Welch Materials

WELCHROM®
QuEChERS

INNOVATIVE | REPRODUCIBLE | RUGGED

WELCH MATERIALS, INC.

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WELCH MATERIALS, INC.

COMPANY PROFILE

Welch Materials is a multinational company specializing in the development and manufacturing of laboratory products. Our extensive range of offerings includes HPLC columns, GC columns, chromatographic packing materials, sample preparation products, protein purification products, laboratory instruments, and general consumables.

Established in August 2003, Welch Materials, Inc. has its headquarters in Songjiang, Shanghai. In addition to our main office, we operate production and research facilities in Jinhua, Zhejiang, and Nanjing, Jiangsu. Furthermore, we have established subsidiary branches in the United States, India, and Canada.

At Welch Materials, Inc., we seamlessly integrate research, production, sales, and service to provide comprehensive laboratory solutions worldwide. Our products have wide-ranging applications in vital industries such as biomedicine, food safety testing, environmental monitoring, and fine chemicals, making a significant contribution to improving people's lives.

In 2018, we proudly obtained the ISO 9001:2015 international quality management system certification, reaffirming our unwavering commitment to maintaining the highest quality standards. Through the implementation of rigorous quality inspection processes and strict adherence to standards, we ensure that each product we produce complies with the most stringent laboratory requirements.


Certificate CN24/00003771

SGS

The management system of

Welch Materials (Zhejiang), Inc.

United Social Credit Code: 91330701581998887G
Business Registration Address: No. 166, Shuanglin South Street, Qibin Community, Wucheng District, Jinhua City, Zhejiang Province, P.R. China
Business Operation Address: No. 166, Shuanglin South Street, Qibin Community, Wucheng District, Jinhua City, Zhejiang Province, P.R. China

has been assessed and certified as meeting the requirements of

ISO 9001:2015

For the following activities

Design and mafacture of liquid chromatographs and liquid chromatographic separation products (fillers)
Manufacture of chromatographic separation products (chromatographic columns and solid phase extraction products)

This certificate is valid from 02 September 2024 until 01 September 2027 and remains valid subject to satisfactory surveillance audits.
Issue 2. Certified since 02 September 2021

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WELCHROM®
QuEChERS

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QuEChERS Method Introduction

QuEChERS(pronounced as "Catchers") is an acronym describing its features of quick, easy, cheap, effective, rugged and safe. This method was first developed by M. Anastassiades et al. (2003) at the U.S. Department of Agriculture.

The QuEChERS method is a pesticide residue analysis technique for multiple target residues in high-moisture matrices (80-95%). It is a streamlined version of SPE with comparable cleanup performance while simplifying the process. QuEChERS method has the advantages of time efficiency, high effectiveness, and cost savings.

In sample matrices such as food and animal products, a wide range of co-extractives—including chlorophyll, lipids, proteins, and other compounds—can significantly interfere with target analyte analysis. The QuEChERS method effectively mitigates these interferences. With a few simple steps. it can complete multi-residue pesticide sample preparation. Nowadays, this method has been widely adopted worldwide. Integrating QuEChERS into your project can significantly reduce the time and labor required for method development and sample preparation.

Comparison between QuEChERS and SPE

Reference Project	Traditional SPE	QuEChERS	Advantages of QuEChERS
Time required to process 6 samples simultaneously	120 min	30 min	Save more than 75% of the time
Solvents used	90 mL	10 mL	Save more than 90% of the solvent
Equipment required	SPE manifold, vacuum pump, rotary evaporator, large container, water bath, etc	Centrifuge, vortexer	Less investment required for equipment

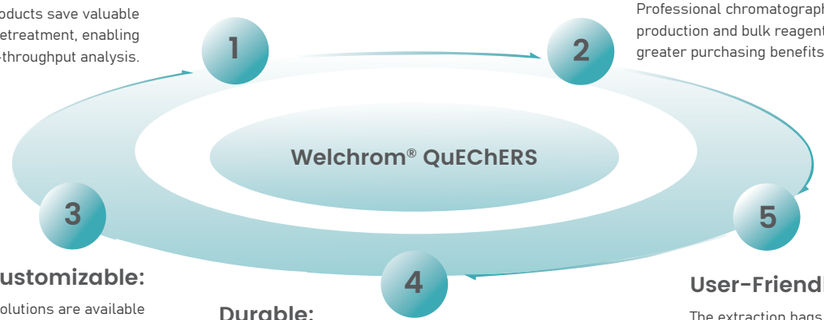
Reasons for Choosing Welchrom® QuEChERS Products

Time-Saving:

Pre-packed products save valuable time for pretreatment, enabling smooth high-throughput analysis.

Cost-Effective:

Professional chromatographic packing material production and bulk reagent sourcing offer greater purchasing benefits.



Customizable:

Tailored solutions are available based on user needs, complemented by professional support from our technical team.

Durable:

Ultra-clean SPE adsorbent and strict quality control ensure reproducibility of results. Anhydrous magnesium sulfate is treated in a muff furnace at high temperatures to enhance dehydration efficiency and remove contaminants that may be introduced in the preparation process.

User-Friendly Packaging:

The extraction bags come in coffee-bag style packaging. And the clean-up tubes are packaged in centrifuge tubes for added convenience during use.

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Evolution of QuEChERS Method

Step 1——Extraction

Original Method, 2003

10 g of homogenized/hydrated sample

10 mL acetonitrile + internal standard, extract by vibration

4 g anhydrous $MgSO_4$ and 1 g NaCl, vigorously shake for 1min and centrifuge at 5000rpm for 5min

AOAC Method, 2007 AOAC 2007.01

15 g of homogenized/hydrated sample

15 mL acetonitrile (containing 1% acetic acid) + internal standard, extract by vibration

6 g anhydrous $MgSO_4$ and 1.5 g NaAc, shake vigorously for 1min and centrifuge at 5000 rpm for 5 min

EN Method, EN 15662

10 g of homogenized/hydrated sample

10 mL acetonitrile + internal standard, extract by vibration

4 g anhydrous $MgSO_4$+1 g NaCl+1 g sodium citrate ($2H_2O$) + 0.5 g disodium citrate ($1.5H_2O$). Shake vigorously for 1 min and centrifuge at 5000 rpm for 5 min

Step 2——Clean up

Take the supernatant

Add into OR clean-up tube, shake for 1 min and centrifuge at 5000 rpm for 1min

GC or LC analysis

Take the supernatant

Add into AOAC clean-up tube, shake for 30 s and centrifuge at 13000 rpm for 5min

GC/MS or LC/MS/MS analysis

Take the supernatant

Add into EN clean-up tube, shake for 30 s and centrifuge at 13000 rpm for 5min

GC/MS or LC/MS/MS analysis

The AOAC and EN methods modified the original OR method by incorporating buffer salts to ensure the efficient extraction of pH-sensitive compounds, reduce the degradation of such sensitive analytes (e.g., pesticides unstable under acidic or alkaline conditions), and broaden the applicability of the method to various food matrices. In the first step of extraction, citrate is used as a buffer to adjust pH to 5-5.5, ensuring sufficient stability for most pesticides that are sensitive to acidic or alkaline environments. For alkaline-sensitive compounds, the stability can be improved by adding a small amount of formic acid after cleanup. For target compounds containing acidic pesticides (phenoxy alcohols), sample injection analysis can be performed directly (skipping the SPE dispersion step), as the acidic group binds to the PSA adsorbent, resulting in reduced recovery rate. For dry samples such as cereals, dried fruits, tobacco, or tea, water should be added before the extraction to reduce the interaction between pesticides and matrices to ensure adequate phase separation. Even high-fat samples such as avocados or olive oil can be analyzed using this method. However, for strongly nonpolar pesticides, the recovery rate is limited to 70% due to their partitioning into the lipid phase. Co-extracted lipids can be removed by freezing or adding a C18E adsorbent.

Nowadays, QuEChERS method has been applied to a growing number of, such as the detection of PAHs in meat products, various veterinary drug residues, dicyandiamide, etc. The development of these applications has greatly reduced the pretreatment workload of chromatographers.

Common Questions for QuEChERS Method

1. How to improve method recovery rate

- Using high-quality homogenization equipment can obtain finer sample particles and improve method precision.
- Using matrix solutions to prepare standard solutions can reduce matrix effect and improve accuracy.
- The addition of isotopic internal standard can effectively monitor recovery rate.
- Maintaining sample moisture content at 80% can effectively improve extraction efficiency.
- Adding extraction bags with buffer salts to alkaline-sensitive pesticides can prevent compound loss.
- Appropriate analytical technique selection should be based on the thermal stability of pesticides. GC or GC/MS can be used for general pesticides, while LC/MS/MS can be used for thermally unstable pesticides.
- Adding toluene in final sample solution can prevent thermal instability losses in the GC liner.
- Adding 1% formic acid in final sample solution can reduce the degradation loss of alkaline-sensitive sensitive compounds.

2. Questions of chromatography

- Adding acetic acid will affect the cleaning effect of PSA adsorbent and leading peak, tailing peak may occur in GC chromatogram. If this happens, a QuEChERS method without acetic acid can be used instead.
- For some samples of complex matrices, if the QuEChERS method cannot effectively purify the samples, multiple methods such as SPE-QuEChERS and QuEChERS-GPC can be used.

Ordering Information of Welchrom® QuEChERS Clean-up Tubes

Method	P/N	Centrifuge Tube	$MgSO_4$	PSA	C18E	GCB	Packing
AOAC General fruits and vegetables	00531-20020	2 mL	150 mg	50 mg	-	-	100 pcs
	00531-20021	15 mL	1200 mg	400 mg			50 pcs
EN General fruits and vegetables	00532-20020	2 mL	150 mg	25 mg	-	-	100 pcs
	00532-20021	15 mL	900 mg	150 mg			50 pcs
AOAC Fruits and vegetables with fats and waxes	00533-20020	2 mL	150 mg	50 mg	50 mg	-	100 pcs
	00533-20021	15 mL	1200 mg	400 mg	400 mg		50 pcs
EN Fruits and vegetables with fats and waxes	00534-20020	2 mL	150 mg	25 mg	25 mg	-	100 pcs
	00534-20021	15 mL	900 mg	150 mg	150 mg	-	50 pcs
AOAC Pigmented fruits and vegetables	00535-20020	2 mL	150 mg	50 mg	-	50 mg	100 pcs
	00535-20021	15 mL	1200 mg	400 mg	-	400 mg	50 pcs
EN Pigmented fruits and vegetables	00536-20020	2 mL	150 mg	25 mg	-	2.5 mg	100 pcs
	00536-20021	15 mL	900 mg	150 mg	-	15 mg	50 pcs
AOAC Fruits and vegetables with pigments and fats	00537-20020	2 mL	150 mg	50 mg	50 mg	50 mg	100 pcs
	00537-20021	15 mL	1200 mg	400 mg	400 mg	400 mg	50 pcs
EN Highly pigmented fruits and vegetables	00538-20020	2 mL	150 mg	25 mg		7.5 mg	100 pcs
	00538-20021	15 mL	900 mg	150 mg		45 mg	50 pcs

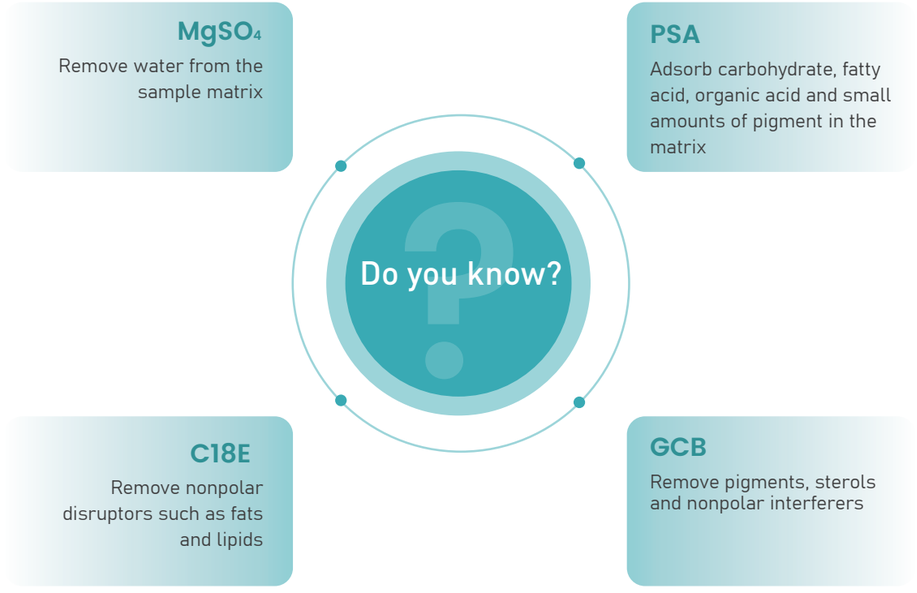
PSA: N-propyl ethylenediamine C18E: Octadecyl bonded silica gel, end-capped GCB: Graphitized carbon black

Note: In this step, the AOAC method specifies sample volumes of 1 mL and 8 mL for 2 mL and 15 mL clean-up tubes, respectively, while the EN method specifies 1 mL and 6 mL.

Ordering Information for Welchrom® QuEChERS Extraction Bags

Method	P/N	$MgSO_4$	NaAcetate	NaCitrate	Disodium Citrate Sesquihydrate	NaCl	Packing
AOAC Method (15 g sample)	00528-20000	6 g	1.5 g				50 pcs
EN Method (10 g sample)	00529-20000	4 g		1 g	0.5 g	1 g	50 pcs
Original Method (10 g sample)	00530-20000	4 g				1.5 g	50 pcs
EN Method (15 g sample)	00529-25000	6 g		1.5 g	0.75 g	1 g	50 pcs
Original Method (15 g sample)	00530-25000	6 g				1.5 g	50 pcs

Usage of various adsorbents in removing major interferences from the matrices



How to Select the Proper Clean-up Tube According to the Sample?

Sample Characteristics			
General fruit and vegetable samples, light color samples.	Samples containing small amounts of chlorophyll and carotenoids, colored samples.	Samples containing high pigment and fat, dark color samples.	Samples with >1% fat and lipid content
Representative fruit: Apple, pear, apricot, cherry, western plum, nectarine, peach, plum, strawberry, pineapple, banana, dried fig, melon, kiwi, mango, papaya, etc.	Representative fruit: Blackberries, blueberries, raisins, elderberries, raspberries, mango, papaya, etc.	Representative fruit: Blackberries, blueberries, seedless fruit, raspberries, red grapes, raisins, mango, papaya, avocado, olives, etc.	Representative fruit: Orange juice, grapefruit, lemon, orange, orange peel, nectarine, orange, banana, avocado, olive, etc.
Representative vegetables: Beet, carrot, celery, horseradish, radish, potato, garlic, onion, eggplant, cucumber, sweet green pepper, tomato, courgette, broccoli, cabbage, asparagus, beans, etc.	Representative vegetables: Green onion, leek, chives, sweet green pepper, red sweet pepper, pumpkin, broccoli, kale, red cabbage, lettuce, coriander, spinach, mint, watercress, large yellow leaves, fresh beans, tea, coffee beans, etc.	Representative vegetables: Green onion, chives, sweet green pepper, red sweet pepper, kale, lettuce, endive, water celery, mint, parsley, cilantro, arugula, spinach, fresh beans, coffee beans, tea leaves, etc.	Representative vegetables: Garlic, onion, wheat, corn, rice, grain, flour, etc.

Welchrom® QuEChERS Extraction Bags			
Citrate or sodium acetate buffer	Citrate or sodium acetate buffer	Citric acid buffer	Citric acid buffer

Welchrom® DQuEChERS Clean-up Tubes			
00531-20020	00535-20020	-	-
00531-20021	00535-20021	00537-20021	00533-20021
00532-20020	00536-20020	00537-20020	00533-20020
00532-20021	00536-20021	-	-

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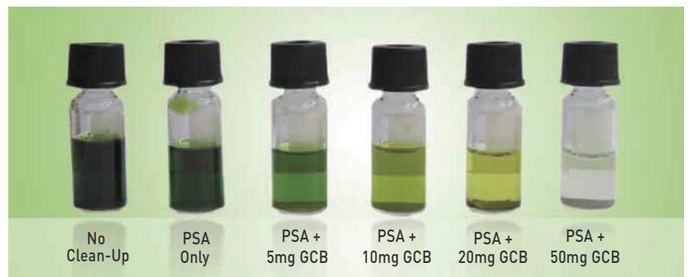
Clean-up Efficiency of Different Concentrations of Welchrom® GraphiCarb in Spinach Extract



Graphitized carbon has a strong adsorption for planar pesticides.

Solutions:

- Reduce the amount of graphitized carbon; this also reduces the adsorption of pigment.
- Optimize the recovery rate by adding a proper amount of toluene (e.g., acetonitrile/toluene = 8/3).

More QuEChERS Customized Products

P/N	Product	Pack	Usage
00551-20000	Extraction bag	5.0 g Na_2SO_4, 50 pcs/pk	Customized product
005PM-055-50	Extraction bag	8.0 g Na_2SO_4, 2.0 g NaCl, 50 pcs/pk	Customized product
005PM-059-50	Extraction bag	4.0 g Na_2SO_4, 1.0 g NaCl, 50 pcs/pk	Tetracycline
005PM-064-50	Extraction bag	5.0 g $MgSO_4$, 50 pcs/pk	Customized product
005PM-018-100	Extraction bag	4 g $MgSO_4$, 100 pcs/pk	Determination of 9 bactericide residues in fruits
00553-20020	Clean-up tube	2 mL, 150 mg $MgSO_4$, 25 mg C18E, 100 pcs/pk	Customized product
00565-20020	Clean-up tube	2 mL, 100 mg $MgSO_4$, 50 mg PSA, 100 mg C18E, 100 pcs/pk	Shanghai Local standard DB 31/2010-2012 (Hot pot base)
00581-20021	Clean-up tube	15 mL 900 mg $MgSO_4$, 300 mg PSA, 300 mg C18E, 300 mg Silica, 90 mg GCB, 50 pcs/pk	General Rule 2341 of the 2020 edition of Chinese Pharmacopoeia Determination of pesticide residues
00588-20020	Clean-up tube	2 mL, 200 mg $MgSO_4$, 50 mg PSA, 100 pcs/pk	Determination of pesticide residues
00590-20020	Clean-up tube	2 mL, 50 mg PSA, 50 mg GCB, 100 pcs/pk	Customized product
00592-20020	Clean-up tube	2 mL, 150 mg $MgSO_4$, 50 mg PSA, 25 mg C18E, 100 pcs/pk	Customized product
00592-20021	Clean-up tube	15 mL, 900 mg $MgSO_4$, 300 mg PSA, 150 mg C18E, 50 pcs/pk	Customized product

More QuEChERS Customized Products

P/N	Product	Pack	Usage
00592-20022	Clean-up tube	50 mL, 900 mg $MgSO_4$, 300 mg PSA, 150 mg C18E, 25 pcs/pk	Customized product
00596-20021	Clean-up tube	15 mL, 900 mg Na_2SO_4, 50 mg PSA, 150 mg C18E, 50 pcs/pk	Used for veterinary drug detection in food
00597-20021	Clean-up tube	15 mL, 900 mg $MgSO_4$, 300 mg PSA, 150 mg GCB, 50 pcs/pk	Customized product
00597-20022	Clean-up tube	50 mL, 900 mg $MgSO_4$, 300 mg PSA, 150 mg GCB, 25 pcs/pk	Customized product
00598-20020	Clean-up tube	2 mL, 150 mg $MgSO_4$, 50 mg PSA, 50 mg C18E, 7.5 mg GCB, for all food types, 100 pcs/pk	Used for all food types
00598-20021	Clean-up tube	15 mL, 1200 mg $MgSO_4$, 400 mg PSA, 400 mg C18E, 45 mg GCB, for all food types, 50 pcs/pk	Used in fruits and vegetables with pigments and fats
005PM-001-50	Clean-up tube	5 mL, 50 mg $MgSO_4$, 50 mg PSA, 50 mg C18E, 100 pcs/pk	Customized product
005PM-002-50	Clean-up tube	5 mL, 50 mg $MgSO_4$, 50 mg PSA, 50 mg C18E, 50 mg GCB, 50 pcs/pk	Customized product
005PM-008-50	Clean-up tube	15 mL, 400 mg $MgSO_4$, 100 mg PSA, 50 mg C18E, 20 mg GCB, 50 pcs/pk	SN/T 3235-2012 (Pork liver and animal tissue sample)
005PM-009-50	Clean-up tube	15 mL, 600 mg $MgSO_4$, 100 mg PSA,40 mg C18E, 50 pcs/pk	SN/T 3235-2012 (Milk and aquatic products sample)
005PM-014-50	Clean-up tube	15 mL, 500mg C18E, 250 mg PSA, 250 mg GCB, 50 pcs/pk	Customized product
005PM-017-100	Clean-up tube	2 mL, 400 mg PSA, 100 pcs/pk	Determination of 9 bactericide residues in fruits
005PM-023-50	Clean-up tube	15 mL, 300 mg $MgSO_4$, 100 mg PSA,100 mg C18E, 50 pcs/pk	NY/T 1380-2007 Determination of 51 kinds of pesticide residues in vegetables and fruits.
005PM-024-50	Clean-up tube	5 mL, 150 mg $MgSO_4$, 75 mg PSA, 50 pcs/pk	Custom products
005PM-025-50	Clean-up tube	15 mL, 800 mg Na_2SO_4, 800 mg PSA, 400 mg C18E, 50 pcs/pk	Detection of anthraquinone in tea leaf
005PM-026-50	Clean-up tube	15 mL, 250 mg Na_2SO_4, 400 mg PSA, 50 pcs/pk	Detection of pesticide residues in vegetables
005PM-027-50	Clean-up tube	15 mL, 250 mg Na_2SO_4, 400 mg PSA, 250 mg C18E, 50 pcs/pk	Detection of pesticide residues in vegetables
005PM-028-25	Clean-up tube	50 mL, 1500 mg Na_2SO_4, 500 mg PSA, 25 pcs/pk	Determination of organophosphorus pesticide residues in plant Foods - National Food Risk Manual for Disease Control
005PM-029-25	Clean-up tube	50 mL, 1500 mg Na_2SO_4, 500 mg PSA, 250 mg C18E, 25 pcs/pk	Determination of organophosphorus pesticide residues in plant Foods - National Food Risk Manual for Disease Control
005PM-031-50	Clean-up tube	15 mL, 300 mg $MgSO_4$, 100 mg PSA, 100 mg GCB, 50 pcs/pk	Customized product
005PM-038-100	Clean-up tube	2 mL, 50 mg PSA, 50 mg C18E, 100 pcs/pk	GB 23200.58-2016 Determination of sulfonamide chloride residues in food

P/N	Product	Pack	Usage
005PM-039-50	Clean-up tube	15 mL, 300 mg $MgSO_4$, 100 mg C18E, 50 pcs/pk	Customized product
005PM-044-50	Clean-up tube	15 mL, 900 mg $MgSO_4$, 150 mg PSA, 150 mg C18E, 150 mg GCB, 50 pcs/pk	Customized product
005PM-045-50	Clean-up tube	15 mL, 885 mg $MgSO_4$, 150 mg PSA, 15 mg GCB, 50 pcs/pk	GB 23200.113-2018 208 kinds of agricultural residues
005PM-046-50	Clean-up tube	15 mL, 1200 mg $MgSO_4$, 400 mg PSA, 200 mg GCB, 400 mg C18E, 50 pcs/pk	GB 23200.113-2018 208 kinds of agricultural residues
005PM-047-50	Clean-up tube	15 mL, 1500 mg $MgSO_4$, 500 mg PSA, 50 pcs/pk	Customized product
005PM-052-100	Clean-up tube	2 mL, 150 mg $MgSO_4$, 40 mg PSA, 15 mg GCB, 100 pcs/pk	GB 23200.110-2018
005PM-053-100	Clean-up tube	2 mL, 150 mg $MgSO_4$, 50 mg C18E, 100 pcs/pk	GB 23200.110-2018
005PM-048-50	Clean-up tube	15 mL, 900 mg $MgSO_4$, 100 mg C18E, 100 mg PSA, 50 pcs/pk	GB5009.265-2016 PAH

Note: More customized products please contact Welch or your local distributors.

QuEChERS-EN Method

Determination of Organophosphorus Residues in Cucumber

1. Range of Application

Applicable for the determination of 7 kinds of organophosphorus pesticide residues (dichlorvos, cadusafos, diazinon, chlorpyrifos, methyl parathion, fenitrothion, triazophos) in cucumber.

Reference standard: BS EN 15662:2008 Foods of Plant Origin-Determination of Pesticide Residues Using GC-MS and/or LC-MS Following Acetonitrile Exraction/Partitioning and Clean-up by Dispersive SPE-QuEChERS Method.

2. Extraction and Cleanup

Weigh 10 g of the sample (pre-homogenized into slurry and mixed) in a 50 ml centrifuge tube with stopper and add 10 mL acetonitrile. Add a 00529-20000 extraction bag and shake vigorously for 1 min. Centrifuge at 4200 r/min for 5 min, then transfer 6 mL of the supernatant into a 00532-20021 clean-up tube. Vortex for 1 min and centrifuge at 4200 r/min for 5 min. Finally filter the supernatant through a 0.22 μm syringe filter and reserve it for GC analysis.

3. Chromatographic Condition

Column	WM-1701, 30 m × 0.32 mm × 0.25 μm
Inlet Temp.	250 °C
Detector (FPD) Temp.	250 °C
Carrier Gas	Nitrogen, make-up gas flow rate: 30 mL/min, hydrogen flow rate: 75 mL/min, air flow rate: 90 mL/min.
Injection	Splitless injection
Carrier Gas Flow Rate	2.0 mL/min
Injection Volume	2 μL
Temperature Program	Initial 60 °C (hold for 5 min), raise to 270 °C at a rate of 10 °C/min and hold for 15 min

4. Chromatograms and Result of Spike Recovery Rate

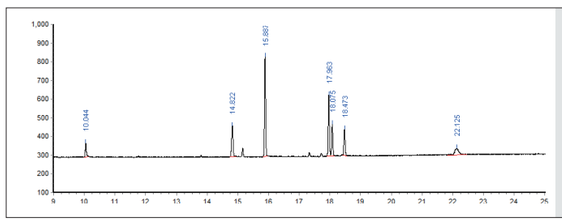


Fig.1. Chromatogram of 7 kinds of organophosphorus standard substance, 0.1 mg/L

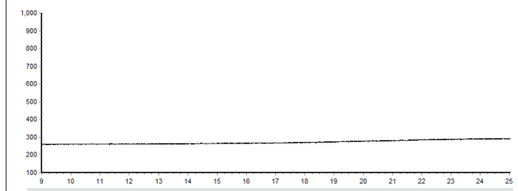


Fig.2. Blank chromatogram of cucumber sample

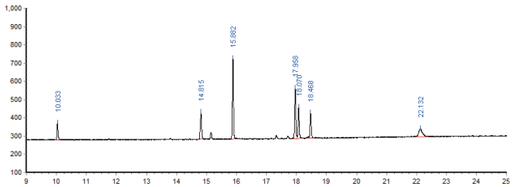


Fig.3. Chromatogram of cucumber spiked sample (100.0 ng/g)

Classification	Spike Level ng/g	Recovery Rate/%	RSD(n=2)/%
Dichlorvos	100	122.98%	3.31
Cadusafos		82.60%	0.83
Diazinon		83.34%	0.22
Chlorpyrifos		87.45%	3.78
Methyl parathion		106.18%	3.85
Fenitrothion		108.76%	5.40
Triazophos		109.81%	2.88

Tab 1: Spike Recovery

Ordering Information of Related Products

P/N	Product	Description
00529-20000	Extraction bag	EN method, 4 g anhydrous magnesium sulfate, 1 g sodium chloride, 1 g sodium citrate, 0.5 g disodium citrate sesquihydrate, 50 pcs/pk
00532-20021	Clean-up tube	EN method, for general fruits and vegetables, 15 ml, 900 mg $MgSO_4$, 150 mg PSA, 50 pcs/pk
03907-32001	GC column	WM-1701, 30 m × 0.32 mm × 0.25 μm

QuEChERS
Determination of Pyrethroid Residues in Vegetables

1. Range of Application

Applicable for the determination of 8 pyrethroid pesticide residues (permethrin, deltamethrin, cypermethrin, fenvalerate, bifenthrin, cyfluthrin, λ-cyhalothrin, and fenpropathrin) in vegetables(enoki mushroom and green bean samples were used in this experiment).

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

GB23200.113-2018: National Standard for Food Safety-Determination of 208 Pesticides and Their Metabolites Residues in Plant-derived Food by GC/MS

2. Extraction and Cleanup

Weigh 15 g of the sample (pre-homogenized into slurry and mixed) a in 50 ml centrifuge tube with stopper and add 15 mL of acetonitrile(containing 1% acetic acid). Add a 00528-20000 extraction bag and shake vigorously for 1 min. Centrifuge at 6000 r/min for 5 min. Transfer 7 mL of the supernatant into a 005PM-046-50 clean-up tube (with 3 mL of toluene added in advance), vortex for 1 min and centrifuge at 6000 r/min for 5 min, then filter supernatant through a 0.22 μm syringe filter and reserve for GC analysis.

3. Chromatographic Condition

Column	WM-5MS, 30 m × 0.25 mm × 0.25 μm
Inlet Temp.	220 °C
Detector (ECD) Temp.	300 °C
Carrier Gas	Nitrogen, make-up gas flow rate: 60 ml/min
Injection	Split injection, split ratio 10:1
Carrier Gas Flow Rate	1.6 mL/min
Injection Volume	5 μL
Temperature Program	Initial 180 °C (hold for 2 min); raise to 200 °C at 4 °C/min, hold for 2 min; raise to 230 °C at 10 °C/min, hold for 2 min; raise to 260 °C at 2 °C/min, hold for 8.5 min; raise to 270 °C at 50 °C/min, hold for 2 min.

4. Chromatograms and Result of Spike Recovery Rate

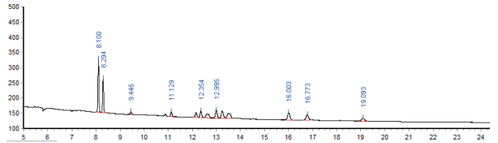


Fig.1. Chromatogram of mixed standards (8 kinds of pyrethroid pesticide standards), 0.1 mg/L

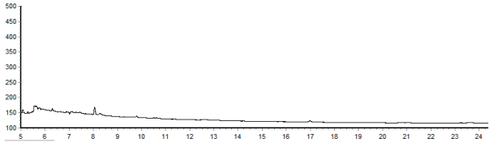


Fig.2. Blank chromatogram of enoki mushroom sample

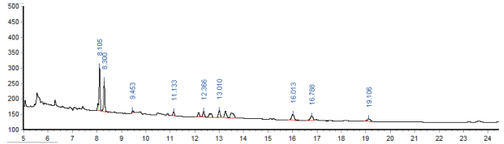


Fig.3. Chromatogram of enoki mushroom spiked sample (100.0 ng/g)

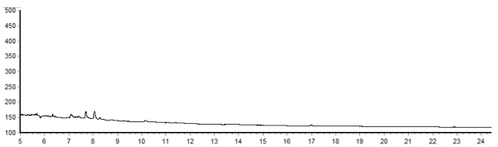


Fig.4. Blank chromatogram of green bean sample

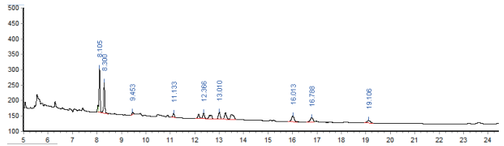


Fig.5. Chromatogram of green bean spiked sample (100.0 ng/g)

Classification	Spike Level ng/g	Enoki mushroom		Green Bean	
		Recovery Rate/%	RSD(n=2)/%	Recovery Rate/%	RSD(n=2)/%
Bifenthrin	100	80.87%	0.54	99.30%	1.24
Fenpropathrin		96.25%	0.70	105.26%	3.78
λ-Cyhalothrin	1000	96.32%	1.46	106.26%	2.30
Permethrin	100	81.81%	1.22	102.12%	3.45
Cyfluthrin		96.12%	2.23	116.58%	0.74
Cypermethrin		93.22%	0.28	111.25%	3.98
Fenvalerate		83.45%	0.55	111.28%	2.71
		78.65%	1.87	98.00%	4.97
Deltamethrin		86.31%	2.51	122.03%	4.84

Tab.1 : Spike Recovery

Ordering Information of Related Products

P/N	Product	Description
03904-22001	GC column	WM-5MS, 30 m × 0.25 mm × 0.25 µm
00528-20000	Extraction bag	AOAC method, 6 g anhydrous magnesium sulfate, 1.5 g Sodium acetate, 50 pcs/pk.
005PM-046-50	Clean-up tube	15 mL, 1200 mg $MgSO_4$, 400 mg PSA, 200 mg GCB, 400 mg C18E, 50 pcs/pk.

QuEChERS-EN Method

Determination of Organochlorine and Pyrethroid Residues in Rice

1. Range of Application

Applicable for the determination of 11 organochlorines [hexachlorobenzene, β-hexachlorocyclohexane, α-hexachlorocyclohexane, γ-hexachlorocyclohexane (lindane), heptachlor, δ-hexachlorocyclohexane, cis-heptachlor exo-epoxide (isomer B), p,p'-DDE, endrin, o,p' - DDT, and p,p'- DDT] and 8 pyrethroids (permethrin, deltamethrin, cypermethrin, fenvalerate, bifenthrin, cyfluthrin, λ-cyhalothrin, and fenpropathrin) pesticide residues in rice.

Reference standard: BS EN 15662:2008 Foods of Plant Origin-Determination of Pesticide Residues Using GC-MS or LC-MS/MS following acetonitrile exraction/partitioning and clean-up by dispersive SPE-QuEChERS-method

2. Extraction and Cleanup

Weigh 10 g of the sample (pre-homogenized into slurry and mixed) into a 50 mL centrifuge tube with stopper and add 10 mL acetonitrile. Add a 00529-20000 extraction bag and shake vigorously for 1 min. Centrifuge at 4200 r/min for 5 min, transfer 6 mL of the supernatant into a 00534-20021 clean-up tube. Vortex for 1 min and centrifuge at 4200 r/min for 5 min, then filter the supernatant with a 0.22 µm syringe filter and reserve for GC analysis.

3. Chromatographic Condition

Column	WM-5MS, 30 m × 0.25 mm × 0.25 µm
Inlet Temp.	220 °C
Detector (ECD) Temp.	300 °C
Carrier Gas	Nitrogen, make-up gas flow rate: 60 mL/min
Injection	Split injection, split ratio 10:1
Carrier Gas Flow Rate	1.6 mL/min
Injection Volume	2 µL
Temperature Program	Initial 180 °C (hold for 2 min); raise to 200 °C at 4 °C/min, hold for 2 min; raise to 230 °C at 10 °C/min, hold for 2 min; raise to 260 °C at 2 °C/min, hold for 8.5 min; raise to 270 °C at 50 °C/min, hold for 2 min.

4. Chromatograms and Result of Spike Recovery Rate

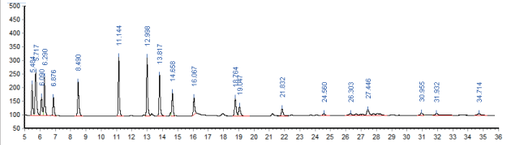


Fig.1. Chromatogram of organochlorine and pyrethroid pesticides mixed standards, 0.1 mg/L

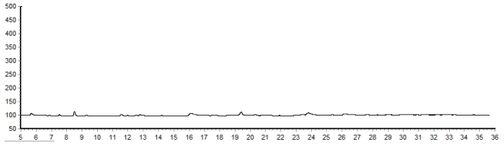


Fig.2. Blank chromatogram of rice sample

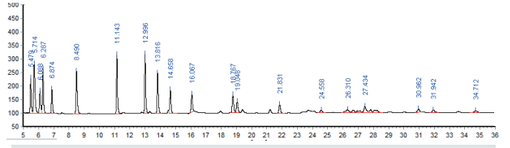


Fig.3. Chromatogram of spiked rice sample (100.0 ng/g)

Classification	Spike Level ng/g	Recovery Rate/%	RSD(n=2)/%
Hexachlorobenzene	100	102.32%	2.26
α-Hexachlorocyclohexane		109.76%	1.68
β-Hexachlorocyclohexane		117.73%	3.80
γ-Hexachlorocyclohexane		109.55%	1.94
Heptachlor		116.02%	0.78
δ-Hexachlorocyclohexane		119.33%	0.93
Heptachlor epoxide		98.76%	1.29
p, p'- DDE		95.37%	0.77
Endrin		100.57%	1.93
o, p'- DDT		100.02%	1.02
p, p'- DDT		106.05%	4.10
Bifenthrin		97.80%	2.65
Fenpropathrin		102.01%	4.42
λ-Cyhalothrin	1000	118.07%	3.19
Permethrin	100	103.27%	0.09
Cyfluthrin		116.25%	1.71
Cypermethrin		120.05%	2.04
Fenvalerate		135.21%	4.82
		127.79%	5.44
Deltamethrin		120.12%	0.74

Tab 1: Spike Recovery

Ordering Information of Related Products

P/N	Product	Description
03904-22001	GC column	WM-5MS 30 m × 0.25 mm × 0.25 μm
00534-20021	Clean-up tube	15 mL, EN method, 900 mg $MgSO_4$, 150 mg PSA, 150 mg C18E, for fruits and vegetables with fats and waxes, 50 pcs/pk
00529-20000	Extraction bag	EN method, 4 g $MgSO_4$, 1 g NaCl, 1 g sodium citrate, 0.5 g disodium citrate sesquihydrate, 50 pcs/pk

QuEChERS-AOAC Method

Determination of Organochlorine and Pyrethroid Residues in Cucumber

1. Range of Application

Applicable for the determination of 11 kinds of organochlorine (hexachlorobenzene, β-hexachlorocyclohexane, α-hexachlorocyclohexane, γ-hexachlorocyclohexane (lindane), heptachlor, δ-hexachlorocyclohexane, cis-heptachlor exo-epoxide (isomer B), p,p'-DDE, endrin, o,p' - DDT, and p,p'- DDT) and 8 kinds of pyrethroid pesticide residues (permethrin, deltamethrin, cypermethrin, fenvalerate, bifenthrin, cyfluthrin, λ-cyhalothrin, and fenpropathrin) in cucumber.

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

2. Extraction and Cleanup

Weigh 15 g of the sample (pre-homogenized into slurry and mixed) in a 50 mL centrifuge tube with stopper and add 15 mL of acetonitrile (contain 1% acetic acid). Add a 00528-20000 extraction bag and shake vigorously for 1 min. Centrifuge at 6000 r/min for 5 min, then transfer 8 mL of the supernatant into a 00533-20021 clean-up tube. Vortex for 1 min and centrifuge at 6000 r/min for 5 min, then filter the supernatant with a 0.22 μm syringe filter and reserve for GC analysis.

3. Chromatographic Condition

Column	WM-5MS, 30 m × 0.25 mm × 0.25 μm
Inlet Temp.	220 °C
Detector (ECD) Temp.	300 °C
Carrier Gas	Nitrogen, make-up gas flow rate: 60 mL/min
Injection	Split injection, split ratio 10:1
Carrier Gas Flow Rate	1.6 mL/min
Injection Volume	2 μL
Temperature Program	Initial 180 °C (hold for 2 min); raise to 200 °C at 4 °C/min, hold for 2 min; raise to 230 °C at 10 °C/min, hold for 2 min; raise to 260 °C at 2 °C/min, hold for 8.5 min; raise to 270 °C at 50 °C/min, hold for 2 min.

4. Chromatogram or Result of Spike Recovery Rate

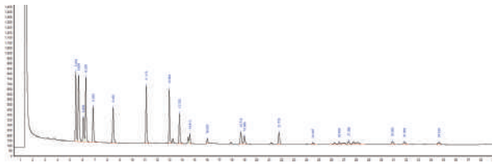


Fig.1. Chromatogram of mixed standards (organochlorine and pyrethroid pesticides standards), 0.1 mg/L

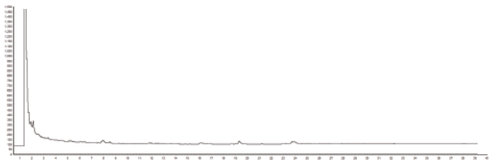


Fig.2. Blank chromatogram of cucumber sample

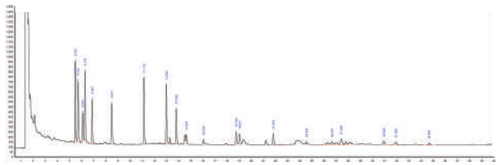


Fig.3. Chromatogram of spiked cucumber sample (100.0 ng/g)

Classification	Spike Level ng/g	Recovery Rate/%	RSD(n=2)/%
Hexachlorobenzene	100	113.29%	3.01
α-Hexachlorocyclohexane		86.17%	0.56
β-Hexachlorocyclohexane		107.54%	4.05
γ-Hexachlorocyclohexane		100.12%	3.37
Heptachlor		114.34%	1.79
δ-Hexachlorocyclohexane		113.83%	1.10
Heptachlor epoxide		111.99%	0.75
p, p '- DDE		106.34%	1.28
Endrin		116.70%	0.40
o, p '- DDT		100.63%	2.38
p, p '- DDT		107.16%	1.86
Bifenthrin		111.68%	0.30
Fenpropathrin		122.75%	0.88
λ-Cyhalothrin	1000	93.98%	0.03
Permethrin	100	125.46%	5.03
Cyfluthrin		129.82%	1.68
Cypermethrin		118.72%	4.09
Fenvalerate		103.71%	3.00
		96.70%	5.18
Deltamethrin		72.12%	5.30

Tab 1: Spike Recovery

Ordering Information of Related Products

P/N	Product	Description
03904-22001	GC column	WM-5MS 30 m × 0.25 mm × 0.25 μm
00528-20000	Extraction bag	AOAC method, 6 g $MgSO_4$, 1.5 g sodium acetate, 50 pcs/pk
00533-20021	Clean-up tube	15 mL, AOAC method, 1200 mg $MgSO_4$, 400 mg PSA, 400 mg C18E, for fruits and vegetables with fats and waxes, 50 pcs/pk

QuEChERS-AOAC Method

Determination of Organochlorine and Pyrethroid Residues in Rice

1. Range of Application

Applicable for the determination of pesticide residues in fruits, vegetables and grains with much fat or wax content.

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

2. Extraction and Cleanup

Weigh 15 g of the sample (comminuted and mixed) into 50 mL centrifuge tube with stopper and add 15 mL acetonitrile(contain 1% acetic acid). Add a 00528-20000 extraction bag and shake vigorously for 1 min. Centrifuge at 6000 r/min for 5 min, transfer 6 mL of the supernatant into a 00533-20021 clean-up tube. Vortex for 1 min and centrifuge at 6000 r/min for 5 min, then filter the supernatant with a 0.22 μm syringeand reserve for GC analysis.

3. Chromatographic Condition

Column	WM-5MS, 30 m × 0.25 mm × 0.25 μm
Inlet Temp.	220 °C
Detector (ECD) Temp.	300 °C
Carrier Gas	Nitrogen, make-up gas flow rate: 60 mL/min
Injection	Split injection, split ratio 10:1
Carrier Gas Flow Rate	1.6 mL/min
Injection Volume	2 μL
Temperature Program	Initial 180 °C (hold for 2 min); raise to 200 °C at 4 °C/min, hold for 2 min; raise to 230 °C at 10 °C/min, hold for 2 min; raise to 260 °C at 2 °C/min, hold for 8.5 min; raise to 270 °C at 50 °C/min, hold for 2 min.

4. Chromatogram or Result of Spike Recovery Rate

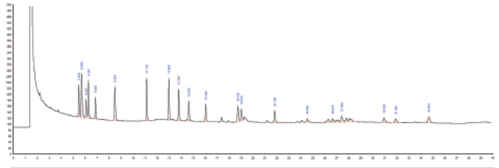


Fig.1. Chromatogram of mixed standards (organochlorine and pyrethroid pesticides standards), 0.1 mg/L

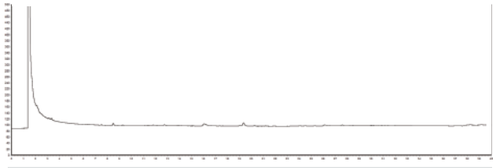


Fig.2. Blank chromatogram of rice sample

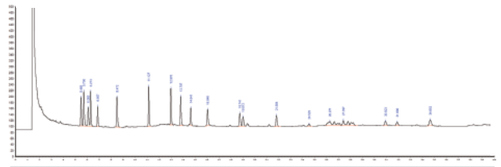


Fig.3. Chromatogram of spiked rice sample (100.0 ng/g)

Classification	Spike Level ng/g	Recovery Rate/%	RSD(n=2)/%
Hexachlorobenzene	100	84.58%	3.38
α-Hexachlorocyclohexane		76.68%	2.53
β-Hexachlorocyclohexane		99.02%	2.60
γ-Hexachlorocyclohexane		88.41%	3.73
Heptachlor		89.89%	1.02
δ-Hexachlorocyclohexane		88.34%	2.20
Heptachlor epoxide		92.22%	1.54
p, p '- DDE		86.38%	0.38
Endrin		93.98%	0.50
o, p '- DDT		88.81%	1.33
p, p '- DDT		108.80%	3.30
Bifenthrin		86.30%	0.70
Fenpropathrin		86.55%	0.21

Classification	Spike Level ng/g	Recovery Rate/%	RSD(n=2)/%
λ-Cyhalothrin	1000	94.32%	0.12
Permethrin	100	75.31%	0.38
Cyfluthrin		89.49%	2.98
Cypermethrin		84.36%	5.16
Fenvalerate		96.63%	0.92
		89.23%	1.50
Deltamethrin		101.67%	5.82

Tab 1: Spike Recovery

Ordering Information of Related Products

P/N	Product	Description
03904-22001	GC column	WM-5MS 30 m × 0.25 mm × 0.25 μm
00528-20000	Clean-up tube	AOAC method, 6 g anhydrous magnesium sulfate, 1.5 g Na-acetate, 50 pcs/pk
00533-20021	Extraction bag	15 mL, AOAC method, 1200 mg $MgSO_4$, 400 mg PSA, 400 mg C18E, for fruits and vegetables with fats and waxes, 50 pcs/pk

QuEChERS-AOAC Method

Determination of Organochlorine and Pyrethroid Residues in Cabbage

1. Range of Application

Applicable for the determination of pesticide residues in fruits, vegetables and grains with more complex pigments.

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

2. Extraction and Cleanup

Weight 15 g of sample (pre-homogenized and mixed) into a 50 mL centrifuge tube with stopper and add 15 mL of acetonitrile containing 1% acetic acid. Add a 00528-20000 extraction bag and shake vigorously for 1 min. Centrifuge at 6000 r/min for 5 min. Transfer 7 mL of the supernatant into a 00537-20021 clean-up tube (in which 3 mL of toluene was added in advance). Vortex for 1 min and centrifuge at 6000 r/min for 5 min, then filter the supernatant with a 0.22 μm syringe filter and reserve for GC analysis.

3. Chromatographic Condition

Column	WM-5MS, 30 m × 0.25 mm × 0.25 μm
Inlet Temp.	220 °C
Detector (ECD) Temp.	300 °C
Carrier Gas	Nitrogen, make-up gas flow rate: 60 mL/min
Injection	Split injection, split ratio 10:1
Carrier Gas Flow Rate	1.6 mL/min
Injection Volume	2 μL
Temperature Program	Initial 180 °C (hold for 2 min); raise to 200 °C at 4 °C/min, hold for 2 min; raise to 230 °C at 10 °C/min, hold for 2 mn; raise to 260 °C at 2 °C/min, hold for 8.5 min; raise to 270 °C at 50 °C/min, hold for 2 min.

4. Chromatogram or Result of Spike Recovery Rate

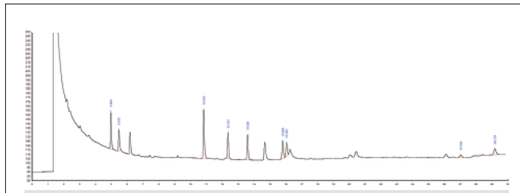


Fig.1. Chromatogram of mixed standards(organochlorine and pyrethroid standards, 0.1 mg/L

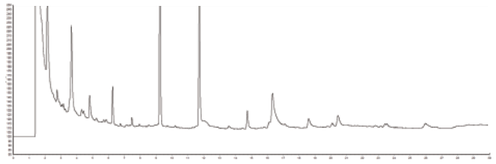


Fig.2. Blank chromatogram of cabbage sample

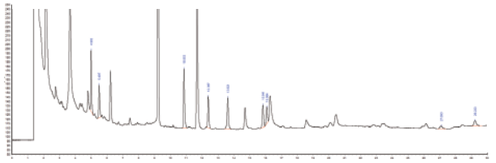


Fig.3. Chromatogram of spiked cabbage sample (100.0 ng/g)

Classification	Spike Level ng/g	Recovery Rate/%	RSD(n=2)/%
α-Hexachlorocyclohexane	100	140.07%	3.49
β-Hexachlorocyclohexane		125.65%	1.78
p, p '- DDE		118.83%	1.63
o, p '- DDT		116.78%	2.72
p, p '- DDT		129.07%	7.43
Bifenthrin		122.19%	1.52
Fenpropathrin		114.79%	0.86
Fenvalerate		114.46%	6.56
Deltamethrin		90.17%	2.33

Tab 1: Spike Recovery

Ordering Information of Related Products

P/N	Product	Description
03904-22001	GC column	WM-5MS 30 m × 0.25 mm × 0.25 μm
00528-20000	Extraction bag	AOAC method, 6 g anhydrous magnesium sulfate, 1.5 g sodium acetate, 50 pcs/pk
00537-20021	Clean-up tube	15 mL, AOAC method, 1200 mg $MgSO_4$, 400 mg PSA, 400 mg C18E, 400 mg GCB, used for fruits and vegetables with pigments and fats, 50 pcs/pk

QuEChERS Method

Determination of Papaverine, Morphine, Narcodine, Codeine and Thebaine in Hot Pot Foods

1. Range of Application

This standard is applicable to the determination of papaverine, morphine, narcodine, codeine and thebaine in hot pot food such as hot pot sauces, soup bases, flavoring oils and solid flavoring powders.

2. Standard

DB 31/2010-2012 Local Standard for Food Safety-Determination of Papaverine, Morphine, Narcodine, Codeine and Thebaine in Hotpot Food by LC-MS/MS.

3. Principles

Dispense samples evenly with water or hydrochloric acid solution and extract with acetonitrile. After salting out, acetonitrile extract should be purified with bonded silicon SPE adsorbent, centrifuged, detected by liquid chromatography-tandem mass spectrometer, then quantified by external standard method.

4. Preparation of Reagents

4.1 Ammonium formate solution (2 mmol/L) : Accurately weigh 0.252 g of ammonium formate , dissolve in appropriate amount of water and dilute to 2 L, mix well.

4.2 Acetonitrile containing 0.1% formic acid: take 1 mL of formic acid, add acetonitrile and dilute to 1 L, shake well, then filter.

4.3 Ammonium formate solution containing 0.1% formic acid: take 1 mL of formic acid, add ammonium formate solution (2 mmol/L), dilute to 1 L, shake well, then filter.

5. Extraction

Hot pot sauce, soup base, flavoring oil

Weigh 2 g of sample (accurate to 0.01 g) in a 50 mL PTFE centrifuge tube with a stopper, add 5 mL water, then vibrate to make it spread evenly. Add 15 mL of acetonitrile, vortex for 1 min, then add a 00528-20000 extraction bag, then vibrate swiftly. Vortex for 1 min, centrifuge at 8000r/min for 5 min, then take the supernatant for purification.

6. QuEChERS Cleanup Process

Transfer the supernatant to a 00565-20020 clean-up tube and vortex for 1 min to mix. Centrifuge the supernatant at 10000 rpm for 2 min. Take the supernatant through a 0.22 μm syringe filter, reserve the filtrate for test.

7. Instrument Condition

7.1 HPLC Conditions

Column: Welch Boltimate® HILIC 2.1×100 mm, 2.7 μm

Mobile phase:

A: 2 mmol ammonium formate solution containing 0.1% formic acid

B: acetonitrile containing 0.1% formic acid

Flow rate: 0.25 mL/min

Column temperature: 30 °C

Gradient elution procedure: in Tab. 1

Injection volume: 2 μL

Time(min)	Mobile Phase A(%)	Mobile Phase B(%)
0	10	90
3	30	70
3.01	10	90
7	10	90

Tab.1 Mobile phase composition and gradient elution

7.2 Mass spectrum conditions

Ion source: ESI+

DL tube temperature: 350 °C

Flow rate of atomizer: 3.0 L /min

Acquisition method: MRM

Instrument type: Shimadzu LC30A+8050MS

Inlet temperature: 300 °C

Heating block temperature: 350 °C

Flow rate of heater: 10.0 L / min

Q1 and Q3 are unit resolutions

Name	Parent Ion (m/z)	Son Ion (m/z)	Deflection Voltage 1(V)	Deflection Voltage 1(V)	Collision Energy (V)
Papaverine	340.1	202.1	-23.0	-21.0	-26.0
		171.1	-24.0	-17.0	-39.0
Morphine	285.9	180.9	-14.0	-19.0	-36.0
		165.1	-10.0	-30.0	-40.0
Narcotine	414.1	220.1	-15.0	-23.0	-23.0
		353.1	-10.0	-24.0	-25.0
Codeine	299.90	215.1	-10.0	-22.0	-26.0
		64.9	-10.0	-26.0	-55.0
Thebaine	312.0	58.1	-11.0	-24.0	-14.0
		249.2	-11.0	-26.0	-16.0

Tab.2 Multiple reaction monitoring(MRM) conditions

4. Chromatogram and Result of Spike Recovery Rate

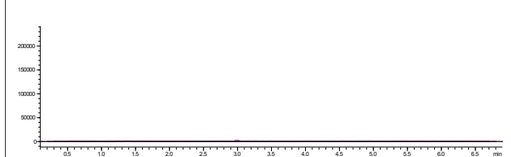


Fig.1. TIC Chromatogram of blank sample

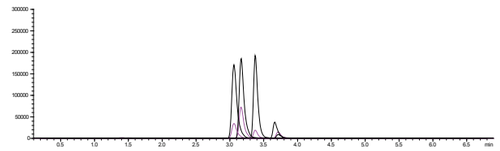


Fig.2. TIC chromatogram of spiked blank sample

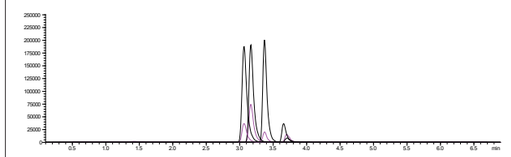


Fig.3. TIC Chromatogram of mixed standards in which papaverine, nacodine and thebaine have a concentration of 2 μg/L, morphine and codeine have a concentration of 5 μg/L

Result of Spike Recovery Rate

Parameters	Concentration (μg/kg)	Recovery Rate/%
Papaverine	15	79.1-85.2
Morphine	37.5	85.4-93.1
Narcotine	15	80.3-87.1
Codeine	37.5	84.1-97.4
Thebaine	15	81.6-87.3

Conclusion

This experiment established HPLC-MS/MS method for the determination of papaverine, morphine, narcodine, codeine and thebaine in hot pot food, samples spiked at 15 μg/kg and 37.5 μg/kg were tested, with recovery rates between 79.1% and 97.4%. The QuEChERS method demonstrated stability, and good reproducibility was shown, indicating that this method is suitable for determining the contents of papaverine, morphine, noscapine, codeine, and thebaine in hot pot foods.

QuEChERS Methos
Determination of Phthalate in Food SN/T 3147-2017

1. Range of Application

Applicable for the determination of phthalates in fatty food (rapeseed oil is selected as the sample matrix in this experiment).

Reference Standard: SN/T 3147-2017 Determination Method for Phthalates in Exported Food.

2. Configuration of Solution

1) Standard storage solution(100 μg/mL): accurately transfer the standard storage solution (1 mg/mL) 1.0 mL and dilute with acetone to 10 mL.

2) Standard working solution(10 μg/mL): accurately transfer 1 mL standard storage solution, dilute to 10 mL with hexane.

3) Hexane saturated with acetonitrile: take 100 mL acetonitrile and 100 mL hexane, mix them well, and take the acetonitrile-saturated hexane in the upper layer.

4) Acetonitrile saturated with hexane: take 100 mL acetonitrile and 100 mL hexane, mix them well, and take the hexane-saturated acetonitrile in the lower layer.

3. Extraction Process

1) Weigh 5 g of the samples in a glass tube and add 2 mL of acetonitrile-saturated hexane, then vortex. Add 4 mL of hexane-saturated acetonitrile, vortex, then ultrasound for 10 mins. Centrifuge for 5 min (3000 r/min), carefully transfer the lower layer solution into another glass tube. Add another 4 mL of hexane-saturated acetonitrile, and repeat the extraction process. Combine the two extraction solutions.

2) Place the extract in a water bath at 40 ℃, blow with nitrogen to nearly dry, accurately add 5 mL of acetonitrile, vortex, then add 50 mg of PSA, 50 mg of C18E and 150 mg of $MgSO_4$. Vortex and centrifuge for 5 min (3000 r/min), and take the supernatant for testing.

4. Precautions

1) Spike level: Add 0.025 mL 10 μg/mL standard to 5 g sample and delute to 5 mL. The spike level is 0.05 mg/kg, and the datum of machine is 0.125 μg/mL.

2) Blow with nitrogen to nearly dry, leaving a drop of liquid.

5. Chromatographic Condition

5.1 Gas chromatography conditions

Column	WM-5MS, 30 m × 0.25 mm × 0.25 μm
Inlet Temp.	260 °C
Carrier Gas	High purity helium (>99.999%)
Injection	Splitless injection
Injection Volume	1 μL
Temperature Program	Initial 60 °C (hold for 1 min); raise to 220 °C at 20 °C/min, hold for 1 min; raise to 250 °C at 5 °C/min, hold for 1 min; raise to 290 °C at 20 °C/min, hold for 7.5 min.

5.2 Mass spectrum conditions

Ionization method	Electron ionization (EI)
Ionization energy	70 eV
Transmission line temperature	280 °C
Ion source temperature	230 °C
Monitoring way	Selected ion monitoring (SIM)
Solvent delay	7.5 min

6. Chromatogram and Spike Recovery Results

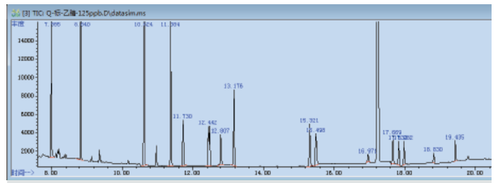


Fig.1. Chromatogram of phthalates standard at 0.125 μg/mL

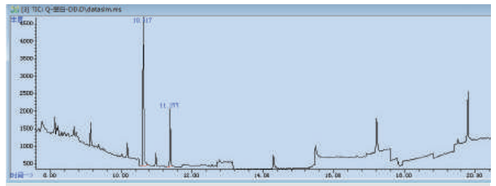


Fig.2. Blank chromatogram

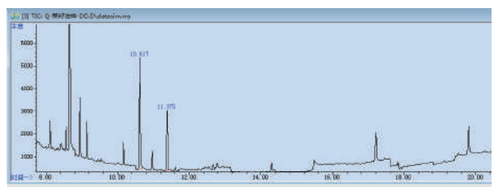


Fig.2. Chromatogram of rapeseed oil sample after cleanup

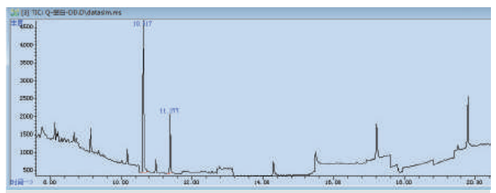


Fig.4. Chromatogram of spiked rapessed oil sample (0.05 mg/kg) after cleanup

No.	Name	Retention Time	Peak Width	Area
1	DMP	7.995	0.026	231015
2	DEP	8.84	0.022	215986
3	DIBP	10.624	0.026	331212
4	DBP	11.384	0.028	240580
5	DMEP	11.73	0.029	80356
6	BMPP	12.442	0.055	144812
7	DEEP	12.807	0.034	65897
8	BPP	13.176	0.032	149986
9	DPP	15.321	0.051	97533
10	DHXP	15.498	0.038	75616
11	DBEP	16.971	0.038	20408
12	DCHP	17.669	0.035	61322
13	DEHP	17.833	0.033	51514
14	DPhP	17.982	0.033	53156
15	DNP	18.83	0.024	16379
16	DNOP	19.435	0.028	38063

Table 1. Correlation peak information of reference substance

No.	Name	Retention Time	Area
1	DMP	91.08%	4.33%
2	DEP	92.83%	0.24%
3	DIBP	64.94%	0.85%
4	DBP	84.56%	2.40%
5	DMEP	98.81%	0.47%
6	BMPP	94.82%	4.31%
7	DEEP	111.22%	4.07%
8	DPP	93.01%	0.12%
9	DHXP	83.75%	0.11%
10	BBP	133.48%	5.74%
11	DBEP	138.31%	1.07%
12	DCHP	87.88%	0.42%
13	DEHP	75.59%	4.82%
14	DPhP	109.14%	1.41%
15	DNP	72.99%	3.40%
16	DNOP	68.25%	0.64%

Table 2: Recovery rates of spiked sample (0.05 mg/kg)

Ordering Information of Related Products

P/N	Product	Description
00821-32291	Caps and septa	Welchrom Pre-slit red PTFE/white Silicone septa, 9mm blue short screw-thread polypropylene cap, 6mm centre hole, 100 pcs/pk
00821-40927	Vials	2 mL wide opening short screw-thread vial with write-on spot, clear, 11.6 × 32mm, 100 pcs/pk
00533-20020	Clean-up tube	2 mL, AOAC method,150 mg $MgSO_4$,50 mg PSA, 50 mg C18E, for fruits and vegetables with fats and waxes,100 pcs/pk
03904-22001	GC column	WM-5MS, 30 m × 0.25 mm × 0.25 μm

QuEChERS Method

Determination of 15 Kinds of PAHs in Foods GB 5009.265-2016

1. Range of Application

Applicable for the determination of 15 PAHs in cereals, cereal products, vegetables and fruits. In this experiment, the selected samples were flour, cabbage and cherry tomatoes.

Reference Standard: GB 5009.265-2016 National Food Safety Standard for the Determination of PAHs in Food.

2. Extraction Steps

Weigh 2 g of samples in a 50 mL centrifuge tube, add 10 mL of n-hexane, then perform ultrasonic extraction for 15 min and centrifuge at 8000 r/min for 5 min. Transfer the supernatant to another 50 mLcentrifuge tube. Continue to add 10 mL n-hexane to the residue, perform ultrasonic extraction for 15 min, followed by centrifugation at 8000 r/min for 5 min. Mix the supernatant and evaporate with nitrogen to near dryness. Add 3 mL of acetonitrile into the centrifuge tube. After fully mixing, slowly blow with nitrogen to remove all the n-hexane, then delute to 2 mL with acetonitrile. Reserve for cleanup.

3. Cleanup Steps

Transfer the liquid to the (005PM-048-50) clean-up tube, and vortex for 5 min, centrifuge at 6000 r/min for 5 min. Then filter the supernatant through a 0.22 μm syringe filter for HPLC analysis.

4. Chromatographic Condition

Chromatographic column: Ultisil® PAH (00210-31043), 4.6×250 mm, 5 μm
Mobile phase: acetonitrile/water
The gradient elution procedure is shown in the table to the right

Flow rate: 1.5 mL/min
Column temperature: 30 °C
Injection volume: 20 μL
Wavelength: the fluorescence detection wavelength gradient is shown in the following table

Time(min)	Mobile Phase A(%)	Mobile Phase B(%)
0	50	50
5	50	50
20	100	0
35	100	0
36	50	50
45	50	50

Tab. 1: Gradient elution procedures

Time(min)	Excitation wavelength	Target
0-16	270/324	Naphthalene, acenaphthylene, fluorene
16-18	248/375	Phenanthrene, anthracene
18-28.9	292/410	Fluoranthrene, pyrene, benzo[a]anthracene, chrysene, benzo[b]fluoranthrene, benzo[k]fluoranthrene, benzo[a]pyrene, dibenz[a,h]anthracene, benzo[g, h, i]pyrene
28.9-45	274/507	Indeno[1, 2, 3-cd]pyrene

Tab.2: Fluorescence detection wavelength gradient procedure

5. Chromatogram and Spike Recovery Results

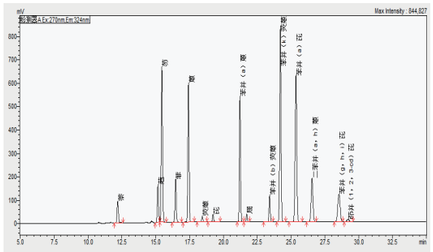


Fig.1. Chromatogram of 15 kinds of PAH standard substances, 20 μg/L

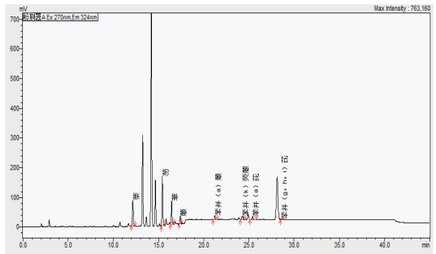


Fig.2. Blank chromatogram of flour

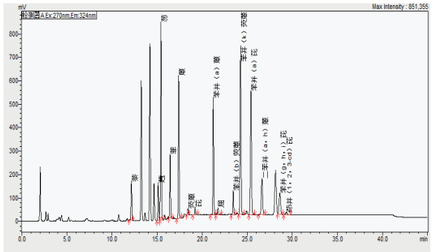


Fig.3. Chromatogram of spiked flour sample (20.0 μg/kg)

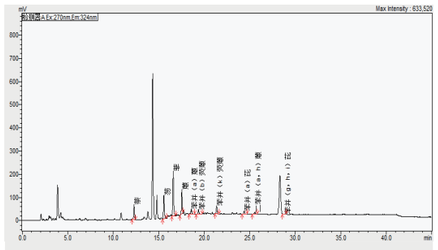


Fig.4. Blank chromatogram of vegetables

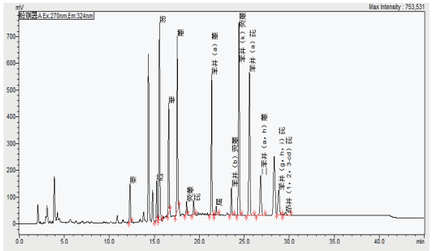


Fig.5. Chromatogram of spiked vegetable sample (20.0 μg/kg)

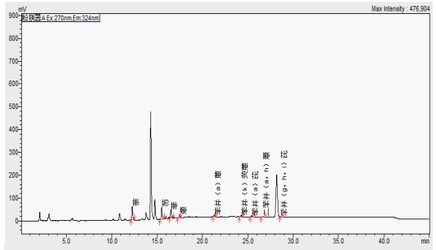


Fig.6. Blank chromatogram of fruits

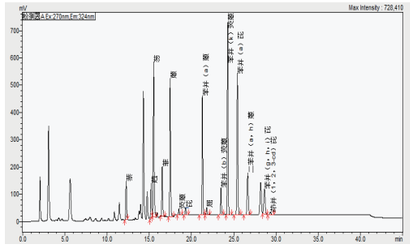


Fig.7. Chromatogram of spiked fruit sample (20.0 μg/kg)

Other matrices: The recovery rates of the vegetable matrices ranged from 74.54% to 107.448%, and the RSD (n=3) ranged from 0.52% to 3.11%. The recovery rate of fruit matrices was 74.22%-92.73%, and the RSD (n=3) was 0.09%-2.95%.

Classification	Spike Level μg/L	Recovery Rate/%	RSD/%
Naphthalene	2.0	82.60%	3.11%
Acenaphthylene		96.34%	1.80%
Fluorene		96.05%	2.83%
Phenanthrene		107.48%	2.88%
Anthracene		92.81%	1.66%
Fluoranthrene		99.13%	1.46%
Pyrene		106.45%	2.37%
benzo[a]anthracene		92.46%	1.94%
Chrysene		100.34%	0.52%
Benzo[b]fluoranthrene		88.25%	1.83%
Benzo[k]fluoranthrene		87.26%	1.69%
Benzo[a]pyrene		85.60%	1.56%
Dibenz[a,h]anthracene		84.59%	1.35%
benzo[g, h, i]pyrene		74.54%	2.55%
Indeno(1, 2, 3-cd)pyrene		81.65%	2.27%

Tab 3: Spike Recovery

Ordering Information of Related Products

P/N	Product	Description
005PM-048-50	Clean-up tube	15 mL, 900 mg $MgSO_4$,100 mg C18E, 100 mg PSA, 50 pcs/pk
00210-31043	HPLC Column	Ultisil® PAH, 5 μm, 4.6×250 mm
00802-02201	Syringe filters	Welchrom NY, 13 mm×0.22 μm, 100 pk

QuEChERS Method

Determination of 15 Kinds of PAHs in Water HJ 478-2009

1. Range of Application

Applicable for the determination of 15 kinds of PAHs in water samples.

Reference Standard: HJ 478-2009 Water Quality-Determination of PAHs Liquid-liquid Extraction and Solid-Phase Extraction Followed by HPLC.

2. Extraction Steps

Measure 20 mL of water sample in a 50 mL centrifuge tube, add 0.2 g sodium thiosulfate, 1 g NaCl and 10 mL n-hexane. Perform ultrasonic extraction for 15 mins, then centrifuge at 6000 r/min for 5 min. Transfer the supernatant to another 50 mL clean centrifuge tube. Add 10 mL of n-hexane in the residue, perform ultrasonic extraction for 15 min, then centrifuge at 6000 r/min for 5 min. Mix the supernatant and evaporate with nitrogen to near dryness, then add 3 mL acetonitrile into the centrifuge tube, mix well, slowly evaporate with nitrogen to eliminate hexane, dilute with acetonitrile to 2 mL. Reserve for cleanup.

3. Cleanup Steps

Transfer the liquid to be purified to 005PM-048-50 clean-up tube, then vortex for 5 min and centrifuge at 6000 r/min for 5 min. Filter the supernatant through a 0.22 μm syringe filter for HPLC analysis.

4. Chromatographic Condition

Column: Ultisil® PAH, 4.6 x 250 mm, 5 μm

Mobile phase: acetonitrile/water

The gradient elution procedure is shown in the table to the right

Flow rate: 1.5 mL/min

Column temperature: 30 °C

Injection volume: 20 μL

Wavelength: the fluorescence detection wavelength gradient is shown in the following table

Time(min)	Mobile Phase A(%)	Mobile Phase B(%)
0	50	50
5	50	50
20	100	0
35	100	0
36	50	50
45	50	50

Tab. 1: Gradient elution procedures

Time(min)	Excitation wavelength	Target
0-16	270/324	Naphthalene, acenaphthylene, fluorene
16-18	248/375	Phenanthrene, anthracene
18-28.9	292/410	Fluoranthrene, pyrene, benzo[a]anthracene, chrysene, benzo[b]fluoranthrene, benzo[k]fluoranthrene, benzo[a]pyrene, dibenz[a,h]anthracene, benzo[g, h, i]pyrene
28.9-45	274/507	Indeno[1, 2, 3-cd]pyrene

Tab 2: Fluorescence detection wavelength gradient procedure

5. Chromatogram or Result of Spike Recovery Rate

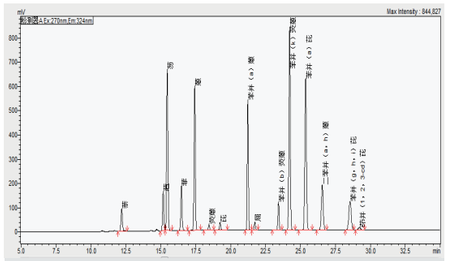


Fig.1. Chromatogram of 15 kinds of PAH standard substances, 20 μg/L

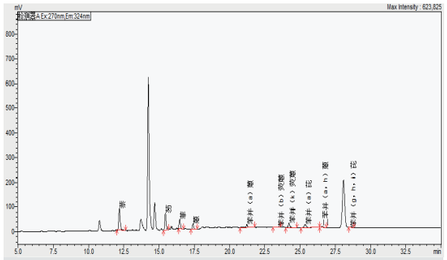


Fig.2. Blank chromatogram of water sample

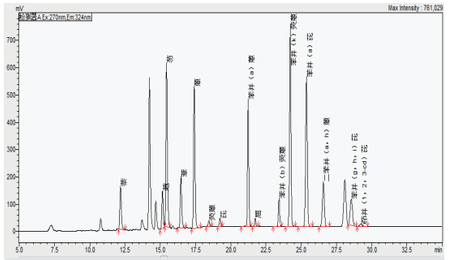


Fig.3. Chromatogram of spiked water sample (2.0 μg/L)

Classification	Spike Level μg/L	Recovery Rate/%	RSD/%
Naphthalene	2.0	76.88%	1.04%
Acenaphthylene		87.51%	0.91%
Fluorene		86.56%	1.42%
Phenanthrene		93.39%	2.02%
Anthracene		91.59%	1.12%
Fluoranthrene		97.53%	0.61%
Pyrene		94.49%	0.62%
Benzo[a]anthracene		89.73%	0.22%
Chrysene		92.36%	0.06%
Benzo[b]fluoranthrene		88.65%	0.17%
Benzo[k]fluoranthrene		87.92%	0.43%
Benzo[a]pyrene		86.59%	0.57%
Dibenz[a,h]anthracene		87.47%	0.34%
Benzo[g, h, i]pyrene		76.84%	1.15%
Indeno(1, 2, 3-cd)pyrene		85.62%	0.84%

Tab 3: Standard recovery of water sample

Ordering Information of Related Products

P/N	Product	Description
005PM-048-50	Clean-up tube	15 mL, 900 mg $MgSO_4$,100 mg C18E, 100 mg PSA, 50 pcs/pk
00210-31043	HPLC column	Ultisil® PAH, 5 μm, 4.6×250 mm
00802-02201	Syringe filters	Welchrom NY, 13 mm×0.22 μm, 100 pk