

Table 2. Targes, and Characteristic Ions (*Quantitative Ions)

Targets	Parent Ion	Daughter Ion	Ion Mode
Aflatoxin B1	313.1	241.1、285*	ESI+
13C17-AFB1	330.1	255、301*	ESI+

Results

Table 1. Spiked Aflatoxin B1 In Peanut Recovery

Target	Spike (µg/kg)	Recovery (%)	Average Recovery (%)	RSD (%)
Aflatoxin B1	1.00	108	105	2.52
		104		
		103		

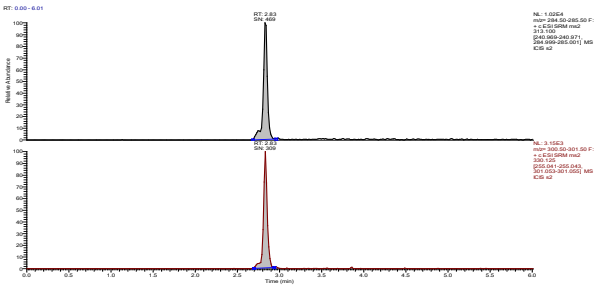


Figure 1. Quantitative Ion Extraction Chromatogram of Aflatoxin B1 Calibration Points with External Standard and Isotope Internal Standard (1.0 µg/L)

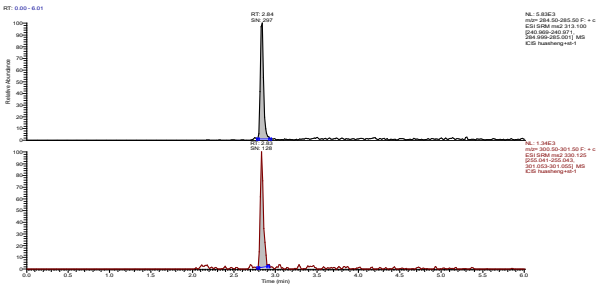


Figure 2. Quantitative Ion Extraction Chromatogram of Aflatoxin B1 with External Standard and Isotope Internal Standard in Peanuts (1.00 µg/kg)

Ordering Information

Cat. #	Description	Qty.
COAFMB103	Copure® Aflatoxin B1 Immunoaffinity Columns, 3 mL	20 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box

Ochratoxin A in Coffee Beans (Copure® MAX Cartridges)

Introduction

Coffee, a daily beverage, is highly reliant on quality and safety. Mycotoxin contamination, notably Ochratoxin A (OTA), is a key risk factor affecting coffee quality. Biocomma established a UPLC-MS/MS method for detection of OTA by Copure® MAX Cartridges with good recovery and precision for your reference. The recoveries were 95-105% with RSD less than 5%.

Experiment

Extraction

Weigh 2.50 g of ground coffee bean into a 50 mL centrifuge tube. Add 25 mL of 30 g/L methanol-sodium bicarbonate solution (50:50, v/v). Vortex for 10 min. Centrifuge at 8000 r/min for 5.0 min, then filter the supernatant with filter paper. Collect 10 mL of the extracted filtrate for purification.

Purification (Copure® MAX Cartridge, 200 mg/ 3 mL)

Activation: activate the Copure® MAX Cartridge by 5 mL of methanol, then 5 mL of 30 g/L methanol-sodium bicarbonate (50:50, v/v).

Loading: add the prepared solution at flow rate of 1~2 drop/s. Drain the cartridge.

Washing: wash the cartridge by 5 mL of washing solution (0.1 mol/L potassium hydroxide: acetonitrile: water = 3:50:47, v/v), 5.0 mL of water, and 5.0 mL of methanol sequentially. Drain the cartridge.

Elution: add 5 mL of eluent solution (methanol: acetonitrile: formic acid: water = 40:50:5:5, v/v). Collect the eluate and evaporate to nearly dryness at 45 ℃ . Add 1.0 mL of acetonitrile-2% acetic acid aqueous solution (50:50, v/v), and vortex to reconstitute. After filter into a sample vial, the sample is ready for analysis.

Preparation of Procedural Blank

Carry out the experiment according to the above steps without sample.

Instrumental Conditions

Instrument: Thermo Scientific UltiMate 3000

Chromatographic column: Commasil® AQ-C18 (4.6 mm*250 mm, 5 um)

Detector: FLD (excitation wavelength 333 nm, emission wavelength 460 nm)

Mobile phase: A: 2% acetic acid water, B: acetonitrile

Flow rate: 1 mL/min

Injection volume: 5 μL

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0	88	12
2.0	88	12
10.0	80	20
12.0	70	30
19.0	50	50
30.0	50	50
31.0	0	100
39.0	0	100
40.0	88	12
45.0	88	12

Results

Table 2. Spiked Ochratoxin A In Coffee Bean Recovery

Target	Spike (μg/kg)	Recovery (%)	Average Recovery (%)	RSD (%)
Ochratoxin A	20.0	97.0	99.0	2.74
		101		
		95.9		

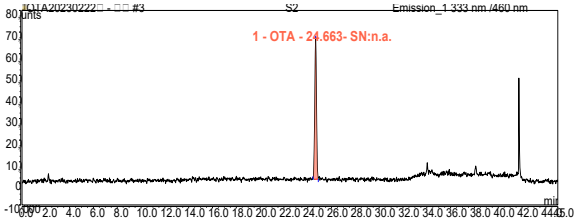


Figure 1. Calibration Point Liquid Chromatogram (5 μg/L)

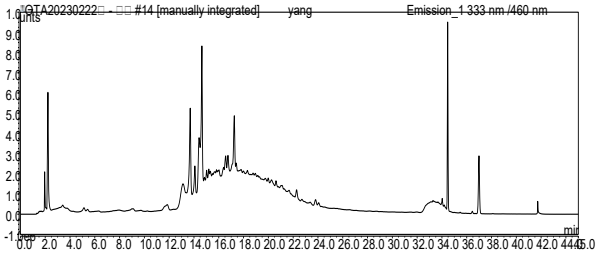
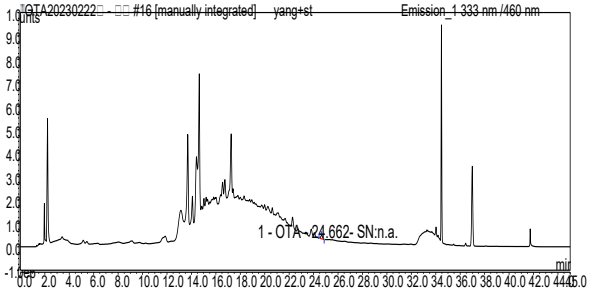


Figure 2. Background Liquid Chromatogram of Coffee Beans



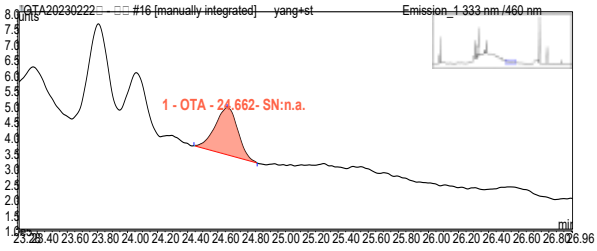


Figure 3. Liquid Chromatogram of Spiked Ochratoxin A in Coffee Beans (20 μg/kg)

Ordering Information

Cat. #	Description	Qty.
COMAX3200	Copure® MAX Cartridge, 200 mg/ 3 mL	50 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
BN24	Biocomma® 24Intelligent Water-Bath Nitrogen Evaporator	1 Set/Ctn.
SF130-22-PTFE-HL	Syringe Filters PTFE-HL Syringe Filters / Φ13mm / 0.22μm / Hydrophilic	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/ red silicone septa, 6 mm hole	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box

Aflatoxin M Group in Milk (Copure® 223 Multifunctional Purification Column)

Introduction

Aflatoxins are toxic and highly carcinogenic metabolites of Aspergillus flavus and Aspergillus parasiticus fungi. When cows consume contaminated feed, aflatoxin B1 will be transformed into aflatoxin M1. A portion of aflatoxin M1 is excreted through urine and milk, while other portions are stored in meat. Biocomma established a rapid LC method using Copure® 223 Multifunctional Purification Plate for detection of aflatoxins M1 and M2 in milk with good recovery and stability for your reference. The recoveries were 90-110% with RSD less than 5% at both 5 ng/g and 10 ng/g concentrations.

Experiment

Extraction

Weigh 4 g of sample into a 50 mL centrifuge tube. Add 10 mL of Acetonitrile and vortex to mix well. Then, extract by ultrasonic treatment for 20 min. Centrifuge at 6000 r/min for 10 min. The supernatant is ready for purification.

Purification (Copure® 223 Multifunctional Purification Column)

Add 10 mL of supernatant to a glass tube. Insert the rubber head of the purification column into the tube, and push to the bottom. Pipette 5 mL of the purified sample to a sample vial or EP tube. Evaporate sample to dryness and redissolve by 1 mL of initial mobile phase. Vortex for 30 seconds to dissolve the residue. After filter by microporous membrane, the sample is ready for analysis.

Instrument Conditions

Equipment: UltiMate 3000 (Thermo Fisher Scientific) with FLD detector.
Chromatographic column: Agilent ZORBAX C18 (4.6 mm×250 mm, 5 μm)
Mobile phase: A: Deionized water, B: Acetonitrile-methanol (50/50)
Mobile phase elution conditions: A: 70%, B: 30%
Flow rate: 1.0 mL/min
Column temperature: room temperature
Injection volume: 20.0 μL
Detection wavelength: excitation wavelength 360 nm, emission wavelength 430 nm
Detector: FLD

Results

Table 1 Aflatoxin M Group Spike Recovery in Milk

Targets	Spike (ng/g)	Copure® 223 Multifunctional Purification Column		Brand A	
		Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
AFT M1	5	93.5	4.11	93.5	4.53
	10	94.1	4.25	89.5	4.55
AFT M2	5	95.1	3.50	95.5	5.31
	10	94.2	4.25	96.4	5.51

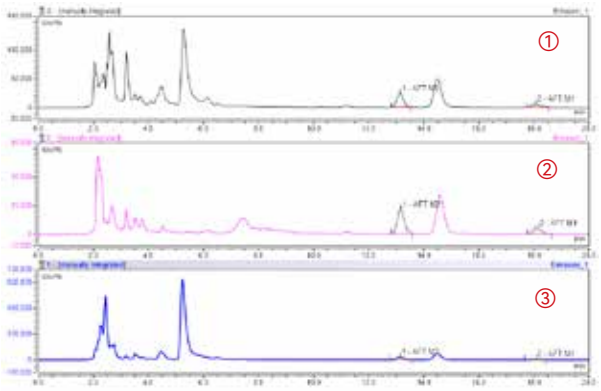


Figure 1. Chromatograms of Spiked Samples Purified by Different Brands at 5 ng/g
① Purified by Brand A
② Purified by Copure® 223 Multifunctional Purification Column
③ Before Purification

Based on Figure 1, the miscellaneous peaks of sample ② are fewer than sample ① . Less interference of impurities indicates better purification effects. The recovery rates of Aflatoxin M1 and M2 are both between 90-110%, and the RSD value is less than 5%, which meet the standard of experimental requirements.

Ordering Information

Cat. #	Description	Application	Qty.
COAF226	Copure® 226 Multifunctional Purification Column	Zearalenone, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF228	Copure® 228 Multifunctional Purification Column	Patulin, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF224	Copure® 224 Multifunctional Purification Column	Zearalenone	25 Pcs/Box
COAF223	Copure® 223 Multifunctional Purification Column	Aflatoxin M1 M2	25 Pcs/Box
COAF229	Copure® 229 Multifunctional Purification Column	Ochratoxin A	25 Pcs/Box
COAF230	Copure® 230 Multifunctional Purification Column	Deoxynivalenol	25 Pcs/Box
COAF302	Copure® 302 Multifunctional Purification Column	Multiple functions	25 Pcs/Box

Zearalenone in Grain Products (Copure® 224 Multifunctional Purification Column)

Introduction

Zearalenone, a non-steroidal mycotoxin from Fusarium fungus, is commonly found in contaminated grains (wheat, corn, oats, barley, etc.). It's a major global crop contaminant, leading to adverse effects such as tumors, estrogen presence, and immune suppression in livestock and poultry that consume contaminated feed, and then poses a risk to human health.

Biocomma established an LC method to detect zearalenone in corn flour using Copure® 224 Multifunctional Purification Column. At 8 ng/g and 16 ng/g concentratios, recoveries were 90-100% with RSD below 10%. This rapid method outperforms competitors, ensuring high recovery, efficient decolorization, and impurity removal. It's a valuable reference for zearalenone detection in corn flour, applicable to grains, wine, soy sauce, vinegar, soybean, rapeseed, and edible vegetable oil.

Experiment

Preparation

Weigh 5 g of sample into a clean tube. Add 1 g of sodium chloride and 20 mL of acetonitrile-water solution (9+1). Shake to mix well. Vortex for 15 min, and centrifuge at 6000 r/min for 5 min. The supernatant is ready for purification.

Purification (Copure® 224 Multifunctional Purification Column)

Add 10 mL of prepared sample to a glass tube. Insert the rubber head of the purification column into the tube, and push to the bottom. Pipette 5 mL of the purified sample to a sample vial or EP tube. Evaporate sample to dryness and redissolve by 1 mL of initial mobile phase. Vortex for 30 seconds to dissolve the residue. After filter by 0.22 μm microporous membrane, the sample is ready for analysis.

Instrument Conditions

Equipment: UltiMate 3000(Thermo Fisher Scientific) with FLD detector.

Chromatographic column: Agilent ZORBAX C18 (4.6 mm×250 mm, 5 μm)

Mobile phase: A: Acetonitrile, B: Deionized water, C: Methanol

Flow rate: 1.0 mL/min

Column temperature: room temperature

Injection volume: 50.0 μL

Detection wavelength: excitation wavelength 274 nm, emission wavelength 440 nm

Detector: FLD

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)	C (%)
0.00	52	40	8
5.00	52	40	8
8.00	95	5	0
11.00	95	5	0
13.00	52	40	8
17.00	52	40	8

Results

Table 2. Zearalenone Spike Recovery in Corn Flour

Targets	Spike (ng/g)	Copure®224 Multifunctional Purification Column		Brand A		Brand B	
		Recovery (%)	RSD/%	Recovery (%)	RSD/%	Recovery (%)	RSD/%
Zearalenone	8	95.5	6.2	98.4	6.0	145	5.1
	16	90.1	6.8	92.1	6.5	125	5.7

Figure 1. Decolorization Effect by Different Brands of Multifunctional Purification Columns

① Corn flour Sample Before Purification

② Purified by Copure® 224 Multifunctional Purification Column

③ Purified by Brand A

④ Purified by Brand B

Based on Figure 1, the pigments are obviously absorbed by Copure® 224 Multifunctional Purification Column. The decolorization ability is similar to Brand A and B.

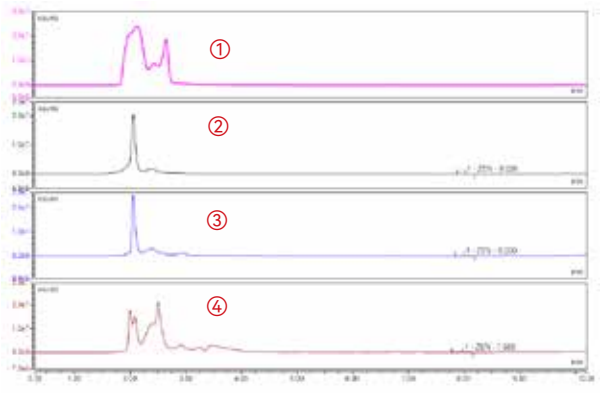


Figure 2. Chromatograms of Spiked Samples Purified by Different Brands
① Corn flour Sample Before Purification
② Purified by Copure® 224 Multifunctional Purification Column
③ Purified by Brand A
④ Purified by Brand B

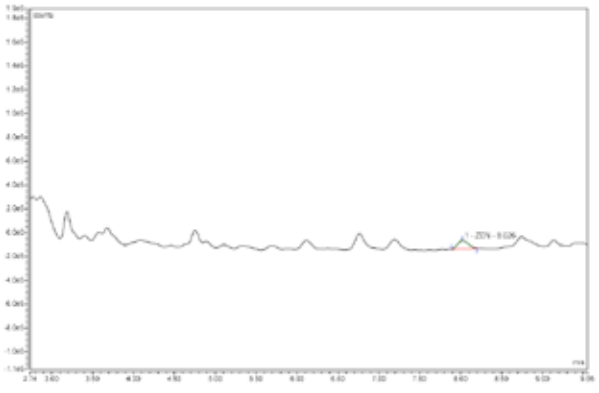


Figure 3. Chromatogram of Spiked Sample (8 ng/g) purified by Copure® 224 Multifunctional Purification Column

Based on Figure 2, the interference of impurities in sample ② are obviously absorbed, therefore miscellaneous peaks are fewer in the TIC chromatogram. The purification effect of Copure® 224 Multifunctional Purification Column is similar to Brand A and better than Brand B.

Ordering Information

Cat. #	Description	Application	Qty.
COAF226	Copure® 226 Multifunctional Purification Column	Zearalenone, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF228	Copure® 228 Multifunctional Purification Column	Patulin, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF224	Copure® 224 Multifunctional Purification Column	Zearalenone	25 Pcs/Box
COAF223	Copure® 223 Multifunctional Purification Column	Aflatoxin M1 M2	25 Pcs/Box
COAF229	Copure® 229 Multifunctional Purification Column	Ochratoxin A	25 Pcs/Box
COAF230	Copure® 230 Multifunctional Purification Column	Deoxynivalenol	25 Pcs/Box
COAF302	Copure® 302 Multifunctional Purification Column	Multiple functions	25 Pcs/Box

Flatoxin B1, Zearalenone and T-2 Toxin in Feed (Copure® 226 Multifunctional Purification Columns)

Introduction

Mycotoxins, produced by molds or fungi during food or feed growth, are harmful secondary metabolites. Over 300 mycotoxins exist naturally, with aflatoxin (AFT), zearalenone (ZEN), and T-2 toxin posing risks to livestock quality. Human ingestion leads to liver damage, cancer, teratogenicity, and immune diseases. Biocomma established a rapid LC-MS method using Copure® 226 Multifunctional Purification Columns for detection of aflatoxin B1, ZEN, and T-2 toxins in feed with good recovery and stability at three different concentrations for your reference.

Experiment

Extraction

Weigh 5.0 g of the sample into a 50 mL centrifuge tube, and add 25 mL of acetonitrile-water solution (84:16). Vortex for 3 min. Then, extract by ultrasonic treatment for 20 min. Shake 2-3 times during this period. Centrifuge at 10000 r/min for 5 min. The sample is ready for purification.

Purification (Copure® 226 Multifunctional Purification Column)

Add 10 mL of prepared sample to a glass tube. Insert the rubber head of the purification column into the tube, and push to the bottom. Pipette 5 mL of the purified sample to a sample vial or EP tube. Evaporate sample to dryness and redissolve by 1 mL of 20% acetonitrile solution. Vortex to dissolve the residue. After filter by 0.22 μm microporous membrane, the sample is ready for analysis.

Preparation of Standard Curve Solution

Prepare blank sample in the same procedures. Add an appropriate amount of standard solution. Evaporate to dryness at 45 °C and dilute by 1 mL of 20% acetonitrile solution to prepare the concentration of 2 ng/mL, 5 ng/mL, 10 ng/mL, 20 ng/mL, 50 ng/mL, and 100 ng/mL.

Instrument Conditions

1.Chromatography Conditions

Instrument: Thermo Fisher TSQ Endura

Chromatographic column: Elite SinoPaK BEH T-C18 (2.1 mm×100 mm,3 μm)

Mobile phase: A: 5 mmol/L Ammonium acetate solution, B: Methanol (contains 0.1% formic acid)

Flow Rate: 0.3 mL/min

Column Temperature: 30℃

Injection volume: 5 μL

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)	C (%)
0.0	10	90	8
1.2	40	60	8
2.1	10	90	0
4.8	10	90	0
5.0	90	10	8
6.0	90	10	8

2.Mass Spectrometry Conditions

Ion source: HESI

Scan mode: positive ion (ESI+) and negative ion mode (ESI-)

Sheath gas pressure: 30 arb

Auxiliary gas pressure: 8 arb

Ion exchange tube: 300 ℃

Auxiliary gas temperature: 350 ℃

Table 2. Targets and Characteristic Ions (*Quantitative Ions)

Targets	Parent Ion	Daughter Ion	Ion mode
Aflatoxin B1	313.1	241.1,285*	ESI+
T-2 toxin	489.1	387.2,245.2*	ESI+
Zearalenone	317.0	273.1,175.1*	ESI-

Results

Table 3. Spike Recovery

Targets	5.00 μg/kg		10.0 μg/kg		20.0 μg/kg	
	Recovery (%)	RSD/% (n=3)	Recovery (%)	RSD/% (n=3)	Recovery (%)	RSD/% (n=3)
Aflatoxin B1	90.9	5.6	92.3	4.4	93.8	4.8
T-2 Toxin	96.2	4.9	104	3.8	101	5.2
Zearalenone	71.6	5.8	78.1	5.1	88.9	6.1

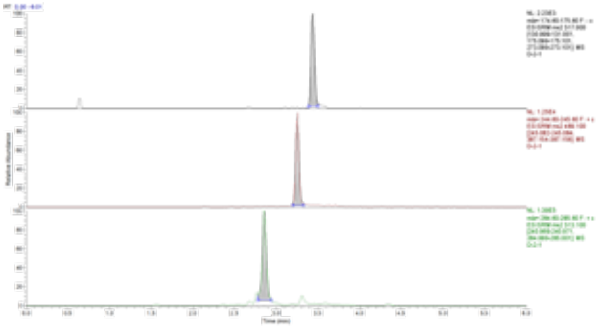


Figure 1. EIC of Targets at 10.0 μg/kg

Ordering Information

Cat. #	Description	Qty.
COAF226	Copure® 226 Multifunctional Purification Column, Application: Zearalenone, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
SDC-3000-D	Biocomma® Multi-Tube Vortex Mixer	1 Set/Ctn.
SF130-22-NL	Syringe Filters NL / Φ13 mm / 0.22 μm / Hydrophilic	100 Pcs/Box
SC2-1	Blue polypropylene screw caps with white PTFE/red silicone septa, 6 mm hole	100 Pcs/Box
V2-AL	Screw-thread vials with write-on spot, amber	100 Pcs/Box
COAF302	Copure® 302 Multifunctional Purification Column, Multiple functions	25 Pcs/Box

Aflatoxins In Vegetable Oil (Copure® 226 Multifunctional Purification Column)

Introduction

Aflatoxin, a secondary metabolite synthesized by fungi such as Aspergillus flavus and Aspergillus parasiticus via the polyketide pathway, typically comprises a difuran ring and an oxinone. Natural aflatoxins B1, B2, G1, and G2 are classified based on their chemical structures. These mycotoxins, present in contaminated crops used for livestock feed, can accumulate in meat, eggs, milk, and other foods, posing health risks through the food chain.

Biocomma established a rapid LC-MS/MS method using Copure® 228 Multifunctional Purification Columns for detection of aflatoxins B1, B2, G1, and G2 in in vegetable oil with good recovery and stability for your reference. The recoveries were 90-100% with RSD less than 5% at three different concentrations. This method is also applicable to cereals, cereal products, nuts, seeds, oils, condiments, infant formulas, and supplementary foods.

Experiment

Extraction

Weigh 5 g of sample into a 50 mL centrifuge tube. Add isotope internal standard and vortex to mix well. Let stand for 30 min. Add 20 mL of acetonitrile-water solution (84+16) and vortex to mix well. Then, extract by ultrasonic treatment for 20 min. Centrifuge at 6000 r/min for 10 min. The supernatant is ready for purification.

Purification

Add 10 mL of supernatant to a glass tube. Insert the rubber head of the purification column into the tube, and push to the bottom. Pipette 5 mL of the purified sample to a sample vial or EP tube. Evaporate sample to dryness and redissolve by 1 mL of initial mobile phase. Vortex for 30 seconds to dissolve the residue. After filter by microporous membrane, the sample is ready for analysis.

Instrument Conditions

1.Chromatography Conditions

Equipment: LC-MS/MS SCIEX Triple Quad™ 4500

Chromatographic column: Waters C18 (2.1 mm×100 mm, 1.7um)

Mobile phase: A:0.1% Formic acid water, B: Acetonitrile

Mobile phase gradient: Initial 80%A, 20%A (0 min~2 min), 20%A (2 min~3 min), 80%A (3 min~3.1min), 80%A (3.1min~5min).

Flow rate: 0.3 mL/min

Column temperature: room temperature

Injection volume: 10.0 μL

2.Mass Spectrometry Conditions

Detection mode: MRM

Table 1. Ion Source Control Conditions

Curtain Gas (CUR)	35.0
Collision Gas (CAD)	9
IonSpray Voltage (IS)	5500
Temperature (TEM)	550
Ion Source Gas1 (GS1)	50.0
Ion Source Gas2 (GS2)	50.0

Table 2. Ion Selection Parameters (*Quantitative Ions)

Targets	Parent ion (m/z)	Daughter ion (m/z)	DP (V)	CE (V)
AFB1	313.000	285.000*	100.000	33.000
	313.000	241.000	100.000	51.000
AFB2	315.000	287.000*	95.000	37.000
	315.000	259.000	89.000	41.000
AFG1	329.000	243.000*	98.000	39.000
	329.000	283.000	91.000	35.000
AFG2	331.000	245.000*	113.000	41.000
	331.000	285.000	103.000	39.000

Results

Table 3. Spiked Aflatoxins in Vegetable Oil Recovery

Targets	Spike (ng/g)	Copure®226 Multifunctional Purification Column		Brand A		Brand B	
		Recovery (%)	RSD /%	Recovery (%)	RSD/%	Recovery (%)	RSD /%
AFB1	0.2	95.7	3.12	95.4	5.25	90.1	5.21
	0.5	94.5	4.47	96.7	5.13	89.1	5.05
	1.0	96.7	4.21	93.2	5.04	89.5	4.52
AFB2	0.2	95.4	3.50	82.1	4.85	95.1	5.11
	0.5	93.5	4.31	92.7	4.75	94.2	5.01
	1.0	94.5	4.52	97.2	3.95	93.4	4.25
AFG1	0.2	95.9	4.15	97.3	4.31	97.2	5.21
	0.5	92.8	4.31	98.2	4.21	95.2	4.35
	1.0	93.9	4.29	88.3	5.25	88.1	3.24
AFG2	0.2	95.1	4.25	95.1	4.75	92.1	3.56
	0.5	97.1	4.25	92.1	3.19	93.5	4.27
	1.0	93.2	4.20	92.2	4.27	91.2	4.13

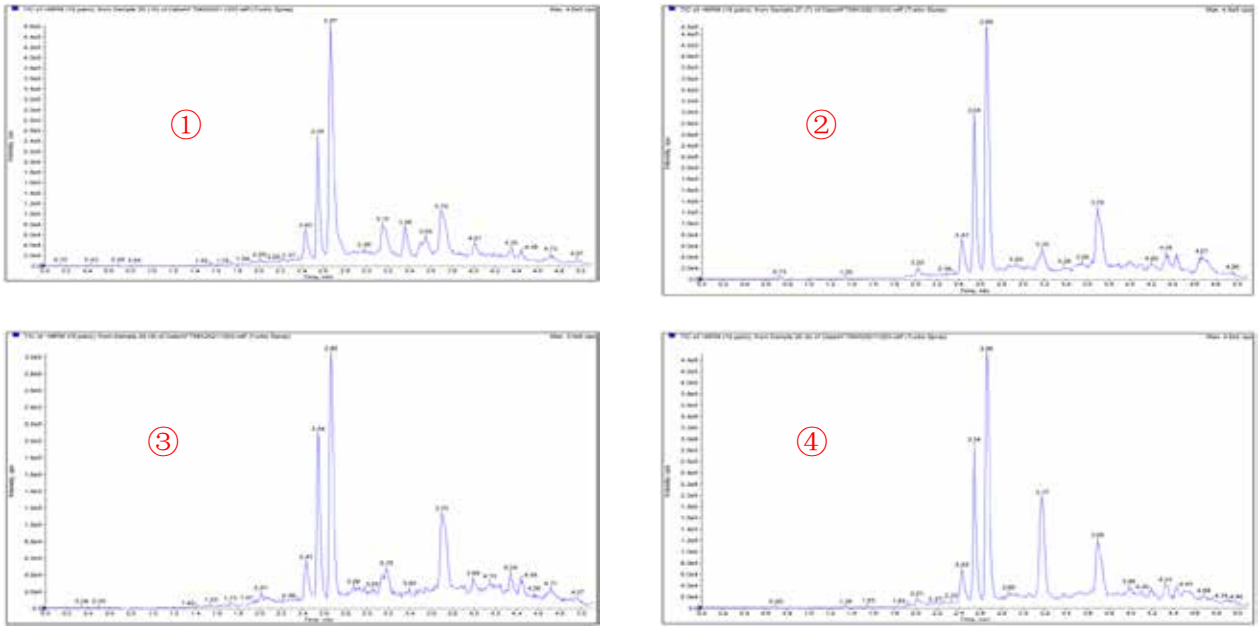


Figure 1. TIC of Spiked Samples by Different Brands of Multifunctional Purification Columns

① Before Purification

② Purified by Copure® 226 Multifunctional Purification Column

③ Purified by Brand A

④ Purified by Brand B

Based on Figure 1, the purification effect of Copure® 226 Multifunctional Purification Column is similar to Brand A and better than Brand B. The recovery rates of aflatoxin in the 24 wells are between 90-100%, and RSD is less than 5%, which meet the standard of experimental requirements.

Ordering Information

Cat. #	Description	Application	Qty.
COAF226	Copure® 226 Multifunctional Purification Column	Zearalenone, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF228	Copure® 228 Multifunctional Purification Column	Patulin, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF224	Copure® 224 Multifunctional Purification Column	Zearalenone	25 Pcs/Box
COAF223	Copure® 223 Multifunctional Purification Column	Aflatoxin M1 M2	25 Pcs/Box
COAF229	Copure® 229 Multifunctional Purification Column	Ochratoxin A	25 Pcs/Box
COAF230	Copure® 230 Multifunctional Purification Column	Deoxynivalenol	25 Pcs/Box
COAF302	Copure® 302 Multifunctional Purification Column	Multiple functions	25 Pcs/Box

Aflatoxins in Corn Flour (Copure® 228 Multifunctional Purification Column)

Introduction

Aflatoxin, a secondary metabolite synthesized by fungi such as Aspergillus flavus and Aspergillus parasiticus via the polyketide pathway, typically comprises a difuran ring and an oxinone. Natural aflatoxins B1, B2, G1, and G2 are classified based on their chemical structures. These mycotoxins, present in contaminated crops used for livestock feed, can accumulate in meat, eggs, milk, and other foods, posing health risks through the food chain.

Biocomma established a rapid LC-MS/MS method using Copure® 228 Multifunctional Purification Columns for detection of aflatoxins B1, B2, G1, and G2 in corn flour with good recovery and stability for your reference. The recoveries were 90-100% with RSD less than 5% at three different concentrations. This method is also applicable to cereals, cereal products, nuts, seeds, oils, condiments, infant formulas, and supplementary foods.

Experiment

Extraction

Weigh 5 g of sample into a 50 mL centrifuge tube. Add isotope internal standard and vortex to mix well. Let stand for 30 min. Add 20 mL of acetonitrile-water solution (84+16) and vortex to mix well. Then, extract by ultrasonic treatment for 20 min. Centrifuge at 6000 r/min for 10 min. The supernatant is ready for purification.

Purification (Copure® 228 Multifunctional Purification Column)

Add 10 mL of supernatant to a glass tube. Insert the rubber head of the purification column into the tube, and push to the bottom. Pipette 5 mL of the purified sample to a sample vial or EP tube. Evaporate sample to dryness and redissolve by 1 mL of initial mobile phase. Vortex for 30 seconds to dissolve the residue. After filter by microporous membrane, the sample is ready for analysis.

Instrument Conditions

1.Chromatography Conditions

Equipment: LC-MS SCIEX Triple Quad™ 4500

Chromatographic column: Waters C18 (2.1 mm×100 mm, 1.7um)

Mobile phase: A:0.1% Formic acid water, B: Acetonitrile

Mobile phase gradient: initial 80%A, 20%A (0 min~2 min), 20%A (2 min~3 min), 80%A (3 min~3.1 min), 80%A (3.1 min~5 min).

Flow rate: 0.3 mL/min

Column temperature: room temperature

Injection volume: 10.0 L

2.Mass Spectrometry Conditions

Detection mode: MRM

Table 1. Ion Source Control Conditions

Curtain Gas (CUR)	35.0
Collision Gas (CAD)	9
IonSpray Voltage (IS)	5500
Temperature (TEM)	550
Ion Source Gas1 (GS1)	50.0
Ion Source Gas2 (GS2)	50.0

Table 2. Ion Selection Parameters (*Quantitative Ions)

Targets	Parent Ion (m/z)	Daughter Ion (m/z)	DP (V)	CE (V)
AFB1	313.000	285.000*	100.000	33.000
	313.000	241.000	100.000	51.000
AFB2	315.000	287.000*	95.000	37.000
	315.000	259.000	89.000	41.000
AFG1	329.000	243.000*	98.000	39.000
	329.000	283.000	91.000	35.000
AFG2	331.000	245.000*	113.000	41.000
	331.000	285.000	103.000	39.000

Results

Table 3. Spiked Aflatoxin in Corn Flour Recovery

Targets	Spike (ng/g)	Copure® 228 Multifunctional Purification Column		Brand A		Brand B	
		Recovery (%)	RSD/%	Recovery (%)	RSD/%	Recovery (%)	RSD/%
AFB1	0.2	94.1	3.1	93.7	5.8	90.1	5.3
	0.5	91.0	4.5	92.5	5.0	88.5	4.5
	1.0	92.1	4.8	91.0	5.4	87.6	5.2
AFB2	0.2	93.2	5.0	89.5	4.2	96.5	6.1
	0.5	95.3	4.1	93.5	4.2	93.1	4.1
	1.0	90.6	4.2	91.4	3.8	95.2	4.8
AFG1	0.2	93.7	4.5	90.8	4.9	92.1	5.9
	0.5	95.2	4.8	96.3	4.8	94.3	4.5
	1.0	94.5	3.9	89.2	5.7	85.2	3.7
AFG2	0.2	95.4	3.5	93.7	4.5	90.7	3.1
	0.5	92.7	2.5	90.2	3.9	91.6	4.5
	1.0	91.3	4.6	91.6	4.7	92.7	4.9

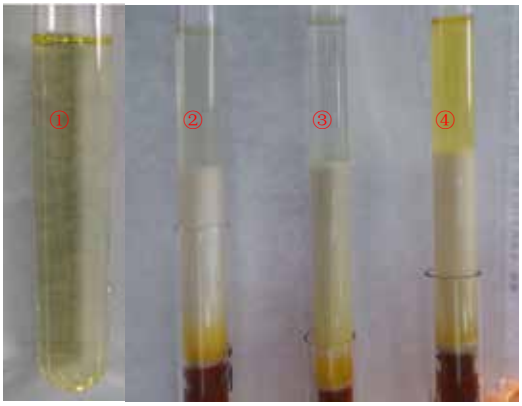


Figure 1. Decolorization Effect by Different Brands of Multifunctional Purification Columns
① Corn flour Sample Before Purification
② Purified by Copure® 224 Multifunctional Purification Column
③ Purified by Brand A
④ Purified by Brand B

Based on Figure 1, the pigments are obviously absorbed by Copure® 224 Multifunctional Purification Column. The decolorization ability is similar to Brand A and much better than Brand B.

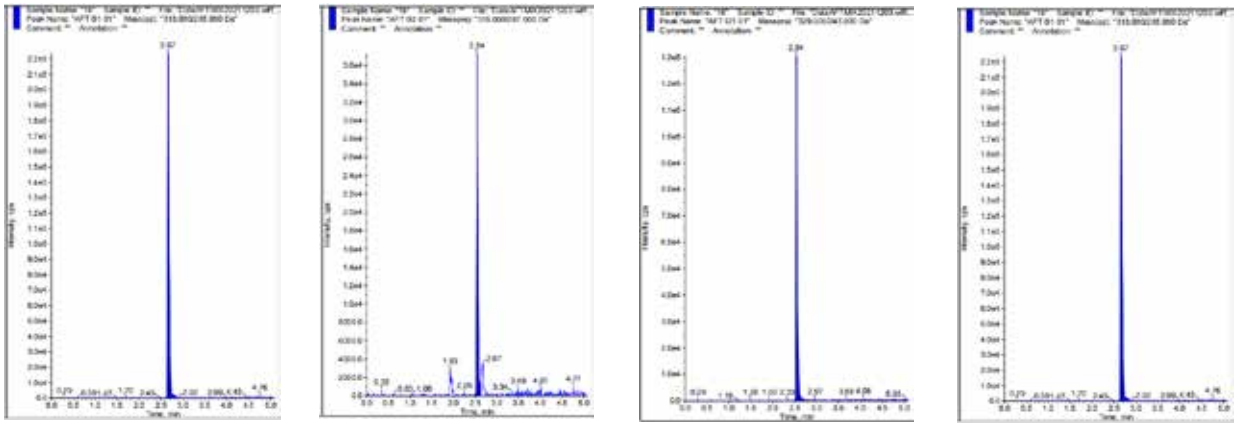


Figure 2. EIC of Spiked Sample by Copure® 228 Multifunctional Purification Column

Ordering Information

Cat. #	Description	Application	Qty.
COAF226	Copure® 226 Multifunctional Purification Column	Zearalenone, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF228	Copure® 228 Multifunctional Purification Column	Patulin, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF224	Copure® 224 Multifunctional Purification Column	Zearalenone	25 Pcs/Box
COAF223	Copure® 223 Multifunctional Purification Column	Aflatoxin M1 M2	25 Pcs/Box
COAF229	Copure® 229 Multifunctional Purification Column	Ochratoxin A	25 Pcs/Box
COAF230	Copure® 230 Multifunctional Purification Column	Deoxynivalenol	25 Pcs/Box
COAF302	Copure® 302 Multifunctional Purification Column	Multiple functions	25 Pcs/Box

10 Mycotoxins in Wheat Flour (Copure® 302 Multifunctional Purification Column)

Introduction

Mycotoxins are toxic secondary metabolites produced by fungi, primarily absorbed by humans through diet. Aspergillus, Penicillium, and Fusarium are the main genera generating the most common mycotoxins found in food, including aflatoxin, ochratoxin A, sterigmatocystin, patulin, fumonisin, zearalenone, deoxynivalenol, NIV, T-2 toxin, etc. Biocomma established an LC-MS/MS method using Copure® 302 Multifunctional Purification Column for detection of 10 mycotoxins in wheat flour with good recovery and stability for your reference.

Experiment

Extraction

Weigh 5 g of sample into a 50 mL centrifuge tube. Add 20 mL of acetonitrile-water-acetic acid solution (80:19:1) and vortex to mix well. Then, extract by ultrasonic treatment for 20 min. Centrifuge at 6000 r/min for 10 min. The supernatant is ready for purification.

Purification (Copure® 302 Multifunctional Purification Column)

Add 10 mL of prepared sample to a glass tube. Insert the rubber head of the purification column into the tube, and push to the bottom. Pipette 5 mL of the purified sample to a sample vial or EP tube. Evaporate sample to dryness at 40 ℃. Dilute to 1 mL by adding acetonitrile-water solution (50:50). Vortex for 30 seconds to dissolve the residue. After filter by microporous membrane, the sample is ready for analysis.

Instrument Conditions

1.Chromatography Conditions

Equipment: UPLC-MS/MS (Thermo Scientific TSQ Endura)
Chromatographic column: Commasil® BEH T-C18 (2.1 mm×100 mm, 3 μm)
Mobile phase: A:5mM Ammonium acetate solution (contains 0.1% formic acid)
Mobile phase: B: Acetonitrile (contains 0.1% formic acid)
Mobile phase gradient: initial 90%A,40%A (0 min~2 min), 10%A (2 min~6 min), 10%A (6min~7 min), 90%A (7 min~8 min), 90%A (8 min~10 min)
Flow rate: 0.3 ml/min
Column temperature: room temperature
Injection volume: 5.0 μL

2.Mass Spectrometry Conditions

Sheath gas pressure: 30 arb
Auxiliary gas pressure: 8 arb
Ion exchange tube: 300 ℃
Auxiliary air temperature: 350 ℃
Detection mode: MRM
Scan method: positive ion mode (ESI+) and negative ion mode (ESI-)

Table 1. Ion Selection Parameters (*Quantitative Ions)

Targets	Peak Time (min)	Parent Ion (m/z)	Daughter Ion (m/z)	RF Lens (V)	CE (V)
Aflatoxin B1	2.98	313.0	285.0	195	33.00
		313.0	241.0*	195	51.00
Aflatoxin B1	2.90	315.0	287.0	176	37.00
		315.0	259.0*	176	41.00
Aflatoxin G1	2.91	329.0	243.0*	192	39.00
		329.0	283.0	192	35.00
Aflatoxin G2	4.09	331.0	245.0*	169	41.00
		331.0	285.0	169	39.00
Fumonisin B1	2.66	722.0	334.2*	298	32.75
		722.0	352.3	298	30.51
Fumonisin B2	2.81	706.1	336.3*	233	31.72
		706.1	354.3	233	30.12
Fumonisin B2	3.57	489	245.1*	217	26.15
		489	387.1	217	20.08
T-2 toxin	3.11	319	69*	93	40.00
		319	283	93	15.00
Ochratoxin A	3.72	404.1	238.9*	132	22.28
		404.1	358.0	132	13.15
Zearalenone	3.82	317	175.1*	99	18.64
		317	187	99	10.23

Note: The detection of zearalenone is in negative ion mode (ESI-), and the others are in positive ion mode (ESI+).

Results

Table 2. Spiked Recovery of 10 Mycotoxins in Wheat Flour

Test	Spike (ng/g))	Copure® 302 Multifunctional Purification Column		Brand A	
		Recovery (%)	RSD/%	Recovery (%)	RSD/%
Aflatoxin B1	0.5	92.5	6.1	85.1	6.2
	1.0	92.1	6.5	83.5	6.5
Aflatoxin B2	0.5	93.5	5.0	89.6	7.1
	1.0	94.5	5.1	90.5	7.5
Aflatoxin G1	0.5	94.0	6.5	92.2	7.5
	1.0	95.5	6.8	96.2	6.5
Aflatoxin G2	0.5	96.5	7.5	91.5	6.4
	1.0	95.8	7.5	93.1	6.1
Fumonisin B1	10	94.2	6.5	92.1	7.3
	20	93.2	6.5	87.5	7.5
Fumonisin B2	10	94.1	5.5	92.1	8.5
	20	92.7	5.7	93.1	8.6
T-2 Toxin	5	96.5	7.5	90.5	6.9
	10	95.8	8.5	89.4	7.5
Vomitoxin	25	91.1	8.1	91.2	5.1
	50	95.2	8.1	92.1	6.5
Ochratoxin A	5	96.1	7.5	82.5	6.3
	10	98.2	7.6	81.4	7.5
Zearalenone	5	95.1	6.3	83.2	8.1
	10	97.4	6.5	84.5	8.5

Based on Table 2, the recovery rates of 10 Mycotoxins are all between 90-110% with RSD less than 10%, which meet the standard of the experimental requirements. The overall evaluation of recovery rate is better than Brand A.

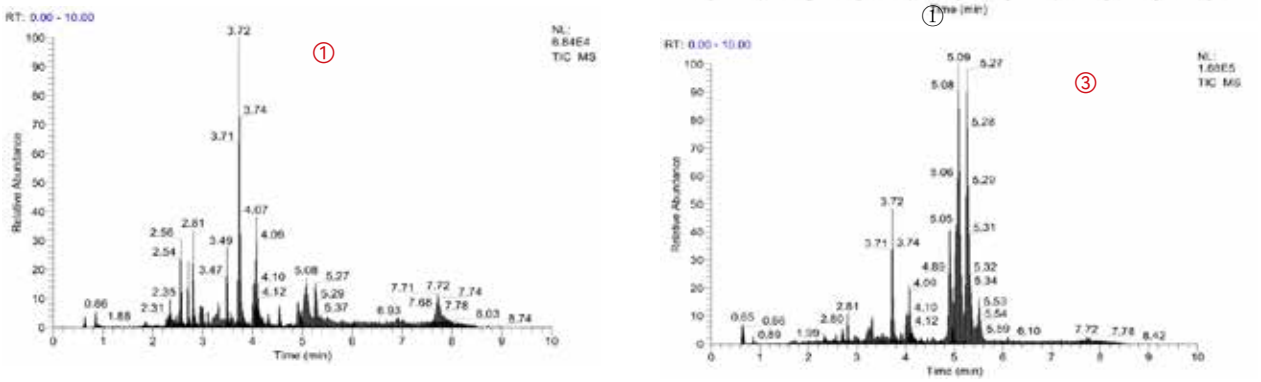


Figure 1. TIC of Wheat Flour Sample

① Before Purification

② Purified by Brand A

③ Purified by Copure® 302 Multifunctional Purification Column

Based on Figure 1, the interference of impurities in sample ③ is obviously reduced, therefore miscellaneous peaks are fewer in the TIC chromatogram. The purification effect of Copure® 302 Multifunctional Purification Column is better than Brand A to meet experiment requirements.

Ordering Information

Cat. #	Description	Application	Qty.
COAF226	Copure® 226 Multifunctional Purification Column	Zearalenone, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF228	Copure® 228 Multifunctional Purification Column	Patulin, Aflatoxin B1 B2 G1 G2	25 Pcs/Box
COAF224	Copure® 224 Multifunctional Purification Column	Zearalenone	25 Pcs/Box
COAF223	Copure® 223 Multifunctional Purification Column	Aflatoxin M1 M2	25 Pcs/Box
COAF229	Copure® 229 Multifunctional Purification Column	Ochratoxin A	25 Pcs/Box
COAF230	Copure® 230 Multifunctional Purification Column	Deoxynivalenol	25 Pcs/Box
COAF302	Copure® 302 Multifunctional Purification Column	Multiple functions	25 Pcs/Box

High-Throughput of Zearalenone in Corn Flour (Copure® 224 Multifunctional Purification Plate)

Introduction

Zearalenone, a non-steroidal mycotoxin from Fusarium fungus, is commonly found in contaminated grains (wheat, corn, oats, barley, etc.). It's a major global crop contaminant, leading to adverse effects such as tumors, estrogen presence, and immune suppression in livestock and poultry that consume contaminated feed, and then poses a risk to human health.

Biocomma established a high-throughput LC method using Copure® 224 Multifunctional Purification Plate for detection of zearalenone in corn flour with good recovery and stability for your reference. The recoveries were 90-110%, and CV value between wells was less than 5% at both 8 ng/g and 16 ng/g concentrations.

Experiment

Extraction

Weigh 5 g of sample into a 50 mL centrifuge tube. Add 1 g of sodium chloride and 20 mL of acetonitrile-water solution (9+1). Vortex for 15 min. Centrifuge at 6000 r/min for 5 min. The supernatant is ready for purification.

Purification (Copure® 224 Multifunctional Purification Plate)

Place a 24 well purification plate on a 24 well collection plate. Add 6 mL of supernatant to the purification plate. Place the 24 well purification plate and the collection plate on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the collection plate. Pipette 4 mL of sample to a clean tube. Evaporate sample to dryness at 40 ℃ and redissolve by 1 mL of initial mobile phase. Vortex for 30 seconds to dissolve the residue. After filter by 0.22 μm microporous membrane, the sample is ready for analysis.

Instrument Conditions

Equipment: UltiMate 3000 (Thermo Fisher Scientific) with FLD detector

Chromatographic column: Agilent ZORBAX C18 (4.6 mm×250 mm, 5 μm)

Mobile phase: A: Acetonitrile, B: Deionized water, C: Methanol

Flow rate: 1.0 mL/min

Column temperature: room temperature

Injection volume: 50.0 μL

Detection wavelength: excitation wavelength 274 nm, emission wavelength 440 nm

Detector: FLD

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)	C (%)
0.00	52	40	8
5.00	52	40	8
8.00	95	5	0
11.00	95	5	0
13.00	52	40	8
17.00	52	40	8

Results

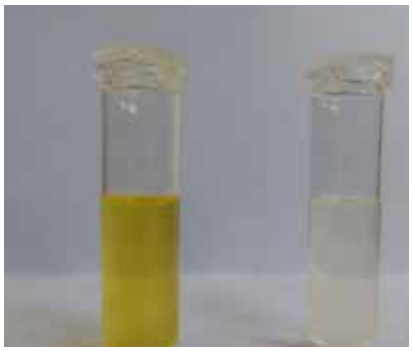


Figure 1. Decolorization Effect

① Before Purification

② Purified by Copure® 226 Multifunctional Purification Plate

Based on Figure 1, the pigments are obviously absorbed by Copure® 226 26 Multifunctional Purification Plate.

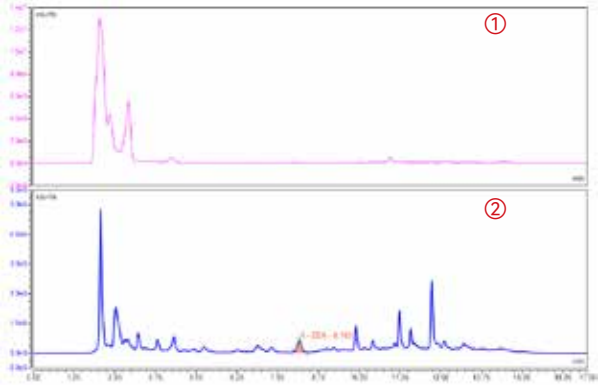


Figure 2. Chromatograms of Corn Flour Sample

① Before Purification

② Purified by Copure® 224 Multifunctional Purification Plate

Table 2. Zearalenone Spike Recovery in Corn Flour

Test	Spike (ng/g)	Copure® 224 Multifunctional Purification Plate	
		Recovery (%, n=24)	CV (%)
Zearalenone	8	96.1	3.64
	16	95.4	4.17

Based on Figure 2 and Table 2, the miscellaneous peaks of purified sample are much fewer indicating obviously absorbed impurities. The recovery rates of aflatoxin in the 24 wells are between 90-110%, and the CV value is less than 5%, which meet the standard of experimental requirements.

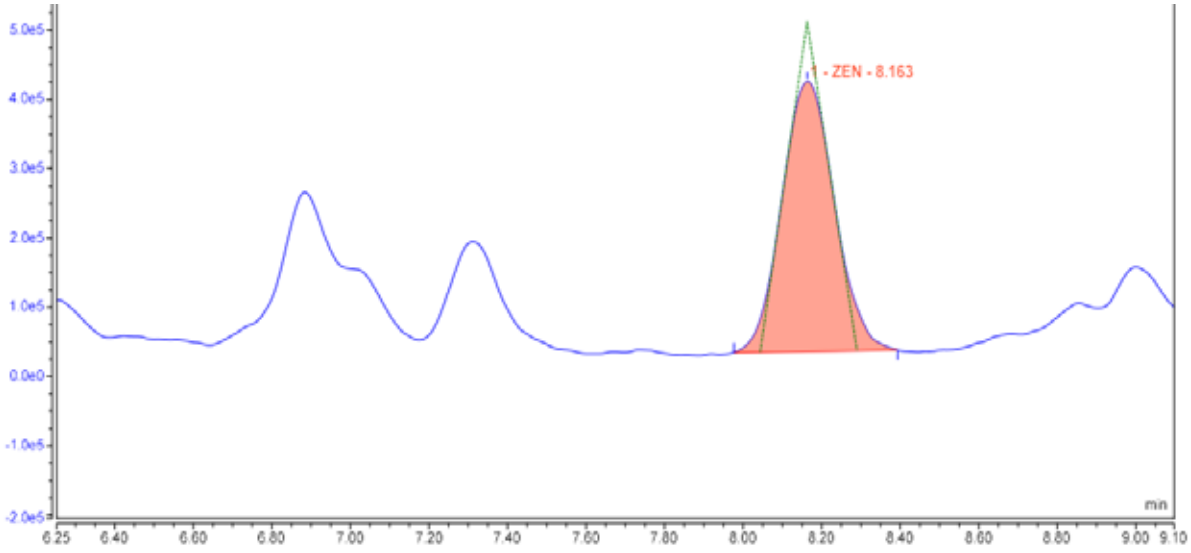


Figure 3. Chromatogram of Spiked Sample at 8 ng/g
(Purified by Copure® 224 Multifunctional Purification Plate)

Ordering Information

Cat. #	Description	Application	Qty.
COAF226-GTL	Copure® 226 Multifunctional Purification Plate	Zearalenone, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF228-GTL	Copure® 228 Multifunctional Purification Plate	Patulin, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF224-GTL	Copure® 224 Multifunctional Purification Plate	Zearalenone	1 Pc/Box
COAF223-GTL	Copure® 223 Multifunctional Purification Plate	Aflatoxin M1, M2	1 Pc/Box
COAF229-GTL	Copure® 229 Multifunctional Purification Plate	Ochratoxin	1 Pc/Box
COAF230-GTL	Copure® 230 Multifunctional Purification Plate	Deoxynivalenol	1 Pc/Box
COAF302-GTL	Copure® 302 Multifunctional Purification Plate	Multiple functions	1 Pc/Box

High-Throughput of Aflatoxins in Corn Flour (Copure® 226 Multifunctional Purification Plate)

Introduction

Aflatoxin, a secondary metabolite synthesized by fungi such as Aspergillus flavus and Aspergillus parasiticus via the polyketide pathway, typically comprises a difuran ring and an oxinone. Natural aflatoxins B1, B2, G1, and G2 are classified based on their chemical structures. These mycotoxins, present in contaminated crops used for livestock feed, can accumulate in meat, eggs, milk, and other foods, posing health risks through the food chain.

Biocomma established a high-throughput LC-MS/MS method using Copure® 226 Multifunctional Purification Plate for detection of aflatoxins B1, B2, G1, and G2 in corn flour with good recovery and stability for your reference. The recoveries were 90-110%, and CV value between wells was less than 5% at both 8 ng/g and 16 ng/g concentrations. This method is also applicable to cereals, cereal products, nuts, seeds, oils, condiments, infant formulas, and supplementary foods.

Experiment

Extraction

Weigh 5 g of sample into a 50 mL centrifuge tube. Add isotope internal standard and vortex to mix well. Let stand for 30 min. Add 20 mL of acetonitrile-water solution (84:16) and vortex to mix well. Then, extract by ultrasonic treatment for 20 min. Centrifuge at 6000 r/min for 10 min. The supernatant is ready for purification.

Purification (Copure® 226 Multifunctional Purification Plate)

Place a 24 well purification plate on a 24 well collection plate. Add 6 mL of supernatant to the purification plate. Place the 24 well purification plate and the collection plate on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the collection plate. Pipette 4 mL of sample to a clean tube. Evaporate sample to dryness at 40 ℃ and redissolve by 1 mL of initial mobile phase. Vortex for 30 seconds to dissolve the residue. After filter by 0.22 μm microporous membrane, the sample is ready for analysis.

Instrument Conditions

1.Chromatography Conditions

Equipment: UPLC-MS/MS (Thermo Scientific TSQ Endura)

Chromatographic column: Thermo C18 (2.1 mm×100 mm, 1.7 um)

Mobile Phase: A: Water (0.1% Formic acid), B: Acetonitrile

Mobile Phase Gradient: Initial 80%A, 20%A (0 min~2 min), 20%A (2 min~3 min), 80%A (3 min~3.1min), 80%A (3.1min~5min)

Flow Rate: 0.3 mL/min

Column Temperature: room temperature

Injection Volume: 5.0 μL

2.Mass Spectrometry Conditions

Detection method: MRM

Table 1. Ion Source Control Conditions

Curtain Gas (CUR)	35.0
Collision Gas (CAD)	9
IonSpray Voltage (IS)	5500
Temperature (TEM)	550
Ion Source Gas1 (GS1)	50.0
Ion Source Gas2 (GS2)	50.0

Table 2. Ion Selection Parameters (*Quantitative Ions)

Targets	Parent ion (m/z)	Daughter ion (m/z)	DP (V)	CE (V)
AFT B1	313.000	285.000*	100.000	33.000
	313.000	241.000	100.000	51.000
AFT B2	315.000	287.000*	95.000	37.000
	315.000	259.000	89.000	41.000
AFT G1	329.000	243.000*	98.000	39.000
	329.000	283.000	91.000	35.000
AFT G2	331.000	245.000*	113.000	41.000
	331.000	285.000	103.000	39.000

Results

Figure 1. Decolorization Effect

① Before Purification

② Purified by Copure® 226 Multifunctional Purification Plate

Based on Figure 1, the pigments are obviously absorbed by Copure® 226 26 Multifunctional Purification Plate.

Table 3. Aflatoxin Spike in Corn Flour Recovery

Targets	Spike (ng/g)	Copure® 226 Multifunctional Purification Plate	
		Recovery (%, n=24)	CV/%
AFT B1	0.5	105	3.92
	1.0	101	2.41
AFT B2	0.5	102	4.12
	1.0	95.8	4.15
AFT G1	0.5	105	2.69
	1.0	104	4.13
AFT G2	0.5	101	4.28
	1.0	95.4	3.75

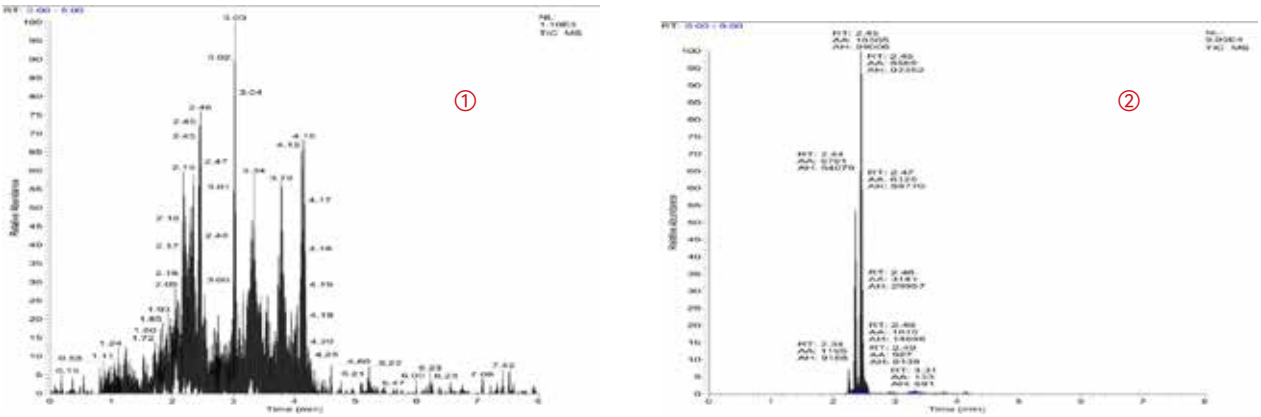


Figure 2. TIC of Corn Flour Sample

① Before Purification

② Purified by Copure® 226 Multifunctional Purification Plate

Based on Table 3 and Figure 2, the miscellaneous peaks of purified sample are much fewer indicating obviously absorbed impurities. The recovery rates of aflatoxin in the 24 wells are between 90-110%, and the CV value is less than 5%, which meet the standard of experimental requirements.

Ordering Information

Cat. #	Description	Application	Qty.
COAF226-GTL	Copure® 226 Multifunctional Purification Plate	Zearalenone, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF228-GTL	Copure® 228 Multifunctional Purification Plate	Patulin, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF224-GTL	Copure® 224 Multifunctional Purification Plate	Zearalenone	1 Pc/Box
COAF223-GTL	Copure® 223 Multifunctional Purification Plate	Aflatoxin M1, M2	1 Pc/Box
COAF229-GTL	Copure® 229 Multifunctional Purification Plate	Ochratoxin	1 Pc/Box
COAF230-GTL	Copure® 230 Multifunctional Purification Plate	Deoxynivalenol	1 Pc/Box
COAF302-GTL	Copure® 302 Multifunctional Purification Plate	Multiple functions	1 Pc/Box

High-Throughput of Patulin in Apple Cider (Copure® 228 Multifunctional Purification Plate)

Introduction

Patulin is commonly found in fruits, fruit products and fruit wine. Patulin is a strong toxin to humans and animals, potentially leading to respiratory issues.

Biocomma established a high-throughput LC method using Copure® 228 Multifunctional Purification Plate for detection of patulin in apple cider with good recovery and stability for your reference. The recoveries were 90-110%, and CV value between wells was less than 5% at both 5 ng/g and 10 ng/g concentrations. This method is also applicable to fruit and vegetable juices and alcoholic foods.

Experiment

Extraction

Weigh 4 g of sample and dilute to 25 mL with acetonitrile. Vortex for 5 min and centrifuge at 6000 r/min for 5 min. The supernatant is ready for purification.

Purification (Copure® 228 Multifunctional Purification Plate)

Place a 24 well purification plate on a 24 well collection plate. Add 6 mL of supernatant to the purification plate. Place the 24 well purification plate and the collection plate on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the collection plate. Pipette 4 mL of sample to a clean tube. Add 20 µL of acetic acid. Evaporate sample to dryness at 40 ℃ and redissolve by 1 mL of initial mobile phase. Vortex for 30 seconds to dissolve the residue. After filter by 0.22 µm microporous membrane, the sample is ready for analysis.

Instrument Conditions

Equipment: UltiMate 3000 (Thermo Fisher Scientific) with DAD detector.

Chromatographic column: Agilent ZORBAX C18 (4.6 mm×250 mm, 5 µm)

Mobile phase: A: Deionized water, B: Acetonitrile

Mobile phase gradient: 5% B (0min~13min), 100% B (13min~15min), 5% B (15min~20min)

Flow rate: 1.0 mL/min

Column temperature: 40 ℃

Injection volume: 20.0 µL

Detection wavelength: 276 nm

Detector: DAD

Results

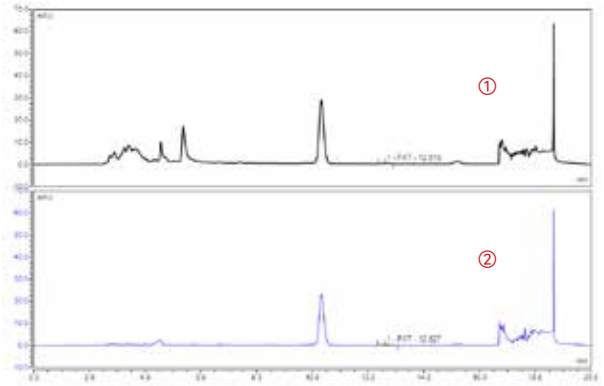


Figure 1. Chromatograms of Samples Before and After Treatment

① Before Purification

② Purified by Copure® 229 Multifunctional Purification Plate

Based on Figure 1, less miscellaneous peaks showing the impurities are obviously absorbed by Copure® 229 Multifunctional Purification Plate. There are no interference peaks next to the target peak, so the quantification is more accurate.

Table 1. Patulin Spike Recovery in Apple Cider

Test	Spike (ng/g)	Copure® 228 Multifunctional Purification Plate	
		Average recovery (%, n=24)	CV/%
Patulin	5	94.5	2.23
	10	95.6	3.45

Based on Table 1, the recovery rates of patulin in the 24 wells are all between 90-110%, and the CV value of the recovery rate between wells is less than 5%, which meet the standard of the experimental requirements.

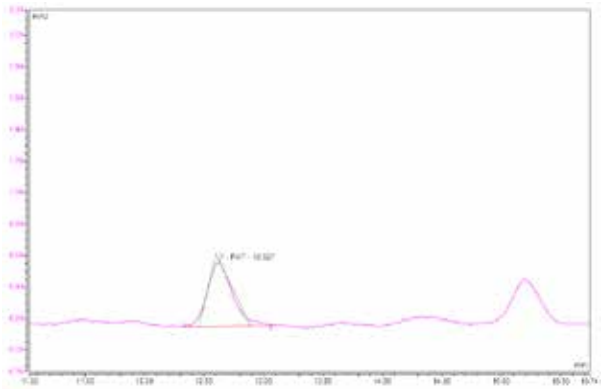


Figure 2. Chromatogram of Spiked Sample at 5 ng/g

(Purified by Copure® 228 Multifunctional Purification Plate)

Ordering Information

Cat. #	Description	Application	Qty.
COAF226-GTL	Copure® 226 Multifunctional Purification Plate	Zearalenone, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF228-GTL	Copure® 228 Multifunctional Purification Plate	Patulin, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF224-GTL	Copure® 224 Multifunctional Purification Plate	Zearalenone	1 Pc/Box
COAF223-GTL	Copure® 223 Multifunctional Purification Plate	Aflatoxin M1, M2	1 Pc/Box
COAF229-GTL	Copure® 229 Multifunctional Purification Plate	Ochratoxin	1 Pc/Box
COAF230-GTL	Copure® 230 Multifunctional Purification Plate	Deoxynivalenol	1 Pc/Box
COAF302-GTL	Copure® 302 Multifunctional Purification Plate	Multiple functions	1 Pc/Box

High-Throughput of Ochratoxin A in Soybean (Copure® 229 Multifunctional Purification Plate)

Introduction

Ochratoxins are metabolites of certain Aspergillus and Penicillium fungi. Ochratoxin A, B, and C differ in chemical structures. Ochratoxin A (OTA) is the most toxic and widely contaminated. Once ingested through contaminated agricultural products, OTA is absorbed through the gastrointestinal system and causes serious diseases.

Biocomma established a high-throughput LC method using Copure® 229 Multifunctional Purification Plate for detection of OTA in soybean with good recovery and stability for your reference. The recoveries were 90-110%, and CV value between wells was less than 5% at both 4 ng/g and 8 ng/g concentrations.

Experiment

Extraction

Weigh 5 g of sample into a 50 mL centrifuge tube. Add 20 mL of acetonitrile-water solution (84:16) and vortex for 15 min to mix well. Centrifuge at 6000 r/min for 5 min. The supernatant is ready for purification.

Purification (Copure® 229 Multifunctional Purification Plate)

Place a 24 well purification plate on a 24 well collection plate. Add 6 mL of supernatant to the purification plate. Place the 24 well purification plate and the collection plate on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the collection plate. Pipette 4 mL of sample to a clean tube. Evaporate sample to dryness at 40 ℃ and redissolve by 1 mL of initial mobile phase. Vortex for 30 seconds to dissolve the residue. After filter by 0.22 μm microporous membrane, the sample is ready for analysis.

Instrument Conditions

Equipment: UltiMate 3000 (Thermo Fisher Scientific) with FLD detector.

Chromatographic column: Agilent ZORBAX C18 (4.6 mm×250 mm,5 μm)

Mobile phase: A: 2% Acetic acid water, B: Acetonitrile

Flow rate: 1.0 mL/min

Column temperature: 30 ℃

Injection volume: 10.0 μL

Detection wavelength: excitation wavelength 333 nm, emission wavelength 460 nm

Detector: FLD

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.00	50	50
1.00	50	50
15.0	50	50

Results

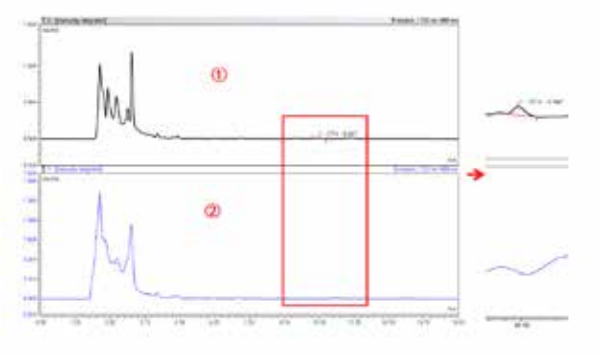


Figure 1. Chromatograms of Samples Before and After Treatment

① Purified by Copure® 229 Multifunctional Purification Plate

② Before Purification

Table 2. Spiked Ochratoxin A in Soybean Recovery

Test	Spike (ng/g)	Copure® 229 Multifunctional Purification Plate	
		Average Recovery (%, n=24)	CV/%
Ochratoxin A	4	91.3	2.32
	8	94.0	3.25

Based on Figure 1, the impurities are obviously absorbed by Copure® 229 Multifunctional Purification Plate. There are no interference peaks next to the target peak, so the quantification is more accurate. Based on Table 2, the recovery rates of Ochratoxin A in the 24 wells are all between 90-110%, and the CV value of the recovery rate between wells is less than 5%, which meet the standard of the experimental requirements.

Ordering Information

Cat. #	Description	Application	Qty.
COAF226-GTL	Copure® 226 Multifunctional Purification Plate	Zearalenone, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF228-GTL	Copure® 228 Multifunctional Purification Plate	Patulin, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF224-GTL	Copure® 224 Multifunctional Purification Plate	Zearalenone	1 Pc/Box
COAF223-GTL	Copure® 223 Multifunctional Purification Plate	Aflatoxin M1, M2	1 Pc/Box
COAF229-GTL	Copure® 229 Multifunctional Purification Plate	Ochratoxin	1 Pc/Box
COAF230-GTL	Copure® 230 Multifunctional Purification Plate	Deoxynivalenol	1 Pc/Box
COAF302-GTL	Copure® 302 Multifunctional Purification Plate	Multiple functions	1 Pc/Box

High-Throughput of Deoxynivalenol in Wheat Flour (Copure® 230 Multifunctional Purification Plate)

Introduction

Deoxynivalenol (DON) is a trichothecene compound widely present in wheat and its derivatives. Once ingested through contaminated agricultural products, DON leads to symptoms of poisoning, including vomiting, posing a significant threat to human health.

Biocomma established a high-throughput LC method using Copure® 230 Multifunctional Purification Plate for detection of DON in wheat flour with good recovery and stability for your reference. The recoveries were 90-110%, and CV value between wells was less than 5% at both 200 ng/g and 400 ng/g concentrations.

Experiment

Extraction

Weigh 2 g of sample into a 50 mL centrifuge tube. Add 20 mL of acetonitrile-water solution (9:1) and vortex for 15 min to mix well. Centrifuge at 6000 r/min for 5 min. The supernatant is ready for purification.

Purification (Copure® 230 Multifunctional Purification Plate)

Place a 24 well purification plate on a 24 well collection plate. Add 6 mL of supernatant to the purification plate. Place the 24 well purification plate and the collection plate on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the collection plate. Pipette 4 mL of sample to a clean tube. Evaporate sample to dryness at 40 ℃ and redissolve by 1 mL of initial mobile phase. Vortex for 30 seconds to dissolve the residue. After filter by 0.22 μm microporous membrane, the sample is ready for analysis.

Instrument Conditions

Equipment: UltiMate 3000 (Thermo Fisher Scientific) with DAD detector.

Chromatographic column: Agilent ZORBAX C18 (4.6 mm×250 mm,5 μm)

Mobile phase: A: Methanol, B: Deionized water

Flow rate: 0.8 mL/min

Column temperature: 35 ℃

Injection volume: 50.0 μL

Detection wavelength: 218 nm

Table 1. Gradient Elution Program

Time (min)	A (%)	B (%)
0.00	20	80
2.00	20	80
10.00	70	30
12.00	70	30
13.50	20	80
16.00	20	80

Results

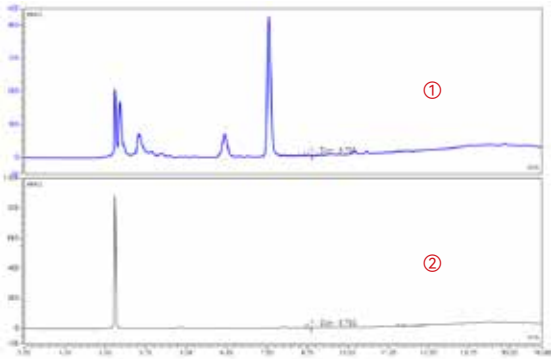


Figure 1. Chromatograms of Samples Before and After Treatment

① Before Purification

② Purified by Copure® 230 Multifunctional Purification Plate

Based on Figure 1, less miscellaneous peaks showing the impurities are obviously absorbed by Copure® 230 Multifunctional Purification Plate. There are no interference peaks next to the target peak, so the quantification is more accurate.

Table 2. Deoxynivalenol Spike Recovery in Wheat Flour

Test	Spike (ng/g)	Copure® 230 Multifunctional Purification Plate	
		Average Recovery (%, n=24)	CV/%
Deoxynivalenol	200	95.9	4.11
	400	91.2	4.25

Based on Table 2, the recovery rates of deoxynivalenol in the 24 wells are all between 90-110%, and the CV value of the recovery rate between wells is less than 5%, which meet the standard of the experimental requirements.

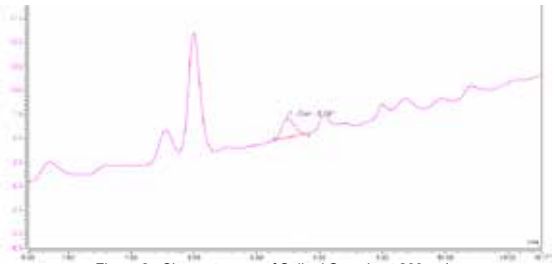


Figure 2. Chromatogram of Spiked Sample at 200 ng/g
(Purified by Copure® 230 Multifunctional Purification Plate)

Ordering Information

Cat. #	Description	Application	Qty.
COAF226-GTL	Copure® 226 Multifunctional Purification Plate	Zearalenone, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF228-GTL	Copure® 228 Multifunctional Purification Plate	Patulin, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF224-GTL	Copure® 224 Multifunctional Purification Plate	Zearalenone	1 Pc/Box
COAF223-GTL	Copure® 223 Multifunctional Purification Plate	Aflatoxin M1, M2	1 Pc/Box
COAF229-GTL	Copure® 229 Multifunctional Purification Plate	Ochratoxin	1 Pc/Box
COAF230-GTL	Copure® 230 Multifunctional Purification Plate	Deoxynivalenol	1 Pc/Box
COAF302-GTL	Copure® 302 Multifunctional Purification Plate	Multiple functions	1 Pc/Box

High-Throughput of 10 Mycotoxins in Corn Flour (Copure® 302 Multifunctional Purification Plate)

Introduction

Mycotoxins are toxic secondary metabolites produced by fungi, primarily absorbed by humans through diet. Aspergillus, Penicillium, and Fusarium are the main genera generating the most common mycotoxins found in food, including aflatoxin, ochratoxin A, sterigmatocystin, patulin, fumonisin, zearalenone, deoxynivalenol, NIV, T-2 toxin, etc. Biocomma established a high-throughput LC-MS/MS method using Copure® 302 Multifunctional Purification Plate for detection of 10 mycotoxins in corn flour with good recovery and stability for your reference.

Experiment

Extraction

Weigh 5 g of sample into a 50 mL centrifuge tube. Add 20 mL of acetonitrile-water-acetic acid solution (80:19:1) and vortex to mix well. Then, extract by ultrasonic treatment for 20 min. Centrifuge at 6000 r/min for 10 min. The supernatant is ready for purification.

Purification (Copure® 302 Multifunctional Purification Plate)

Place a 24 well purification plate on a 24 well collection plate. Add 6 mL of supernatant to the purification plate. Place the 24 well purification plate and the collection plate on the Biocomma® Positive Pressure 24 Processor. Turn on the gas. Make sure the wells of purification plate are positioned directly under the gas vent. Adjust the gas flow until all the sample filter into the collection plate. Pipette 4 mL of sample to a clean tube. Add 20 µL of acetic acid. Evaporate sample to dryness at 40 ℃ . Re-dilute to 1 mL by acetonitrile-water solution (50:50). Vortex for 30 seconds to dissolve the residue. After filter by 0.22 µm microporous membrane, the sample is ready for analysis.

Instrument Conditions

1.Chromatography Conditions

Equipment: UPLC-MS/MS (Thermo Scientific TSQ Endura)

Chromatographic column: Commasil® BEH T-C18 (2.1 mm×100 mm,3 µm)

Mobile phase: A:5mM Ammonium acetate solution (contains 0.1% formic acid)

Mobile phase: B: Acetonitrile (contains 0.1% formic acid)

Mobile phase gradient: initial 90%A,40%A (0 min~2 min), 10%A (2 min~6 min), 10%A (6min~7 min), 90%A (7 min~8 min), 90%A (8 min~10 min)

Flow rate: 0.3 ml/min

Column temperature: room temperature

Injection volume: 5.0 µL

2.Mass Spectrometry Conditions

Sheath gas pressure: 30 arb

Auxiliary gas pressure: 8 arb

Ion exchange tube: 300 ℃

Auxiliary air temperature: 350 ℃

Detection method: MRM

Scan method: positive ion mode (ESI+) and negative ion mode (ESI-)

Targets	Peak Time (min)	Parent Ion (m/z)	Daughter Ion (m/z)	RF Lens (V)	CE (V)
Aflatoxin B1	2.98	313.0	285.0	195	33.00
		313.0	241.0*	195	51.00
Aflatoxin B1	2.90	315.0	287.0	176	37.00
		315.0	259.0*	176	41.00
Aflatoxin G1	2.91	329.0	243.0*	192	39.00
		329.0	283.0	192	35.00
Aflatoxin G2	4.09	331.0	245.0*	169	41.00
		331.0	285.0	169	39.00
Fumonisin B1	2.66	722.0	334.2*	298	32.75
		722.0	352.3	298	30.51
Fumonisin B2	2.81	706.1	336.3*	233	31.72
		706.1	354.3	233	30.12
Fumonisin B2	3.57	489	245.1*	217	26.15
		489	387.1	217	20.08
T-2 toxin	3.11	319	69*	93	40.00
		319	283	93	15.00
Ochratoxin A	3.72	404.1	238.9*	132	22.28
		404.1	358.0	132	13.15
Zearalenone	3.82	317	175.1*	99	18.64
		317	187	99	10.23

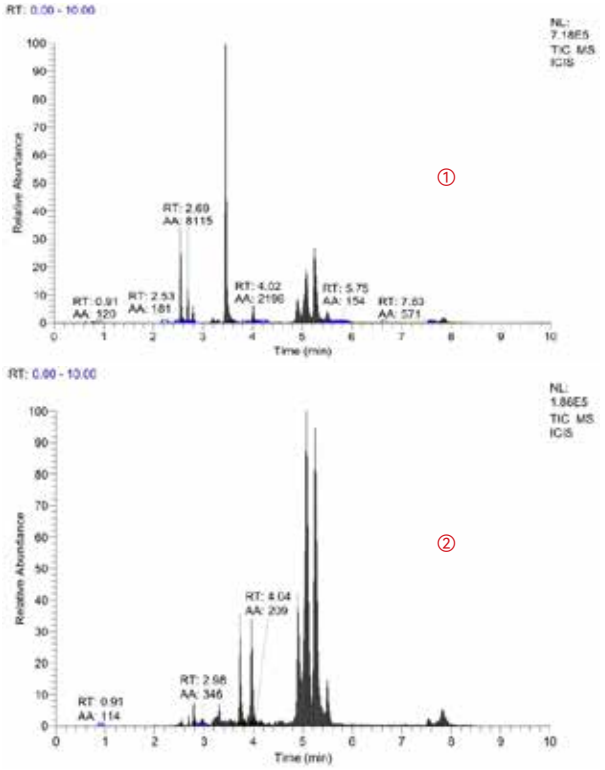
Note: The detection of zearalenone is in negative ion mode (ESI-), and the others are in positive ion mode (ESI+).

Results

Table 2. Spiked Recovery of 10 Mycotoxins in Corn Flour

Targets	Spike (ng/g)	Copure® 302 Multifunctional Purification Plate	
		Average Recovery (%, n=24)	CV/%
Aflatoxin B1	0.5	91.0	4.5
	1.0	90.5	4.8
Aflatoxin B2	0.5	92.3	5.0
	1.0	94.7	5.1
Aflatoxin G1	0.5	92.1	5.6
	1.0	93.5	5.1
Aflatoxin G2	0.5	95.1	6.0
	1.0	94.1	6.5
Fumonisin B1	10	93.2	7.1
	20	94.5	7.5
Fumonisin B2	10	93.5	8.0
	20	94.6	6.5
T-2 Toxin	5	95.2	5.5
	10	94.2	5.5
Vomitoxin	25	91.2	7.1
	50	90.6	6.5
Ochratoxin A	5	90.1	6.7
	10	91.2	5.9
Zearalenone	5	91.3	7.1
	10	90.5	6.5

Based on Table 2, the recovery rates of 10 Mycotoxins are all between 90-110% with RSD less than 10%, which meet the standard of the experimental requirements.



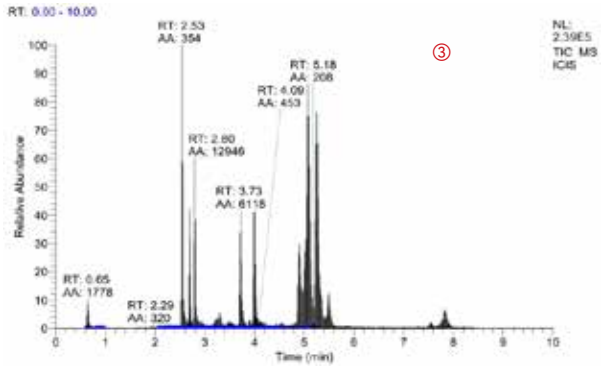
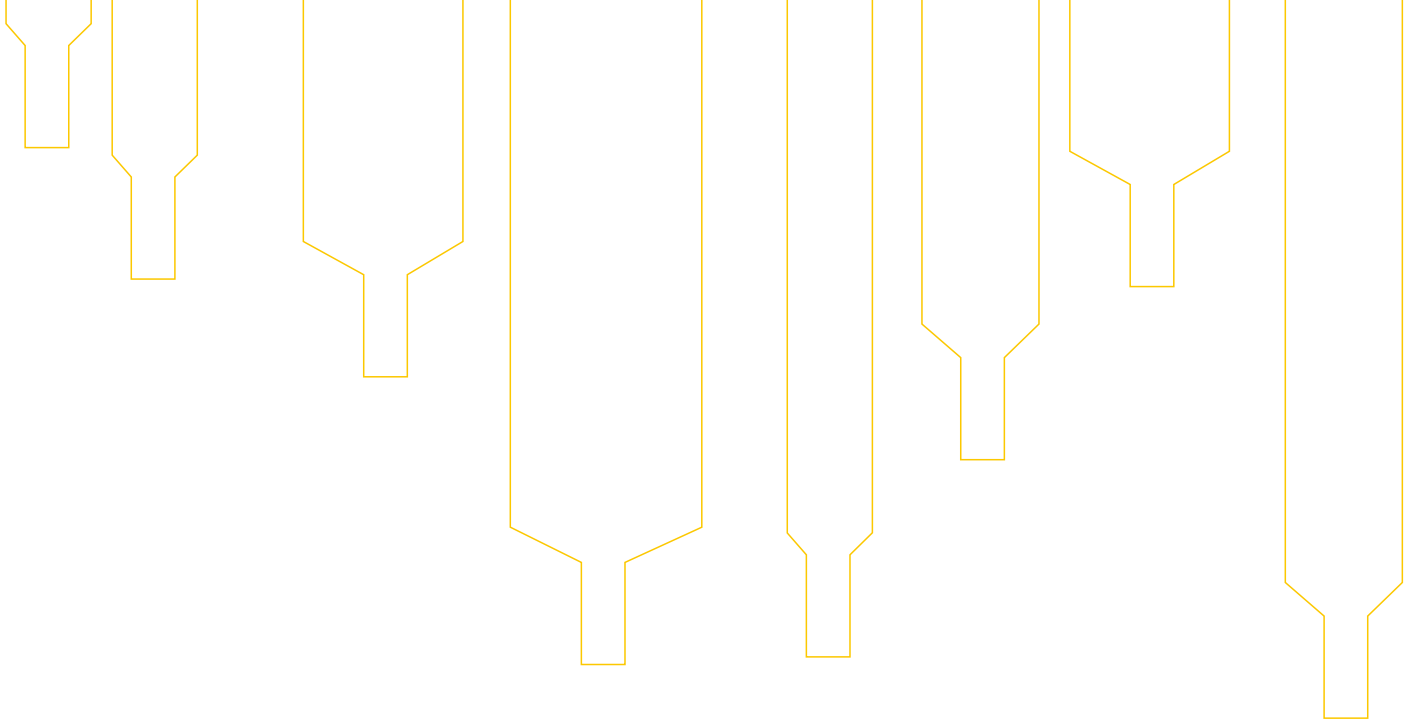


Figure 1. TIC of Corn Flour Sample
① Before Purification
② Solvent Standard Solution
③ Purified by Copure® 302 Multifunctional Purification Plate

Based on Figure 1, the interference of impurities is obviously reduced, and the target signal response value increased. As a result, the quantification is accurate to meet the standard of the experimental requirements.

Ordering Information

Cat. #	Description	Application	Qty.
COAF226-GTL	Copure® 226 Multifunctional Purification Plate	Zearalenone, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF228-GTL	Copure® 228 Multifunctional Purification Plate	Patulin, Aflatoxin B1, B2, G1, G2	1 Pc/Box
COAF224-GTL	Copure® 224 Multifunctional Purification Plate	Zearalenone	1 Pc/Box
COAF223-GTL	Copure® 223 Multifunctional Purification Plate	Aflatoxin M1, M2	1 Pc/Box
COAF229-GTL	Copure® 229 Multifunctional Purification Plate	Ochratoxin	1 Pc/Box
COAF230-GTL	Copure® 230 Multifunctional Purification Plate	Deoxynivalenol	1 Pc/Box
COAF302-GTL	Copure® 302 Multifunctional Purification Plate	Multiple functions	1 Pc/Box



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