Agela Technologies

BONNA-AGELA TECHNOLOGIES

Cleanert® SPE

Media and Accessories

- SPE Cartridges and Plates
- SPE Bulk Media
- Clean-up Cartridges for Ion Chromatography
- Accessories
- Applications

Bonna-Agela Technologies

BETTER SOLUTIONS FOR CHROMATOGRAPHY

SPE Columns

Cleanert® SPE Cartridges

Cleanert® IC
Clean-up Cartridges

96-Well Plates

HPLC Columns

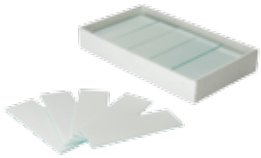
Venusil® & Promosil® HPLC Columns

Venusil® AA Kit

Flash Chromatography Products

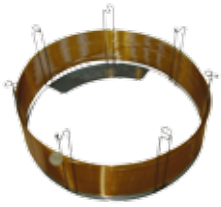
Claricep™ Flash Columns

CHEETAH™ Flash Purification System



TLC Plates

Consumables Products



GC Columns

Clarinert™ Syringe Filters

Sample Accessories

Bonna-Agela Technologies — A Global Supplier for Chromatography Solutions

2012 Message From Bonna-Agela Technologies

As Bonna-Agela is poised to enter its fifth year with confidence and pride in its innovative separation, purification, and sample preparation products, we would like to thank our many loyal customers for your continuous support and trust. With your support and our effort in delivering the highest quality products to you, our company has grown remarkably. This has allowed us to expand our research and development effort, and thus introduce more innovative products to better service your application needs.

We had tremendous accomplishments: We cataloged over one thousand different products. Our manufacturing and R&D operation were certified in compliance with ISO 9001 and passed many quality audits by customers and distributors, including VWR International. As a global wide company, we not only have our own international sales force but have also formed a marketing alliance with VWR. This will allow us to reach higher goals and to provide our customers with even better quality products and faster service in the new year.

Our mission statement and commitment:

- Provide products with our innovative technologies at the best performance to cost ratio.
- Deliver products with guaranteed quality.
- Provide global support with quick responses.

How to Place Orders

Our office is open from 9:00 am to 6:00 pm Eastern Standard Time, Monday through Friday.

To place an order or receive a quote, you may choose from the following contacts:

Bonna-Agela Technologies US
2038A Telegraph Rd.
Wilmington, DE 19808, USA
Tel: (302) 438 8798
Fax: (302) 636 9339
Website:http://www.bonnaagela.com
E-mail: info@bonnaagela.com

Bonna-Agela India
G-212, Second Floor , Sector-63 , Noida-201301 (U.P) , India
Phone : 91-120-4225466-71
Fax : 91-120-4225465

Bonna-Agela China
179 South Street, Teda West Zone, Tianjin 300462, China
Tel:+86(22)25321032/7023
Fax:+86(22)25321033

Please include the following information with your order or request: Account number (if you have one), purchase order number, contact name, organization name, shipping and billing address, telephone number, fax number or email address, product number, brief description and quantity, method of payment and preferred method of delivery. A written confirmation will be sent to you by email or fax. We accept business checks, wire transfers and major credit cards as methods of payment.

Checks:
Please make checks payable to:
Bonna-Agela Technologies Inc. and send to:
Bonna-Agela Technologies Inc.
2038A Telegraph Road, Wilmington, DE 19808, USA

Wire Transfer:
Please contact us by phone, fax or email for account information.

Credit Cards: (USA Only)
Please include card type and number, expiration date, and card holder name. Due to security concerns, please do not email the information. Please call or send a fax to provide your credit card information.

Terms and Conditions

PLEASE READ THESE TERMS BEFORE ORDERING. IF YOU HAVE ANY QUESTIONS, PLEASE DO NOT HESITATE TO CONTACT US AND OUR STAFF WILL BE GLAD TO ASSIST YOU.

Acceptance and Availability
All orders placed are subject to the agreement of Bonna-Agela Technologies Inc. The catalogue does not constitute an engagement of the company to sell all listed products. You are guaranteed to be notified at the time of ordering if the ordered items are in back-order or discontinued.

Price and Payment
The prices are in effect at the time of printing. Bonna-Agela Technologies reserves the right to change the prices without notice, though we do our best to provide our customers with advance notice. The prices quoted at the time of ordering will be guaranteed. The general payment term is net 30 days, F.O.B., Newark, Delaware,USA. However we reserve the right to ask for prepayment if customers' account information is not satisfactory. A 1.5% per month service charge will be added to delinquent accounts. If a purchase order is less than $1000.00, a $50.00 extra charge will be added to the invoice.

Changes
Bonna-Agela Technologies reserves the right to change product specifications, quantities, designs or prices without prior notice and without liability for such changes.

Shipping Policy
The standard shipping method is 2-day FedEx within the United States and Canada. We will try to accommodate requests for other shipping methods if they are available. All shipping and handling charges will be billed separately. Should you receive damaged goods, it is imperative that you notify us immediately and save all packing materials for inspection by the carrier.

Application
All products in this catalog should be used for laboratory or manufacturing use only. They are not intended for direct medicinal or food use. Bonna-Agela Technologies assumes no liability for any misuse of the products.

Returns
Bonna-Agela Technologies tries to accommodate all requests for returns of unused goods. However, return of some items may be restricted by the original manufacturers. Please contact us for return authorization before returning any items. A restocking charge may apply to certain products.

Warranty
All Bonna-Agela Technologies products are warranted to be free of defects in materials and workmanship. They are not warranted for any other particular purpose. Bonna-Agela Technologies shall not under any circumstance be liable for any incidental, consequential or compensatory damage in conjunction with its products. The maximum liability shall not exceed the invoice price of the product.

Best Value
Guaranteed Product Quality
Innovation to Benefit Customers

SPE Columns and Wellplates

SPE Columns and Wellplates

SPE Columns and Wellplates

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SPE Columns and Wellplates

Introduction

Solid phase extraction (SPE) is one of the most used techniques in sample preparation. It is replacing the conventional time-consuming liquid-liquid extraction (LLE) method. The process of SPE is a miniature version of liquid column chromatography. SPE, utilizing the same type of stationary phases as used in liquid chromatography columns, are practiced in different mechanisms including normal phase, reversed phase, ion-exchange, affinity chromatography and mixed-mode in the fields of agricultural and food, bio-analytical, environmental and forensic applications. Bonna-Agela as a leader of chromatographic media products offers a variety of polymer and silica based SPE products.

Cleanert® SPE Cartridges, Well Plates and Media

OMM SPE Products (Optimized Molecular Modification)— PEP series, PAX, PCX, PWAX, PWCX and PS are all based on polystyrene/divinylbenzene while each phase has different functionality and unique selectivity. They are highly recommended for the extraction of a wide range of compounds in pharmaceutical, agricultural, food, and environmental industries. Average particle diameter: 40-60 μm and 30 μm; Average pore size: 70 Å; Specific surface area: 600 m^2/g.

MAS (Multi-functional Impurity Adsorption SPE)
MAS is a simple sample treatment method that applies multi-functional impurity adsorption to remove most of the interferences in samples. It achieves a faster and easier approach in comparing to SPE, and it can be used in clinical and food analysis.

SLE (Solid Supported Liquid/liquid Extraction) Cartridges and Well-plates
Specially treated diatomite materials are packed in columns and well plates. The liquid/liquid extraction is run on the surface of the materials, which is easily automated in parallel processing for time saving.

Bonna-Agela is one of the very few original manufacturers of diatomite for chromatography. Bonna-Agela can provide diatomite at different pH values, as well as a variety of surface modification, to meet different application needs. The particle size distribution was narrowed and the surface activity was controlled to avoid unwanted adsorption of analytes.

Bonded Silica SPE Cartridges

Functionalities include AQ C18, C18, C18-N, C8, NH_2, COOH, Silica, PSA, PRS, SCX, SAX; All of them are made of high quality and low metal silica particles. Using the special surface modification methodology, the activity of silica surface is reduced largely, which in turn will reduce the tailing of compounds and will ensure high recovery and reproducibility.

Characteristics of irregular particles:
Average particle diameter: 40-60 μm
Average pore size: 60 Å
Volume of pore: 0.8 mL/g
Specific surface area: 480 m^2/g

Characteristics of spherical particles:
Average particle diameter: 40-60 μm
Average pore size: 60 Å
Volume of pore: 0.9 mL/g
Specific surface area: 600 m^2/g

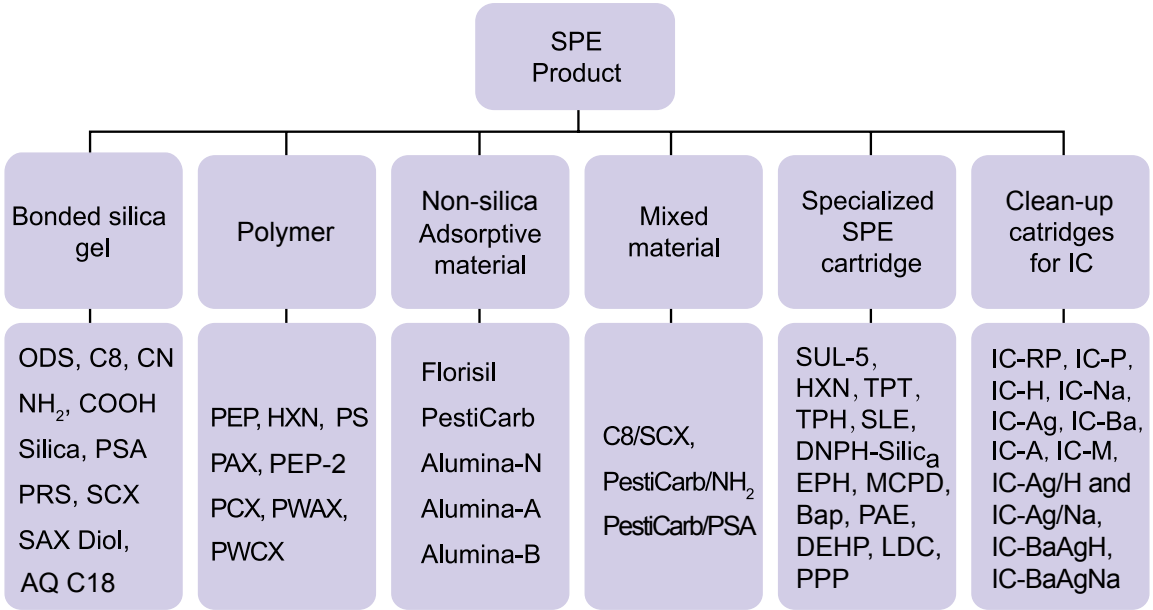
Non-silica Adsorption Phase Cartridges-Florisil, PestiCarb, Alumina (N, A, B). They are commonly used to remove polar interference from non-polar samples. Cleanert adsorption sorbents have high purity, high recovery and good reproducibility. They are widely used in sample preparation for environmental and food analyses.

Mixed and Layered Phases-C8/SCX, PestiCarb/NH_2, PestiCarb/PSA

Specialized Phases- SUL-5 (Determination of five sulfonamides in pork)
TPH—Triple-phase for herb (Pesticide residues analysis)
HXN (Determination of 10 sulfonylureas herbicide residues in soil samples
MCPD (chloropropanol detection in food)
PAE (plasticizer detection in food containing fats and oils)
Bap (benzopyrene detection in food containing fats and oils)
LRC (Large receiver column)
PPP (Protein precipitation plate)
TPT—Triple-phase for tea leaves (Pesticide residues analysis)
DNPH-Silica (Aldehyde ketone analysis in air)
DEHP (plasticizer detection in aqueous sample)
EPH (Extractable petroleum hydrocarbon analysis)
LDC (Large disk column for water analysis)

Clean-up Cartridge for Ion Chromatography: IC-RP, IC-P, IC-H, IC-Na, IC-Ag, IC-Ba, IC-A, IC-M, IC-Ag/H and IC-Ag/Na; IC-BaAgH, IC-BaAgNa; Remove matrix interferences such as phenolics, metals, cations, anions, or hydrophobic substances encountered in many ion chromatography applications

Specialized cartridge and wellplate format: Deep array 96-well plate series; Glass tube and PTFE frit without plasticizer.



Classification According to the Type of the Products

1. SPE Cartridges (Figure1)

Figure 1 SPE cartridge

- The common SPE cartridges consist of three parts: high purity polypropylene tube, porous PE frit and packing materials. (40-60 μm, 90 μm).
- Common specification: 100 mg/1 mL, 200 mg/3 mL, 500 mg/3 mL and1 g/6 mL etc. Take 100 mg/1 mL of cartridge as an example, 100 mg is the quality of packings and 1 mL is the volume of the tube.
- Disposable usage: SPE is disposable to avoid cross-contamination.

2. 96-well plates (Figure 2)

Figure 2 96-well plate

96-well plate is the product for high throughput applications. Each well contains a small amount of sorbents (10-100 mg) with max. volume of every well is 2 mL . With the main applications in the cleanup of multi-sample in the area of bio-analysis and clinical analysis, it is compatible with the automated sample handing work station for high throughput operation.

3. Micro array 96-well plate series (Figure 3)

Figure 3 Cleanert® micro array 96-well plate

Micro array 96-well plate allows a small amount of sample loading and elution.
Array series provide base plate and individual removal wells, and it allows different SPE materials in one plate, which is suitable for method development.

4. Deep array 96-well plate (Figure 4)

Figure 4 Cleanert® deep array 96 well plates

Deep array 96-well plate have a bigger room for sample and elution solution than regular 96-well plates.

5. Clean-up Cartridges for Ion-chromatography (Figure 5)

Figure 5 Cleanert® IC cartridges

Cleanert® IC series are used for removing matrix interferences such as phenolics, metals, cations, anions, or hydrophobic substances encountered in many ion chromatography applications.

6. MAS-QuEChERS (Figure 6)

Figure 6 Cleanert® MAS cartridge and plates

MAS (Multi-mechanism Adsorption SPE) -QuEChERS, which is uses a centrifuge tube format, has been widely applied in pesticide and veterinary drug residue analysis in vegetable, fruit, grain, and animal tissues.

7. Bulk Media (Figure 7)

Figure 7 Bulk media

Bulk packing materials are available for customized uses in sample preparation.

8. Cartridge Format

General cartridge

Flangeless cartridge

LRC cartridge

LDC cartridge

Glass cartridge

Featured Products

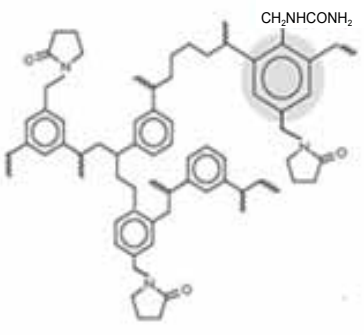
OMM Technology (Optimized Molecular Modification) for SPE

Bonna-Agela SPE products have been developed based on a thorough understanding of interactive natures of chemical molecules. Our SPE products can thus better meet customer's needs. Our R&D results demonstrated that the adsorption/desorption property of the polymeric SPE materials is regulated by the types of the functional groups and the degree of substitution of the surface modification.

In general, modification with electron donor groups will help to retain the electron-deficient molecules, while modification with electron-withdrawing groups will prefer to retain the electron-rich molecules. Different SPE materials have been developed by incorporating proper types of functional groups and the degree of substitutions on the surface, and thus providing optimized and balanced performance for all types of molecules.

Cleanert® PEP-2 *NEW !*

Cleanert® PEP-2 is made of polydivinylbenzene on which the surface is functionalized with vinyl pyrrolidone and urea. In addition to a balanced hydrophilic and hydrophobic property, the PEP-2 is also an electron donor in a polar-polar interaction, as well as a strong hydrogen donor and acceptor in hydrogen bonding. As a result, the PEP-2 can retain most of acidic, basic and neutral polar compounds without adjusting the pH of the samples. PEP-2 has stronger retention of polar compounds than PEP.



Particle Characteristics

Functionalized polymer sorbents; Average particle size: 40-60 µm. (30 µm are optional)
Average pore size: 70 Å; Surface area: 600 m^2/g.

Suggestion Processing Method

Neutral and Basic Compounds (metoprolol)		Acidic Compounds (salicylic acid)	
Condition	4 mL MeOH, 4 mL Water	Condition	4 mL MeOH, 4 mL Water
Sample Loading	1 mL sample (aqueous)	Sample Loading	1 mL sample (aqueous)
Wash	2 mL Water (or 5% organic solvent)	Wash	2 mL Water (or 5% organic solvent)
	4 min drying time		4 min drying time
Elute	Elution with 4 mL MeOH (no need to adjust the pH)	Elute	Elution with 4 mL MeOH (add some acid can be helpful for strong acid compounds elution)

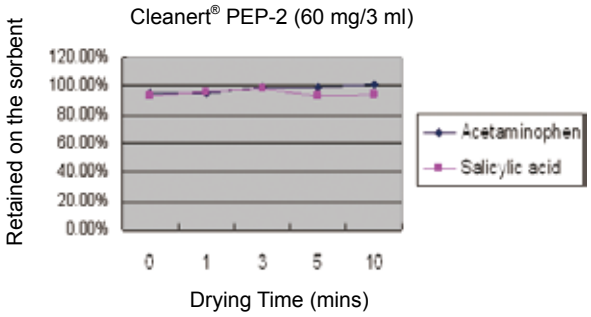
Ordering Information

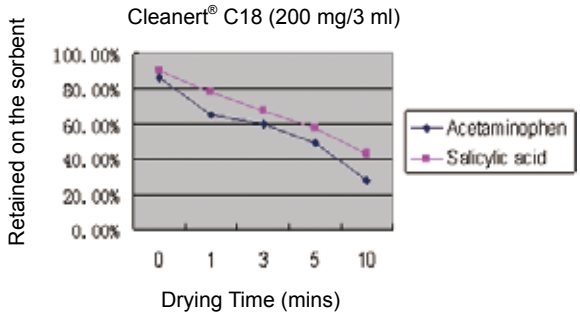
Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PEP-2	30 mg	1 mL	100	PE0301-2
	60 mg	3 mL	50	PE0603-2
	30 mg	1 mL, flangeless	100	PE0301-2R
	60 mg	3 mL, flangeless	50	PE0603-2R
	100 mg	3 mL	50	PE1003-2
	200 mg	6 mL	30	PE2006-2
	500 mg	6 mL	30	PE5006-2
	500 mg	6 mL, flangeless	30	PE5006-2R
	30 mg/well	2 mL	96-well plate	PE0302-2W
	50 mg/well	2 mL	96-well plate	PE0502-2W
	10 g	-	-	PE0010-2
	100 g	-	-	PE0100-2

Good Water-Wettability

The Cleanert® PEP-2 (polar polymer) sorbent is a unique hydrophilic-lipophilic balanced material. It provides excellent wettability since the hydrophobic surface of the sorbent is highly modified with polar functional groups. The SPE mechanism follows the reversed phase separation principle.

Effect of Drying on Recovery - PEP-2 Versus C18 Sorbents



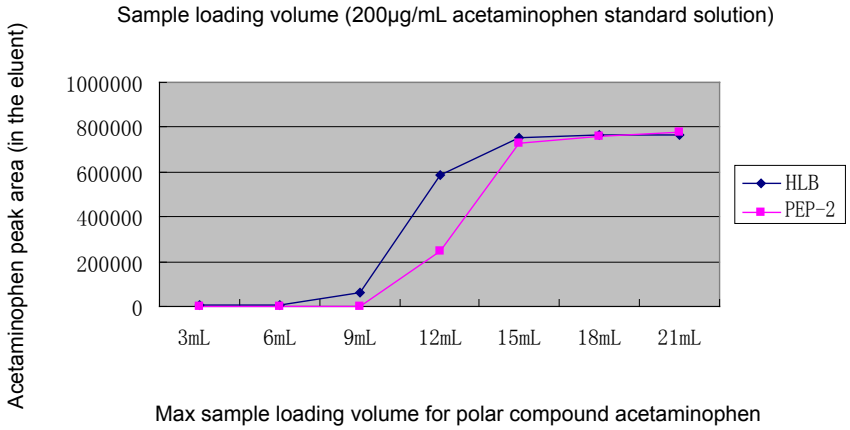


High Retention for Polar Compounds and Acid Compounds

The electron donating and with-drawing functionalities of the sorbent allow it to have enhanced retention for polar compounds.

	PEP	PEP-2	Brand P	Brand W
Caffeine	98.58%	100.37%	100.12%	103.53%
Metoprolol	80.11%	88.25%	91.23%	90.05%
Salicylic acid	21.36%	109.73%	8.03%	18.79%

The recoveries of three compounds on different SPE materials

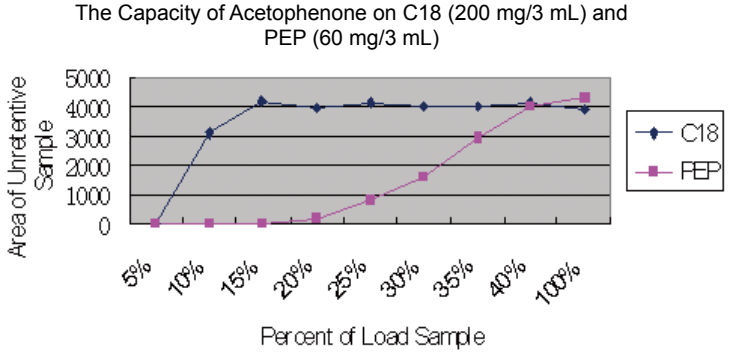


Cleanert® PEP

Cleanert® PEP is made of polydivinylbenzene on which the surface is functionalized with vinyl pyrrolidone. The material has a balanced hydrophilic and hydrophobic property and can be used in the entire pH range of 1-14.
PEP can be used to extract a variety of polar and non-polar compounds. Some highly hydrophilic compounds which have little retention on C18 columns, such as chlorinated phenols, phosphate esters and drug metabolites, can be effectively retained on PEP.

High Capacity

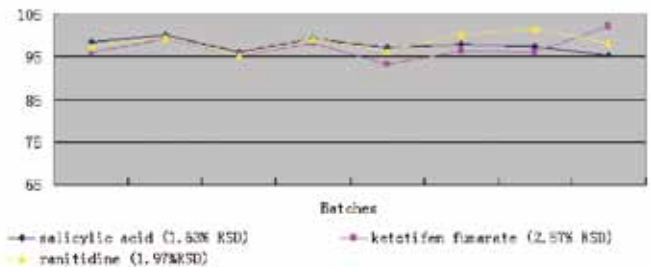
The Cleanert® PEP series sorbents have 2-3 x more surface area and show a dramatic increase of sample capacity compared to silica-based C18. The volume of elution solvents can be reduced, and subsequently the total operation time (including solvent evaporation) will be shortened.



Excellent Batch-to-Batch Reproducibility

The OMM technology offers consistent surface modification, resulting in excellent batch-to-batch reproducibility.
Multiple batches of Cleanert PEP series have been successfully used for various compounds with consistent results.

Batch to batch reproducibility of PEP

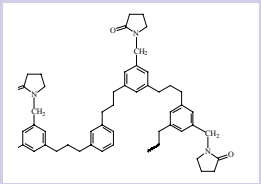


Particle Characteristics

Functionalized polymer sorbents; Average particle size: 70-90 µm (40-60 µm and 30 µm are optional) .
Average pore size: 70 Å; Surface area: 600 m^2/g.

For Applications, see page 59~60, 90~93

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PEP 70-90 µm 	30 mg	1 mL	100	PE0301
	60 mg	1 mL	100	PE0601
	60 mg	3 mL	50	PE0603
	30 mg	1mL, flangeless	100	PE0301-R
	60 mg	3mL, flangeless	50	PE0603-R
	100 mg	3 mL	50	PE1003
	200 mg	6 mL	30	PE2006
	500 mg	6 mL	30	PE5006
	500 mg	6mL, flangeless	30	PE5006-R
	10 g		bottle	PE0010
	100 g		bottle	PE0100
	30 mg	2 mL	96-well plate	PE0302-W
	50 mg	2 mL	96-well plate	PE0502-W

Cleanert® PAX (RP/Strong Anion Exchange)

Description

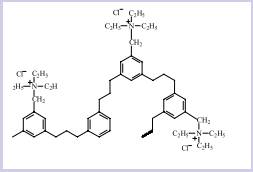
It is designed to overcome the limitations of traditional silica based mixed-mode SPE sorbents such as C18/SAX. It is a RP/strong anion exchange mixed-mode polystyrene/divinylbenzene sorbent, stable from pH 0-14.

Particle Characteristics

Based on functioalized polystyrene/divinylbenzene;
Average Particle Diameter: 40-60 µm (30 µm are optional); Average Pore Size: 70 Å; Porosity: 1.2 mL/g; Specific Surface Area: 600 m^2/g

For applications, see page 63, 96, 101, 114

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PAX 	30 mg	1 mL	100	AX0301
	60 mg	1 mL	100	AX0601
	60 mg	3 mL	50	AX0603
	30 mg	1mL, flangeless	100	AX0301-R
	60 mg	3mL, flangeless	50	AX0603-R
	100 mg	3 mL	50	AX1003
	200 mg	6 mL	30	AX2006
	500 mg	6 mL	30	AX5006
	500 mg	6mL, flangeless	30	AX5006-R
	30 mg/well	2 mL	96-well plate	AX0302-W
	50 mg/well	2 mL	96-well plate	AX0502-W
	10 g	-	-	AX0010
	100 g	-	-	AX0100

Cleanert® PWAX (RP/Weak Anion Exchange)

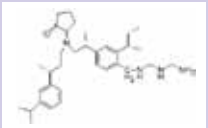
Description

Cleanert® PWAX provides the dual modes of retention, weak anion exchange and reversed phase on a stable polymer sorbent, which improves the retention for acidic analytes.

Particle Characteristics

Based on partially functionalized aminopolystyrene/divinylbenzene;
Average Particle Diameter: 40-60 µm; Average Pore Size: 70 Å; Porosity: 1.2 mL/g; Specific Surface Area: 600 m^2/g.

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PWAX 	30 mg	1 mL	100	WA0301
	60 mg	3 mL	50	WA0603
	30 mg	1 mL, flangeless	100	WA0301-R
	60 mg	3 mL, flangeless	50	WA0603-R
	100 mg	3 mL	50	WA1003
	200 mg	6 mL	30	WA2006
	500 mg	6 mL	30	WA5006
	500 mg	6 mL, flangeles	30	WA5006-R
	30 mg/well	2 mL	96-well plate	WA0302-W
	50 mg/well	2 mL	96-well plate	WA0502-W
	10 g	-	-	WA0010
	100 g	-	-	WA0100

Suggested Method for PAX and PWAX

Condition	Equilibration	Load	Wash 1	Wash 2	Elute	Evaporate
Methanol	Water	Load sample	2% $NH_3 \cdot H_2O$ in water	methanol	2% formic acid in methanol	and Reconstitute

Cleanert® PCX (RP/Strong Cation Exchange)

Description

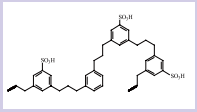
Cleanert® PCX is a mixed-mode, strong cation exchange sorbent which provides dual retention modes of reverse-phase and cation-exchange. It has high surface area and a wide usable pH range of 0-14.

Particle Characteristics

Based on Functionalized polystyrene/divinylbenzene;
Average Particle Diameter: 40-60 µm (30 µm are optional); Average Pore Size: 70 Å; Porosity: 1.2 mL/g; Specific Surface Area: 600 m^2/g.

For applications, see page 54, 82, 85, 97

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PCX 	30 mg	1 mL	100	CX0301
	60 mg	1 mL	100	CX0601
	60 mg	3 mL	50	CX0603
	30mg	1 mL, flangeless	100	CX0301-R
	60mg	3 mL, flangeless	50	CX0603-R
	100 mg	3 mL,	50	CX1003
	200 mg	6 mL	30	CX2006
	500 mg	6 mL	30	CX5006
	500mg	6 mL, flangeless	30	CX5006-R
	10 g	bottle		CX0010
	100 g	bottle		CX0100
	30 mg	2 mL	96-well plate	CX0302-W
	50 mg	2 mL	96-well plate	CX0502-W

Cleanert® PWCX (RP/Weak Cation Exchange)

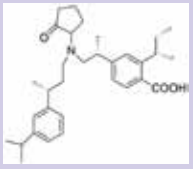
Description

Cleanert® PWCX provides dual modes of retention, weak cation exchange and reversed phase on a stable polymer sorbent, which improves the retention for basic analytes.

Particle Characteristics

Based on partially functionalized polystyrene/divinylbenzene;
Average Particle Diameter: 40-60 µm (30 µm are optional); Average Pore Size: 70 Å; Porosity: 1.2 mL/g; Specific Surface Area: 600 m^2/g.

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PWCX 	30 mg	1 mL	100	WC0301
	60 mg	3 mL	50	WC0603
	30 mg	1 mL, flangeless	100	WC0301-R
	60 mg	3 mL, flangeless	50	WC0603-R
	100 mg	3 mL	50	WC1003
	200 mg	6 mL	30	WC2006
	500 mg	6 mL	30	WC5006
	500 mg	6 mL, flangeless	30	WC5006-R
	30 mg/well	2 mL	96-well plate	WC0302-W
	50 mg/well	2 mL	96-well plate	WC0502-W
	10 g	-	-	WC0010
	100 g	-	-	WC0100

Suggested Method for PCX and PWCX

Condition Methanol	Equilibration Water	Load Load sample	Wash 1 2% formic acid in water	Wash 2 methanol	Elute 2% $NH_3 \cdot H_2O$ in methanol	Evaporate and Reconstitute

Cleanert® PS

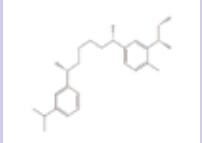
Description

Cleanert® PS is made of non-substituted polydivinylbenzene. It has larger surface areas (>600 m^2/g.) and thus greater capacity than reversed phase bonded silica. Cleanert PS can be used for the extraction of non-polar and polar compounds.

Particle Characteristics

Based on polystyrene/divinylbenzene; Average Particle Diameter: 40-60 µm;
Average Pore Size: 70 Å; Porosity: 1.2 mL/g; Specific Surface Area: 600 m^2/g.

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PS 	30 mg	1 mL	100	PS0301
	60 mg	1 mL	100	PS0601
	60 mg	3 mL	50	PS0603
	30 mg	1 mL, flangeless	100	PS0301-R
	60 mg	3 mL, flangeless	50	PS0603-R
	100 mg	3 mL	50	PS1003
	200 mg	6 mL	30	PS2006
	500 mg	6 mL	30	PS5006
	500 mg	6 mL, flangeless	30	PS5006-R
	10 g	bottle		PS0010
	100 g	bottle		PS0100
	30 mg	2 mL	96-well plate	PS0302-W
	50 mg	2 mL	96-well plate	PS0502-W

Cleanert® Micro array 96-well plate

Micro array 96-well plates can be packed with less sorbent, which allows a small amount of sample loading and elution solvent. The micro array 96-well plates use a special array design; loose cartridges use an internally tapered well design and make a high sorbent bed. Even a small mass of sorbent is embedded in it, no breakthrough will happen. All the cartridge can be removed from the base plate, and assembled by hand; Differen sorbents can be used in one base plate, suitable for method development.
The Micro array well plate optimizes the configuration of the sorbents (PEP-2, PCX, PAX, PWCX, and PWAX), using 30um particle size sorbent to enhance the efficiency of column.
Micro plates, which allow low-elution volume, enable sensitive, robust, reproducible results without time-consuming evaporation and reconstitution.

Innovative features of micro array well plate

- Minimum eluent
- No evaporation and reconstitution
- Suitable for small sample loading volumes

Cleanert® micro array 96 well plates

Ordering Information

Material	Sorbent	Vol	Specification	Cat. Number
PEP	5 mg	1 mL/well	Micro array 96-well plate	PE00501-MW
	10 mg	1 mL/well	Micro array 96-well plate	PE0101-MW
PEP-2	5 mg	1 mL/well	Micro array 96-well plate	PE00501-2MW
	10 mg	1 mL/well	Micro array 96-well plate	PE0101-2MW
PAX	5 mg	1 mL/well	Micro array 96-well plate	AX00501-MW
	10 mg	1 mL/well	Micro array 96-well plate	AX0101-MW
PCX	5 mg	1 mL/well	Micro array 96-well plate	CX00501-MW
	10 mg	1 mL/well	Micro array 96-well plate	CX0101-MW
PWAX	5 mg	1 mL/well	Micro array 96-well plate	WA00501-MW
	10 mg	1 mL/well	Micro array 96-well plate	WA0101-MW
PWCX	5 mg	1 mL/well	Micro array 96-well plate	WC00501-MW
	10 mg	1 mL/well	Micro array 96-well plate	WC0101-MW

Cleanert® MAS (Multi-function Impurity Adsorption SPE)

Cleanert® MAS-C

Description

Cleanert® MAS-C is a simplified sample preparation for bioanalysis which applies multi-functional adsorption to remove multiple interferences, while not retaining analytes. It combines protein precipitation with SPE method to reduce proteins and phospholipids which interfere with analytes in bioanalysis.

Method

Protocol of 96-well plate:

● Condition: Insert a 96-well collection plate into the vacuum manifold. Place the manifold collar and 96-well MAS plate onto the manifold. Add 0.5 mL of acetonitrile to each well to activate the sorbent and wash the well.

● Load: Add 10-50 µL of plasma (or serum, urine, etc.) sample to each well

● Protein precipitation: Add 0.5mL of eluant, for example acetonitrile (or acetonitrile with 0.01%-1% $NH_3 \cdot H_2O$ for amphoteric drugs) to each well quickly. Waiting for 3 minute (vortex maybe applied if needed), protein precipitation and drug extraction are completed simultaneously.

● Elution: Apply pressure to each well to collect eluate by vacuum or centrifugation. Then the eluate will be analyzed by LC/MS.

● The demonstrated procedure is based on the plate with 30mg packing material each well. It may be adjusted to meet the special demand in practical application. The plate of 60mg packing material is designed to treat 100µL sample while the plate of 30mg packing material is for 50µL sample or less ones. For 100µL sample, 1mL eluant is needed zfor protein precipitation.

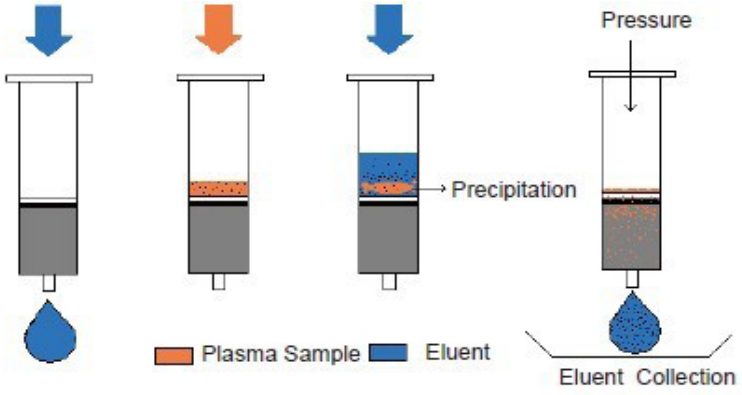
Protocol of cartridge:

● Condition: Place a MAS cartridge onto the manifold. Add 0.5-1 mL of acetonitrile to each well to activate the sorbent;

● Load: Add 10-50 µL of plasma (or serum, urine, etc.) sample to each cartridge;

● Protein precipitation: Add 0.5-1mL of eluant, for example acetonitrile (or acetonitrile with 0.01%-1% $NH_3 \cdot H_2O$ for amphoteric drugs) to each cartridge quickly. Waiting for 3minute (vortex maybe applied if needed), protein precipitation and drug extraction are completed simultaneously.

● Elution: Apply pressure to each cartridge to collect eluate by vacuum or centrifugation. Then the eluate will be analyzed by LC/MS.



Characteristics of the Method

● In conventional SPE method, interference matrices are adsorbed and then target compounds eluted, but the MAS are only to adsorb impurities
● MAS employ the sorbent modified with different functional groups that could, simultaneously remove a variety of impurities in one step.
● MAS has good separation ability and selectivity for the proteins, peptides, amino acids, phospholipids, and such biological interference matrix.
● By choosing suitable conditions (solvent and pH), most of the biological interference can be removed, and to ensure strong water-soluble substances tested with more than 70% recovery rate, also for further separation of LC-MS detection.

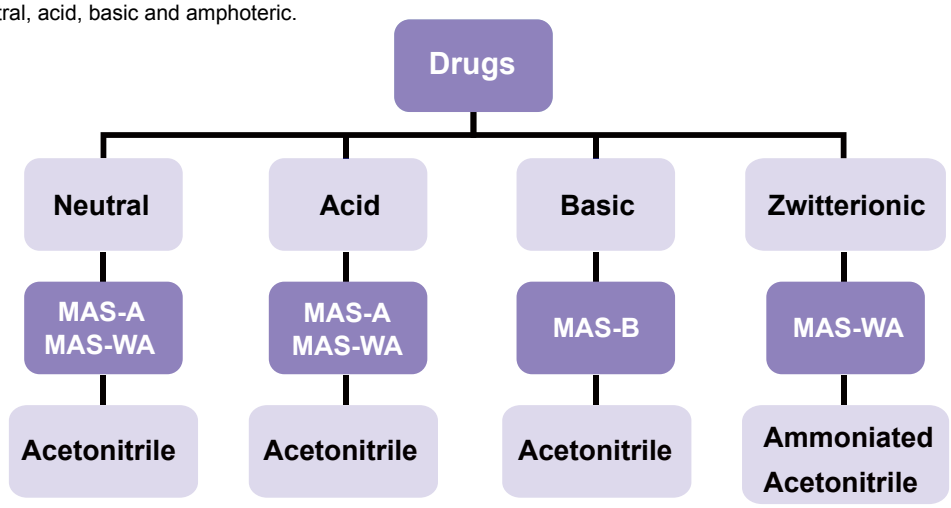
Benefit

In LC-MS/MS analysis, matrix such as protein, phospholipid, fat and some surfactants will cause serious matrix effect. Compared to traditional methods including liquid-liquid extraction (LLE), solid-phase extraction (SPE) and protein precipitation (PPT),
MAS-C is especially suitable for the treatment of polar compounds which can't be done with SPE.

	LLE	SPE	PPT	MAS
Interferences Removal	-	+	-	+
Operation	-	-	+	+

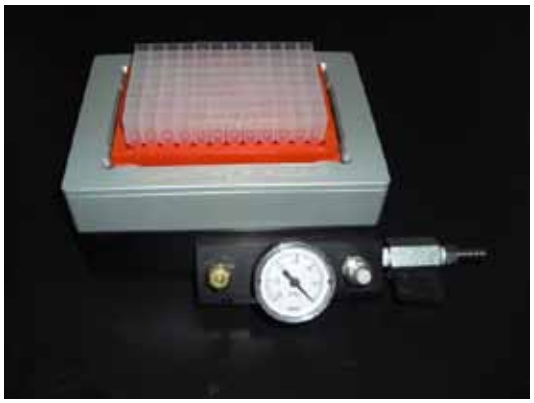
Selection Guide

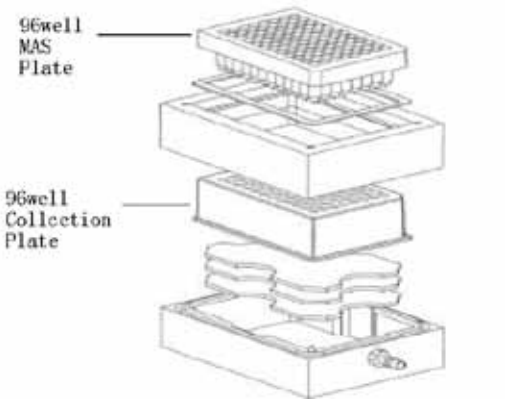
Cleanert® MAS is divided into two kinds, named MAS-A (including MAS-WA) and MAS-B to cover all the analytes. All the analytes can be divided into neutral, acid, basic and amphoteric.



For application, see page 79~84

Procedures

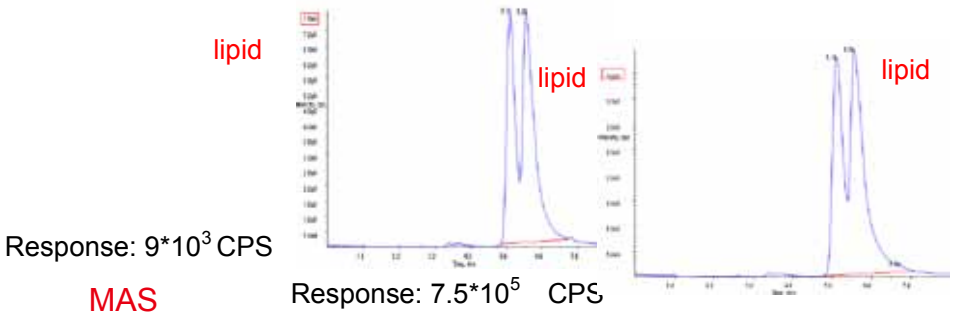




The vacuum manifold complemented to MAS plate

96-well plate MAS method for sample pretreatment of DMPK is extremely simple and rapid, mainly divided into the following four steps. The demonstrated procedure is based on the plate with 30mg packing material each well. It may be adjusted to meet the special demand in practical application. The plate of 60mg packing material is designed to treat 100μL sample while the plate of 30mg packing material is for 50μL sample or less ones. For 100μL sample, 1mL eluant is needed in step 3.

Removal of Phospholipids MAS vs PPT and SPE



The peak area of phospholipids lpid by three method (HPLC/MS analysis)

PPT= protein precipitation

Ordering Information

Production	Part No.	Spec.	Application	Package
MAS SPE cartridge	MSC-B-0301	1 mL	Used for clean-up of basic and neutral compounds in plasma and biological samples (For vacuum or positive pressure)	100
	MSC-B-0601	1 mL		100
	MSC-B-0301-F	1 mL	Used for clean-up of basic and neutral compounds in plasma and biological samples (For centrifugation)	100
	MSC-B-0601-F	1 mL		100
	MSC-A-0301	1 mL	Used for clean-up of acidic compounds in plasma and biological samples (For vacuum or positive pressure)	100
	MSC-A-0601	1 mL		100
	MSC-WA-0301	1 mL	Used for clean-up of weak acidic compounds in plasma and biological samples (For vacuum or positive pressure)	100
	MSC-WA-0601	1 mL		100
	MSC-A-0301-F	1 mL	Used for clean-up of acidic compounds in plasma and biological samples (For centrifugation)	100
	MSC-A-0601-F	1 mL		100
MAS 96 well plate	MS-B-0302W	30 mg/2 mL/well	Used for clean-up of basic and neutral compounds in plasma and biological samples (For vacuum or positive pressure)	2
	MS-B-0302W-F	30 mg/2 mL/well	Used for clean-up of basic and neutral compounds in plasma and biological samples (For centrifugation)	2
	MS-A-0302W	30 mg/2 mL/well	Used for clean-up of acidic compounds in plasma and biological samples (For vacuum or positive pressure)	2
	MS-A-0302W-F	30 mg/2 mL/well	Used for clean-up of acidic compounds in plasma and biological samples (For centrifugation)	2
	MS-WA-0302W	30 mg/2 mL/well	Used for clean-up of weak acidic compounds in plasma and biological samples (For vacuum or positive pressure)	2
	MS-WA-0302W-F	30 mg/2 mL/well	Used for clean-up of weak acidic compounds in plasma and biological samples (For centrifugation)	2

Cleanert® MAS-Q (QuEChERS)

Description

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method was first introduced by USDA and German scientists in 2003 and it becomes increasingly popular in the area of multi-residue pesticide analysis in food and agricultural products.

Bonna-Agela's QuEChERS product

MAS-Q (Multi-mechanism Adsorption SPE-QuEChERS) is an application of modified QuEChERS method which is suitable for most of the pesticides residue analysis, drug or antibiotic residue analysis in vegetable, fruit, grain, and animal issues;
MAS-Q method is based on dispersed solid phase extraction, and it applies multi-function impurity adsorption to remove as many as interferences and leave the analytes in the supernatant. The products are available in 2 mL, 15 mL and 50 mL centrifuge tube sizes, with or without centrifuge tubes.
MAS-Q extraction tubes are packed in anhydrous sealed packets. This enables you to add the salts after adding organic solvent to your sample. Adding the salts directly to the food sample may cause exothermic reactions that affect the recoveries of some heat-sensitive analytes.

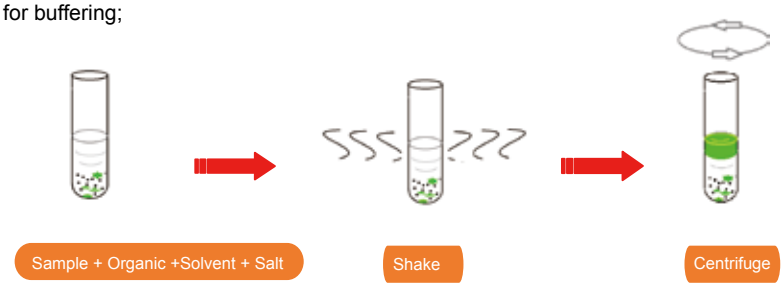
Advantage of MAS-Q

- Saving time, easy to operate;
- Economic and effective;
- Bulk sorbents and salts are also available for customization.

Recommended Standard Operating Procedure for MAS-Q

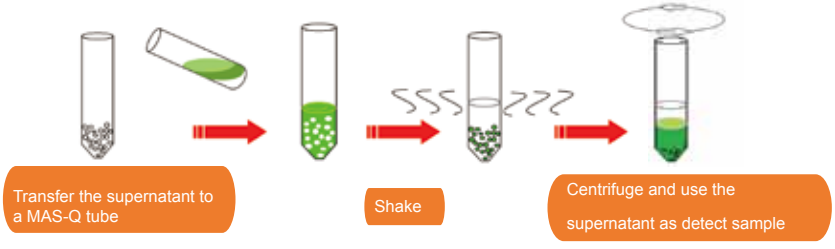
- Step 1: Extraction

Tubes containing salts enable you to extract the analytes of interest into the organic layer. The tube often sealed with $MgSO_4$ and NaCl, or other salts for buffering;



- Step 2: Clean-Up

Select a proper MAS-Q tube follow the description. Put the supernatant of extract tubes into a clean-up tube that containing SPE sorbent and $MgSO_4$. The sorbent will get rid of the intefering matrix, and the $MgSO_4$ helps to remove excess water.



QuEChERS has been used in traditional application areas, such as pesticide residue analysis in vegetable and fruit.
New applications are developed such as veterinary drugs and food additive analysis, including:

- Melamine determination in fish, milk and eggs by HPLC
- Simultaneous QuEChERS-HPLC analysis of clopidol, diclazuril and sulfonamides in animal tissues
- Determination of pesticide residues in honey by Modified MAS-QuEChERS Extraction

Ordering Information

Step one Extraction	Step two Clean-up	
	AOAC 2007.01	
	General fruit and vegetable	2 mLCentrifuge tube for 1 mL sample 50 mg PSA, 150 mg $MgSO_4$ P/N: MS-PA0250 15 mLCentrifuge tube for 8 mL sample 400 mg PSA, 1200mg $MgSO_4$ P/N: MS-PA1012
AOAC 2007.01 -AOAC Method 6 g MgSO4 (anhydrous) ; 1.5 g NaAc P/N: MS-MG5051	Fruit and vegetables with fats and waxes	2 mLCentrifuge tube for 1 mL sample 50 mg PSA, 50mg C18, 150 mg $MgSO_4$ P/N: MS-9PA0203 15 mLCentrifuge tube for 8 mL sample 400 mg PSA, 400mg C18, 1200mg $MgSO_4$ P/N: MS-9PA1011
	Pigmented fruits and vegetables	2 mLCentrifuge tube for 1 mL sample 50 mg PSA, 50 mg PC, 150 mg $MgSO_4$ P/N: MS-PP0250 15 mLCentrifuge tube for 8 mL sample 400 mg PSA, 400 mg PC, 1200 mg $MgSO_4$ P/N:MS-PP1550
EN 15662 -European and Mini-Multiresidue Method 4 g $MgSO_4$, 1 g NaCl, 1 g NaCitrate, 0.5 g disodium citrate sesquihydrate P/N: MS-NMS5050	EN 15662	
	General fruit and vegetable	2 mLCentrifuge tube for 1 mL sample 25 mg PSA, 0.15 g, $MgSO_4$(anhydrous) P/N: MS-PA0251 15 mLCentrifuge tube for 6 mL sample 150 mg PSA, 0.9 g $MgSO_4$(anhydrous) P/N:MS-PA1011
Original Unbuffered Method 6 g $MgSO_4$ (anhydrous) ; 1.5 g NACL P/N: MS-MG5051 4 g MgSO4(anhydrous); 1 g NACL P/N: MS-MG5055	Fruit and vegetables with fats and waxes	2 mLCentrifuge tube for 1 mL sample 25 mg PSA, 25 mg C18, 0.15 g $MgSO_4$(anhydrous) P/N: MS-9PA0204 15 mLCentrifuge tube for 6mL sample 150 mg PSA, 150 mg C18, 0.9 g $MgSO_4$(anhydrous) P/N: MS-9PA1210
	Pigmented fruits and vegetables	2 mLCentrifuge tube for 1 mL sample 25 mg PSA, 2.5 mg PC, 0.15 g $MgSO_4$(anhydrous) P/N: MS-PP0251 15 mLCentrifuge tube for 8 mL sample 150 mg PSA, 15 mg PC, 0.9 g $MgSO_4$(anhydrous) P/N: MS-PP1511

Step one Extraction	Step two Clean-up	
	Customized	
15 mL Centrifuge tube $MgSO_4$ (anhydrous) 6 g, NaCl 1 g P/N: MS-MG5053	Pesticide residues in Honey	15 mL Centrifuge tube $MgSO_4$ (anhydrous)1 g, C18 200 mg P/N: MS-181010
		15 mL Centrifuge tube C18 200 mg, $MgSO_4$ (anhydrous)1 g, PSA 100 mg P/N: 9PA1110

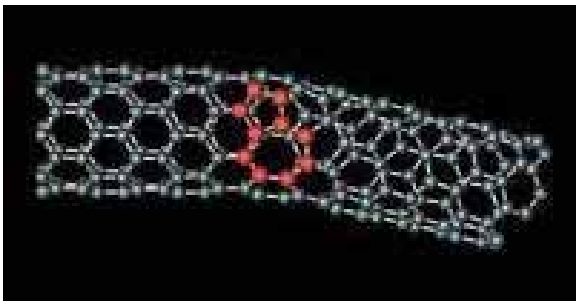
A New Carbon Nanotube Material: Cleanert® NANO

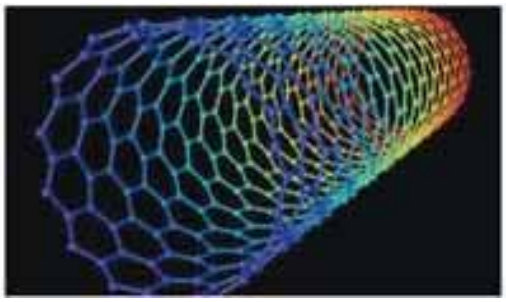
—— A Terminator Of Traditional SPE Analysis Of Pesticide Residues

Cleanert® NANO was developed from a carbon nano material for purification. It has the following characteristics.

- Functionalized nano material has better affinity to remove colorants and fatty acids.
- Controlled surface deactivation shows no over-adsorption to drugs to ensure sample recovery.
- Layer stacked carbon nanotubes structure increases specific surface area, thus enhancing loading capacity

These characteristics allow much less use of the material and improve specificity and selectivity. The amount of material used per sample is about only 1/10 to 1/5 when comparing to traditional PSA, C18 or GCB SPE.
It can be used widely in anlysis of multiple-pesticide residues in fruit, vegetable and other agro-products. Because of its small amount used, it can be packed into different format styles for fast analysis and in situ sampling on laboratory applications.





Product formats

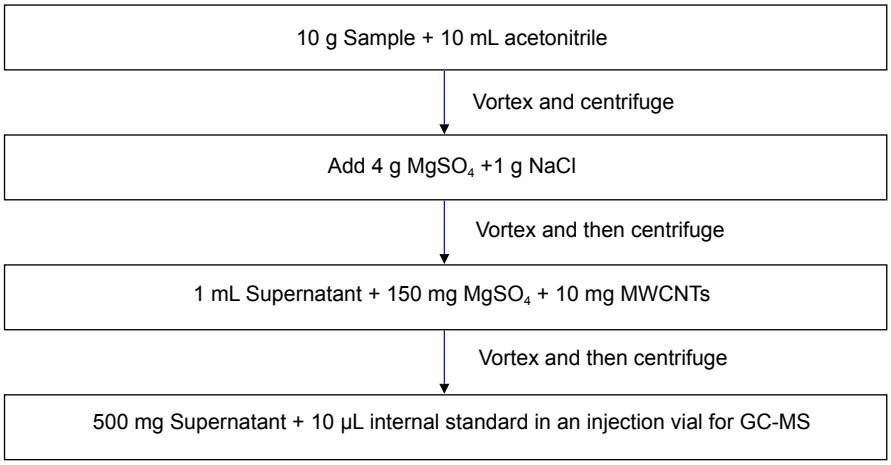
Centrifuge Tube Style
Easy to operate (such as centrifuging and batch processing)

SPE cartridge
2-Step operation: Conditioning, loading and eluting.
Easy to fit into automated workstation for high throughput processing

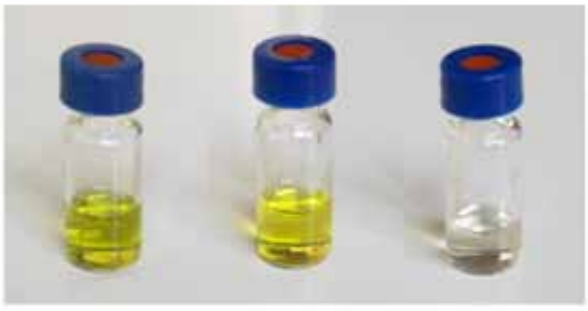
Application 1

Cleanert® NANO procedure to cleanup kale, spinac, grape and orange samples: 30 pesticides were used in the recovery test.

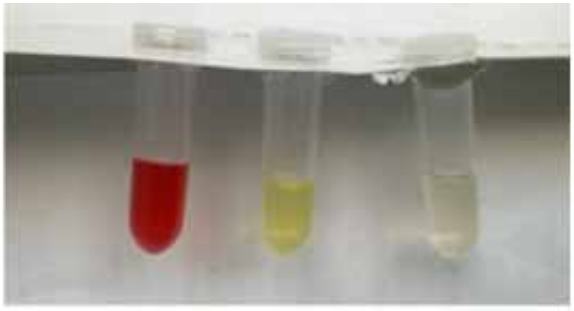
Procedure



Results



Comparison of Cleanup for kale sample: Before cleanup (left), PSA (middle) and MWCNT (right)



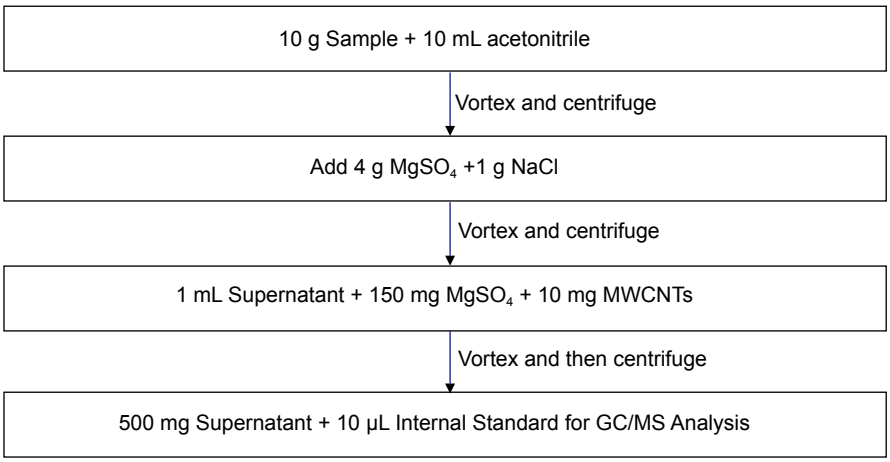
Cleanup of grape sample: Before cleanup (left), PSA (middle) and MWCNT (right)

Recovery of spiked samples: 30 Pesticides were spiked to kale, spinach and orange samples to test the cleanup effect of Cleanert NANO. For the pesticides spiked, the deviation of recovery is from 71% to 110%. For kale, spinach and orange samples, the recoveries fall in 71-109%, 72-110% and 72-110%, respectively.

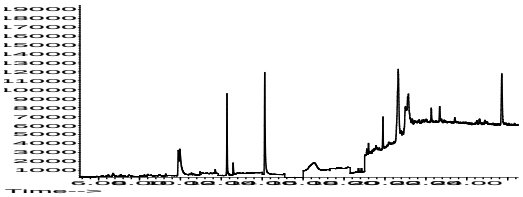
Application 2

A Cleanert® NANO cleanup procedure for chive, onion, ginger and garlic samples is shown in the following. In these examples, 14 Pesticides were spiked for recovery test in the mentioned samples.

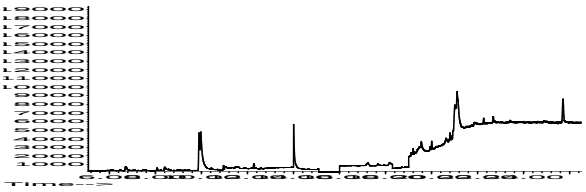
Procedure



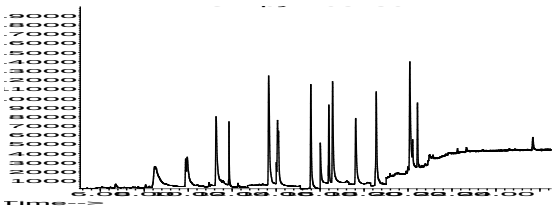
Results



a) PSA (chive blank)



b) MWCNTs (chive blank)



c) MWCNTs (spiked at 0.1 mg/Kg)

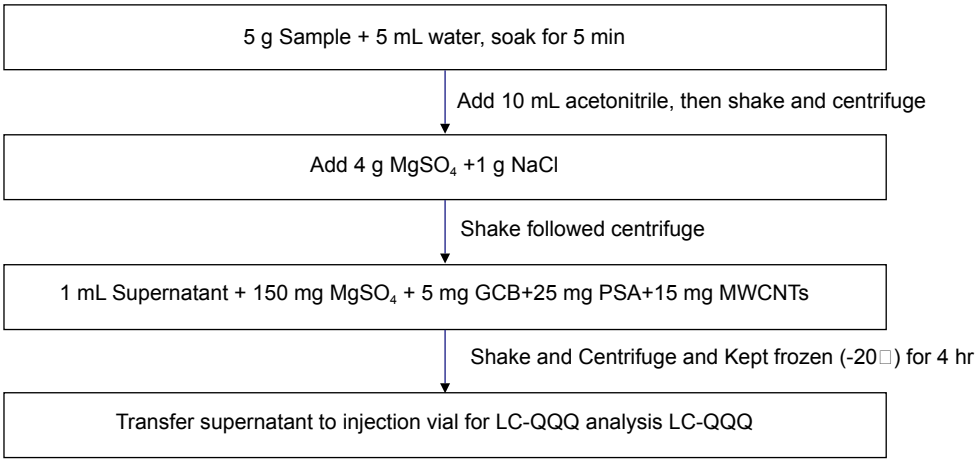
Comparison of total ion current (TIC) profiles of chive samples

Recovery of spiked samples: For 14 pesticides spiked, the recovery is in the range from 78 to 110% in tea leaf samples. The ranges of recovery for chive, onion, ginger and garlic samples, are 78-109%, 81-107% and 81-110% and 79-104%, respectively, and RSD for these samples is less than 14%. The LOQ and LOD of the analysis are 0.004 mg/Kg and 0.001-0.006 mg/Kg, respectively.

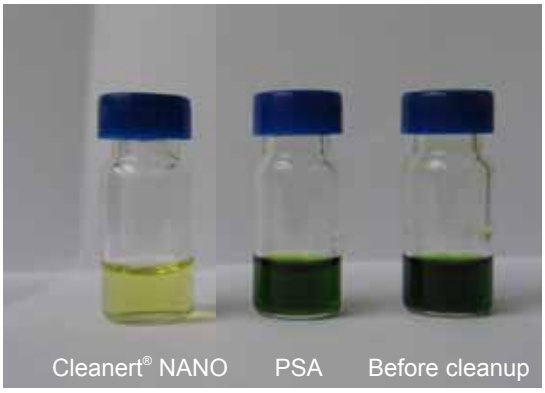
Application 3

A cleanup application of Cleanert® NANO on tea leaves is shown in the following. Tea leaf samples used in this application include Taiping Houkui, Tie Guan Yin and Pu'er brands. The procedure is shown below.

Procedure



Results (A comparison of cleanup with Cleanert NANO)



A comparison of cleanup with Cleanert® NANO

Recovery of spiked samples: For 34 pesticides spiked, the recovery is in the range from 78 to 110% in tea leaf samples. The ranges of recovery for Taiping Houkui, Tie Guan Yin and Pu'er tea leaves are 70 %-105 %, 78 %-107 % and 72%-109%, respectively, and RSD for these samples is less than 14%. The LOQ and LOD of the analysis are 0.3 ug/Kg and 0.1-1.5 ug/Kg, respectively.

Ordering Information

Product Name	Description	Specification, Package	Part Number
Nano tube for general vegetable and fruit	Light colored samples such as fruit and vegetables;	Syringe filter style, 1 mL, 50/PK;	IC-NN1010-V
		Centrifuge Tube Style, 15 mL; 50/PK	MS-NANO-V
Nano tube for Complex Samples	Complex sample such as Tea leaves, herbs;	Syringe filter style, 1 mL, 50/PK;	IC-NN1510-C
		Centrifuge Tube Style, 15 mL; 50/PK	MS-NANO-C
Nano tube for samples containing sulfide	Onion, ginger, garlic and chive, leek;	Syringe filter style, 1 mL, 50/PK;	IC-NN1510-S
		Centrifuge Tube Style, 15 mL; 50/PK	MS-NANO-S

Cleanert® SLE (Solid Supported Liquid/liquid Extraction) Technology and Sorbents

Description

Cleanert® SLE (Solid supported Liquid-liquid Extraction) plates and cartridges contain a modified diatomaceous earth, which provides an ideal surface with large specific area and low activity. Cleanert SLE is often used in bioanalytical,clinical, forensic, environmental and agrochemical fields and, it also can replace most of the Liquid/Liquid extraction (LLE) method.

Agela technologies can supply various specification of diatomaceous earth to meet different requirements, such as:
- Different buffered material: SLE-basic, SLE-neutral
- Different particle size: SLE- 100-200 mesh, SLE- 20-80 mesh
- Different surface modification: SLE-Inert, SLE-CM

Particles Characteristics.

Treated diatomiteous earth materials, Average particle diameter: 20-80 mesh;
3 different pH values: pH 3, pH 7 and pH 9;
Capability of water absorption: 0.8-1 mL/g.

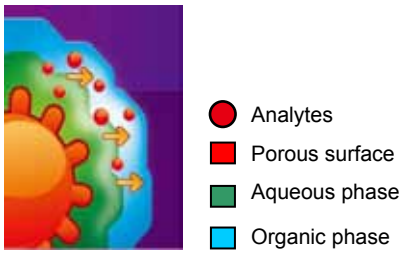
Mechanism

Diatomaceous earth using in the Cleanert SLE is a kind of porous material with strong absorption ability for water. When blood plasma was loaded onto the material, the sample is adsorbed onto the micropore and forms a thin liquid film onto the surface. Microcosmic extraction will occur between the organic solvent and the sample solution when eluent was added. The analytes are eluted while matrix such as phospholipids and proteins are retained on the polar surface, thus improving productivity and recovery.

Advantages over Liquid/Liquid Extraction

1. Reducing sample volume, solvents and time;
2. Easier parallel operation and automation;
3. Removing phospholipids;
4. Avoiding emulsification.

For applications, see page 102, 108, 109, 122



Method Process

Step 1: Apply the aqueous sample to the dry cartridge (No need to activated SLE product) Wait for 5-10 minutes;
Step 2: Add organic extraction solvent and collect the eluent. Ensure that the final eluent is water-immiscible.
Step 3: The collected eluent can be analyzed directly or dried and reconstituted.

Cleanert® SLE plates and cartridges usually are used under gravity, a pulse of vacuum is useful to sample loading of the aqueous sample through the upper hydrophobic frit and for a final draw of organic solvent after elution. Hydrophilic frit is available for SLE-AQ series. Figure 1 below shows the recommended procedure for plates and cartridges. Table 1 shows recommended buffer and elution volumes depending on sample loading volume for plate format product. Table 2 shows recommended buffer and elution volumes depending on sample loading volume for cartridge format product.

Tips: Thickness samples are proposed to be diluted with an equal volume of buffer or water. 1M ammonium buffer (pH 9-10) are recommended for basic analytes and 1M phosphate buffers (pH 2-3) are recommended for acidic analytes. MTBE is widely used as elution solvent, and ethyl acetate, DCM and mixed solvents are also good choices for elution solvent. For some very polar compounds, Agela suggests that more extreme pH control may be necessary to improve recovery. Elution with 2-3 times aliquots instead of one improves extraction efficiency and recovery.

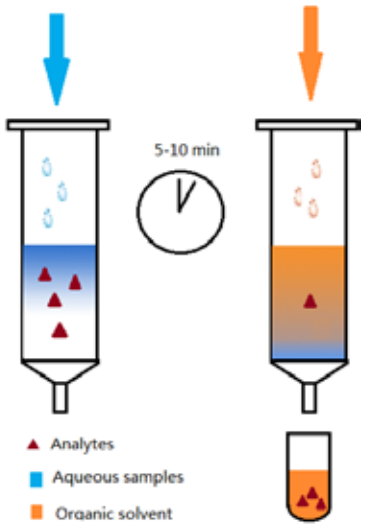


figure 1 Recommended precedure for SLE product

Table 1 Recommended buffer and elution volumns depending on initial sample volume (wellplates)

Sample Volume	<100 μL	100-200 μL	200 μL	300 μL	400 μL	>400 μL
	Suggest to diluted the sample with water (1:1) when sample volume is below 200 μL;					
Recommended SLE product	200 mg/2 mL; 96-well plate	200 mg/2 mL 96-well plate	300 mg/2 mL; 96-well plate	300 mg/2 mL; 96-well plate	400 mg/2 mL; 96-well plate	500 mg/3 mL or 600 mg/3 mL; 96-deep array well plate
	Apply vacuum (0.5 bar) for seconds make the sample pass through the upper frit; Wait for 5-10 minutes for sample absorb;					
Extraction Solvent	1 x 1 mL or 2 x 600 μL	1 x 1 mL or 2 x 600 μL	1 x 1 mL or 3 x 700 μL	1 x 1 mL or 3 x 700 μL	3 x 700 μL or 4 x 550 μL	3 x 1 mL or 4 x 800 μL
	Apply vacuum (0.5 bar) for 2 minutes to complete elution if necessary, then concentrate the elution and reconsititute as required;Don't make the sorbent bed dry between eluting					

Table 2 Recommended buffer and elution volumns depending on initial sample volume (cartridges)

Sample Volume	<200 μL	500 μL - 1 mL	1 - 2 mL	2 - 4 mL	8 - 10 mL	10 mL - 20 mL
Recommended SLE product	200 mg/3 mL cartridge;	1 g/6 mL cartridge;	2 g/12 mL cartridge;	4 g/25 mL cartridge;	10 g/60 mL cartridge;	20 g/60 mL cartridge;
	Wait for 10 minutes for sample absorb;The suggested ratio of sample volume and sorbent amount between 0.8:1-1:1;					
Extraction Solvent	1 x 1 mL or 2 x 0.5 mL	1 x 6 mL or 2 x 3 mL	2 x 6 mL or 3 x 4 mL	2 x 6 mL or 3 x 6 mL	4 x 15 mL	4 x 20 mL
	Apply vacuum (0.5 bar) for 2 minutes to complete elution, then concentrate the elution and reconsititute as required					

Ordering Information

Material	Description	Specification, Package	Cat. Number
Special treated diatomite SLE -AQ series (Neutral)	20-80 mesh; Neutral SLE cartridges with hydrophilic frit	200 mg/3 ml, 50 /Pk	HC2003Q-7
		500 mg/3 ml, 50 /Pk	HC5003Q-7
		500 mg/6 ml, 30 /Pk	HC5006Q-7
		1 g/6 mL, 30 /Pk	HC0006Q-7
		2 g/12 ml, 20 /Pk	HC200012Q-7
		4 g/25 ml, 15 /Pk	HC400025Q-7
		10 g/60 mL, 10 /Pk	HC1000060Q-7
		20 g/60 mL, 10 /Pk	HC2000060Q-7
	20-80 mesh; Neutral SLE well plates with hydrophilic frit	200 mg/2 ml/well, 2 /Pk	HC2002Q-7W
		300 mg/2 ml/well, 2 /Pk	HC3002Q-7W
		400 mg/2 ml/well, 2 /Pk	HC4002Q-7W
	20-80 mesh; Neutral SLE deep array well plates with hydrophilic frit	500 mg/3 ml/well, 1 /Pk	HC5003Q-7DW
	100-200mesh; Neutral SLE well plates with hydrophilic frit	200 mg/2 ml/well, 2 /Pk	HC2002SQ-7W
		300 mg/2 ml/well, 2 /Pk	HC3002SQ-7W
		400 mg/2 ml/well, 2 /Pk	HC4002SQ-7W
	100-200mesh; Neutral SLE deep array well plates with hydrophilic frit	500 mg/3 ml/well, 1 /Pk	HC5003SQ-7DW
Special treated diatomite SLE series Basic	20-80mesh; Basic SLE cartridges with hydrophilic frit	200 mg/3 ml, 50 /Pk	HC2003Q-9
		500 mg/3 ml, 50 /Pk	HC5003Q-9
		500 mg/6 ml, 30 /Pk	HC5006Q-9
		1 g/6 mL, 30 /Pk	HC0006Q-9
		2 g/12 ml, 20 /Pk	HC200012Q-9
		4 g/25 ml, 15 /Pk	HC400025Q-9
		10 g/60 mL, 10 /Pk	HC1000060Q-9
		20 g/60 mL, 10 /Pk	HC2000060Q-9
	20-80mesh; Basic SLE well plates with hydrophilic frit	200 mg/2 ml/well, 2 /Pk	HC2002Q-9W
		300 mg/2 ml/well, 2 /Pk	HC3002Q-9W
		400 mg/2 ml/well, 2 /Pk	HC4002Q-9W
	20-80mesh; Basic SLE deep array well plates with hydrophilic frit	500 mg/3 ml/well, 1 /Pk	HC5003Q-9DW
	100-200mesh; Basic SLE deep array well plates with hydrophilic frit	200 mg/2 ml/well, 2 /Pk	HC2002SQ-9W
		300 mg/2 ml/well, 2 /Pk	HC3002SQ-9W
		400 mg/2 ml/well, 2 /Pk	HC4002SQ-9W
	100-200mesh; Basic SLE deep array well plates with hydrophilic frit	500 mg/3 ml/well, 1 /Pk	HC5003SQ-9DW
Special treated diatomite SLE bulk sorbent	20-80mesh; Neutral SLE bulk sorbent	1 kg/Package	HC1001000-7
	20-80mesh; Basic SLE bulk sorbent	1 kg/Package	HC1001000-9
	20-80mesh; Neutral SLE bulk sorbent	18 kg/Package	HC018-7
	20-80mesh; Basic SLE bulk sorbent	18 kg/Package	HC018-9

Special Products

Cleanert® PAE and DEHP *NEW !*

—— Plasticizers Detection in Food and Water

Food contamination by phthalates has caused considerable hazard to food safety in Asia and other regions in the world. The most popular used plasticizers are phthalates series, which may affect the liver, kidney and may cause cancer. Bonna-Aglea as a provides Cleanert® PAE and DEHP to analyze phthalates in different matrices including food containing fats and oil, and aqueous samples.

For application, see page 70

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number	Type of Samples
Cleanert® PAE and DEHP	500 mg	6 mL	30	DEHP5006-G	Soft drink, wine, and water
	Glass bottle	40 mL	15	MS-PAE40	Low-fat food, such as milk
	Glass bottle	40 mL	15	MS-PAE40-C	Soy sauce
	3 g	6 mL	30	PAE30006-G	High-fat food, such as edible oil
	3 g	6 mL	30	PAE30006-C	sauces rich in oil

Cleanert® TPT *NEW !*

—— Triple Phases SPE for Tea Products

Description

Cleanert® TPT constitutes three type of materials, which employ different mechanisms and entail interactions with colorants, organic acids, bases and polyphenols as well as nonpolar interfering substances. By mixing different materials together, we can achieve a better cleanup result for complicated matrices. Cleanert® TPT is used for extraction and analysis of pesticides from tea products. It can remove most of interferents without adsorbing pesticide residues.

Determination of 519 pesticides and related chemicals residues in tea leaves—GC-MS method

1) Extraction
   Weigh 5 g of tea leaves in a centrifuge tube, and then add 15 mL of acetonitrile. The solution is homogenized and then centrifugated at 4200 r/min for 5 min. Transfer the supernatant, and extract the residue with 15 mL of acetonitrile again and centrifugate. Combine the two supernatants and evaporate to 1 mL for further purification.
2) Purification
   Purification method for GC-MS:
   Load 2 cm high of anhydrous sodium sulfate in Cleanert® TPT cartridge. Wash the cartridge with 10 mL of acetonitrile/toluene (3:1, v/v). Load the concentrated sample onto the Cleanert® TPT cartridge. Wash the sample bottle with 2 mL of acetonitrile/toluene (3:1, v/v) 3 times and combine the solutions into the cartridge.

Determination of 448 pesticides and related chemicals residues in tea leaves—LC-MS-MS method. (Cleanert® TPT, P/N: TPT200010)

Use the same procedure illustrated above except sample amount is 2 g. Evaporate the collected eluate to 0.5 mL by rotary evaporator in water bath at 40°C. Dry under a stream of nitrogen at 35°C. Redissolve the residue in 1 mL of acetonitrile/water (3:2, v/v). Filter the solution through 0.2 μm membrane for LC-MS/MS analysis.

For the full length of application, see page 66

Cleanert MCPD *NEW !*

Description

MCPD use high-pure diatomite earth to support LLE on the sorbent surface, enhance efficiency and clean effective

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleaenert MCPD	5 g	25 mL	20	EC500025

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® TPT	1 g	6 mL	30	TPT0006
	2 g	12 mL	20	TPT200010

Cleanert® TPH *NEW !*

—— Triple Phases SPE for Herb Samples

Description

Cleanert® TPH means triple phase SPE for Herb samples, and it is a composite of three types of sorbent with different mechanisms. By mixing different materials together, we achieve a better result of clean up, particularly for complicated matrices. Cleanert TPH is mainly used for extraction and detection of pesticides from Chinese Herb Medicines such as Ramulus Mori, Honeysuckle and the Fruit of Chinese Wolfberry. It can reduce effectively the interference without adsorbing any pesticide residues.
Cleanert® TPH has been used in Chinese national standard methods, determinating of 488 Pesticide Residues and Related Chemicals Residues and 413 Pesticide Residues and Related Chemicals Residues in the Ramulus Mori, Honeysuckle and the Fruit of Chinese Wolfberry with GS-MS and LC-MS/MS respectively.
The process includes ACN extraction, loading the sample onto the pre-activated column, and the subsequent elution by ACN- toluene (3+1).

For the full length of application, see page 66

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® TPH	1 g	6 mL	30	TPH0006
	2 g	12 mL	20	TPH200010

Cleanert® Bap *NEW !*

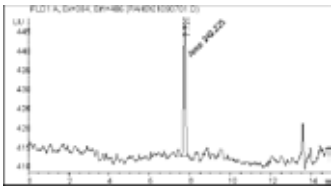
—— Benzopyrene Detection in Edible Oil

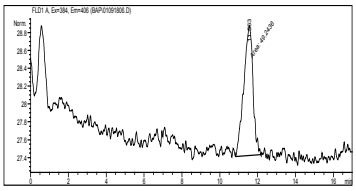
Description

Cleanert® Bap is a chromatographic column pre-packed with Alumina material aiming to separate benzopyrene and the triglyceride in oil.

Chromatographic condition

Column: Venusil® PAH, 5.0 μm, 4.6 mm × 250 mm
(P/N: VP952505-L)
Mobile phase: ACN : Water = 95 : 5;
Flow rate: 1.0 mL/min;
Injection volume: 20 μL
Fluorescence detector: Emission 406 nm, Excitation 384 nm





Cleanert ACA *NEW !*

—— Acrylamide analysis in water

Description

Cleanert ACA using carbon material to concentrate polar substances in water sample. Currently methods using Oasis HLB, ENVI Carb have a problem that acrylamide easy to be overloaded

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert ACA	2 g	30 mL	15	ACA200030

Cleanert® DNPH-Silica

Description:

Cleaner® DNPH-Silica prepared by acidified dinitrophenylhydrazine reagent coated on silica is used for collection of air samples. Aldehydes and ketones react in situ to form hydrazone derivatives; these are then eluted and quantitated by HPLC analysis. DNPH-Silica can be used in EPA Method TO–11A; ASTM D5197 for carbonyl compounds in air and JPMOE Official Methods for aldehydes: odor in out door air and in exhaust gas.

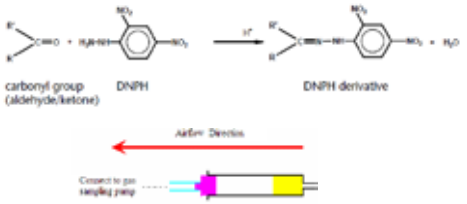
Specification

- Ave Background value: ≤0.2 μg (calculated by formaldehyde)
- Max sampling quantity: >75 μg (calculated by formaldehyde)
- Max Sampling Pressure: 0.15 MPa
- Recoveries: >90%

Background comparison
Red: Cleanert® DNPH-Silica
Blue: Brand G

Mechanism

The most sensitive and specific method for analyzing aldehydes and ketones is based on their reaction with 2,4-dinitrophenylhydrazine (DNPH) and subsequent analysis of the hydrazone derivatives by HPLC



Operation

Using a vacuum pump, an air sample is drawn through the new Cleanert® DNPH-silica cartridge. The aldehydes and ketones react with the DNPH and form the hydrazone derivative, which is retained on the cartridge. Later, the hydrazones are eluted from the cartridge with acetonitrile and analyzed by HPLC

HPLC Codition:
Column: Venusil® XBP C18, 4.6 × 250 mm, 5 μm (P/N: VX952505-0);
Mobile Phase: water:acetonitrile = 40 : 60;
Flow Rate: 1 ml/min; Tempreture: room temperature; Detector: UV 360 nm;

Ordering Information

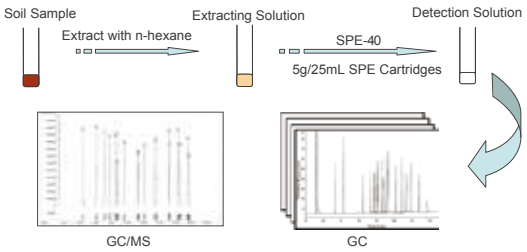
Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert DNPH	200 mg	1 mL	1	IC-DN2001

Cleanert® EPH *NEW !* —— for Extractable Petroleum Hydrocarbons

Description

Cleanert® EPH uses a special silica material to separate aliphatic hydrocarbon from aromatics in Environmental samples. The column was used in New Jersey Department of Environmental Protection Site Remediation Program, extractable petroleum hydrocarbons methodology. The method can also be adopted onto a fully automatic SPE working station. (SPE-10 and SPE-40 .

For application, see page 86



Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® EPH	5 g	25 mL	15	SI500025-30

Cleanert SLE-OD *NEW !*

Description

Aromatic amine detection in textile sample. China national method “Textiles-Determination of the banned azo colourants”.

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert SLE	20 g	60 mL	10	GB/T17592-2006

Cleanert® LDC *NEW !*

—— Large Disk for Water Analysis

Description

Cleanert® LDC is a specially designed SPE format with a much larger cross-section area to allow a large flow rate through the column, which is mostly useful to concentrate trace chemical substances from a large volume of aqueous samples, such as in water analyses.

● The special cartridge design is suitable for water concentration, and it can replace SPE disk in most of the analysis.

● Application: AQ C18 20-30 μm material used in EPA 525 method; In addition to three packing materials offered as standard products, Cleanert® LDC can be customized with any sorbents to meet customers' needs.

Ordering Information

Material	Vol	Tubes/box	Cat. Number
Cleanert® LDC AQ C18	200 mL	4	L182000100
Cleanert® LDC PEP-2	200 mL	4	LPE000100-2
Cleanert® LDC AQ C18-N	200 mL	4	L182000100-N

Cleanert LRC *NEW !*

—— Large Receiver Column

Description

Cleanert® LRC is designed to load more solvent in SPE process, specially for applications using a small amount of sorbents but a large volume of eluents.

All kinds of SPE material can be customized using LRC tube; Suggested specifications for Polymers are from 30 mg to 150 mg; Silica materials are from 100 mg to 300 mg;

Ordering Information

Material	Vol	Tubes/box	Cat. Number
Cleanert® LRC C18	200 mg/10 mL	20	L1820010
Cleanert® LRC PEP-2	60 mg/10 mL	20	LPE0610-2

Cleanert® SPE in Glass Tubes *NEW !*

Description

Cleanert® glass column series can avoid the residue interference from the plastic tubes and are recommended to be used for trace analysis of food and environmental samples. leave space PTFE frit are optional; All kinds of SPE material can be customized using glass tube;

For application, see page 70

Ordering Information

Material	Vol	Tubes/box	Cat. Number
Cleanert® C18 glass	200 mg/3 mL	50	182003-G
Cleanert® C18 glass	500 mg/6 mL	30	185006-G
Cleanert® PEP glass	60 mg/3 mL	50	PE0603-G
Cleanert® PEP glass	150 mg/6 mL	30	PE1506-G
Cleanert® PCX glass	60 mg/3 mL	50	CX0603-G

Conventional Products

Bonded Silica SPE

Cleanert® ODS C18 (End-capped)

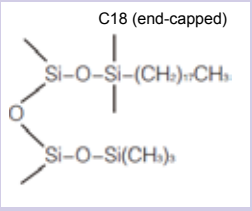
Description

Cleanert® ODS C18 products columns and plates are packed with octadecylsilane bonded silica sorbents. The sorbent is double endcapped and has a high bonding density (%C > 17). These products can be used for desalting biomolecules, such as proteins and DNAs.

Particle Characteristics

Based on Silica; C%: 18-19%; Average Particle Diameter: 40-60 μm; Average Pore Size: 60 Å;
Porosity: 0.8 mL/g; Specific Surface Area: 600 m^2/g. (Sperical Silica)

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® S C18 (Sperical Endcapped) 	100 mg	1 mL	100	S181001
	200 mg	3 mL	50	S182003
	500 mg	3 mL	50	S185003
	500 mg	6 mL	30	S185006
	1000 mg	6 mL	30	S180006
	2000 mg	12 mL	20	S18200012
	10 g	bottle		S180010
	100 g	bottle		S180100
	50 mg	2 mL	96-well plate, 2/pk	S180502-W
	100 mg	2 mL	96-well plate, 2/pk	S181002-W

Cleanert® ODS C18-N (Non-end-capped)

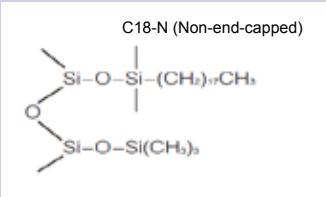
Description

Cleanert ODS C18-N is a type of C18 sorbent bonded to silica without end-capping modification, providing extra silanol residuals around the 'root' of alkyl chain on silica surface.The extra silanol silica of the sorbent provide additional polar interactions associated with surface silanol groups which enhance the retention of basic compounds.

Particle Characteristics

Based on Silica; C%: 17-18%; Average Particle Diameter: 40-60 μm; Average Pore Size: 60 Å;
Porosity: 0.8 mL/g; Specific Surface Area: 600 m^2/g (Sperical Silica);

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® S C18-N (Spherical Non-end-capped) 	100 mg	1 mL	100	S181001-N
	200 mg	3 mL	50	S182003-N
	500 mg	3 mL	50	S185003-N
	500 mg	6 mL	30	S185006-N
	1000 mg	6 mL	30	S180006-N
	2000 mg	12 mL	20	S18200012-N
	10 g	bottle		S180010-N
	100 g	bottle		S180100-N
	50 mg	2 mL	96-well plate, 2/pk	S180502-N-W
	100 mg	2 mL	96-well plate, 2/pk	S181002-N-W

Cleanert® C8 (Octyl)

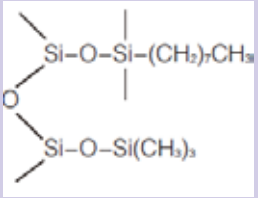
Description

The property of Cleanert® C8 products is similar to Cleanert® ODS C18 products. However, this sorbent is slightly less retentive than Cleanert® C18, which accelerates the elution of more hydrophobic substance. Cleanert® C8 is successfully used for the extraction of both water-soluble and fat-soluble vitamins from serum, as well as the desalting of biological macromolecules.

Particle Characteristics

Based on Silica; C%: 9-10%; Average Particle Diameter: 40-60 μm;
Average Pore Size: 60 Å; Porosity: 0.8 mL/g; Specific Surface Area: 600 m^2/g (Sperical Silica);

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® S C8 (Sperical) 	100 mg	1 mL	100	S081001
	200 mg	3 mL	50	S082003
	500 mg	3 mL	50	S085003
	500 mg	6 mL	30	S085006
	1000 mg	6 mL	30	S080006
	10 g	bottle		S080010
	100 g	bottle		S080100
	50 mg/well	2 mL	96-well plate	S080502-W
	100 mg/well	2 mL	96-well plate	S081002-W

Cleanert® CN (Cyanopropyl)

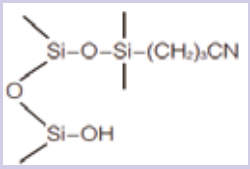
Description

Cleanert® Cyano(CN) SPE is silica based sorbent bonded with cyanopropyl functional groups. This polar sorbent exhibits both polar and non-polar interactions. It can be used for extraction of both polar and non-polar molecules in either normal phase or reversed phase mode.

Particle Characteristics

Based on Spherical Silica; C%: 5-6%; Average Particle Diameter: 40-60 μm;
Average Pore Size: 60Å; Porosity: 0.8mL/g; Specific Surface Area: 600 m^2/g

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® CN 	100 mg	1 mL	100	CN1001
	200 mg	3 mL	50	CN2003
	500 mg	3 mL	50	CN5003
	500 mg	6 mL	30	CN5006
	1 g	6 mL	30	CN0006
	50 mg/well	2 mL	96-well plate	CN0502-W
	100 mg/well	2 mL	96-well plate	CN1002-W
	10 g	-	-	CN0010
	100 g	-	-	CN0100

Cleanert® NH_2 (Aminopropyl)

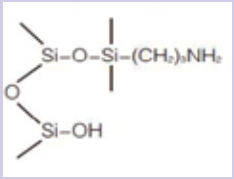
Description

Cleanert® NH_2 products are silica based sorbent bonded with aminopropyl funtional group. This sorbent can be used in either normal phase or reversed phase mode. It retains the analytes either by a polar adsorption (from non-polar solution) or by weak anion exchange (from aqueous solution). pKa=9.8 .

Particle Characteristics

Based on Irregular Silica; Average Particle Diameter: 40-60 μm;
Average Pore Size: 60 Å; Porosity: 0.8 mL/g; Specific Surface Area: 480 m^2/g

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® NH_2 	100 mg	1 mL	100	NH1001
	200 mg	3 mL	50	NH2003
	500 mg	3 mL	50	NH5003
	500 mg	6 mL	30	NH5006
	1 g	6 mL	30	NH0006
	50 mg/well	2 mL	96-well plate	NH0502-W
	100 mg/well	2 mL	96-well plate	NH1002-W
	10 g	-	-	NH0010
	100 g	-	-	NH0100

Cleanert® PSA {(N-aminoethyl) Aminopropyl}

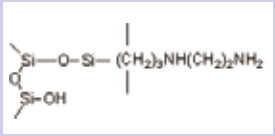
Description

Cleanert® PSA SPE is similar to Cleanert® NH_2. It has two amino groups with pKa = 10.1 and 10.9, respectively. This sorbent is an anion exchanger slightly stronger than Cleanert® NH_2. It can be used for the extraction of metal ions by chelating interactions. It is also commonly used to remove organic acids, pigments and metal ions from organic samples such as vegetables and fruits.

Particle Characteristics

Based on Irregular Silica; Average Particle Diameter: 40-60 μm;
Average Pore Size: 60 Å; Porosity: 0.8 mL/g; Specific Surface Area: 480 m^2/g

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PSA 	100 mg	1 mL	100	PA1001
	200 mg	3 mL	50	PA2003
	500 mg	3 mL	50	PA5003
	500 mg	6 mL	30	PA5006
	1 g	6 mL	30	PA0006
	50 mg/well	2 mL	96-well plate	PA0502-W
	100 mg/well	2 mL	96-well plate	PA1002-W
	10 g	-	-	PA0010
	100 g	-	-	PA0100

Cleanert® SAX (Strong Anion Exchanger)

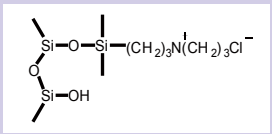
Description

Cleanert® SAX SPE products are packed with silica based sorbent bonded with a quaternary amine. This strong anion exchanger is used to extract compounds capable of carrying a negative charge from both aqueous and non-aqueous solutions. They are ideally suitable to extraction of weak acids and desalting of biological macromolecules.

Particle Characteristics

Based on Spherical Silica; C%: 9-10%; Average Particle Diameter: 40-60 μm; Average Pore Size: 60 Å;
Porosity: 0.8 mL/g; Specific Surface Area: 600 m^2/g The Ion Exchange Degree: 0.5 meq/g.

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® SAX 	100 mg	1 mL	100	SA1001
	200 mg	3 mL	50	SA2003
	500 mg	3 mL	50	SA5003
	500 mg	6 mL	30	SA5006
	1 g	6 mL	30	SA0006
	50 mg/well	2 mL	96-well plate	SA0502-W
	100 mg/well	2 mL	96-well plate	SA1002-W
	10 g	-	-	SA0010
	100 g	-	-	SA0100

Cleanert® COOH (Weak Cation Exchanger)

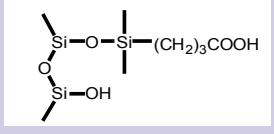
Description

Cleanert® COOH SPE products consist of a propyl carboxylic acid on the inner silica surface. The pKa of the carboxylic acid group is approximately 3.8. It is a useful sorbent for quaternary ammonium salt and other strong cations.

Particle Characteristics

Based on Spherical Silica; C%: 5-6%; Average Particle Diameter: 40-60 μm; Average Pore Size: 60 Å;
Porosity: 0.8 mL/g; Specific Surface Area: 600 m^2/g

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® COOH 	100 mg	1 mL	100	CH1001
	200 mg	3 mL	50	CH2003
	500 mg	3 mL	50	CH5003
	500 mg	6 mL	30	CH5006
	1 g	6 mL	30	CH0006
	50 mg/well	2 mL	96-well plate	CH0502-W
	100 mg/well	2 mL	96-well plate	CH1002-W
	10 g	-	-	CH0010
	100 g	-	-	CH0100

Cleanert® PRS (Propane Sulfonic Acid)

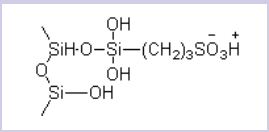
Description

Cleanert® PRS SPE sorbent is a silica gel based strong cation exchanger. This sorbent, consisting of a propane sulfonic acid, has slightly less exchange capability than SCX. It can be applied to the extraction of weak cations, such as pyridine, with high recovery.

Particle Characteristics

Based on Spherical Silica; Average Particle Diameter: 40-60 μm; Average Pore Size: 60 Å;
Porosity: 0.8 mL/g; Specific Surface Area: 600 m^2/g
The Ion Exchange Degree: 0.3 meq/g.

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert PRS 	100 mg	1 mL	100	PR1001
	200 mg	3 mL	50	PR2003
	500 mg	3 mL	50	PR5003
	500 mg	6 mL	30	PR5006
	1 g	6 mL	30	PR0006
	50 mg/well	2 mL	96-well plate	PR0502-W
	100 mg/well	2 mL	96-well plate	PR1002-W
	10 g	-	-	PR0010
	100 g	-	-	PR0100

Cleanert® SCX (Strong Cation Exchanger)

Description

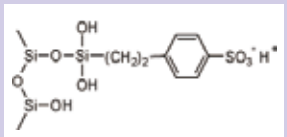
Cleanert® SCX sorbent is a strong cation exchanger based on silica gel, with benzene sulfonic acid. The sorbent is used to extract positively charged basic compounds or remove the salt from biological samples. It can also be mixed with C18 sorbent to extract the organic bases.

Particle Characteristics

Based on Spherical Silica; Average Particle Diameter: 40-60 μm; Average Pore Size: 60 Å;
Porosity: 0.8 mL/g; Specific Surface Area: 600 m^2/g; The Ion Exchange Degree: 0.5 meq/g.

For applications, see page 67

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® SCX 	100 mg	1 mL	100	SC1001
	200 mg	3 mL	50	SC2003
	500 mg	3 mL	50	SC5003
	500 mg	6 mL	30	SC5006
	1 g	6 mL	30	SC0006
	50 mg/well	2 mL	96-well plate	SC0502-W
	100 mg/well	2 mL	96-well plate	SC1002-W
	10 g	-	-	SC0010
	100 g	-	-	SC0100

Cleanert® Silica

Description

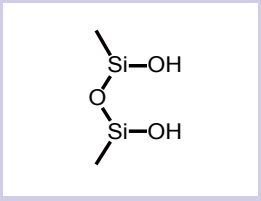
Cleanert® Diol is a Silica SPE product has unbonded, activated irregular silica as sorbent. This sorbent exhibits high polar interactions and is used to retain polar interference and to pass through weak-polar or non-polar compounds of interest such as oil samples. In addition, the silanol groups are ionzable at intermediate pH, so it can be used as a weak cation exchanger.

Particle Characteristics

Irregular Silica; Average Particle Diameter: 40-60 μm; Average Pore Size: 60 Å;
Porosity: 0.8 mL/g; Specific Surface Area: 480 m^2/g

For application, see page 63

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® Silica 	100 mg	1 mL	100	SI1001
	200 mg	3 mL	50	SI2003
	500 mg	3 mL	50	SI5003
	500 mg	6 mL	30	SI5006
	1 g	6 mL	30	SI0006
	50 mg/well	2 mL	96-well plate	SI0502-W
	100 mg/well	2 mL	96-well plate	SI1002-W
	10 g	-	-	SI0010
	100 g	-	-	SI0100

Cleanert® Diol

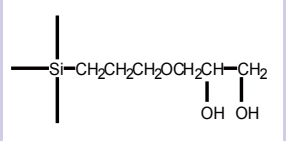
Description

Cleanert® Silica based dihydroxy SPE. It is used to extract polar analytes from non-polar solutions. It is a neutral sorbent and extracts compounds by forming hydrogen bonding or polar-polar interaction. As an example, it can be used to extract THC.

Particle Characteristics

Based on Spherical Silica; C%: 5-6%; Average Particle Diameter: 40-60 μm; Average Pore Size: 60 Å;
Porosity: 0.8 mL/g; Specific Surface Area: 600 m^2/g

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® Diol 	100 mg	1 mL	100	DI1001
	200 mg	3 mL	50	DI2003
	500 mg	3 mL	50	DI5003
	500 mg	6 mL	30	DI5006
	1 g	6 mL	30	DI0006
	50 mg/well	2 mL	96-well plate	DI0502-W
	100 mg/well	2 mL	96-well plate	DI1002-W
	10 g	-	-	DI0010
	100 g	-	-	DI0100

Non-silica Adsorption Phase Cartridges

Cleanert® Florisil (Magnesia Silica)

Description

Cleanert® Florisil is a highly selective adsorbent, which contains silica (84%), magnesium oxide (15.5%), and sodium sulfate (0.5%). It was used for AOAC, EPA and other methods designed for pesticide residues, separation, internal secretion and the separation of oil, PCBs, PAHs, and the the separation of nitrogen compounds and antibiotic substances in hydrocarbons. For example, it has been used in pesticide analysis, removal of pigment, for analysis method NY761.

Particle Characteristics

Adsorption Sorbents; Average Particle Diameter: 60-100 mesh (100-200 mesh optional);
Average Pore Size: 80 Å; Specific Surface Area: 290 m^2/g

For applications, see page 68

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® Florisil	100 mg	1 mL	100	FS1001
	200 mg	3 mL	50	FS2003
	500 mg	3 mL	50	FS5003
	500 mg	6 mL	30	FS5006
	1 g	6 mL	30	FS0006
	2 g	6 mL	30	FS20006
	50 mg/well	2 mL	96-well plate	FS0502-W
	100 mg/well	2 mL	96-well plate	FS1002-W
	10 g	-	-	FS0010
	100 g	-	-	FS0100

Cleanert® PestiCarb (Graphitized Carbon Black)

Description

Cleanert® PestiCarb is made of graphitized carbon by a distinct surface modification process, and has been used for sample cleanup in pesticide residues in plants or animal tissues. This sorbent can effectively reduce the background noise and increase sensitivity.

Particle Characteristics

Adsorption Sorbents; Average Particle Size: 120~400 mesh.

For application, see page 114

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PestiCarb	100 mg	1 mL	100	PC1001
	200 mg	3 mL	50	PC2003
	500 mg	3 mL	50	PC5003
	500 mg	6 mL	30	PC5006
	1 g	6 mL	30	PC0006
	50 mg/well	2 mL	96-well plate	PC0502-W
	100 mg/well	2 mL	96-well plate	PC1002-W
	10 g	-	-	PC0010
	100 g	-	-	PC0100

Cleanert® Alumina N (Aluminium Oxide; Neutral)

Description

Cleanert® Alumina N sorbent (pH = 7.5) can adsorb molecules by interaction with the aluminum metal center. The neutralized surface allows interaction with compounds whose heteroatoms are electronegative (e. g. N, S, P) or with an electron-rich highly aromatic structure.

Particle Characteristics

Adsorption Sorbents;
Average Particle Size: 100-200 mesh; Average Pore Size: 80 Å;

For applications, see page 84, 85, 116

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® Alumina N	100 mg	1 mL	100	AL1001-N
	200 mg	3 mL	50	AL2003-N
	500 mg	3 mL	50	AL5003-N
	500 mg	6 mL	30	AL5006-N
	1 g	6 mL	30	AL0006-N
	50 mg/well	2 mL	96-well plate	AL0502-N-W
	100 mg/well	2 mL	96-well plate	AL1002-N-W
	10 g	-	-	AL0010-N
	100 g	-	-	AL0100-N

Cleanert® Alumina A (Aluminium Oxide; Acidic)

Description

Cleanert® Alumina A sorbent (pH = 4.5) can be used as a strong polar absorbent or a mild cation exchanger. This sorbent is processed with a special deactivation procedure which ensures high analytes recovery.

Particle Characteristics

Adsorption Sorbents;
Average Particle Size: 100-200 mesh; Average Pore Size: 80 Å;

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® Alumina A	100 mg	1 mL	100	AL1001-A
	200 mg	3 mL	50	AL2003-A
	500 mg	3 mL	50	AL5003-A
	500 mg	6 mL	30	AL5006-A
	1 g	6 mL	30	AL0006-A
	50 mg/well	2 mL	96-well plate	AL0502-A-W
	100 mg/well	2 mL	96-well plate	AL1002-A-W
	10 g	-	-	AL0010-A
	100 g	-	-	AL0100-A

Cleanert® Alumina B (Aluminium Oxide; Basic)

Description

Cleanert® Alumina B products (pH = 10.0) can be used to remove organic acids and phenols in sample matrix. They have been pre-treated by special deactivation to ensure high analytes recovery.

Particle Characteristics

Adsorption Sorbents;
Average Particle Size: 100-200 mesh; Average Pore Size: 80 Å.

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® Alumina B	100 mg	1 mL	100	AL1001-B
	200 mg	3 mL	50	AL2003-B
	500 mg	3 mL	50	AL5003-B
	500 mg	6 mL	30	AL5006-B
	1 g	6 mL	30	AL0006-B
	50 mg/well	2 mL	96-well plate	AL0502-B-W
	100 mg/well	2 mL	96-well plate	AL1002-B-W
	10 g	-	-	AL0010-B
	100 g	-	-	AL0100-B

Mixed and Layered Phases

Cleanert® PestiCarb/NH_2

Description

Cleanert® PestiCarb/NH_2 SPE column is packed with 500 mg PestiCarb and 500 mg NH_2. It hasbeen widely used in analysis of pesticide residues, esp. for the Japanese Positive List System. It can be used in pesticide residue analysis, coloring matter, fatty acid and hydroxybenzene.

Particle Characteristics

Refer to Cleanert® Pesticarb and Cleanert® NH_2

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® PestiCarb/NH_2	500 mg/500 mg	6 mL	30	PN0006
	300 mg/500 mg	6 mL	30	PN8006

Cleanert® C8/SCX

Description

Clearnert® C8/SCX is a mixed-mode SPE based on silica of C8 and strong cation-exchange. It is usually used for the extraction of basic drugs from urine or blood.It has been widely used in drug abuse and forensic analysis.

Particle Characteristics

Refer to Cleanert® C8 and Cleanert SCX

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleaner® C8/SCX	50 mg	1 mL	100	CS0501
	130 mg	3 mL	50	CS1303
	300 mg	3 mL	50	CS3003
	500 mg	6 mL	30	CS5006
	1 g	6 mL	30	CS0006
	50 mg/well	2 mL	96-well plate	CS0502-W
	100 mg/well	2 mL	96-well plate	CS1002-W
	10 g	-	-	CS0010
	100 g	-	-	CS0100

Specialized Phases

Cleanert® HXN (Mid Polar Polymers Specially for Sulfonyl Urea Samples)

Description

Cleanert® HXN is also made of polydivinylbenzene having surface modified with vinylpyrrolidone. This sorbent is specially designed to extract sulfonyl ureas from water and soil at ppb level. It is less polar than Cleanert® PEP and can also be used to extract, enrich and clean up samples from mid polar to high polar compounds.

Particle Characteristics

Based on polystyrene/divinylbenzene; Average Particle Diameter: 40-60 μm;
Average Pore Size: 70 Å; Porosity: 1.2 mL/g; Specific Surface Area: 600 m^2/g

For detail, see page 94

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® HXN	30 mg	1 mL	100	HX0301
	60 mg	1 mL	100	HX0601
	60 mg	3 mL	50	HX0603
	100 mg	3 mL	50	HX1003
	200 mg	6 mL	30	HX2006
	500 mg	6 mL	30	HX5006
	10 g		bottle	HX0010
	100 g		bottle	HX0100
	30 mg	2 mL	96-well plate	HX0302-W
	50 mg	2 mL	96-well plate	HX0502-W

Cleanert® SUL-5 (Specific Columns for Sulfonamides)

Description

Cleanert® SUL-5 (specific columns for sulfonamides) is specially designed for the extraction of five sulfonamides (SM2,SMM,SMZ,SDM. SQ) in pork.

For application, see page 56

Ordering Information

Material	Sorbent	Vol	Tubes/box	Cat. Number
Cleanert® SUL-5	2 g	12 mL	20	SUL-5

Cleanert® IC:

Sample Clean-up Cartridges for Ion Chromatography

Description

Cleanert® IC series are used for removing matrix interferences such as phenolics, metalions, cations, anions, or hydrophobic substances encountered in many ion chromatography applications. They can increase organic matrix and reduce interferential ion in the sample, thus increasing the lifetimes of the analytical columns. They also can be used for some trace-level determinations.
The IC cartridges are designed as luer inlet and outlet for easy operation. They also can connect several cartridges together for complex matrix. The sample solution can be passed through the IC cartridge by syringe, and also by vacuum manifold connected with an empty SPE cartridge as funnel.

For application, see page 123

Type	Ion-exchange capability	Application	Spec.	Cat. No.
IC-ODS	—	To remove the hydrophobe from biology samples.	1 cc, 50/pk 2.5 cc, 50/pk	IC-1810 IC-1825
IC-RP	—	To remove substances such as aromatic dyes, some aromatic carboxylic acids, hydrocarbons, and surfactants from sample matrices.	1 cc, 50/pk 2.5 cc, 50/pk	IC-RP10 IC-RP25
IC-P	—	To remove the phenolic fraction of humic acids, tannic acids, lignins, anthocyanins, and azodyes from samples.	1 cc, 50/pk 2.5 cc, 50/pk	IC-P10 IC-P25
IC-A	0.7 meq/1 cc	To remove anion contaminant and neutralize the strongly acidic sample solution.	1 cc, 50/pk 2.5 cc, 50/pk	IC-A10 IC-A25
IC-H	2.0-2.2 meq/1 cc	To remove high levels of alkaline earths and transition metals from sample matrices and in the neutralization of highly alkaline samples such as sodium hydroxide or sodium carbonate.	1 cc, 50/pk 2.5 cc, 50/pk	IC-H10 IC-H25
IC-Na	2.0-2.2 meq/1 cc	To remove high levels of alkaline earths and transition metals from sample matrices without acidifying the sample. This ensures good recovery of acid labile analytes such as nitrite.	1 cc, 50/pk 2.5 cc, 50/pk	IC-Na10 IC-Na25
IC-Ag	2.0-2.2 meq/1 cc	To remove chloride, bromide, and iodide from sample matrices. An IC- H cartridge should be used after the IC- Ag cartridge to remove dissolved Ag.	1 cc, 50/pk 2.5 cc, 50/pk	IC-Ag10 IC-Ag25
IC-Ba	2.0-2.2 meq/1 cc	To remove $SO4^{2-}$, the cartridge should be activated with solution contain Cl- when the concertration of anion in the sample is too low.	1 cc, 50/pk 2.5 cc, 50/pk	IC-Ba10 IC-Ba25
IC-M	0.4 meq/1 cc	To remove transition metals and matrix elimination of alkali and alkaline earth metals.	1 cc, 50/pk 2.5 cc, 50/pk	IC-M10 IC-M25
IC-Ag/H	—	A layered cartridge containing IC-Ag and IC-H,	1 cc, 50/pk 2.5 cc, 50/pk	IC-AgH10 IC-AgH25
IC-Ag/Na	—	A layered cartridge containing IC-Ag and IC-Na,	1 cc, 50/pk 2.5 cc, 50/pk	IC-AgNa10 IC-AgNa25
IC-Ba/Ag/H	—	A layered cartridge containing IC-Ba, IC-Ag and IC-H,	1 cc, 50/pk 2.5 cc, 50/pk	IC-Ba/Ag/H10 IC- Ba/Ag/H 25
IC-Ba/Ag/Na	—	A layered cartridge containing IC-Ba, IC-Ag and IC-Na,	1 cc, 50/pk 2.5 cc, 50/pk	IC-BaAgNa10 IC-BaAgNa25

SPE Accessories and Supplies

Cleanert® PPP

—— Protein Precipitation Plate

Description

Cleanert® Protein Precipitation Plate product for DMPK research is manufactured with the reagents of the highest purity. Every lot is subjected to a wide range of rigorous quality control tests and confirmed to our strict specification.The graded frit design is optimized to efficiently remove most of the interference from plasma samples without plugging or breakthrough.PPP Plus combine the 0.45um PTFE filter with the precipitation frit, sample no need to filtration otherwise.

The 96-well plate can be accomplished with most of robotics, also minimizing sample handling. The plates are compatible with Bonna-Agela 96 well vacuum manifold, and also can be used in centrifuges.

Operation

- Loading about 50 µl plasma sample
- Add 3 times solvents as elute solution and shake the plate to help precipitation
- Apply vacuum or centrifuge to get the solution through the plate

Ordering Information

Product	Specification	Package	Cat.No
Cleanert® PPP	1 mL	100 /pk	96CD01
Cleanert® PPP	2 mL/well	2 /pk	96CD2025
Cleanert® PPP Plus	2 mL/well	2 /pk	96CD2025-F

SPE Vacuum Manifolds

Description

SPE Vaccum manifolds for SPE sample preparation, filtration and elution are available in 12 and 24 port configurations. These manifolds permit consistent extraction and filtration results. Multiple sample processing with these manifolds consists of a clear glass chamber and lid, to which a vacuum is applied to draw solvents and sample through an SPE column, cartridge, or disk. The lid is CNC machined, solvent resistant, low extractable virgin polypropylene. The lid is autoclavable, and will not wrap. The female Luer inlets and male Luer outlets are molded of pure polypropylene. Adjustable racks placed in the glass chamber will accommodate a variety of sample collection vessels, including test tubes, autosampler vials, volumetric flasks, and Erlenmeyer flasks. Eluants are deposited directly into the collection vessel of choice via polypropylene, or optional stailness steel or Teflon needles.

Cat No.:VM12/24

Ordering Information

Product Name	Specification, Package	Cat. No.
Vaccum manifold	12 positions	VM12
	24 positions	VM24
Stopcocks	12 pieces/ package	A81213
Teflon Needles	12 pieces/ package	A80100

SPE-10 Automatic Processing Station

- Designed for sample preparation in analysis of food and environmental samples;
- Process up to 6 samples per batch without attendance;
- Up to 5 solvents can be used for column conditioning and multi-step elution;
- Two fractions can be collected for each sample;
- Accommodate 1-25 mL SPE cartridges;
- Flow rate can be 1-20 mL/min;
- Maximum 100 mL sample can be loaded;
- Exhaustive sample injection by positive pressure;
- Easy operation with panel or by computer;
- 20 programs can be stored in the machine.

Cat No.:SPE-10

Qdaura™ Automated SPE Workstation

The Qdaura™ Automated SPE Workstation is a 4-channel parallel system specifically designed for high throughput samples processing. It is a powerful system, automating routine SPE procedures from conditioning, sample loading, washing, elution to collection. The built-in program facilitates rinse step during extraction to eliminate cross-contamination. Each thread of the 4-parallel channel can handle 6 samples in a given method that 24 samples in total can be prepared in a single run. It is an indispensable tool for applications in the area of agricultural and food chemistry, environmental monitoring,clinical testing and forensic analysis.

- ♦ 4 Channels in parallel
- ♦ 6 Samples per channel
- ♦ Touch screen PC
- ♦ Designed for bench top application
- ♦ High precision and reproducibility
- ♦ Zero cross-contamination
- ♦ Eliminate human errors
- ♦ High throughput

Large Receiver SPE Vacuum Manifold

Description

This device is specially designed to collect large volumes of eluent from SPE procedures. It accommodates six samples in parallel and it also provides connection to a vacuum pump for expedited operation. Standard size spin steaming bottle can be connected to the manifold for large amount eluent collection.

Cat No.:VM06

96-Well Plate Vacuum Manifold

Cat. No: VM96

Description

1. Flow rate controlled by vacuum
2. Display operational vacuum
3. Anti-corrosion design
4. Depth-adjusting modular

Suitable for various hydraulic plates and eluents.

Positive Pressure SPE Work-stations

192-position positive pressure SPE work-station

Cat.No: EZY-P192

Description

Two 96-well plate could be run on the device at the same time with the same flow rate for each plate. the flow is continuously controlled and taken effect immediately according to the rate without connecting with electric power, gas, or nitrogen gas.

Features & Benefits

- 96 Positive Pressure Processor provides uniform flow for all extraction steps.
- Provides even pressure to each 96 well plate. Even flow can be maintained whether you are using one, or two 96-well plates.
- Dual flow regulators. Allows users to set 2 different pressures for extraction and drying the plates.
- Uniform drying of extraction plate bed is achieved by flowing nitrogen or moisture-free air at 25 psi.

48-position positive pressure SPE work-station

Cat.No:EZY P48

Description

The work-station can process SPE columns of 1 ml, 3 ml, and 6 ml capacity in batches of 1 to 48 samples. It provides a positive pressure for solid phase extraction using dry inert pressurized air, nitrogen or other inert gases. The standard hardware provided with the unit is designed for 1ml columns, with elution using 12 x 75 mm test-tubes.
The 48 Positive Pressure Processor is designed as an ideal accessory for solid phase extraction applications with the ability to provide set pressure levels for conditioning, sample transfer, wash steps, along with the line pressure for drying prior to the elution step.

Features & Benefits

Modular rack design, allowing quick interchangeability between 1 ml, 3 ml and 6 ml columns, eluting into 12 x 75 mm, 13 x 100 mm and 16 x 100 mm test-tube racks respectively.
Uniform gas distribution through the manifold ensures uniform pressure and uniform liquid flow at each SPE tubes. The uniform flow, ensures uniform flow, with some SPE columns open along with reproducible SPE runs from column to column. It also ensures uniform flow rate when less than 48 columns are used.
Liquid flow rate in the columns changes quickly and proportionately when gas pressure is changed. The pressurized air source can be replaced with other inert, dry gases like nitrogen and helium.

Ordering Information

Cat.No.	Description	Qty/Pk
APSP-48	Positive Pressure Processor-48 Position	1
APSP-22	Rack for 1 ml SPE Columns	1
APSP-23	Rack for 3 ml SPE Columns	1
APSP-24	Rack for 6 ml SPE Columns	1
APSP-19	Collection Tube Rack, 12 x 75 mm Tubes	1
APSP-20	Collection Tube Rack, 13 x 100 mm Tubes	1
APSP-21	Collection Tube Rack, 16 x 100 mm Tubes	1
APSP-25	Collection Vial Rack, 12 x 32 mm Auto-Sampler	1
APSP-26	Waste Bin	1
APSP-27	48-Column Sealing Gasket	1
APSP-28	Gas Supply Adapter	1

Solvent Evaporator

Product Features

- Combination of heating and vacuum for speedy evaporation of a variety of solvents at relative lower temperature
- Simple operation, no programming set-up needed
- Adjustable gas pressure and flow
- Multi-size block interchangeable, accepts tubes OD from 11 to 29 mm
- Optional automatic shutoff, for safe and fast parallel enrichment of various samples
- Small footprint, only 18(W) x 16(D) inch bench area
- Broad customization configurations
- Economic and reliable

1.Height post; 2.Vacuum vent;
3.Needle piercing gas manifold;
4.Sample tubes;
5.Temperature controller;
6.System flow controller;

Technical Specifications

Dimension	31(W) x 22(H) x 42(D) mm
Weight	7 kg
Power	220 V, 50-60 Hz, 560 W
Working environment	0 to 50°C, Humidity <85% RH
Well dimension	16 mm
Number of Well Plate	30
Temperature	RT-150°C
Temperature accuracy	+-1°C
Gas flow	0-8 liter/min
Needle	Stainless steel, 1.6 (OD) x 150 (L)mm
Gas/vacuum connection fitting	Hose barb 1/8 flow port

Multi-Needle manifold with individually controlled flow

Ordering Information

Cat.No.	Description
NV30-G	30-position Solvent evaporator, including a multi-needle manifold, inert gas flow module, heater & controller, 1 year limited warranty.
NV15-G	15-position Solvent evaporator, including a change multi-needle to multi-port manifold, inert gas flow module, heater & controller, 1 year limited warranty.
	Replace with: Note: Multi-well blocks and matching manifold plates need ordering separately.

Empty Columns and Accessories

Ordering Information

Product name	Specification, Package	Cat. No.
Empty Cartridge	1 mL,100 pieces/package	AZ001
	3 mL,100 pieces/package	AZ003
	6 mL,100 pieces/package	AZ006
	12 mL,100 pieces/package	AZ012
	25 mL, 50 pieces/package	AZ030
	60 mL, 50 pieces/package	AZ060
	150 mL, 25 pieces/package	AZ150
Frit	Application for 1-mL cartridge, 100 pieces/package	AS001-A
	Application for 3-mL cartridge, 100 pieces/package	AS003-A
	Application for 6-mL cartridge, 100 pieces/package	AS006-A
	Application for 12-mL cartridge, 100 pieces/package	AS012-A
	Application for 25-mL cartridge, 50 pieces/package	AS030-A
	Application for 60-mL cartridge, 50 pieces/package	AS060-A
	Application for 150-mL cartridge, 25 pieces/package	AS150-A
General adapter	Application for 3-mL, 6-mL, and 12 mL cartridge, 12 pieces/package	A80115
IC cartridge	1-mL IC cartridge	AZ-IC-1
	1-mL IC cartridge (with frit)	AZ-IC-1T
	2.5-mL IC cartridge	AZ-IC-2.5
	2.5-mL IC cartridge (with frit)	AZ-IC-2.5T

Large Volume Sampling cartridge

Ordering Information

Product name	Specification	Cat. No.
Large Loading Column (30 mL)	1 piece/package	A82030
Large Loading Column (60 mL)	1 piece/package	A82060
Water Loading Pipeline	1 piece/package	A80116

96-Welll Filtration Plates and Collection Plates

Ordering Information

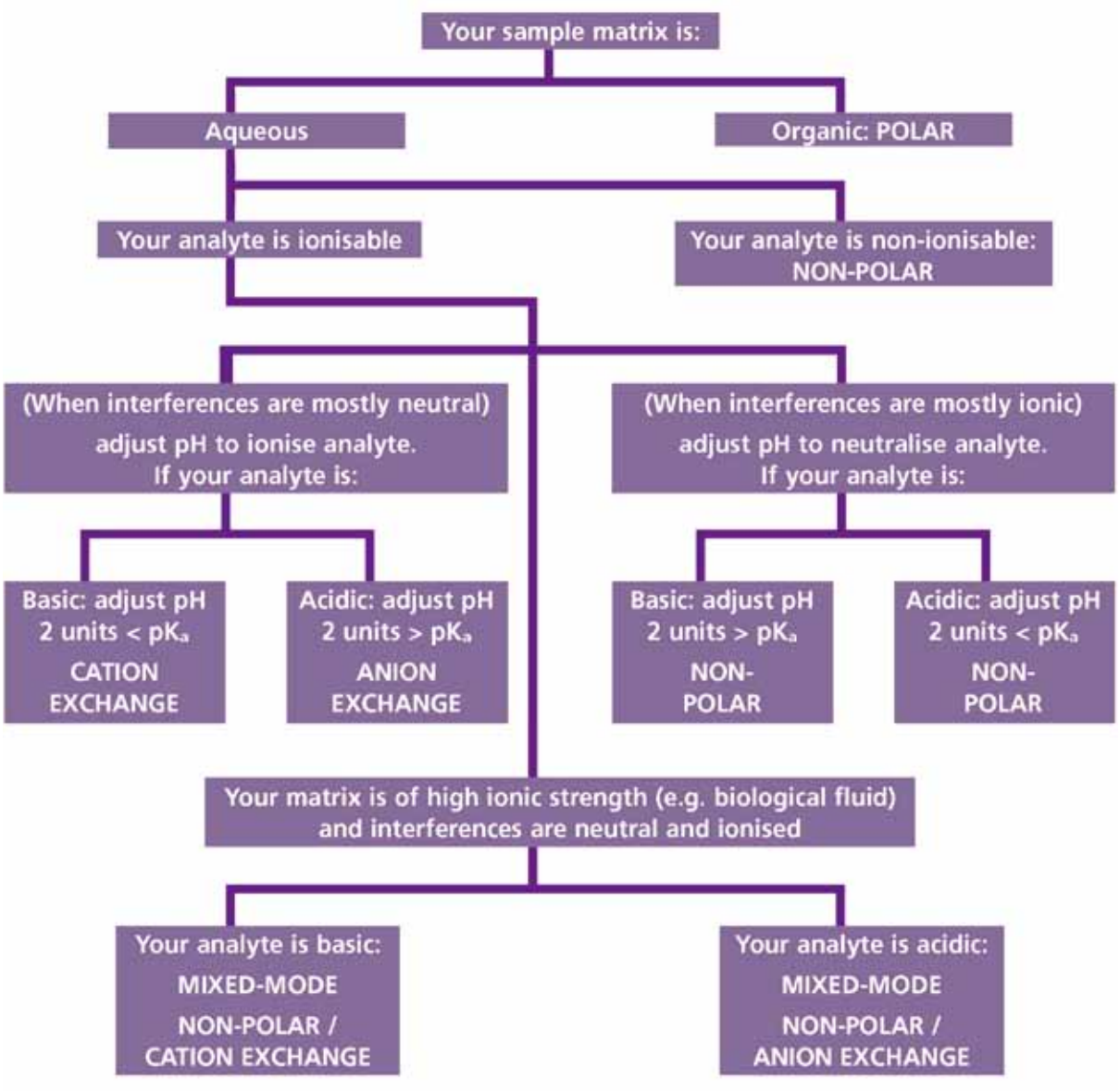
Product name	Specification	Description	Quantity	Cat. No.
1 mL 96-Well collection plate	8 × 12 pores	square pore and circular bottom	1	96SP1036
2 mL 96-Well collection plate	8 × 12 pores	square pore and circular bottom	1	96SP2036
2 mL 96-Well collection plate	8 × 12 pores	circular pore and bottom	1	96SP2036-Y
96-well silica pad	8 × 12 pores	square pore	1	96GP2036

Method Development of SPE Procedures

Many factors impact SPE procedures. For SPE method deveopment, however, here are some selection guides.

The Selection of Sorbent Retention Mechanism

The guide on this page briefly outlines the decision making process required to choose a suitable extraction mechanism.



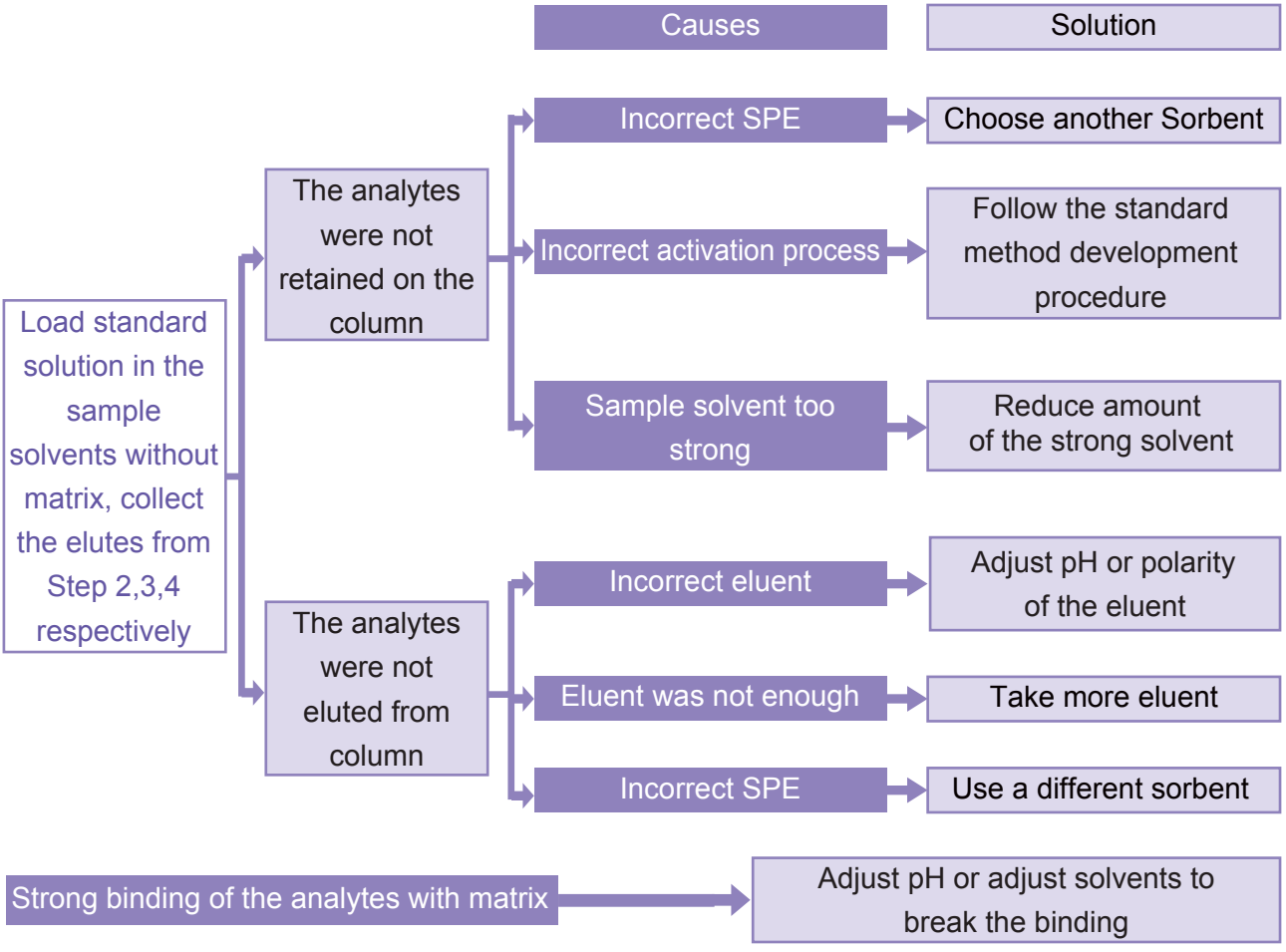
SPE Product Cross Reference Table

This guide directs the selection of an appropriate sorbent or chemistry for a SPE procedure.

	Agela	Waters	Supelco	Aglient	Varian
C18 (end capping)	Cleanert® C18	Sep-pak C18	ENVI-18	—	Bond Elut C18
C18 (without end capping)	Cleanert® C18-N	—	—	AccuBOND C18	Bond Elut C18-OH
C8	Cleanert® C8	Sep-pak C8	ENVI-8	AccuBOND C8	Bond Elut C8
Cyano group	Cleanert® CN	Sep-pak CN	LC-CN	AccuBOND CN	Bond Elut CN
Amino group	Cleanert® NH_2	Sep-pak NH_2	LC- NH_2	AccuBOND NH_2	Bond Elut NH_2
Propyl ethylene diamine	Cleanert® PSA	—	—	—	Bond Elut PSA
Quaternary ammonium salt (Strong anion-exchange cartridge)	Cleanert® SAX	—	LC- SAX	AccuBOND SAX	Bond Elut SAX
Carboxyl group (Weak cation-exchange cartridge)	Cleanert® COOH	—	LC- WCX	—	Bond Elut CBA
Propylsulfonic acid	Cleanert® PRS	—	—	—	Bond Elut PRS
Benzene sulfonic acid (Strong cation-exchange cartridge)	Cleanert® SCX	—	LC- SCX	AccuBOND SCX	Bond Elut SCX
Silica gel	Cleanert® Silica	Sep-pak Silica	LC- Silica	AccuBOND Silica	Bond Elut Silica
Diol	Cleanert® Diol	Sep-pak Diol	LC-Diol	AccuBOND Diol	Bond Elut Diol
Polystyrene/divinyl-benzene	Cleanert® PS	—	ENVI-Chrom P	AccuBOND ENV PS-DVB	—
Polar polymer cartridge	Cleanert® PEP-2	Oasis HLB	—	—	Bond Elut® Plexa
Mixed anion-exchange cartridge	Cleanert® PAX	Oasis MAX	—	—	—
Mixed cation-exchange cartridge	Cleanert® PCX	Oasis MCX	—	—	—
Cartridge specialized for sulfonylureas	Cleanert® HXN	—	—	—	—
Magnesium silicate (Florisil)	Cleanert® Florisil	Sep-pak Florisil	LC Florisil	—	Bond Elut FL
Graphitized carbon	Cleanert® PestiCarb	—	ENVI Carb	—	—
Neutral alumina	Cleanert® Alumina N	Sep-pak Alumina N	LC- Alumina N	AccuBOND Alumina N	Bond Elut Alumina N
Acidic alumina	Cleanert® Alumina A	Sep-pak Alumina A	LC- Alumina A	AccuBOND Alumina A	Bond Elut Alumina A
Basic alumina	Cleanert® Alumina B	Sep-pak Alumina B	LC- Alumina B	AccuBOND Alumina B	Bond Elut Alumina B
Mixed graphitized carbon and amino group cartridge	Cleanert® PestiCarb/NH_2	Sep-pak Carb/ NH_2	ENVI Carb/ NH_2	—	—
Cartridge specialized for sulfanilamides	Cleanert® SUL-5	—	—	—	—
DNPH-Silica cartridge (specialized for pretreatment of aldehydes and ketones in air)	Cleanert® DNPH-Silica	Sep-pak DNPH-Silica	—	—	—
Solid supported liquid/liquid Extraction columns	Cleanert® SLE	—	—	—	Chem Elut SLE

Trouble Shooting

Low Recovery



High Deviation

SPE was dry during activation (Only for C18, C18-N, C8)	Re-activated the cartridge or change another SPE cartridge
SPE cartridge was over loading	Reduce the sample amount or increase the sorbent amount
Flow rate was too high	Reduce the flow rate below 5 mL/min
The solution used in four steps was not miscible to each other	Dry the column when change different solution
The washing solution was too strong	Change the polarity of the washing solution
The loading solution was too strong	Change the polarity and pH of the loading solution

Clogging

The sample contains colloidal particles	Centrifuge or filtration before SPE
The sample was sticky	Dilute the sample

Standard Method Development Procedure

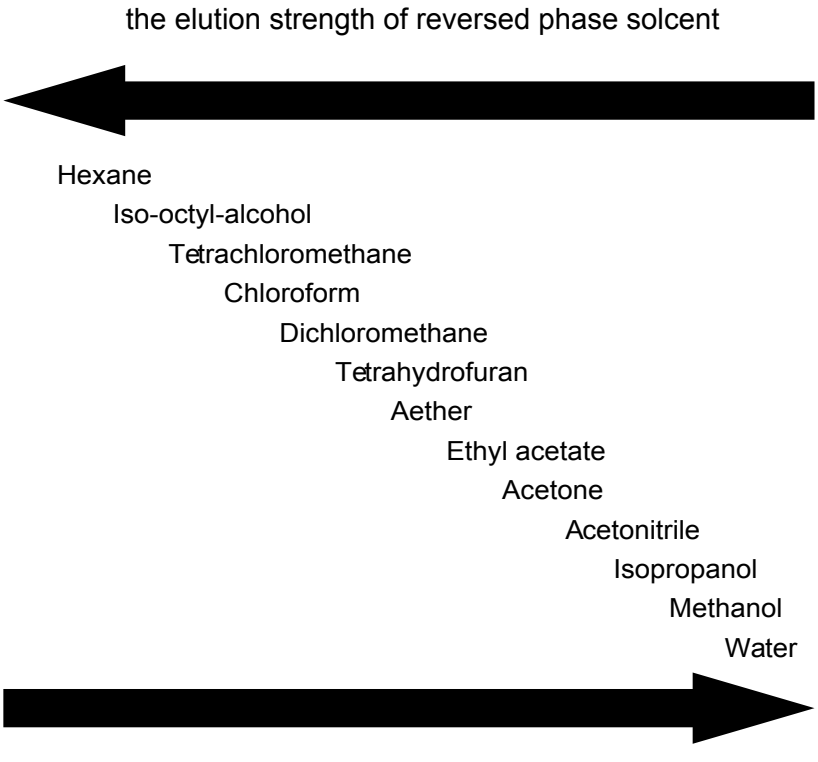
Properties	Sorbent Phase Type			
	Normal Phase	Reversed-Phase	Ion Exchange Phase or Mix-phase	
Typical Sorbents	Silica Florisil Amine (NH_2) Cyano (CN) Diol	PEP series C18 C18-N C8	PAX SAX PWAX	PCX SCX PWCX
Sorbent Polarity	High	Low	High	
Matrix Properties	Organic	Organic or Aqueous	Organic or Aqueous	
Analyte Properties	Slightly to Moderately Polar	Non-Polar or Polar	Acidic	Basic
Retention	Polar Non-Polar	Non-Polar polar	Ionic Strength	
Step 1: Activation	Sample solvent or other organic solvents	Water-miscible organic solvents followed by Water	Water-miscible organic solvents followed by aqueous solution with pH adjusted	
Step 2: Sample Loading	Load the sample or with dilution in low polarity solvents	Load the sample or with dilution in high polarity solvents	Load sample or with dilution in high polarity solvents with pH adjusted	
Step 3: Washing	Washing with low-polar solvents	Washing with mixture of aqueous solution or buffer with a small amount polar solvent	Washing with Polar organic solvents followed by aqueous solvents with pH adjustment to maintain analytes ionized	
Step 4: Elution	Eluting with mixture of non-polar and polar solvents	Eluting with non-polaror polar organic solvents	Eluting with polar solvents with pH adjustment	

Size, Capacity And Elution Volume In SPE Process

For the normal phase and reversed phase SPE cartridges, the weight of sample can not exceed the 5% of sorbent weight. For the ion-exchange mode, the capacity of the ion-exchange must be considered.
The table below is the capability and eluted parameter of SPE:

Specification	Quality of Loading Sample	The Minimum Volume of Elution
50 mg/1 mL	2.5 mg	125 µL
100 mg/1 mL	5 mg	250 µL
200 mg/3 mL	10 mg	500 µL
500 mg/6 mL	25 mg	1.2 mL
1 g/6 mL	50 mg	2.4 mL

The Selection of Ideal Elution Solvent



Applications

1. Application in Veterinary Drug Residues

Determination of Four β- Agonist Drugs Residues (Clenbuterol Hydrochloride, Salbutamol, Cimaterol and Ractopamine etc.) in Animal Tissues (Cleanert® PCX, P/N: CX1506)

Material

(1) SPE Cartridge: Cleanert® PCX (150 mg/6 mL);
(2) Four β- agonist drugs: Clenbuterol hydrochloride, Salbutamol, Cimaterol and Ractopamine;

Experiment

Sample preparation
Blank samples are spiked with standard solution at certain concentration, and then extracted by liquid-liquid partition.

PCX Procedure
- Condition the PCX cartridge with 5 mL methanol, 5 mL of deionized water and 5 mL of 30 mM HCl solution sequentially.
- Load sample concentrate onto the cartridge.
- Wash the loaded cartridge with 5 mL water followed by 5 mL methanol, and discard the eluate.
- Dry the cartridge by passing through nitrogen gas.
- Elute the cartridge with 5 mL methanol containing 4% ammonia, and collect the eluate into a glass test tube, and then dry the eluate at 50 Celsius under a gentle flow of nitrogen (ca. 1 mL/min).

Derivatization and detection
Heat the sample tube in an oven at 50 Celsius for a few minutes to remove water. Add 100 µL of toluene and 100 µL of N,O-Bis (trimethylsilyl) trifluoroacetamide (BSTFA) to the test tube. Vortex mix for 20 s. Seal the test tube and place it in oven at 80°C for 1 h. Cool and add 300 µL of toluene to the test tube. The solution is ready for GC-MS analysis (GC column: DA-5MS, 30 m×0.25 mm×0.25 µm, P/N: 1525-3002).

Results

Recovery
Samples of pig liver, spiked with 1, 2, 5, 10, and 100 ug/L, respectively, were extracted by liquid-liquid partitioning. Four batches of samples were tested. In each batch, 5 replications were used for each concentration. The results of the recovery test are listed in the following table.

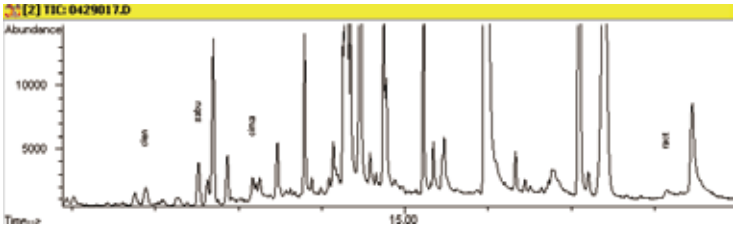
Results of analysis of pig liver samples:

Spiked concentration (μg/L)	Measured concentration (μg/L)	Mean measured concentration (μg/L)	Mean percent recovery (%)	RSD (%)
1	0.75	0.72	72.40	5.93
	0.67			
	0.72			
	0.70			
	0.78			
2	1.62	1.63	81.30	1.23
	1.66			
	1.60			
	1.61			
	1.64			
5	4.02	4.24	84.80	4.16
	4.10			
	4.27			
	4.38			
	4.43			
10	8.24	8.45	84.45	2.81
	8.35			
	8.77			
	8.62			
	8.25			
100	90.24	9.12	91.15	2.86
	87.15			
	91.77			
	92.62			
	93.95			

Repeatability Experiment
Results of analysis of pig liver samples:

Batch	Spiked concentration (μg/L)									
	1		2		5		10		100	
	Mean recovery %	RSD%	Mean recovery %	RSD%	Mean recovery %	RSD%	Mean recovery %	RSD%	Mean recovery %	RSD%
1	72.40	5.93	81.30	3.49	84.80	6.16	84.45	3.59	91.15	2.86
2	75.37	6.12	80.47	5.37	84.74	7.55	87.46	4.68	90.05	3.86
3	70.09	7.85	80.80	6.57	83.10	8.17	83.21	5.39	89.53	4.16
4	76.73	4.90	78.50	8.35	82.90	5.11	85.95	5.72	88.27	5.93
Average	73.65	6.20	80.25	5.95	83.88	6.75	85.27	4.84	89.75	4.20
RSD%	12.95		10.79		9.43		7.00		5.75	

Figure: Typical total ion chromatography (TIC) of spiked pig liver samples at six concentrations: 0.5 μg/L, 1 μg/L, 2 μg/L, 5 μg/L, 10 μg/L and 100 μg/L: liver + 1 ppb (pcx)



Determination of Five Sulfonamides in Pork (Cleanert® SUL-5, P/N: SUL-5)

Material

SPE cartridge: Cleanert® SUL-5 (2 g/12 mL)
Five sulfonamides: sulphadimidine (SM2), Sulfamonomethoxine (SMM), Sulfamethoxazolum (SMZ), Sulfadimoxine (SDM), and Sulfaquinoxaline (SQ).

Experiment

Spike sample was prepared by diluting 50 or 100 uL of working solution in 3.00 mL acetonitrile/water (95:5, v/v). Use the follow steps for the experiment:

1) Add 5 mL acetonitrile/water (95:5, v/v) to activate the cartridge
2) Load the sample (3 mL) onto the cartridge
3) Wash the cartridge with 5 mL acetonitrile/water (95:5, v/v), and discard the eluate.
4) Elute the cartridge with 10 mL acetonitrile/water (7:3, v/v), and collect the eluate.
5) Inject 20 uL of the collected eluate to HPLC for analysis.

Cartridge batch	Drugs	Recovery (%)			Mean recovery (%)	RSD (%)
Batch 1	SM_2	94.37	96.10	95.19	95.22	0.91
	SMM	96.97	95.38	96.10	96.15	0.83
	SMZ	89.95	90.96	98.50	93.14	5.01
	SDM	94.66	98.15	95.08	95.96	1.99
	SQ	93.48	91.85	90.55	91.96	1.59
Batch 2	SM_2	94.18	95.49	101.13	96.93	3.81
	SMM	97.55	88.94	96.90	94.46	5.07
	SMZ	91.73	87.87	94.46	91.35	3.62
	SDM	87.22	99.60	97.87	94.90	7.06
	SQ	92.18	94.80	93.17	93.38	1.43
Batch 3	SM_2	95.08	94.76	94.52	94.79	0.30
	SMM	99.63	95.11	95.96	96.90	2.48
	SMZ	96.40	98.16	87.26	93.94	6.23
	SDM	96.79	96.66	94.55	96.00	1.31
	SQ	96.32	91.38	92.49	91.93	0.85

Sample spiked recovery.
Add 5 g (accurate to 0.01 g) of tissue sample to a centrifuge tube containing 10 g of anhydrous sodium sulfate. Add 25 mL of acetonitrile. Homogenize at 10000 r/min for 1 min and centrifugate at 3000 r/min for 5 min. Dissolve the residue in 25 mL of acetonitrile and centrifugate at 3000 r/min for 5 min. Combine the supernatants of two centrifugations. Add 30 mL of hexane and vortex mix for 10 min. Centrifugate at 3000 r/min for 5 min and discard the supernatant layer. Add 10 mL of n-propanol and evaporate to dryness under reduced pressure at the temperature below 50°C. Dissolve the residue in 3 mL of 95% acetonitrile and load onto basic alumina cartridge. Wash the cartridge with 5 mL of 95% acetonitrile and then elute with 10 mL of 70% acetonitrile. Collect the eluate for HPLC analysis.

Results

Cartridge retention test

SUL-5 retention test was conducted at two concentrations, 100 and 200 ug/Kg.The results are summarized in Tables 1 and 2. The mean recovery ranges of SM2, SMM, SMZ, SDM, and SQ are 92.47~99.37%, 93.69~99.44%, 88.61~96.27%, 90.87~96.06% and 91.83~95.92%, respectively. The intra-batch RSD of Cleanert cartridge is 0.30~9.38%, showing good stability.

Sample recovery

Table 3 shows the results of recovery test of spiked samples with fortified concentration 100 and 200 ug/Kg, respectively. 3. The mean recovery and intra-batch RSD for spiked pork samples (100 and 200 μg/kg) are 80.62~94.49% and 3.98~7.79%, respectively, indicating that the Cleanert Series SPE cartridge has high recovery and good stability.

Table 1 Cartridge retention recovery of Cleanert SUL-5 cartridge (corresponding to 100 μg/kg sulfonamides in tissues)

Cartridge batch	Drugs	Recovery (%)			Mean recovery (%)	RSD (%)
Batch 1	SM_2	94.37	96.10	95.19	95.22	0.91
	SMM	96.97	95.38	96.10	96.15	0.83
	SMZ	89.95	90.96	98.50	93.14	5.01
	SDM	94.66	98.15	95.08	95.96	1.99
	SQ	93.48	91.85	90.55	91.96	1.59
Batch 2	SM_2	94.18	95.49	101.13	96.93	3.81
	SMM	97.55	88.94	96.90	94.46	5.07
	SMZ	91.73	87.87	94.46	91.35	3.62
	SDM	87.22	99.60	97.87	94.90	7.06
	SQ	92.18	94.80	93.17	93.38	1.43
Batch 3	SM_2	95.08	94.76	94.52	94.79	0.30
	SMM	99.63	95.11	95.96	96.90	2.48
	SMZ	96.40	98.16	87.26	93.94	6.23
	SDM	96.79	96.66	94.55	96.00	1.31
	SQ	96.32	91.38	92.49	91.93	0.85

Table 2 Cartridge retention test recovery of Cleanert SUL-5 cartridge (corresponding to 200 μg/kg sulfonamides in tissues)

Cartridge batch	Drugs	Recovery (%)			Mean recovery (%)	RSD (%)
Batch 3	SM_2	99.66	99.26	98.75	99.22	0.46
	SMM	97.46	100.25	96.37	98.03	2.04
	SMZ	96.35	94.42	97.06	95.94	1.42
	SDM	94.97	94.86	98.34	96.06	2.06
	SQ	98.22	91.96	97.59	95.92	3.59
Batch 4	SM_2	100.45	85.90	91.06	92.47	7.97
	SMM	94.67	88.58	97.83	93.69	5.02
	SMZ	95.96	81.25	88.63	88.61	8.30
	SDM	99.83	82.87	89.90	90.87	9.38
	SQ	96.21	87.89	91.40	91.83	4.54
Batch 5	SM_2	98.69	100.31	99.11	99.37	0.85
	SMM	101.51	100.78	96.04	99.44	2.99
	SMZ	91.35	98.78	98.68	96.27	4.43
	SDM	92.75	97.29	97.88	95.97	2.92
	SQ	84.27	98.61	98.20	93.69	8.71

Table 3 Results of recovery test of spiked samples

Spiked concentration (μg/kg)	Drugs	Recovery (%)					Mean Recovery (%)	RSD (%)
100	SM2	93.39	97.95	98.87	90.33	98.27	94.49	4.83
	SMM	97.46	90.66	94.33	81.83	93.64	92.40	6.17
	SMZ	83.78	88.45	94.40	80.24	82.04	84.87	6.56
	SDM	91.26	96.75	91.79	87.57	85.88	90.28	4.30
	SQ	88.44	85.14	92.61	81.03	79.57	85.06	5.69
200	SM2	80.85	77.37	88.39	90.53	73.80	82.02	7.79
	SMM	91.79	88.87	96.23	92.96	87.01	90.70	3.98
	SMZ	79.60	77.27	87.34	86.98	75.44	81.52	6.09
	SDM	84.31	72.35	84.52	81.92	82.53	80.62	5.79
	SQ	91.93	81.86	91.83	87.89	86.17	87.76	4.33

Determination of Terramycin, Tetracycline and Aureomycin in Aquatic Products and Meat (Cleanert® PS, P/N: PS2003)

Material

Cleanert® PS (200 mg/3 mL)

Experimental

Procedure

- To a 100 mL centrifuge tube, add 5 g homogenized sample
- Add 2 x 20 mL citric acid/EDTA solution to extract target compounds. Vibrate the mixture in a mechanical shaker for 15 minutes.
- Centrifuge the mixture for 10 minute at 4000 rpm.
- Transfer the supernatant into a separating funnel, and add 20 mL hexane (to remove fat), and the shake the mixture for 5 minutes.
- Transfer the aqueous substratum into a sample flask for SPE cleanup.

Note: Citric acid/EDTA solution was prepared by dissolving 1.86 g EDTA-2Na into a mixture of 307 mL 0.1 M acid buffer solution and 193 mL 0.5 M disodium hydrogen phosphate solution.

Cleanert® PS Procedure

- Activate the PS cartridge by sequentially adding 10 mL methanol, 10 mL water and 5 mL EDTA-2Na solution.
- Load sample solution prepared from above procedure; and then wash the cartridge with 10 mL water, and discard the eluate.
- Dry the cartridge via positive pressure.
- Elute the cartridge with 10 mL methanol and collect the eluate.

Sample concentration and reconstitution

- Evaporate the sample eluate under rotovap to nearly dryness.
- Reconstitute the sample residue with 1 mL HPLC mobile phase.
- Filter sample through 0.45 um membrane before HPLC analysis.

Condition of HPLC
Column: Unisol C18, 5 μm, 4.6 × 250 mm
Mobile phase: A:B = 77:23

A: Iminazole buffer solution—dissolve 68.08 g of iminazole, 10.72 g of magnesium acetate and 0.37 g of EDTA2Na in 100 mL of water; modify pH to 7.2 with glacial acetic acid; bring the solution to 1000 mL with water.

B: Acetonitrile

Results

Compound	Recovery-1	Recovery-2
Terramycin	128%	137%
Tetracycline	91.8%	97.4%
Aureomycin	88.5%	87.1%
Doxycycline	97.3%	95.3%

Determination of Tetracycline in Honey (Cleanert® PEP, Cleanert® COOH, P/N: PE5006, CH5003)

Material

Cleanert PEP, 500 mg/6 mL
Cleanert COOH, 500 mg/3 mL

Experimental

Sample preparation
Dissolve 6.00 g honey sample in 30 mL extract solution; Vortex the mixture until the solution is clear; then the sample is ready for cleanup.
(Extract solution: Dissolve 10.5 citric acid and 8.88 g disodium hydrogen phosphate and 30.3 g EDTA-2Na in ca. 800 mL water; adjust pH to 4.0 by adding 2 M HCl and bring up total volume to 1000 mL with additional water if needed.)

Cleanert® PEP and COOH cleanup
(1) Cleanert® PEP (500 mg/6 mL)
- Add 5 mL methanol and then 5 mL water
- Load sample solution and then wash the cartdrige with 5 mL methanol/water (5:95, v/v)
- Dry the cartridge under positive pressure

(2) Cleanert® COOH (500 mg/3 mL)
Condition: 5 mL of ethyl acetate
(3) Cleanert® PEP (top) – Cleanert® COOH (down) in series
Washing: ethyl acetate (15 mL), remove Cleanert® PEP cartridge, dry Cleanert® COOH cartridge (5 min)
Elution: mobile phase (0.01 M oxalic acid: acetonitrile: methanol = 350:100:50) 4.5 mL
Dilute the eluate with 0.01 M oxalic acid in volumetric flask to 5 mL. Shake the volumetric flask to obtain homogeneous solution. Filter the solution before analyzed by UPLC.

Determination of Chloramphenicol Residue in Aquatic Products by Gas Chromatography (Cleanert® C18, P/N: S180006)

Material

Cleanert C18, 1 g/6 mL
DA-5, 30 m×0.53 mm×1.5 μm, P/N: 0153-3015.)

Experimental

Sample preparation
For Fish: remove scales and skins, take the back muscles.
For Shrimp and Crab: remove the head, shell and limbs, take the eatable muscles.
Cut the muscle samples into small pieces (no bigger than 0.5 cm × 0.5 cm × 0.5 cm) and mix. Freeze in fridge for later use.

Extraction

Put 5.00 g fresh or fully defrozen sample into a glass centrifuge tube, and then add 20 mL ethyl acetate. Homogenize the mixture for 1 minute to extract target compound. Centrifuge the mixture at 4000 rpm for 3 minutes, and then tranfer ethyl acetate layer to a 100-mL pear-shaped flask. Add 10 mL ethyl acetate to the sample sediment to extract one more time following the previous procedure. Evaporate the combined extract to dryness under a rotovap at 40 Celsius.

Degreasing

- Add 1 mL methanol to reconstitute sample in the pear-shaped bottle from last step.
- Add 2 × 15 mL n-hexane and 2 × 25 mL 4% NaCl solution into the sample to remove lipids. For each replicate, first vortex the mixture for 1 minute followed by 2 minute centrifuging at 4000 rpm. Discard the hexane layer; and then combine the aqueous layer for next step operation.
- Add 2 × 15 mL ethyl acetate to the aqueous layer; then vortex the mixture for 2 minutes; and then centrifuge the mixture at 3000 rpm for 3 minutes.
- Collect the ethyl acetate layer; and pass the solution through a bed of sodium sulfate anhydrous to remove water content.
- Rotovap the solution to nearly dryness at 40 Celsius.
- Dissolve the sample residue in 2 mL ethyl acetate for SPE procedure.

Cleanert® C18 (1 g/6 mL) Procedure

- Sequentially add 5 mL each of methanol, chloroform, methanol and water, respectively, to activate and to condition C18 cartridge.
- Load the sample on the C18 cartridge; and adjust the elution speed to below 1 mL/min.
- Wash the cartridge with 6 mL water and discard eluate.
- Dry the cartridge under positive pressure.
- Elute the cartridge with 5 mL acetonitrile and collect the eluate.
- Evaporate the sample solution under nitrogen to nearly dryness in a 50 Celsium sand bath.
- Rinse the sample vail with 1 mL ethylacetate; and then evaporate the solvent again under nitrogen to dryness.

Derivatization

Add 100 µL of derivatization reagent (N,O-bis (trimethylsilyl) trifluoroacetamide (BSTFA) /trimethylchloro-silane (TMCS), 99/1, v/v) to the dried residue. Cap the tube and vortex and mix for 10 s. Allow to react in oven at 70 for 30 min. Remove the excess reagent under nitrogen stream in sand bath at 50~55°C until the tube is just dried. (Note: Too long time of evaporation may result in loss of analytes.) Add 0.5 mL of n-hexane, vortex and mix for 10 s for the analysis of GC (DA-5, 30 m×0.53 mm×1.5 µm, P/N: 0153-3015.)

Determination of Nitrofuran Residues with LC-MS (Cleanert® PEP, P/N: PE0603)

Material

Cleanert® SPE Cartridge: PEP (60 mg/3 mL)

Experimental

Sample preparation

(1) Treatment of milk powder and milk

1) Sample treatment: add 15 mL of solution mixed by trichloroacetic acid and water (2 and 15 mol) in milk powder of 1 g (milk of 5 g); add the internal standard and mixed standard; hydrolyze in water bath at 37.5°C for 5 h; centifuge at 4000 rpm for 5 min and then get the supernatant for usage.
2) Initial cleanup and derivation of the sample: activate the Cleanert® PEP cartridge with 5 mL of methanol and 5 mL of water; pass the treated supernatant in SPE cartridge and wash the cartridge with 5 mL of trichloroacetic acid; collect the solution into another test tube; derive the derivating agent of 100 µl (dissolve 20 mg 2-nitrobenzaldehyde in dimethylsulfoxide of 1 mL) at 37.5°C for 16 h in the water bath (overnight).

(2) Treatment of the pork, beef, chicken, pork liver, aquatic product and honey sample.

1) Bulk processing of samples: add 15 mL of mixed solution of methanol and water in 2 g of pork, beef, chicken, pork liver and aquatic product sample respectively (5 g of honey) and vortex; centrifuge at 4000 rpm for 5 min and add the interior label and mixed label into the supernate.

2) Deriving add 1 mL of derivating agent (dissolve 20 mg of 2-nitrobenzaldehyde in 1 mL dimethylsulfoxide) into the supernate; derivate the solution in the water bath of 37.5°C for 16 h (overnight); add dipotassium phosphate of 5 mL (pH = 7.4) and centrifuge at 4000 rpm for 10 min. (add 5 mL of n-haxane to the supernate if there is too much fat in the sample; adsorb and remove n-haxane by vibration for 2 min and centrifuging at 4000 rpm for 10 min).

Cleanert® PEP Cleanup

(1) Activation: activate the cartridge with 5 mL of methanol and 5 mL of water.

(2) Sample loading: add the buffer solution of dipotassium phosphate 5 mL in the derivative solution; adjust the pH to 7.4 with 1 mol/L of sodium hydroxide solution; centrifuge at 4000 r/min for 10 min; keep the supernate (add 5 mL of n-haxane to the supernate if there is too much fat in the sample; absorb and remove n-haxane by vibration for 2 min and centrifuging at 4000 rpm for 10 min) going through the PEP cartridge with the flow rate less than 2 mL/min.

(3) Washing: elute SPE cartridge with 10 mL of water and remove all outflow; dry SPE cartridge of PEP for 15 min under the negative pressure of 65 kPa with the vaccum pump.

(4) Elution: elute the tested sample with 5 mL of ethylacetate into 25 mL-brown centrifuge tube.

(5) Concentration: dry the eluate with nitrogen at 40°C; dissolve the solution and fix the volume to 1.0 mL with the sample of constant volume solution; filter through the filter membrane of 0.2 μm by LC-MS after mixing.

Determination of 19 Quinolone Residues in Honey by LC-MS/MS (Cleanert® PAX, P/N: AX0603)

Material

(1) Quinolone standards: Enrofloxacin (ENR), Ciprofloxacin (CIP), Norfloxacin (NOR), Ofloxacin (OFL), Flumequine (FLU), Oxolinic acid (OXO), Difloxacin HCl (DIF), Sarafloxacin HCl (SAR), Sparfloxacin (SPA), Danofloxacin (DAN), Fleroxcain (FLE), Marbofloxacin (MAR), Enoxacin (ENO), Orbifloxacin (ORB), Pipemidic acid (PIP), Pefloxacin (PEF), Lomefloxacin (LOM), Cinoxacin (CIN), Nalidixic acid (NAL). The purities of all above standards are all ≥99%.

(2) SPE cartridge: Cleanert® PAX (60 mg/3 mL).

(3) Internal standard stock solution of deuterated Norfloxacin (NOR-D5): dissolve proper amount of NOR-D5 standard in methanol to obtain internal standard stock solution of 100 μg/mL. Dilute proper amount of the internal standard stock solution with methanol to obtain working solution at the concentration of 1 μg/mL and store at 4°C.

Experimental

Cleanert® PAX Procedure

Weigh 5 g (accurate to 0.01 g) of sample and add to a 50 mL of centrifuge tube with stopper. Add 50 μL of 1 μg/mL internal standard solution and 5 mL of 0.1 mol/L sodium hydroxide solution. Vortex mix to dissolve the honey thoroughly. Activate Cleanert PAX SPE mini-cartridge with 5 mL of methanol followed by 3 mL of water. Load the sample solution onto the mini-column. Wash the cartridge sequentially with water and methanol. Elute with 3 mL of methanol containing 5% formic acid. Collect the eluate and evaporate to dryness by rotary evaporator in water bath at 40°C. Dilute to 1.0 mL with 20% methanol in water. Filter through 0.45 μm membrane to sample vial for LC-MS analysis.

Determination

(1) LC conditions:

a) Column: Unisol C18, 3 μm, 150 mm × 2.0 mm i.d or equivalent;
b) Mobile phase: methanol + water containing 0.1% formic acid;
c) Flow rate: 0.20 mL/min;
d) Gradient elution procedure: (omitted);
e) Temperature: room temperature;
f) Injection volume: 25 μL;

(2) MS methods: (omitted)

Please download the details at the website of Bonna-Agela: www.bonnaagela.com

Determination of Nitroimidazole Drugs and Metabolites Residues in Royal Jelly with LC-MS/MS (Cleanert® PAX, P/N: AX0603)

Material

(1) Standards: Metronidazole (MNZ), Dimetridazole (DMZ) and related metabolite-2 - hydroxymethyl--1 - methyl-5–nitroimidazole (HMMNI), Ipronidazole (IPZ) and related metabolite-2-(2-hydroxy isopropyl)-1-methyl-5 –nitroimidazole (IPZOH), Ronidazole (RNZ). The purities of all above standards are all ≥99%.

(2) SPE cartridge: Cleanert® PAX (60 mg/3 mL)

(3) Preparation of Internal standard solution of deuterated Norfloxacin (NOR-D5): dissolve proper amount of NOR-D5 standard in MeOH to obtain internal standard solution at 100 μg/mL. Dilute proper amount of the internal standard solution with MeOH to a concentration of 1 μg/mL and store at 4°C.

Experimental

Sample Extraction and Cleanert® PAX Cleanup

Add 5 g (accurate to 0.01 g) of sample to a 50 mL centrifuge tube with cap. Add 50 μL of mixture of three internal standards (1.3) and 10 mL of 0.5 mol/L sodium hydroxide solution. Mix for 15 s to dissolve the sample. Add 10 mL of ethyl acetate and mix for 30 s. Centrifugate at 2500 r/min for 3 min. Transfer the supernatant ethyl acetate layer to a 50 mL glass test tube. Add 10 mL of ethyl acetate again and repeat the extraction procedures. Combine the ethyl acetates and evaporate to dryness by rotary evaporator in water bath at 40°C. Dissolve the residue with 5 mL of acetonitrile containing 10% formic acid. Activate the SPE cartridge with 3 mL of methanol and 3 mL of water. Load the sample onto the catridge. Wash the cartridge with 3 mL of water and draw almost to dryness. Elute with 3 mL of methanol containing 5% ammonia. Collect the eluate and evaporate to dryness by rotary evaporator in water bath at 45°C. Dilute with 1.0 mL of water containing 20% methanol. Filter through membrane to sample vial for LC-MS/MS analysis.

Determination

(1) Reference conditions for LC analysis;

a) Column: C18 (end capped), 3 μm, 150 mm × 2.0 mm i.d or equivalent;
b) Mobile phase: methanol (A) + 5 mmol/L ammonium acetate(B);
c) Flow rate: 0.20 mL/min;
d) Gradient elution procedure: (omitted);
e) Temperature: room temperature;
f) Injection volume: 25 μL;

Please download the details from the website of Bonna-Agela: www.bonnaagela.com

Determination of Glucocorticoids Drugs Residues in Animal-derived Foods with LC-MS (Cleanert® Silica, P/N: SI5006)

Material

Cleanert Silica 500 mg/6 mL

Experimental

Extraction

(1) Muscle tissue sample

Weigh 5 g (±0.05 g) of tissue sample in a 50 mL centrifuge tube. Add 30 mL of ethyl acetate and 10 g of anhydrous sodium sulfate. Homogenize for 1 min and vibrate on shaker at 200 r/min for 20 min. Centrifugate at 4200 r/min for 10 min and take the ethyl acetate layer. Add 25 mL of ethyl acetate to the residue. Homogenize for 1 min and vibrate on shaker at 200 r/min for 20 min. Centrifugate at 4200 r/min for 10 min and take the ethyl acetate layer. Combine the ethyl acetate layers and evaporate by rotary evaporator in water bath at 40°C almost to dryness. Dissolve the residue in 1 mL of ethyl acetate and 5 mL of n-hexane for cleanup.

(2) Bovine milk, egg sample

Weigh 5 g (±0.05 g) of bovine milk or egg sample in a 50 mL centrifuge tube. Add 30 mL of ethyl acetate. Homogenize for 1 min and vibrate on shaker at 200 r/min for 20 min. Centrifugate at 4200 r/min for 10 min and take the ethyl acetate layer. Add 25 mL of ethyl acetate to the residue. Homogenize for 1 min and vibrate on shaker at 200 r/min for 20 min. Centrifugate at 4200 r/min for 10 min and take the ethyl acetate layer. Combine the ethyl acetate layers and evaporate by rotary evaporator in water bath at 40°C almost to dryness. Dissolve the residue in 1 mL of ethyl acetate and 5 mL of n-hexane for cleanup.

Cleanert® Silica Cleanup

Load the extract onto silica cartridge activated with 6 mL of n-hexane. Wash the cartridge with 6 mL of n-hexane and dry it. Elute with 6 mL of n-hexane-acetone (6/4 v/v). Evaporate the eluate under a stream of nitrogen at 50°C to dryness. Dissolve the residue in 0.5 mL of water containing 20% acetonitrile and transfer to a 1.5 mL centrifuge tube. Centrifugate at 4200 r/min for 20 min. Filter supernatant layer through 0.22 μm membrane for HPLC-MS/MS analysis.

Determination of Zearanol Drugs Residues in Animal-derived Foods with LC-MS (Cleanert® NH_2, PAX, P/N: NH5006, AX1506)

Material

Cleanert® NH_2: 500 mg/6 mL, Cleanert® PAX: 150 mg/6 mL

Experimental

Muscle tissue sample

1) Extraction: Weigh 5 g (±0.05 g) of tissue sample in a 50 mL centrifuge tube. Add 15 mL of methanol and vortex mix for 1 min. Centrifugate at 4000 r/min for 10 min and transfer the supernatant layer to another centrifuge tube. Extract again and combine the extracts. Add 20 mL of n-hexane and shake 20 times by hand. Centrifugate at 3000 r/min for 5 min and discard the n-hexane layer. Add 20 mL of n-hexane again to repeat the degreasing process. Transfer the underlayer to a 100 mL pear-shaped bottle and evaporate by rotary evaporator in water bath at 50°C almost to dryness. Add 5 mL of ethyl acetate and vortex mix for 1 min. Stand for 10 s and transfer the supernatant to the same centrifuge tube. Wash the pear shape tube once again with 10 mL of n-hexane. Combine the solutions for later use.
2) Cleanert® NH_2 cleanup: load 2 g of anhydrous sodium sulfate on the NH_2 cartridge and knock to uniformity with a glass stick. Activate the cartridge with 5 mL of ethyl acetate followed by 5 mL of n-hexane. Load the prepared sample onto the cartridge. Wash sequentially with 5 mL of n-hexane and 5 mL of n-hexane-ethyl acetate (60/40 v/v). Elute sequentially with 4 mL of n-hexane-ethyl acetate (20/80 v/v) and 4 mL of ethyl acetate. Combine the eluates and dry under a stream of nitrogen at 50°C. add 0.5 mL of acetonitrile to the residue and vortex mix for 1 min. Add 0.5 mL of water and mix. Filter the solution through 0.2 μm organic membrane for LC-MS/MS analysis.

Liver tissue sample

1) Weigh 5 g (±0.05 g) of sample in a 50 mL centrifuge tube. Add 15 mL of methanol and vortex mix for 1 min. Centrifugate at 4000 r/min for 5 min and transfer the supernatant layer to another centrifuge tube. Extract again and combine the two extracts. Add 10 mL of n-hexane and shake by hand. Centrifugate at 3000 r/min for 5 min and discard the supernatant n-hexane layer. Dry the underlayer under a stream of nitrogen at 50°C. Add 5 mL of n-hexane and vortex mix for 1 min. Add 20 mL of n-hexane and vortex mix for 30 s. Centrifugate at 4000 r/min for 5 min and take the supernatant for later use.
2) Cleanup: load 2 g of anhydrous sodium sulfate on the NH_2 cartridge and knock to uniformity with glass stick. Load the prepared sample onto the cartridge activated with 5 mL of ethyl acetate followed by 5 mL of n-hexane. Wash sequentially with 5 mL of n-hexane and 5 mL of n-hexane-ethyl acetate (45/55 v/v). Elute sequentially with 5 mL of n-hexane-ethyl acetate (20/80 v/v) and 5 mL of ethyl acetate containing 2% methanol. Combine the eluates and dry under a stream of nitrogen at 50°C.
Add 0.5 mL of acetonitrile to the residue and vortex mix for 1 min. Add 0.5 mL of water and mix. Add 2 mL of n-hexane and vortex mix for 30 s. Centrifugate at 9000 r/min for 5 min. Filter the underlayer through 0.2 μm organic membrane for LC-MS/MS analysis.

Bovine milk sample

1) Extraction: add 5.0 mL of sample to a 50 mL centrifuge tube. Add 0.1 mL of 18% H_2SO_4 solution and vortex mix to uniformity. Stand for 10 min. Add 10 mL of n-hexane and 20 mL of acetonitrile and vortex mix at 300 r/min for 10 min. Centrifugate at 4000 r/min for 10 min and discard the n-hexane layer. Transfer 12.5 mL of the extract to a centrifuge tube. Evaporate under a stream of nitrogen at 50°C to less than 0.1 mL. Add 10 mL of water and adjust with 5 mol/L sodium hydroxide to pH=11. Centrifugate at 9000 r/min for 5 min for later use.
2) Cleanert® PAX cleanup: activate and equilibrate Cleanert PAX SPE cartridge sequentially with 2 mL of methanol and 2 mL of water. Load the sample onto the cartridge. Wash sequentially with 1 mL of methanol-ammonia-water (5/5/90 v/v/v) and 0.5 mL of methanol. Elute with 4 mL of 2% ethyl acetate. Collect the eluate and dry under a stream of nitrogen at 50°C.add 0.5 mL of acetonitrile to the residue and vortex mix for 1 min. Add 0.5 mL of water and mix. Filter the solution through 0.2 μm organic membrane for LC-MS/MS analysis.

Egg sample

1) Extraction: add 5 g (±0.05 g) of sample to a 50 mL centrifuge tube. Add 10 mL of acetonitrile and vortex mix for 1 min. Centrifugate at 9000 r/min for 5 min. Transfer the supernatant to another centrifuge tube. Extract again and combine the two extracts. Transfer 12.5 mL of the extract to a centrifuge tube. Evaporate under a stream of nitrogen at 50°C to less than 0.1 mL. Add 10mL of water and adjust pH = 11.0 to with 5 mol/L sodium hydroxide. Centrifugate at 9000 r/min for 5 min for later use.
2) Cleanup: activate and equilibrate Cleanert® PAX SPE cartridge sequentially with 2 mL of methanol and 2 mL of water. Load the sample onto the cartridge. Wash sequentially with 1 mL of methanol-ammonia-water (5/5/90 v/v/v) and 0.5 mL of methanol. Elute with 4 mL of 2% ethyl acetate. Collect the eluate and dry under a stream of nitrogen at 50°C. Add 0.5 mL of acetonitrile to the residue and vortex mix for 1 min. Add 0.5 mL of water and mix. Filter the solution through 0.2 μm organic membrane for LC-MS/MS analysis.

Determination of β-estradiol Residues in Muscles of Fish and Shellfish with Deuterium Isotope by GC-MS (Cleanert® C18, P/N: S185003)

Material

Cleanert® C18, 500 mg/3 mL

Experimental

Extraction

Muscle tissues of fish or shellfish are minced by domestic blender and stored in refrigerator at -18°C for later use. Transfer 5.00 g of sample, accurately weighed, into a 50 mL centrifuge tube. Add 100 μL of internal standard working solution and 5 mL of sodium acetate buffer. Homogenize the mixture at 18000 r/min twice, by homogenizer, each time for 30 s. Add 10 mL of acetonitrile and vortex mix for 1 min. Ultrasonic extract at room temperature for 15 min. Centrifugate the mixture at 10000 r/min, 4°C, for 10 min. Transfer the supernatant to another centrifuge tube. Add 10 mL of acetonitrile to the residue and repeat the extraction steps. Combine the supernatants.

Cleanert® C18 cleanup

Add 10 mL of n-hexane to the supernatant. Cap the tube and shake tempestuously for 1~2 min. Centrifugate at 1000 r/min at 4°C for 5 min. Discard the n-hexane layer and wash the underlayer with n-hexane again. Transfer the remaining solution to a pear-shaped bottle. Add 0.5 mL of n-propanol and evaporate to dryness by rotary evaporator in water bath at 45°C. Add 1 mL of acetonitrile to the residue and wash the bottle for 1 min. Transfer the solution to a 5 mL syringe. Repeat the extraction with 1 mL of acetonitrile and combine the solution to the syringe. Filter the solution through organic membrane. Dilute the filtered solution with water to 10 mL. Load the sample solution onto the C18 SPE cartridge (activated sequentially with 6 mL of methanol, 3 mL of 0.1% acetic acid and 3 mL of water) at the flow rate of 1~2 mL/min. Wash the C18 cartridge with 3 mL of water then elute the cartridge with 9 mL of acetonitrile. Collect the eluate and dry under nitrogen stream.

Derivatization

For sample,accurately add 100 μL of MSTFA2DTE2TM IS derivatization reagent to the residue. Cap the tube and vortex mix for 1 min. Allow to react in oven at 60°C for 30 min and then cool to room temperature. Analyze the sample by GC-MS within 48 h. For standard solution, β-estradiol and internal standard working solution should be added to a tube in which the sample deactivation is conducted.
(Agilent, U.S.) then mix the solution with vortex and dry under nitrogen stream before derivatization as above.

GC/ MS conditions

DA-5MS capillary column (25 m × 0.32 mm × 0.52 μm);
column temperature: 120°C (2 min)---- 250°C (15°C/min), 300°C (5 min, 5°C/min);
Carrier gas: He (≥99. 999%);
Flow rate: 1. 0 mL /min;
Injector port temperature: 250°C;
Splitless injection volume: 1 μL;
EI source temperature: 230°C;
Quadrupole temperature: 150°C;
Interface temperature: 280°C;
Ionization voltage: 70 Ev;
Solvent detention time: 3 min;
Electron motiplier voltage: 1106 V;
Mass scan range: 40~500 amu.

2. Pesticide Residues

Analysis of Mult-pesticide Residues in Tea Leaves by GC-MS and LC-MS/MS(Cleanert® TPT, P/N: TPT200010)

Material

Cleanert® TPT cartridge 2 g/12 mL

Experimental

Extraction

Weigh 5 g (accurate to 0.01 g) of tea leaves sample in a 80 mL centrifuge tube, add 15 mL of acetonitrile. The solution is homogenized at 15000 r/min for 1 min, then centrifugated at 4200 r/min for 5 min. Transfer the supernatant into a 100 mL pear-shaped bottle. Extract the residue with 15 mL of acetonitrile and centrifugate. Combine the two supernatants and evaporate to 1 mL by rotary evaporator in water bath at 40°C for further cleanup.

Cleanert® TPT cleanup

Method for GC/MS: load 2 cm high of anhydrous sodium sulfate onto Cleanert® TPT cartridge. Wash the cartridge with 10 mL of acetonitrile/toluene (3:1, v/v), and place it on a fixed rack mounted with a pear-shaped bottle under it. Load the concentrated sample onto the Cleanert® TPT cartridge. Wash the sample bottle with 2 mL of acetonitrile/toluene (3:1, v/v) 3 times and combine the solutions onto the cartridge. Add a 50 mL liquid reservoir on the cartridge and wash the cartridge with 25 mL of acetonitrile/toluene (3:1, v/v). Collect all the eluate in a pear-shaped bottle and evaporate to 0.5 mL by rotary evaporator in water bath at 40°C. Exchange the solvent with 5 mL of n-hexane twice and obtain 1 mL of solution. Add 40 µL of internal standard solution and mix for GC-MS analysis.

Method for LC-MS/MS: Except that the sample amount is 2 g, the sample extraction and cleanup method is the same as above. Evaporate the collected eluate to 0.5 mL by rotary evaporator in water bath at 40°C. Dry under a stream of nitrogen at 35°C. Redissolve the residue in 1 mL of acetonitrile/water (3:2, v/v). Filter the solution through 0.2 µm membrane for LC-MS/MS analysis.

Analysis of Mult-pesticide Residues in Ramulus Mori, Honeysuckle and the Fruit of Chinese Wolfberry with GS-MS and LC-MS/MS Respectively (Cleanert® TPH, P/N: TPH200010)

Material

Cleanert® TPH, 2 g/12 mL

Experimental

(1) Extraction

Weigh 5 g (accurate to 0.01 g) of honeysuckle, medlar samples, or 2.5 g (accurate to 0.01 g) of lotus leaves, ramuli mori samples in 50 mL centrifuge tubes. Add 15 mL of acetonitrile. (For medlar sample, another 5 mL of water is needed). Homogenize at 15000 r/min for 1 min. Add 2 g of sodium chloride and homogenize again for 1 min. Centrifugate at 4200 r/min for 5 min and transfer the supernatant into a 150 mL pear-shaped bottle. Add 15 mL of acetonitrile to the centrifuge tube again and homogenize for 1 min. Centrifugate at 4200 r/min for 5 min and combine the supernatant to the pear-shaped bottle. Evaporate to 1~2 mL by rotary evaporator in water bath at 40°C for further cleanup.

(2) Cleanup

Load 2 cm high of anhydrous sodium sulfate onto Cleanert® TPH cartridge. Place it on a fixed. Wash the cartridge with 10 mL of hexane/acetone (4:6, v/v). When the washing solution reaches the upper surface of the sodium sulfate, load the concentrated sample (2.1) onto the cartridge. Collect the eluate in a pear-shaped bottle. Wash the pear-shaped bottle (2.1) with 2 mL of hexane/acetone(4:6, v/v) 3 times. Combine the washing solutions onto the cartridge. Add a 25 mL liquid reservoir onto the cartridge. Wash the cartridge with 25 mL of hexane/acetone to elute the pesticides and related compounds. Collect the eluate and evaporate by rotary evaporator in water bath at 40°C almost to dryness. Redissolve the residue in 1 mL of hexane. Add 40 µL of internal standard solution and mix. Filter the solution through 0.2 µm membrane for GC-MS analysis.

LC-MS/MS

(1) Extraction

Weigh 2 g (accurate to 0.01 g) of honeysuckle, medlar, (lotus leaf) and ramuli mori samples in 50 mL centrifuge tubes. Add 15 mL of acetonitrile. (For medlar sample, another 5 mL of water is needed). Homogenize at 15000 r/min for 1 min. Add 2 g of sodium chloride and homogenize again for 1 min. Centrifugate at 4200 r/min for 5 min and transfer the supernatant into a 150 mL pear-shaped bottle. Add 15 mL of acetonitrile to the centrifuge tube again and homogenize for 1 min. Centrifugate at 4200 r/min for 5 min and combine the supernatant to the pear-shaped bottle. Evaporate to 1~2 mL by rotary evaporator in water bath at 40°C for further cleanup.

(2) Cleanup

Fill a 2 cm bed height of sodium sulfate anhydrous onto the head of TPH cartridge. Place it on a fixed rack. Wash the cartridge with 10 mL of acetonitrile/toluene (3:1, v/v). When the washing solution reaches the upper surface of the sodium sulfate, load the concentrated sample (3.1) onto the cartridge. Collect the eluate in a pear-shaped bottle. Wash the pear-shaped bottle (3.1) with 2 mL of acetonitrile/toluene (3:1, v/v) 3 times. Combine the washing solutions onto the cartridge. Add a 25 mL liquid reservoir on the cartridge. Wash the cartridge with 25 mL of acetonitrile/toluene (3:1, v/v) to elute the pesticides and related compounds. Collect the eluate and evaporate to 1~2 mL by rotary evaporator in water bath at 40°C. Dry under a stream of nitrogen. Redissolve the residue in 1 mL of acetonitrile/toluene (3:1, v/v). Filter the solution through 0.2 µm membrane for LC-MS/MS analysis.

Determination of Cyromazine Residues in Vegetables with HPLC (Cleanert® SCX, P/N: SC5006)

Material

Cleanert® SCX (500 mg/6 mL)

Experimental

Sample preparation

Mince the edible part of vegetable sample. Collect the minced sample by quartation into a PE container and store it at 16 to 20 Celsius. Defreeze the sample at room temperature before use.

Sample extraction

Weigh 20.00 g sample into a beaker, and then add 50 mL mixture of 0.05 M ammonium acetate solution and acetonitrile (1:4, v/v).
Homogenize the sample mixture at 14000 rpm for 2 minutes.
Filter the sample slurry through a Buchner funnel into a 100 mL volumetric flask.
First rinse of the sample beaker with 35 mL ammonium/acetonitrile solution, and then homogenize the remaining sample at 14000 rpm for 30 seconds, and transfer the mixture into the funnel.
Add additional ammonium/acetonitrile solution into the collecting flask to bring up volume to 100 mL and mix well the sample solution.
Pipette 10 mL sample solution into a round-bottom flask, and then rotovap the sample at 40 Celsius to remove the organic solvent.
Adjust the pH of the sample concentrate to 2 with 0.1 M HCl solution, and the concentrate is ready for SPE cleanup.

SCX Procedure

- Precondition SCX cartridge by adding 5 mL methanol first followed by 5 mL de-ionized water.
- Add sample mixture from the last step to the cartridge, and discard the eluate.
- Wash the cartridge first with 5 mL methanol and then with 5 mL de-ionized water, and discard the eluate and dry the cartridge.
- Elute the cartridge three times subsequently with 5 mL 5% ammonina/methanol a time, and collect the eluate into a round-bottom flask.
- Evaporate the collected eluate to nearly dryness with a rotovap at 40 Celsius, then blow the sample to dryness under nitrogen.
- Reconstitute the sample residue with 1 mL acetonitrile/water (93:7, v/v), and filter the solution through 0.45 um membrane into a HPLC vial.

Detection

Column: Venusil® NH_2 column, 4.6 × 250 mm, 5 µm
Mobile phase: acetonitrile/water = 97:3 (v/v)
UV: 215 nm
Flow rate: 1.0 mL/min
Injection volume: 10 µL
Column temperature: 35°C
Please download the details at the website: www.bonnaagela.com

Determination of Organophosphorus, Organic Chloride and Carbamates Residues in Vegetables (Cleanert® Florisil, P/N: FS0006)

Material

Cleanert® Florisil (1 g/6 mL)

Experimental

Sample preparation

Cole plants or tomato samples are minced into small pieces followed by blending using a food processor. Store the samples in subpackages at 16°C~ -20°C.

Extraction

Weigh 25 g of sample accurately and place in homogenizer. Add 50 mL of acetonitrile and homogenize for 2 min. Filter the solution through filter paper into a 100 mL graduated cylinder with cap containing 5~7 g sodium chloride in it. Collect 40~50 mL of filtrate. Cap the graduated cylinder and shake for 1 min. Stand at room temperature for 30 min to allow acetonitrile and water phases separated.

Florisil cleanup

Transfer 10 mL of acetonitrile solution into a 150 mL beaker. Heat the beaker in water bath at 80°C and evaporate the solution under nitrogen or air stream almost to dryness. Add 2 mL of n-hexane and cover the beaker with aluminium foil for further cleanup. Pre-washand condition Florisil cartridge sequentially with 5 mL of acetone/hexane (10:90, v/v) and 5 mL of hexane. When the solvent reaches the adsorption layer of the cartridge, load the sample in beaker onto the cartridge. Collect the eluate in a 15 mL centrifuge tube. Wash the beaker with 5 mL of acetone/hexane(10:90, v/v) twice. Combine the solutions onto the cartridge and collect the eluates into the centrifuge tube. Evaporate under nitrogen stream in water bath at 50°C to less than 5 mL. Dilute to 5 mL with hexane again. Vortex mix and transfer the solution to two 2 mL sample vials for GC analysis. (column: DA-50+, 30 m × 0.53 mm × 1.0 µm, P/N: 5053-3010, DA-1, 30 m × 0.53 mm × 1.5 µm, P/N: 0153-3015).

Results

Recoveries of tomato and cole samples, with low and high levels of pigments respectively, are studied (spiked concentrations: 0.1 mg/kg and 0.2 mg/kg). For each concentration level, experiments are repeated three times. The results are summarized in Table 1.

Table 1 Recoveries of tomato and cole samples

Pesticides	Spiked concentration 0.1 mg/kg		Spiked concentration 0.2 mg/kg		Average
	Tomato	Cole	Tomato	Cole	
Chlorothalonil	72.6	68.8	70.0	70.9	70.6
Ketotriazole	88.6	84.7	87.8	82.7	88.4
Cyfluthrin	91.0	97.6	97.1	98.0	93.4
Cypermethrin	81.8	77.7	82.0	83.0	81.1
Fenvalerate	77.0	72.0	78.0	79.8	76.7
Fenpropathrin	77.7	77.1	81.2	79.0	78.5

Determination of 466 Pesticide Residues in Vegetables and Fruits (Cleanert PestiCarb/NH_2, Cleanert C18, P/N: PN0006, S18200012)

Material

Cleanert C18, 2 g/12 mL; Cleanert PestiCarb/NH_2, 500 mg/500 mg/6 mL

Experimental

Extraction

Weigh 20 g (accurate to 0.01 g) of sample in a 80 mL centrifuge tube. Add 40 mL of acetonitrile and homogenize at 15000 r/min for 1 min. Add 5 g of sodium chloride and homogenize for 1 min again. Centrifugate at 3000 r/min for 5 min and take 20 mL (corresponding to 10 g of sample) of the supernatant for further cleanup.

Cleanert C18 and PestiCarb/NH_2 cleanup

(1) Activate Cleanert C18 cartridge with 10 mL of acetonitrile. Load the sample (2) onto the cartridge and elute with 15 mL of acetonitrile. Collect the solutions in a pear-shaped bottle and evaporate to 1 mL by rotary evaporator in water bath at 40°C for later cleanup.

(2) First fill PestiCarb/NH_2 cartridge with sodium sulfate anhydrous to a 2 cm bed height; and use a pear-shaped bottle under the cartridge for collection. Wash the cartridge with 4 mL of acetonitrile/toluene (3:1, v/v). When the washing solution reaches the upper surface of the sodium sulfate, load the concentrated sample (3.1) onto the cartridge. Wash the pear-shaped bottle (3.1) with 2 mL of acetonitrile/toluene (3:1, v/v) 3 times. Combine the washing solutions onto the cartridge. Wash the cartridge with 25 mL (divided into several times) of acetonitrile/toluene (3:1, v/v). Collect the eluate in pear-shaped bottle and evaporate to 0.5 mL by rotary evaporator in water bath at 40°C. Add 5 mL of n-hexane to exchange the solvent and evaporate by rotary evaporator in water bath at 40°C. Repeat the solvent exchange process and obtain 1 mL of sample. Add 40 µL of internal standard solution and mix for GC-MS analysis. (Column: DA1701, 30 m × 0.25 mm × 0.25 µm, P/N: 6125-3002)

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3. Detection of Food Additives

Detecting Phthalates in Different Food Matrices

Introduction

Phthalic acid esters or phthalates are commonly used as plasticizers. Although phthalates are being phased out in many developed countries over health concern, occurrence of such substances recently in food products in other parts of the world has drawn considerable publicity over the same health issue. SPE products from Bonna-Agela provide effective tools to rapidly process food samples for detection and analysis of such substances. Detail procedure and methodology are illustrated here for such applications with plenty of real-life samples.

Experimental

Soft drink

Sample	Bottled water, mineral water, tea, juice and functional drink
Condition	5 mL of methanol, 5 mL of water
Sample Loading	10 mL liquid sample
Interference Elution	5 mL water contained 5% methanol, vacuum for 20 min
Analyte Elution	5 mL of methanol.
Eluent Treatment	Evaporate the eluent to dryness with N_2 and bring to a volume of 1mL with methanol
Analytical Method	HPLC
Note	This method is valid when the concentration of phthalates is too low for direct HPLC analysis. For GC/
	MS analysis, n-hexane liquid/liquid extraction is more convenient.
Products	
SPE Cartridge	Cleanert® DEHP; 500 mg/6 mL; Cat. No: DEHP5006-G, 30/pk
HPLC Column	Venusil® XBP C18-L; 4.6 × 250 mm, 5 μm, 150 Å; Cat. No: VX952505-L

Low-fat liquid food

Sample	Milk, jam or syrup
Pre-treatment	Add 2 mL of sample into the glass tube, then add 4 mL of acetonitrile/methyl tert-butyl ether (v/v = 9:1), then vortex for 2 min
Extraction	Add the Cleanert® MAS-PAE sorbents, vortex for 2 min, centrifuge for 4 min at 4000 rpm
	Filter the supernatant with special syringe filters
Analytical Method	GC/MS
Products	
SPE Cartridge	Cleanert® MAS-PAE; Cat. No: MS-PAE40, 15/pk
Syringe Filters	PAE Syringe filters; Cat.No: PAE041345-T; 100/pk
GC Column	DA-5MS; 30 m × 0.25 mm × 0.25 μm; Cat. No: 1525-3002, 1/pk
Vials	40 mL 95 × 27.5 mm; AV7200-7; 100/pk

Low-fat solid food

Sample	Milk powder, biscuits, cakes, jelly or candy
Pre-treatment	Take 1 g of powder samples and 2 mL of water into a centrifuge tube, vortex the tube for 2 min
Extraction	Add Cleanert® MAS-PAE sorbents, vortex the tube for 2 min, centrifuge for 5 min at 4000 rpm, filter the supernatant with special syringe filters
Analytical Method	GC/MS
Products	
SPE Cartridge	Cleanert® MAS-PAE; Cat. No: MS-PAE40; 15/pk
Syringe Filters	Clarinert™ PAE Syringe filters; Cat.No: PAE041345-T; 100/pk
GC Column	DA-5MS; 30 m × 0.25 mm × 0.25 μm; Cat. No: 1525-3002; 1/pk

High-fat food

Sample	Animal fat
Pre-treatment	Dissolve 0.2 g of sample with 1 mL of n-hexane
Condition	5 mL of n-hexane
Interference Elution	7 mL of n-hexane
Analyte Elution	Ethyl acetate: hexane (50:50, v/v) (3 mL × 2)
Eluent Treatment	Blow to nearly dryness with nitrogen at 40°C add 1 mL of acetonitrile for stripping, vortex for 3 min, ultrasound for 3 min, centrifugal for 5 min at 4000 rpm, and take the supernatant.
Analytical Method	GC/MS
Products	
SPE Cartridge	Cleanert® PAE, Cat. No:PAE30006-G, 30/pk
GC Column	DA-5MS, 30 m × 0.25 mm × 0.25 μm, Cat. No: 1525-3002, 1/pk

Sample	Animal fat
Pre-treatment	Dissolve 0.2 g of sample with 1 mL of n-hexane
Condition	5 mL of n-hexane
Interference Elution	7 mL of n-hexane
Analyte Elution	Ethyl acetate: hexane (50:50, v/v) (3 mL × 2)
Eluent Treatment	Blow to nearly dryness with nitrogen at 40°C add 1 mL of acetonitrile for stripping, vortex for 3 min, ultrasound for 3 min, centrifugal for 5 min at 4000 rpm, and take the supernatant.
Analytical Method	GC/MS
Products	
SPE Cartridge	Cleanert® PAE, Cat. No:PAE30006-G, 30/pk
GC Column	DA-5MS, 30 m × 0.25 mm × 0.25 μm, Cat. No: 1525-3002, 1/pk

Sample	Vegetable oil, cheese or other animal tissues
Pre-treatment	Add 0.5 g of sample in a sealed glass tube
Extraction	Extract with n-hexane under ultrasonic, centrifuge for 5 min, take the supernatant, if the sample contains water, anhydrous sodium sulfate should be added
Condition	5 mL of n-hexane
Interference Elution	3 mL of n-hexane
Analyte elution	3 mL × 2 of ethyl acetate: hexane (50:50, v/v).
Eluent Treatment	Blow to nearly dryness with N_2, add 1 mL of acetonitrile for stripping, vortex for 3 min, ultrasound for 3 min, centrifugal for 5 min at 4000 rpm, take the supernatant
Analytical Method	GC/MS
Products	
SPE Cartridge	Cleanert® PAE, Cat. No:PAE30006-G, 30/pk
GC Column	DA-5MS, 30 m × 0.25 mm × 0.25 μm, Cat. No: 1525-3002, 1/pk

Complex Samples

Sample	Instant noodle sauces (rich in oil)
Extraction	Take 0.5 g of sample, add 5 mL of n-hexane, vortex for 3 min, add 500 mg of anhydrous sodium sulfate, vortex for 3 min, centrifuge for 5 min at 4000 rpm, take the supernatant
Condition	5 mL of n-hexane
Interference Elution	3 mL × 2 of n-hexane
Analyte Elution	Blow to nearly dryness, add 1 mL acetonitrile for stripping, vortex for 3 min, ultrasound for 3 min, centrifuge for 5 min at 4000 rpm, take the supernatant
Analytical Method	GC/MS
Products	
SPE Cartridge	Cleanert® PAE; Cat.No: PAE30006-C; 30/pk
GC Column	DA-5MS, 30 m × 0.25 mm × 0.25 μm, Cat. No: 1525-3002, 1/pk

Sample	Soy sauce
Pre-treatment	Add 2 mL of sample into a Cleanert MAS-PAEc cartridge
Extraction	Add 4 mL of acetonitrile: toluene = 9:1 (v/v), vortex for 2 min, add Cleanert MAS-PAEc sorbents, vortex the cartridge for 2 min, centrifugal for 5 min at 4000 rpm, filter the supernatant with syringe filter
Analytical Method	GC/MS
Products	
SPE Cartridge	Cleanert® MAS-PAEc; Cat.No: MS-PAE40-C; 15/pk
GC Column	DA-5MS, 30 m × 0.25 mm × 0.25 μm, Cat. No: 1525-3002, 1/pk

Appendix 1 The detection of 15 pthalate esters with HPLC

Column	Venusil® XBP C18-L, 4.6 × 250 mm, 5 μm, 150 Å (Cat. No: VX952505-L)
Mobile Phase	A: water; B: methanol: acetonitrile = 50:50
Flow Rate	1.0 mL/min
Wavelength	242 nm
Loading	50 μL
Sample	15 pthalate esters
Concentration	10 ppm
Solvent	40% mobile phase A
ColumnTemperature	30 °C

Elution gradient:

Time / min	A / %	B / %
0	60	40
2	50	50
10	40	60
12	30	70
20	30	70
31	0	100
40	0	100
40.01	60	40

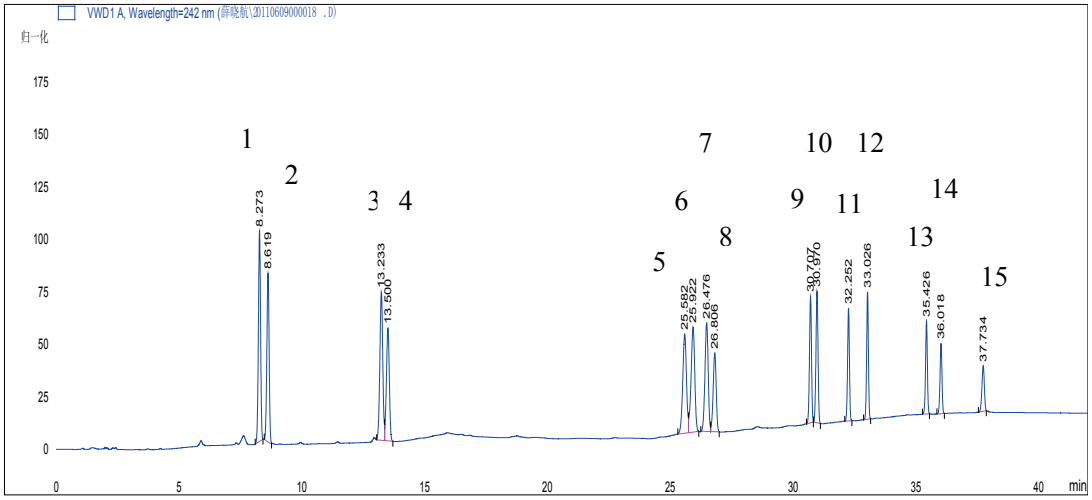


Fig 1. The chromatogram for pthalate esters standard (sample concentration: 10 ppm)
(1-DMP, 2-DEP, 3-DBP, 4-DEHP, 5-BBP, 6-DEHP, 7-DMEP, 8-DBEP, 9-DPP, 10-BMPP, 11-DEEP, 12-DCHP, 13-DIBP, 14-DNP, 15-DINP)

15 Pthalates were separated as shown in Figure 1 with good resolution.
Detection limit: 0.5 ppm
Linear relationship: Y = 26.8X-26.6, R2 = 0.941

Appendix 2 The detection of 15 pthalate esters with GC/MS

Instrument	Agilent 7890/5975 GC/MS
GC Column	DA-5MS 30 m × 0.25 mm × 0.25 μm (1525-3002)
Injection	250 °C, splitless injection
Temperature Program	50 °C (1 min) 20 °C/min 220 °C (1 min) 5 °C/min 280 °C (4 min)
Loading Volume	1 μL
Flow Rate	1 mL/min
Inlet Temperature	280
Ionization Mode	EI
Ionization Energy	70eV
Solvent Delay	7 min
Monitoring Methods	SIM mode, ion monitored as below

No	Retention Time (min)	Name	SIM
1	8.265	DMP	163, 77
2	9.135	DEP	149, 177
3	10.888	DIBP	149, 223
4	11.637	DBP	149, 223
5	11.979	DMEP	59, 149, 193
6	12.72	BMPP	149, 251
7	13.044	DEEP	45, 72
8	13.41	DPP	149, 237
9	15.552	DHXP	104, 149, 76
10	15.694	BBP	149, 91
11	17.153	DBEP	149, 223
12	17.81	DCHP	149, 167
13	18.056	DEHP	149, 167
14	20.444	DNOP	149, 279
15	22.98	DNP	57, 149, 71

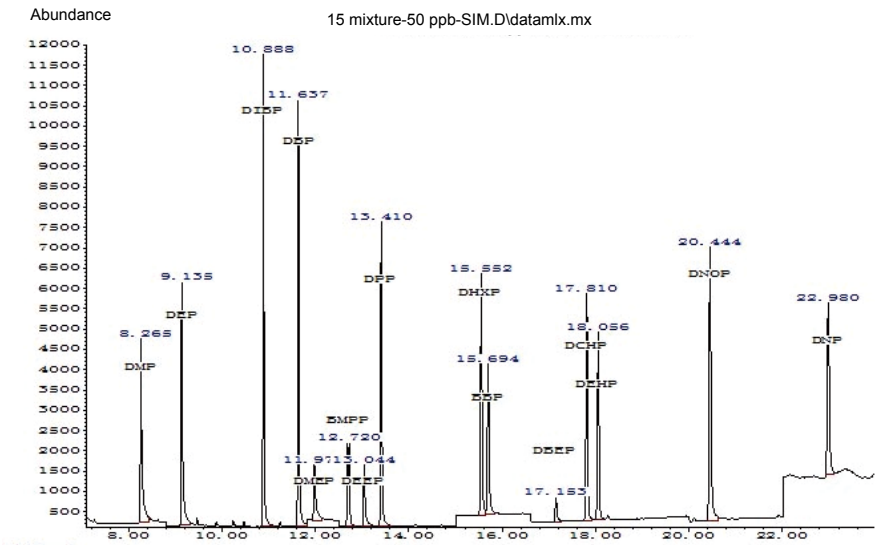


Figure 2. Detection of 15 pthalates using GC-MS method

Figure 2 shows the separation profile of 15 pthalates using GC-MS. Each component is well resolved, and the detection limit reaches ppb level.

Appendix 3 The recoveries of 15 phthalate esters in vegetable oil

Validation of SPE procedures for 15 phthalates in vegetable oil shown with method recovery data when samples were spiked at 500 ug/L.

No	Retention time/min	Sample	Recovery
1	8.308	DMP	149.97%
2	9.185	DEP	93.49%
3	10.96	DIBP	125.70%
4	11.716	DBP	136.89%
5	12.064	DMEP	90.84%
6	12.778	BMPP	82.29%
7	13.144	DEEP	106.38%
8	13.518	DPP	88.14%
9	15.686	DHXP	75.32%
10	15.844	BBP	89.56%
11	17.295	DBEP	105.05%
12	17.967	DCHP	72.94%
13	18.206	DEHP	124.27%
14	20.625	DNOP	78.19%
15	23.297	DNP	75.27%

Detecting Acrylamide in Potato Chips with HPLC (Cleanert® ACA, P/N: ACA50012)

Overview

On April, 2002, researchers from Swedish National FDA and University of Stockholm first reported that acrylamide have been detected on many oil-fried and baked starchy foods, such as French fries, potato chips and so on, and the contents of acrylamide exceeded more than 500 times of the maximum permitted content of acrylamide in drinking water. The similar results have been reported by researchers from Norway, United Kingdom, Swiss, and USA etc. Although there is no sufficient evidence that acrylamide intake through food has a significant relationship with the occurrence of any type of human tumors, the investigation results on professionals who contact acrylamide frequently and on people who contact acrylamide occasionally show that acrylamide has neurotoxicity. This article describes a method for detecting acrylamide in potato chips.

Material

SPE cartridge

Cleanert ACA 500mg/12 mL (P/N: ACA50012);

Reagents and Standards

n-Hexane (HPLC grade, 4 L/bottle, P/N: AH216), Watsons Water, Acrylamide (1000 ppm, MeOH solution)..

Instrumentals and Consumables

Agilent 1200, N2 blow-dry machine, Ultrasound instrument, high-speed centrifuge, PTFE syringe filter 0.22 μm (P/N: AS041320-T), 50 mL centrifuge tube (P/N: LXG0050), 12 Position Vacuum manifold SPE (P/N: VM12).

Experimental

Sample Preparation

Weigh 2 grams potato chips, ground to very fine powder, put into a 50mL centrifuge tube. Add 30 mL ultra-pure water (Watsons Water), vortex oscillated and Ultrasonic vibrated for 10min, then centrifuged at 6000 rpm. Collect 15 mL supernatant solution, put into a new 50mL centrifuge tube, add 5 mL n-hexane into the tube, and vortex oscillated for a minute, then centrifuged at 6000 rpm for 10 min. Disposal of the upper layer of n-hexane, and repeat the above n-hexane extraction steps one more time. The resultant 15 mL sample solution is ready for next step.

SPE Method

Conditioning: 5 mL MeOH and 5 mL H_2O (Vacuum filtration)
Sample Loading: 15 mL sample solution from step 3.1(add internal standard before ultrasound extraction)
Washing: 5 mL Watsons Water
Vacuum filtration 30min, dried the column
Elution: 3 mL MeOH (Vacuum filtration, collect)
At 40°C, N2 blow dry to 100-200 μL, then add Watsons Water to dilute to 2 mL, filtered by 0.22 μm PTFE syringe filter, then sample is ready for test.

HPLC Method

HPLC Column: Unisol C18, 4.6 × 250 mm, 5.0 μm, 100 Å (P/N: VA952505-0);
Mobile phase: Watsons Water;
Flow rate: 1.0 mL/min;
Injection sample: 20 μL;
UV Wavelength: 210 nm;
Column temperature: R. T.

Method Validation

Calibration Curve

Dilute the standard samples to the following concentration: 1, 2, 5, 10, 20, 50 μg/mL. A calibration curve is generated according to the peak areas at different concentrations.

Sample Analysis

Analyze the samples from step 3.1, and quantify with external standards.

Formulation

Acrylamide contents calculated by the following formula: $w = \frac{c \times V}{m}$
Among them:
w: content of acrylamide in sample. Unit is μg/g.
c: concentration of acrylamide in pre-treatments solution calculated from calibration curve. Unit is μg/mL.
V: the final diluted volume of acrylamide extracted from potato chips. Unit is mL.
m: weighed potato chips mass. Unit is gram.

Table1. Recovery of Acrylamide (R=5)

	10 μg/mL	5 μg/mL	1 μg/mL
	Recovery	Recovery	Recovery
Parallel experiment 1	85.42 %	87.30 %	86.54 %
Parallel experiment 2	88.52 %	86.14 %	87.21 %

Ordering information

Product	Specification	Cat.No
Cleanert® ACA	500 mg/12 mL	ACA50012
Unisol C18	4.6 mm × 250 mm,5.0 μm,100 Å	VA952505-0
SPE manifold	12-position	VM12
Clarinert Syringe filters	PTFE 0.22 μm	AS041320-T
Centrifuge tube	—	LXG0050
n-Hexane	HPLC grade, 4 L/bottle	AH216

Determination of 3-chloro-1,2-propanediol in soy sauce samples with supported liquid extraction and GC-MS(Cleanert MCPD P/N: MCPD250012)

Introduction

Chloropropanol is a kind of compound which chlorine atom replace hydroxyl on the glycerin, including 4 kinds of compounds which are 3-chloro-1,2-propanedio l(3-MCPD), 2-chloro-1,3-propanediol (2- MCPD), 1,3-dichloro-propanol (1,3-DCP) and 2,3-dichloro-1-propanol (2, 3-DCP). These compounds poison people's liver, kidney, nervous system blood circulation system, make people suffer from neuropathy heart attack even cancer. 3-MCPD is the typical substance of chloropropanol which contaminate food. The Joint Committee of Experts of Food Additives and Contaminant confirm the Tolerable Daily Intake (TDI) as 2 μg/kg provisionally [2]; European Union make maximum intake limits as 20 μg/kg; America, Japan and other countries make the limit standard of chloropropanol in soy sauce. Contaminants of chloropropanol are often occur in many kinds of food, such as soy sauce, soup bases, biscuit, sausage and so on. It is evident in soy sauce and hydrolyzed vegetable protein. The level of soy sauce is mainly based on content of amino acid N in the soy sauce industry standard of China. Hydrolyzed vegetable protein (HVP) has been added into products without authorization to increase the delicate flavors of the soy sauce, shortened the period of fermentation, boost profits by some manufacturers, which cause severe contamination in soy sauce. The leading unqualified causes for soy sauce is over-standard of chloropropanol in the Food hygiene inspection organizing by Ministry of Public Health in 2006.

This paper use the Chinese national standard as reference. It is modified property of infusorial earth which for sample preparation, improved the process of SLE and developed Cleanert MCPD for 3-chloro-1,2-propanediol successfully. The result show that the method satisfied requirement of the experiment which exhibit more simple and efficient. It is a significant improvement for the mass soy sauce sample analysis

Material

Instrument: Agilent GC-MS 7890-5975c; Vortex mixer; Ultrasonic machine; Organomation; Thermostat; Balance (sense of 0.0001g);
Material: Standard of 3 -MCPD; Ethyl Acetate, Acetone and Hexane are HPLC grade; N- (Heptafluoro-n-butyryl)- imidazole (HFBI); Anhydrous Sodium Sulfate; Ultrapure Water; Sodium Chloride; Cleanert MCPD, 2.5 g/12 mL;

Experimental

Standard Solution

Weigh 0.1 g (accurate to 0.1 mg) 3-MCPD and make up to 100 mL in a volumetric flask with ethyl acetate to give a solution of approximately 1 mg/mL. Diluted the stock solution with Acetone to the required concentration as 1 ng/mL,10 ng/mL, 50 ng/mL, 100 ng/mL, 500 ng/mL and 1μg/mL.

Saturated Sodium Chloride Solution

Dissolve 290 g of sodium chloride in water to make 1000 mL, ultrasound for 20 min.

GC operating conditions

Column: DA-5MS(30 m*0.25 mm*0.25 μm); Injection port temperature: 230 ℃ , Splitless; Injection volume: 1 μL; Carrier gas: helium gas (purity ≥ 99.999 %) at flow rate of 1 mL/min; Interface temperature: 250 ℃ ; Temperature program: 50℃ (1 min) 2 ℃ /min 82 ℃ , post run 260 ℃ for 3 min.

MS operating conditions

Ionization mode: EI; Ionization voltage: 70 eV; Ion source: 230 ℃ ; Quadrupole: 150 ℃ ; Solvent delay: 7 min; Determination mode: SIM, selected ions (m/z): 253, 275, 289, 291, 453.

Sample Extraction

Weigh 2.5 g of soy sauce into Cleanert MCPD, elute the cartridge by 15mL ethyl acetate after 10 min standing, collect the elute in a 20mL vial and concentrate to nearest dryness (not dryness) by nitrogen-blow concentrator at 35 ℃ , add 2mL hexane, shake up.

Derivatization

Derivatization of test sample

Add 50 μL HFBI into hexane solution, tighten the stopper, mix thoroughly on a vortex mixer for 20 s. Keep for 30 min at 70℃ and let it cool to room temperature. Add 2 mL of saturated sodium chloride solution, tighten the stopper, mix thoroughly on a vortex mixer for 1 min, standing for 2 min, transfer organic phase to a clean sample vial. Repeat the step of “Add 2 mL of saturated sodium chloride solution…” again. Transfer the organic phase to a 2 mL sample vial, add a small amount of anhydrous sodium sulfate to remove residual water, and then use for GC/MS analysis.

Derivatization of standard working solutions

Pipet 2 mL of each of the standard working solutions into 6 individual 10 mL sample vials. Proceed the procedure as in 2.2.5.1 from the step of adding 50 μL HFBI…, and then analyze by GC/MS.

Result

Liner Range and LOD

Adopt external standard method for quantitative. Inject 1μL of derivatized standard working solution into GC/MS and measure the 3-MCPD derivative peak area under the condition set in 2.2.3. Plot peak area versus the concentration of each standard to construct the calibration curve and obtain regression equation as y=4.8712x-2.7648 (r^2=0.9993), the liner range is 1~1000 ng/mL. The detection limit of 3-MCPD is 0.3 μg/kg according to the principle of S/N=3.

Chromatogram of standard solution

Chromatogram of 3-MCPD standard solution show as figure 1 which operated under the chromatographic conditions set in 2.2.3.

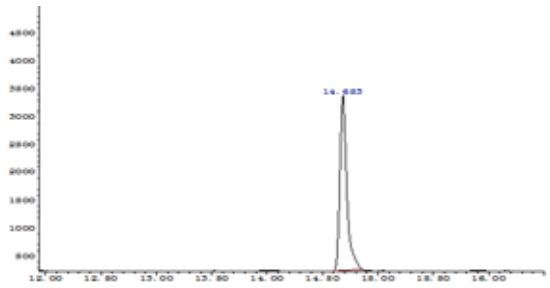


Figure 1 Chromatogram of standard solution(concentration is 50 ng/mL)

Chromatogram of spike sample

Weigh 6 samples of soy sauce accurately, spike 0.1mL of standard solution with concentration of 1μg/mL into 5 samples individual. Proceed the procedure as 2.2.4~2.2.5, and then analyze by GC/MS under the conditions set in 2.2.3. Chromatograms of samples show as figure2 and figure 3.

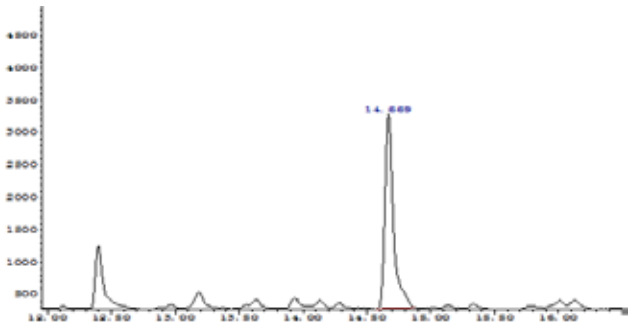


Figure 2 Chromatogram of spiked soy sauce sample (concentration is 50 ng/mL)

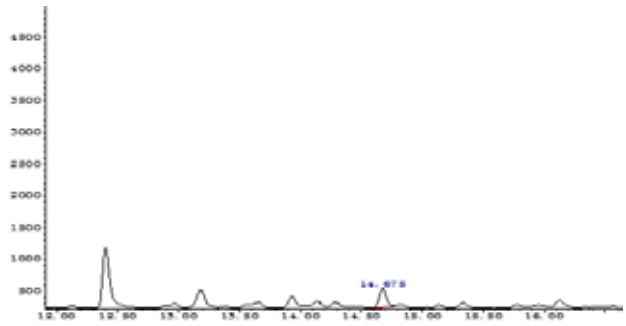


Figure 3 Chromatogram of soy sauce sample

Recovery and Precision

Proceed 3 levels of concentration as 5 ng/mL, 50 ng/mL, 200 ng/mL, respectively. Parallel processed 5 samples of each spike concentration. Result of spike recovery and precision is shown in table1.

Table 1 Recovery and precision

Concentration/ ng/mL	NO.1 (%)	NO.2 (%)	NO.3 (%)	NO.4 (%)	NO.5 (%)	Recovery (%)	RSD (%, n=5)
5	88	83.9	90.5	83.6	92.1	87.6	4.36 %
50	96.3	90.4	90.1	95.5	91.1	92.7	3.21 %
200	98.9	91.2	95.4	92.7	96.2	94.9	3.18 %

The results show that Cleanert MCPD is suitable for purifying and extracting 3-MCPD from soy sauce samples. The spiked recovery and RSD are able to meet requirement of trace analysis. This scheme presents more easy-to-handle by comparing with <GB/T 18782-2002> and < GB/T 5009.191-2006>. The amount of reagent is only one-twentieth of GB/T method which benefits environment and operator's healthy. Time costing in this scheme is shorter than GB/T method. All the characteristics prove that the method is more suitable for the determination of 3-MCPD in a large number of real soy sauce samples.

Detection of Benzo(a)pyrene in edible oils by HPLC (Cleanert Bap P/N: bap2260)

Material

Cleanert Bap, 22 g/ 60 m

Sample Preparation

(Cleanert Bap, P/N: Bap2260)
Weigh 0.3 g oil sample, dissolved by 5mL n-hexane and mixed well on vortex by oscillating.

Experimental

SPE Method

Activation: Add about 30 mL n-hexane to activate Cleanert® Bap catridge. (Note: during n-hexane drop-out process, keep adding n-hexane to the upper column to avoid n-hexane liquid level lower than the upper sieve. In this way air would be avoided into the column!)
Sample loading: Add the dissolved oil sample to the pre-activated Cleaner® Bap catridge.. Notice to avoid the upper sieve dried during the operation.
Elution: Add 80 mL n-hexane to the column, and use a 150 mL rotary evaporation bottle to collect the elution, until 80 mL n-hexane drop out completely by gravity. In order to ensure recovery, one can also increase the elution volume of n-hexane up to 120 mL.

Concentration

Rotary evaporation: put the eluted solution into a 45 ℃ water bath, rotary evaporated to dryness; if oil droplets are still observed in the remained solution, indicating a higher fat content in the sample and purification is incomplete. Need to add 80 mL n-hexane to the oil droplets to obtain a new sample, and take a new Cleanert® Bap catridge, then repeat the above purification process.

Nitrogen blow: Use add-up to 10 mL n-hexane to leach the rotary evaporation bottle three times. Combine eluents to a nitrogen blowpipe into, and blow dried by nitrogen. Add 300μL n-hexane to nitrogen blowpipe; mix well in the vortex oscillator. Notice to avoid blowing nitrogen gas flow too large during the process, which might result in spillage; avoid hexane evaporated during vortex process. Transfer the above 300μL hexane to the insert of 1.5mL vial, and inject to LC-MS for analysis.

HPLC Method

HPLC column: Venusil® PAH 250mm × 4.6 mm, 5.0 μm (P/N: VP952505-L).
Flow rate: 1.0 mL/min; Mobil phase: ACN: H2O = 95:5
Fluorescence detector: Emission 406 nm, Excitation 384 nm

Results

This method uses Cleanert® Bap SPE cartridge for the purification treatment of benzo (a) pyrene in a vegetable oil. When benzo (a) pyrene is as low as 5 μg/kg at sample, 99.49 % recovery is achieved. The chromatogram of benzo (a) pyrene analyzed by using Venusil® PAH column is shown as follows:

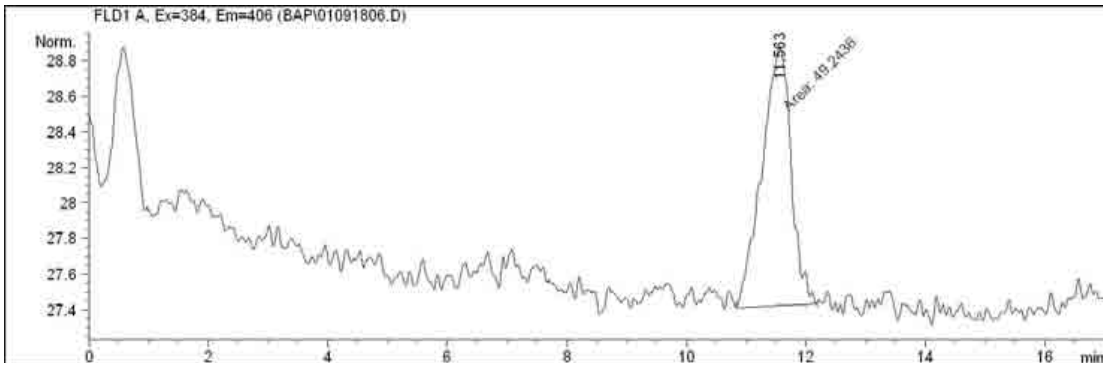


Figure 1 Chromatogram of 10 μg/L benzo (a) pyrene standard analyzed by Venusil® PAH HPLC column.

Ordering information

Product name	Packing	Cat. Number
LC-10F HPLC system	10ml/min, gradient, 200-800 nm double wavelength detector	FL-LC010GS
Cleanert® Bap SPE cartridge	22 g/60mL, 10 /pk	Bap2260
Venusil PAH HPLC column	4.6 m × 250 mm, 5 μm	VP952505-L
PTFE micro-pore syringe filter	0.22 μm, diameter 13 mm,100 /pk	AS041320
Disposable sterilized syringe	5 mL, 100 /pk	ZSQ-5ML
1.5 mL vial	100/ pk	1109-0519
Cap for 1.5 mL vial	100 /pk	0915-1819
MeOH	4×4 L/case	AH230-4
CH3CN	4×4 L/case	AH015-4

Determination of Melamine in Eggs (Cleanert® PCX, P/N: CX0603)

Material

(1) Instruments and reagents
Chromatographic column (Venusil® ASB C8, 4.6 × 250 mm, 5 μm), SPE(mixed cation exchange) (Cleanert® PCX, 60 mg/3 mL), SPE manifolds of 12 port configuration, HPLC, high speed centrifuge, ultrasonic oscillators, vortex mixer, analytical balance(one out of ten thousand) , solvent filtrator with organic and aqueous filtering membrane of 0.45 μm and vaccum pump, acetonitrile (HPLC level), standard substance of melamine (≥99.0%), space citric acid (analytical reagent), heptane sulfonic acid sodium salt (HPLC grade), water (over second distilled water).
(2) HPLC condition
Column: Venusil® ASB C8, 4.6 × 250 mm, 5 μm.
Mobile phase: acetonitrile: 10 mM/L citric acid + 10 mM/L heptane sulfonic acid sodium salt buffer solution = 7:93 (pH = 3.0).
Detection: 240 nm; flow rate: 1 mL/min; Injection: 20 μL.

Experimental

Sample preparation

Prepare freshly whipped egg sample by mechanical blending for 3 minutes. Weigh 1.0 g of the sample each time into a centrifuge tube for experiment. Spike samples with 10, 20 and 100 μL of 100 mg/L melamine stock solution, respectively, to obtain three fortified samples at concentrations of 1, 2, and 10 mg/Kg. To each sample, add 10 mL 1% trichloroacetic acid and 2 mL 5% lead acetate solutions, respectively. Mix the sample thoroughly and sonicate it for 20 minutes, and then centrifuge the sample at 8000 rpm for 10 minutes.

Blank sample is prepared with the same procedure as illustrated above without fortification.

Cleanert® PCX (60 mg/3 mL) cleanup

- Activate the SPE cartridge by adding 3 mL methanol followed by 3 mL water, then discard the eluate.
- Transfer supernate of the sample into the cartridge.
- Wash the loaded cartridge first with 3 mL water, and then with 3 mL methanol; dry the cartridge and discard the eluate.
- Add 5 mL 5% ammonium methanol (v/v) to elute the cartridge and collect the eluate.
- Evaporate the collected eluate under nitrogen at 50 Celsius.
- Reconstitute the sample with 1 mL mobile phase and filter through 0.45 μm membrane for HPLC analysis.

Results

Figures 1 and 2 show the HPLC profiles of negative blank and spiked samples, respectively. The PCX cleanup procedure removes interference effectively and allows clear detection of melamine. The HPLC method was confirmed by 6 replicate injections of standard melamine solution at 1 and 5 mg/L, respectively. The results are summarized in Table 1, showing the consistence of the method. Calibration curve was established as shown in Table 2 and Figure 3. Recovery data of the spiked samples are shown in Table 3.

Table1 stable data of retention time and peak area ratio

Density (mg/mL)	Indicator	1#	2#	3#	4#	5#	6#	Mean value	RSD%
1.0	Retention time (min)	18.830	18.829	18.829	18.838	18.840	18.834	18.833	0.026
	Peak area ratio	89	81	84	88	84	80	84	4.286
5.0	Retention time (min)	18.949	18.952	18.947	18.949	18.950	18.946	18.949	0.011
	Peak area ratio	423	440	438	439	437	438	436	1.461

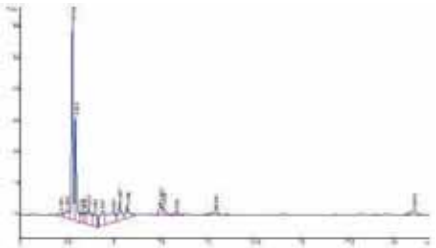


Figure 1, blank sample

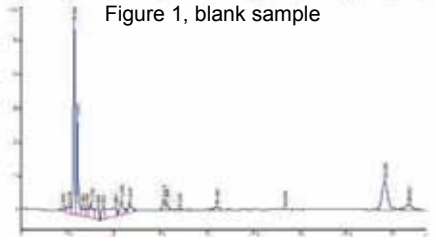


Figure 2 Profile of sample spiked with 10 ppm melamine.

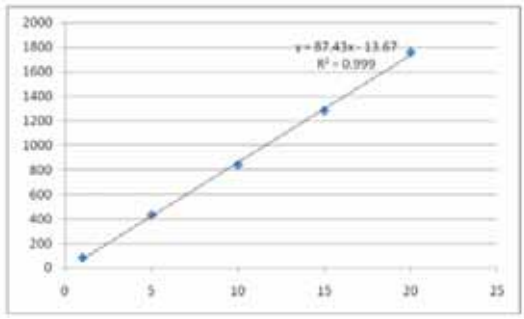


Figure 3 Regression curve of density and peak area ratio

Table 2 Experimental data of standard adjusted curve

Levels (mg/Kg)	Replicate 1	Replicate 2	Mean
1.0	89	79	84
5.0	423	440	431
10.0	832	844	838
15.0	1265	1299	1282
20.0	1689	1823	1756

Table 3 shows that the recovery ratio of melamine in the eggs is relatively good with the method

Concertration (mg/kg)	Peak area	Content	Recovery (%)
1.0	19.9	1.158892	115.89
1.0	21.0	1.214532	121.45
2.0	41.7	2.261587	113.08
2.0	40.8	2.216062	110.80
10.0	188.8	9.702247	97.02
10.0	219.6	11.26018	112.60

Melamine detection in the eggs with the above method has the following advantages:
Excellent matrix purity performance;
Less impurity interference;
Good peak symmetry;
Easy operation and high accuracy.

Determination of Sudan Red in Foods with HPLC (Cleanert® Alumina-N, P/N: AL5006-N)

Material

Cleanert® Alumina-N (500 mg/6 mL)

Experimental

Red chili and other powder samples
Put 1.00 to 5.00 g sample in a flask and then add 15 mL n-hexane. Extract the sample under sonication for 5 minutes followed by filtration. Use additional 10 mL hexane by batch to rinse the sample residue several times to colorless. Rotovap the combined supernatant to obtain sample concentrate in less than 5 mL.
Cleanert® Alumina-N Cleanup: Slowly transfer the concentrate to Alumina-N cartridge to reduce broadening of the pigment band. Wash the loaded cartridge with 10-30 mL hexane until the eluate is colorless, and discard the eluate. Elute the cartridge with 60 mL n-hexane/acetone (95:5, v/v) and collect the eluate. Evaporate the collected eluate in a rotovap. Transfer the final concentrate to a vial and bring up to 5 mL using acetone. Filter the solution through 0.45 um membrane before analysis.
Red chili oil, hot pot ingredients, cream and other oil samples
Directly dissolve 0.50 to 2.00 g oil sample with ca. 10 mL n-hexane in a beaker. Follow the 'Cleanert® Alumina-N Cleanup' procedure illustrated above to prepare sample.
Chili sauce, tomato sauce and other sample with high content of water
Weigh 10.00 to 20.00 g sample into a centrifuge tube and add 10 to 20 mL water and mix well the mixture, and then add 30 mL n-hexane/acetone (3:1, v/v). Homogenize the mixture for 5 minutes followed by centrifuging at 3000 rpm for 10 minutes. Transfer the n-hexane layer. Extract the sample paste again using 2 x 20 mL n-hexane. Dry the combined extract over 5 g sodium sulfate in a funnel and collect the solution. Evaporate the solution to dryness under rotovap. Reconstitute the residue in 5 mL hexane. Follow the procedure 'Cleanert® Alumina-N Cleanup' illustrated above to cleanup the sample for analysis.

Sausage and other meat products

Weigh 10~20 g (accurate to 0.01 g) of smashed sample in a triangular flask. Add 60 mL of n-hexane and homogenize for 5 minutes. Filter and obtain clear filtrate. Add 20 mL of n-hexane to repeat the extraction twice. Combine the n-hexane solutions of 3 times and dehydrate with 5 g of anhydrous sodium sulfate. Filter the solution and evaporate to less than 5 mL by rotary evaporator. Follow the procedure 'Cleanert® Alumina-N Cleanup' illustrated above to further process the sample.

Please download at: www.bonnaagela.com

Determination of Malachite Green and Crystal Violet Residues in Aquatic Products with HPLC-MS (Cleanert® Alumina-N, Cleanert® PCX, P/N: AL0006-N, CX0603)

Material

Alumina Neutral cartridge: Cleanert® Alumina-N, 1 g/6 mL, activate with 5 mL of acetonitrile.
Cation exchange cartridge: Cleanert® PCX, 60 mg/3 mL, activate sequentially with 3 mL of acetonitrile and 3 mL of formic acid solution.

Experimental

Fresh aquatic product

1) Extraction

Weigh 5.00 g of smashed sample in a 50 mL centrifuge tube. Add 200 µL of mixed internal standard solution and 11 mL of acetonitrile. Sonicate for 2 min; homogenate at 8000 r/min for 30 s; centrifugate at 4000 r/min for 5 min. Transfer the supernatant into a 25 mL colorimetric tube. Wash the blade of homogenizer with 11 mL of acetonitrile in another 50 mL centrifuge tube for 10 s and transfer the solution to the former centrifuge tube. Mash the sediments in the centrifuge tube with a glass rod and vortex mix for 30 s. Sonicate for 5 min and centrifugate at 4000 r/min for 5 min. Combine the supernatant into the 25 mL colorimetric tube, dilute with acetonitrile to 25 mL. Shake up for later use.

2) Alumina-N Cleanup

Load 5 mL of sample onto the activated Alumina Neutral cartridge, and wash the cartridge with 4 mL of acetonitrile. Collect the eluate in a KD–concentrator. Evaporate the solution to approximately 1 mL by rotary evaporator in water bath at 45°C, dilute with acetonitrile to 1 mL. Ultrasonicate the solution for 5 min and then add 1 mL of 5 mmol/L ammonium acetate, ultrasonicate for 1 min. Filter the sample solution through 0.2 µm membrane for HPLC-MS analysis.

Processed aquatic products

1) Extraction

Weigh 5 g of mashed sample in a 100 mL centrifuge tube. Add 200 µL of mixed internal standard solution, 1 mL of 0.25 g/mL hydroxylamine hydrochloride, 2 mL of 1 mol/L p-toluenesulfonic acid, 2 mL of 0.1 mol/L ammonium acetate and 40 mL of acetonitrile, sequentially. Homogenize at 10000 r/min for 2 min and centrifugate at 3000 r/min for 3 min. Transfer the supernatant into a 250 mL separatory funnel. Extract the residue with 20 mL of acetonitrile again and combine the supernatant. Add 30 mL of dichloromethane and 35 mL of water to the separatory funnel and shake for 2 min. Stand to let two layers separated. Transfer the underlayer to a 150 mL pear-shaped flask. Extract with 20 mL of dichloromethane again and combine dichloromethane layers. Evaporate by rotary evaporator in water bath at 45°C almost to dryness.

2) Coupled Cleaner® Alumin-N and PCX procedure

Connect Cleanert® Alumina-N cartridge (top) with Cleanert® PCX (down). Vortex mix to dissolve the residue (c) in 6 mL of acetonitrile (divided into three times) and load the solutions sequentially onto the connected cartridges. Keep the flow rate in Cleanert® PCX cartridge below 0.6 mL/min. Wash Cleanert® Alumina-N cartridge with 2 mL of acetonitrile and discard the eluate. Wash the Cleanert® PCX cartridge with 3 mL of 2% (V/V) formic acid, 3 mL of acetonitrile sequentially and discard the effluent. Elute with 4 mL of 5% ammonium acetate in methanol at the flow rate of 1 mL/min. Collect the eluate in a 10 mL scaled test tube and dilute with water to 10 mL. Filter the sample solution through 0.2 µm filter membrane for HPLC-MS analysis.

4. Determination of Environmental Pollutants

Optimizing the Determination of Extractable Petroleum Hydrocarbons (EPH, P/N: SI500025-30) by SPE-10 Automated Processing Station

Introduction

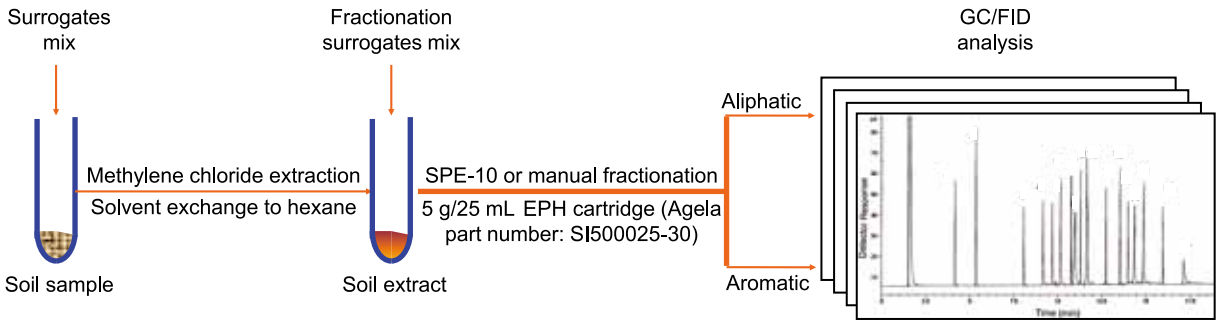
Extractable Petroleum Hydrocarbons (EPH) methodology was recently released by the New Jersey
Department of Environmental Protection (NJDEP) to replace the Total Petroleum Hydrocarbons (TPH) method based on Freon 113 extraction and IR. This method allows aliphatic and aromatic C9-C44 hydrocarbons to be measured separately. It can be used for quantitative analysis of environmental samples (water, soil, sediment and sludge) for residues of crude oils, diesel, sludge and many other types.

Silica gel cleanup and fractionation of aliphaticsand aromatics is a critical and sensitive step in this method. Slight changes, such as the volumes of eluting solvents used, fractionation equipments (including the preparation of the silica gel columns) and fractionation techniques, will have tremendous impact on the proportion of the hydrocarbons separated into their respective aliphatic and aromatic fractions.

Here we present a method based on an automated sample processing station and commercial silica cartridges for rapid, reproducible and efficient fractionation procedure.

Experimental

A. EPH methodSummary



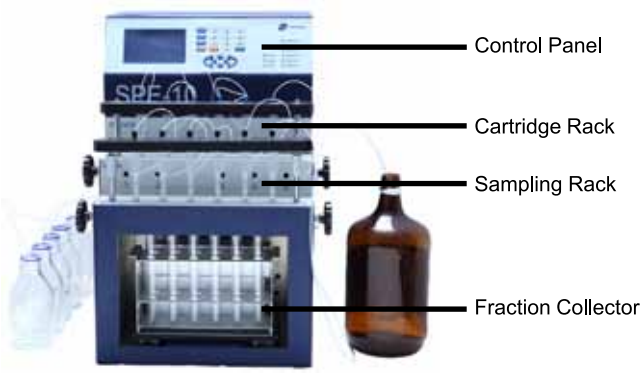
10 Gram soil sample was mixed with pelletized diatomaceous earth, a drying agent, and was loaded into an extraction cell. After adding 100 µL of 1000 ppm surrogate mix (1-chlorooctadecane and o-terphenyl), sample was heated and pressurized with methylenechloride. Extract was collected, dried, concentrated to 1 mLand solvent exchanged to hexane.

With the addition of 1 mL 100 ppm fractionation surrogates mix in hexane (2-bromonaphthlene and 2-fluorobiphenyl), 1 mL extract was fractionated through a 5 g/25 mL silica cartridge (Bonna-Agela Technologies, P/N SI500025-30). Recovered fractions of the aliphatics and aromatics were then concentrated to 1 mL and analyzed separately by Gas Chromatography/Flame Ionization Detector (GC/FID).

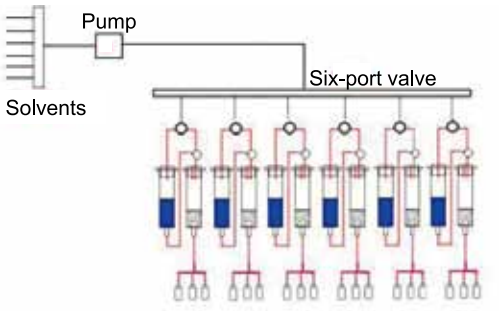
A method blank (MB) was done in the same way except that 10 g diatomaceous earth was used to monitor the contamination of the extraction procedure. Method blank spike (MBS) and duplicate (MBSD) analysis were also performed similarly but in addition to the 100 µL surrogate, 100 µL of the 1000 ppm spike mix (all of the aliphatic and aromatic compounds) was added before fractionation.

B. SPE-10 automated workstation

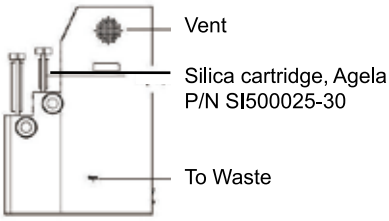
a. Front view



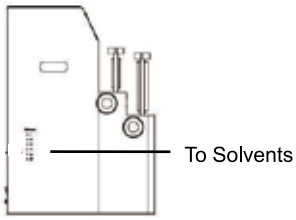
b. System diagram



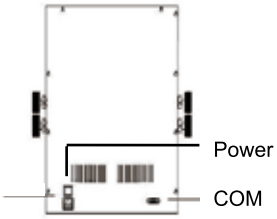
c. Right view



d. Left view



e. Back view

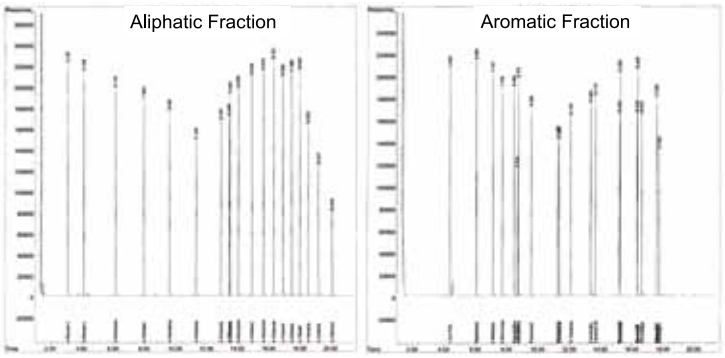


SPE-10 can process up to 6 samples per batch sequentially with automatic 2-fraction collection. The system employs positive pressure delivering constant flow with high precision and good reproducibility. In operation, the introduction of sample does not go through pump and thus prevents clogging with unfiltered samples. The benefits of system include:
-Automatic operation, saves up to 90% cost of labor and solvent.
-Independent channel design, eliminates cross contaminations.
-Combinatorial workstation, provides parallel control for up to 10 units to maximize throughput.

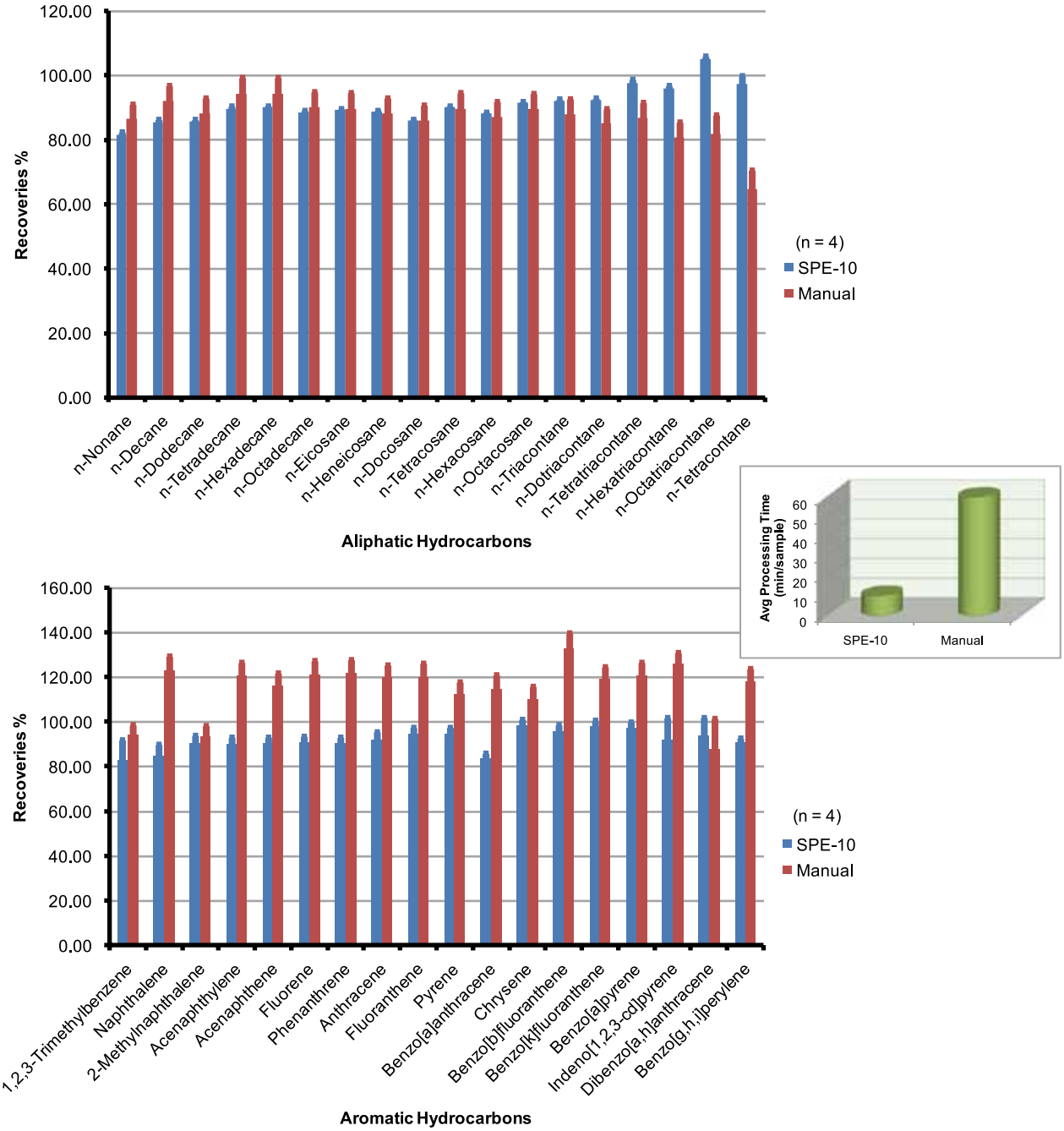
Results

200 ppm Fractionating Check Solution (FCS) 1 was used to monitor the fractionation efficiency of the silica gel cartridge as well as the SPE-10 unit and establish the optimum hexane volume required to efficiently elute the aliphatic fraction without significant aromatic breakthrough (naphthalene and 2-methylnaphthalene were not detected in aliphatic fraction, data now shown).

D. Representative FCS gas chromatogramsF.



E. Fractionation efficiency



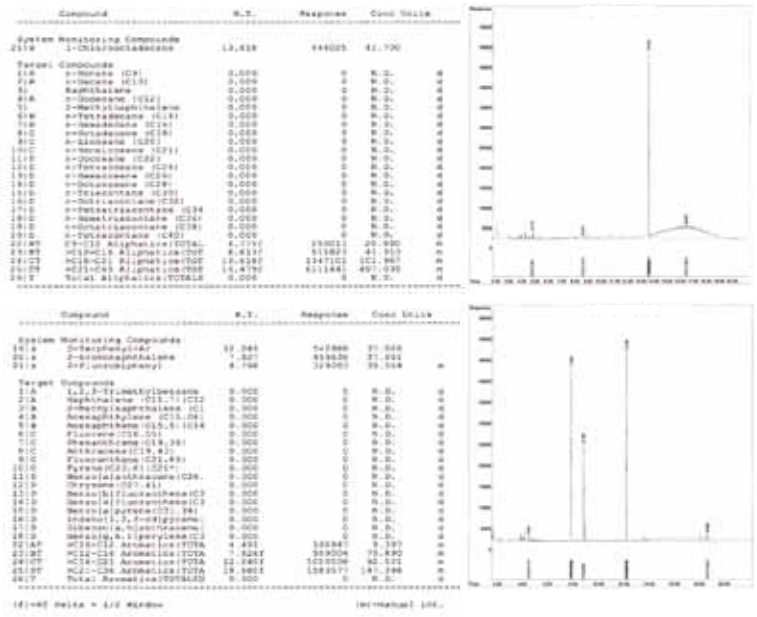
Comparing with manual fractionation on the same batch of silica cartridges, SPE-10 provides comparable recoveries, if not better, with improved reproducibility. Error bars in plots represent one standard deviations.

Average sample processing time by SPE-10, including adding samples and rinsing channels in between different batches of samples, is about 10 min/sample; while during manual fractionation, one would have to monitor the whole procedure for 60 min/sample.

F. Optimized SPE-10 method

Step	Action	Flow Rate (mL/min)	Volume (mL)	Description
0	Elute 1 (hexane)	10	4.5	Add 4.5 mL hexane to silica cartridge.
1	Elute 4 (air)	10	4.5	Blow dry silica cartridge. Step 0-1 is used to minimize the possible differences between silica cartridge packings.
2	Elute 1 (hexane)	20	9.0	Rewet the silica cartridge with 9.0 mL hexane. Cartridge needs to be wet before adding samples for best aliphatic recoveries.
3	Add sample	10	1.0	Add 1.0 mL of sample onto cartridge.
4	Collect 1 (hexane)	5	15.0	Elute and collect aliphatic fraction with 15.0mLhexane. Flow rate and hexane volume is optimized. If fractionation is done manually, flow needs to be controlled as “drop-by-drop” for best aliphatic recoveries.
5	Collect 2 (methylene chloride)	10	30.0	Elute and collect aromatic fraction with 30.0 mL methylene chloride.
6	Elute 4 (air)	10	4.5	Push out any remaining aromatic fraction on the cartridge with 4.5 mL air.

G. Quantitationreport of a real soil sample



Summary

SPE-10 automated processing station provides high fractionation capability with improved reproducibility comparing with manual fractionation.

Processing time per sample was reduced to 1/6 of the manual fractionation time and this relieves the laboratory labor of babysitting the whole process.

Selected Reference

1. Analysis of Extractable Petroleum Hydrocarbon Compounds (EPH) in Aqueous and Soil/Sediment/Sludge Matrices, New Jersey Department of Environmental Protection, Office of Data Quality, August 2010, Revision 3.

Determination of Phenols in Water (Cleanert® PEP, P/N: PE0603)

Material

SPE Cartridge: Cleanert® PEP 60 mg/3 mL
7 types of phenols: phenol, 4-nitrophenol, metacresol, 2-chlorophenol, 2,4-bitin, 2,4,6- trichlorophenol, pentachlorophenol

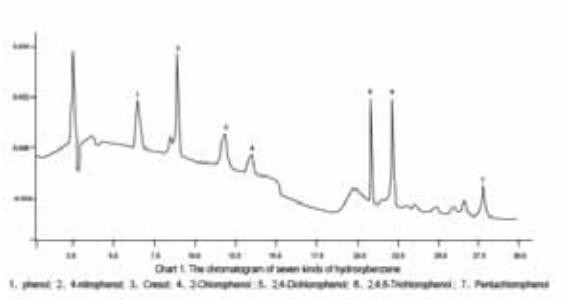
Experimental

Cleanert® PEP procedure

1) Activation: activate the Cleaner® PEP cartridge sequentially with methyl tertiary butyl ether of 3 mL (10:90, V/V), methanol of 3 mL and deionized water of 3 mL at 5 mL/min
2) Wash: wash the cartridge with deionized water of 10 mL at 5 mL/min and dry it for 20 min by vaccum
3) Elution: elute the methanol of 2 mL and methy tertiary butyl ether (10:90, V/V) by two steps and collect the elution to the fine tip flask
4) Concentration: concentrate the collected elution of 2 mL with a stream of nitrogen to 1 mL

HPLC conditions

Column: Unisol C18 (4.6 × 150, 5 μm)
Mobile phase: A: 1% acetic acid
B: 1% acetic methyl alcohol
Detector: UV



Time	Mobile phase ratio	Flow rate(mL/min)	Detection wavelength(nm)
0-15 min	A:B = 50:50	1	275
15-30 min	A:B = 15:85	1.8	295

Results

Recovery of the 7 types of phenol seen in Table 1

Table 1 Result of recovery of the phenols

	Mark			Average value	Standard deviation	Average recovery (%)
	1	2	3			
phenol	1.367	1.541	1.524	1.477	0.096	100.3
4-nitrophenol	1.229	1.308	1.430	1.322	0.101	90.0
metacresol	1.294	1.540	1.548	1.461	0.144	106.3
2-chlorophenol	0.527	0.684	0.641	0.617	0.081	100.6
2,4- bitin	1.305	1.613	1.621	1.513	0.180	92.8
2,4,6- trichlorophenol	1.365	1.609	1.511	1.495	0.123	90.3
pentachlorophenol	1.259	1.487	1.472	1.406	0.128	95.6

SPE Methods for Polycyclic Aromatic Hydrocarbons (PAHs) in Water (Cleanert® PEP, P/N: PE0603)

Material

Cleanert® PEP, 60 g/3 mL

Target components

Naphthalene, acenaphthene, acenaphthylene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, chrysene, benzo (a) anthracene, benzo (k) fluoranthene, benzo (a, h) fluoranthene, Benzo (a) pyrene, Benzo (g, h, i) pyrene and indeno (1, 2, 3-cd) pyrene.

Experimental

Sample preparation

Add 20 mL of 10% nitric acid into 1 L of water

PEP cleanup

Activation: isopropanol 5 mL, water 5 mL, sequentially
Sample Loading: Load the prepared sample on PEP cartridge
Washing: eluant (water 300 mL + methanol 700 mL + Na_2HPO_4 2.1 g + KH_2PO_4 2.04 g) 5 mL
Drying: dry the cartridge by vacuum pump for 30 min.
Elution: eluant (isopropanol 90 mL + acetic acid 10 mL + toluene 200 mL + petroleum ether 1 L)
Reconstitute sample to 4 mL with methanol after evaporation of the collected eluant.

HPLC conditions

Column:Venusil® PAH, 4.6 × 250 mm, 5 μm, 200 Å
Sample: soluble in methanol: dichloromethane (1:1) 16PAHs sample, dilute 10times with methanol and dichloromethane (1:1)
Flow Rate: 1.2 mL/min
Injection: 10 μL
Temperature: 30°C
Wavelength: 254 nm
Gradient:

Time (min)	Methanol (%)	Water (%)
0	85	15
2	85	15
7	95	5
40	95	5

SPE Method to Cleanup Aqueous Sample of Nitrobenzene (Cleanert PEP, P/N: PE5006)

Material

Cleanert PEP, (500 mg/6 mL)

Experiment

Sample preparation

Adjust pH of water sample to neutral. Add methanol to each sample to obtain solution containing 0.5% methanol.

PEP SPE method

- PEP cartridge activation: Set PEP cartridge on SPE system. Wash the cartridge with 3 mL of n-hexane; add 5 mL of methanol; load 10 mL of water onto the cartridge before it is dry. Keep the cartridge wet and activated.
- Sample enrichment: Transfer definite amount of water sample to a separatory funnel connected with PEP cartridge. Turn on the SPE vacuum system; let the water sample flow through the activated cartridge at the flow rate of below 5 mL/min. Keep the liquid level at least 1 cm above the adsorption bed during the extraction process. After all the sample passes the cartridge, wash the internal wall of separating funnel with 10 mL of ultra pure water and keep vacuum for 20 min.
- Elution of SPE cartridge: Put test tube rack and receiving tubes in the extraction cylinder of vacuum multiple tube system. Elute the cartridge with n-hexane of 10 mL/acetone (90:10, V/V) and collect the outflow into the receiving tubes, concentrate to 1.0 mL by nitrogen evaporators at 40°C for analysis.
- Drying cartridge pretreatment: Add 5 g of anhydrous sodium sulfate to a drying cartridge with sieve plate. Wash the cartridge with 10 mL of acetone, n-hexane, and acetone sequentially to purify the drying cartridge before use.
- Elution: Put test tube rack and receiving tubes in the extraction cylinder of vacuum multiple tube system. Connect the drying cartridge between cartridge holding part of SPE system and extraction cartridge. Load 1 mL of acetone on the cartridge (do not let the cartridge dry in this procedure and let the acetone and packing material balance for 2 minutes). Elute the extraction cartridge with 10 mL of hexane/acetone (90/10, V/V) and let the flow pass the drying cartridge (connected to PEP cartridge). Collect the eluate in receiving tubes. Concentrate the eluate under nitrogen stream at 40°C to 1.0 mL for further analysis. (separated layers caused by residual water can affect the efficiency of evaporation under nitrogen stream).

SPE Cleanup Procedure for Bentazone in Water (Cleanert® PEP, P/N: PE5006)

Material

Cleanert® PEP, (500 mg/6 mL)

Experimental

Sample preparation

Adjust the pH of sample with 0.5 mL of H_2SO_4

PEP cleanup

- Activation: Wash the PEP cartridge with 5 mL of furanidine, 5 mL of methanol and 5 mL of water sequentially.
- Sample Loading: Load 500 mL of water sample onto the cartridge at a flow rate of below 5 mL/min.
- Washing: Wash the cartridge with 5 mL of pure water and dry the cartridge under nitrogen stream for 20 min. Then wash the cartridge with 0.9 mL of methanol and discard the eluate.
- Elution: Elute the cartridge with 3 mL of furanidine at the flow rate of below 1 mL/min. Collect the eluate and concentrate to 3 mL for HPLC determination. Or dehydrate by anhydrous sodium sulfate cartridge and evaporate under a stream of nitrogen to 1 mL for HPLC determination.

Results

Recovery of spiked tap water sample is beyond 85 %.

SPE Methods of 2, 4-D in Water (Cleanert® PEP, P/N:PE5006)

Material

Cleanert® PEP, 500 mg/6 mL.

Experimental

Sample preparation
Adjust the pH of water sample with 0.5 mL H_2SO_4 to 1.5~2.0

Cleanert® PEP cleanup
- Activation: activate the PEP cartridge with 5 mL of methanol and 5 mL of water sequentially.
- Sample loading: Load 500 mL of water sample on the cartridge at a flow rate of below 5 mL/min.
- Washing: Wash the cartridge with 5 mL of pure water and dry the cartridge under nitrogen stream for 20 min. Wash the cartridge with 0.8 mL of methanol (stand for 2 min to ensure methanol to roak packing material thoroughly) and discard the eluate.
- Elution: Elute the cartridge with 3 mL of furanidine at a flow rate of below 1 mL/min. Collect the eluate and concentrate to 3 mL for HPLCdetermination. Or dehydrate with anhydrous sodium sulfate cartridge and evaporate under a stream of nitrogen to 1 mL for HPLC determination.

Results

Recovery of spiked tap water sample is above 80%.

SPE Methods of Chlorophenol in the Water (Cleanert® PEP, P/N: PE5006)

Material

Cleanert® PEP, (500 mg/6 mL)

Experimental

Sample preparation
Adjust the pH of 500 mL of water sample with 0.5 mol/L H_2SO_4 to 1.5~2.0

PEP method
- Activation: activate the PEP cartridge with 5 mL of methanol and 5 mL of water sequentially.
- Sample loading: load water sample onto the cartridge at a flow rate of below 5 mL/min.
- Washing: Wash the cartridge with 5 mL of pure water and dry the cartridge under nitrogen stream for 20 min. Wash the cartridge with 0.8 mL of methanol (stand for 2 min to ensure methanol to soak packing material thoroughly) and discard the eluate.
- Elution: Elute the cartridge with 5 mL of furanidine. Collect the eluate and concentrate under nitrogen stream at 30°C to 1 mL for HPLC determination.

Results

Recovery of chlorophenol is between 75-90%.

Determination of 10 Sulfonylureas Herbicide Residues in Soil Samples with HPLC-MS (Cleanert® HXN, P/N: HX1003)

Material

(1) SPE cartridge: Cleanert HXN, 100 mg/3 mL.
(2) 10 Sulfonylurea herbicides:nicosulfuron,ethidimuron, metsulfuron-methyl, sulfometuron methyl, chlorsulfuron, ethametsulfuron-methyl, tribenuron-methyl, bensulfuron methyl, pyrazosulfuron-ethyl, chlorimuron-ethyl.
(3) Standard solution: dissolve the sulfonylurea herbicides in acetonitrile as standard solution. Dilute the standard solution with acetonitrile to obtain standard working solution for later use.

Experimental

Extraction

Weigh 10.0 g of air-dried soil (filtered with 20 mesh sieve) in a centrifuge tube with cap. Add 10 mL of extractive solution (pH = 7.8, 0.2 M phosphate buffer: methanol (8:2, V/V)). Vortex mix for 3 min, ultrasonicate for 5 min, and centrifugate at 4000 r/min for 10 min. Repeat the extraction procedures twice. Combine the supernatants of three times and adjust pH with 85% phosphoric acid to 2.5.

Cleanert® C18 cleanup and sample concentration

Purify the sample solution with Cleanert® C18 cartridge (100 mg/3 mL): Activate the SPE cartridge with 5 mL of methanol (soak the cartridge for 30 min and then wash). Wash the cartridge with 5 mL of extractive solution (pH adjusted to 2.5 with 85% phosphoric acid). Load the sample onto the cartridge at the flow rate of 1 mL/min. Dry the cartridge under vacuum for 10 min after all the sample flows out. Elute with 3 mL of acetonitrile/phosphate buffer (pH = 7.8) (9/1, V/V). Collect the eluate and evaporate under nitrogen stream to 1 mL.

HPLC conditions

Column: Venusil® ASB C18 (250 mm × 4.6 mm, 5 μm);
Mobile phase: acetonitrile-methanol-water (0.2% acetic acid); flow rate: 1 mL/min; gradient elution procedure is listed in Table 1.
UV: 254 nm; column temperature: 30; injection volume: 10 uL.

Table 1. Gradient elution procedure

Time (min)	Water containing 0.2% acetonitrile (%)	Acetonitrile (%)	Methanol (%)
0.00	80	10	10
14.00	10	45	45
16.00	4	48	48
18.00	80	10	10

Results

Figure 1 and 2 show total ion chromatogram and extracted ion chromatogram of 10 sulfonylurea herbicides by HPLC-MS, respectively; the spiked recoveries of 10 sulfonylurea herbicides in soil samples at three concentration levels (0.01, 0.1, 1 μg/mL) are summarized in Table 1.

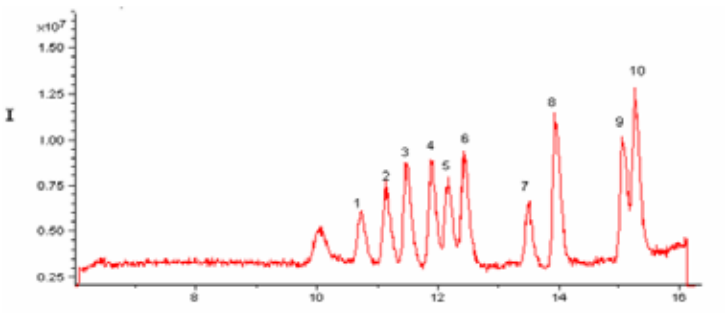


Figure 1. Total ion chromatogram of 10 sulfonylurea herbicides

1. nicosulfuron;
2. ethidimuron;
3. metsulfuron-methyl;
4. sulfometuron methyl;
5. chlorsulfuron;
6. ethametsulfuron-methyl;
7. tribenuron-methyl;
8. bensulfuron methyl;
9. pyrazosulfuron-ethyl;
10. chlorimuron-ethyl

Table 1 Recovery of 10 sulfonylurea herbicides in spiked soil samples at three concentration levels

Sulfonylurea herbicides	Spiked concentration (μg/mL)	Recovery (%)	R.S.D (%)
Nicosulfuron	0.01	94.52	7.22
	0.1	97.31	4.07
	1	84.83	0.02
Ethidimuron	0.01	88.20	0.11
	0.1	87.43	9.95
	1	82.69	0.02
Metsulfuron-methyl	0.01	104.52	2.81
	0.1	87.57	0.25
	1	83.14	0.05
Sulfometuron methyl	0.01	95.52	8.43
	0.1	94.128	4.49
	1	80.16	0.14
Chlorsulfuron	0.01	91.41	5.37
	0.1	85.53	1.65
	1	89.26	0.03
Ethametsulfuron-methyl	0.01	102.67	12.85
	0.1	92.58	14.90
	1	87.72	0.01
Tribenuron-methyl	0.01	34.79	2.20
	0.1	16.57	12.10
	1	11.00	0.03
Bensulfuron methyl	0.01	90.09	8.58
	0.1	83.34	2.15
	1	84.01	0.03
Pyrazosulfuron-ethyl	0.01	101.28	12.24
	0.1	85.98	1.65
	1	86.04	0.03
Chlorimuron-ethyl	0.01	86.70	7.71
	0.1	85.54	8.10
	1	100.53	0.03

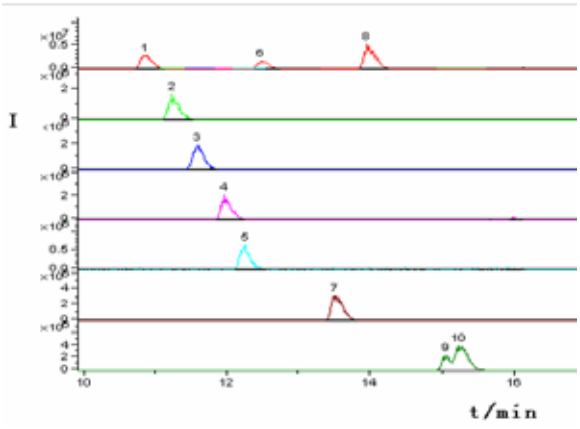


Figure 2. Extractive ion chromatogram of 10 sulfonylurea herbicides

5. Detection of Drug Metabolites

Analysis of Oleic Acid and Its Metabolites in Blood Plasma by LC-MS (Cleanert® PAX, P/N: AX0301)

Material

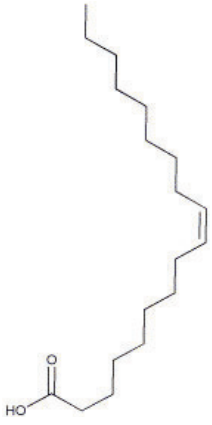


Figure 1. Oleic acid structure

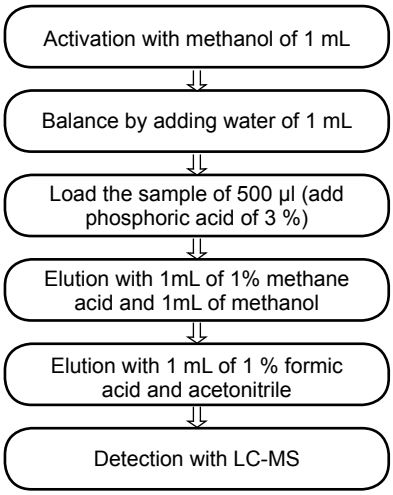


Figure 2. Sample preparation using Cleanert® PAX cleanup

Experimental

Instrument: API Qtrap 3200 (from Applied Biosystem in USA), LC-20A HPLC (from Shimadzu Corporation in Japan).
Mass spectra conditions: electric ion spray sources; detection of anion model; multiple reaction monitoring.
Ionic reaction for quantitative analysis are m/z 281.2→ m/z 281.2 (oleic acid), m/z 315.2→ m/z 315.2 (oleic acid metabolite) and m/z 269.2→m/z 269.2 (internal label C17)
Mobile phase: ACN: 3 mmol/L ammonium acetate = 85:15

Results

Density	10 ng/mL (n = 3)	100 ng/mL (n = 3)	2500 ng/mL (n = 3)
Recovery of oleic acid(%)	77	87	91
RSD	2.9	0.9	0.2

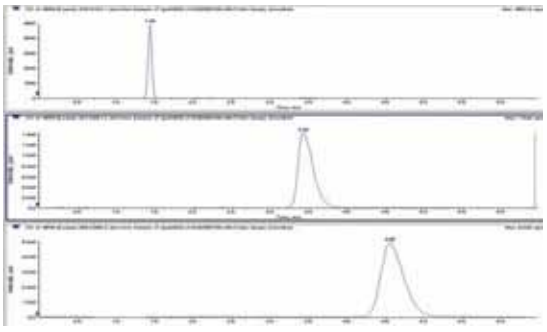


Figure 3. Chromatographic profiles of oleic acid

Rapid Analysis of Pseudoephedrine in Human Plasma Using LC-MS (Cleanert® PCX, P/N: CX0301)

Material

Pseudoephedrine formula (Figure 1)

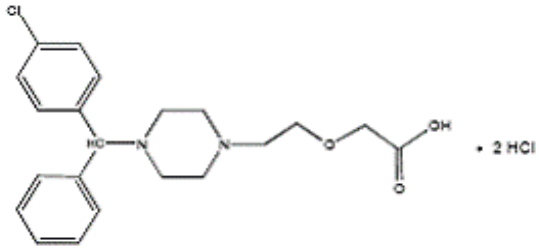


Figure 1. Pseudoephedrine formula

Cleanup (Cleanert® PCX, 30 mg/1 mL)

Activation: first add 2 mL methanol followed by 2 mL water.
Sample loading: load the serum diluted with formic acid of 2%.
Washing: first with 1 mL water and then with 1 mL methanol.
Elution: elute targeted sample with 1 mL of 5% ammonia water and methanol and collect.

Experimental

Instrument: API Qtrap 3200 (Applied Biosystems, USA); LC-20A HPLC (Shimadzu Corporation, Japan)
Mass spectrum conditions: electric ion spray sources; detection of anion model; multiple reaction monitoring.
Ionic reaction for quantitative analysis are m/z 166.0 → m/z 148.1 (Pseudoephedrine) and m/z 235.3 → m/z 86.1 (internal label, lidocaine)

Results

Concertration	10 ng/mL (n = 3)	100 ng/mL (n = 3)	2500 ng/mL (n = 3)
Recovery of Pseudoephedrine (%)	77	79	85
RSD	3.5	1.9	0.5
Recovery of Lidocaine (%)	88	92	87
RSD	4.9	2.1	0.3

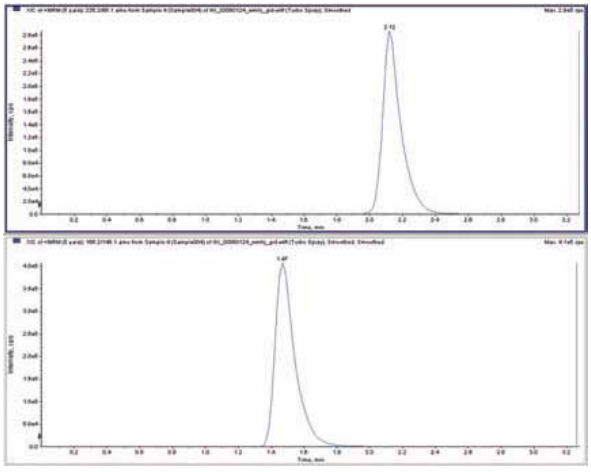
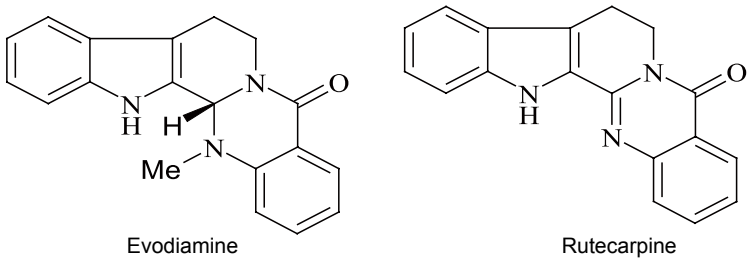


Figure 2. Chromatogram map of pseudoephedrine and internal label of lidocaine

Evodianmine and Rutaecarpine in Human Serum (Cleanert® C18, P/N: S182003)

Material

Cleanert® C18, 200 mg/3 mL, P/N: S182003
Sample: human serum, sample concentration: 50 ng/mL (diluted 2-fold with water)



Evodiamine Rutecarpine

Figure 1. Structural formula of two extracts from Tetradium ruticarpum (evodiamine and rutecarpine)

Experimental

C18 SPE Cleanup

- Cartridge activation: activate the SPE cartridge with 2 mL of methanol followed by 2 mL of water.
- Sample loading: load 2 mL of sample at the flow rate of 0.5 mL/min.
- Washing: wash the SPE cartridge twice with 1 mL of water, then dry the cartridge under nitroge.
- Elution: elute with 1 mL of acetic ether twice, and collect the eluate.

Evaporate the eluate to dryness under nitrogen at 40°C. Dilute with 1mL of methanol-water (85/15).
Filter the solution through 0.45 μm filter membrane for LC-MS analysis.

LC-MS conditions

Instrument: Agilent 1100
Column: Venusil® XBP-C18, 4.6 x 150 mm, 5 μm, P/N: VX951505-0;
Mobile phase: methanol:water = 85:15;
Flow rate: 0.5 mL/min; Column temperature: 25°C;
Injection volume: 10 μl; MS: ESI;

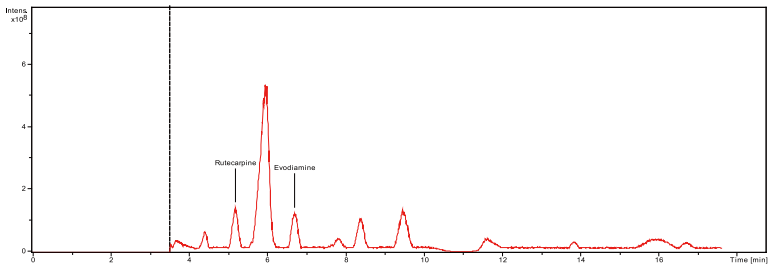


Figure 2. Total ion chromatogram of Evodiamine and Rutecarpine in human serum

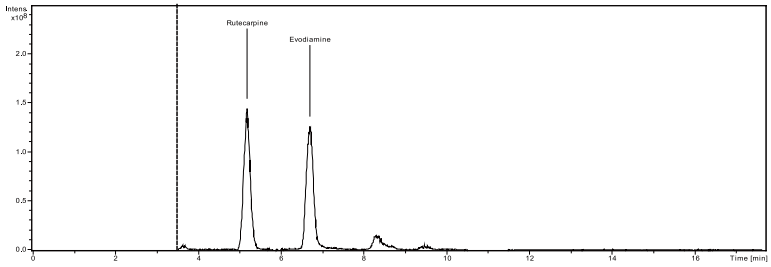


Figure 3. Total ion chromatogram of Evodiamine and Rutecarpine in human serum

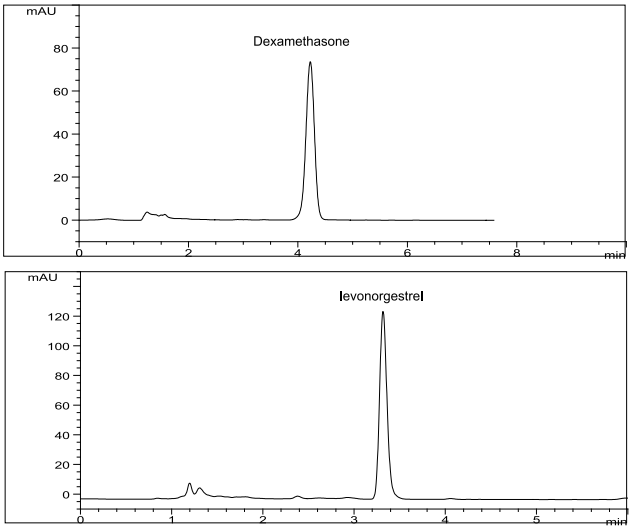
Drug Ingredients in the Serum by SPE (Cleanert® PEP, P/N: PE0603)

Material

Cleanert® PEP, P/N: PE0603, 60mg/3mL.

Experimental

Cartridge activation: 3 mL methanol followed by 3 mL water
Sample loading: load spiked serum sample onto SPE cartridge
Washing: 3 mL water or 5% methanol/water (5:95, v/v)
Elution: methanol 3 mL



Results

Analytes	Recovery (%)
Dexamethasone	97.9
Ethinylestradiol	96.3
Hydrocortisonμm	74.0
Triamcinolone	71.9
Levonorgestrel	93.9
Ganciclovir	54.1
Prednisone Acetate	98.6
Cefalexin	58.6
Cefradine	45.6

Determination of Sulpiride in Human Plasma by SPE and HPLC (Cleanert® C18, P/N: S181001)

Material

Cleanert® C18, 100 mg/1 mL, P/N: S181001

Experimental

Sample preparation
Transfer 0.75 mL of blood plasma accurately to a 2 mL centrifuge tube. Add 10 μL of internal standard solution of metoclopramide and vortex mix.

C18 SPE procedure
●Activation: activate with 2 mL of methanol, equilibrate with 2 mL of water
●Sample loading: load the prepared blood plasma sample onto the cartridge
●Washing: wash the cartridge with 1mL of water after all sample solution flows out
●Elution: methanol 2 mL, collect the eluate and dry under nitrogen in water bath at 55°C. Dissolve the residue in 100μL of methanol. Centrifugate at 3000 r/min for 5 min and take 20 μL of the supernatant for analysis.

Determination of IFO in Serum by SPE and HPLC (Cleanert® C18, P/N: S181001)

Material

(1) SPE cartridge: Cleanert® C18, 100 mg/1 mL.
(2) Cyclophosphamide solution as internal standard: add 0.2 g of cyclophosphamide to a 100 mL volumetric flask. Dissolve and dilute to volume with mobile phase solution. Store this internal standard solution (2 mg·mL-1) in refrigerator at 4°C.

Experimental

Pretreatment of Blood Sample

Add 12.5 μL of internal standard solution to 0.5 mL of serum sample for later use.

C18 cleanup

●Activation: Activate SPE cartridge with 2 mL of acetonitrile, followed by 2 mL of physiological saline.
●Sample loading: Load the prepared sample onto SPE cartridge.
●Washing: Wash the cartridge with 1 mL of physiological saline followed by 1 mL of 5% acetonitrile solution. Dry the cartridge after all eluant flows out.
●Elution: Elute with 0.5 mL of acetonitrile. Collect the eluate and take 20 μL of eluate for further determination.
Figure 1 shows the chromatogram of ifosfamide (retention time: 11.6 min) and cyclophosphamide (retention time: 12.8 min). Both peaks are in good shape without interference of impurities.

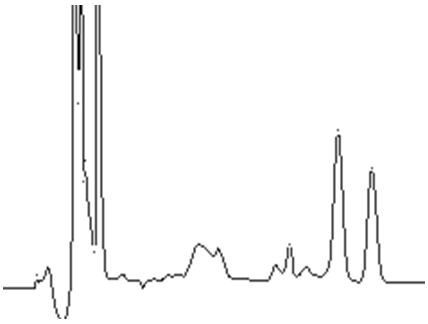


Figure 1. Chromatogram of ifosfamide and cyclophosphamide spiked in blank serum

HPLC Conditions

Column: Venusil® ASB C18 (4.6 mm × 250 mm, 5 μm);
Mobile phase: acetonitrile: water (25:75); Flow rate: 1 mL·min-1;
UV: 200 nm; Injection volume: 20 μL; Column temperature: room temperature.

Results

Table 1. Recovery and precision of ifosfamide (n = 5)

Spiked concentration	Recovery of extraction	Recovery of method	RSD (%)	
(μg·mL-1)	(%)	(%)	Inter-day	Intra-day
100	92.0±1.8	101.2±3.5	1.1	2.1
50	93.6±5.6	107.2±1.5	2.9	4.1
5	89.3±1.3	95.3±4.3	2.2	2.4

Please download the details of the article at the website of Bonna-Agela: www.bonnaagela.com

Detection of Uretic Residues in Animal Urine by HPLC-MS/MS(Cleanert® PAX, P/N: AX0603)

Material

(1) SPE cartridge: Cleanert® PAX, 60 mg/3 mL

Experimental

Sample pretreatment

Transfer 2 mL of sample accurately to a 50 mL centrifuge tube. Adjust pH with 5 mol/L hydrogen chloride solution to 3.5±0.5. Add 1 mL of 5% lead acetate solution and 5 mL of water-saturated acetic ether. Vortex mix and vibrate on shaker for 10 min. Centrifugate at 5000 rpm for 5 min and transfer the supernatant to another 50 mL centrifuge tube. Add 5 mL of water-saturated acetic ether to the aqueous underlayer. Vortex mix and vibrate on shaker for 10 min. Centrifuge at 5000 rpm for 5 min and combine the supernatants. Dry the solution under nitrogen at 50°C. Dissolve the residue in 3 mL of acetonitrile-2% ammonia (10:90, V/V) for later use.

Cleanert® PAX cleanup

Load the sample onto Cleanert® PAX cartridge, which is already activated first with 3 mL methanol and then followed with 3 mL 2% ammonia solution. Wash the SPE cartridge with 3 mL of 2% ammonia, methanol and 5% formic acid, sequentially. Dry the cartridge. Elute the cartridge with 3 mL of methanol containing 5% formic acid. Dry the eluate under nitrogen. Dissolve the residue in 1.00 mL of acetonitrile-0.3% ammonia (10:90, V/V). Vortex mix and filter the solution through 0.22 μm membrane for LC-MS/MS analysis.

Results

Table 1 Recovery of eight diuretics spiked in bovine urine (n = 5)

Spiked concentration (μg/L)	Measured parameter	Drugs							
		Chlorath-iazide	Dihydro-chlorot-hiazide	Hydrofl-umethia-zide	Chlort-alido-ne	Trichlor-methiaz-ide	Methy-clothi-azide	Furose-mide	Etacrynic Acid
20	Mean recovery (%)	88.9	80.0	91.2	71.1	86.6	89.9	95.3	99.8
	Intra-batch RSD (%)	6.7	7.9	5.3	6.9	10.1	7.6	8.8	7.1
	Inter-batch RSD (%)	9.3	10.3	13.5	7.7	15.6	9.9	12.7	13.3
50	Mean recovery (%)	87.2	86.7	86.0	78.6	93.3	91.5	97.7	90.8
	Intra-batch RSD (%)	9.5	6.7	9.8	4.6	7.2	6.5	10.0	8.3
	Inter-batch RSD (%)	11.3	8.5	13.8	7.7	9.8	7.4	13.9	9.8
100	Mean recovery (%)	86.3	93.6	95.6	81.2	101.1	82.6	106.5	109.0
	Intra-batch RSD (%)	6.6	7.9	4.7	8.6	5.2	3.9	5.8	9.3
	Inter-batch RSD (%)	8.9	10.7	6.4	11.8	9.6	7.7	13.0	12.8

Full length article can be downloaded at: www.bonnaagela.com

6. Application of MAS-C Protein Precipitation Columns and SLE in Analyzing Drug Metabolites in Serum Samples

MAS stands for Multi-function Impurity Adsorption SPE. Composite adsorption materials functionalized with different chemistries are used in this method. Thus most interfering matrices in biological samples are adsorbed and strong water soluble components of interest are remained in sample solution. By this method, the sample can be purified and the components of interest can be enriched. The key of this application is to choose appropriate separation material which has good selectivity to the interfering biological matrices, such as proteins, peptides, amino acids, phospholipids and so on. After optimizing experimental conditions (selection of solvents and optimization of pH), MAS can remove most interfering biological matrices in samples and retain the strong water soluble analytes with a recovery higher than 70%. It offers the probability for highly sensitive detection in LC-MS.

The key point of this method is to use dispersed solid phase extraction. First, put pretreated sample into a centrifuge tube and add extractive solvent, like acetonitrile. Then add SPE sorbent, such as C18, PestiCarb, PSA, Alumina-N and so on, and some water removal materials like anhydrous sodium sulfate or magnesium sulfate. Shake up and centrifugate. The supernatant is collected and analyzed. This method, which can replace the cleanup method with SPE mini-cartridge, has following advantages.
① It is fast and simple, with which extraction and cleanup are completed in one step;
② The analyte lost in sample emulsification and concentration procedures is avoided;
③ It is cost effective. However, the weakness is that the detection limit is not satisfactory. Besides, water cannot be removed completely, resulting in the loss of extraction efficiency.

MAS method is simple, fast and economical. Bonna-Agela Technologies now can provide a series of centrifuge tubes containing accurately weighed SPE packing materials to support the MAS products and method kits which are most widely used.

Series of MAS-C mainly employ the MAS-products of 96-well plate to wipe off the protein and phospholipid in drug analysis, and the packings with the specifical adsorption to the endogenous impurities to realize the precipitation of protein and extraction of drugs in one step with special membrance.

The series include MAS-A and MAS-B with the suitable scope as below:
Series of MAS-A is suitable for pretreatment on the acidic drugs in serum and removal of protein and phospholipids;
Series of MAS-B is suitable for pretreatment on the alkaline and neutral drugs in the serum and removal of protein and phospholipids.

Comparison of the Cleanup Capability of MAS, PPT, and traditional SPE Method for analyzing Amlodipine in Serum Sample

Material

(1) Cleanert® MAS-B (60 mg/1 mL)
(2) SPE Cartridge (60 mg/1 mL), Brand W

Experimental

MAS-SPE

- Activation: 2 mL of 2% formic acid and acetonitrile: methanol (80:20).
- Sample: loading: plasma of 200 µL + 50 µL of 200 ng/mL standard amLodipine.
- Elution: 1mL of 1% formic acid and acetonitrile, 2 mL of 2% formic and acetonitrile: methanol (80:20) N_2, constant volume of 0.5 mL.

PPT

Add the sample of 200 μL into 5 mL-centrifuge tube, spike 1 mL of 1 % formic and acetonitrile and 2 mL of 2 % formic and acetonitrile: methanol (80:20), vibrate for 3 min at 10000 rad/min and centrifuge for 15 min, dry the supernate with N_2 to 0.5 mL.

SPE

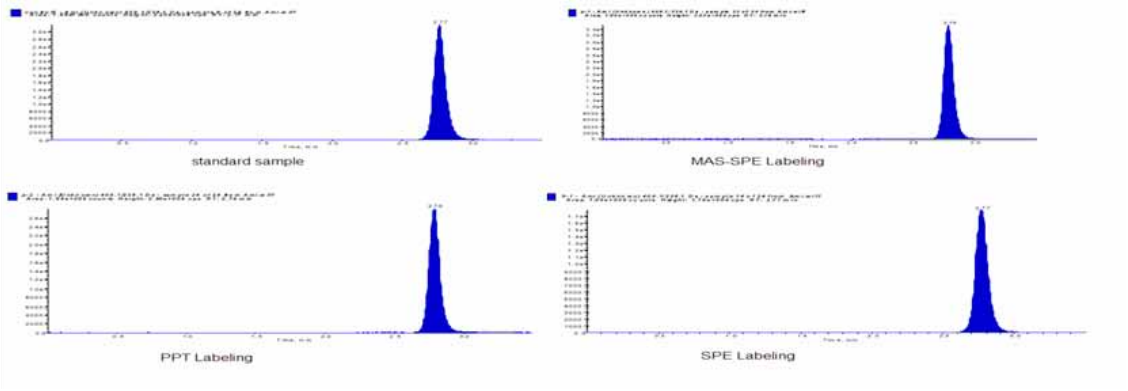
- Activation: methanol of 2 mL, water of 2 mL.
- Sample: loading: plasma of 200 μL + 50 μL of 200 ng/mL amLodipine.
- Washing: 2 mL of 5 % solution of methanol and water.
- Elution: 2 mL of methanol.

HPLC conditions

Column: Venusil ASB C18 (2.1 × 150, 3 μm);
Mobile phase: 10 mmol of ammonium acetate buffer: acetonitrile = 40 : 60;
Flow rate: 0.2 mL/min;
Column temperature: 25°C;
Sample loading: 5 μL;
Detector: ABI4000 QT(amLodipine: MRM 409.1/238.1 m/z.)

Results

(1) Original experiment Chromatogram
(2) Recovery



	Recovery (%)	Mean recovery
MAS-SPE	83.2	82.9
MAS-SPE	82.6	
PPT	81.2	82.4
PPT	83.6	
SPE	59.2	64.5
SPE	70.1	

Propranolol in the Serum by MAS-LC-MS (Cleanert® MAS-B, P/N: MSC-B-0601)

Material

Cleanert MAS-B (60 mg/1 mL)

Experimental

MAS-SPE

- Activation: 2 mL of 2 % formic and acetonitrile: methanol (80:20).
- Sample: loading: 200 µL of plasma + 50 µl of 200 ng/mL propranolol.
- Elution: 1 mL of 1 % formic and acetonitrile, 2 mL of 2% formic and acetonitrile: methanol (80:20) dry with N_2 to 0.5 mL.

PPT

Add 200 µL of sample into 5 mL-centrifuge test, spike 1 mL of 1 % formic and acetonitrile and 2 mL of 2 % formic and acetonitrile: methanol (80 : 20), vibrate for 3 min at 10000 rad/min, centrifuge for 15 min. Dry the supernate with N_2 to 0.5 mL.

HPLC conditions

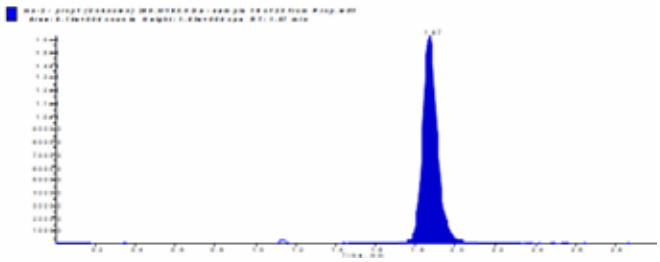
Column: Venusil® ASB C18 (2.1 × 150, 3 µm);
Mobile phase: 10 mmol of ammonium acetate buffer: acetonitrile = 40:60;
Flow rate: 0.2 mL/min; Temperature: 25°C;
Sample loading: 5 µL;
Detector: ABI4000 QT (propranolol: MRM 260.1/183.0 m/z).

Results

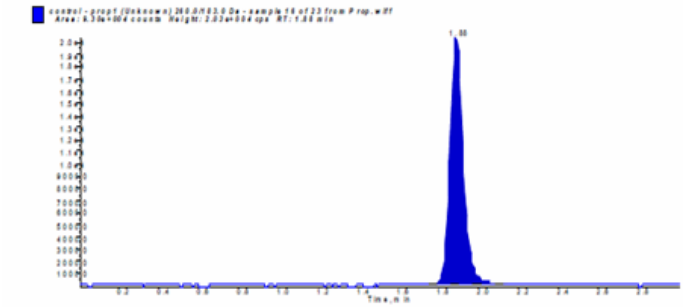
(1) Original Chromatogram
(2) Recovery (Figure1)

Figure 1 Spiked Recovery result

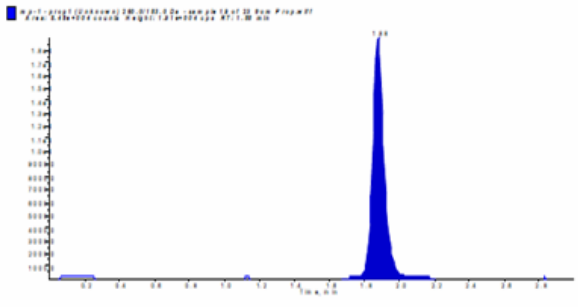
	Recovery (%)	Mean recovery
MAS-SPE	91.2	90.0
MAS-SPE	88.8	
PPT	78.3	82.9
PPT	87.5	



PPT (Spiked)



Standard sample



MAS-SPE (Spiked)

Simultaneous Detection of Glipizide and Glibenclamide with MAS (Cleanert® MAS-B, P/N: MSC-A-0601)

Material

Cleanert® MAS-B (60 mg/1 mL)

Experimental

MAS-SPE

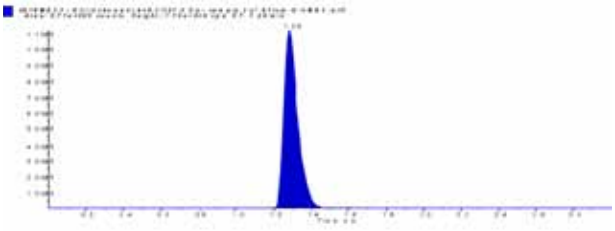
- Activation: 2 mL of 2 % formic and acetonitrile: methanol (80 : 20).
- Sample loading: 200 µL of plasma + 50 µl of 200 ng/mL propranolol.
- Elution: 1 mL of 1 % formic and acetonitrile, 2 mL of 2 % formic and acetonitrile:methanol (80:20), dry with N_2 to 0.5 mL.

HPLC conditions

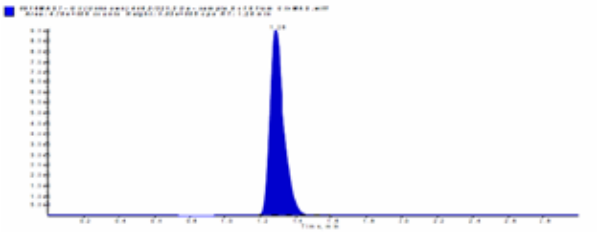
Column: Venusil ASB C18 (2.1 × 150, 3 µm);
Mobile phase: 10mmol ammonium acetate buffer: acetonitrile = 20 : 80;
Flow rate: 0.2 mL/min;
Temperature: 25°C;
Sample loading: 5 uL;
Detector: ABI4000 QT glipizide: MRM 446.3/321.1 m/z; glucovance: MRM 464.2/369.0 m/z;

Results

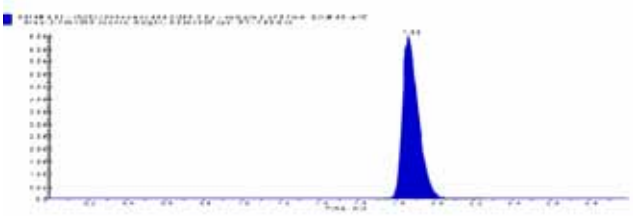
Original Chromatogram



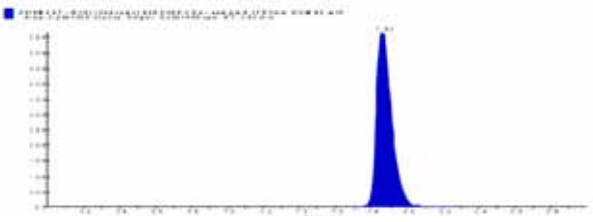
Sample of glipizide



Spiked Glipizide



Sample of glucovance



Spiked Glucovance

Recovery

	Recovery (%)	Mean recovery
Spiked Glipizide	83.8	80.6
Spiked Glipizide	77.4	
Spiked Glucovance	88.9	78.6
Spiked Glucovance	68.3	

Hydrochlorothiazide in Bovine Plasma (Cleanert® MAS-A, P/N: MSC-A-0601)

Material

Cleanert® MAS-A (60 mg/1 mL)

Experimental

MAS-SPE

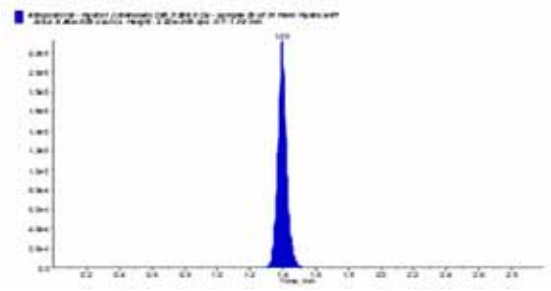
- Activation: 2 mL of 5% ammoniation and acetonitrile.
- Sample: loading: 200 µL of plasma + 50 µL of 200 ng/mL hydrochlorothiazide.
- Elution: 2 mL of 5 % ammoniation and acetonitrile; dry with N_2, constant volume of 0.5 mL.

HPLC conditions

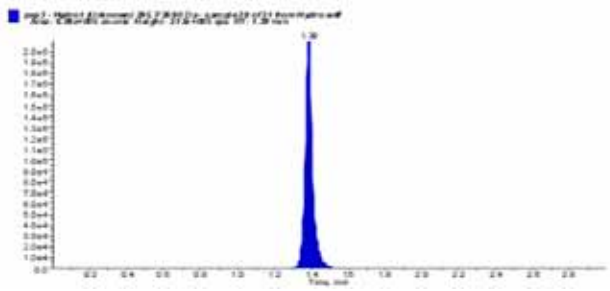
Column: Venusil ASB C18 (2.1 × 150, 3 µm)
Mobile phase: 10 mmol of ammonium acetate buffer: acetonitrile = 10 : 90;
Flow rate: 0.2 mL/min;
Temperature: room temperature;
Sample loading: 2 µL;
Detector: ABI4000 QT (Target: MRM 295.7/269.0 m/z)

Results

Chromatogram



Standard Chromatogram



Spiked Sample Chromatogram

Recovery

	Recovery (%)	Mean recovery (%)
Spiked Hydrochlorothiazide	76.5	75.5
Spiked Hydrochlorothiazide	74.9	
Spiked Hydrochlorothiazide	75.1	

Nadifloxacin in Bovine Serum (Cleanert® MAS-A, P/N: MSC-A-0601)

Material

Cleanert® MAS-A, (60 mg/1 mL)

Experimental

MAS-SPE

- Activation: 3 mL of acetronitrile.
- Sample loading: 0.5 mL.
- Elution: 5 mL of 2% formic and acetonitrile; dry with N_2 to1 mL.

HPLC conditions

Column: Venusil ASB C18 (4.6 × 150, 5 μm);
Mobile phase: 1% of acetic acid:methanol = 45 : 55;
Wavelength: 280 nm;
Flow rate: 1 mL/min;
Temperature: room temperature;
Sample loading: 20 μL;

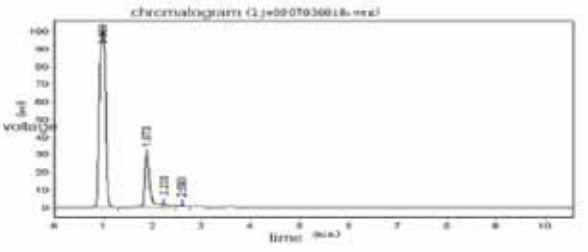


Figure 1. Chromatogram of Bovine Serum Blank

Results

Original experimental Chromatogram
Spiked Recovery (Table1)

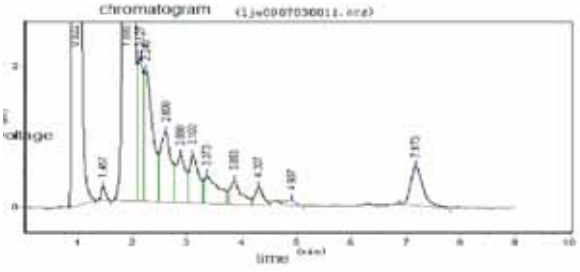


Figure 3. Sample Spiked with 0.5 ppm Standard

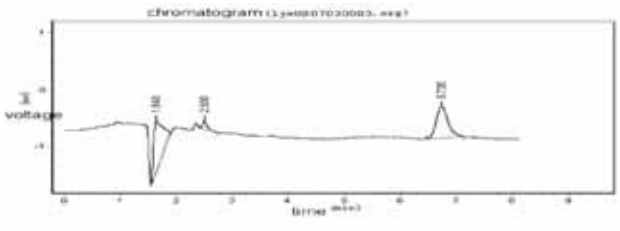
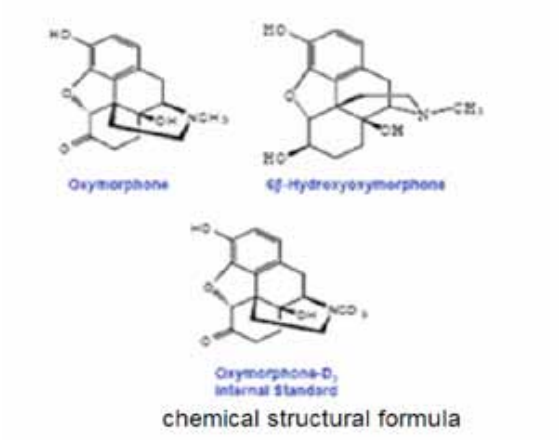


Figure 2. Chromatogram of 0.5 ppm Standard

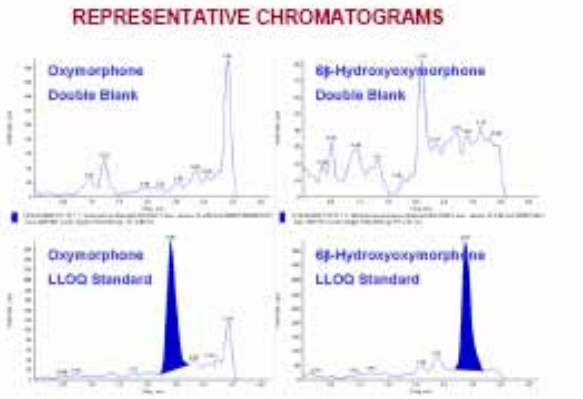
Spiked concertration	Recovery (%)	Mean recovery(%)
0.5ppm of sample passing cartridge	85.2	82.6
1ppm of sample passing cartridge	92.7	95.0
2ppm of sample passing cartridge	96.6	99.1

Extraction of Oxymorphone and its Isomeride in Human Plasma by Cleanert® SLE (P/N: HC2002)

Oxymorphone is a semisynthetic opium agonist, and beta-hydroxyoxymorphone is an important metabolite of the drug. These compounds are isomeride, which are difficult to be separated, and they require extremely low limit of detection.



chemical structural formula



Experimental

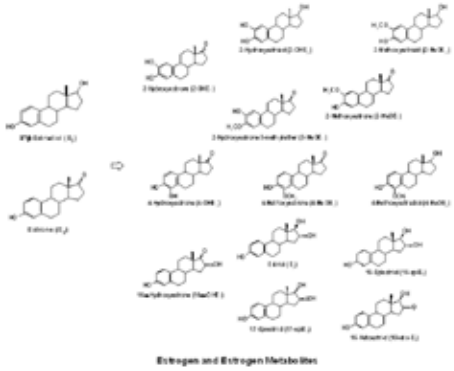
Quickly add 400 µL of plasma spiked with internal standard substance into the pretreated products of Cleanert SLE (200 mg/well/2 mL, P/N: HC2002-W), and process the sample with Tomtec processing station and elute with 1.4 mL of acetic acid and ethyl ester, dry the supernate with N_2 at 40°C and dissolve the treated sample with 200 µL of first class water for LC-MS/MS analysis.
Mass spectrum scanning: first grade of API scanning for 4000 times, TIS positive pole MRM detection:
m/z 302.2 (M + H) +→ 227.20 (oxymorphone)
m/z 304.30 (M + H) + → 268.2 (6β-hydroxyoxymorphone)
Mobile phase: A: water :ammonia water / 100 : 0.05 (v:v)
B:methanol: ammonia water / 100 : 0.05 (v:v)
Gradient elution rised from 25% of B to 100% of B
Flow rate: 300 µL/min
LC Column: Durashell C18, 3 µm, 3.0 x 50 mm

	oxymorphone	hydroxy oxymorphone
Day to day precision(% CV)	1.4—10.8	2.2—10.1
Day to day accuracy(% RE)	-6.9—1.2	-9.2—0.7
Day to day precision(% CV)	2.2—8.1	3.6—8.4
Day to day accuracy(% RE)	-4.4—-0.2	-6.7—-1.7
Mean recovery	82.7%	76.2%

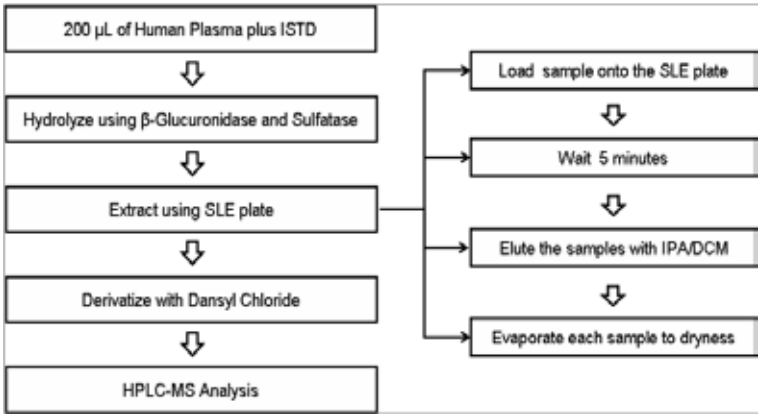
Estrogens and Metabolites Analysis in Human Plasma (SLE-hipure 300 mg/2 mL/well; P/N: HC3002YQ-7W)

Material

SLE-hipure 300 mg/ 2ml/well; P/N: HC3002YQ-7W



Experimental

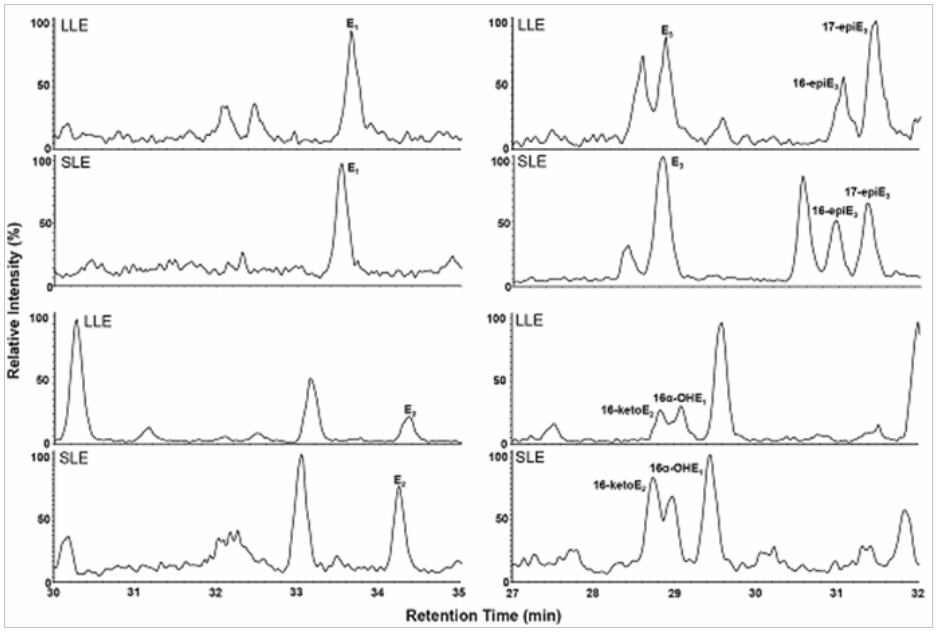


LC-MS CONDITIONS
HPLC: Shimazu solvent delivery system LC-10ADVP
HPLC column: Synergi Hydro-RP, 2 × 150 mm2, 4 µm, 80 A
Mobile Phase A: 0.1% FA in water ; Mobile Phase B: Methanol
Flow rate: 0.4 mL/min MS/MS Detection
Mass spectrometer: Sciex API 4000; Ionization mode: ESI Positive

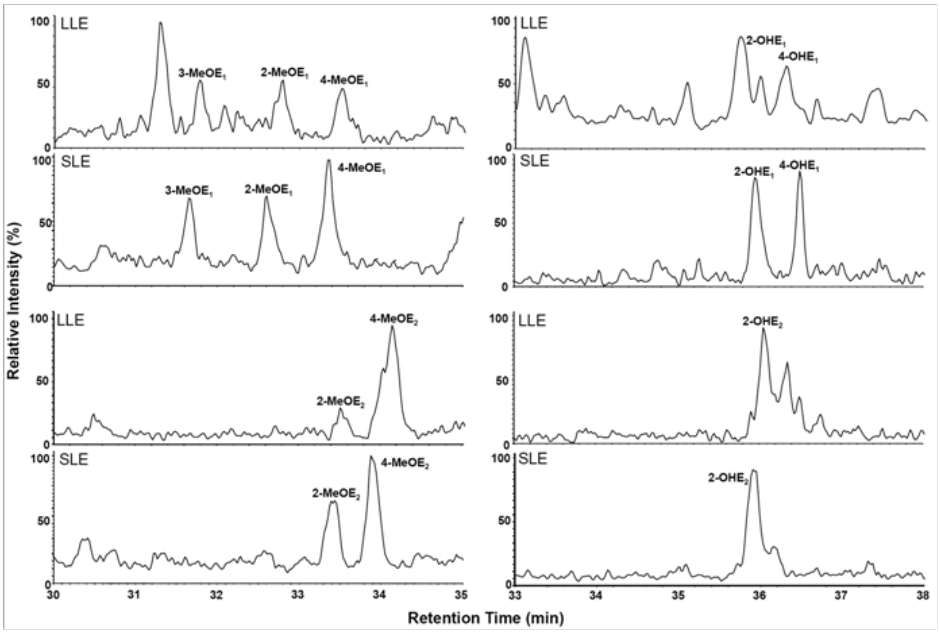
Results

Time (minute)	Module	Function	Value (%)
5.00	Pumps	Pump B Conc.	30
41.00	Pumps	Pump B Conc.	98
46.00	Pumps	Pump B Conc.	98
47.00	Pumps	Pump B Conc.	30
57.00	Controller	STOP	

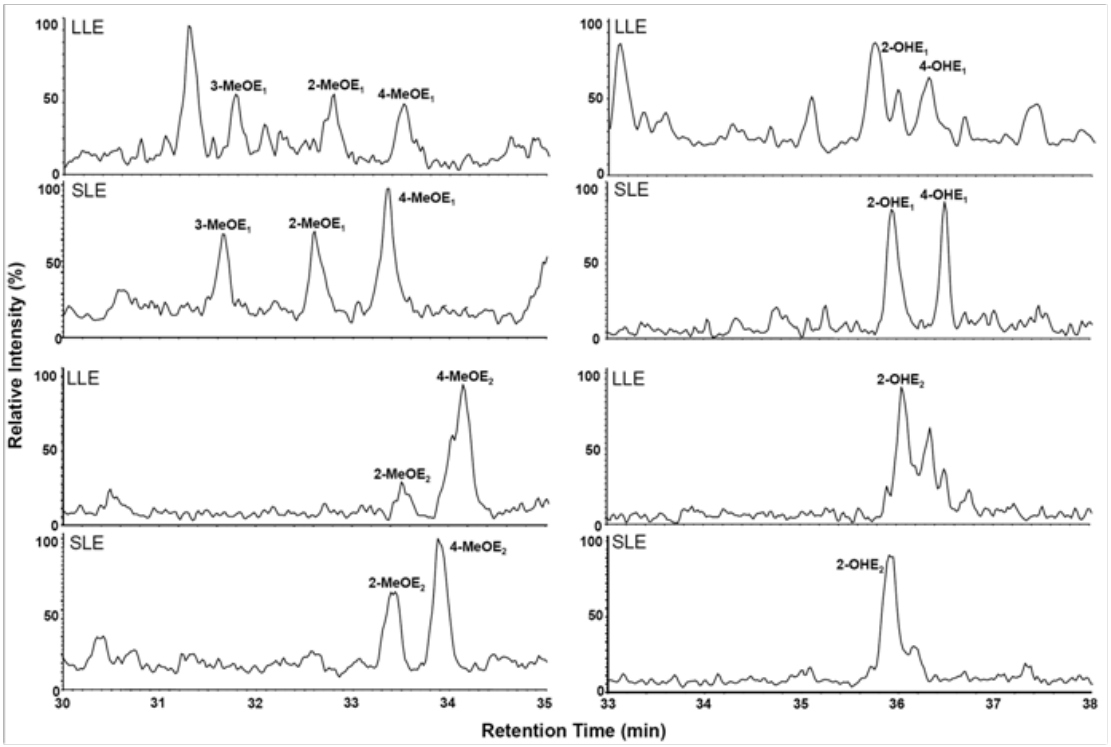
Chromatograms of estrogens and EMs



LLE and SLE method comparison at 10 pg/mL (LLOQ).



LLE and SLE method comparison at 10 pg/mL (LLOQ).



LLE and SLE method comparison at 10 pg/mL (LLOQ).

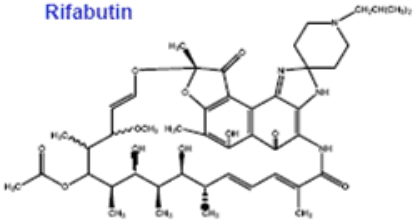
Short-term Stability and Dilution Integrity Results

Nominal Conc. (pg/mL) Freeze and Thaw (n=6)		E1	E2	E3	16-ketoE2	16a-OHE1	16-epiE3
30.0	Conc.	31.7	31.5	31.7	30.2	30.8	30.6
	Accuracy (%)	105.6	105.1	105.7	100.7	102.5	102.1
	CV (%)	4.6	5.8	8.3	7.8	7.5	5.8
7000	Conc.	6985	6750	6845	6665	6523	6968
	Accuracy (%)	99.8	96.4	97.8	95.2	93.2	99.5
	CV (%)	0.7	2.8	2.3	5.6	3.2	2.3
Dilution (n=6							
20000	Conc.	19225	18675	19400	18217	18300	19642
	Accuracy (%)	96.1	93.4	97.0	91.1	91.5	98.2
	CV (%)	3.1	2.4	2.4	5.7	4.2	2.5

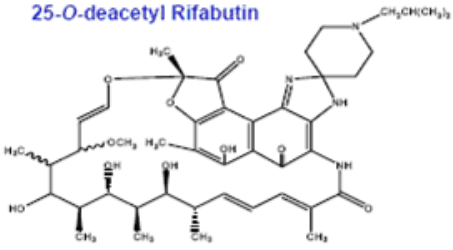
Nominal Conc. (pg/mL)							
Freeze and Thaw (n=6)		17-epiE3	3-MeOE1	2-MeOE1	4-MeOE1		
30.0	Conc.	30.5	28.3	29.9	32.2		
	Accuracy (%)	101.5	94.4	99.8	107.4		
	CV (%)	6.7	7.4	6.6	4.3		
7000	Conc.	7128	7382	7315	6485		
	Accuracy (%)	101.8	105.5	104.5	92.6		
	CV (%)	2.3	1.7	3.8	7.8		
Dilution (n=6							
20000	Conc.	20200	20408	22017	18083		
	Accuracy (%)	101.0	102.0	110.1	90.4		
	CV (%)	2.9	2.0	4.1	2.4		

Analysis of Rifamycin Derivant in Serum by SLE (Cleanert® SLE wellplate, P/N: HC2002)

Rifabutin

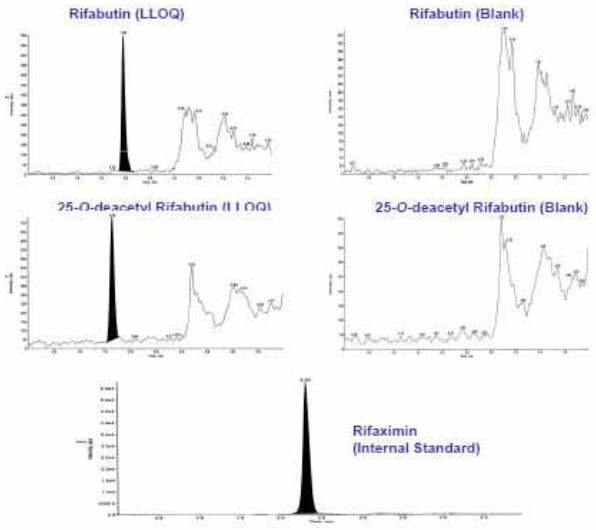


25-*O*-deacetyl Rifabutin



Structure

CHROMATOGRAMS



Experimental

SLE procedure is easier and faster than SPE in method development, and it can be transplanted from LLE. The method can be adopted to high throughput operation using the format such as 96-well microtiter plate. It has been firstly used as alternative cleanup procedure in pharmacokinetic studies.

- Cleanert® SLE wellplate (200 mg/well/2 mL, P/N: HC2002-W) SLE;
- Sample Loading: 50 µL of plasma;
- Elution of targeted sample: 1.4 mL of methyl tert-butyl ether/acetic acid and ethyl ester (1:1, v/v);
- Concentration of collected liquid: dry with N_2 400 µL of methanol:water:acetic acid/45:55:0.1 (v:v:v);

LC-MS analysis: Venusil XBP-C4 analytical cartridge, LC-MS-MS, sample loading of 5 µL.

Mean recovery (%)	Rifabutin: 62.7%; 25-O-deacetyl
	Rifabutin: 66.6%
Mean recovery of internal standard solution (%)	60.7%
QC Day to day precision (%CV)	Rifabutin: 2.0-11.8; 25-O-deacetyl
	Rifabutin: 2.1-9.9
QC Day to day accuracy (%RE)	Rifabutin: -7.6-6.8; 25-O-deacetyl
	Rifabutin: -11.0-6.4
QC Day to day precision (%CV)	Rifabutin: 4.2-9.4; 25-O-deacetyl
	Rifabutin: 3.8-9.3
QC Day to day accuracy (%RE)	Rifabutin: -4.3-3.9; 25-O-deacetyl
	Rifabutin: -6.7-5.0

7. Sample Cleanup Using Mas-Q in Analysis of Pesticides and Veterinary Drugs

MAS-Q series have developed based on the dispersion of matrix, applying to the fast detection of pesticide residue, veterinary residue and food additives. The procedure is fast and straightforward

(1) Add sample solution into a centrifuge tube

(2) Mix the sample with MAS-Q media

(3) Supernate after centrifuging is ready for concentration or direct injection for analysis.

Melamine in Fish, Milk and Eggs by MAS-HPLC (MAS Purified Tube for Melamine, P/N: MS-SPM5001)

Material

Instruments: L6-1 series HPLC (Beijing purkinje general instrument Co., Ltd.); sample preparation method for melamine determination, including HCl 0.1 mol/L, 6% sulfosalicylic acid, mixed anion exchange packing material Cleanert PAX; Venusil SCX-M, 5 μm, 4.6 × 250 mm strong cation exchange cartridge (the achievement of Chinese `11 th Five-Year Plan` supported science and technology project, Bonna-Agela Technologies) and its guard cartridge; needle type filters (Bonna-Agela Clarinert, 0.22/0.45 μm, nylon); melamine standard (>99%); homogenizer (T25 Basic, IKA).

Reagents: acetonitrile (chromatographic grade), potassium dihydrogen phosphate (analytical grade), ultrapure water.

Experimental

HPLC conditions

Column: Venusil® SCX-M, 4.6 × 250 mm, 5 μm, 300 Å;

Mobile phase: potassium dihydrogen phosphate (0.050 mol/L):acetonitrile = 70:30;

Flow rate: 1.5 mL/min; Column temperature: ambient temperature; UV wavelength: 240 nm;

All injection volume is 20 μL if not noted otherwise.

Preparation of melamine working standard solutions

(1) Stock standard solution of melamine: 1.00×10^3 mg/L.

Weigh 100 mg (accurate to 0.1 mg) of melamine standard and dissolve completely in water. Dilute with water to 100 mL and mix.

(2) Working standard solutions

1) Standard solution A: 2.00×10^2 mg/L.

Take 20.0 mL of standard stock solution of melamine (1.3.1) accurately into a 100 mL volumetric flask. Dilute with water to volume and mix for use.

2) Standard solution B: 0.50 mg/L.

Take 0.25 mL of standard solution A (1.3.2.1) into a 100 mL volumetric flask. Dilute with water to volume and mix for use.

3) Working standard solutions

Take different volumes of standard solution A (1.3.2.1) into volumetric flasks according to Table 1. Dilute with water to volume and mix. Filter the solutions through 0.45 μm membrane for determination. Take different volumes of standard solution B (1.3.2.2) into volumetric flasks according to Table 2. Dilute with water to volume and mix. Filter the solutions through 0.45 μm membrane for determination.

Table 1 Preparation of working standard solutions (high concentration)

Volume of standard solution A (mL)	0.1	0.25	1.00	1.25	5.00	12.5
Volumetric flask volume (mL)	100	100	100	50	50	50
Final concentration (mg/L)	0.20	50	2.00	5.00	20.0	50.0

Table 2 Preparation of working standard solutions (low concentration)

Volume of standard solution B (mL)	1.00	2.00	4.00	20.0	40.0
Volumetric flask volume (mL)	100	100	100	100	100
Final concentration (mg/L)	0.005	0.01	0.02	0.10	0.20

Sample preparation using MAS cartridge

(1) Milk sample

Take 15.0 g of milk into a 25 mL volumetric flask. Add 7 mL of 0.1 mol/L HCl, 3.0 mL of 60 g/L sulfosalicylic acid. Add the sample into the melamine MAS Tube (S/N:MS-SPM5001) After vortex mixing for 2 min and centrifugation at 1000 r/min, collect the supernatant and filter it through 0.45 μm membrane for LC analysis.

(2) Egg sample

Take 0.5 g of well mixed egg sample and add 10 mL of 0.1 mol/L HCl for 10 min-ultrasound. Add 3 mL of 60 g/L sulfosalicylic acid and 2 mL of 0.1 mol/L HCl . Add the sample into the melamine MAS tube (S/N:MS-SPC5001). After vortex mixing and centrifugation at 1000 r/min for 10 min, collect the supernatant and filter it through 2 μm membrane for LC analysis.

(3) Fish sample

Take 1.0 g of chopped meat of fish back and 10 mL of 0.1 mol/L HCl. After ultrasound for 10 min , add 3.0 mL of 60 g/L sulfosalicylic acid and 2 mL of 0.1 mol/L HCl. Transfer the sample into MAS tube (S/N:MS-SPC5001). After vortex mixing and centrifugation at 1000 r/min for 10 min, collect the supernatant and filter it through 2 μm membrane for LC analysis.

Results

(1) Linear range

The curve of low concentration is made by 5 standard samples in different concentrations - 0.005 mg/L, 0.01 mg/L, 0.02 mg/L, 0.1 mg/L, 0.2 mg/L. The standard working curve is shown as below.

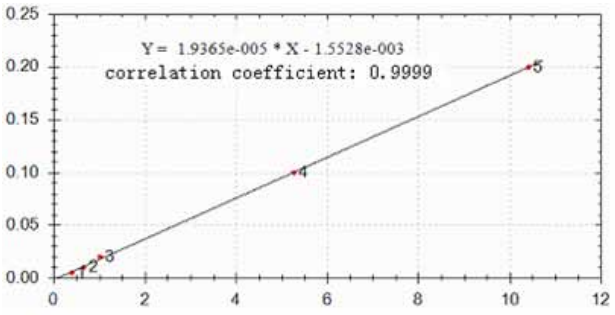


Figure 1. Linearity of low concentration samples

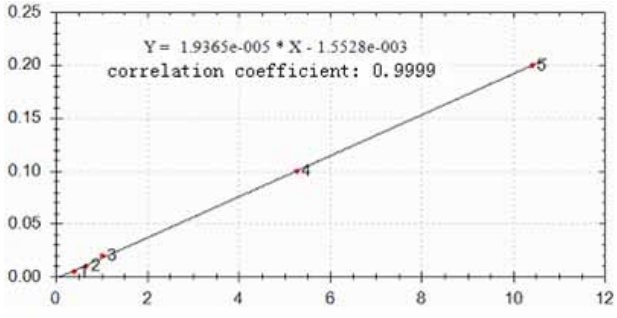


Figure 2. Linearity of high concentration samples

The curve of high concentration is made by 6 standard samples of different concentrations - 0.2 mg/L, 0.5 mg/L, 2 mg/L, 5 mg/L, 20 mg/L, 50 mg/L. The standard working curve is shown as below.

Results show good linearity of melamine at concentrations between 0.005 mg/L~ 50.0 mg/L.

(2) Reproducibility

Analyze 20 μL of 5 mg/L standard solution 6 times. Calculate the RSD of retention time and peak area. The results are listed in the table 3. Table 3 and figure 3 show good reproducibility of this method.

Table 3 Repeatability (precision) of retention time and peak area

Item \ Mumber	1#	2#	3#	4#	5#	6#	Average	RSD%
Rentention time/min	6.817	6.833	6.817	6.817	6.858	6.808	6.825	0.26
Peak area/μAu·s	571360	574492	569813	572187	574525	575593	572995	0.39

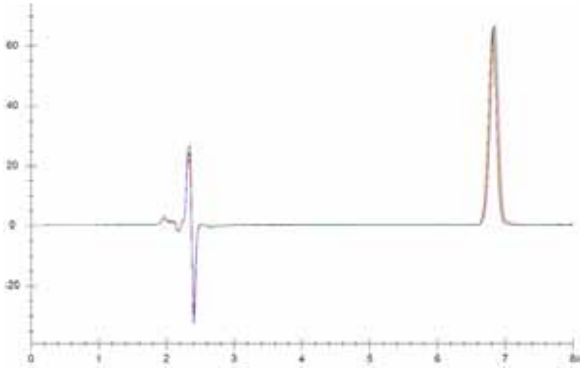


Figure 3. Chromatogram showing repeatability

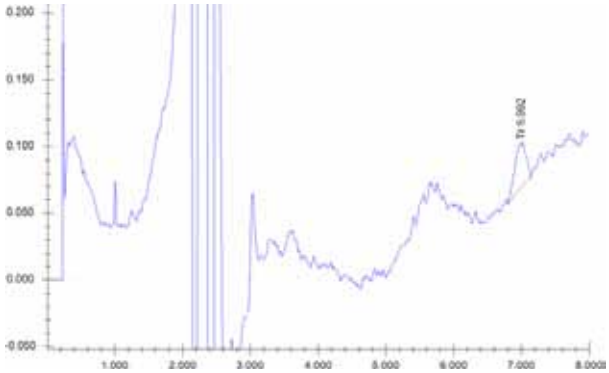


Figure 4. Chromatogram of 0.005 mg/L sample (indicating limit of detection)

(3) Limit of detection

Limit of detection (LOD) is estimated to be 0.0032 mg/L based on the peak height of response from 0.005 mg/L sample while considering signal to noise (S/N) ratio is 3.

(4) Real samples

1) Milk sample

A) Figure 5 shows the chromatogram of blank milk sample.

B) Recovery of spiked milk sample.

Figure 6 shows the chromatograms of spiked milk samples at concentrations of 1.0 mg/kg, 5.0 mg/kg and 10.0 mg/kg.

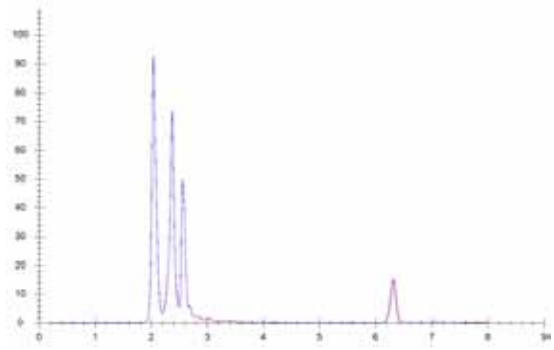


Figure 5. Chromatogram of blank milk sample

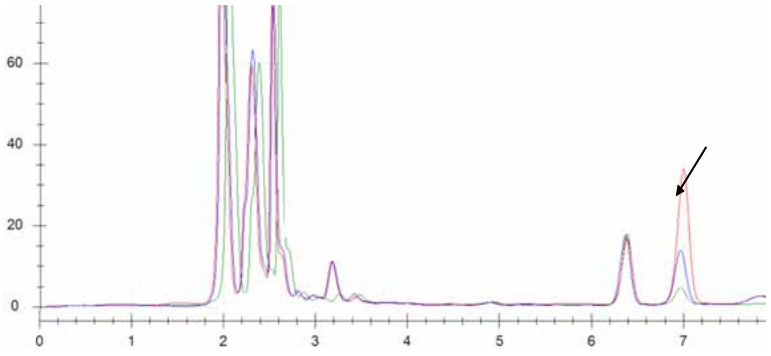


Figure 6. Chromatograms of spiked milk samples at different concentration levels

The recoveries of melamine obtained from above chromatograms are listed in table 4.

Table 4 Results of recovery of melamine spiked in milk

Sample	Amount of spiked melamine (mg/kg)	Amount of measured melamine (mg/kg)	Recovery(%)
Blank milk	0.00	-	-
1#	1.00	1.05	105.0%
2#	5.00	5.18	103.6%
3#	10.00	9.04	90.4%

The results show that the MAS cleanup method of melamine in milk has a good recovery and impurities are effectively removed. The melamine can be well separated from impurities.

2) Fish sample

A) Figure 7 shows the chromatogram of blank fish sample.

B) Recovery of spiked fish sample.

Figure 8 and 9 show the chromatograms of spiked fish samples at concentrations of 1.0 mg/kg, 5.0 mg/kg and 10.0 mg/kg.

From the chromatograms, the recoveries of melamine spiked in fish samples are listed in table 5.

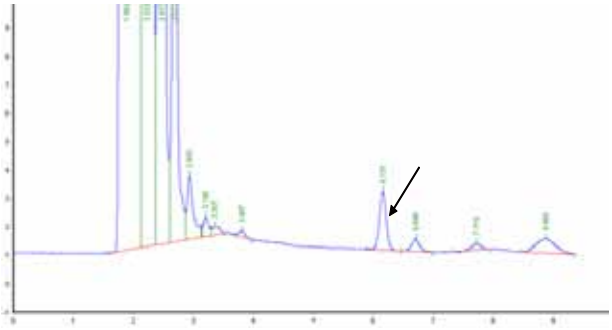


Figure 7. Chromatogram of blank fish sample

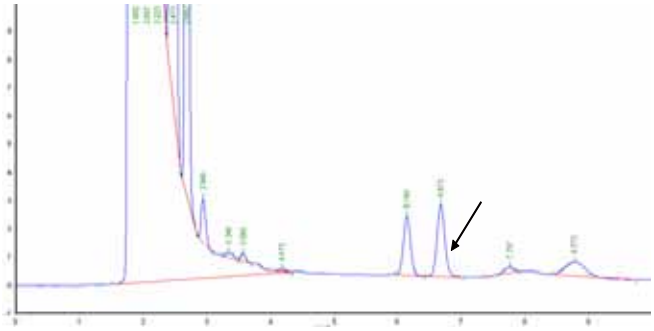


Figure 8. Chromatogram of spiked fish sample (1.0 mg/kg)

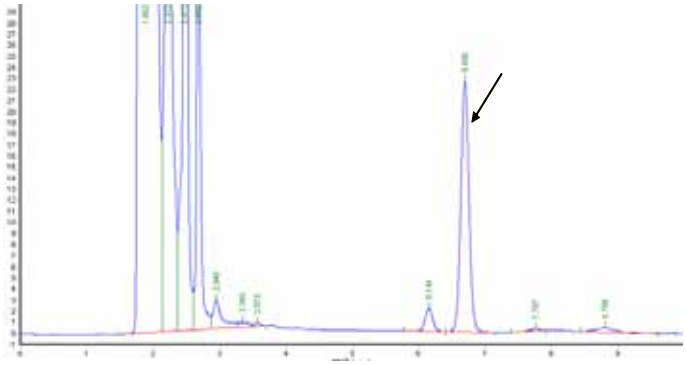


Figure 9. Chromatogram of spiked fish sample (10.0 mg/kg)

Table 5 Results of recovery of melamine spiked in fish

Sample	Amount of spiked melamine (mg/kg)	Amount of measured melamine (mg/ Kg)	Recovery(%)
Blank	0.00	-	-
1#	1	0.98	98.0%
3#	5	5.81	116.2%
5#	10	9.67	96.7%

The results show that the MAS pretreatment method of melamine in fish has a good recovery and impurities are effectively removed. The melamine can be well separated from impurities.

3) Egg sample

A) Figure 10 shows the chromatogram of blank egg sample.

B) Recovery of spiked egg sample.

Figure 11 shows the chromatograms of spiked egg samples at concentrations of 1.0 mg/kg, 5.0 mg/kg and 10.0 mg/kg.

The recoveries of melamine spiked in egg samples are listed in table 6.

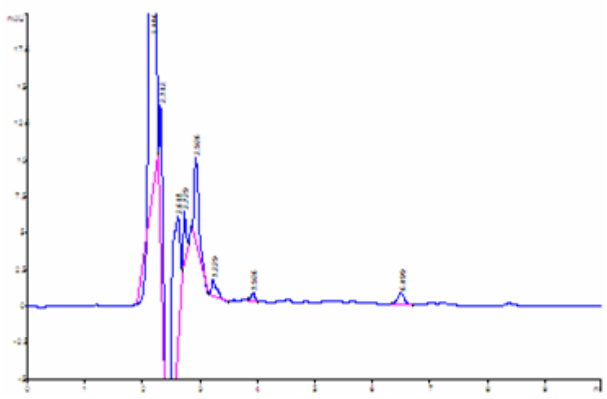


Figure 10. Chromatogram of blank egg sample

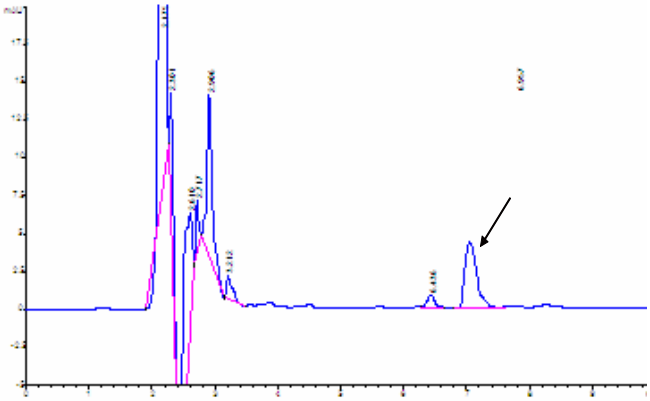


Figure 11. Chromatogram of spiked egg sample (5.0 mg/Kg)

Table 6 Recovery of melamine spiked in egg samples

Sample	spiked melamine (mg/kg)	measured melamine (mg/ Kg)	Recovery (%)
Blank	0.00	-	-
1#	1	0.89	89.0%
3#	5	5.55	111.0%
5#	10	10.48	104.8%

The results show that the MAS cleanup method of melamine in egg sample has a good recovery and impurities are effectively removed. The melamine can be well separated from impurities.

Conclusions

The above experiment results show that the MAS sample pretreatment method, which utilizes mixed anion exchange packing Cleanert PAX as extraction material, provides a fast and accurate approach for treatment of milk, fish and egg samples. Cation exchange column is used for HPLC analysis. The whole analysis method can be used for melamine in different samples, with low limit of detection, high repeatability, wide linear range and good recovery, which totally meets the demands of fast melamine analysis.

QuEChERS Application in Fast Analysis of Multiple Pesticide Residues (Cleanert® PSA, C18, PestiCarb, NH_2, P/N: PA0010, 180010, PC0010, NH0010)

Material

PSA absorbent: Cleanert® PSA
C18 absorbent : Cleanert® ODS C18
Graphite carbon black absorbent: Cleanert® PestiCarb
NH_2 absorbent : Cleanert® NH_2

Experimental

Sample preparation

Take edible parts of samples, mince and mix. Weigh 15 g (accurate to 0.01 g) of sample and place it into a 100mL plastic centrifuge tube. Add 15 mL of 0.1% acetic acid / acetonitrile solution, 6.0 g of anhydrous magnesium sulfate, 1.5 g of sodium acetate and homogenize.

Centrifugate at 5000 r/min for 5 min. Take 10mL of organic phase accurately into a 15 mL plastic centrifuge tube. Dry the solution under nitrogen stream. Vortex mix to dissolve the residue in 2.0 mL of 0.1% acetic acid / acetonitrile solution. According to the interference of sample matrix, select and weigh proper amount of absorbents like C18, PSA, graphite carbon black or NH_2, and place into another 15 mL plastic centrifuge tube. Transfer 2 mL of the above dissolved solution to the centrifuge tube. Vortex mix for 2 min and centrifuge at 5000 r/min for 3 min. Take the supernatant with a disposable syringe and filter through 0.45 μm membrane for analysis.

GC analysis

The treated sample can be analyzed with GC-FPD for organophosphorus pesticide determination, GC-ECD for organochlorine pesticides determination or GC-MS for multi-residue organophosphorus, organochlorine, carbamate et al. Here GC-MS is applied for simultaneous multiple residues. The conditions of instrument are as follows:

Column: Bonna-Agela DA-35MS capillary column, (30 m × 0.25 mm × 0.25 μm, P/N: 3525-3002);

Temperature programming in column box: 50°C (for 2 min), 10°C/min to 180°C (hold on for 1 min), 3°C/min to 250°C (hold on for 1 min), 2°C/min to 270°C (hold on for 15 min);

Inlet temperature: 250°C;

Carrier gas: He (>99.999%), constant flow, flow rate 1.0 mL/min;

Injection volume: 1 μL;

Injection type: Splitless injection. After 0.8 min switch the splitting valve on.

Electron impact ionization source: 70 eV;

Temperature of ionization soure: 250°C

Temperature of GC/MS interface: 250°C

Selected ion monitoring: For each compound, select one ion for quantitative analysis and 2~3 ions for qualitative analysis. All the ions to be determined in each group are monitored respectively at different time, according to their retention times.

Results

Recovery and precision experiments are carried out with spiked spinach, cabbage and yellow peach samples at three concentration levels. For each level, experiments are repeated six times. At the concentrations between 0.05 mg/kg~1.0 mg/kg, the recovery and RSD are 65%~120% and 1%~13.5%, respectively.

Discussion

(1) Improvement of sample extraction

In a typical QuEChERS cleanup for analysis of multiple pesticide residues, 'shaking' the sample mixture is employed to extract target compounds, and 2 mL aliquot of the organic solution is taken for analysis. In this study, however, it is found that homogenization for sample extraction improves QuEChERS procedure with higher efficiency. This is useful for samples that contain high content of fibers. On the other hand, this study also shows that a concentration step is necessary to enhance the detectability of target compounds. 10 mL Aliquot was used in the procedure, which results in a 5 magnitude concentration comparing to 2 mL. The drying effect of anhydrous magnesium and sodium sulfates was compared. Before sample introduction to GC analysis, magnesium sulfate anhydrous is more effective to remove water content in the sample solution of reconstitution This is helpful to reduce interfering background when using electron capture detector (ECD) in the GC analysis.

(2) Improvement of sample cleanup

It is shown that Cleanert PSA is more effective to remove fatty acids in sample matrices while Cleanert C18 and graphite carbon black (PestiCarb) have good cleanup performance on pigments, sterols and vitamins. Cleanert NH_2 is more adsorptive to interference than PSA. Therefore, besides the evaluation of PSA in this paper, Cleanert® C18, NH_2 and PestiCarb were also tested. Even though the amount of sorbents used cannot be specified explicitly for given sample matrices, it always falls into the range from 100 to 350 mg. The extract of spinach was clear and colorless, for example, with our improved QuEChERS procedure while the solution was greenish with the 'standard' QuEChERS. In the recovery experiment, we found that C18 has no impact upon recovery. But PSA, NH_2 and PestiCarb could reduce recovery of some pesticides if the amount of sorbent used was too much since the analytes can be strong adsorbed. In practice, it is recommended that the type and the amount of sorbents should be screened and optimized based on the properties of target compounds and nature of matrices.

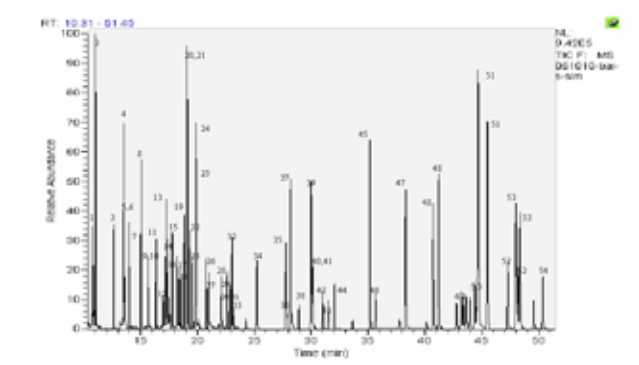


Figure 1. GC-EI-MS SIM spectrum of spiked cabbage sample

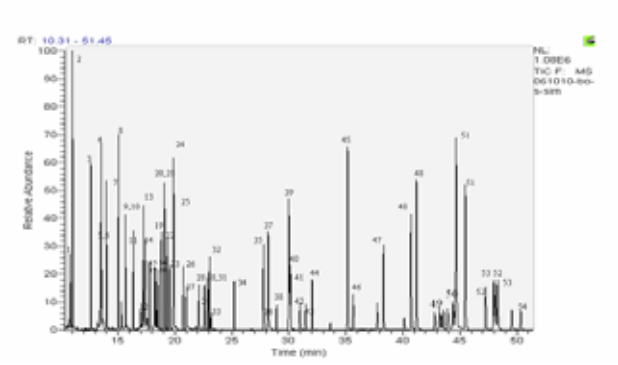


Figure 2. GC-EI-MS SIM spectrum of spiked spinach sample

The spiked concentration is the level 3 in table 4.

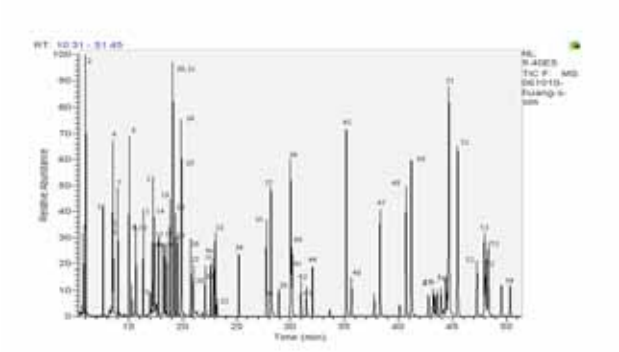


Figure 3. GC-EI-MS SIM spectrum of spiked yellow peach sample

Full length of this paper is available at www.bonnaagela.com: Imidacloprid, tebufenozide, avennectins and hexythiazox in vegetables by QuEChERS-GC.

Simultaneous QuEChERs-HPLC Analysis of Clopidol, Diclazuril and Sulfonamides in Animal Tissues (Cleanert® PSA, C18, Alumina-N, P/N: PA0010,180010, AL0010-N)

Material

PSA absorbent: Cleanert® PSA
C18 absorbent: Cleanert® C18
Neutral alumina absorbent: for chromatography (roast at 600°C in muffle furnace before use)

Experimental

Sample preparation with QuEChers method

Weigh 5 g (accurate to 0.01 g) of minced and mixed sample in a 50 mL centrifuge tube. Add 14 mL of acetonitrile/chloroform (10/1) solution, 1.0 mL of 10% sodium sulfate solution and homogenize. Centrifugate at 5000 r/min for 5 min. Take 10 mL of the extract precisely and concentrate or evaporate almost to dryness under nitrogen stream. Dilute with acetonitrile/chloroform (10/1) to 2 mL. Choose and add proper amount of C18, PSA or alumina absorbents. Vortex mix for 2 min to purify sample by dispersed solid phase extraction. Centrifugate at 5000 r/min for 3 min. Transfer 1.0 mL of the supernatant precisely into another 15 mL centrifuge tube. Dry under nitrogen stream and vortex dissolve the residue in 1.0 mL of acetonitrile/water (12/88). Filter through 0.45 μm membrane for analysis.

HPLC Conditions

Column: Venusil® ASB C18 (5 μm, 3.9 × 150 mm)
Mobile phase: A: acetonitrile; B: acetic acid/water (3/1000)
Gradient elution: see table 1
Flow rate: 1.0 mL/min; injection volume: 20 μL;
Column temperature: 40°C; UV: 270 nm.

Table 1 Program of gradient elution

Time/min	A %	B %	Gradient Curve
0.00	12	88	linear
8.50	12	88	linear
8.51	35	65	linear
15.00	35	65	linear
15.01	60	40	linear
20.00	60	40	linear
20.01	12	88	linear
25.00	12	88	linear

Results

(1) Linear range and limit of detection (LOD)
The limits of detection of different samples could not be exactly the same due to the difference of recovery even at the same spiked concentration. LOD of 7 compounds could reach: 0.05 mg/kg for clopidol, 0.05 mg/kg for sulfonamides, 0.10 mg/kg for diclazuril.
(2) Recovery and precision
Recovery and precision experiments are carried out with spiked chicken meat and liver samples at three concentration levels, respectively. For each level, experiments are repeated six times. At the concentrations between 0.10 mg/kg~1.0 mg/kg, the recovery and RSD are 65%~100% and 1%~10%, respectively. Chromatograms of spiked chicken meat and liver samples are shown as figure 1 and 2, respectively.

Discussion

The original QuEChERS method for pesticide residues in vegetables needs 15 g of sample and uses shaking for extraction. The extract is taken directly for next cleanup. The improved method for veterinary drug residues in animal tissues uses 5 g of sample and extraction by homogenization has better extraction efficiency. Take 10mL of the extract and evaporate almost to dryness by rotary evaporator. Dissolve the residue and dilute with extractive solvent to 2 mL for next cleanup. The improved method enriches more analytes than the original one. Therefore the sensitivity could be improved by 5 times theoretically.
PSA is used in the original QuEChERS method to remove fatty acids and pigments. As animal tissues have large amounts of proteins and fats, C18 and neutral alumina absorbent also need to be considered during the cleanup process. Sample cleanup is carried out by mixed dispersed solid phase extraction. The amount of different adsorbents, which usually is between 100 and 250 mg, is adjustable with different sample matrices. As for recovery, C18 and neutral alumina have strong adsorption towards diclazuril, thus reducing its recovery. But these adsorbents can hardly adsorb other 6 veterinary drugs. PSA could adsorb 7 compounds to different extents. In practice, it is best to choose appropriate absorbent and its amount according to the properties of matrix and the target compounds.

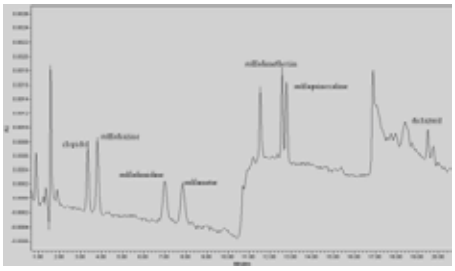


Figure 1. Chromatogram of spiked chicken meat sample

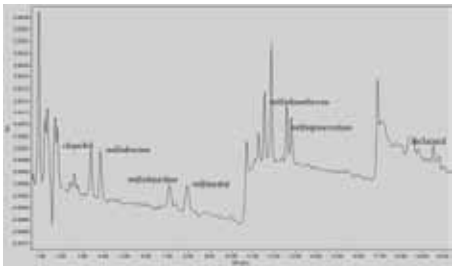


Figure 2. Chromatogram of spiked chicken liver sample

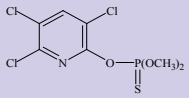
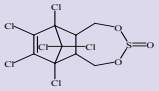
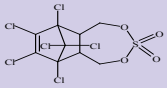
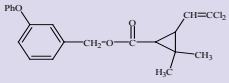
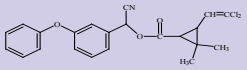
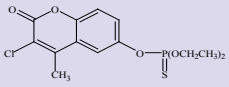
For more details please download the paper at www.bonnaagela.com

Determination of Pesticide Residues in Honey by Modified QuEChERS Extraction

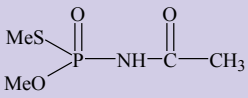
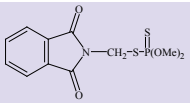
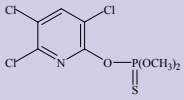
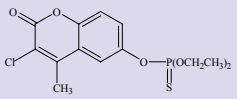
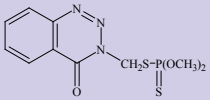
Dispersive sample preparation, referred as “QuEChERS”, is a sample and straightforward sample extraction technique. It can be used for multi-residue pesticide analysis in a wide variety of food and agricultural products.
Bonna-Agela’s MAS-Q is a well proven, high throughput method. After study of more than 60 of the fruit, vegetable samples (contain 27 of different sample matrix) and honey samples, the recoveries are very well proved for both Bonna-Agela’s high quality SPE sorbents and MAS-Q method.

Material

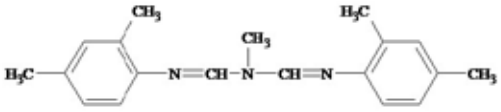
honey
Analytes: 10 pesticides

Organochlorines	Molecular Structure	Type
Chlorpyrifos Methyl		Insecticides
Endosulfans		Insecticides
Endosulfan sulfate		Insecticides
Permethrins		Insecticides (Pyrethroid)
Cypermethrins		Insecticides (Pyrethroid)
Coumaphos		Insecticides

Organophosphates

Acephate		Insecticides
Phosmet		Insecticides
Chlorpyrifos Methyl		Insecticides
Coumaphos		Insecticides
Azinphos Methyl		Insecticides

Organonitrogen Pesticide: Amitraz



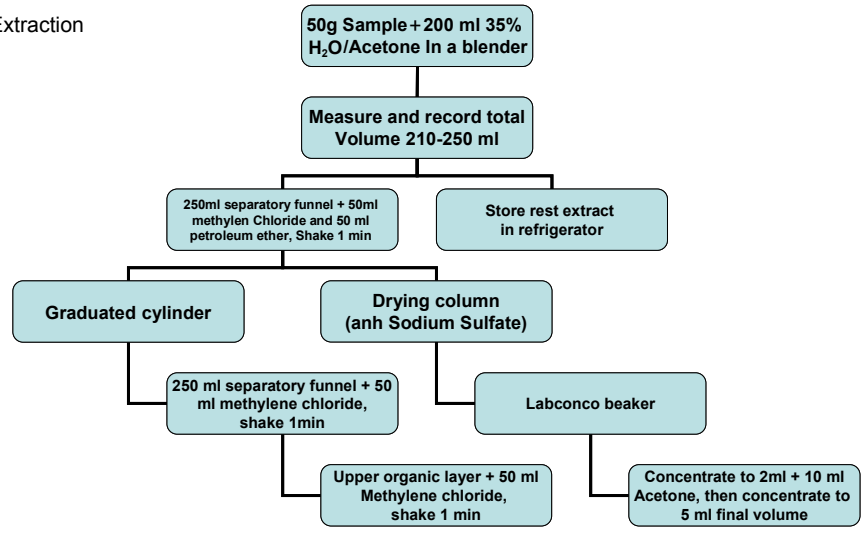
The New Challenge of LOD: Fluvalinate

Tolerance Level in Honey:

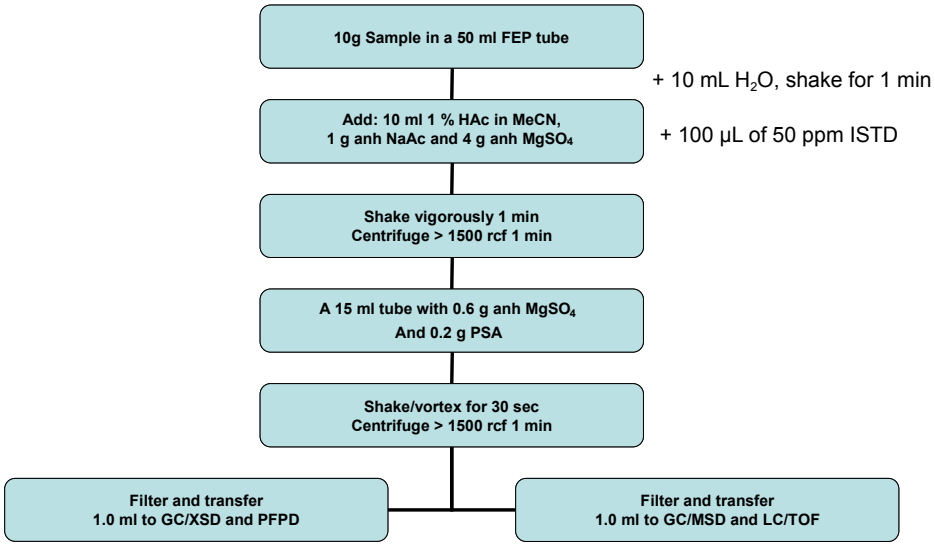
Fluvalinate	0.05 ppm
Coumaphos	0.1 ppm
Amitraz	1.0 ppm

Experimental

The Old Luke method for Honey Extraction

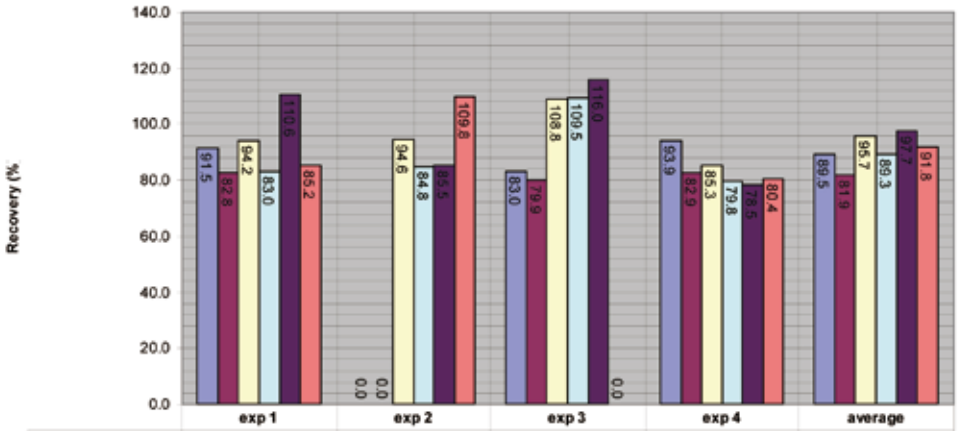


The new QuEChERS method for honey extraction



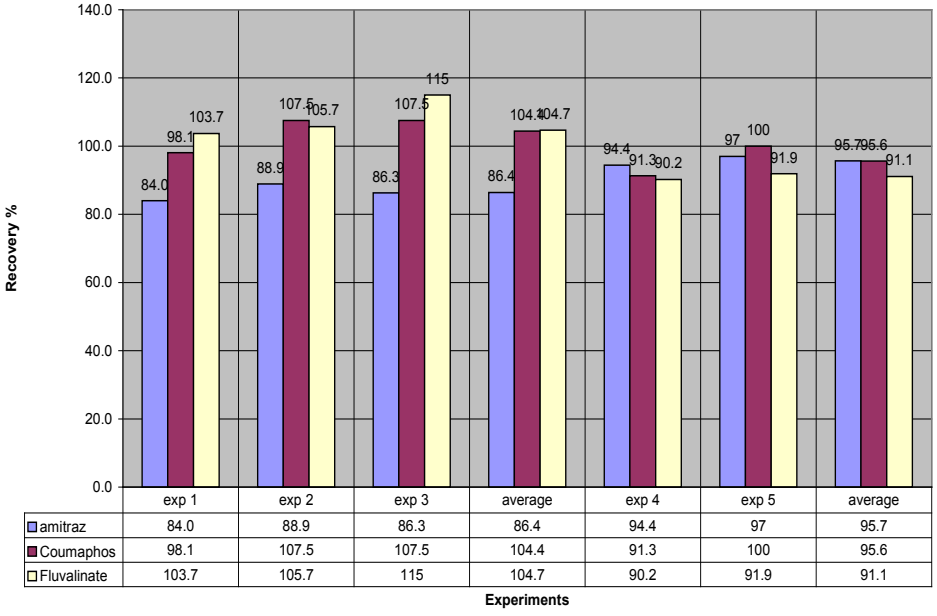
Results

Pesticide Recoveries by PFPD
Amitraz Recovery by MSD



	exp 1	exp 2	exp 3	exp 4	average
m-CPRF	91.5	0.0	83.0	93.9	89.5
endosulfans	82.8	0.0	79.9	82.9	81.9
endosulfan sulfate	94.2	94.6	108.8	85.3	95.7
permethrins	83.0	84.8	109.5	79.8	89.3
cypermethrins	110.6	85.5	116.0	78.5	97.7
coumaphos	85.2	109.8	0.0	80.4	91.8

LC/MS/TOF recovery



	exp 1	exp 2	exp 3	average	exp 4	exp 5	average
amitraz	84.0	88.9	86.3	86.4	94.4	97	95.7
Coumaphos	98.1	107.5	107.5	104.4	91.3	100	95.6
Fluvalinate	103.7	105.7	115	104.7	90.2	91.9	91.1

Conclusion

Method	Sample (gram)	Solvent (ml)	Experiment time	Equipment	Toxicity	Others
Old Luke	50	400	4 hrs	250 ml Glass separate funnels, Drying columns	Methylene Chloride	A lot of Dish washings And Waste Most tubes
QuEChERS	10	10	1 hr	Tubes	MeCN	disposable, and less waste Environment
Advantages	Save 80% sample	Save 97% solvent	Save 75% time	Cost Cut	Less toxic	Friendly

8. Analysis of Banned Azo Dyes in Textiles

Banned azo dyes in textiles (Cleanert SLE Azo dyes Extraction Column, P/N: GB/T17592-2006)

Reduce the textiles in citrate buffer solution by sodiumdithionate to obtain forbidden aromatic amines that possibly exist. Extract the aromatic amines by proper liquild-liquid partition cartridge. After concentration, dilute to volume with proper organic solvent for determination by GC-MS. If necessary, choose one or more other methods to confirm the existence of isomers. HPLC/DAD or GC/MS is employed for quantification.

Materials

(1) Cleanert® SLE extraction cartridge
20 cm × 2.5 cm (i.d.) polypropylene cartridge, packed with 20 g of diatomite.
(2) Citrate buffer (0.06 mol/L, pH = 6.0)
Dissolve 12.526 g of citric acid and 6.320 g of sodium hydroxide in water and dilute to 1000 mL.
(3) Sodiumdithionate solution
200 mg/mL sodiumdithionate in water, fresh prepared with solid sodiumdithionate ($Na_2S_2O_4$≥85%) before use.

Experimental

Sample Preparation

Cut representative sample into small pieces of 5 mm × 5 mm and mix. Transfer 1.0 (accurate to 0.01 g) of sample into reactor and add 16 mL of citrate buffer at 70±2°C. Seal the reactor and shake up until all samples are soaked in liquid. Put the reactor in water bath at 70±2°C for 30 min to soak the textiles thoroughly. Add 3.0 mL of sodiumdithionate solution, seal and shake up. After another 30 min in water bath, cool the reactor to room temperature in 2 min.

Extraction and Concentration

(1) Extraction:
Press the sample with a glass rod into the reactor and transfer the liquid into diatomite extraction cartridge. Allow to adsorb for 15 min. Elute the cartridge with ether four times (20 mL × 4). For each time, combine the ether and eluate, and load onto the cartridge. Control the flow rate. Collect the eluate in a round-bottom flask.
(2) Concentration:
Evaporate the eluate to 1 mL by rotary evaporator at 35°C and dry under a slow stream of nitrogen.

GC-MS Analysis

Capillary column: DA-5MS, 30 m × 0.25 mm × 0.25 μm (P/N: 1525-3002), or a corresponsive one.
Injection temperature: 250°C
Column temperature: 50°C (0.5 min) 20°C/min 150°C (8 min) 20°C/min 230°C (20 min) 20°C/ min 260°C (5 min)
MS interface temperature: 270°C; MS scan range: 35~350 amu; Injection mode: splitless; Carrier gas: He(≥99.999%); Flow rate: 1.0 mL/min; Injection volume: 1 μL; Ionization source: EI; Ionization voltage: 70 eV

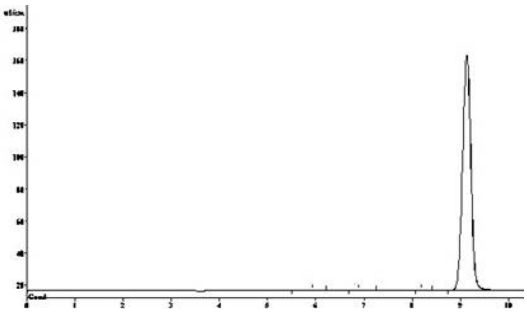
9. Removal of Interfering Ions and Organic Impurities

Nitrites in Food (Cleanert® IC-Ag and Na, P/N: IC-Ag10, IC-Na10)

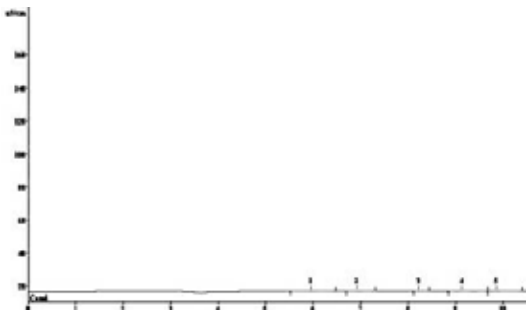
To monitor nitrites in food, ion chromatography is gradually replacing diazonium-coupled spectrophotometric analysis for its convenience and accuracy. During sample preparation process, impurities in extracted water solution such as particles, organic interference and cloride ions must be removed before analysis.

First particles are eliminated by MCM syringe filters. And then, Cleanert® IC-RP cartridge is employed to remove organic compounds in samples to avoid contamination of ion chromatography cartridge; Cleanert® IC-Ag and Na cartridges are combined to remove Cl^-, which can affect the peak shape of NO^{3-}.

The comparison of chromatograms before and after sample pretreatment is shown in the following figures to demonstrate the elimination effect of $C^{l-}$by Cleanert IC-Ag and Na cartridge.



Chromatogram of untreated sample



Chromatogram of sample treated by Cleanert® IC-Ag and Na cartridge

Cleanup of Water Samples from Oilfield Using Cleanert IC Cartridges (Cleanert® IC-RP, P/N: IC-RP10)

During oil exploration process, ion content in oil field water of different drilling depth must be monitored. MCM syringe filter can effectively trap unwanted particles in the water supernatant after centrifugation, and then Cleanert® IC-RP is followed to remove organic contaminants from oilfield.

SPE cartridges from Bonna-Agela can be easily used for batch sampling in a SPE manifold or workstation even though manual sampling might be still needed for random sampling.

Agela Technologies

Bonna-Agela Technologies
2038A Telegraph Rd.
Wilmington, DE 19808, USA
Tel: (302) 438 8798
Fax: (302) 636 9339
E-mail: info@bonnaagela.com

Bonna-Agela India
#212, 2nd Floor, Sector-63, Noida, Uttar Pradesh
Tel: 91120-4225466/67
Fax: 91120-4225465

Bonna-Agela China
179 South Street, Teda West Zone, Tianjin 300462, China
Tel: +86 (22) 25321032/7023
Fax: +86 (22) 25321033

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