Quality, Innovation, Competitive Price

Welchrom® QuEChERS

www.welch-us.com

Introduction

Welch Materials is a multinational company that develops and manufactures chromatography consumables including analytical and preparative HPLC columns, Solid Phase Extraction (SPE) columns, GC columns, bulk packing materials, and protein purification products.

Welch Materials was established in 2003 at Shanghai, China and Welch Materials (Zhejiang) was opened in 2011 at Jinhua, Zhejiang, China. Welch has established operations at Welch Materials, Inc., at West Haven, CT, USA and Welch Materials, India PVT. Ltd., at Gurgaon.

Welch strength lies in our deep experience in particle surface modification science. We are experts in bonding chemistry and innovative packing materials for chromatography applications. Utilizing and optimizing our resources, we have developed many innovative products including five series of HPLC columns including Ultisil®, Welchrom®, Xtimate®, Topsil®, and Boltimate™ and market and support these products on worldwide basis.

Certificate of Registration

This certifies that the Quality Management System of

Welch Materials Inc.(Zhejiang)

Unified Social Credit Code: 91330701581660807G
No.168, Shuanglin South Street, Qiubin Community, Wucheng District,
Jinhua City, Zhejiang Province, China 321016

has been assessed by NSF-ISR and found to be in conformance to the following standards(s):

ISO 9001:2015

Scope of Registration:
Manufacture of Chromatographic Separation Products (Chromatographic Column, Chromatographic Packing Materials and Solid Phase Extraction Products), Design of Chromatographic Separation Products (Chromatographic Packing Materials)

Certificate Number: CNQM50357477
Certificate Issue Date: 09-SEP-2019
Registration Date: 03-SEP-2018
Expiration Date*: 02-SEP-2021

Tom Chestnut,
Sr Vice President - ISR,
NSF-ISR, Ltd.

The certificate information can be searched on the website of CNCA (www.cnca.gov.cn).
The certified organization must do the regular surveillance audit and get valid audit conclusion to maintain the effective of the certificate

NSF International Strategic Registrations

789 North Dixboro Road, Ann Arbor, Michigan 48105 | (888) NSF-9000 | www.nsf-isr.org

Page 1 of 2

ANNEX PAGE FOR CERTIFICATE REGISTRATION NUMBER:
CNQM50357477
CERTIFICATE ISSUE DATE: *09-SEP-2019*
CERTIFICATE EXPIRATION DATE: *02-SEP-2021*

Welch Materials Inc.(Zhejiang)

No.168, Shuanglin South Street, Qiubin Community, Wucheng District,
Jinhua City, Zhejiang Province, China 321016

Remote Location:	Scope:
Welch Materials Inc. Unified Social Credit Code: 91310000753805145A Building 10, Zijingyuan, Qidi Caobejing(Zhongchao) Technology Park, No. 85, Mingnan Rd, Songjiang District, Shanghai City, China 201203	Sales of Chromatographic Separation Products

NSF International Strategic Registrations

789 North Dixboro Road, Ann Arbor, Michigan 48105 | (888) NSF-9000 | www.nsf-isr.org
This Annex is only Valid in connection with the above-mentioned certificate issued by NSF-ISR

Page 2 of 2

Contents

QuEChERS Method Introduction

QuEChERS(sounds like "Catchers") is the abbreviation describing its features of quick, easy, cheap, effective, rugged and safe. This method was first developed by Department of Agriculture M. Anastassisdes et al. (2003) in U.S. Finally, QuEChERS method was formally proposed after multiple verification and improvement.

The QuEChERS method is a residue detection method for multiple target objects in the analysis of pesticide residues in the matrix with high water content (80-95%). It is the fast version of SPE with the similar purification effect, but its treatment steps are more concise. QuEChERS method has the characteristics of time saving, high efficiency and economical. There may be a large number of chlorophyll, lipid, steroid and other compounds in sample matrix of food, animal products and so on. These extracts can seriously interfere with the analysis of target substance, while QuEChERS method can effectively reduce these problems. With a few simple steps. it can complete the preparation of pesticide residues and other samples. Nowadays, this method has been widely accepted worldwide, if it is applied to your project, you will find that it can significantly reduce the time required for method development and pretreatment.

Comparative Advantage

Reference Project	Traditional SPE	QuEChERS	Merits 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.
- Economical: As the professional chromatographic packing materials manufacturer, we offer preferential price by bulk purchasing of our reagents.
- Durable: ultra-clean SPE adsorbent and strict quality control to ensure reproducibility of results. Anhydrous magnesium sulfate is treated by muff furnace at high temperature to improve the dehydrating effect and remove contaminants that may be introduced in the self-made process.
- Customized: We provide customized service according to the needs of users, and professional technical services of our technical team.
- Humanized packaging: The extraction bag is packaged by coffee bag, and the clean-up tube is packaged by vertical pipe rack, which is very convenient to use.

Development Course of QuEChERS Method

Step 1——Extraction

Original Method, 2003	AOAC Method, 2007 AOAC 2007.01	EN Method, EN 15662
10 g homogenized/hydrated sample	15 g homogenized/hydrated sample	10 g homogenized/hydrated sample
⬇	⬇	⬇
10 ml acetonitrile + internal standard, extract by vibration	15 ml acetonitrile (containing 1% acetic acid) + internal standard, extract by vibration	10 ml acetonitrile + internal standard, extract by vibration

↓	↓	↓
4 g anhydrous $MgSO_4$ and 1 g NaCl, vigorously shake for 1 min and centrifuge at 5000 rpm for 5 min	6 g anhydrous $MgSO_4$ and 1.5 g NaAc, shake vigorously for 1min and centrifuge at 5000 rpm for 5min	4 g anhydrous $MgSO_4$+1 g NaCl+1 g sodium citrate (2 H_2O) +0.5 g disodium hydrogen citrate shake (1.5 H_2O) vigorously for 1 min and centrifuged at 5000 rpm for 5 min

Step 2——Purification

Take the supernatant	Take the supernatant	Take the supernatant
↓	↓	↓
Add into OR clean-up tube, shake for 1 min and centrifuge at 5000 rpm for 1 min	Add into AOAC clean-up tube, shake for 30s and centrifuge at 13000 rpm for 5min	Add into EN clean-up tube, shake for 30s and centrifuge at 13000 rpm for 5min
↓	↓	↓
GC or LC analysis	GC/MS or LC/MS/MS analysis	GC/MS or LC/MS/MS analysis

AOAC, EN methods add buffer salts in the original OR method to ensure effective extraction of pH-sensitive compounds, to reduce degradation of sensitive compounds (such as pesticides that are not stable to alkali or acid), and to expand the application range of broad spectrum of this method for food matrices. In the first step of extraction, citrate is used as a buffer to adjust pH to 5-5.5, allowing most pesticides that are not stable to acid or alkali to remain stable. For alkali-sensitive compounds, the stability can be improved by adding a small amount of formic acid after purification. 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 pesticide and matrices to ensure adequate phase separation. Even high-fat samples such as avocados or olive oil can be analyzed this way. However, for strong nonpolar pesticides, the recovery rate can only reach 70% due to phase separation entered lipid phase. Those lipids extracted together can be removed by freezing or adding a C18E adsorbent.

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

Common Questions for QuEChERS Method

1. How to improve recovery rate of method?

- Using high-quality homogeneous device can obtain smaller sample particles and improve the precision of the method.
- Using matrix solution to prepare standard solution can reduce matrix effect and obtain higher accuracy.
- The addition of isotopic internal standard can effectively monitor the recovery rate.
- Making the water content of the sample to be tested reach 80% can effectively improve the extraction efficiency.
- Alkali sensitive pesticides can be added with buffer salt, extraction and extraction bags to prevent loss.
- According to the thermal stability of pesticides, appropriate analytical means should be adopted. GC or GC/MS can be used for general pesticides, while LC/MS/MS can be used for thermal unstable pesticides.
- Toluene is added to the final sample solution to prevent thermal instability in the GC liner.
- Adding 1% formic acid to the final sample solution can reduce the degradation loss of alkali sensitive compounds.

2. Questions of chromatography

- Adding acetic acid will affect the purification effect of PSA adsorbent and leading peak and 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 purification methods such as SPE-QuEChERS and QuEChERS-GPC can be used.

Welchrom® QuEChERS Ordering Information for Extraction Bags

Method	$MgSO_4$	Na-acetate	Na-citrate	NaCitrate Sesquihydrate	NaCl	Pack Size	P/N
AOAC Method (15g sample)	6 g	1.5 g	1 g	0.5 g		50	00528-20000
EN Method (10g sample)	4 g		1.5 g	0.75 g	1 g	50	00529-20000
Original Method (10g sample)	4 g				1 g	50	00530-20000
EN Method (15g sample)	6 g				1.5 g	50	00529-25000
Original Method (15g sample)	6 g				1.5 g	50	00530-25000

Welchrom® QuEChERS Ordering Information of Clean-up Tubes

Method	P/N	Centrifuge Tube	$MgSO_4$	PSA	C18E	GCB	Pack Size
AOAC method general fruits and vegetables	00531-20020	2 ml	150 mg	50 mg	-	-	100
	00531-20021	15 ml	1200 mg	400 mg			50
EN method general fruits and vegetables	00532-20020	2 ml	150 mg	25 mg	-	-	100
	00532-20021	15 ml	900 mg	150 mg			50
AOAC method fruits and vegetables with fats and waxes	00533-20020	2 ml	150 mg	50 mg	50 mg	-	100
	00533-20021	15 ml	1200mg	400 mg	400 mg		50
EN method waxy or fatty fruits and vegetables	00534-20020	2 ml	150 mg	25 mg	25 mg	-	100
	00534-20021	15 ml	900 mg	150 mg	150 mg	-	50
AOAC method pigmented fruits and vegetables	00535-20020	2 ml	150 mg	50 mg	-	50 mg	100
	00535-20021	15 ml	1200 mg	400 mg	-	400 mg	50
EN method pigmented fruits and vegetables	00536-20020	2 ml	150 mg	25 mg	-	2.5 mg	100
	00536-20021	15 ml	900 mg	150 mg	-	15 mg	50
AOAC method fruits and vegetables with pigments and fats	00537-20020	2 ml	150mg	50 mg	50 mg	50 mg	100
	00537-20021	15 ml	1200 mg	400 mg	400 mg	400 mg	50
EN method fruits and vegetables with pigments and fats	00538-20020	2 ml	150 mg	25 mg		7.5 mg	100
	00538-20021	15 ml	900 mg	150 mg		45 mg	50

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

Note: In this step, for 2 ml and 15 ml clean-up tubes in AOAC method, the specified sample volumes are 1ml and 8ml, while 1 ml and 6ml in EN method.

Several different types of adsorbents are used to remove major interferences from the matrices	
$MgSO_4$	Remove water from the sample matrix
PSA	Adsorb the carbohydrate, fatty acid, organic acid and some pigment in the matrix
C18E	Remove nonpolar disruptors such as fats and lipids
GCB	Remove pigments, sterols and nonpolar interferers

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.	High pigment and fat sample, dark color sample.	The high fat samples contained > 1% fat and lipids
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.	Representative fruit: Blackberries, blueberries, seedless fruit, raspberries, red grapes, raisins, mango, papaya, avocado, olives.	Representative fruit: Orange juice, grapefruit, lemon, orange, orange peel, nectarine, orange, banana, avocado, olive
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	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® Disperse solid phase clean-up tube			
00531-20020	00535-20020	-	-
00531-20021	00535-20021	00537-20020	00533-20020
00532-20020	00536-20020	00537-20021	00533-20021
00532-20021	00536-20021	-	-

Purification Effects of Different Concentrations of Welchrom® GraphiCarb in Spinach Extract

No Clean-Up	PSA Only	PSA + 5 mg GCB	PSA + 10 mg GCB	PSA + 20 mg GCB	PSA + 50 mg GCB

Graphitized carbon has a strong adsorption for planar pesticides.

Solutions:

Reduce the amount of graphitized carbon, which also reduces the adsorption of pigment.

The recovery rate can be optimized by adding proper amount of toluene (such as acetonitrile/toluene =8/3).

More QuEChERS Customized Products

P/N	Product	Pack	Usage
00551-20000	Extraction bag	5.0 g $MgSO_4$, 50 pcs/pk	Customized product
005PM-055-50	Extraction bag	8.0 g $MgSO_4$, 2.0 g NaCl, 50 pcs/pk	Customized product
005PM-059-50	Extraction bag	4.0 g $MgSO_4$, 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	2015 Edition Of Chinese Pharmacopoeia 2341
00588-20020	Clean-up tube	2 ml, 200 mg $MgSO_4$, 50 mg PSA, 100 pcs/pk	Customized product
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
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 containing pigments and lipids
005PM-001-50	Clean-up tube	5 ml, 50 mg $MgSO_4$, 50 mg PSA,50 mg C18E, 100 pcs/pk	Custom products
005PM-002-50	Clean-up tube	5 ml, 50 mg $MgSO_4$, 50 mg PSA, 50 mg C18E, 50 mg GCB, 50 pcs/pk	Custom products
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 (Pig liver and animal group weave 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	Custom products
005PM-016-50	Clean-up tube	15 ml, 1 g $MgSO_4$, 50 pcs/pk	Pharmacopoeia 2015 edition four agricultural residue detection 2341
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 multiple residues of pesticides in 51 kinds of 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
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
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

QuEChERS-EN Methods for Determining the Organophosphorus Pesticide Residues in Cucumber

1. Range of Application

It is suitable for the determination of 7 kinds of organophosphorus in cucumber (dichlorvos, thiophosphorus, diazine phosphorus, chlorpyrifos, methyl parathion, borer thiophosphorus, triazophos).

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

2. Extraction and Purification

Weigh 10 g sample (pre-mashed into slurry and mixed) in 50 ml centrifuge tube with plug and add 10 mL acetonitrile. Add 00529-20000 extraction bags and shake violently for 1min. Centrifuge at 4200 r/min for 5 min, then absorb 6 mL of the supernatant into the 00532-20021 clean-up tube. Mix the supernatant by vortex for 1 min and centrifuge at 4200 r/min for 5 min. Finally filter the supernatant with a 0.22 μm syringe filter and keep it for GC analysis.

3. Chromatographic Condition

Column	WM-1701, 30 m×0.32 mm×0.25 μm
Inlet Temp.	250℃
Detector (FPD) Temp.	250℃
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	Keep 60℃ for 5 min, rise to 270℃ at 10℃/min for 15 min

4. Chromatogram or Result of Spike Recovery Rate

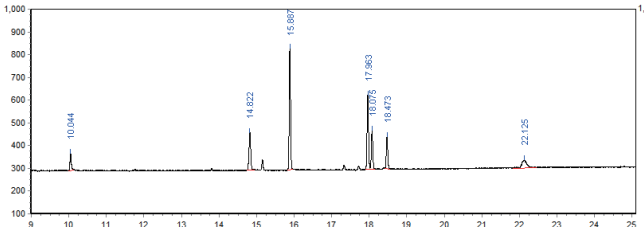

Fig.1. Chromatogram of 7 kinds of organophosphorus standard substance, 0.1 mg/L

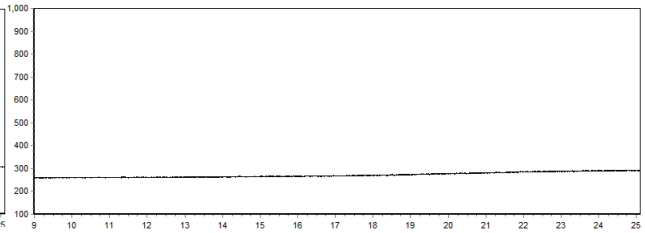

Fig.2. Blank chromatogram of cucumber sample

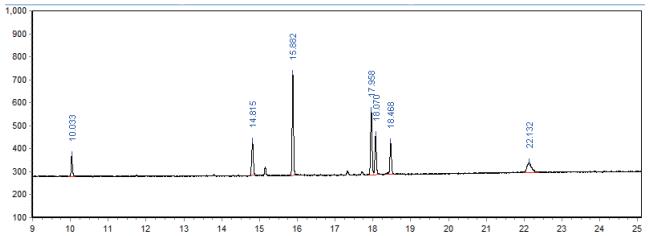

Fig.3. Chromatogram of cucumber sample added a spike of 100.0 ng/g

Classification	Spike Level ng/g	Recovery Rate/%	RSD（n=2）/%
Dichlorvos	100	122.98%	3.31
Sulfur phosphorus line		82.60%	0.83
Diazine phosphorus		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

5. Ordering Information

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 hydrogen citrate, 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 Pesticide Residues in Vegetables

1. Range of Application

It's suitable the determination of pyrethroid pesticide residues of eight substances in vegetables (permethrin, deltamethrin, cyperme-thrin, fenvalerate, bifenthrin, flufenpropathrin, highly effective flufenpropathrin and fenpropathrin).

Reference standard: AOAC Official Method 2007.01: Pesticide in Foods by Acetonitrile Extraction and Partitioning with Sulfate GB 23200.113-2018 National Standard for Food Safety-Determination of 208 Pesticides and Their Metabolites Residues in Plant-derived Food by GC/MS

2. Extraction and Purification

Weigh 15 g sample (the sample is pre-mashed into slurry and mixed) in 50 ml centrifuge tube with plug and add 15 mL acetonitrile containing 1% acetic acid. Add 00528-20000 extraction bags and shake violently for 1 min. Centrifuge the supernatant at 6000 r/min for 5 min to be purified.

Absorb 7 mL of supernatant into 005PM-046-50 clean-up tube (3 mL toluene needs to be added in advance), vortex for 1 min, centrifuge at 6000 r/min for 5 min, then filter supernatant with a 0.22 μm syringe filter and left for GC analysis.

3. Chromatographic Condition

Column	WM-5MS, 30 m×0.25 mm×0.25 μm
Inlet Temp.	220℃
Detector (FPD) Temp.	300℃
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	Keep 180℃ for 2 min, rise to 200℃ for 2min at 4℃/min, and rise to 230℃ /min at 10℃/min and keep for 2 min. Rise to 260℃ at 2℃/min, keep 8.5 min and rise to 270℃ for 2 min at 50℃/min

4. Chromatogram or Result of Spike Recovery Rate

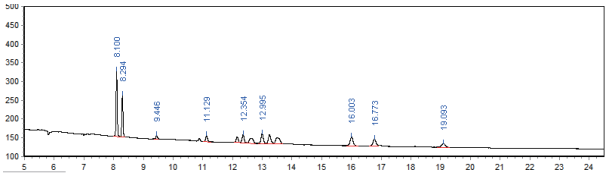

Fig.1. Chromatogram of 8 kinds of pyrethroid pesticide standard substance of mixed standards, 0.1 mg/L

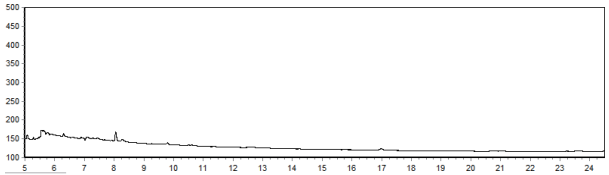

Fig.2. Blank chromatogram of flammulina velutiae sample

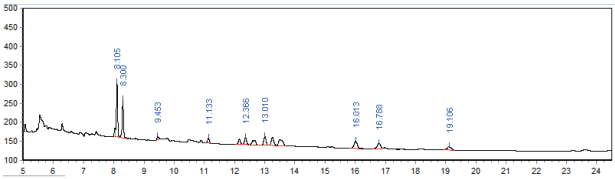

Fig.3. Chromatogram of flammulina velutiae sample added a spike of 100.0 ng/g

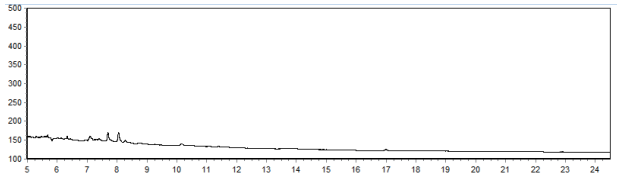

Fig.4. Blank chromatogram of green bean sample added a spike of 100.0 ng/g

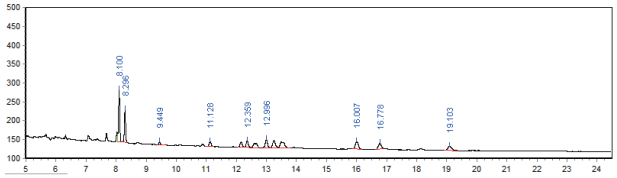

Fig.3. Chromatogram of green bean sample added a spike of 100.0 ng/g

Classification	Spike Level ng/g	Flammulina Velutiae		Green Beans	
		Recovery Rate/%	RSD（n=2）/%	Recovery Rate/%	RSD（n=2）/%
Biphenyl chrysanthemum ester	100	80.87%	0.54	99.30%	1.24
Armor cyanogen chrysanthemum ester		96.25%	0.70	105.26%	3.78
Lambda-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
Bromine permethrin		86.31%	2.51	122.03%	4.84

Tab.1 : Spike Recovery

5. Ordering Information

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 acetic acid sodium, 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 Methods for Determining Organochlorine and Pyrethroid Pesticide Residues in Rice

1. Range of Application

It's suitable for the determination of 11 organochlorines (hexachlorobenzene, β- benzene hexachloride, α- benzene hexachloride, γ- benzene hexachloride (Lin), heptachlor, δ- benzene hexachloride, heptachlor epoxide (outside heptachlor epoxide B) and p, p '- DDE, endrin, o, p' - DDT, p, p '- DDT) organochlorine and the determination of pyrethroid pesticide residues of eight substances (permethrin and deltamethrin, cypermethrin, fenvalerate, bifenthrin ester, fluorine cypermethrin and efficient with ester, armor cyanogen chloride fluorine chrysanthemum ester).

Reference standards: BS EN 15662:2008 Foods of Plant Origin-Determination of Pesticide Residues Using GC-MS or LC-MS/MS

2. Extraction and Purification

Weigh 10g sample (pre-mashed into slurry and mixed) in 50ml centrifuge tube with plug and add 10mL acetonitrile. Add 00529-20000 extraction bags and shake violently for 1min. Centrifuge the supernatant at 4200 r/min for 5 min, absorb 6 mL of the supernatant into the 00534-20021 clean-up tube. Vortex for 1min and centrifuge at 4200 r/min for 5 min, then filter supernatant with a 0.22 μm syringe filter and left for GC analysis.

Clean-up tube: 15 mL centrifuge tube -150 mg PSA+150 mg C18E+900 mg $MgSO_4$.

Extraction bag: 1 g sodium citrate +1 g NaCl +0.5 g disodium citrate sesquihydrate +4 g $MgSO_4$

3. Chromatographic Condition

Column	WM-5MS, 30 m×0.25 mm×0.25 μm
Inlet Temp.	220℃
Detector (FPD) Temp.	300℃
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	Keep 180℃ for 2 min, rise to 200℃ for 2 min at 4℃/min, and rise to 230℃ /min at 10℃/min and keep for 2 min. Rise to 260℃ at 2℃/min, keep 8.5 min and rise to 270℃ for 2 min at 50℃/min

4. Chromatogram or Result of Spike Recovery Rate

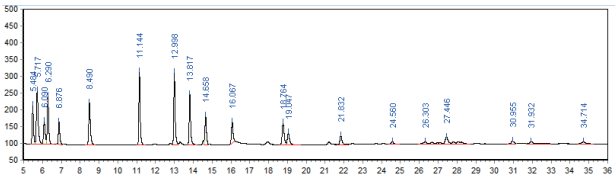

Fig.1. Chromatogram of organochlorine and pyrethroid pesticides standard substance of mixed standards, 0.1 mg/L

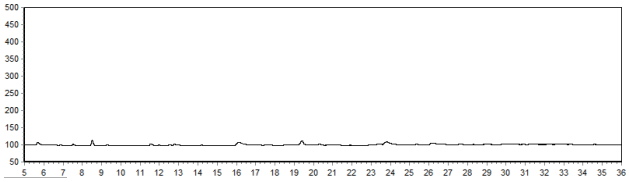

Fig.2. Blank chromatogram of rice sample

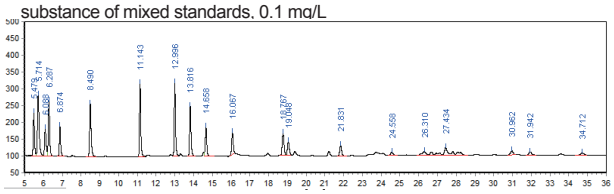

Fig.3. Chromatogram of rice sample added a spike of 100.0 ng/g

Classification	Spike Level ng/g	Recovery Rate/%	RSD（n=2）/%
Hexachlorobenzene	100	102.32%	2.26
α- benzene hexachloride		109.76%	1.68
β- benzene hexachloride		117.73%	3.80
γ- benzene hexachloride		109.55%	1.94
Heptachlor		116.02%	0.78
δ- benzene hexachloride		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
Biphenyl chrysanthemum ester		97.80%	2.65
Armor cyanogen chrysanthemum ester		102.01%	4.42
Lambda-cyhalothrin	1000	118.07%	3.19
Permethrin	100	103.27%	0.09
Cyfluthrin		116.25%	1.71

Cypermethrin	100	120.05%	2.04
Fenvalerate		135.21%	4.82
		127.79%	5.44
Bromine permethrin		120.12%	0.74

Tab 1: Spike Recovery

5. Ordering Information

P/N	Product	Description
03904-22001	Gas capillary 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 fatty and waxy fruits and vegetables, 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-EN Methods for Determining Organochlorine and Pyrethroid Pesticide Residues in Cucumber

1. Range of Application

It is suitable for the determination of many kinds of pesticide residues in common fruits and vegetables.
Reference standards: BS EN 15662:2008 Foods of Plant Origin-Determination of Pesticide Residues GC-MS and/or LC-MS/MS Following Acetonitrile Exraction/Partitioning and Clean-up by Dispersive SPE-QuEChERS-method.

2. Extraction and Purification

Weigh 10 g sample (pre-mashed into slurry and mixed) in 50ml centrifuge tube and add 10 mL acetonitrile. Add 00529-20000 extraction bags and shake violently for 1 min. Centrifuge the supernatant at 4200 r/min for 5 min, absorb 6 mL of the supernatant into the 00532-20021 clean-up tube. Vortex for 1 min and centrifuge at 4200 r/min for 5 min, then filter supernatant with a 0.22 μm syringe filter and left for GC analysis.
Clean-up tube: 15 mL centrifuge tube -150 mg PSA+900 mg $MgSO_4$.
Extraction bag: 1 g sodium citrate +1 g NaCl +0.5 g disodium citrate sesquihydrate+4 g $MgSO_4$

3. Chromatographic Condition

Column	WM-5MS, 30 m×0.25 mm×0.25 μm
Inlet Temp.	220℃
Detector (FPD) Temp.	300℃
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	Keep 180℃ for 2 min, rise to 200℃ for 2 min at 4℃/min, and rise to 230℃/min at 10℃/min and keep for 2 min. Rise to 260℃ at 2℃/min, keep 8.5 min and rise to 270℃ for 2 min at 50℃/min

4. Chromatogram or Result of Spike Recovery Rate

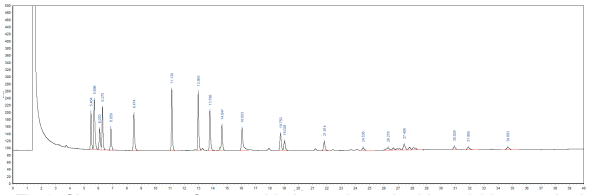

Fig.1. Chromatogram of organochlorine and pyrethroid pesticides standard substance of mixed standards, 0.1 mg/L

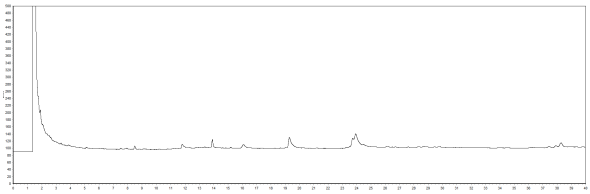

Fig.2. Blank chromatogram of cucumber sample

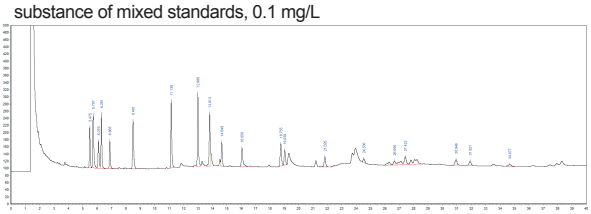

Fig.3. Chromatogram of cucumber sample added a spike of 100.0 ng/g

Classification	Spike Level ng/g	Recovery Rate/%	RSD（n=2）/%
Hexachlorobenzene	100	102.32%	2.26
α- benzene hexachloride		109.76%	1.68
β- benzene hexachloride		117.73%	3.80
γ- benzene hexachloride		109.55%	1.94
Heptachlor		116.02%	0.78
δ- benzene hexachloride		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
Biphenyl chrysanthemum ester		97.80%	2.65
Armor cyanogen chrysanthemum ester		102.01%	4.42
Lambda-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
Bromine permethrin		120.12%	0.74

Tab 1: Spike Recovery

5. Ordering Information

P/N	Product	Description
03904-22001	GC column	WM-5MS 30 m×0.25 mm×0.25 μm
00532-20021	Clean-up tube	15 ml, EN method, 900 mg $MgSO_4$, 150 mg PSA, for general fruits and vegetables, 50 pcs/pk
00529-20000	Extraction bag	EN method, 4 g $MgSO_4$, 1 g NaCl, 1 g sodium citrate, 0.5 g disodium hydrogen citrate, 50 pcs/pk

QuEChERS-AOAC Methods for Determining Organochlorine and Pyrethroid Residues in Cucumber

1. Range of Application

It's suitable for the determination of eleven kinds of organochlorine in cucumber (hexachlorobenzene, β-benzene hexachloride , α- benzene hexachloride, γ- benzene hexachloride (Lin), heptachlor, δ- benzene hexachloride, heptachlor epoxide (outside heptachlor epoxide B) and p, p '- DDE, endrin, o, p' - DDT, p, p '- DDT) and the determination of pyrethroid pesticide residues of eleven substances in cucumber (permethrin and deltamethrin, cyperme-thrin, fenvalerate, bifenthrin ester, fluorine cypermethrin and efficient with ester, armor cyanogen chloride fluorine chrysanthe-mum ester).

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

2. Extraction and Purification

Weigh 15 g sample (pre-mashed into slurry and mixed) in 50 ml centrifuge tube with plug and add 15 mL acetonitrile containing 1% acetic acid. Add 00528-20000 extraction bags and shake violently for 1 min. Centrifuge the supernatant at 6000 r/min for 5 min, absorb 8 mL supernatant into 00533-20021 clean-up tube. Vortex for 1 min and centrifuge at 6000 r/min for 5 min, then filter supernatant with a 0.22 μm syringe filter and left for GC analysis.

3. Chromatographic Condition

Column	WM-5MS, 30 m×0.25 mm×0.25 μm
Inlet Temp.	220℃
Detector (FPD) Temp.	300℃
Carrier Gas	Nitrogen, make-up gas flow rate: 60 ml/min
Injection	Split stream sampling, split ratio 10:1
Carrier Gas Flow Rate	1.6 mL/min
Injection Volume	5 μL
Temperature Program	Keep 180℃ for 2 min, rise to 200℃ for 2min at 4℃/min, and rise to 230℃ /min at 10℃/min and keep for 2 min. Rise to 260℃ at 2℃/min, keep 8.5 min and rise to 270℃ for 2 min at 50℃/min

4. Chromatogram or Result of Spike Recovery Rate

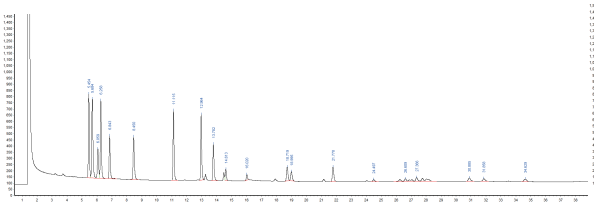

Fig.1. Chromatogram of organochlorine and pyrethroid pesticides standard substance of mixed standards, 0.1 mg/L

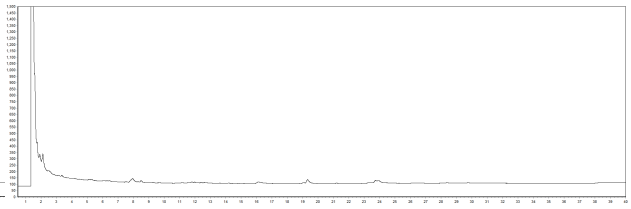

Fig.2. Blank chromatogram of cucumber sample

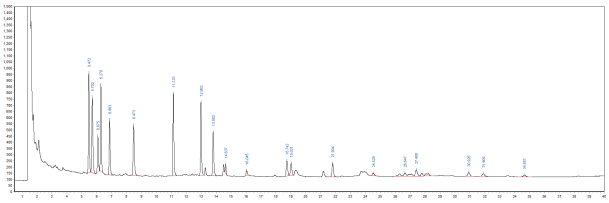

Fig.3. Chromatogram of rice sample added a spike of 100.0 ng/g

Classification	Spike Level ng/g	Recovery Rate/%	RSD（n=2）/%
Hexachlorobenzene	100	113.29%	3.01
α- benzene hexachloride		86.17%	0.56
β- benzene hexachloride		107.54%	4.05
γ- benzene hexachloride		100.12%	3.37
Heptachlor		114.34%	1.79
δ- benzene hexachloride		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
Biphenyl chrysanthemum ester		111.68%	0.30
Armor cyanogen chrysanthemum ester		122.75%	0.88
Lambda-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
Bromine permethrin		72.12%	5.30

Tab 1: Spike Recovery

5. Ordering Information

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 Na-acetate, 50 pcs/pk
00533-20021	Clean-up tube	15 ml, AOAC method, 1200 mg $MgSO_4$, 400 mg PSA, 400 mg C18E, for fatty and waxy fruits and vegetables, 50 pcs/pk

QuEChERS-AOAC Methods for Determining Organochlorine and Pyrethroid Residues in Rice

1. Range of Application

It is suitable for the determination of pesticide residues in fruits, vegetables and grains containing much fat and waxy.
Reference standard: AOAC Official Method 2007.01: Pesticide in Foods by Acetonitrile Extraction and Partitioning with Sulfate

2. Extraction and Purification

Weigh 15 g sample (pre-crushed and mixed) into 50 ml centrifuge tube with plug and add 15 mL acetonitrile containing 1% acetic acid. Add 00528-20000 extraction bag and shake violently for 1 min. Centrifuge the supernatant at 6000 r/min for 5 min, absorb 6 mL supernatant into the 00533-20021 clean-up tube. Vortex for 1 min and centrifuge at 6000 r/min for 5 min, then filter supernatant with a 0.22 μm syringe filter and left for GC analysis.
Clean-up tube: 15 mL centrifuge tube -400 mg PSA+400 mg C18E+1200 mg $MgSO_4$.
Extraction bag: 1.5 g anhydrous sodium acetate +6 g anhydrous magnesium sulfate

3. Chromatographic Condition

Column	WM-5MS, 30 m×0.25 mm×0.25 μm
Inlet Temp.	220℃
Detector (FPD) Temp.	300℃
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	Keep 180℃ for 2 min, rise to 200℃ for 2 min at 4℃/min, and rise to 230 ℃ /min at 10℃/min and keep for 2 min. Rise to 260℃ at 2℃/min, keep 8.5 min and rise to 270℃ for 2 min at 50℃/min

4. Chromatogram or Result of Spike Recovery Rate

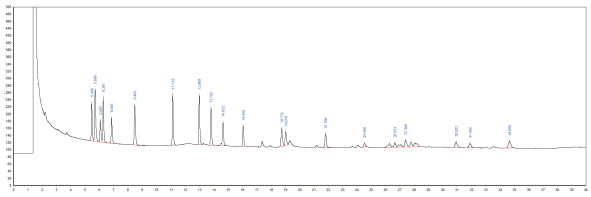

Fig.1. Chromatogram of organochlorine and pyrethroid pesticides standard substance of mixed standards, 0.1 mg/L

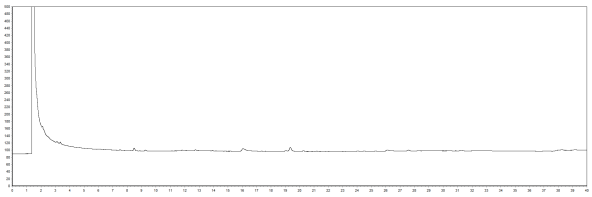

Fig.2. Blank chromatogram of rice sample

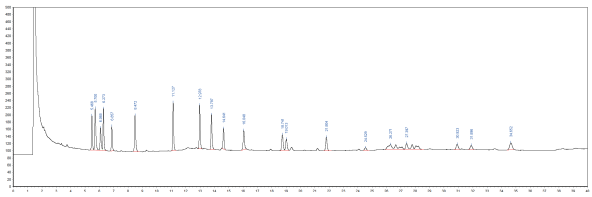

Fig.3. Chromatogram of rice sample added a spike of 100.0 ng/g

Classification	Spike Level ng/g	Recovery Rate/%	RSD（n=2）/%
Hexachlorobenzene	100	84.58%	3.38
α- benzene hexachloride		76.68%	2.53
β- benzene hexachloride		99.02%	2.60
γ- benzene hexachloride		88.41%	3.73
Heptachlor		89.89%	1.02
δ- benzene hexachloride		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
Biphenyl chrysanthemum ester		86.30%	0.70
Armor cyanogen chrysanthemum ester		86.55%	0.21
Lambda-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
Bromine permethrin		101.67%	5.82

Tab 1: Spike Recovery

5. Ordering Information

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 fatty and waxy fruits and vegetables, 50 pcs/pk

QuEChERS-AOAC Methods for Determining Organochlorine and Pyrethroid Residues in Cabbage

1. Range of Application

It is suitable 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 Purification

Weigh 15 g sample (pre-crushed and mixed) into 50 ml plug centrifuge tube and add 15 mL acetonitrile containing 1% acetic acid. Add 00528-20000 extraction bags and shake violently for 1 min. After centrifugation at 6000 r/min for 5 min, 7 mL supernatant should be absorbed into 00537-20021 clean-up tube (in which 3 mL toluene was added in advance). Vortex for 1 min and centrifuge at 6000 r/min for 5 min. Filter supernatant with a 0.22 μm syringe filter and left for GC analysis.

Clean-up tube: 15 mL centrifuge tube-400 mg PSA+400 mg C18E+400 mg GCB+1200 mg $MgSO_4$.
Extraction bag: 1.5 g anhydrous sodium acetate +6 g anhydrous magnesium sulfate

3. Chromatographic Condition

Column	WM-5MS, 30 m×0.25 mm×0.25 μm
Inlet Temp.	220℃
Detector (FPD) Temp.	300℃
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	Keep 180℃ for 2 min, rise to 200℃ for 2min at 4℃/min, and rise to 230℃ /min at 10℃/min and keep for 2 min. Rise to 260℃ at 2℃/min, keep 8.5 min and rise to 270℃ for 2 min at 50℃/min

4. Chromatogram or Result of Spike Recovery Rate

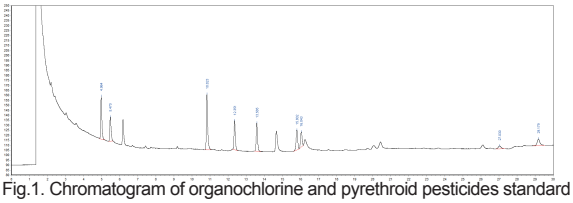

Fig.1. Chromatogram of organochlorine and pyrethroid pesticides standard substance of mixed standards, 0.1 mg/L

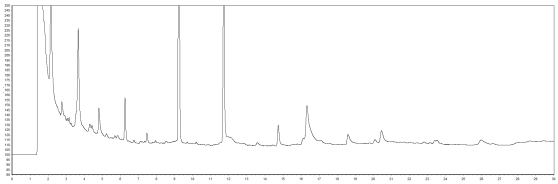

Fig.2. Blank chromatogram of cabbage sample

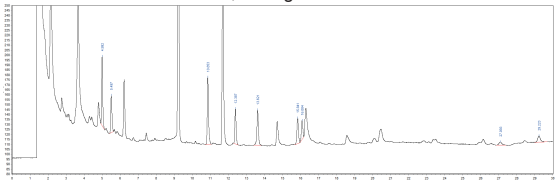

Fig.3. Chromatogram of cabbage sample added a spike of 100.0 ng/g

Classification	Spike Level ng/g	Recovery Rate/%	RSD（n=2）/%
α- benzene hexachloride	100	140.07%	3.49
β- benzene hexachloride		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
Bromopermethrin		90.17%	2.33

Tab 1: Spike Recovery

5. Ordering Information

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 containing pigments and lipids, 50 pcs/pk

QuEChERS Methods for Determining Papaverine, Morphine, Narcodine, Codeine and Thebaine in Hotpot Food

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 sauce, soup, flavoring oil and solid flavoring powder.

2. Extraction and Purification

DB31 2010-2012 Local Standard for Food Safety-Determination of Papaverine, Morphine, Narcodine, Codeine and Thebaine in Hotpot Food LC-MS

3. Chromatographic Condition

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 ammonium formate dissolved in appropriate amount of water to have a constant volume of 2 L, mix for preparation.
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 Steps

Hot pot sauce, soup, flavoring oil
Weigh 2 g sample (accurate to 0.01 g) in 50 mL polytetrafluoroethylene (PTFE) with centrifugal pipe plug, add 5 mL water, then vibrate to make it spread evenly. Add 15 mL of acetonitrile, vortex for 1 min, continue to add 6 g anhydrous magnesium sulfate and the mixture of anhydrous sodium acetate powder 1.5 g (00528-20000), then vibrate fast. Vortex for 1 min, centrifuge at 8000 r/min for 5 min, then take supernatant to be purified.

6、QuEChERS Purification Steps

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

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: 0.1% acetonitrile formate

Flow rate: 0.25 mL/min
Column temperature: 30℃
Gradient elution procedure: in Tab. 1
Injection volume: 2 L

Time/min	A Phase / %	B Phase / %
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℃
Flow rate of atomizer: 3.0 L /min
Acquisition method: MRM
Instrument type: Shimazu LC30A+8050MS

Inlet temperature: 300℃
Heating block temperature: 350℃
Flow rate of heater: 10.0 L / min
Q1 and Q3 are unit resolutions
Shimazu LC30A ULTRA HPLC /8050 triple tandem quadrupole liquid mass spectrometry instrument

Name	Parent Ion (M/Z)	Son Ion (m/z)	Deflection Voltage 1(V)	Deflection Voltage 2 (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

8. Chromatogram or Result of Spike Recovery Rate

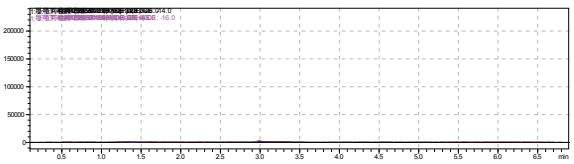

Fig.1. Blank chromatogram of TIC sample

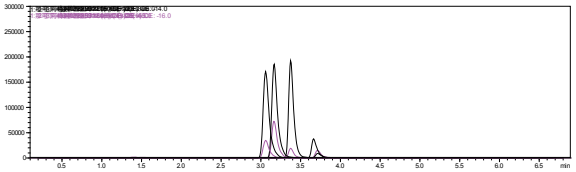

Fig.2. Blank chromatogram of TIC sample added a spike

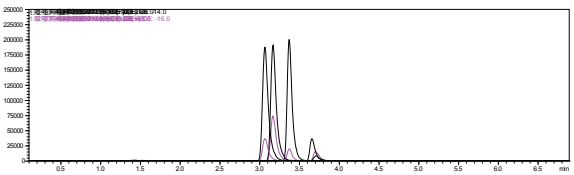

Fig.3. Chromatogram of TIC standard substance solution of mixed standards in which papaverine, nacodine and Thebaine have a concentration of 2 μg/L morphine and codeine have a concentration of 5 μg/L

9. 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

10. Conclusion

This experiment established HPLC-MS/MS method for detection of papaverine, morphine, narcodine, codeine and thebaine in hot pot food. Samples which added a spike of 15 or 37.5 μg of were tested, and the recovery rate was between 79.1% and 97.4%. QuEChERS method is stable and its reproducibility is good, indicating this method can be used to determine the content of papaverine, morphine, narcodine, codeine and thebaine in hot pot food.

QuEChERS Methods for Determining Phthalate in Food SN/T 3147-2017

1. Range of Application

It is suitable for the determination of oil phthalates (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: accurately transfer the standard storage solution (1 mg/mL 1.1 mL) 1.0 mL and dilute with acetone to 10 mL with concentration of 100 g/mL.
2) Standard working solution: accurately transfer 1 mL standard storage solution to have a constant volume to 10 mL with hexane, and the concentration is 10 g/mL.
3) N-hexane saturated with acetonitrile: take 100 mL of acetonitrile and 100 mL of n-hexane, mix them well, and take the n-hexane which is saturated with acetonitrile in the upper layer.
4) Acetonitrile saturated with n-hexane: take 100 mL acetonitrile and 100 mL n-hexane, mix them well, and take acetonitrile which is saturated with n-hexane in the lower layer.

3. Extraction Steps

1) Weigh 5 g of the samples in the glass tube and add 2 mL n-hexane which is saturated with acetonitrile. After vortex, add 4 mL acetonitrile which is saturated with n-hexane with 10 min ultrasound time. Centrifuge for 5 min (3000 r/min), carefully remove the lower solution into another glass tube. Add 4 mL n-hexane-saturated acetonitrile, and repeat the extraction to mix two extracts.
2) Place the extract in a water bath at 40℃, blow with nitrogen to nearly dry, then accurately add 5 mL acetonitrile, 50 mg PSA, 50 mg C18E and 150 mg $MgSO_4$. Centrifuge for 5 min (3000 r/min) after vortex mixing, and take the supernatant for testing.

4. Precautions

1) Spike level: Add 0.025 mL at 10 μg/mL to 5 g sample to have a constant volume of 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℃
Temperature Program	The initial column temperature was 60 ℃ for 1 min. Rise the temperature to 220 ℃ /min at 20 ℃/min for 1 min. Then rise to 250℃ by 5 ℃/min, keep 1 min. Continue to rise to 290℃ at 20 ℃/min for 7.5 min
Carrier Gas	High purity helium (>99.999%)
Injection	Split injection
Injection Volume	1 μL

5.2 Mass spectrum conditions

Ionization methods	Electron bombardment ionization source (EI)
Ionization energy	70 eV
Transmission line temperature	280 ℃
Ion source temperature	230 ℃
Monitoring way	Ion scan (SIM)
Solvent delay	7.5 min

6. Chromatogram or Spike Recovery Results

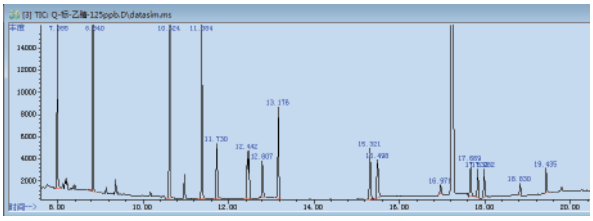

Fig.1. Blank chromatogram of phthalates standard substance at 0.125 μg/mL

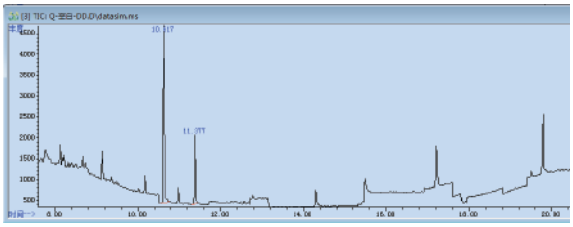

Fig.2. Chromatogram of blank reagent

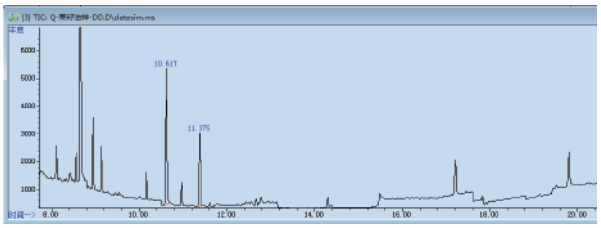

Fig.2. Chromatogram of rapeseed oil samples after purification

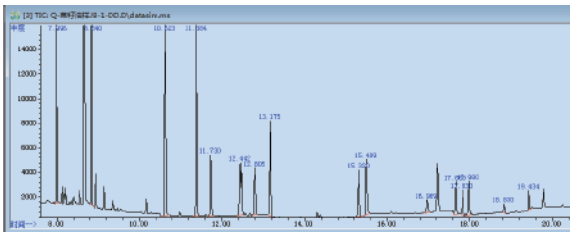

Fig.4. Chromatogram of rapeseed oil samples added a spike of 0.05 mg/kg after purification

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

No.	Name	Retention Time	Peak Width	Area
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	DPP	13.176	0.032	149986
9	DHXP	15.321	0.051	97533
10	BBP	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 table of sample added a spike of 0.05 mg/kg

7. Ordering Information

P/N	Product	Description
00821-32291	Caps and septas	Pre-slit red PTFE/white silicone septa, 9 mm blue short screw-thread polypropylene cap, 6 mm centre hole, 100 pcs/pk
00821-40927	Vials	2 ml wide opening short screw-thread vial with write-on spot, clear, 100 pcs/pk

00533-20020	Clean-up tube	2 ml, AOAC method,150 mg $MgSO_4$, 50 mg PSA, 50 mg C18E, for fatty and waxy fruits and vegetables,100 pcs/pk
03904-22001	GC column	WM-5MS, 30 m×0.25 mm×0.25 μm

QuEChERS Methods for Determining 15 Kinds of PAHs in Foods GB 5009.265-2016

1. Range of Application

It is suitable 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 with ultrasonic extraction for 15 min and centrifuge at 8000 r/min for 5 min. Transfer the supernatant to another centrifuge tube of 50 mL. Continue to add 10 mL n-hexane to the residue with ultrasonic extraction for 15 min, followed by centrifugation at 8000 r /min for 5 min. Mix the supernatant and blow with nitrogen to nearly dry, and add 3 mL of acetonitrile in centrifuge tube. After fully mixing, slowly blow with nitrogen to remove all the n-hexane, then have a constant volume of 2 ml with acetonitrile. Finally, keep it for purification.

3. Purification Steps

Transfer liquid to be purified to the (005PM-048-50) centrifuge tube, and vortex for 5 min, centrifuge at 6000 r/min for 5 min. Then the supernatant needs to be filtered 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 following table

Time/min	Mobile phase A/ acetonitrile	Mobile phase B/ water
0	50	50
5	50	50
20	100	0
35	100	0
36	50	50
45	50	50

Tab. 1: Gradient elution procedures

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

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, terylene, benzo (b), Fluoranthrene, benzo (k) fluoranthrene, Benzo (a) pyrene, II Anthracene (a, h), benzo (g, h, i) pyrene
28.9-45	100	Ninhydrin (1, 2, 3-cd) pyrene

Tab.2: Fluorescence detection wavelength gradient procedure

5. Chromatogram or Spike Recovery Results

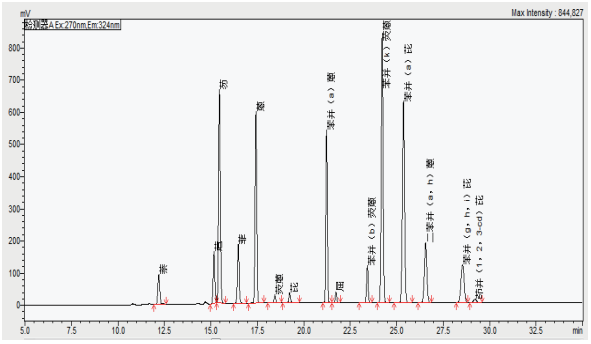

Fig.1. Chromatogram of 15 kinds of PAH standard substances, 20 μg/L

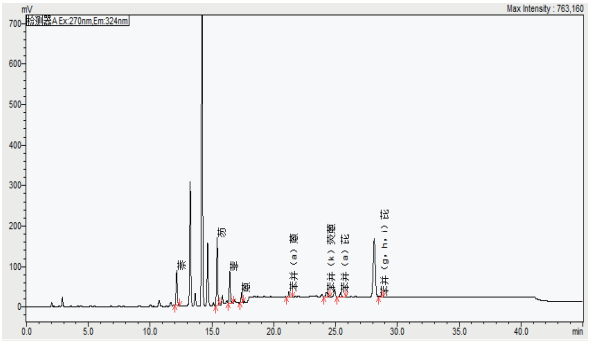

Fig.2. Blank chromatogram of flour

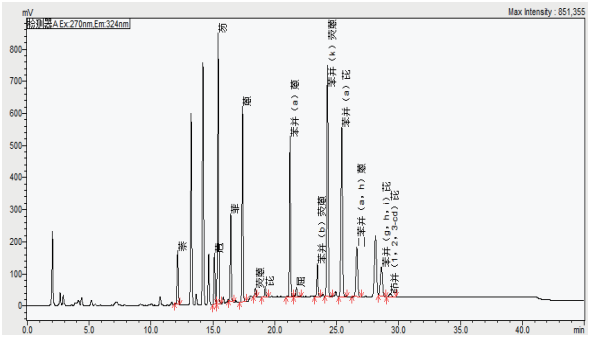

Fig.3. Chromatogram of flour sample added a spike of 20.0 μg/kg

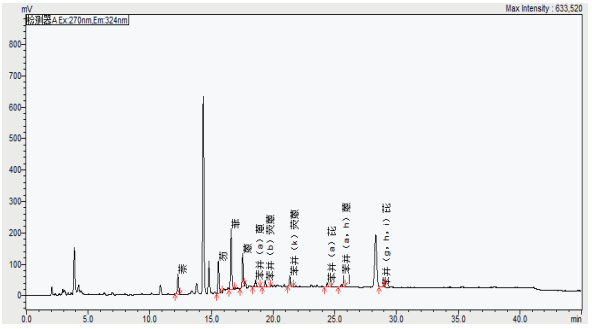

Fig.4. Blank chromatogram of vegetables

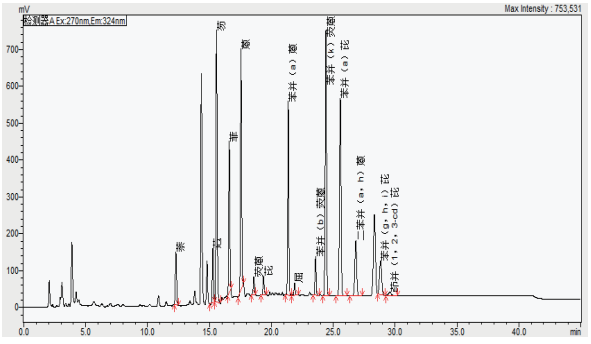

Fig.5. Chromatogram of vegetable sample added a spike of 20.0 μg/kg

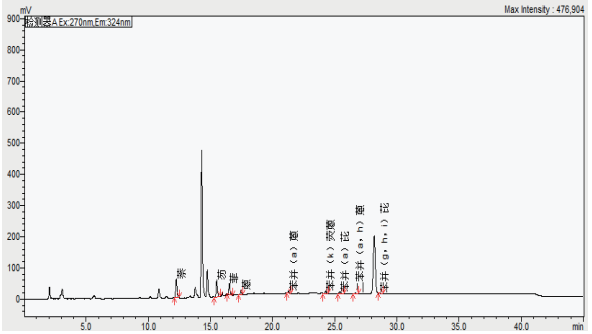

Fig.6. Blank chromatogram of fruits

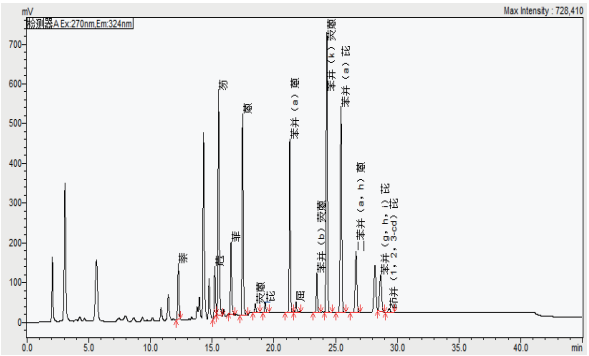

Fig.7. Chromatogram of fruit sample added a spike of 20.0 μg/kg

Other substrates: The recovery rate of the experimental vegetable substrates ranged from 74.54% to 107.448%, and the RSD (n=3) value ranged from 0.52% to 3.11%. The recovery rate of fruit matrix 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%
Acenaphthene		96.34%	1.80%
Fluorene		96.05%	2.83%
Phenanthrene		107.48%	2.88%
Anthracene		92.81%	1.66%
Fluoranthene		99.13%	1.46%
Pyrene		106.45%	2.37%
Benzanthracene		92.46%	1.94%
Tracy		100.34%	0.52%
Benzo (b) fluoranthene		88.25%	1.83%
Benzene (k) fluoranthene		87.26%	1.69%
Benzo (a) pyrene		85.60%	1.56%
Diphenyl (a, h) anthracene		84.59%	1.35%
Benzo (G, H, I) pyrene		74.54%	2.55%
Ninhydrin (1, 2, 3-CD) pyrene		81.65%	2.27%

Tab 3: Spike Recovery

6. Ordering Information

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	NY, 13 mm×0.22 μm, 100 pk

QuEChERS Methods for Determining 15 Kinds of PAHs in Water HJ 478-2009

1. Range of Application

It is suitable 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 HPLC

2. Extraction Steps

Take 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. Through 15 min of ultrasonic extraction, centrifuge at 6000 r/min for 5 min. Transfer the supernatant to another 50 mL of clean centrifuge tube and continue to add 10 mL of n-hexane in the residue, keep ultrasonic extraction for 15 min, then centrifuge at 6000 r/min for 5 min. Mix the supernatant and blow with nitrogen to nearly dry, then add 3 mL acetonitrile into the centrifuge tube.

3. Purification Steps

Transfer the liquid to be purified to 005PM-048-50 centrifuge tube, then vortex for 5 min and centrifuge at 6000 r/min for 5 min. Let the supernatant be filtered 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 following table

Time/min	Mobile phase A/ acetonitrile	Mobile phase B/ water
0	50	50
5	50	50
20	100	0
35	100	0
36	50	50
45	50	50

Tab 1: Mobile phase elution procedures

Flow rate: 1.5 mL/min

Column temperature: 30 ℃

Injection volume: 20 L

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

Time/min	Excitation wavelength	Target
0-16	270/324	Naphthalene, acenaphthylene, fluorene
16-18	248/375	Phenanthrene, anthracene
18-28.9	292/410	Fluoranthene, pyrene, benzo (a) fluoranthene, Terylene, benzo (b) fluoranthene, benzo (k) fluoranthene, benzo (a) pyrene, dibenzo (a, h) anthracene, benzo (g, h, i) pyrene
28.9-45	100	Ninhydrin (1, 2, 3-CD) pyrene

Tab 2: Fluorescence detection wavelength gradient procedure

5. Chromatogram or Spike Recovery Results

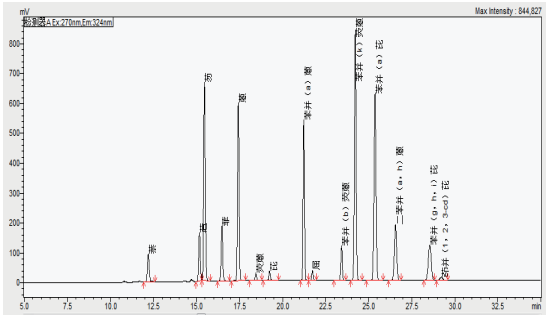

Fig.1. Chromatogram of 15 kinds of PAH standard substances, 20 μg/L

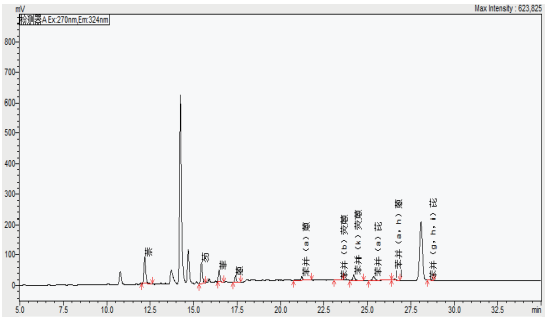

Fig.2. Blank chromatogram of water sample

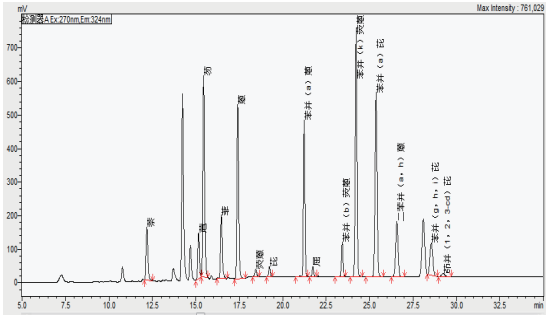

Fig.3. Chromatogram of water sample added a spike of 2.0 μg/L

Classification	Spike Level μg/L	Recovery Rate/%	RSD/%
Naphthalene	2.0	76.88%	1.04%
Acenaphthene		87.51%	0.91%
Fluorene		86.56%	1.42%
Phenanthrene		93.39%	2.02%
Anthracene		91.59%	1.12%
Fluoranthene		97.53%	0.61%
Pyrene		94.49%	0.62%
Benzanthracene		89.73%	0.22%
Tracy		92.36%	0.06%
Benzo (b) fluoranthene		88.65%	0.17%
Benzene (k) fluoranthene		87.92%	0.43%
Benzo (a) pyrene		86.59%	0.57%
Diphenyl (a, h) anthracene		87.47%	0.34%
Benzo (g, h, i) pyrene		76.84%	1.15%
Ninhydrin (1, 2, 3-cd) pyrene		85.62%	0.84%

Tab 3: Standard recovery of water sample

6. Ordering Information

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	NY, 13 mm×0.22 μm, 100 pk

Bridging Separation Techniques

Welch Materials.Inc.
Email: info@welchmat.com
web: www.welch-us.com