

NEW Content See Inside))

Agilent Bond Elut QuEChERS Food Safety
Applications Notebook: Volume 2

PROVEN APPROACHES FOR TODAY'S FOOD ANALYSIS CHALLENGES

The Measure of Confidence



Agilent Technologies

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Food Safety Overview

Reliable food safety testing begins with reliable sample preparation

Dear Valued Customer,

Today's consumers demand foods and beverages that are safe, high-quality, and nutritious. Your food laboratory's work lays the foundation for meeting that demand.

Agilent's comprehensive analytical solutions deliver on that promise

From inspection and product development to quality assurance and packaging, Agilent instruments, systems, columns, and supplies help your lab meet the toughest standards. In-depth experience, broad knowledge, and creative people, along with our insight into industry trends and global regulations, address your challenges.

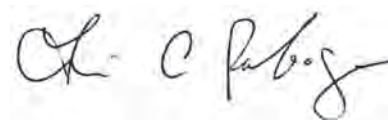
Agilent Bond Elut QuEChERS and SPE Sample Preparation products: your first step in food safety analysis

Agilent Bond Elut Sample Preparation products help you confidently extract and concentrate samples from complex matrixes, to deliver fast, accurate, and reproducible results.

The Bond Elut family of products supports your food sample preparation needs with a broad set of formats and products manufactured to strict quality standards:

- **Bond Elut QuEChERS Pre-Packed Extraction and Dispersive SPE Kits** enable preparation of food samples for multi-residue, multi-class organic contaminant analysis in just a few simple steps, with consistent quality and high performance built into the kits.
- **The Bond Elut SPE product range**, comprising over 40 different polymeric and silica-based functionalities, comes in a variety of cartridge and plate formats.
- **Bulk sorbents** support method development and lab-level customization of QuEChERS sample prep approaches.

Our team of scientists continues to support your food testing needs, so be sure to check our QuEChERS page for new applications and product developments at agilent.com/chem/QuEChERS. Accuracy starts here.



Trisa Robarge
Sample Preparation Product Manager



What is QuEChERS?

Developed by United States Department of Agriculture in 2003, QuEChERS (pronounced “catchers”) stands for **Q**uick, **E**asy, **C**heap, **E**ffective, **R**ugged and **S**afe – the qualities that describe this sample preparation method for food substances. The technique is very simple, involves a minimum of steps, and is effective for the cleanup of complex samples.

QuEChERS is a technique that was developed for multi-class, multi-residue pesticides analysis in fruits and vegetables, but more recently has expanded its scope to other trace contaminants in other non-vegetable foods such as meat and fish. Methods for hundreds of pesticides in a variety of fruits, vegetables, meat, and for dry materials such as beans and nuts have been published. “Official” methods are now available and a standardization of the technique on a worldwide basis is taking place. In the United States, the Association of Official Analytical Chemists (AOAC) has published its 2007.01 Method while the European equivalent, the EN 15662 2007, uses similar methodology.

The practice of QuEChERS involves two steps:

1. An extraction step that is based on partitioning via salting-out extraction involving an equilibrium between an aqueous and an organic layer, and
2. A dispersive solid-phase extraction (SPE) step that involves further cleanup using various combinations of salts and porous sorbents to remove interfering substances.

In the dispersive SPE step, the use of porous sorbents such as primary-secondary amine (PSA), C18, and graphitized carbon black help to remove a variety of matrix compounds that are co-extracted in Step 1. The most popular analytical methodology to measure extracted analytes is either LC/MS or GC/MS or their tandem equivalents.

Tips and Tricks

To support the wide range of QuEChERS product options, we offer a number of applications featuring the QuEChERS approach. This guide includes many of these applications. You’ll find the applications grouped according to the standard method associated with the application, such as AOAC, EN, or the original method, along with a section for other approaches. A handy index also lets you search this guide based on matrix type and analyte class.



QuEChERS Extraction Kits

Step 1: Extraction

Choose the extraction salt packet based on your method of analysis, AOAC or EN. The buffered extraction salts are amenable for more labile pesticides. Adding solvent and then salts to a homogenized fruit or vegetable sample (10 g or 15 g) enables you to extract the pesticides of interest into the organic layer. Agilent pre-packages its QuEChERS salts and buffers in anhydrous packages. This allows you to add them after adding your solvent to the sample, as specified in QuEChERS methodologies.

In the table below, the “CH” products contain the appropriately-sized ceramic homogenizers for those particular kits. For more information on ceramic homogenizers see page 13.



QuEChERS extraction kits are available with or without 50 mL centrifuge tubes and caps and includes $MgSO_4$, NaCl, or other salts for buffering; pre-weighed in anhydrous packets.



Ceramic homogenizers cut extraction time by up to 70%. They are available with QuEChERS kits or separately.

QuEChERS Extraction Kits

| Method | Buffered | Contents | Ceramic Homogenizers | with 50 mL Tubes 50 / pk | Packets Only | |
|----------------------------|----------|---|----------------------|-----------------------------|--------------|-----------|
| | | | | | 50 / pk | 200 / pk |
| AOAC 2007.01 | Yes | 6 g $MgSO_4$; 1.5 g NaAcetate | Yes | 5982-5755CH | | |
| | | | No | 5982-5755 | 5982-6755 | 5982-7755 |
| Original (10 g samples) | No | 4 g $MgSO_4$; 1 g NaCl | Yes | 5982-5550CH | | |
| | | | No | 5982-5550 | 5982-6550 | 5982-7550 |
| Original (15 g samples) | No | 6 g $MgSO_4$; 1.5 g NaCl | Yes | 5982-5555CH | | |
| | | | No | 5982-5555 | 5982-6555 | 5982-7555 |
| EN 15662 | Yes | 4 g $MgSO_4$; 1 g NaCl; 1 g NaCitrate; 0.5 g disodium citrate sesquihydrate | Yes | 5982-5650CH | | |
| | | | No | 5982-5650 | 5982-6650 | 5982-7650 |
| Acrylamides* | No | 4 g $MgSO_4$; 0.5 g NaCl | No | 5982-5850 | | |
| Veterinary Drugs | No | 4g Na_2SO_4 , 1 g NaCl | No | 5982-0032 | | |

* Procedure from K. Mastovska and S. J. Lehotay, J. Agric. Food Chem. 2006, 54, 7001-7008.

QuEChERS Dispersive Kits

Step 2: Dispersive SPE Cleanup

Select the Dispersive SPE kit suited to the type of food being analyzed and the method you are following. In this step, an aliquot of the sample extract from Step One is added to a 2 mL or 15 mL centrifuge tube containing a small amount of SPE sorbent and MgSO₄. The sorbent will pull out interfering matrix materials from the sample, while the MgSO₄ helps remove excess water and improve analyte partitioning. Select kits are now available with ceramic homogenizers (two per tube). Their part numbers are designated by a CH.



QuEChERS Dispersive Kits, Fruits and Vegetables

| Kit | Size | Unit | AOAC 2007.01 Method | European Method |
|--|-------|--------|---|--|
| | | | Kit Contents Part No. | EN 15662 Kit Contents Part No. |
| General fruits and vegetables: Removes polar organic acids, some sugars and lipids | 2 mL | 100/pk | 50 mg PSA 150 mg MgSO ₄ 5982-5022 5982-5022CH | 25 mg PSA 150 mg MgSO ₄ 5982-5021 5982-5021CH |
| | 15 mL | 50/pk | 400 mg PSA 1200 mg MgSO ₄ 5982-5058 5982-5058CH | 150 mg PSA 900 mg MgSO ₄ 5982-5056 5982-5056CH |
| Fruits and vegetables with fats and waxes: Removes polar organic acids, some sugars, more lipids and sterols | 2 mL | 100/pk | 50 mg PSA 50 mg C18EC 150 mg MgSO ₄ 5982-5122 5982-5122CH | 25 mg PSA 25 mg C18EC 150 mg MgSO ₄ 5982-5121 5982-5121CH |
| | 15 mL | 50/pk | 400 mg PSA 400 mg C18EC 1200 mg MgSO ₄ 5982-5158 5982-5158CH | 150 mg PSA 150 mg C18EC 900 mg MgSO ₄ 5982-5156 5982-5156CH |

Part numbers ending in CH indicate tubes containing ceramic homogenizers.

(Continued)



QuEChERS Dispersive Kits, Fruits and Vegetables

| Kit | Size | Unit | AOAC 2007.01 Method | European Method EN 15662 |
|--|-------|--------|---|---|
| | | | Kit Contents Part No. | Kit Contents Part No. |
| Pigmented fruits and vegetables: Removes polar organic acids, some sugars and lipids, and carotenoids and chlorophyll; not for use with planar pesticides | 2 mL | 100/pk | 50 mg PSA 50 mg GCB 150 mg MgSO ₄ 5982-5222 5982-5222CH | 25 mg PSA 2.5 mg GCB 150 mg MgSO ₄ 5982-5221 5982-5221CH |
| | 15 mL | 50/pk | 400 mg PSA 400 mg GCB 1200 mg MgSO ₄ 5982-5258 5982-5258CH | 150 mg PSA 15 mg GCB 885 mg MgSO ₄ 5982-5256 5982-5256CH |
| Highly pigmented fruits and vegetables: Removes polar organic acids, some sugars and lipids, plus high levels of carotenoids and chlorophyll; not for use with planar pesticides | 2 mL | 100/pk | | 25 mg PSA 7.5 mg GCB 150 mg MgSO ₄ 5982-5321 5982-5321CH |
| | 15 mL | 50/pk | | 150 mg PSA 45 mg GCB 855 mg MgSO ₄ 5982-5356 5982-5356CH |
| Fruits and vegetables with pigments and fats: Removes polar organic acids, some sugars and lipids, plus carotenoids and chlorophyll; not for use with planar pesticides | 2 mL | 100/pk | 50 mg PSA 50 mg GCB 150 mg MgSO ₄ 50 mg C18EC 5982-5421 5982-5421CH | |
| | 15 mL | 50/pk | 400 mg PSA 400 mg GCB 1200 mg MgSO ₄ 400 mg C18EC 5982-5456 5982-5456CH | |

Part numbers ending in CH indicate tubes containing ceramic homogenizers.






QuEChERS Dispersive Kits: Other Food Methods

| Kit | Size | Unit | AOAC 007.01 Method | European Method EN 15662 |
|---|-------|--------|--|--------------------------|
| | | | Kit Contents Part No. | Kit Contents Part No. |
| Other Food Methods: Removes biological matrix interferences, including hydrophobic substances (fats, lipids) and proteins | 2 mL | 100/pk | 25 mg C18 150 mg MgSO ₄ 5982-4921 5982-4921CH | |
| | 15 mL | 50/pk | 150 mg C18 900 mg MgSO ₄ 5982-4956 5982-4956CH | |
| All Food Types: Removes all matrix interfering materials including polar organic acids, lipids, sugars, proteins, carotenoids and chlorophyll | 2 mL | 100/pk | 50 mg PSA 50 mg C18 7.5 mg GCB 150 mg MgSO ₄ 5982-0028 5982-0028CH | |
| | 15 mL | 50/pk | 400 mg PSA 400 mg C18 45 mg GCB 1200 MgSO ₄ 5982-0029 5982-0029CH | |
| Animal Origin Food: Removes matrix interferences such as polar organic salts, sugars, lipids and proteins | 15 mL | 50/pk | 50 mg PSA 150 mg C18EC 900 mg Na ₂ SO ₄ 5982-4950 | |

Part numbers ending in CH indicate tubes containing ceramic homogenizers.










Suggested Bond Elut QuEChERS Dispersive Kit by Food Type and Method

| Group commodity | Commodity | General fruits and vegetables; EN or AOAC | Fruits and vegetables with fats and waxes; EN or AOAC | Pigmented fruits and vegetables; EN or AOAC | Highly pigmented fruits and vegetables; EN | Fruits and vegetables with pigment and fats; AOAC only |
|--|-----------------|---|---|--|---|--|
| Use with | | Lightly colored samples | Samples containing > 1% fat/lipids | Colored samples (chlorophyll, carotenoids), no planar pesticides | Highly colored samples (chlorophyll, carotenoids), no planar pesticides | Colored samples that also contain fats or waxes |
| Fruits | | | | | | |
|  <p>Citrus fruits</p> | citrus juices | | | | | |
| | grapefruit | | | | | |
| | lemon/lime | | | | | |
| | orange | | | | | |
| | orange peel | | | | | |
| | nectarine | | | | | |
| | tangerine | | | | | |
|  <p>Pome fruits</p> | apple | | | | | |
| | apple, dried | | | | | |
| | apple juice | | | | | |
| | apple sauce | | | | | |
| | pear | | | | | |
| | quince | | | | | |
|  <p>Stone fruits</p> | apricot | | | | | |
| | apricot, dried | | | | | |
| | apricot nectar | | | | | |
| | cherry | | | | | |
| | mirabelle | | | | | |
| | nectarine | | | | | |
| | peach | | | | | |
| | peach, dried | | | | | |
| | plum | | | | | |
| | plum, dried | | | | | |
|  <p>Soft and small fruits</p> | blackberry | | | | | |
| | blueberry | | | | | |
| | cranberry | | | | | |
| | currant | | | | | |
| | elderberry | | | | | |
| | gooseberry, red | | | | | |
| | grapes, green | | | | | |
| | grapes, red | | | | | |
| | raisin | | | | | |
| | raspberry | | | | | |
| | strawberry | | | | | |
|  <p>Other fruits</p> | avocado | | | | | |
| | banana | | | | | |
| | fig, dried | | | | | |
| | kiwi | | | | | |
| | mango | | | | | |
| | melon | | | | | |
| | olives | | | | | |
| | papaya | | | | | |
| | pineapple | | | | | |












(Continued)

Suggested Bond Elut QuEChERS Dispersive Kit by Food Type and Method

| Group commodity | Commodity | General fruits and vegetables; EN or AOAC | Fruits and vegetables with fats and waxes; EN or AOAC | Pigmented fruits and vegetables; EN or AOAC | Highly pigmented fruits and vegetables; EN | Fruits and vegetables with pigment and fats; AOAC only |
|---|-------------------------------|---|---|--|---|--|
| Use with | | Lightly colored samples | Samples containing > 1% fat/lipids | Colored samples (chlorophyll, carotenoids), no planar pesticides | Highly colored samples (chlorophyll, carotenoids), no planar pesticides | Colored samples that also contain fats or waxes |
| Vegetables | | | | | | |
|  Root and tuber vegetables | beets | | | | | |
| | black salsify | | | | | |
| | carrot | | | | | |
| | celeriac | | | | | |
| | horseradish | | | | | |
| | parsley root | | | | | |
| | potato | | | | | |
| | radish | | | | | |
|  Leek plants | chive | | | | | |
| | garlic | | | | | |
| | leek | | | | | |
| | onion | | | | | |
| | scallion | | | | | |
| | shallot | | | | | |
|  Fruiting vegetables | aubergine/eggplant | | | | | |
| | cucumber | | | | | |
| | pepper, sweet green | | | | | |
| | pepper, sweet red | | | | | |
| | pumpkin | | | | | |
| | tomato | | | | | |
| | zucchini (courgette) | | | | | |
|  Broccoli | broccoli | | | | | |
| | brussel sprouts | | | | | |
| | cabbage, chinese | | | | | |
| | cabbage, red | | | | | |
| | cabbage, savoy | | | | | |
| | cabbage, white | | | | | |
| | cauliflower | | | | | |
| | kale | | | | | |
| | kohlrabi | | | | | |
| | | | | | | |
|  Leafy vegetables and herbs | arugula, rucola | | | | | |
| | basil | | | | | |
| | cilantro | | | | | |
| | cress | | | | | |
| | endive | | | | | |
| | lamb's lettuce | | | | | |
| | lettuce varieties | | | | | |
| | parsley | | | | | |
| | spinach | | | | | |
| | | | | | | |
|  Stem vegetables | artichokes | | | | | |
| | asparagus | | | | | |
| | celery | | | | | |
| | leek | | | | | |
| | rhubarb | | | | | |
|  Legumes | beans, lentils, peas, (dried) | | | | | |
| | beans, lentils, peas, (fresh) | | | | | |

(Continued)

Suggested Bond Elut QuEChERS Dispersive Kit by Food Type and Method

| Group commodity | Commodity | General fruits and vegetables; EN or AOAC | Fruits and vegetables with fats and waxes; EN or AOAC | Pigmented fruits and vegetables; EN or AOAC | Highly pigmented fruits and vegetables; EN | Fruits and vegetables with pigment and fats; AOAC only |
|--|---------------------------|---|---|--|---|--|
| Use with | | Lightly colored samples | Samples containing > 1% fat/lipids | Colored samples (chlorophyll, carotinoids), no planar pesticides | Highly colored samples (chlorophyll, carotinoids), no planar pesticides | Colored samples that also contain fats or waxes |
| Animal-Sourced Foods | | | | | | |
|  Meats | beef, chicken, pork, veal | | | | | |
| | kidney, liver | | | | | |
|  Seafood | bivalve, shellfish | | | | | |
| | finfish | | | | | |
|  Dairy | dairy | | | | | |
| Other Foods | | | | | | |
|  Cereals | corn, rice, wheat | | | | | |
| | flour, grain, etc. | | | | | |
|  Tea/coffee | coffee beans | | | | | |
| | tea leaves | | | | | |
|  Dried spices | curry, peppers | | | | | |
| | leek plants | | | | | |
| | peppercorn seeds | | | | | |
|  Oils | canola, olive | | | | | |
| | citrus | | | | | |
|  Baby food | baby food | | | | | |
| Other | | | | | | |
|  Agricultural products | cocoa solids | | | | | |
| | cotton, hemp | | | | | |
| | tobacco | | | | | |
|  Soil | soil | | | | | |
|  Whole blood | whole blood | | | | | |

QuEChERS Ceramic Homogenizers

Ceramic homogenizers increase your overall lab productivity and give you greater confidence in your results. Make analyte extraction easier with these benefits:

- Cut the required extraction time from 60 seconds to as little as 20 seconds – a time savings of 70% per sample
- Maintain high, reproducible extractions in one-third of the time
- Minimize variation between technicians
- Break up salt agglomerates and maintain a consistent grinding of homogenized material

The same ceramic homogenizers available in our QuEChERS kits are also available for bulk purchase, providing excellent grinding capabilities of your samples.

QuEChERS ceramic homogenizers

| Description | Unit | Part No. |
|-------------------------------------|--------|-----------|
| Ceramic homogenizer for 50 mL tubes | 100/pk | 5982-9313 |
| Ceramic homogenizer for 15 mL tubes | 100/pk | 5982-9312 |
| Ceramic homogenizer for 2 mL tubes | 200/pk | 5982-9311 |



Standards for QuEChERS Products

In addition to our industry-leading QuEChERS kits, Agilent makes your analysis easier by providing standards for the most commonly used regulatory methods, including AOAC and EN.

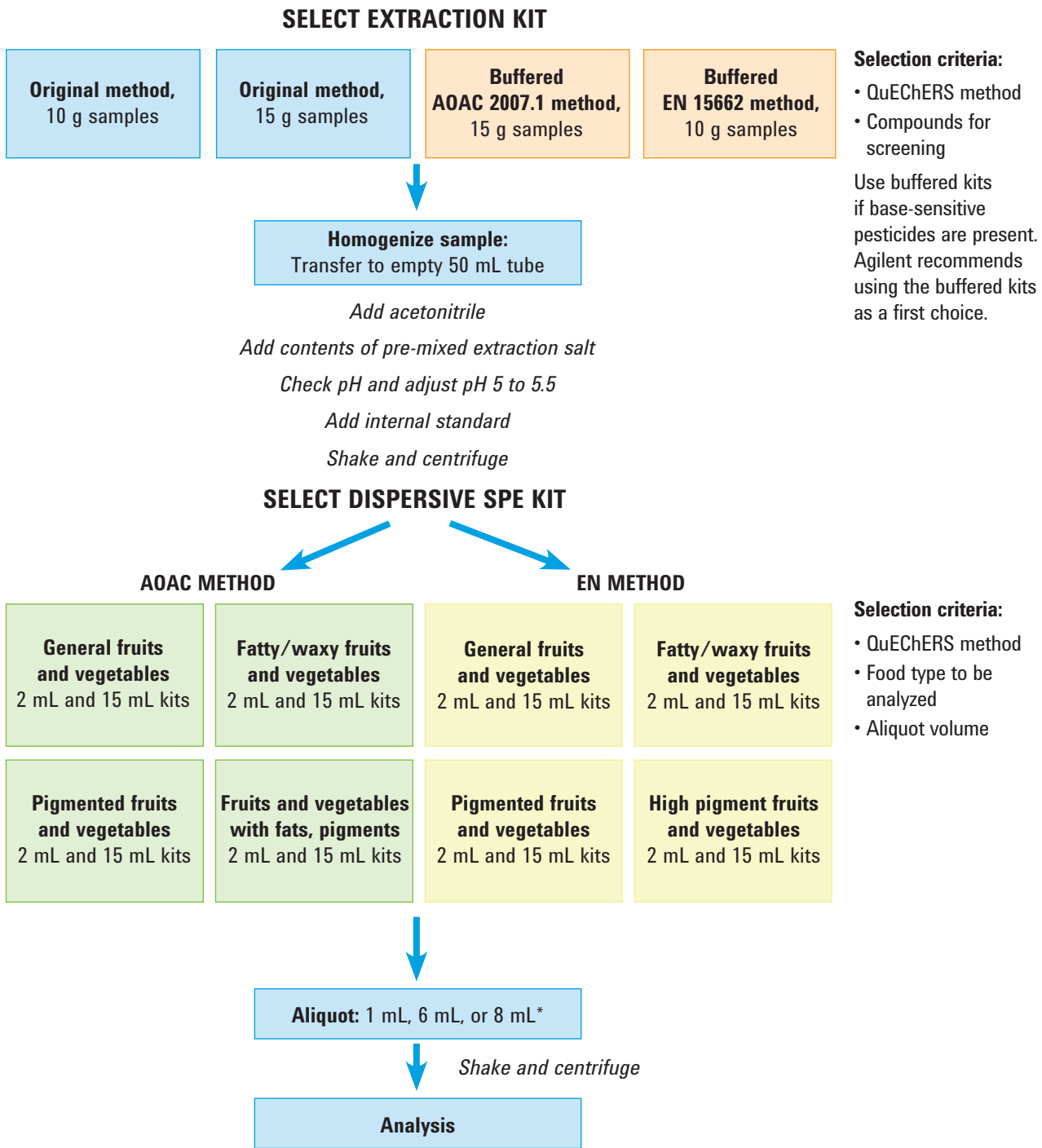
- Save time and avoid inconvenience of making standards
- Available for GC and LC instruments
- Ready to use for QuEChERS extractions – no dilutions required

QuEChERS Standards

| Description | Concentration | Kit Contents | Part No. |
|--|--------------------------|---|-----------|
| HPLC and GC Internal Standard, AOAC Method | 1,000 µg/mL | Parathion-d10 (diethyl-d10), alpha-BHC-d6 (alpha-HCH-d6) | 5190-0502 |
| QC Solution, AOAC Method | 500 µg/mL | Triphenyl phosphate | 5190-0503 |
| HPLC Internal Standard, EN Method | 100 µg/mL | Tris (1,3-dichloroisopropyl) phosphate, nicarbazin | 5190-0500 |
| GC Internal Standard, EN Method | 5,000 µg/mL | (2,2',5,5'-tetrachlorobiphenyl), triphenylmethane, tris (1,3-dichloroisopropyl) phosphate | 5190-0501 |
| QC Surrogate for GC Standard, EN Method | 500 µg/mL 1,000 µg/mL | (2,2',3,4,4',5'-hexachlorobiphenyl) Anthracene-d10 | 5190-0499 |

Agilent Bond Elut Recommended Standard Operating Procedure for QuEChERS

Here are the basic steps to complete the QuEChERS method on any fruit or vegetable sample. In just a few easy steps, you'll prepare your sample for multi-class, multi-residue pesticide analysis.



*Aliquot size is specified by the method, and kits are created for these specific amounts. For pesticides with acidic groups (phenoxycarboxylic acids), analyze directly by LC/MS/MS at this point (skip the dispersive SPE stage). These acidic groups interact with the PSA that is part of the dispersive SPE step.

Original QuEChERS Method

Analysis of Pesticide Residues in Apple by GC/MS using Agilent Bond Elut QuEChERS Kits for Pre-injection Cleanup (Publication 5990-4468EN)

Introduction

This application describes the use of QuEChERS, an effective sample preparation approach to investigate the extraction of 15 multi-class pesticides in apples. The pesticides were chosen to represent typical types of volatile/semi-volatile pesticides that might be found in a typical fruit sample at levels normally encountered. The version of the QuEChERS non-buffered extraction method dates back to the original publication in 2003. For analysis, it uses GC/MS with selective ion monitoring (SIM) to measure pesticides down to the 10 ng/g levels.



Instrument conditions

GC/MS conditions

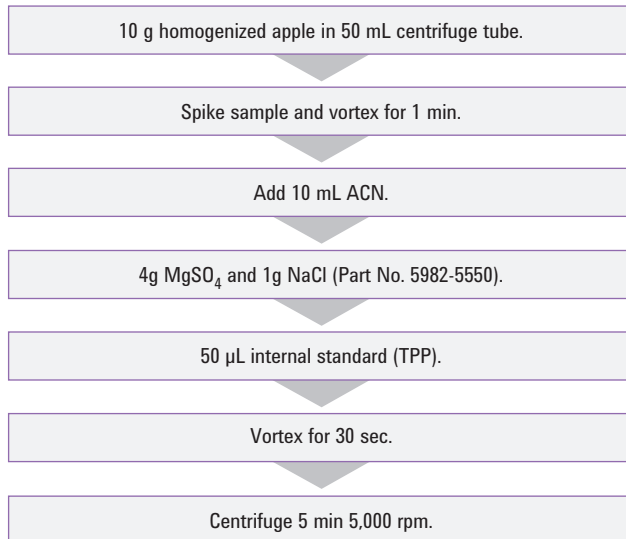
| | |
|---------------------------|--|
| Column: | Agilent J&W HP-5ms Ultra Inert, 30 m x 0.25 mm, 0.25 µm (Part No. 190915-433UI) |
| Injection source: | Manual |
| Inlet: | Splitless |
| Carrier gas: | Helium in constant flow mode |
| Oven temperature program: | 70 °C (2 min), 25 °C/min to 150 °C (0 min), 3 °C/min to 200 °C (0 min), 8 °C/min to 280 °C (7 min) |
| Injection volume: | 1 µL |

MS conditions

| | |
|---|--------------------------------------|
| Tune file: | Atune.u |
| Mode: | SIM |
| Source, quad, transfer line temperature: | 230 °C, 150 °C, 280 °C, respectively |
| Solvent delay: | 4 min |
| Multiplier voltage: | Autotune voltage |

QuEChERS Procedure

QuEChERS Extraction Procedure (Step 1)



QuEChERS Dispersive SPE (Step 2) and Analysis

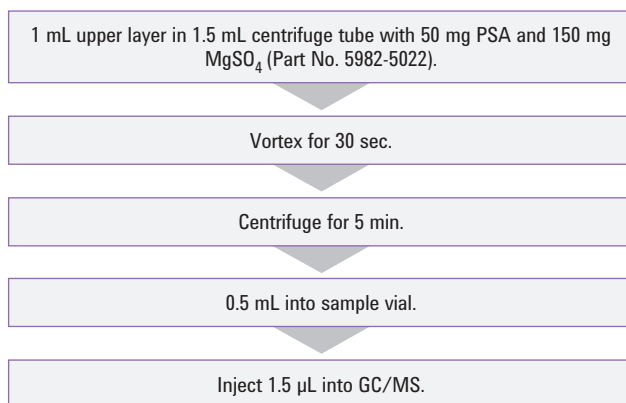


Figure 1. QuEChERS extraction procedure for general fruits and vegetables.

Products used in the above application

Agilent Bond Elut QuEChERS Non-Buffered Extraction Kit. Part No. 5982-5550.

Agilent Bond Elut QuEChERS Dispersive Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5022.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 15 mL. Part No. 5982-5058.

Agilent J&W HP-5ms Ultra Inert GC Capillary Column, 30 m x 0.25 mm, 0.25 µm. Part No. 190915-433UI.

Results

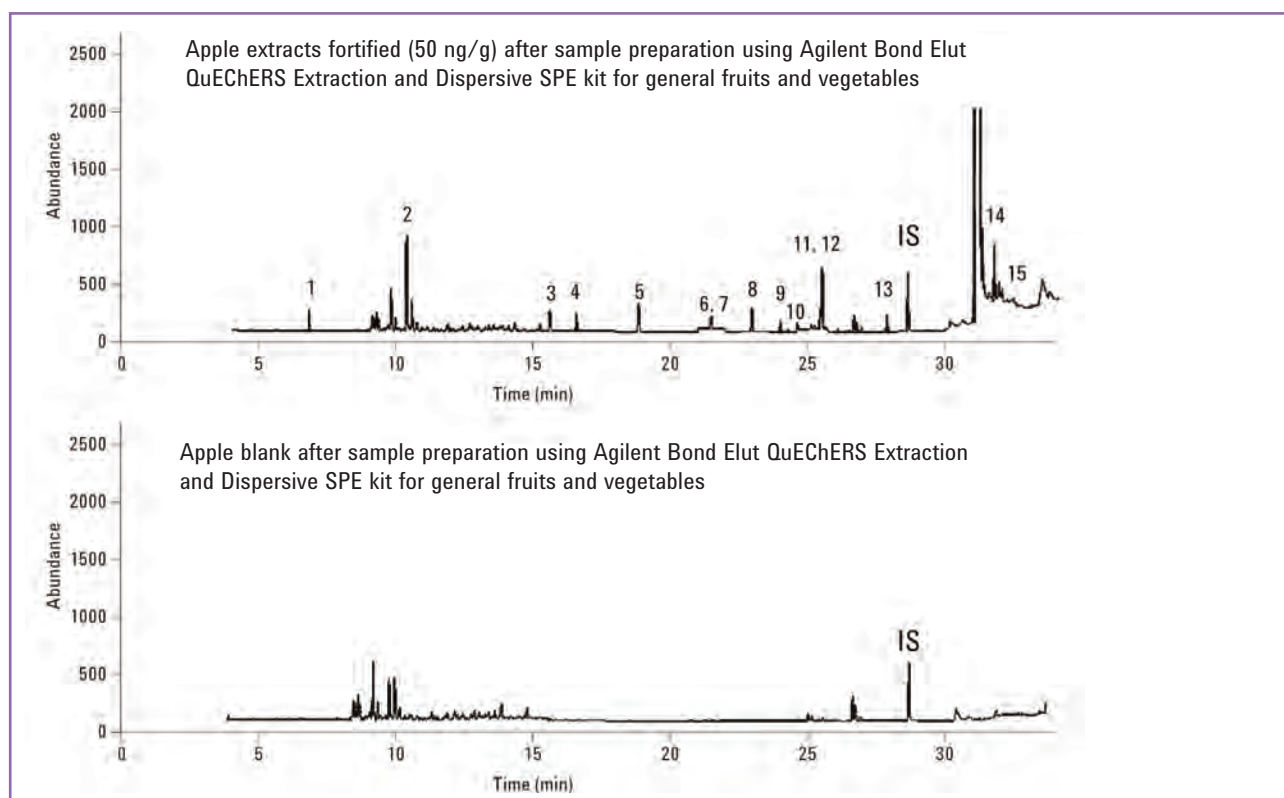


Figure 2. Comparison of blank apple extract to a fortified apple extract.

Table 1. Recovery and reproducibility of pesticides in apple using the original QuEChERS method (n=4)

| Pesticide | Low-QC 10 ng/g | | Mid-QC 50 ng/g | | High-QC 200 ng/g | |
|------------------------|----------------|-----|----------------|------|------------------|-----|
| | Recovery | RSD | Recovery | RSD | Recovery | RSD |
| Dichlorvos | 102.8 | 5.0 | 96.7 | 10.8 | 99.4 | 2.8 |
| <i>o</i> -Phenylphenol | 92.0 | 6.1 | 79.6 | 6.8 | 89.5 | 6.3 |
| Lindane | 97.9 | 2.0 | 88.5 | 9.7 | 92.6 | 4.2 |
| Diazinone | 90.5 | 9.1 | 98.8 | 5.5 | 102.1 | 4.4 |
| Methyl-chlorpyrifos | 88.7 | 7.1 | 90.0 | 4.3 | 98.5 | 3.1 |
| Chlorpyrifos | 93.5 | 6.5 | 95.6 | 4.0 | 100.2 | 1.2 |
| Dichlorobenzophenone | 90.3 | 5.0 | 89.1 | 6.4 | 99.4 | 0.6 |
| Heptachlor-epoxide | 87.0 | 3.2 | 85.6 | 5.4 | 95.4 | 3.9 |
| γ -Chlordane | 92.3 | 3.5 | 90.0 | 6.8 | 95.9 | 2.0 |
| α -Chlordane | 95.5 | 4.7 | 85.8 | 6.9 | 93.5 | 2.6 |
| Dieldrin | 99.4 | 4.2 | 93.6 | 5.3 | 99.9 | 1.8 |
| DDE | 94.5 | 4.2 | 87.1 | 5.7 | 92.7 | 1.9 |
| Endosulfan sulfate | 97.8 | 2.3 | 90.8 | 2.8 | 99.5 | 2.3 |
| Permethrin | 100.7 | 4.8 | 93.0 | 3.4 | 97.6 | 2.1 |
| Coumaphos | 72.5 | 4.5 | 79.6 | 3.5 | 96.6 | 3.0 |

To review this Application Note in its entirety, please search for 5990-4468EN at agilent.com/chem

EN Methods

Analysis of Pesticide Residues in Apple using Agilent Bond Elut QuEChERS European Standard EN Kits by LC/MS/MS Detection (Publication 5990-3938EN)

Introduction

This application describes the use of QuEChERS, an effective sample preparation approach described in the European Committee Standard (EN) for extraction and cleanup of 16 multiple class pesticide residues of interest in apple. The target pesticides in the apple extracts are then determined by liquid chromatography coupled to an electrospray ionization tandem mass spectrometer (LC-ESI-MS/MS) operating in positive ion multiple reaction monitoring (MRM) mode.



Instrument conditions

HPLC conditions

| | | | |
|---------------------|--|------|--------------------|
| Column: | Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl, 3.0 x 150 mm, 3.5 µm (Part No. 959963-312) | | |
| Flow rate: | 0.3 or 0.5 mL/min | | |
| Column temperature: | 30 °C | | |
| Injection volume: | 10 µL | | |
| Mobile phase: | A: 5 mM Ammonium acetate, pH 5.0 in 20:80 MeOH/H ₂ O B: 5 mM Ammonium acetate, pH 5.0 in ACN | | |
| Needle wash: | 1:1:1:1 ACN:MeOH:IPA:H ₂ O (0.2% FA.) | | |
| Gradient: | Time (min) | % B | Flow rate (mL/min) |
| | 0 | 20 | 0.3 |
| | 0.5 | 20 | 0.3 |
| | 8.0 | 100 | 0.3 |
| | 10.0 | 100 | 0.3 |
| | 10.01 | 20 | 0.5 |
| | 12.0 | 100 | 0.5 |
| | 13.0 | STOP | |
| Post run: | 4 min | | |
| Total cycle time: | 17 min | | |

MS conditions

Positive mode

| | |
|------------------|----------|
| Gas temperature: | 350 °C |
| Gas flow: | 10 L/min |
| Nebulizer: | 40 psi |
| Capillary: | 4,000 V |

QuEChERS Procedure

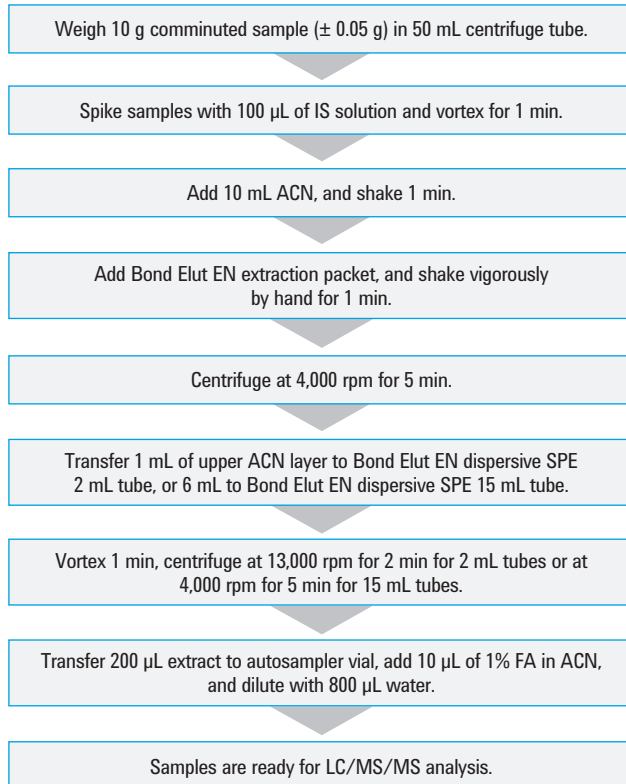


Figure 1. QuEChERS EN sample preparation procedure for pesticides in apple.

Products used in the above application

Agilent Bond Elut QuEChERS EN Method Extraction Kit. Part No. 5982-5650.

Agilent Bond Elut QuEChERS EN Dispersive SPE Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5021 or 15 mL. Part No. 5982-5056.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 µm. Part No. 959963-312.

Results

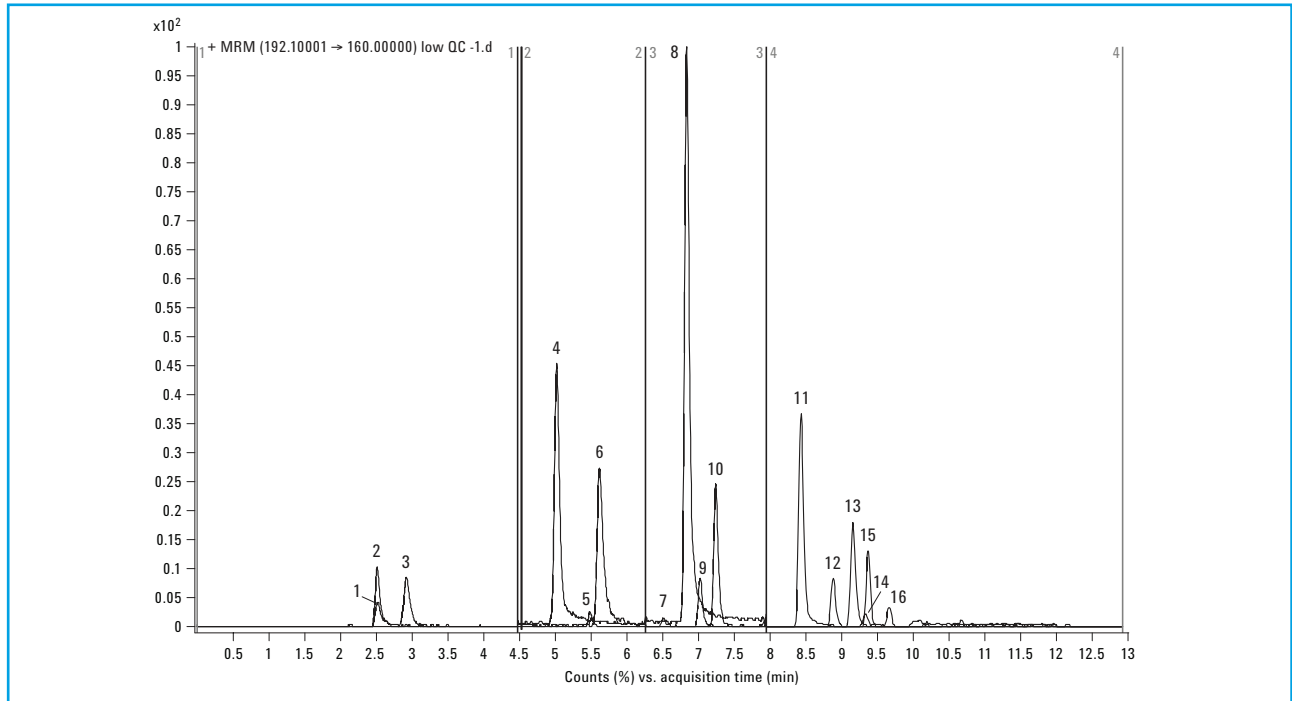


Figure 2. Chromatogram of 10 ng/g fortified apple extract. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Dichlorvos, 8. Propoxur, 9. Thiophanate methyl, 10. Carbaryl, 11. Ethoprophos, 12. Penconazole, 13. Cyprodinil, 14. Dichlorfluaniid, 15. Kresoxim methyl, 16. Tolyfluaniid.

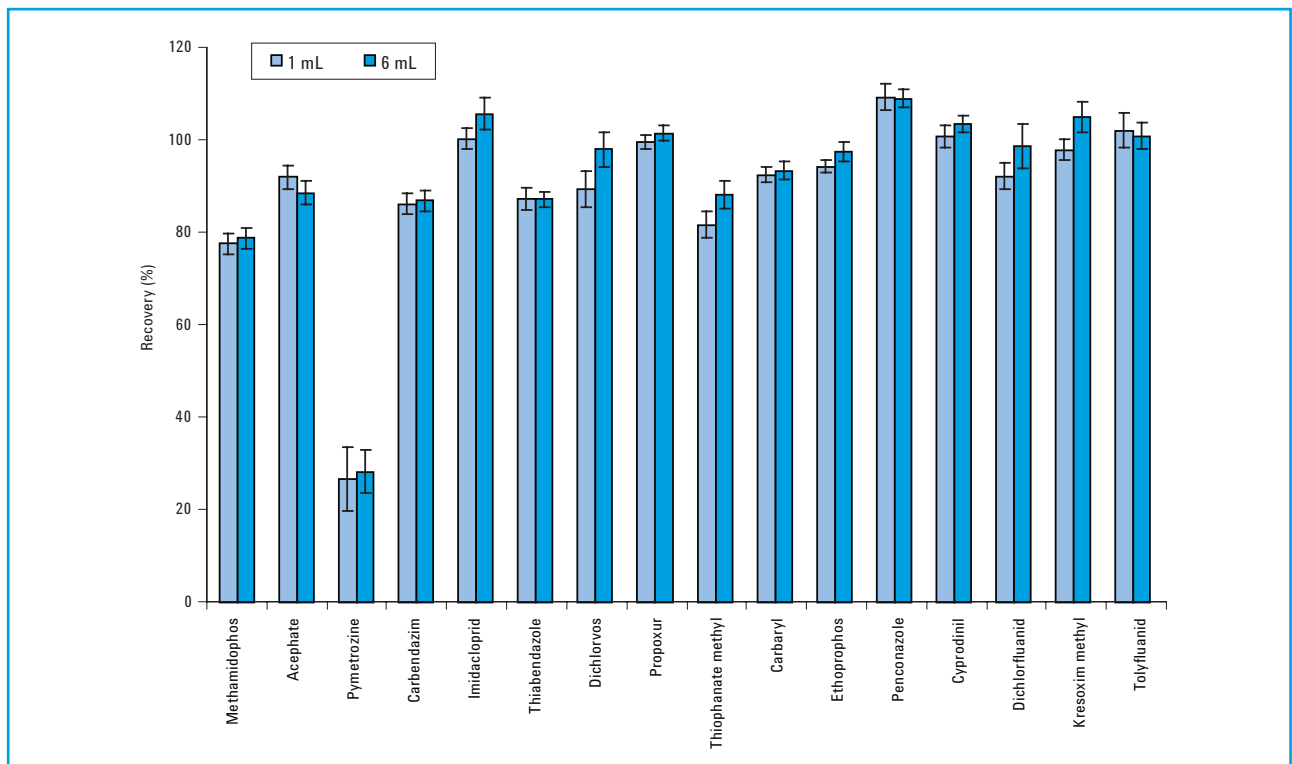


Figure 3. Results comparison of 1 mL dispersive SPE and 6 mL dispersive SPE.

To review this Application Note in its entirety, please search for 5990-3938EN at agilent.com/chem

Analysis of Pesticide Residues in Apple using Agilent Bond Elut QuEChERS EN Kits by GC/MS (Publication 5990-4073EN)

Introduction

This application describes the use of QuEChERS, an effective sample preparation approach described in the European Committee (EN) for extraction and cleanup of 17 GC-amenable multiple pesticide class residues in apple. The method involves initial extraction in an aqueous/acetonitrile system, an extraction/partitioning step after the addition of salt, and a cleanup step using dispersive solid phase extraction (dispersive SPE). The target pesticides in the apple extracts were then analyzed by gas chromatography/mass spectrometry (GC/MS) operating in selective ion monitoring (SIM) mode.

Instrument conditions

GC conditions

| | |
|----------------------------|--|
| Column: | Agilent J&W HP-5ms Ultra Inert 15 m x 0.25 mm, 0.25 µm (Part No. 19091S-431UI) |
| Inlet: | Splitless |
| Inlet liner: | Helix double-taper, deactivated (Part No. 5188-5398) |
| Carrier gas: | Helium |
| Inlet pressure: | 20.18 psi (constant pressure mode) during run 1.0 psi during backflush |
| Inlet temperature: | 250 °C |
| Injection volume: | 1.0 µL |
| Purge flow to split vent: | 30 mL/min at 0.75 min |
| Oven temperature program: | 70 °C (1 min), 50 °C/min to 150 °C (0 min), 6 °C/min to 200 °C (0 min), 16 °C/min to 280 °C (6 min) |
| Post run: | 3 min |
| Capillary flow technology: | Purged Ultimate Union (Part No. G3186B) – used for backflushing the analytical column and inlet |
| Aux EPC gas: | Helium plumbed to Purged Ultimate Union |
| Aux EPC pressure: | 4.0 psi during run, 80.0 psi during backflush |
| Connections: | Between inlet and Purged Ultimate Union (Part No. G3186B) |
| Restrictor: | 65 cm x 0.15 mm x 0.15 µm DB-5ms Ultra Inert |
| Connections: | Between the Purged Ultimate Union and the MSD |

MS conditions

| | |
|--|---|
| Tune file: | Atune.u |
| Mode: | SIM (refer to Table 2 for settings in detail) |
| Source, quad, transfer line temperatures: | 230 °C, 150 °C, and 280 °C, respectively |
| Solvent delay: | 2.30 min |
| Multiplier voltage: | Autotune voltage |

QuEChERS Procedure

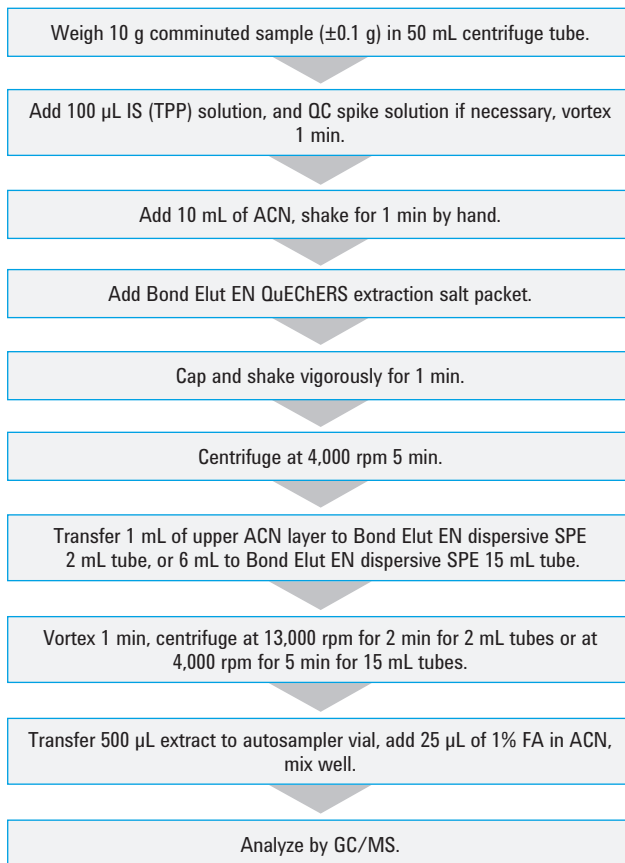


Figure 1. Agilent Bond Elut QuEChERS EN extraction procedure for pesticides in apple.

Results

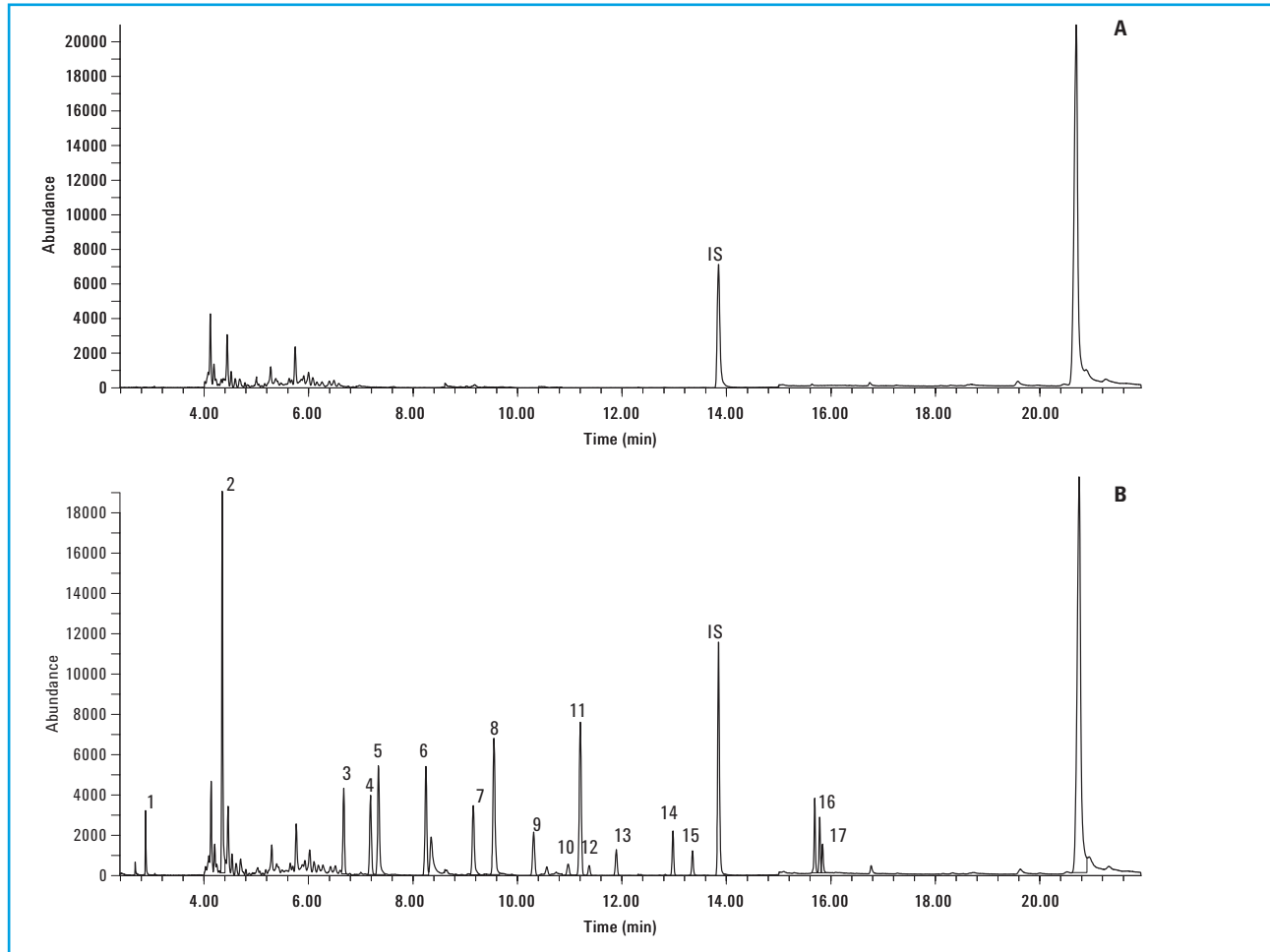


Figure 2. GC/MS chromatogram of apple extract. (A) apple extract blank; (B) 50 ng/g fortified apple extract. Peak Identification: 1. Dichlorvos, 2. *o*-Phenylphenol, 3. Lindane, 4. Diazinon, 5. Chlorothalonil, 6. Chlorpyrifos-methyl, 7. Dichlofluanid, 8. Dichlorobenzophenone, 9. Heptachlor epoxide, 10. γ -Chlordane, 11. DDE, 12. α -Chlordane, 13. Dieldrin, 14. Ethion, 15. Endosulfan sulfate, 16. Permethrin, 17. Coumaphos. IS: Triphenyl phosphate (TPP).

Products used in the above application

Agilent Bond Elut QuEChERS EN Method Extraction Kit. Part No. 5982-5650.

Agilent Bond Elut QuEChERS EN Method Dispersive SPE Kit for General Fruits and Vegetables,
2 mL. Part No. 5982-5021 or 15 mL. Part No. 5982-5056.

Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm, 0.25 μ m. Part No. 19091S-431UI.

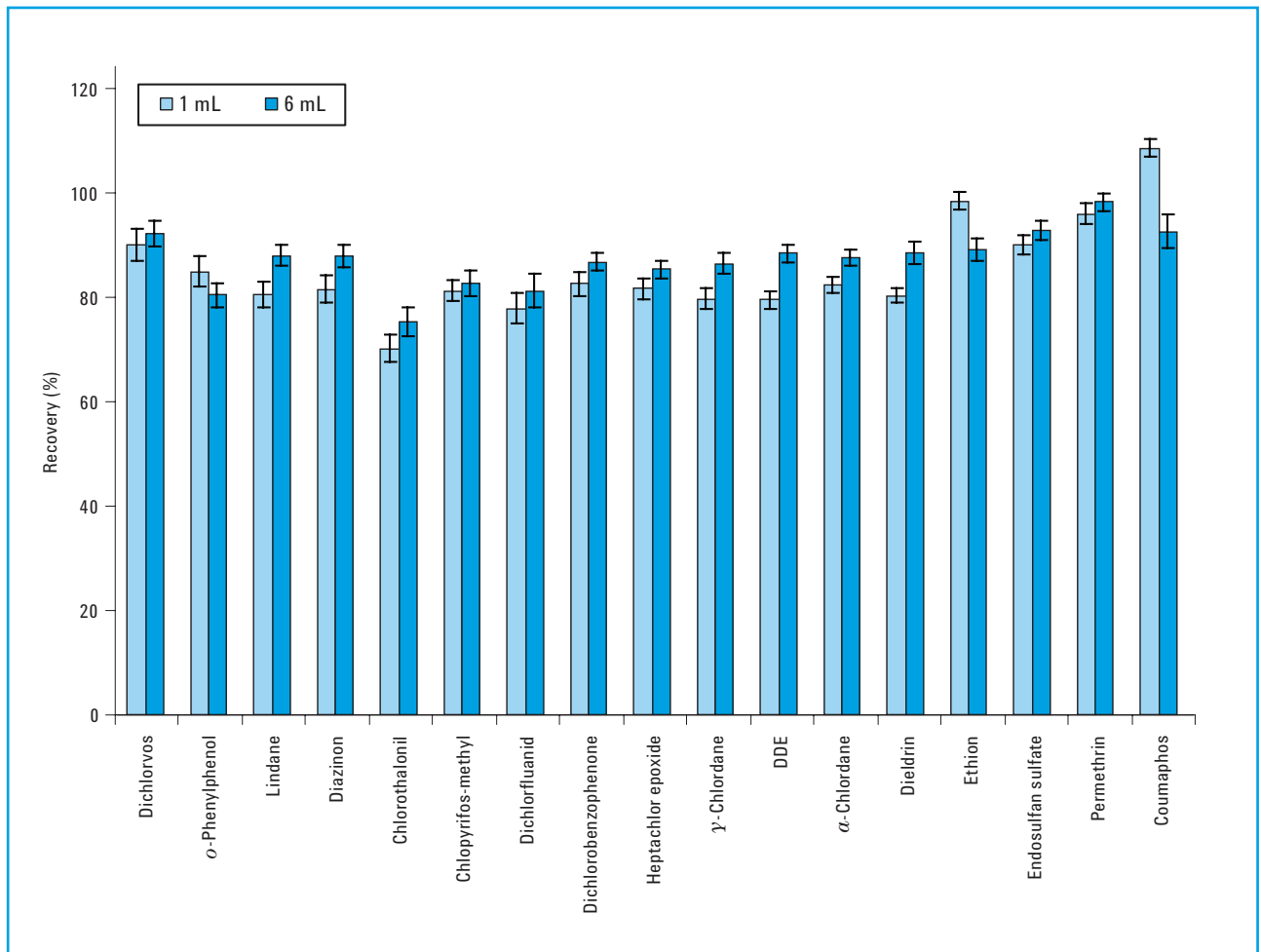


Figure 3. Recovery and precision results of 1 and 6 mL sample volumes employing Agilent Bond Elut Dispersive SPE, 2 and 15 mL kits, respectively.

Analysis of Pesticide Residues in Spinach using Agilent Bond Elut QuEChERS EN Kit by LC/MS/MS Detection (Publication 5990-4395EN)

Introduction

This application describes the use of the QuEChERS EN sample preparation approach for extraction and cleanup of 13 pesticide residues representing various classes in spinach. Because spinach is considered a highly pigmented matrix, the EN dispersive SPE kit for highly pigmented fruits and vegetables is selected. Graphitized carbon black (GCB) in the amount of 7.5 mg/mL of ACN extract is added to the kit. The target pesticides in the spinach extracts are then determined by liquid chromatography coupled to an electrospray ionization tandem mass spectrometry (LC-ESI-MS/MS) operating in positive ion multiple reaction monitoring (MRM) mode.

Instrument conditions

HPLC conditions

| | | | |
|---------------------|--|----------------|--------------------|
| Column: | Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl, 3.0 x 150 mm, 3.5 μm (Part No. 959963-312) | | |
| Flow rate: | 0.3 mL/min | | |
| Column temperature: | 30 °C | | |
| Injection volume: | 10 μL | | |
| Mobile phase: | A: 5 mM Ammonium acetate, pH 5.0 in 20:80 MeOH:H ₂ O B: 5 mM Ammonium acetate, pH 5.0 in ACN | | |
| Needle wash: | 1:1:1 ACN:MeOH:IPA:H ₂ O w/0.2% FA | | |
| Gradient: | Time (min) | % Acetonitrile | Flow rate (mL/min) |
| | 0 | 20 | 0.3 |
| | 0.5 | 20 | 0.3 |
| | 8.0 | 100 | 0.3 |
| | 10.0 | 100 | 0.3 |
| | 13.0 | STOP | |
| Post run: | 4 min | | |
| Total cycle time: | 17 min | | |

MS conditions

Positive mode

| | |
|------------------|----------|
| Gas temperature: | 350 °C |
| Gas flow: | 10 L/min |
| Nebulizer: | 40 psi |
| Capillary: | 4,000 V |

QuEChERS Procedure

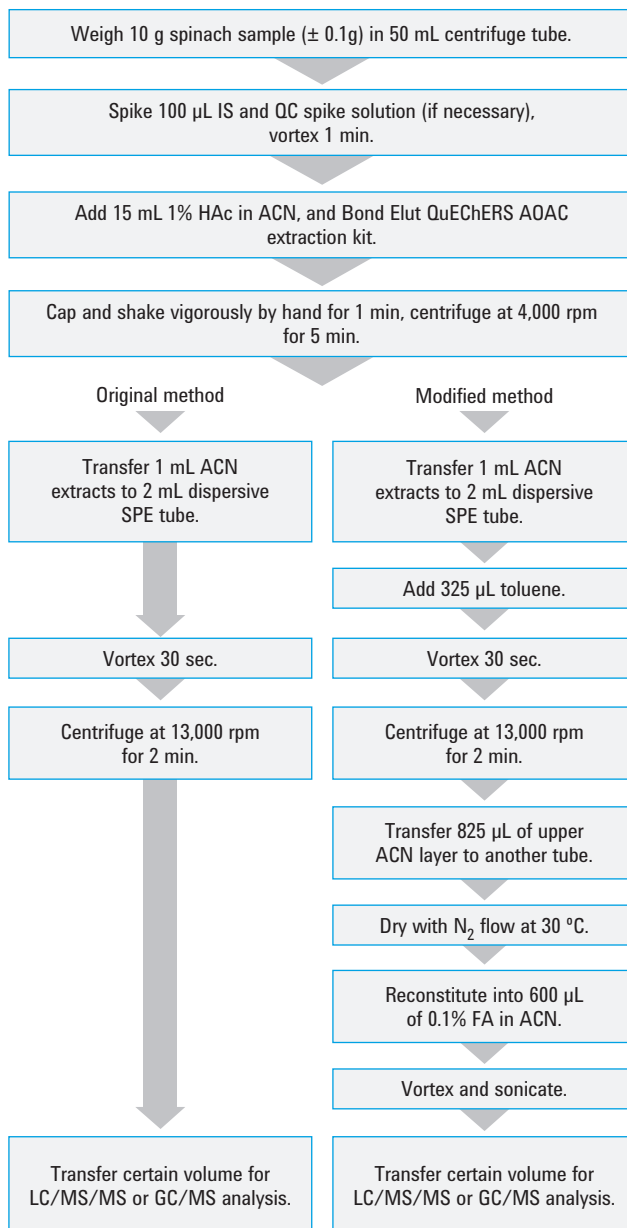


Figure 1. QuEChERS EN procedure (original and modified dispersive SPE, 2 mL size) for pesticide residues in spinach.

Results

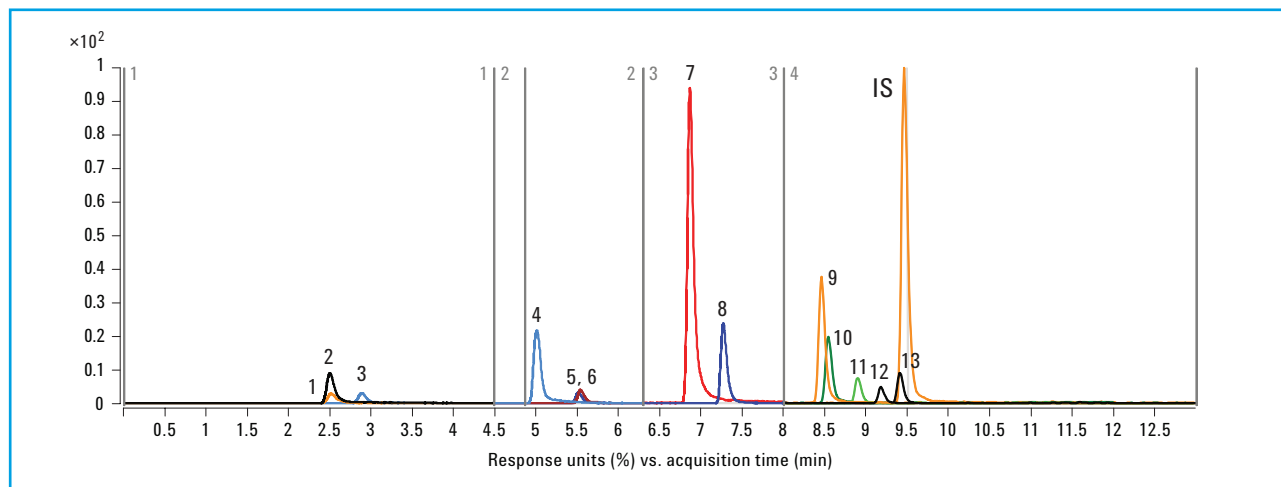


Figure 2. MRM chromatogram of 50 ng/g fortified sample of spinach processed by EN method. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Propoxur, 8. Carbaryl, 9. Ethoprophos, 10. Imazalil, 11. Penconazole, 12. Cyprodinil, 13. Kresoxim methyl, IS: Internal Standard, TPP.

Table 1. Recovery and reproducibility of pesticides in fortified spinach with 6 mL dispersive SPE tube (Part No. 5982-5356)

| Analytes | 10 ng/g fortified QC | | 50 ng/g fortified QC | | 200 ng/g fortified QC | |
|-----------------|----------------------|-----------|----------------------|-----------|-----------------------|-----------|
| | Recovery | RSD (n=6) | Recovery | RSD (n=6) | Recovery | RSD (n=6) |
| Methamidophos | 85.0 | 8.3 | 87.7 | 2.7 | 95.0 | 9.4 |
| Acephate | 88.6 | 5.1 | 84.6 | 3.1 | 94.6 | 9.3 |
| Pymetrozine* | 68.7 | 3.7 | 65.7 | 1.5 | 71.9 | 10.8 |
| Carbendazim* | 94.0 | 5.4 | 91.4 | 2.7 | 53.5 | 9.3 |
| Imidacloprid | 102.0 | 8.9 | 85.4 | 6.1 | 100.1 | 7.7 |
| Thiabendazole* | 77.2 | 4.4 | 77.6 | 2.4 | 79.2 | 9.7 |
| Propoxur | 98.2 | 5.7 | 96.3 | 1.8 | 93.9 | 7.2 |
| Carbaryl | 98.5 | 3.6 | 94.0 | 1.7 | 97.4 | 7.2 |
| Ethoprophos | 102.3 | 6.0 | 95.3 | 1.7 | 91.0 | 6.8 |
| Imazalil | 88.8 | 6.4 | 86.8 | 2.8 | 93.5 | 7.7 |
| Penconazole | 104.5 | 2.5 | 96.4 | 2.0 | 84.6 | 5.5 |
| Cyprodinil* | 101.5 | 4.2 | 92.2 | 2.4 | 86.8 | 7.6 |
| Kresoxim methyl | 99.7 | 6.1 | 97.4 | 1.6 | 95.3 | 6.9 |

*Pesticides with planar structure.

Products used in the above application

Agilent Bond Elut QuEChERS EN Extraction Kits. Part No. 5982-5650.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kits for Pigmented Fruits and Vegetables.

Part Nos. 5982-5321 and 5982-5356.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 µm. Part No. 959963-312.

To review this Application Note in its entirety, please search for 5990-4395EN at agilent.com/chem

Determination of Acrylamide in Cooking Oil by Agilent Bond Elut QuEChERS Acrylamide Kit and HPLC-DAD (Publication 5990-5988EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach for the analysis of acrylamide in cooking oil, employing methacrylamide as the internal standard.

Acrylamide occurs naturally as a byproduct of the cooking process and in heat-treated foods. The acrylamide recoveries ranged from 84 to 93.8%.

The analysis was performed on an Agilent 1200 Infinity Series equipped with a binary pump and a diode array detector (DAD) set at 210 nm.

Instrument conditions

HPLC conditions

| | |
|---------------------|---|
| Column: | Agilent ZORBAX HILIC Plus, 4.6 × 50 mm, 3.5 μm |
| Flow rate: | 0.2 mL/min |
| Column temperature: | 30 °C |
| Injection volume: | 5 μL |
| Mobile phase: | Isocratic elution: A: 3% 5 mM Acetic acid B: 97% Acetonitrile |
| Run time: | 10 min |
| Post time: | 3 min |
| Detection: | DAD at 210 nm |

QuEChERS Procedure

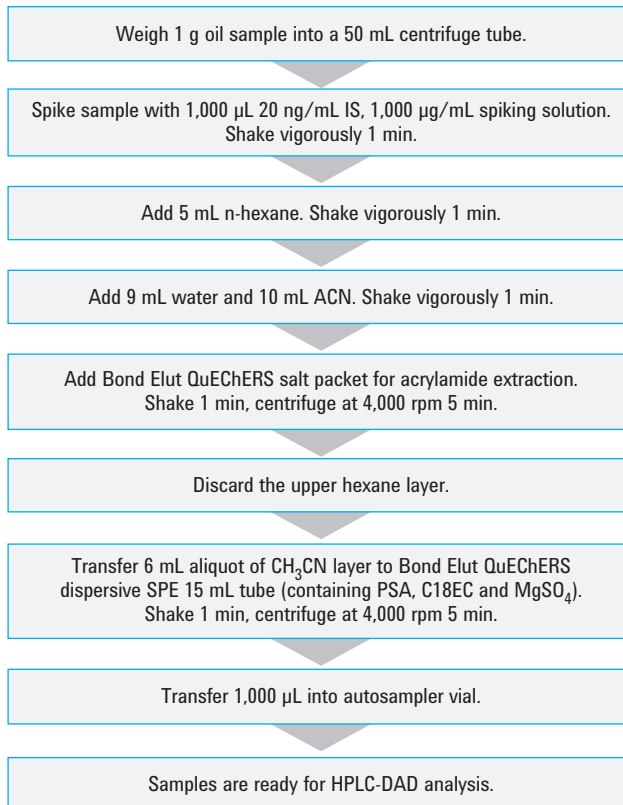


Figure 1. QuEChERS sample preparation procedure for acrylamide in cooking oil.



Results

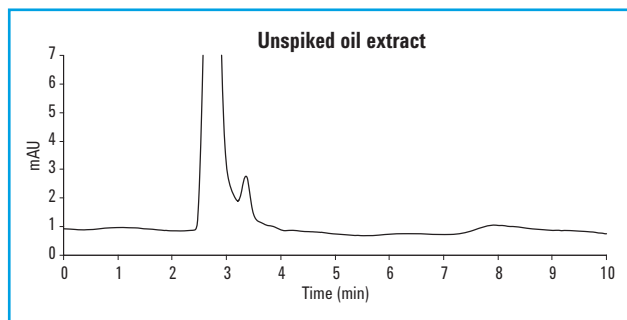


Figure 2. Chromatogram of the blank oil extract.

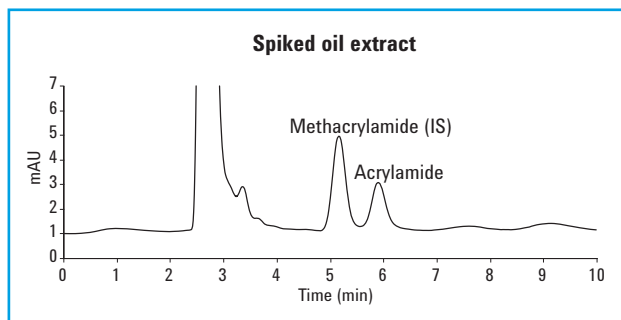


Figure 3. Chromatogram of the spiked oil extract.

Table 1. Recoveries and RSDs for the acrylamide in oil sample (n=6)

| Concentration (ng/mL) | %Recovery (n=6) | %RSD (n=6) |
|-----------------------|-----------------|------------|
| 500 | 84.0 | 3.2 |
| 1,000 | 93.8 | 2.2 |
| 2,000 | 92.2 | 1.5 |

Products used in the above application

Agilent Bond Elut QuEChERS Extraction Kit for Acrylamides. Part No. 5982-5850.

Agilent Bond Elut QuEChERS EN Dispersive SPE Kit. Part No. 5982-5156.

Agilent ZORBAX HILIC Plus Column, 4.6 × 50 mm, 3.5 μm. Part No. 959943-901.

To review this Application Note in its entirety, please search for 5990-5988EN at agilent.com/chem

A Blind Study of Pesticides in Fruiting Vegetables by Agilent Bond Elut QuEChERS Extraction Kits and Agilent 5975T LTM GC/MSD (Publication 5990-6323EN)

Introduction

This application describes the use of the QuEChERS EN sample preparation approach for the identification of target pesticides in fruiting vegetables. Cherry tomato and cucumber were used as pesticide matrixes. The analysis was performed on an Agilent 5975T LTM GC/MSD system equipped with an Agilent 7693 Automatic Liquid Sampler. RTL software is a feature of Agilent MSD ChemStation.

Instrument conditions

Instrumentation

| | |
|---------------|--|
| Column: | Agilent J&W HP-5ms LTM, 30 m × 0.25 mm, 0.25 µm |
| Guard column: | 1 m column with same phase as analytical column, connected to the injector |
| GCMS system: | 5975T LTM |
| Inlet: | Split/splitless |
| Autosampler: | Agilent 7693 |

Experimental conditions

| | |
|----------------------------|---|
| Inlet temperature: | 280 °C |
| Injection volume: | 1 µL |
| Injection mode: | Splitless |
| Carrier gas: | Helium |
| Head pressure: | 26.878 psi, constant pressure mode |
| Method: | RT locked to chlorpyrifos methyl at 16.593 min |
| LTM oven temperature: | 70 °C (2 min), 25 °C/min, 150 °C (0 min), 3 °C/min, 200 °C (0 min), 8 °C/min, 280 °C (10 min) |
| Transfer line temperature: | 270 °C |
| MSD interface: | 270 °C |
| Ion source: | 230 °C |
| Quad temperature: | 150 °C |
| Ionization mode: | EI |
| Scan mode: | Full scan, 50–550 u |
| EMV mode : | Gain factor |
| Gain factor : | 5.00 |
| Resulting EM voltage: | 1129 V |
| Solvent delay: | 3 min |

QuEChERS Procedure

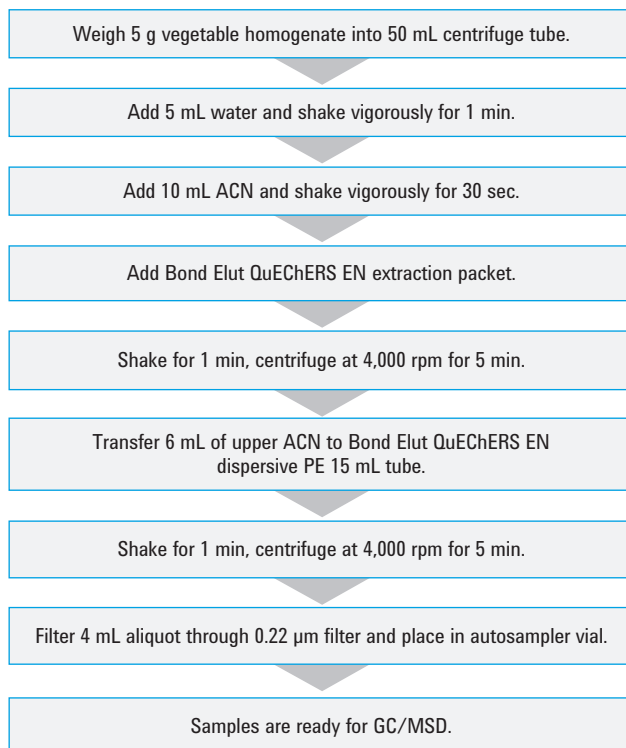


Figure 1. QuEChERS EN procedure for pesticides in fruiting vegetables.

Results

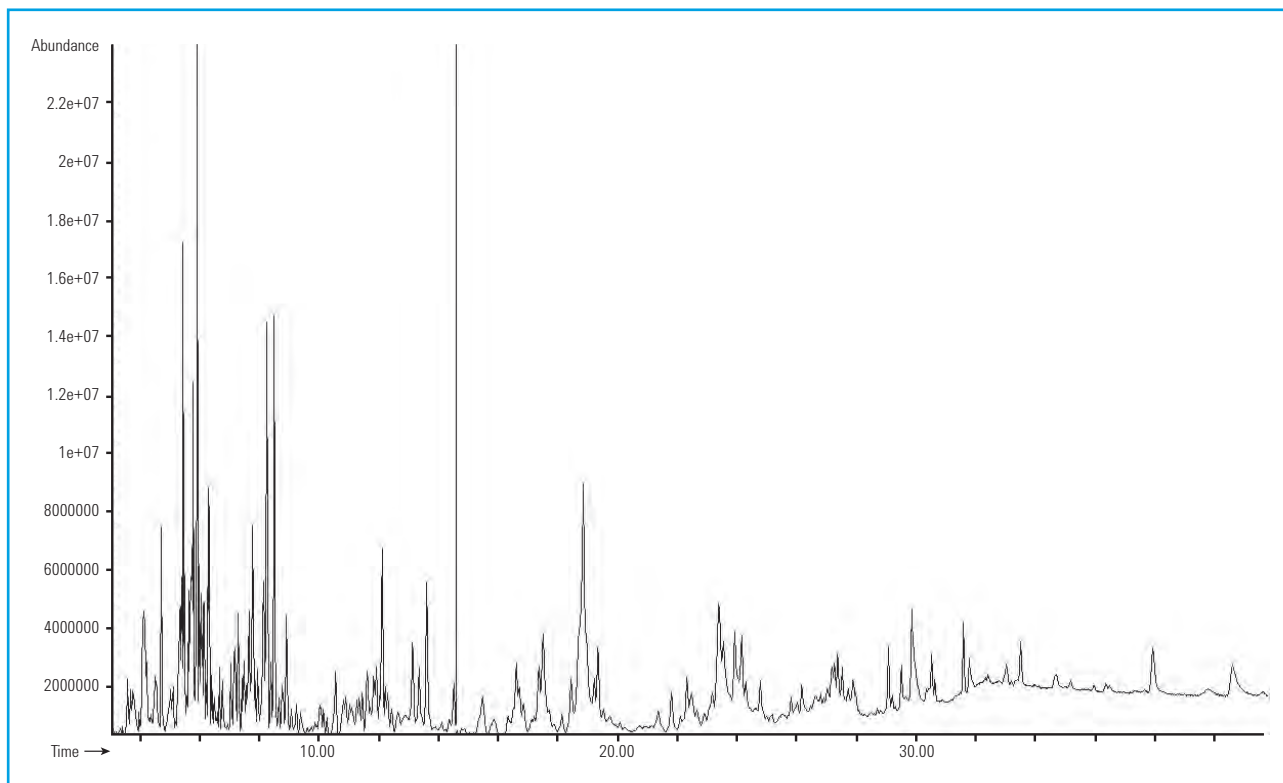


Figure 2. MA 0.2 to 1.0 $\mu\text{g/g}$ amount of pesticide spiked in cherry tomato. All of the targets could be found by DRS software within 2 min.



Products used in the above application

Agilent Bond Elut QuEChERS EN Extraction Kits. Part No. 5982-5650.

Agilent Bond Elut QuEChERS EN Dispersive SPE Kit. Part No. 5982-5156.

Agilent J&W HP-5ms LTM Column, 30 m x 0.25 mm, 0.25 μm . Part No. 29091S-433LTM.

To review this Application Note in its entirety, please search for 5990-6323EN at agilent.com/chem

Analysis of Pesticide Residues in Green Tea Using Agilent Bond Elut QuEChERS EN Kit by LC/MS/MS Detection (Publication 5990-6400EN)

Introduction

This application describes the use of the QuEChERS EN sample preparation approach for the extraction and cleanup of 12 pesticide residues representing various pesticide classes in green tea. Green tea is considered to be a highly pigmented sample since it contains high levels of chlorophyll. The presence of target pesticides was determined by liquid chromatography with an Agilent 1200 Infinity Series coupled to an Agilent 6410 Triple Quadrupole MS system with electrospray ionization operating in MRM mode. Mean recoveries ranged between 87% and 108% (average of 93.5%).

Instrument conditions

HPLC conditions

| | | |
|---------------------|---|----------------|
| Column: | Agilent Poroshell 120 EC-C18, 2.1 × 100 mm, 2.7 μm, (Part No. 695775-902) | |
| Flow rate: | 0.4 mL/min | |
| Column temperature: | 30 °C | |
| Injection volume: | 10 μL | |
| Mobile phase: | A: 5 mM FA in water B: 5 mM FA in ACN | |
| Needle wash: | 1:1:1 ACN:MeOH:IPA:H ₂ O w/0.2% FA | |
| Gradient: | Time (min) | % Acetonitrile |
| | 0 | 5 |
| | 1 | 5 |
| | 3 | 50 |
| | 7 | 90 |
| | 8 | 90 |
| | 8.2 | 5 |
| | 9 | 5 |
| Post run: | 2 min | |
| Total cycle time: | 11 min | |

MS conditions

Positive mode

| | |
|------------------|----------|
| Gas temperature: | 350 °C |
| Gas flow: | 10 L/min |
| Nebulizer: | 40 psi |
| Capillary: | 3,500 V |

QuEChERS Procedure

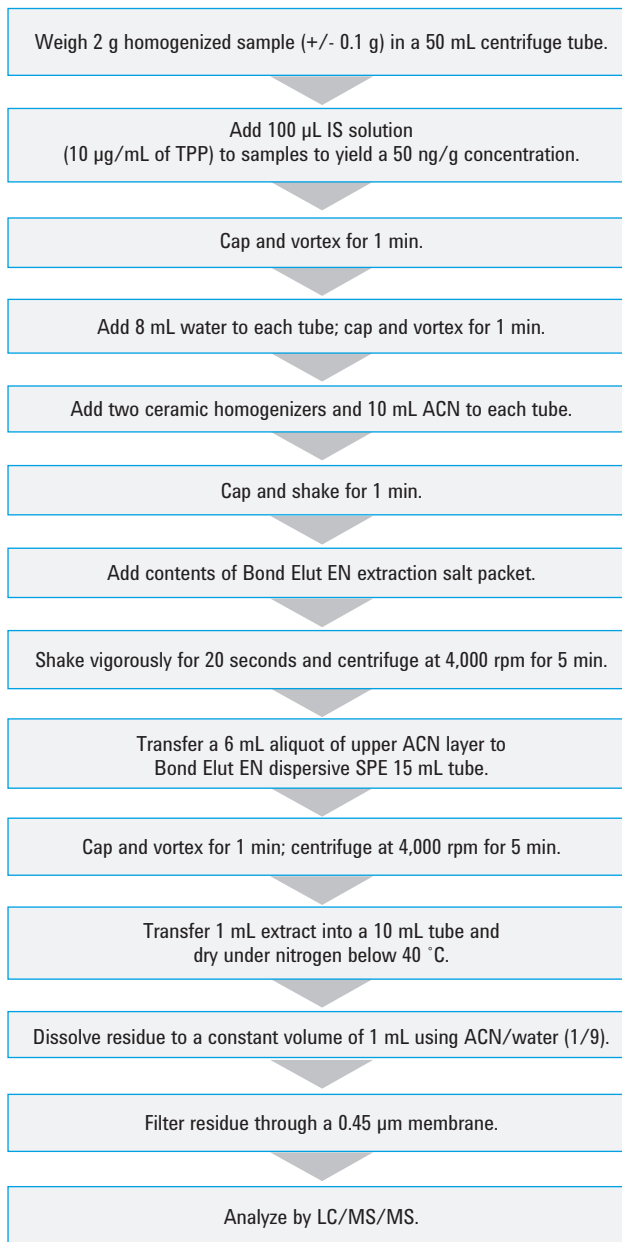


Figure 1. QuEChERS EN procedure for pesticides in green tea.

Results

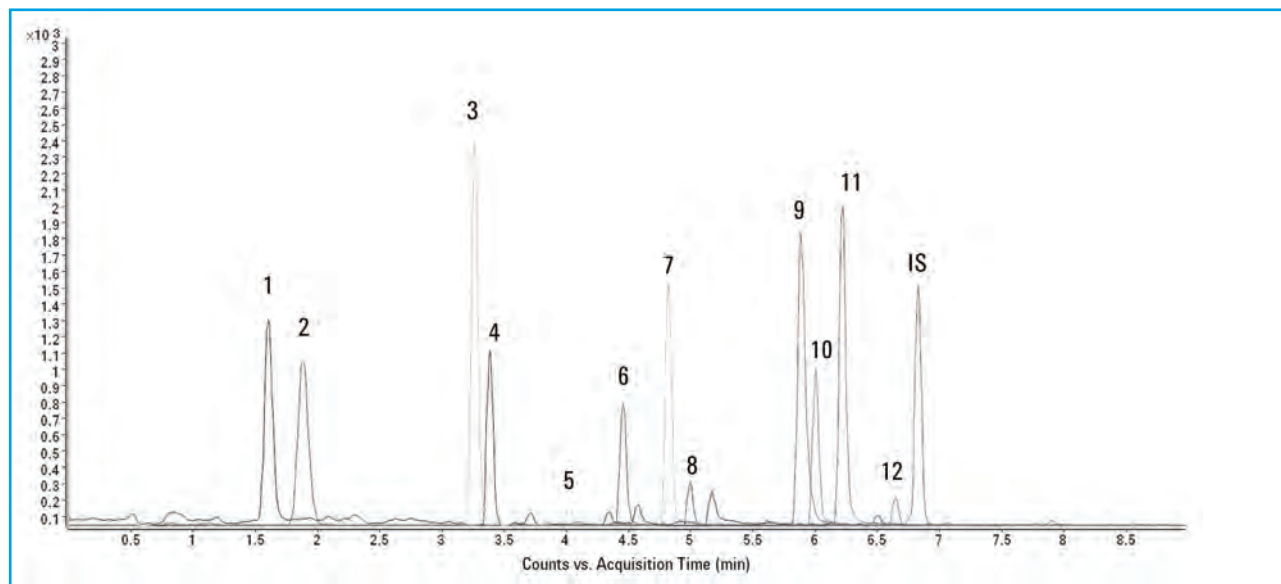


Figure 2. MRM chromatograms of 50 ng/g fortified sample processed by EN method. Peak identification: 1. Acephate, 2. Pymetrozine, 3. Carbendazim, 4. Thiabendazole, 5. Imidacloprid, 6. Imazalil, 7. Propoxur, 8. Carbaryl, 9. Cyprodinil, 10. Ethoprophos, 11. Penconazole, 12. Kresoxim-methyl, IS: TPP.

Table 1. Recovery and reproducibility of pesticides in fortified green tea with Agilent Bond Elut QuEChERS

| Analytes | 10 ng/g fortified QC | | 50 ng/g fortified QC | | 200 ng/g fortified QC | |
|-----------------|----------------------|-----------|----------------------|-----------|-----------------------|-----------|
| | Recovery | RSD (n=6) | Recovery | RSD (n=6) | Recovery | RSD (n=6) |
| Acephate | 80.5% | 5.4% | 91.7% | 2.9% | 88.9% | 8.2% |
| Pymetrozine | 43.1% | 3.0% | 42.2% | 3.4% | 43.4% | 9.8% |
| Carbendazim | 114.6% | 11.6% | 97.6% | 2.0% | 105.0% | 6.2% |
| Thiabendazole | 98.1% | 6.9% | 90.4% | 2.4% | 81.7% | 5.8% |
| Imidacloprid | 104.3% | 11.7% | 108.6% | 2.5% | 93.9% | 7.9% |
| Imazalil | 97.5% | 4.4% | 87.8% | 5.6% | 92.4% | 4.6% |
| Propoxur | 98.1% | 2.4% | 110.2% | 1.7% | 107.8% | 3.9% |
| Carbaryl | 89.7% | 11.4% | 104.9% | 3.3% | 108.1% | 5.2% |
| Cyprodinil | 84.9% | 2.1% | 92.5% | 3.7% | 93.9% | 5.5% |
| Ethoprophos | 103.4% | 3.1% | 111.2% | 3.2% | 104.9% | 5.7% |
| Penconazole | 108.7% | 2.9% | 94.3% | 4.5% | 89.8% | 3.3% |
| Kresoxim-methyl | 105.7% | 12.4% | 96.4% | 2.5% | 99.2% | 5.5% |

Products used in the above application

Agilent Bond Elut QuEChERS EN Extraction Kits. Part No. 5982-5650.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kits for Pigmented Fruits and Vegetables. Part No. 5982-5356.

Agilent Poroshell 120 EC-C18 Column, 2.1 x 100 mm, 2.7 µm, Part No. 695775-902.

To review this Application Note in its entirety, please search for 5990-6400EN at agilent.com/chem

Determination of Hormones in Shrimp by Agilent 1290 Infinity LC with Agilent Poroshell 120 LC Column and Agilent Bond Elut QuEChERS for Sample Preparation (Publication 5990-6589EN)

Introduction

This application describes the use of the QuEChERS EN sample preparation approach for the extraction of 13 hormones in shrimp. A method for the determination of hormones in shrimp was developed using an Agilent 1290 Infinity LC with an Agilent Poroshell 120 EC-C18 column. Method recoveries ranged from 91.6 to 107.2%.

Instrument conditions

HPLC conditions

| | | | |
|-----------------------|---|---------|----------------|
| Column: | Agilent Poroshell 120 EC-C18, 3.0 x 100 mm, 2.7 μ m (Part No. 695975-302) | | |
| Instrument: | Agilent 1290 Infinity LC with DAD detector | | |
| Flow rate: | 0.8 mL/min | | |
| Column temperature: | 30 °C | | |
| Injection volume: | 10 μ L | | |
| Detection wavelength: | 230 nm | | |
| Mobile phase: | Water-acetonitrile gradient | | |
| Gradient: | Time (min) | % Water | % Acetonitrile |
| | | 0 | 80/20 |
| | 6 | 50 | 50 |
| | 8 | 10 | 90 |

QuEChERS Procedure

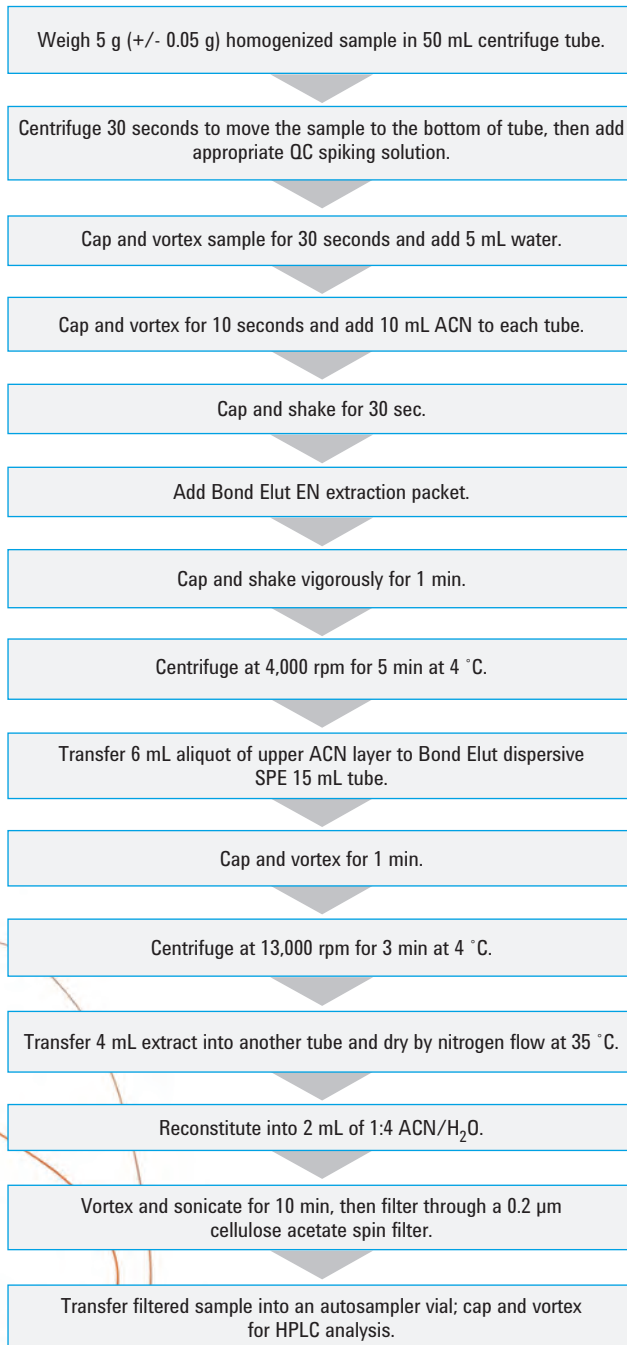


Figure 1. QuEChERS EN procedure for hormones in shrimp.



Results

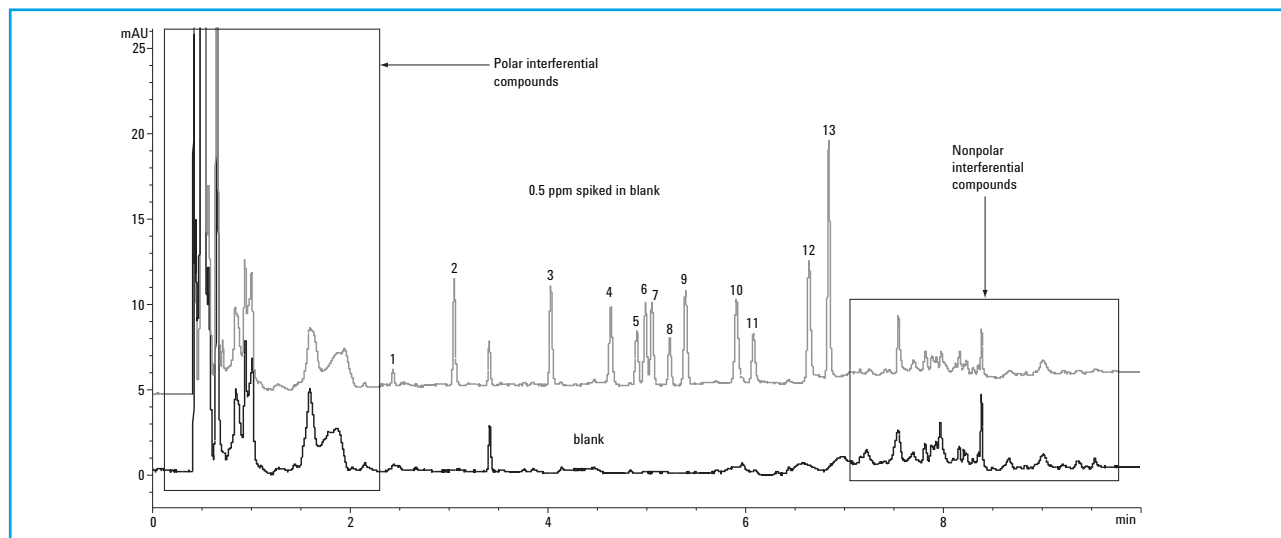


Figure 2. Chromatograms of blank sample and 0.5 ppm spiked in blank sample.

Table 1. Recovery and reproducibility for the QuEChERS method developed for hormone determination in shrimp

| Analytes | Concentration | Recovery (%) | % RSD (n=6) |
|-------------------------|---------------|--------------|-------------|
| Estriol | 0.5 | 107.2 | 3.5 |
| | 10 | 98.2 | 0.98 |
| Prednisone | 0.5 | 97.6 | 2.3 |
| | 10 | 101.7 | 0.58 |
| Dexamethasone | 0.5 | 101.8 | 0.96 |
| | 10 | 96.1 | 1.2 |
| Boldenone | 0.5 | 98.9 | 1.5 |
| | 10 | 96.2 | 1.8 |
| Hydrocortisone | 0.5 | 103.5 | 1.5 |
| | 10 | 92.3 | 0.23 |
| Fludrocortisone acetate | 0.5 | 104.3 | 1.9 |
| | 10 | 91.8 | 0.17 |
| Metandienone | 0.5 | 100.0 | 1.4 |
| | 10 | 95.6 | 0.25 |
| Estradiol | 0.5 | 99.4 | 1.3 |
| | 10 | 97.8 | 0.54 |
| Testosterone | 0.5 | 98.0 | 0.85 |
| | 10 | 98.2 | 0.15 |
| Methyltestosterone | 0.5 | 97.1 | 0.99 |
| | 10 | 92.1 | 0.63 |
| Estrone | 0.5 | 103.4 | 1.2 |
| | 10 | 92.5 | 0.68 |
| Diethylstilbestrol | 0.5 | 100.9 | 1.9 |
| | 10 | 97.3 | 0.79 |
| Hexestrol | 0.5 | 98.5 | 1.6 |
| | 10 | 91.6 | 0.81 |

Products used in the above application

Agilent Bond Elut QuEChERS EN Extraction Kits. Part No. 5982-5650.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kits for Drug Residues in Meat, 15 mL. Part No. 5982-4956.

Agilent Poroshell 120 EC-C18 Column, 3.0 x 100 mm, 2.7 μ m. Part No. 695975-302.

To review this Application Note in its entirety, please search for 5990-6589EN at agilent.com/chem

Analysis of Pesticide Residues in Rice with Bond Elut QuEChERS Extraction Kits and Agilent J&W HP-5ms Ultra Inert GC Column (Publication 5990-8108EN)

Introduction

This application describes the use of the QuEChERS EN sample preparation approach for the extraction and cleanup of 57 GC-amenable multiple pesticide class residues in rice. Agilent developed GC and GC/MS RTL databases (Part No. G1672AA) that include 962 pesticides, metabolites, and suspected endocrine disruptors. The experiments were performed on an Agilent 7890 GC equipped with 5975C inert MSD, and Agilent 7683 Automatic Liquid Sampler (ALS). Separation of the compounds was achieved on an Agilent J&W HP-5ms Ultra Inert GC column. Most of the recoveries ranged between 80 and 110%.

Instrument conditions

GC conditions

| | |
|-------------------------|--|
| Column: | Agilent J&W HP-5ms Ultra Inert, 30 m × 0.25 mm, 0.25 µm (Part No. 19091S-433UI) |
| Inlet temperature: | 250 °C |
| Carrier gas: | Helium, constant pressure mode |
| Retention time locking: | Chropyrifos-methyl locked to 16.596 min |
| Injection mode: | Splitless, purge flow 50 mL/min at 0.75 min |
| Injection volume: | 1 µL |
| Oven: | 70 °C (2 min), 25 °C/min to 150 °C (0 min), 3 °C/min to 200 °C, 8 °C/min to 280 °C (10 min), postrun: 320 °C (5 min) |

MS conditions

| | |
|----------------|--|
| Solvent delay: | 4 min |
| MS temp: | 230 °C (source); 150 °C (quad) |
| Transfer line: | 280 °C |
| MS libraries: | Agilent RTL Pesticide Library (G1672AA) and NIST08 Mass Spec Library |
| MS: | EI, SIM/Scan |
| Scan mode: | Mass range (50-550 amu) |

QuEChERS Procedure

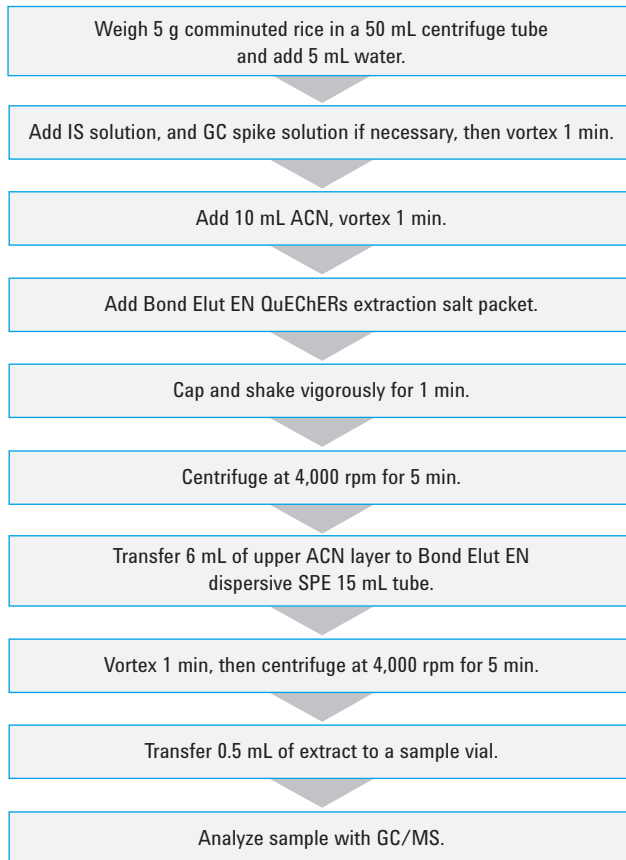


Figure 1. Agilent Bond Elut QuEChERS EN extraction procedure for pesticides in rice.

Results

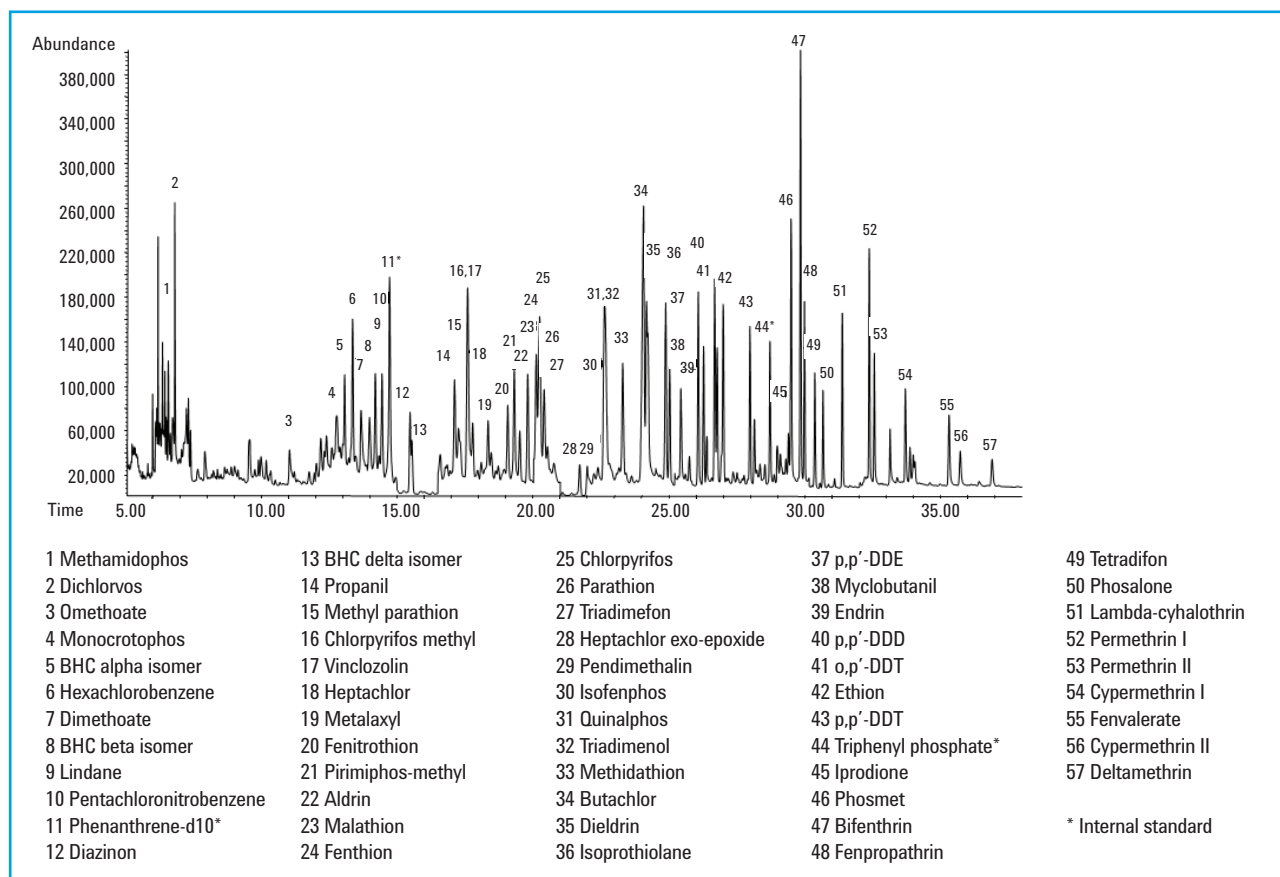


Figure 2. Total ion chromatogram (TIC) of pesticides in rice at 200 ng/mL.



Products used in the above application

Agilent Bond Elut QuEChERS EN Extraction Kits. Part No. 5982-5650.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kits. Part No. 5982-5156.

Agilent J&W HP-5ms Ultra Inert Column, 30 m x 0.25 mm, 0.25 μ m. Part No. 19091S-433UI.

To review this Application Note in its entirety, please search for 5990-8108EN at agilent.com/chem

AOAC Methods

Analysis of Pesticide Residues in Apples using Agilent Bond Elut QuEChERS AOAC Kit by LC/MS/MS Detection (Publication 5990-3937EN)

Introduction

This application describes the use of QuEChERS, Association of Analytical Communities (AOAC) Official Method 2007.01 sample preparation approach for extraction and cleanup of 16 pesticide residues in apple.

The 5 ng/g limit of quantitation (LOQ) for pesticides in apple shown in this application was well below the maximum residue limits (MRLs). The spiking levels for the recovery experiments were 10, 50, and 200 ng/g. Mean recoveries ranged between 76 and 117% (95.4% on average), with RSD below 15% (4.3% on average).



Instrument conditions

HPLC conditions

| | | | |
|---------------------|--|------|--------------------|
| Column: | Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl, 3.0 x 150 mm, 3.5 µm (Part No. 959963-312) | | |
| Flow rate: | 0.3 or 0.5 mL/min | | |
| Column temperature: | 30 °C | | |
| Injection volume: | 10 µL | | |
| Mobile phase: | A: 5 mM NH ₄ OAc, pH 5.0 in 20:80 MeOH/H ₂ O B: 5 mM NH ₄ OAc, pH 5.0 in ACN | | |
| Needle wash: | 1:1:1:1 ACN:MeOH:IPA:H ₂ O (0.2% FA) | | |
| Gradient: | Time (min) | % B | Flow rate (mL/min) |
| | 0 | 20 | 0.3 |
| | 0.5 | 20 | 0.3 |
| | 8.0 | 100 | 0.3 |
| | 10.0 | 100 | 0.3 |
| | 10.01 | 20 | 0.5 |
| | 12.0 | 100 | 0.5 |
| | 13.0 | STOP | |
| Post run: | 4 min | | |
| Total cycle time: | 17 min | | |

MS conditions

Positive mode

| | |
|------------------|----------|
| Gas temperature: | 350 °C |
| Gas flow: | 10 L/min |
| Nebulizer: | 40 psi |
| Capillary: | 4,000 V |

QuEChERS Procedure

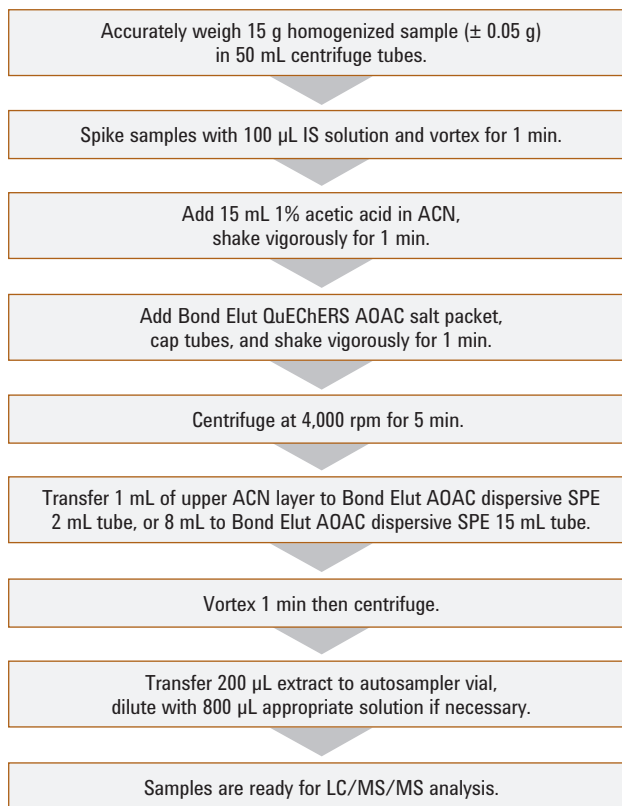


Figure 1. QuEChERS AOAC sample preparation procedures for pesticides in apple.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5022 or 15 mL. Part No. 5982-5058.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 µm. Part No. 959963-312.

Results

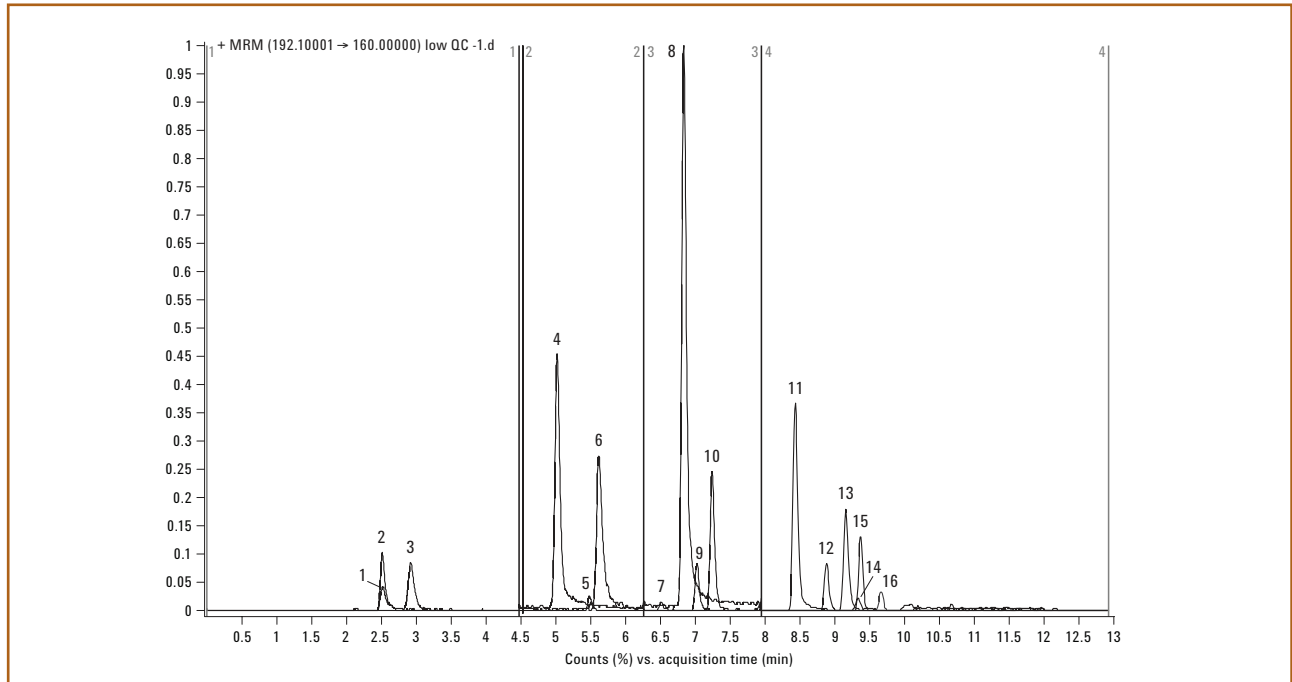


Figure 2. Chromatogram of 10 ng/g fortified apple extract. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Dichlorvos, 8. Propoxur, 9. Thiophanate methyl, 10. Carbaryl, 11. Ethoprophos, 12. Penconazole, 13. Cyprodinil, 14. Dichlofluanid, 15. Kresoxim methyl, 16. Tolyfluanid.

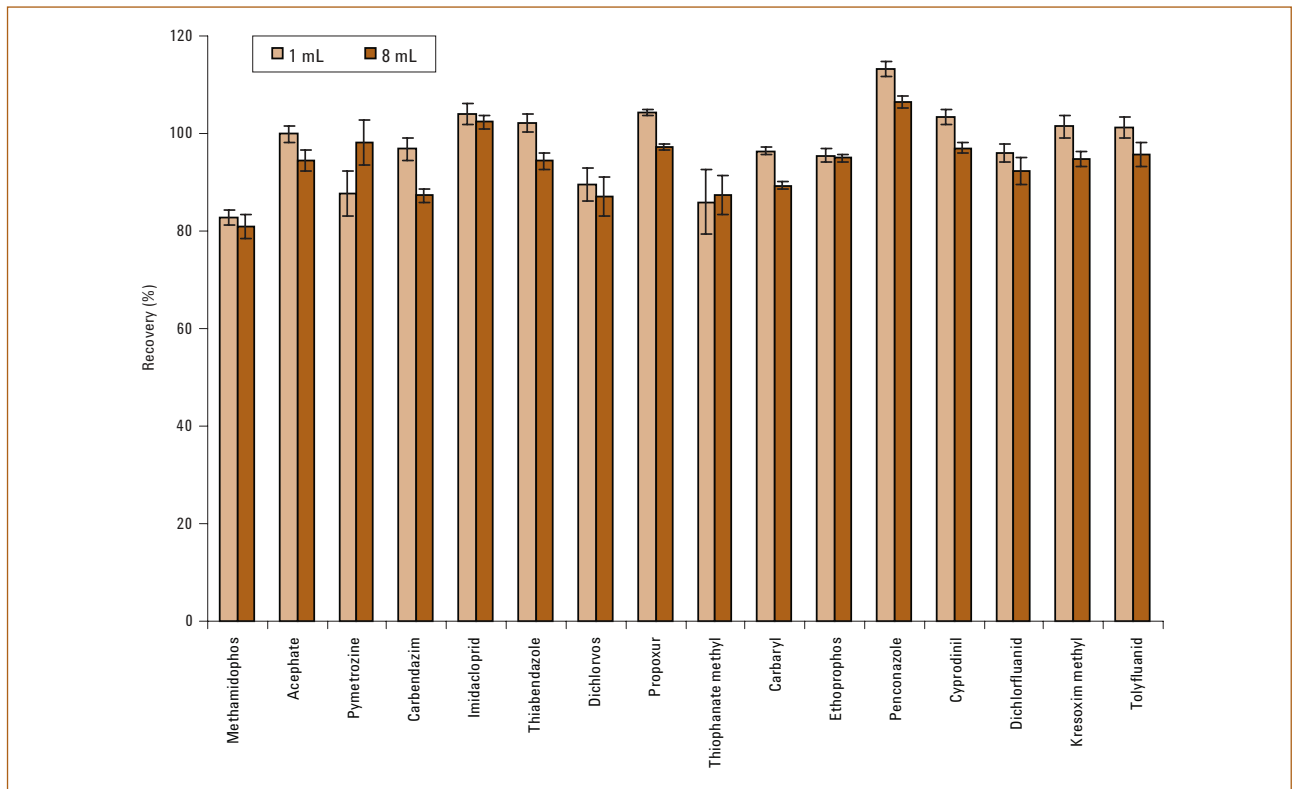


Figure 3. Results comparison of 1 mL and 8 mL dispersive SPE sample volume.

Analysis of Pesticide Residues in Apple using Agilent Bond Elut QuEChERS AOAC Kits by GC/MS (Publication 5990-4068EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach for extraction and cleanup of 17 GC-amenable pesticide residues from multiple classes in apple. The target pesticides in the apple extracts were then analyzed by gas chromatography/mass spectrometry (GC/MS) operating in selective ion monitoring (SIM) mode.

Instrument conditions

GC conditions

| | |
|---------------------------|---|
| Column: | Agilent J&W HP-5ms Ultra Inert, 30 m x 0.25 mm, 0.25 µm (Part No. 19091S-433UI) |
| Autosampler: | Agilent 7683 Automatic Liquid Sampler |
| Inlet: | Splitless |
| Carrier gas: | Helium in constant pressure |
| Retention time locking: | Chlorpyrifos-methyl locked to 16.596 min (nominal column head pressure = 22.0 psi) |
| Oven temperature program: | 70 °C (2 min), 25 °C/min to 150 °C (0 min), 3 °C/min to 200 °C (0 min), 8 °C/min to 280 °C (11.5 min) |
| Injection volume: | 1.0 µL |

MS conditions

| | |
|--|---|
| Tune file: | Atune.u |
| Mode: | SIM (refer to Table 2 of App Note for settings in detail) |
| Source, quad, transfer line temperature: | 230 °C, 150 °C, and 280 °C, respectively |
| Solvent delay: | 3 min |
| Multiplier voltage: | Autotune voltage |

QuEChERS Procedure

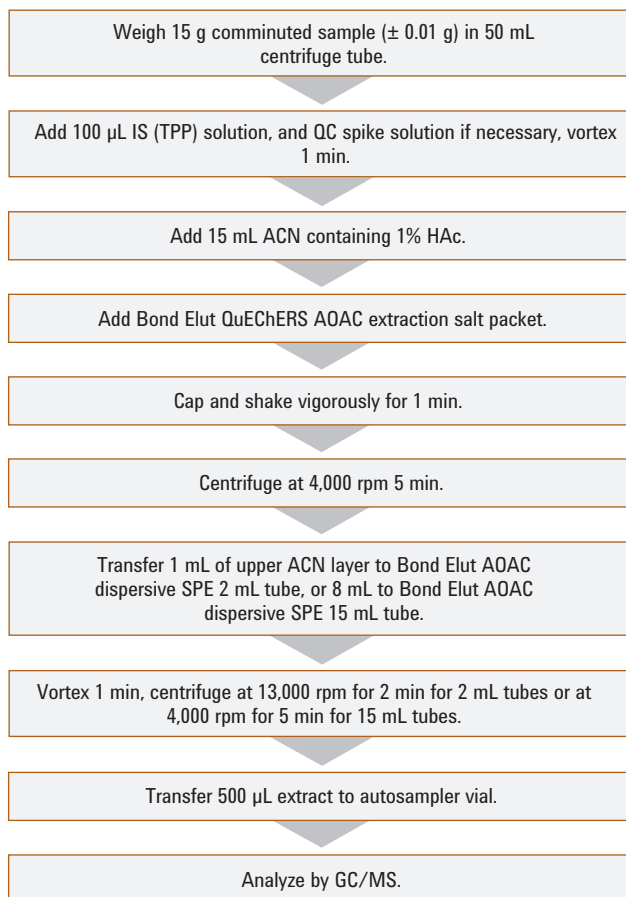


Figure 1. Agilent Bond Elut QuEChERS AOAC extraction procedure for pesticide residues in apple.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5022 or 15 mL. Part No. 5982-5058.

Agilent J&W HP-5ms Ultra Inert GC Column, 30 m x 0.25 mm, 0.25 µm. Part No. 19091S-433UI.

Results

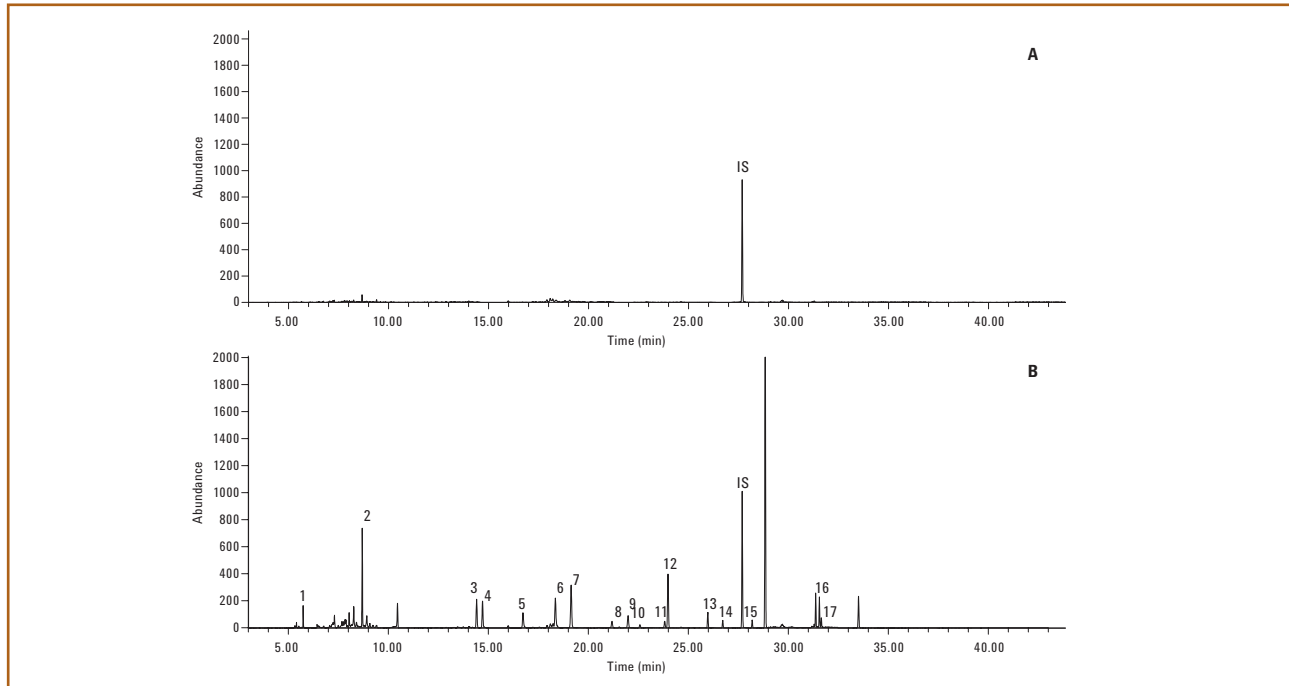


Figure 2. GC/MS chromatogram of apple extract. (A) apple extract blank; (B) 50 ng/g fortified apple extract. Peak Identification: 1. Dichlorvos, 2. *o*-Phenylphenol, 3. Diazinon, 4. Chlorothalonil, 5. Carbaryl, 6. Dichlofluanid, 7. Dichlorobenzophenone, 8. Folpet, 9. γ -Chlordane, 10. Endosulfan, 11. Dieldrin, 12. DDE, 13. Ethion, 14. Endosulfan sulfate, 15. Endrin ketone, 16. Permethrin, 17. Coumaphos. IS: Triphenyl phosphate (TPP).

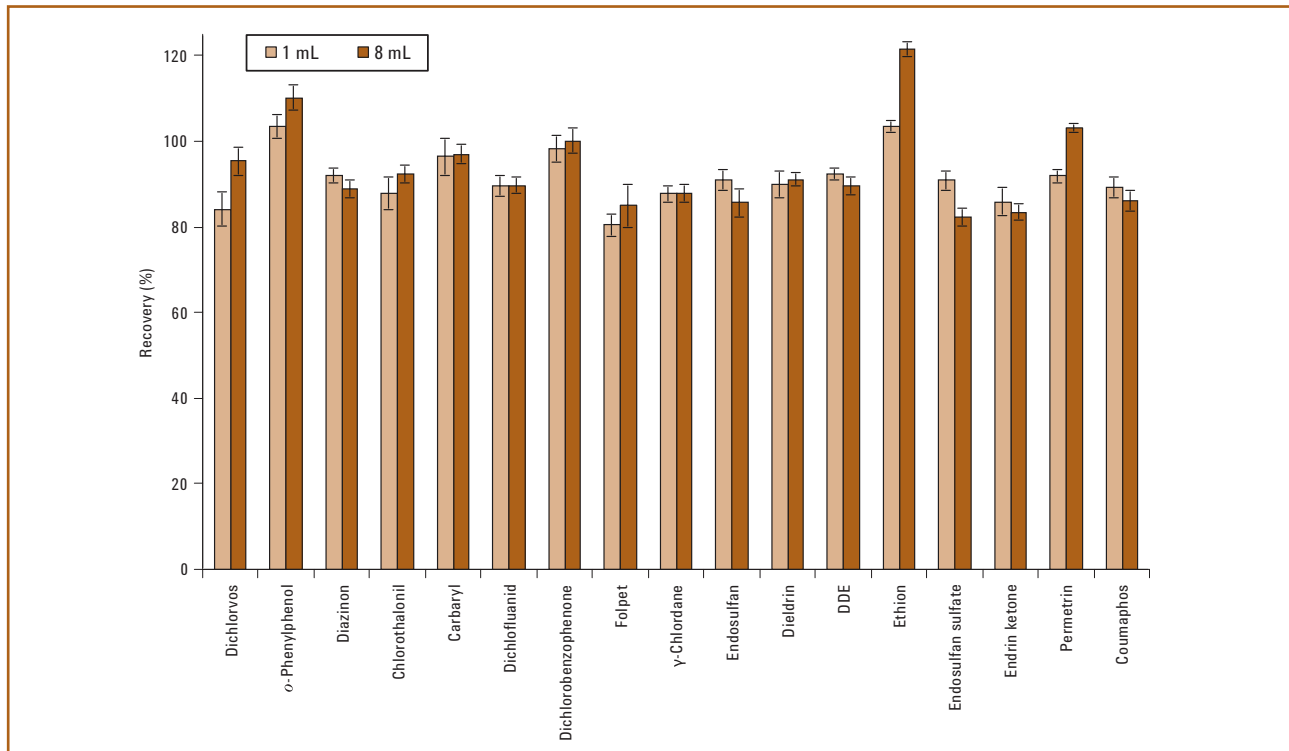


Figure 3. Exceptional recoveries and precision for 1 and 8 mL sample volumes employing Agilent Bond Elut dispersive SPE, 2 and 15 mL kits, respectively.

To review this Application Note in its entirety, please search for 5990-4068EN at agilent.com/chem

Analysis of Pesticide Residues in Spinach using Agilent Bond Elut QuEChERS AOAC Kits by GC/MS (Publication 5990-4305EN)

Introduction

This application describes the use of the QuEChERS AOAC sample preparation approach for extraction and cleanup of 18 GC-amenable multiple pesticide class residues in spinach. To address the significant loss of planar pesticides caused by graphitized carbon black (GCB) in dispersive SPE, a modified method with addition of toluene was employed for the planar pesticides. The target pesticides in the spinach extracts were then analyzed by gas chromatography/mass spectrometry (GC/MS) operating in selective ion monitoring (SIM) mode.

Instrument conditions

GC conditions

| | |
|----------------------------|---|
| Column: | Agilent J&W HP-5ms Ultra Inert, 15 m x 0.25 mm, 0.25 μ m (Part No. 19091S-431UI) |
| Inlet: | Splitless |
| Inlet liner: | Helix double-taper, deactivated (Part No. 5188-5398) |
| Carrier gas: | Helium |
| Inlet pressure: | 19.6 psi (constant pressure mode) during run 1.0 psi during backflush |
| Inlet temperature: | 250 $^{\circ}$ C |
| Injection volume: | 1.0 μ L |
| Purge flow to split vent: | 30 mL/min at 0.75 min |
| Oven temperature program: | 70 $^{\circ}$ C (1 min), 50 $^{\circ}$ C/min to 150 $^{\circ}$ C (0 min), 6 $^{\circ}$ C/min to 200 $^{\circ}$ C (0 min), 16 $^{\circ}$ C/min to 280 $^{\circ}$ C (6 min) |
| Post run: | 3 min |
| Capillary flow technology: | Purged Ultimate Union (Part No. G3186B) – used for backflushing the analytical column and inlet |
| Aux EPC gas: | Helium plumbed to Purged Ultimate Union |
| Aux EPC pressure: | 4.0 psi during run, 80.0 psi during backflush |
| Connections: | Between inlet and Purged Ultimate Union (Part No. G3186B) |
| Restrictor: | 65 cm x 0.15 mm, 0.15 μ m DB-5ms Ultra Inert |
| Connections: | Between the Purged Ultimate Union and the MSD |

MS conditions

| | |
|---|--|
| Tune file | Atune.u |
| Mode | SIM |
| Source, quad, transfer line temperature | 230 $^{\circ}$ C, 150 $^{\circ}$ C, and 280 $^{\circ}$ C, respectively |
| Solvent delay | 2.30 min |
| Multiplier voltage | Autotune voltage |

QuEChERS Procedure

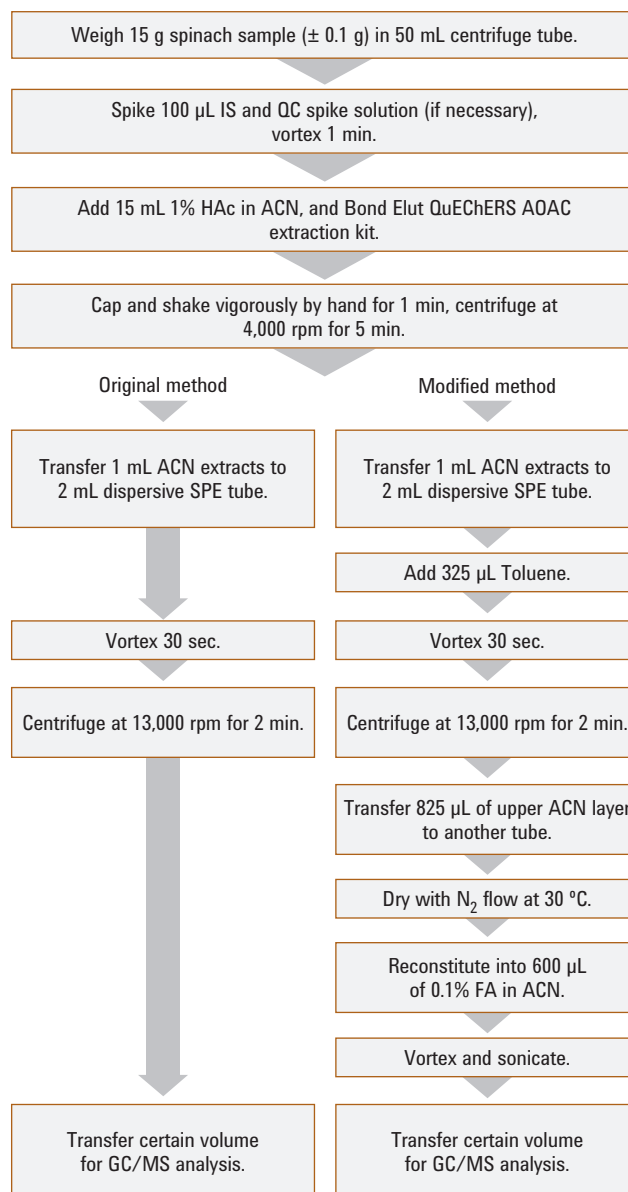


Figure 1. QuEChERS AOAC extraction procedure (original and modified dispersive SPE, 2 mL size) for spinach sample.

Results

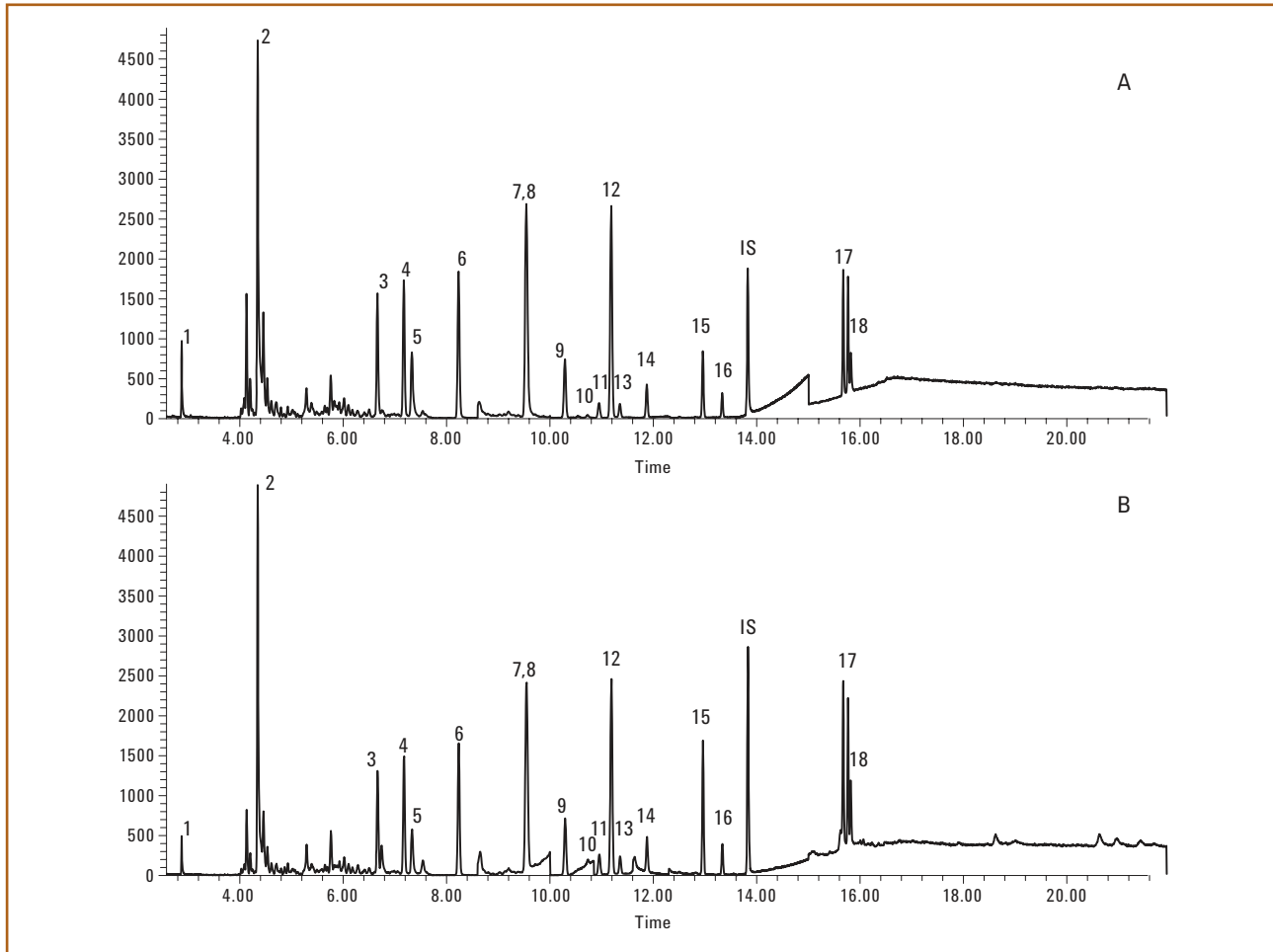


Figure 2. GC/MS chromatograms of 50 ng/g fortified spinach sample extracts processed by original dispersive SPE (A) and modified dispersive SPE (B). Peak identification: 1. Dieldrin, 2. *o*-Phenylphenol, 3. Lindane, 4. Diazinon, 5. Chlorothalonil 6. Chlorpyrifos methyl 7. Dichlorobenzophenone, 8. Chlorpyrifos, 9. Heptachlor epoxide, 10. Folpet, 11. α -Chlordane, 12. DDE, 13. γ -Chlordane, 14. Dieldrin, 15. Ethion, 16. Endosulfan sulfate, 17. Permethrin, 18. Coumaphos. IS: Internal Standard, TPP.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 2 mL. Part No. 5982-5222 or 15 mL. Part No. 5982-5258.

Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm, 0.25 μ m. Part No. 19091S-431UI.

Table 1. Spinach analysis using AOAC dispersive SPE, 1 mL sample volume, 2 mL tube, LC/MS/MS results

| Pesticide | Low QC (10 ng/g) | | Mid QC (50 ng/g) | | High QC (200 ng/g) | |
|------------------------|------------------|-----|------------------|------|--------------------|-----|
| | Recovery | RSD | Recovery | RSD | Recovery | RSD |
| Dichlorvos | 94.0 | 3.0 | 91.7 | 10.5 | 80.9 | 4.6 |
| <i>o</i> -Phenylphenol | 95.0 | 2.2 | 92.0 | 7.9 | 78.7 | 3.8 |
| Lindane | 83.7 | 3.1 | 93.9 | 12.2 | 91.8 | 3.3 |
| Diazinon | 97.3 | 4.3 | 95.6 | 9.9 | 91.8 | 3.3 |
| Chlorothalonil* | 47.5 | 6.8 | 44.9 | 6.6 | 49.4 | 4.3 |
| Chlorpyrifos methyl | 74.1 | 4.6 | 71.7 | 4.5 | 72.2 | 5.8 |
| Dichlorobenzo phenone* | 97.5 | 7.6 | 66.8 | 3.9 | 68.8 | 6.8 |
| Chlorpyrifos | 88.3 | 3.0 | 79.6 | 3.5 | 77.0 | 3.5 |
| Heptachlor epoxide | 74.9 | 1.9 | 81.6 | 11.7 | 78.2 | 3.9 |
| Folpet* | NA | NA | 98.8 | 6.0 | 77.7 | 6.7 |
| γ -Chlordane | 106.0 | 4.9 | 112.2 | 3.3 | 93.6 | 5.3 |
| DDE | 80.3 | 2.2 | 86.8 | 9.6 | 75.4 | 3.5 |
| α -Chlordane | 107.6 | 4.2 | 108.4 | 3.5 | 91.6 | 3.7 |
| Dieldrin | 99.7 | 2.6 | 93.7 | 9.6 | 78.9 | 3.4 |
| Ethion | 91.4 | 3.4 | 100.0 | 5.0 | 107.4 | 7.6 |
| Endosulfan sulfate | 93.7 | 4.8 | 97.3 | 8.8 | 89.8 | 4.3 |
| Permethrin | 84.7 | 5.7 | 74.8 | 9.9 | 84.6 | 6.0 |
| Coumaphos* | 98.4 | 5.5 | 84.2 | 9.5 | 81.2 | 3.2 |

*Results from modified dispersive SPE method.



To review this Application Note in its entirety, please search for 5990-4305EN at agilent.com/chem

Analysis of Pesticide Residues in Spinach using Agilent Bond Elut QuEChERS AOAC Kit by LC/MS/MS Detection (Publication 5990-4248EN)

Introduction

This application describes the use of the QuEChERS AOAC sample preparation approach for the extraction and cleanup of 13 pesticide residues representing various pesticide classes in spinach. To address the significant loss of planar pesticides caused by graphitized carbon black (GCB) in dispersive SPE, a modified method with the addition of toluene was employed. With the combination of original and modified dispersive SPE, the method was validated in terms of recovery and reproducibility for all of the analytes of interest.

Instrument conditions

HPLC conditions

| | | | |
|---------------------|--|------|--------------------|
| Column: | Agilent ZORBAX Solvent Saver Eclipse Plus Phenyl-Hexyl, 3.0 x 150 mm, 3.5 µm (Part No. 959963-312) | | |
| Flow rate: | 0.3 or 0.5 mL/min | | |
| Column temperature: | 30 °C | | |
| Injection volume: | 10 µL | | |
| Mobile phase: | A: 5 mM NH ₄ OAc, pH 5.0 in 20:80 MeOH/H ₂ O B: 5 mM NH ₄ OAc, pH 5.0 in ACN | | |
| Needle wash: | 1:1:1 ACN:MeOH:Isopropyl alcohol (IPA):H ₂ O w/0.2% FA | | |
| Gradient: | Time (min) | % B | Flow rate (mL/min) |
| | 0 | 20 | 0.3 |
| | 0.5 | 20 | 0.3 |
| | 8.0 | 100 | 0.3 |
| | 10.0 | 100 | 0.3 |
| | 10.01 | 20 | 0.5 |
| | 13.0 | STOP | |
| Post run: | 4 min | | |
| Total cycle time: | 17 min | | |

MS conditions

Positive mode

| | |
|------------------|----------|
| Gas temperature: | 350 °C |
| Gas flow: | 10 L/min |
| Nebulizer: | 40 psi |
| Capillary: | 4,000 V |

QuEChERS Procedure

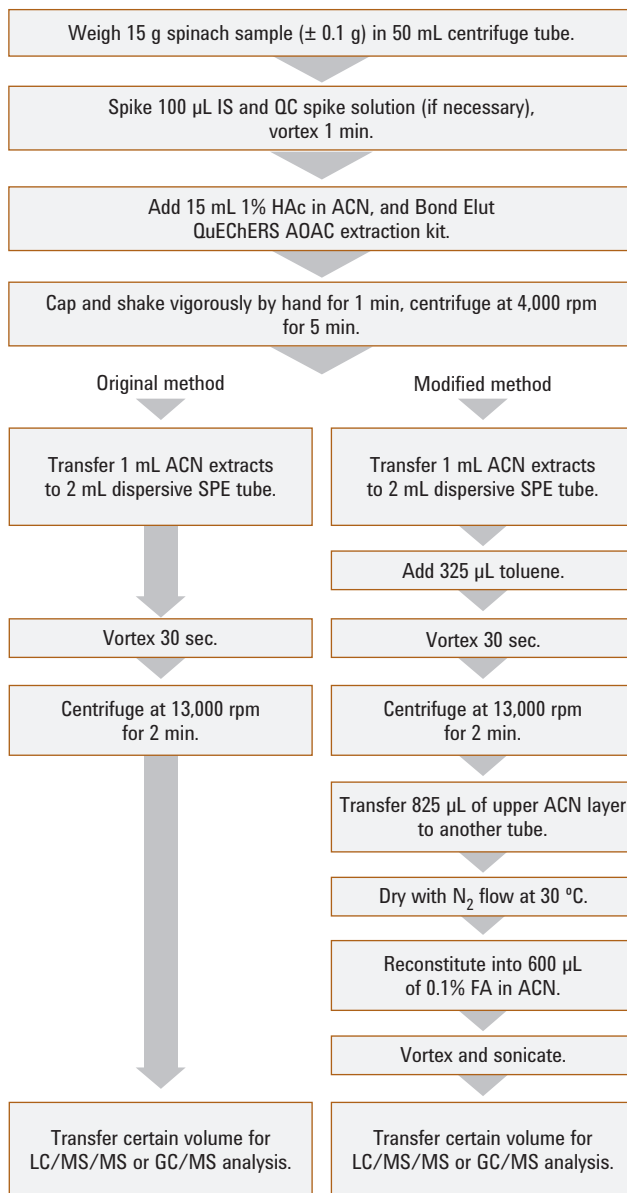


Figure 1. QuEChERS AOAC extraction procedure (original and modified dispersive SPE, 2 mL size) for a spinach sample.

Results

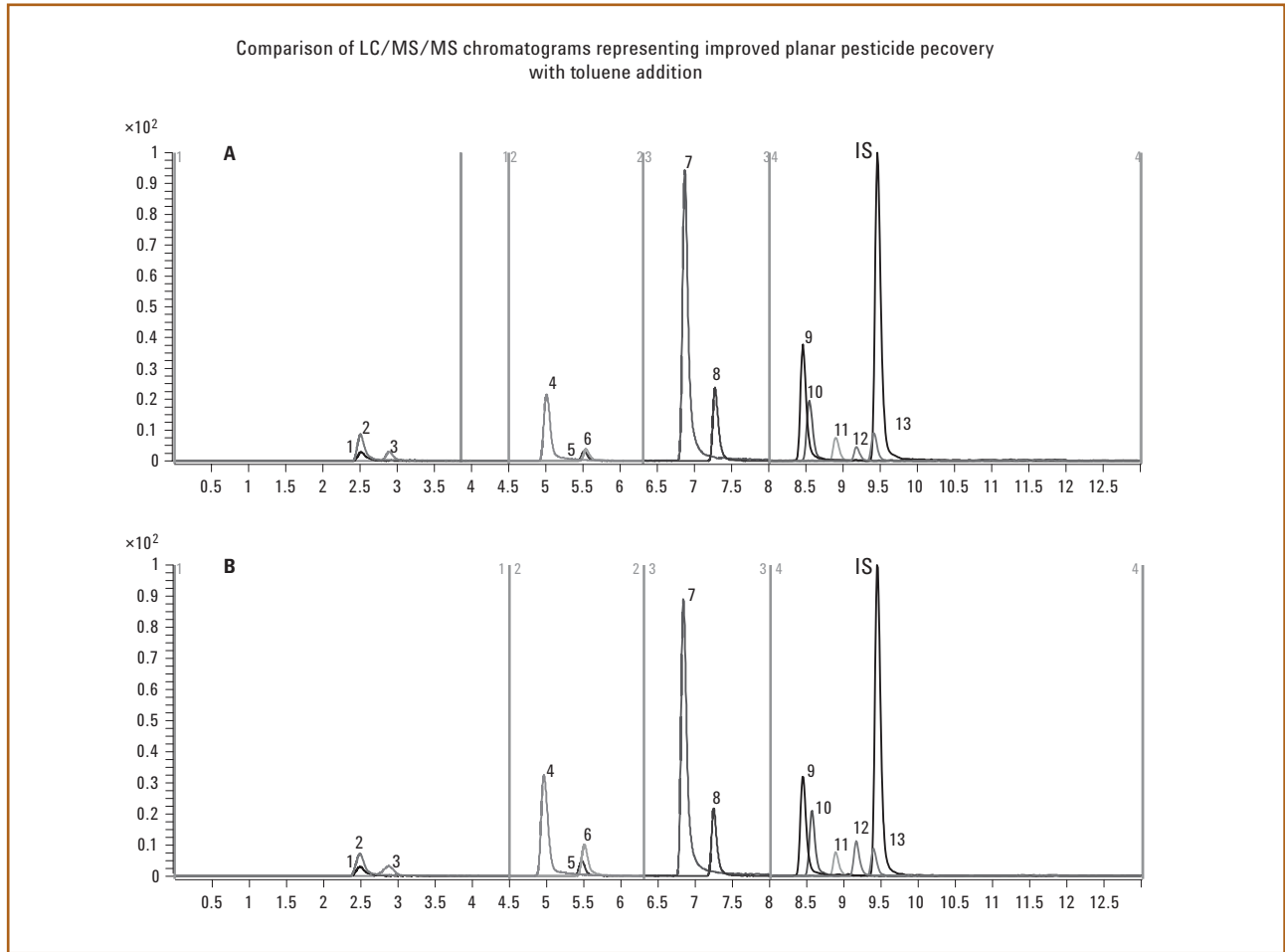


Figure 2. LC/MS/MS chromatograms of 50 ng/g fortified spinach sample extracts processed by original dispersive SPE (A) and modified dispersive SPE (B). Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Propoxur, 8. Carbaryl, 9. Ethoprophos, 10. Imazalil, 11. Penconazole, 12. Cyprodinil, 13. Kresoxim methyl IS: Internal Standard, TPP.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 2 mL. Part No. 5982-5222 or 15 mL. Part No. 5982-5258.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 μ m. Part No. 959963-312.

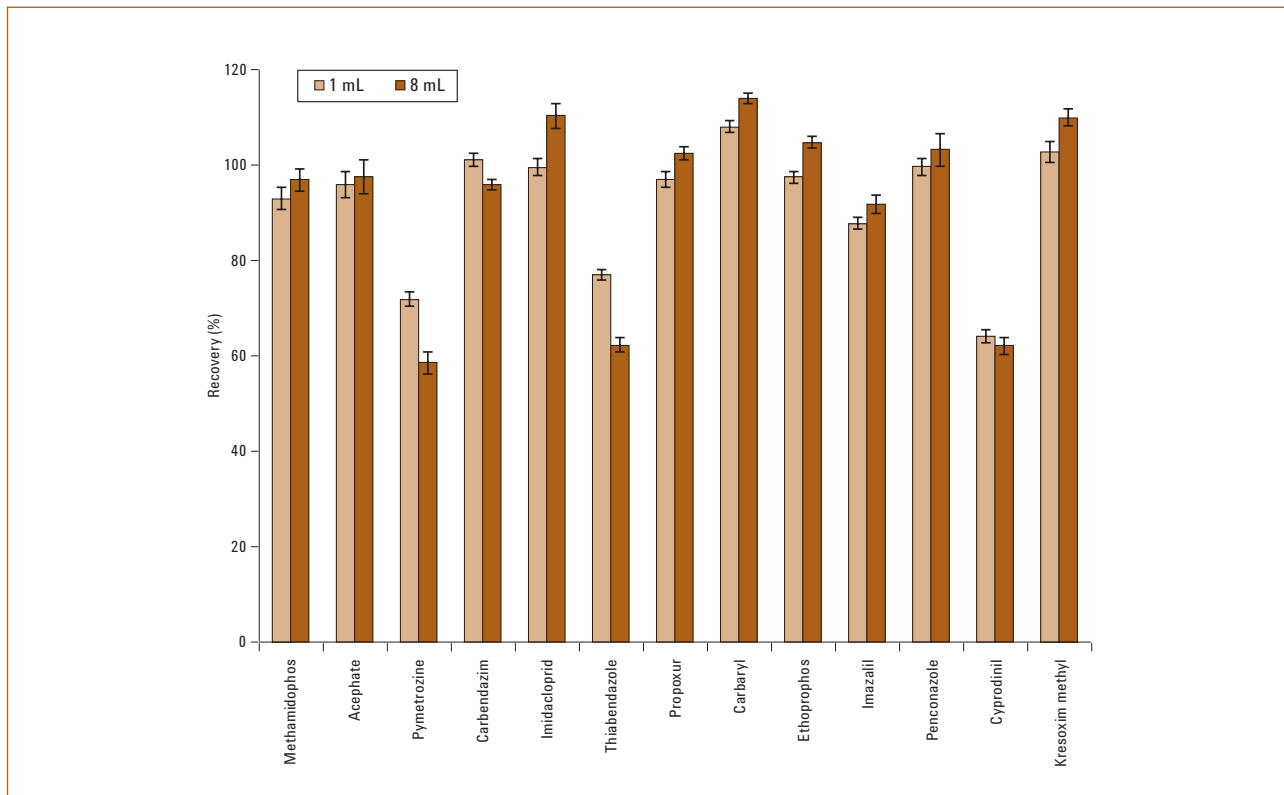


Figure 3. Recovery and precision results for 1 mL dispersive SPE and 8 mL dispersive SPE.



Optimizing Recoveries of Planar Pesticides in Spinach using Toluene and Agilent Bond Elut QuEChERS AOAC Kits with Graphitized Carbon (Publication 5990-4247EN)

Introduction

This application describes the impact of toluene addition in the dispersive solid phase extraction (SPE) step on the analysis of pesticides in spinach using Agilent Bond Elut QuEChERS AOAC kits for highly pigmented fruits and vegetables. With the modified AOAC method, the eight problematic pesticides generated substantially improved recoveries, 50% to 300%, and < 10% RSD. Using GC/MS and LC/MS/MS, the method provides an alternative extraction process for challenging pesticides in highly pigmented foods.

QuEChERS Procedure

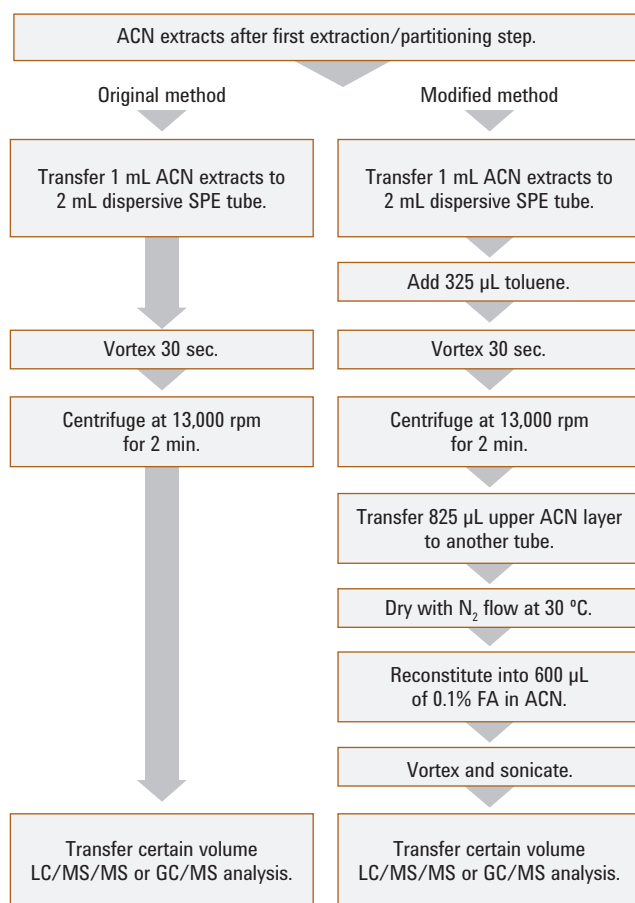


Figure 1. Dispersive SPE procedures of original method (without toluene) and modified method (with toluene).

Instrument conditions

HPLC conditions

| | | | |
|---------------------|--|------|--------------------|
| Column: | Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl, 3.0 x 150 mm, 3.5 µm (Part No. 959963-312) | | |
| Flow rate: | 0.3 or 0.5 mL/min | | |
| Column temperature: | 30 °C | | |
| Injection volume: | 10 µL | | |
| Mobile phase: | A: 5 mM ammonium acetate, pH 5.0 in 20:80 MeOH/H ₂ O B: 5 mM ammonium acetate, pH 5.0 in ACN | | |
| Needle wash: | 1:1:1:1 ACN/MeOH/IPA/H ₂ O w/0.2% FA | | |
| Gradient: | Time (min) | % B | Flow rate (mL/min) |
| | 0 | 20 | 0.3 |
| | 0.5 | 20 | 0.3 |
| | 8.0 | 100 | 0.3 |
| | 10.0 | 100 | 0.3 |
| | 10.01 | 20 | 0.5 |
| | 13.0 | STOP | |
| Post run: | 4 min | | |
| Total cycle time: | 17 min | | |

GC conditions

| | |
|----------------------------|---|
| Column: | Agilent J&W HP-5ms Ultra Inert GC, 15 m x 0.25 mm, 0.25 µm (Part No. 19091S-431UI) |
| Inlet: | Splitless |
| Inlet liner: | Helix double-taper, deactivated (Part No. 5188-5398) |
| Carrier gas: | Helium |
| Inlet pressure: | 19.6 psi (constant pressure mode) during run 1.0 psi during back flush |
| Inlet temperature: | 250 °C |
| Injection volume: | 1.0 µL |
| Purge flow to split vent: | 30 mL/min at 0.75 min |
| Oven temperature program: | 70 °C (1 min), 50 °C/min to 150 °C (0 min), 6 °C/min to 200 °C (0 min), 16 °C/min to 280 °C (6 min) |
| Post run: | 3 min |
| Capillary flow technology: | Purged Ultimate Union (Part No. G3186B) – used for backflushing the analytical column and inlet |
| Aux EPC gas: | Helium plumbed to Purged Ultimate Union |
| Aux EPC pressure: | 4.0 psi during run, 80.0 psi during backflush |
| Connections: | Between inlet and Purged Ultimate Union (Part No. G3186B) |
| Restrictor: | 65 cm x 0.15 mm, 0.15 µm DB-5ms Ultra Inert |
| Connections: | Between the Purged Ultimate Union and the MSD |

Results

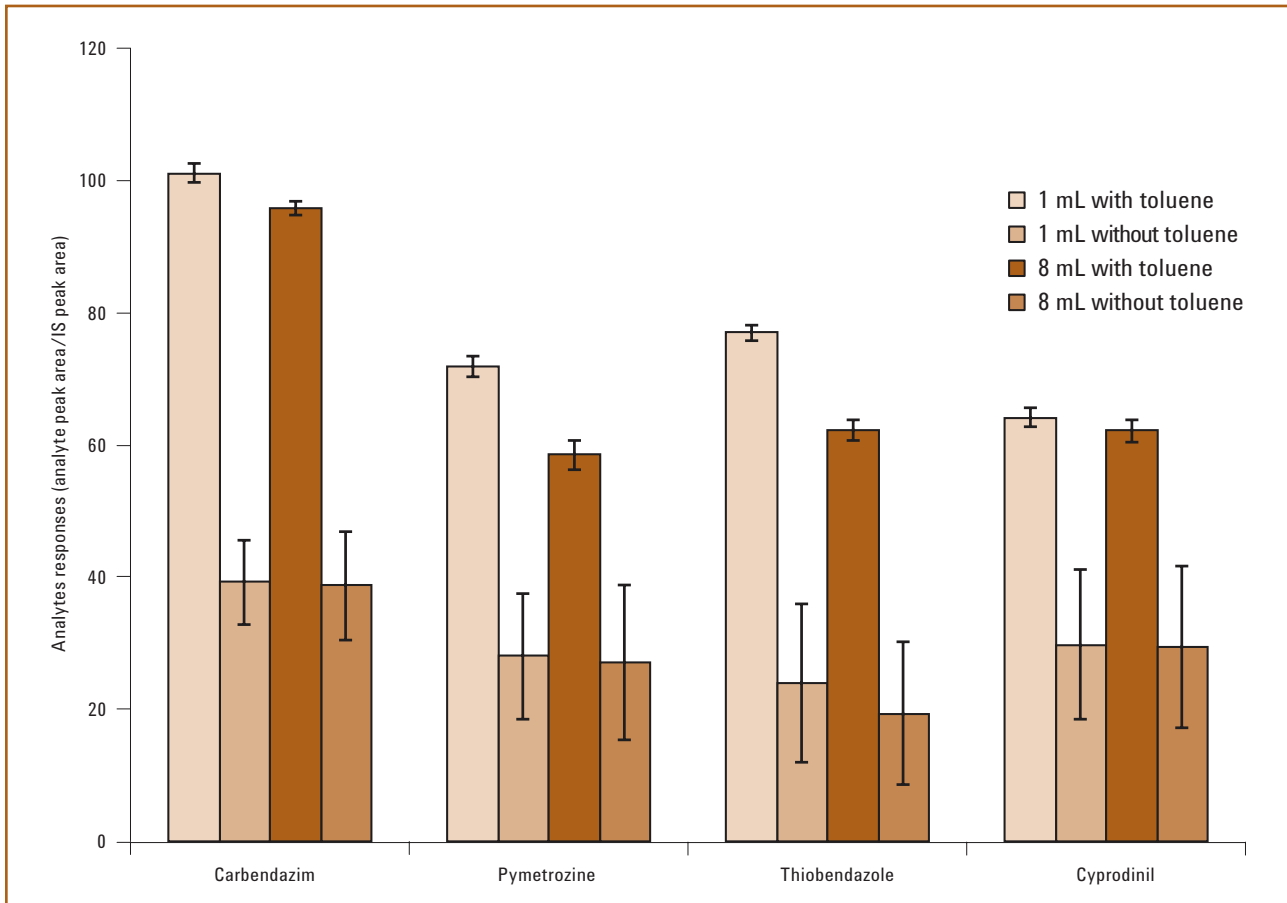


Figure 2. Results comparison of 1 mL and 8 mL dispersive SPE with the modified method (with toluene) and the original method (without toluene).

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables,
2 mL. Part No. 5982-5222 or 15 mL. Part No. 5982-5258.

Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm x 0.25 μ m. Part No. 19091S-431UI.

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 μ m. Part No. 959963-312.

Table 1. Effects on certain pesticides using a modified dispersive SPE cleanup with addition of toluene

| Analytes | Original method (without toluene) | | Modified method (with toluene) | | Impact with modified method | Detection method |
|----------------------|-----------------------------------|-----------|--------------------------------|-----------|-----------------------------|------------------|
| | Recovery | RSD (n=6) | Recovery | RSD (n=6) | | |
| Carbendazim | 38.9 | 14.6 | 98.5 | 2.5 | Positive | LC/MS/MS |
| Thiabendazole | 21.8 | 19.7 | 69.7 | 2.7 | Positive | LC/MS/MS |
| Pymetrozine | 27.6 | 21.2 | 65.2 | 3.7 | Positive | LC/MS/MS |
| Cyprodinil | 29.6 | 23.4 | 63.1 | 3.2 | Positive | LC/MS/MS |
| Chlorthalonil | 21.1 | 16.4 | 47.3 | 5.9 | Positive | GC/MS |
| Coumaphos | 30.1 | 24.0 | 87.9 | 6.1 | Positive | GC/MS |
| Dichlorobenzophenone | 53.7 | 4.5 | 77.7 | 6.1 | Positive | GC/MS |
| Folpet | 62.0 | 14.6 | 88.2 | 6.3 | Positive | GC/MS |
| Dichlorvos | 88.8 | 6.0 | 20.4 | 89.8 | Greatly negative | GC/MS |
| o-Phenylphenol | 88.6 | 4.6 | 73.7 | 7.4 | Slightly negative | GC/MS |
| Diazinon | 94.9 | 5.9 | 81.3 | 4.0 | Slightly negative | GC/MS |
| Chlordane | 103.9 | 4.5 | 101.3 | 4.5 | None | GC/MS |
| Permethrin | 81.4 | 7.2 | 83.3 | 5.1 | None | GC/MS |
| Acephate | 95.5 | 5.6 | 99.8 | 4.7 | None | LC/MS/MS |
| Carbaryl | 108.0 | 2.5 | 109.1 | 1.9 | None | LC/MS/MS |
| Propoxur | 97.0 | 3.19 | 6.7 | 2.5 | None | LC/MS/MS |



To review this Application Note in its entirety, please search for 5990-4247EN at agilent.com/chem

Determination of Pesticides in Baby Food by UHPLC/MS/MS using the Agilent 1290 Infinity LC and the Agilent 6460 Triple Quadrupole LC/MS (Publication 5990-5028EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach for extraction and cleanup of 40 pesticides in baby food at levels below the maximum residue level (MRL) (10 µg/kg fruit or vegetable) specified in EC Regulation 396/2005. The qualitative and quantitative analysis of pesticides at trace levels in baby food matrixes using UHPLC and triple quadrupole MS was demonstrated.

The method and extraction performance were evaluated in terms of repeatability, linearity and sensitivity. Moreover the influence of the additional dispersive SPE cleanup was investigated. Detection limits were between 500 ng/kg and 10 ng/kg (ppt), which is much lower than the MRL of 10 µg/kg (ppb) imposed by the European Union.

Instrument conditions

Method parameters

| | | |
|---------------|---|----------|
| Column: | Agilent ZORBAX Eclipse Plus RRHD C18, 2.1 x 150 mm, 1.8 µm | |
| Mobile phase: | A: 0.05% (w/v) Ammonium formate + 0.01% (v/v) Formic acid in water B: Methanol | |
| Flow rate: | 0.5 mL/min | |
| Gradient: | Time (min) | % B |
| | 0 to 5 | 10 to 65 |
| | 5 to 6.5 | 65 to 95 |
| | 6.5 to 8.5 | 95 |
| | 8.5 to 10 | 10 |
| Temperature: | 45 °C | |
| Injection: | 2 µL, with needle wash (flushport, 5 s, water/methanol 1/1) | |
| Detection: | MS/MS | |
| Ionization: | Electrospray, positive ionization | |

Jet Stream parameters

| | |
|-------------------------|----------|
| Drying gas temperature: | 250 °C |
| Drying gas flow: | 10 L/min |
| Nebulizer pressure: | 30 psig |
| Sheath gas temperature: | 340 °C |
| Sheath gas flow: | 11 L/min |
| Capillary voltage: | 4500 V |
| Nozzle voltage: | 500 V |

Acquisition

| | |
|--------------|-------------------------|
| Dynamic MRM: | See Table 1 of app note |
| Delta EMV: | 50 |
| Cycle time: | 200 ms |

QuEChERS Procedure

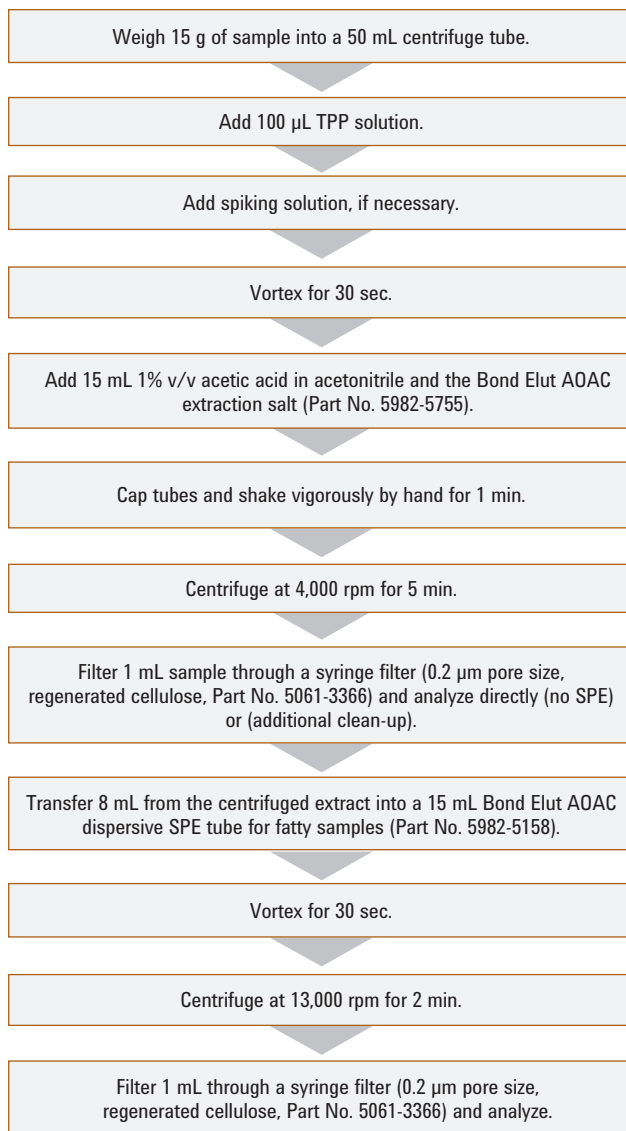


Figure 1. Agilent Bond Elut QuEChERS AOAC extraction procedure for pesticides in baby food.

Results

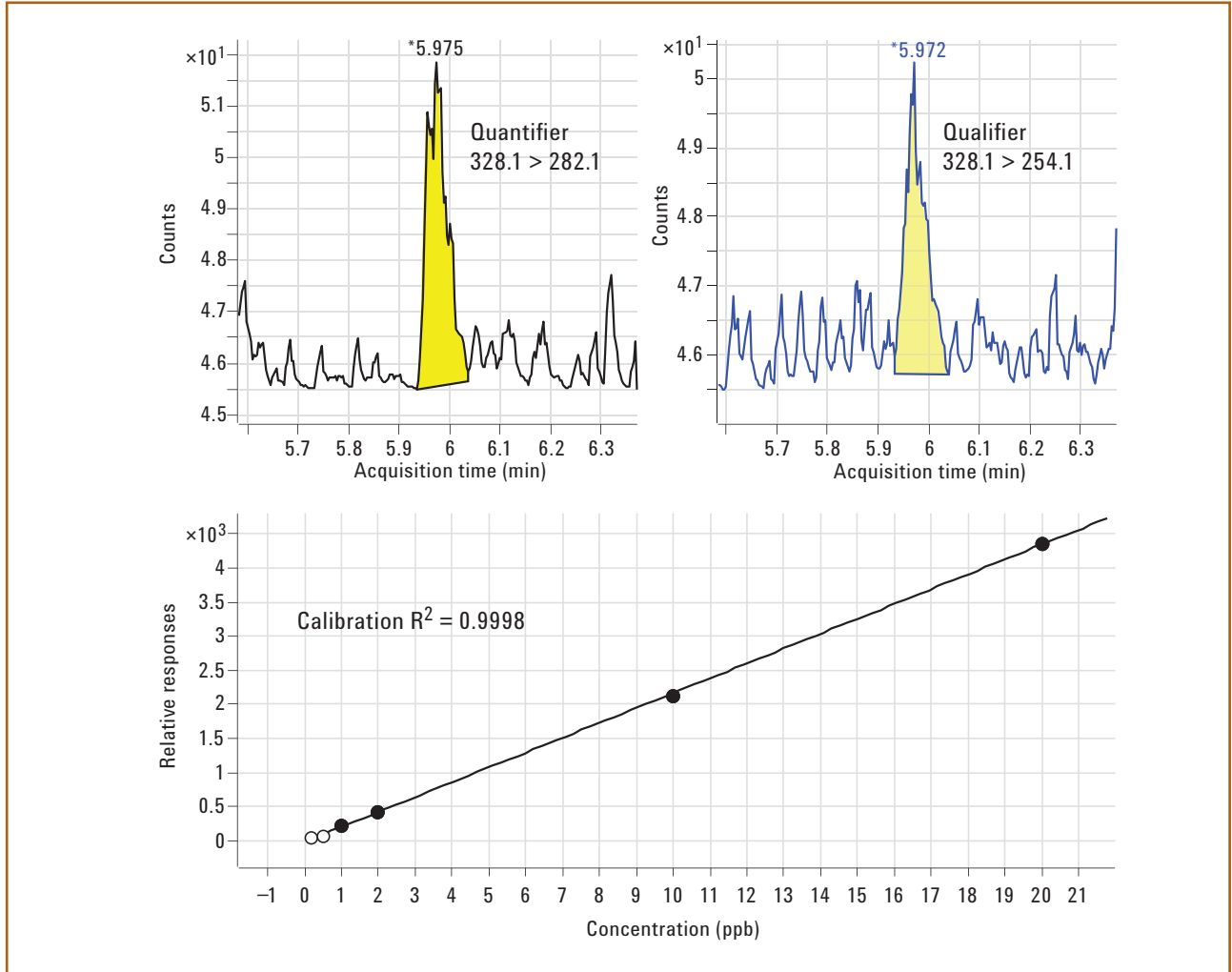


Figure 2. Ion traces for two transitions at the LOD (0.5 ppb standard solution) and calibration curve for fluzifop in baby food.



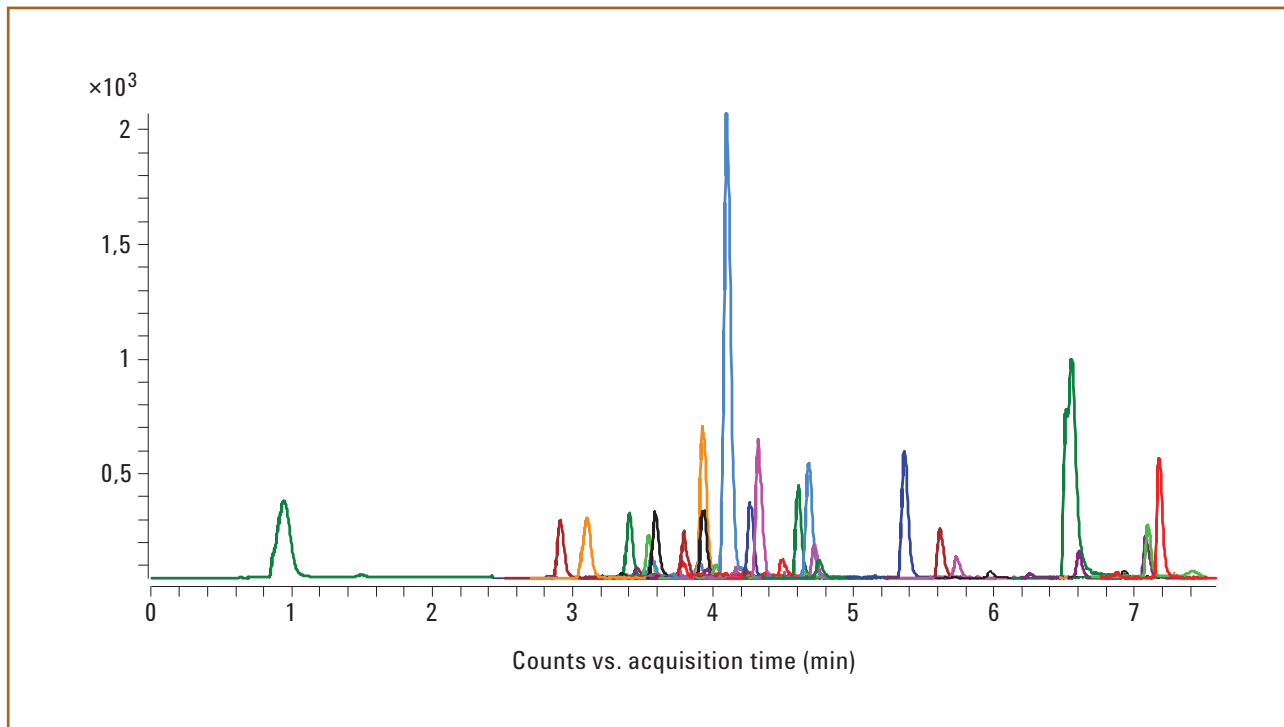


Figure 3. MRM of an extract of baby food sample 2 spiked with 1 ppb (only quantifier transitions are shown). No dispersive SPE performed on the sample. The transition for the internal standard is not shown.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut AOAC Dispersive SPE Tube for Fatty Samples. Part No. 5982-5158.

Agilent ZORBAX Eclipse Plus RRHD C18 Column, 2.1 x 150 mm, 1.8 μm . Part No. 959759-902

To review this Application Note in its entirety, please search for 5990-5028EN at agilent.com/chem

Determination of Sulfonamide Residues in Chicken Muscle by Agilent Bond Elut QuEChERS AOAC Kit and HPLC-FLD (Publication 5990-5395EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach for the determination of sulfonamide drugs in chicken muscle with HPLC-FLD after a pre-column derivatization with fluorescamine. The recoveries ranged from 76.8% to 95.2% and an HPLC-Fluorescence detection (FLD) method was developed and validated for the determination of nine sulfonamides.

The analysis was performed on an Agilent 1200 Infinity Series equipped with a binary pump and a fluorescence detector (FLD) set at $\lambda_{\text{ex}} = 405 \text{ nm}$ and $\lambda_{\text{em}} = 495 \text{ nm}$. Separation of the compounds was achieved on an Agilent ZORBAX Eclipse Plus C18 column. The data was processed by HPLC 2D ChemStation Software.

Instrument conditions

HPLC conditions

| | | |
|---------------------|--|-----|
| Column: | Agilent ZORBAX Eclipse Plus C18, 4.6 × 75 mm, 3.5 μm | |
| Flow rate: | 1 mL/min | |
| Column temperature: | 25 °C | |
| Injection volume: | 5 μL | |
| Mobile phase: | A: 0.05 M Sodium acetate pH 4.5 B: MeOH | |
| Gradient: | Time (min) | % B |
| | 0 | 35 |
| | 35 | 41 |
| | 50 | 55 |
| Detection: | Ex = 405 nm Em = 495 nm | |

QuEChERS Procedure

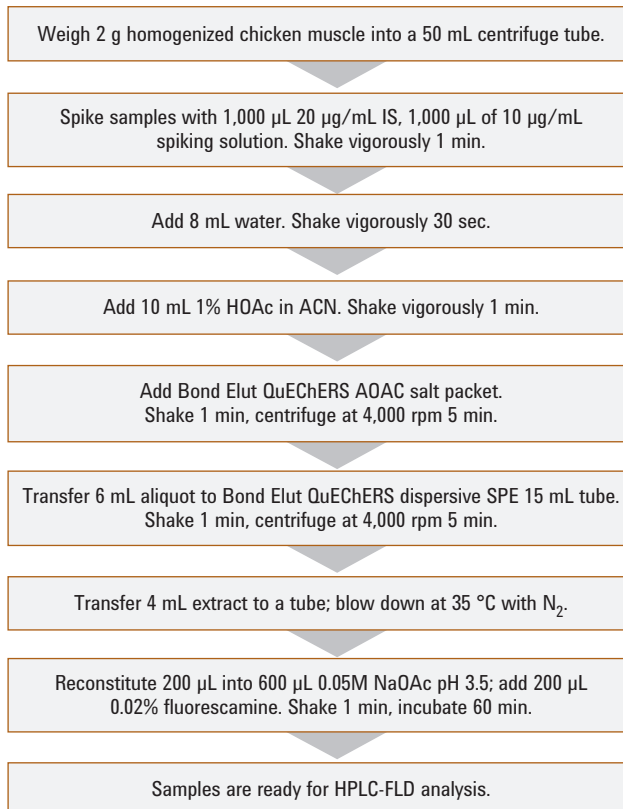


Figure 1. QuEChERS AOAC sample preparation procedure for sulfonamides in chicken.

Results

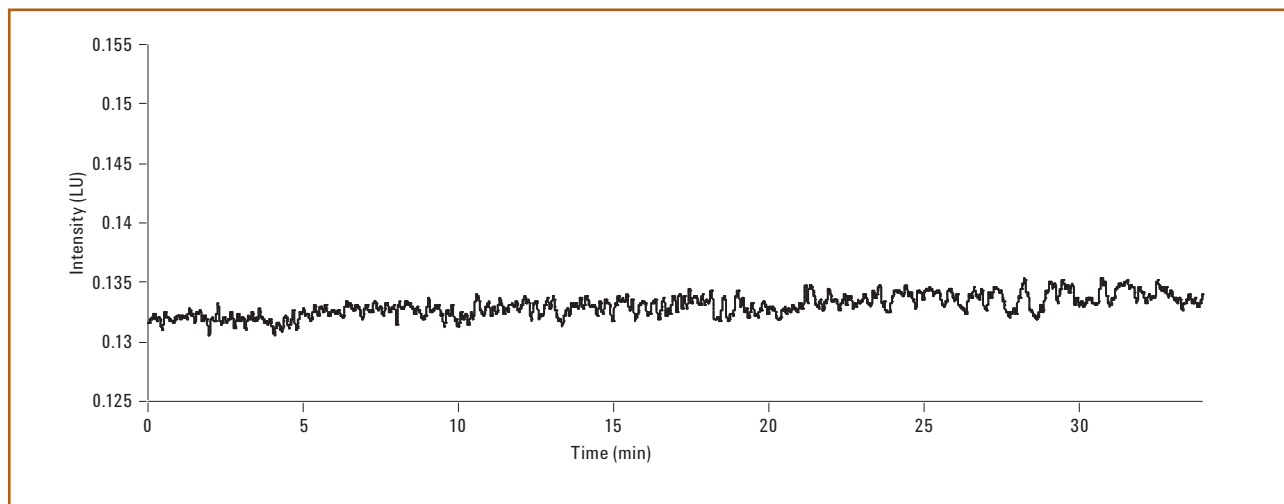


Figure 2. Chromatogram of the blank chicken muscle extract.

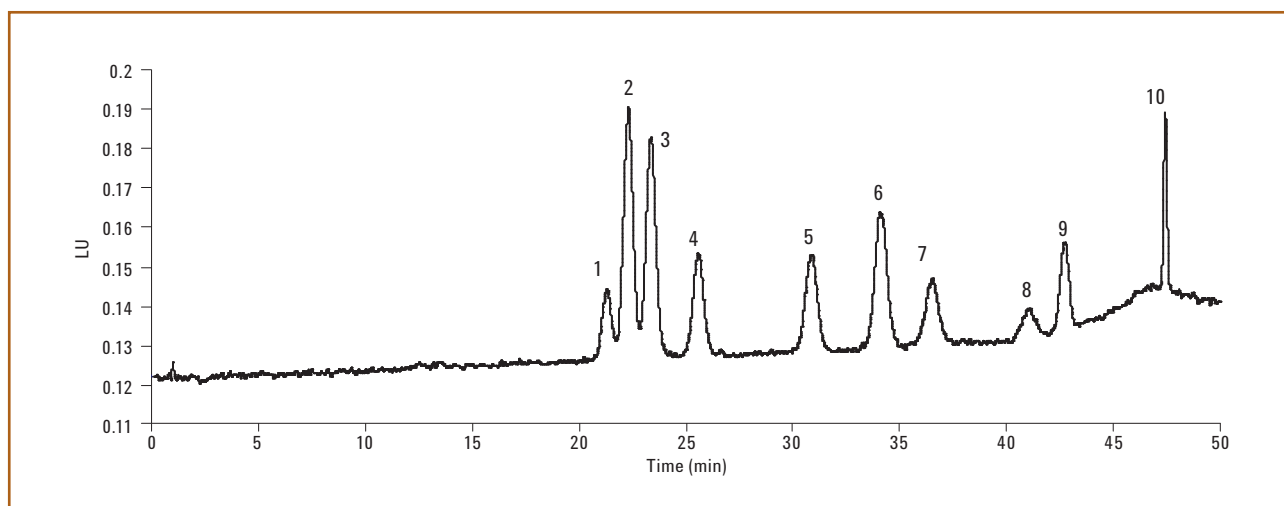


Figure 3. Chromatogram of the spiked chicken muscle extract at 50 ng/g level: 1. Sulfadiazine ; 2. Sulfathiazole; 3. Sulfapyridine (IS); 4. Sulfamerazine; 5. Sulfamethazine; 6. Sulfamethizole; 7. Sulfamethoxyppyridazine; 8. Sulfachloropyridazine; 9. Sulfamethoxazole; 10. Sulfadimethoxine.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit. Part No. 5982-5158.

Agilent ZORBAX Eclipse Plus C18 Column, 4.6 x 75 mm, 3.5 μ m. Part No. 959933-902.

To review this Application Note in its entirety, please search for 5990-5395EN at agilent.com/chem

Analysis of Polycyclic Aromatic Hydrocarbons in Fish with Agilent Bond Elut QuEChERS AOAC Kit and HPLC-FLD (Publication 5990-5441EN)

Introduction

This application describes the use of the QuEChERS AOAC sample preparation approach for the determination of sixteen polycyclic aromatic hydrocarbons (PAHs) in fish fillets. The analyte recoveries ranged from 83.4% to 101% with relative standard deviations ranging from 0.6 to 1.9% at three different fortification levels.

The analysis was performed on an Agilent 1200 Infinity Series equipped with a binary pump and a fluorescence detector (FLD) and data was processed by HPLC 2D ChemStation software. High recoveries with excellent precision were attained. Therefore, this method is suitable for quality control testing for PAHs in real samples.

Instrument conditions

HPLC conditions

Column: Agilent ZORBAX Eclipse PAH C18, 4.6 × 50 mm, 1.8 μm

Flow rate: 0.8 mL/min

Column temperature: 18 °C

Injection volume: 5 μL

Mobile phase: A: Deionized H₂O
B: CH₃CN

| Gradient | Time (min) | % B |
|----------|------------|-----|
| | 0 | 60 |
| | 1.5 | 60 |
| | 7 | 90 |
| | 13 | 100 |

Detection: UV at 230 nm (Acy) and varying fluorescence excitation (Ex) and emission (Em) wavelengths

Wavelengths:

| Time (min) | Ex/Em wavelengths (nm) | PAH detected |
|---------------------|------------------------|----------------------------|
| 0 - 5 (dark blue) | 260/352 | Nap, Ace, Flu, Phe, Chr |
| 0 - 14 (red) | 260/420 | Ant, Pyr, BeP, DahA, BghiP |
| 0 - 14 (light blue) | 260/460 | Fln, 1,2-BaA,BeA, BkF, InP |

QuEChERS Procedure

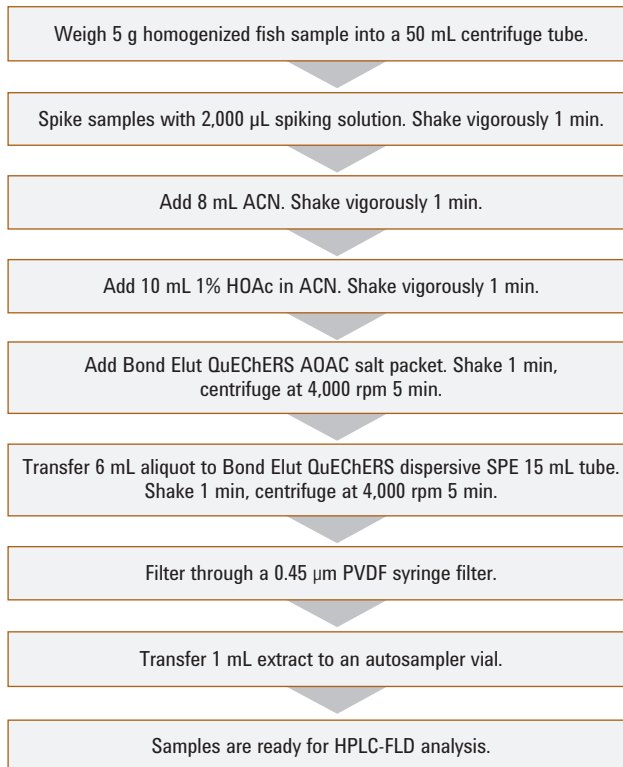


Figure 1. QuEChERS AOAC sample preparation procedure for PAHs in fish.

Results

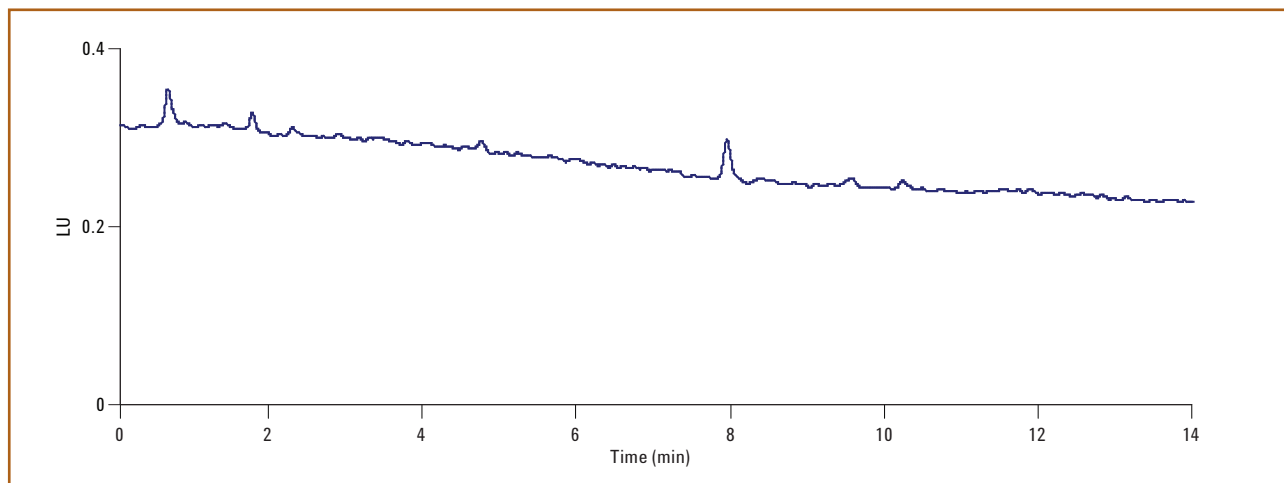


Figure 2. Chromatogram of the blank fish extract. Chromatographic conditions are shown in Table 1 of the app note. The baseline chromatogram used the following excitation/emission wavelengths: 260-nm/352-nm. The other excitation/emission conditions showed no other interferences.

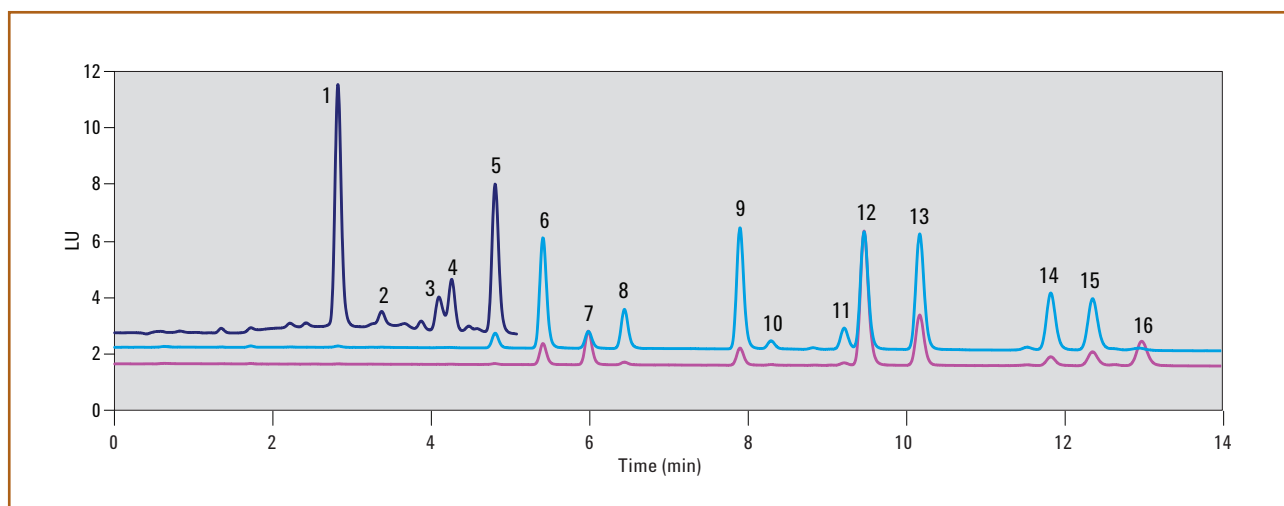


Figure 3. Overlay HPLC – FLD chromatograms of the spiked fish sample containing: 1. Nap; 2. Acy; 3. Ace; 4. Flu; 5. Phe; 6. Ant; 7. Fln; 8. Pyr; 9. BaA; 10. Chr; 11. BeP; 12. BeA; 13. BkF; 14. DahA; 15. BghiP; 16. InP. The spiking level for this sample was level 1 (see Table 3 of the app note). The blue portion of the chromatogram used the following excitation/emission wavelengths: 260-nm/352-nm; the red portion: 260-nm/420-nm; the light blue portion: 260-nm/440-nm. For acenaphthylene, UV detection at 230-nm was used.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut AOAC Fatty Dispersive SPE Kit, 15 mL. Part No. 5982-5158.

Agilent ZORBAX Eclipse PAH C18 Column, 4.6 × 50 mm, 1.8 μm. Part No. 959941-918.

To review this Application Note in its entirety, please search for 5990-5441EN at agilent.com/chem

Analysis of Acrylamide in Fried Potatoes using Agilent Bond Elut QuEChERS and LC/MS/MS (Publication 5990-5940EN)

Introduction

This application describes the use of a QuEChERS Acrylamide extraction step combined with an AOAC dSPE cleanup sample preparation approach for the analysis of acrylamide in fried potatoes, using LC/MS/MS for detection and $^{13}\text{C}_3$ -acrylamide as the internal standard. The acrylamide recoveries ranged from 97% to 116%. An Agilent 1200 Infinity Series equipped with an Agilent 6460 Triple Quadrupole LC/MS was used to perform the analysis.

A simple and fast method based on Agilent Bond Elut QuEChERS for acrylamide extraction and cleanup with LC/MS/MS analysis was developed with high extraction yields and excellent precision.

Instrument conditions

LC/MS/MS conditions

| | |
|-------------------------|--|
| Column: | Reversed C18, 2.1 × 150 mm, 3 μm |
| Column temperature: | 30 °C |
| Isocratic mode (%B): | 2.5% Methanol/97.5% of 0.1% Formic acid |
| Flow rate: | 0.2 mL/min |
| Injection volume: | 10 μL |
| Run time: | 7 min |
| Post run time: | 3 min |
| Mass spectrometer: | Positive electrospray ionization mode with jet stream technology |
| Capillary voltage: | 4,000 |
| Nozzle voltage: | 500 V |
| Sheath gas temperature: | 325 °C at 5 L/min |
| Drying gas temperature: | 350 °C at 11 L/min |

QuEChERS Procedure

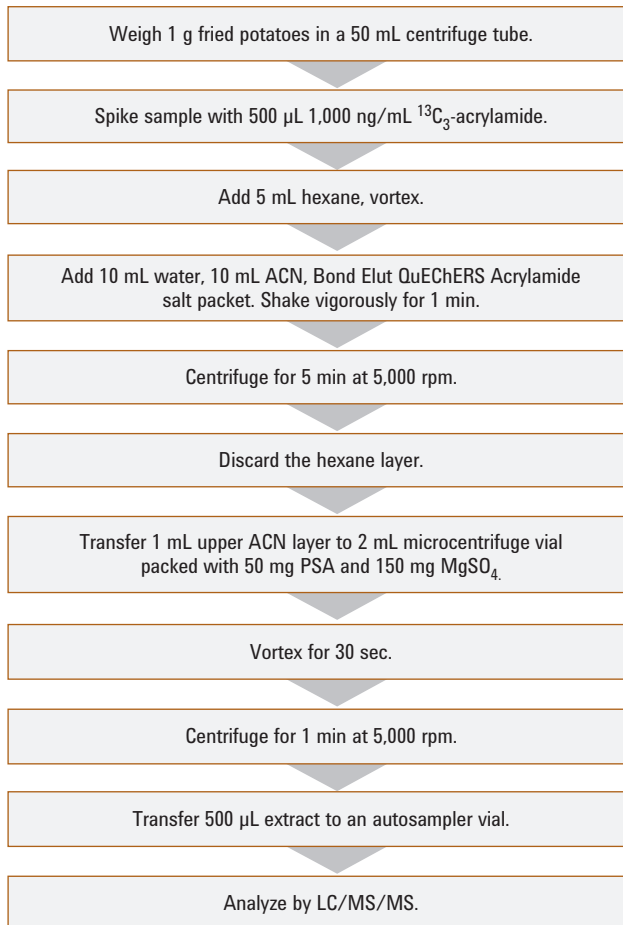
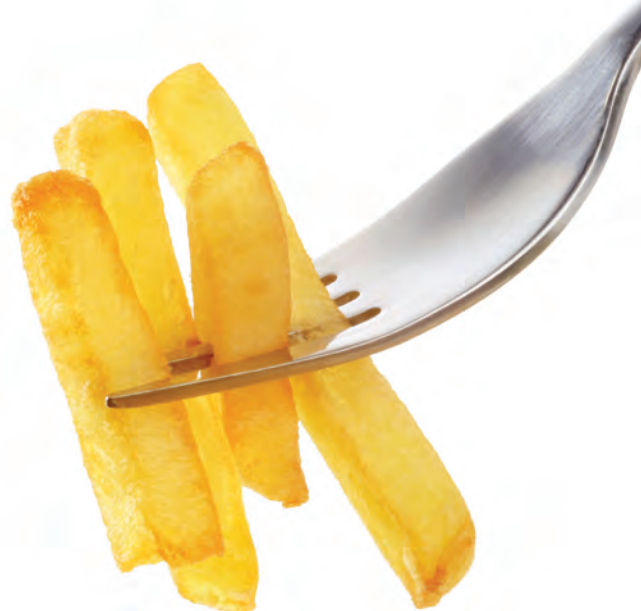


Figure 1. QuEChERS sample preparation procedure for acrylamides in fried potatoes.



Results

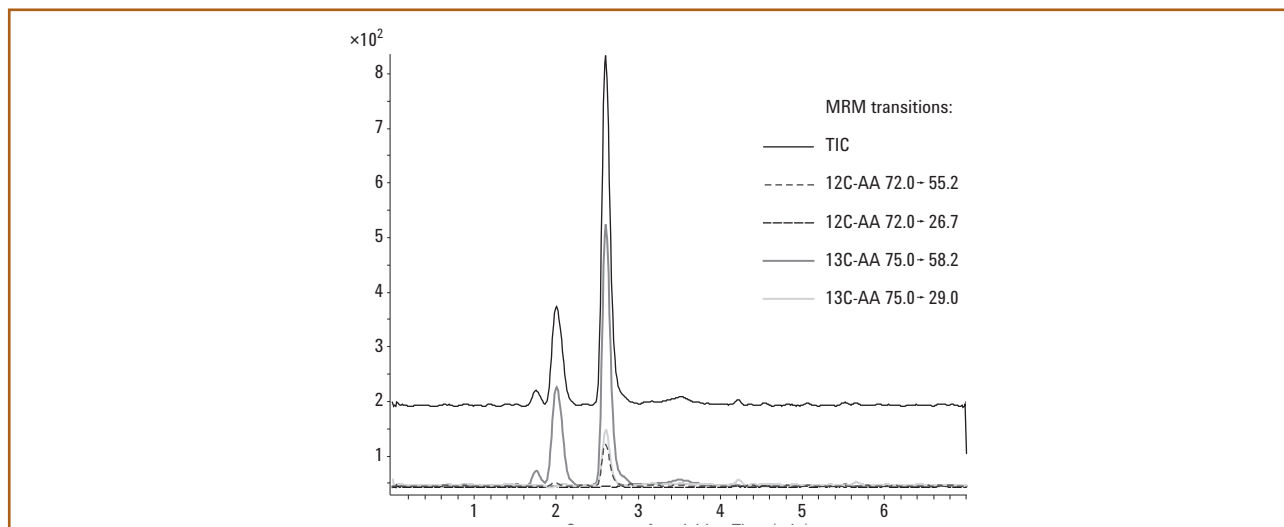


Figure 2. Chromatogram of the 10 ng/mL acrylamide standard and 500 ng/mL internal standard $^{13}\text{C}_3$ -acrylamide.

Table 1. Recoveries and RSDs for acrylamide in spiked fried potato samples and 1:1 water:acetonitrile (n=3)

| Matrix | Concentration of acrylamide spike (ng/mL) | %Recovery (n=3) | %RSD (n=3) |
|----------------|---|--------------------------------|------------|
| 1:1 Water:ACN | 50 | 116.6 | 4.07 |
| 1:1 Water:ACN | 100 | 114.06 | 4.85 |
| Fried potatoes | 100 | 97.14 (after blank correction) | 5.04 |
| Fried potatoes | 200 | 97.50 (after blank correction) | 2.55 |

Products used in the above application

Agilent Bond Elut QuEChERS Extraction Kit for Acrylamides. Part No. 5982-5850.

Agilent Bond Elut AOAC Dispersive SPE Kit, 2 mL. Part No. 5982-5022.

To review this Application Note in its entirety, please search for 5990-5940EN at agilent.com/chem

GC/ μ ECD Analysis and Confirmation of PCBs in Fish Tissue with Agilent J&W DB-35ms and DB-XLB GC Columns (Publication 5990-6236EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach for the extraction and cleanup of 19 PCB (polychlorinated biphenyl) congeners in fish tissue. Spiked recoveries ranged between 72 and 116%. An Agilent 7890A GC system equipped with dual μ ECD detection and dual capillary GC column was used, enabling simultaneous identification and confirmation from a single injection. The GC was also fitted with an unpurged two-way splitter capillary flow technology (CFT) device.

QuEChERS Procedure

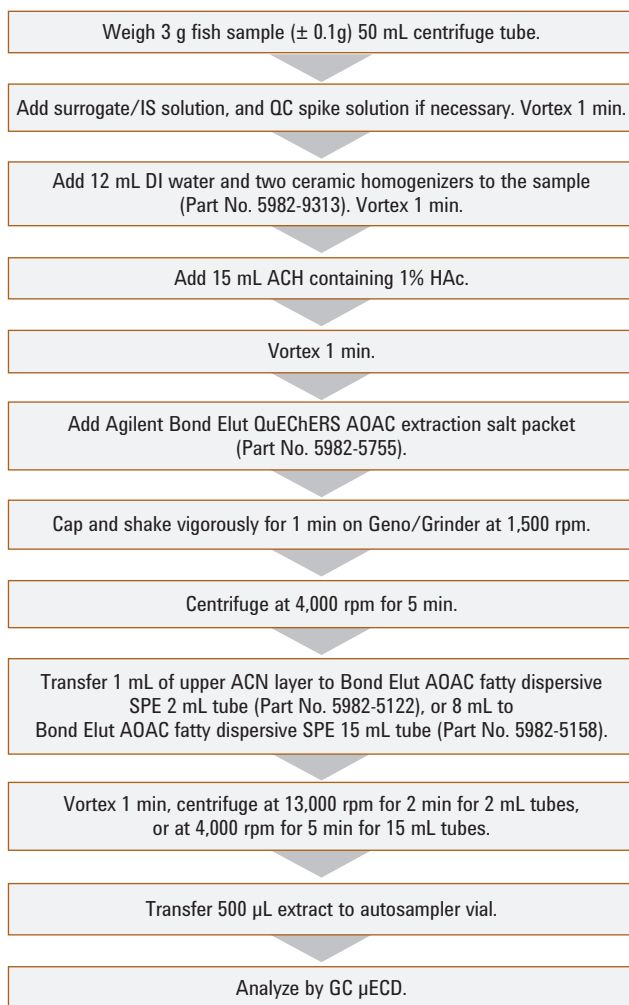


Figure 1. Agilent Bond Elut QuEChERS modified AOAC extraction procedure for fish.

Instrument conditions

HPLC conditions

| | |
|-----------------|---|
| Column 1: | Agilent J&W DB-35 ms, 30 m \times 0.25 mm, 0.25 μ m (Part No. 122-3832) |
| Column 2: | Agilent J&W DB-XLB, 30 m \times 0.25 mm, 0.50 μ m (Part No. 122-1236) |
| GC: | Agilent 7890A equipped with dual μ ECD detection |
| Sampler: | Agilent 7873B 5.0 μ L syringe (Part No. 5181-1273) |
| CFT device: | 2-way unpurged splitter capillary flow technology (Part No. G3181B) |
| Carrier: | Hydrogen 85 cm/s, constant flow 3.5 mL/min |
| Injection: | 1.0 μ L splitless; 250 $^{\circ}$ C, Purge flow 50 mL/min at 0.3 min, Gas saver 50 mL/min at 2 min |
| Oven: | 110 $^{\circ}$ C (0.1 min), 25 $^{\circ}$ C/min to 200 $^{\circ}$ C (0.5 min), 10 $^{\circ}$ C/min to 240 $^{\circ}$ C (0.5 min), 30 $^{\circ}$ C/min to 325 $^{\circ}$ C (1.5 min) |
| Injection: | 1 μ L, 250 $^{\circ}$ C splitless, purge 50 mL/min at 0.3 min, gas saver 50 mL/min on at 2 min |
| Dual μ ECD: | 350 $^{\circ}$ C, N ₂ makeup; constant column + makeup = 30 mL/min |

Flow path supplies

| | |
|----------------|--|
| Vials: | Amber screw top glass vials (Part No. 5183-2072) |
| Vial caps: | Blue screw caps (Part No. 5182-0717) |
| Vial inserts: | 100 μ L glass/polymer feet (Part No. 5181-8872) |
| Syringe: | 5 μ L (Part No. 5181-1273) |
| Septum: | Advanced Green (Part No. 5183-4759) |
| Inlet seal: | Gold plated inlet seal (Part No. 5188-5367) |
| Inlet liners: | Deactivated dual-taper direct connect (Part No. G1544-80700) |
| Ferrules: | 0.4 mm id short; 85/15 Vespel/graphite (Part No. 5181-3323) |
| CFT fittings: | Internal nut (Part No. G2855-20530) |
| CFT ferrules: | SilTite ferrules, 0.25 mm id (Part No. 5188-5361) |
| 20x Magnifier: | 20x Magnifier loop (Part No. 430-1020) |

Results

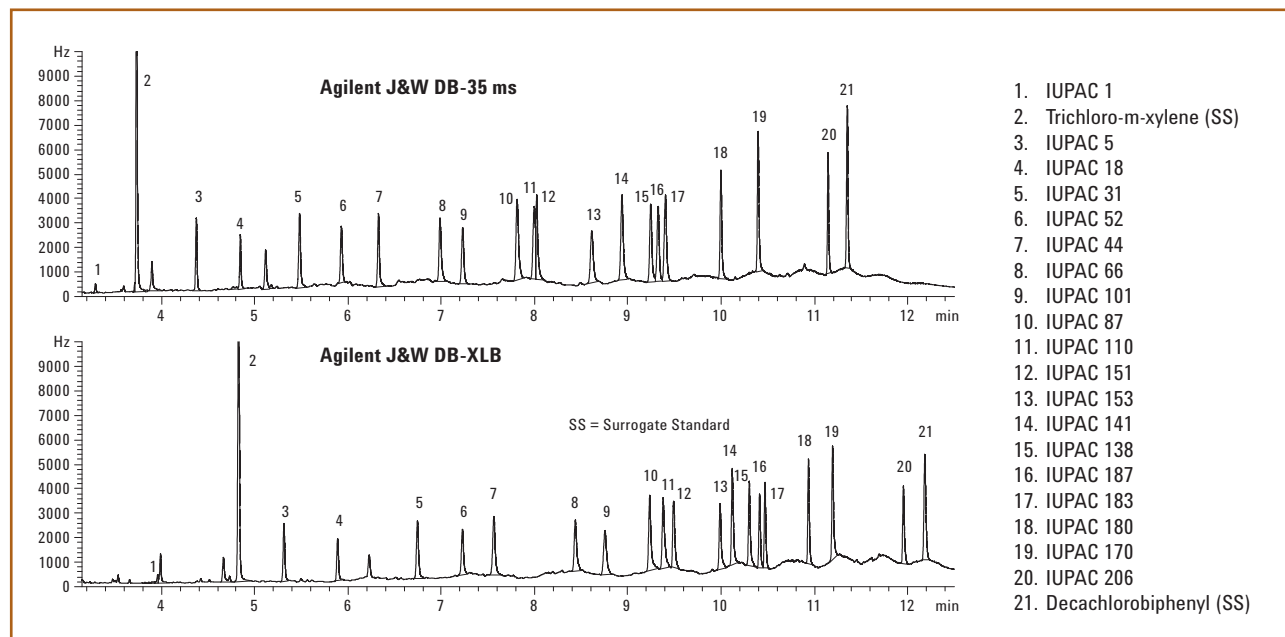


Figure 2. GC/ μ ECD chromatogram of the 50 ng/mL fortified fish extract analyzed on Agilent J&W DB-35ms and DB-XLB GC columns. Chromatographic conditions are listed in Table 1 of app note.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Fatty Sample Dispersive SPE,
2 mL Tube, Part No. 5982-5122 or 15 mL Tube, Part No. 5982-5158.

Agilent J&W DB-35ms Column, 30 m x 0.25 mm, 0.25 μ m. Part No. 122-3832.

Agilent J&W DB-XLB Column, 30 m x 0.25 mm, 0.50 μ m. Part No. 122-1236.

To review this Application Note in its entirety, please search for 5990-6236EN at agilent.com/chem

Analysis of Pesticide Residues in Lettuce and Apple Samples using Agilent Bond Elut QuEChERS AOAC Extraction Kit with Universal Dispersive SPE (Publication 5990-6558EN)

Introduction

This application describes the use of the AOAC buffered extraction method followed by the use of the universal dispersive SPE method for preparing lettuce and apple samples for residue analysis by GC/MS. Twenty-six pesticides of different classes were studied. The analysis was performed using an Agilent 7890 GC System with an Agilent 5975C Series GC/MSD using selective ion monitoring (SIM) mode. All compounds were free of interferences and gave excellent linearity.

Instrument conditions

GC conditions

| | |
|---------------------------|--|
| Column: | Agilent J&W HP-5ms Ultra Inert, 30 m x 0.25 mm, 0.25 μ m (Part No. 190915-433UI) |
| Injection source: | Agilent 7683 Automatic Liquid Sampler with 100 sample tray |
| Inlet: | Splitless |
| Carrier gas: | Helium in constant flow mode |
| Oven temperature program: | 70 °C (2 min), 25 °C/min to 150 °C (0 min), 3 °C/min to 200 °C (0 min), 8 °C/min to 280 °C (7 min) |
| Injection volume: | 1 μ L |

MS conditions

| | |
|--|--------------------------------------|
| Tune file: | Atune.u |
| Mode: | SIM |
| Source, quad, transfer line temperature: | 230 °C, 150 °C, 280 °C, respectively |
| Solvent delay: | 4 min |
| Multiplier voltage: | Autotune voltage |

QuEChERS Procedure

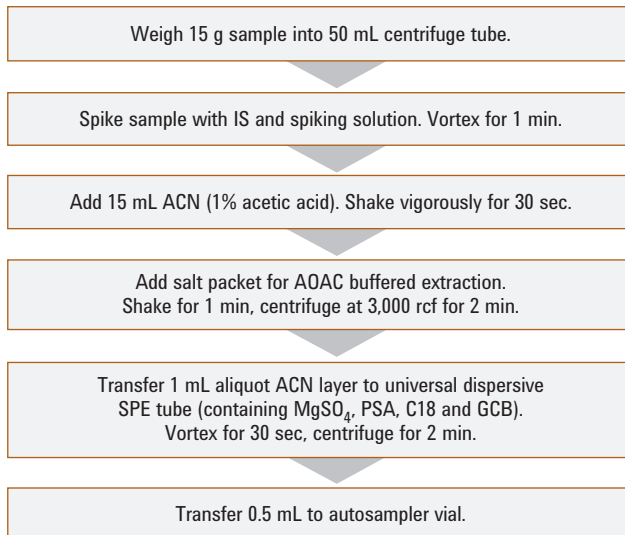


Figure 1. QuEChERS sample preparation procedure for pesticides in lettuce and apple.



Results

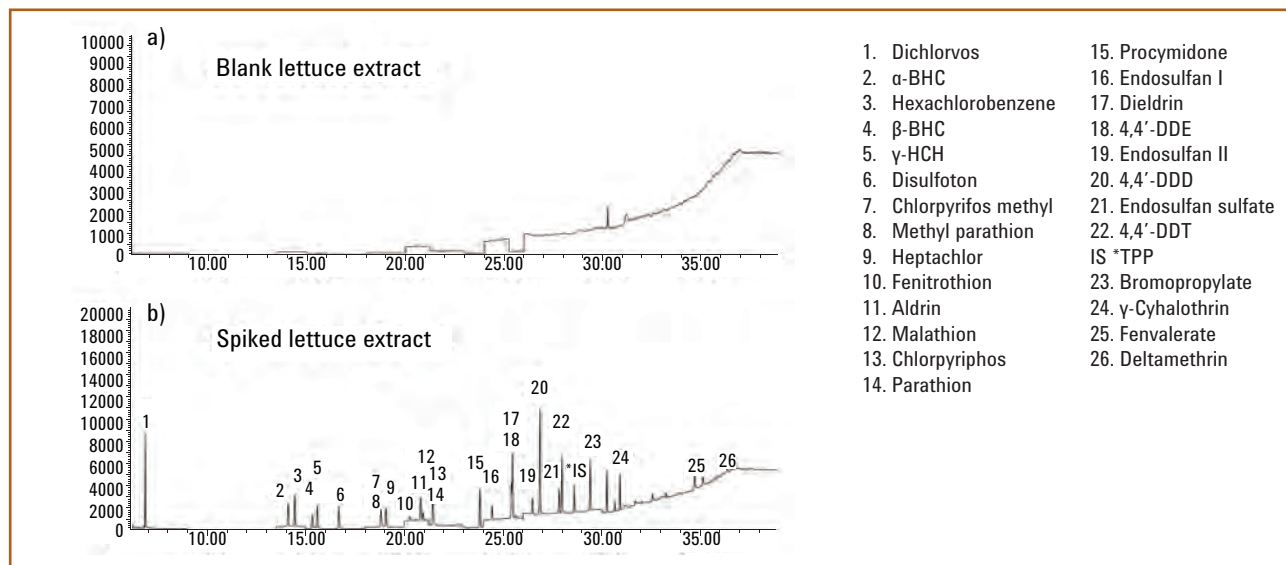


Figure 2. GC/MS of lettuce extracts. (a) blank lettuce extract and (b) spiked lettuce extract after QuEChERS sample preparation.

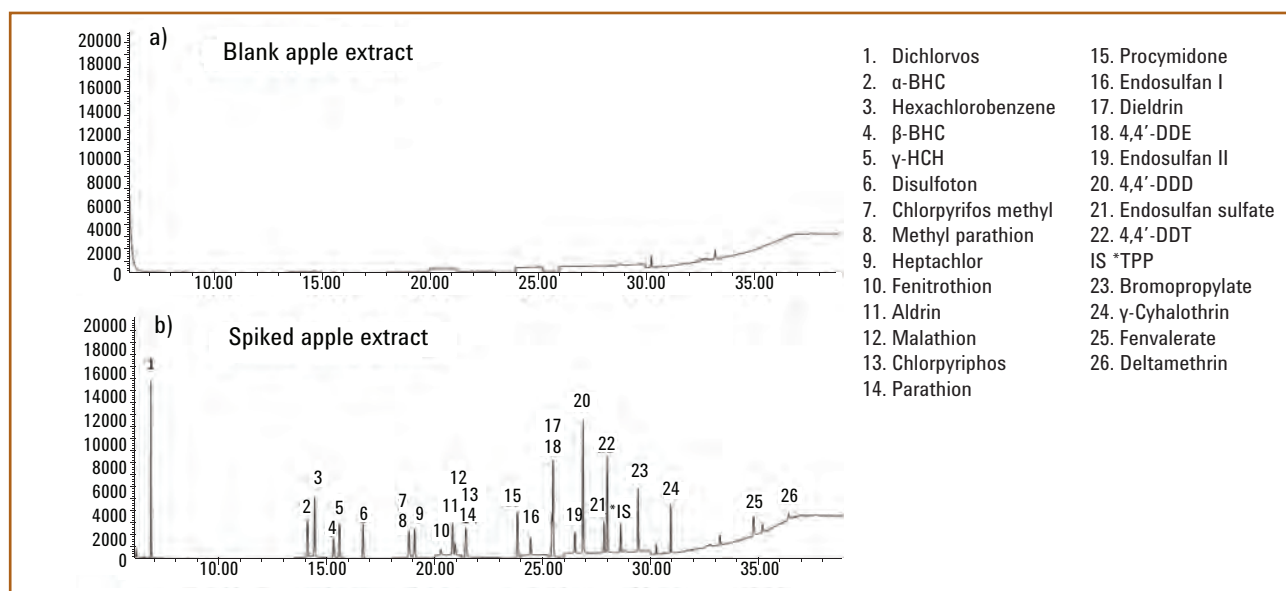


Figure 3. GC/MS of apple extracts (a) blank apple extract and (b) spiked apple extract after QuEChERS sample preparation.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No.5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive Kit for All Food Types. Part No. 5982-0029.

Agilent J&W HP-5ms Ultra Inert Column, 30 m x 0.25 mm, 0.25 μ m. Part No. 190915-433UI.

To review this Application Note in its entirety, please search for 5990-6558EN at agilent.com/chem

Organophosphorus Pesticides in Apple Matrix by GC/MS/FPD using an Agilent J&W DB-35ms Ultra Inert GC Column (Publication 5990-7165EN)

Introduction

This application details a quick and effective analytical method for the determination of low ppm and trace level organophosphorus (OP) pesticides residues in apple extract. Using an ultra-inert mid-polarity GC column, an inert flow path with backflushing, and QuEChERS sample cleanup, good results were achieved, even for the more polar OP pesticides. An Agilent 7890 GC with an Agilent 5975C MSD equipped with a flame photometric detector and Agilent 7683B Automatic Liquid Sampler were used for this series of experiments.

QuEChERS Procedure

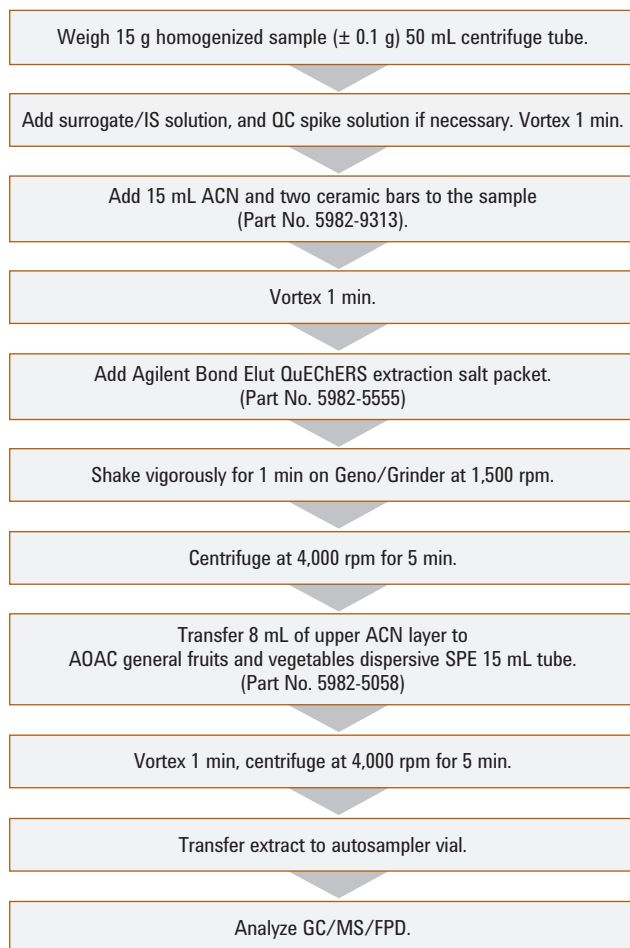


Figure 1. Agilent QuEChERS extraction procedure for apple samples.

Instrument conditions

| GC conditions | |
|--------------------|--|
| Column: | Agilent J&W DB-35ms Ultra Inert, 20 m × 0.18 mm, 0.18 μm (Part No.121-3822UI) |
| GC/MSD: | Agilent 7890 GC/Agilent 5975C Series GC/MSD |
| Sampler: | Agilent 7683B Automatic Liquid Sampler, 5.0 μL syringe (Part No. 5181-1273) |
| CFT device: | Purged 2-way splitter (Part No. G3180B) Split ratio 3:1 MSD:FPD |
| MSD restrictor: | 1.2 m × 0.15 mm id deactivated fused silica tubing |
| FPD restrictor: | 1.4 m × 0.15 mm id deactivated fused silica tubing |
| PCM 1: | 3.8 psi constant pressure |
| Inlet: | 1 μL splitless; 250 °C, purge flow 60 mL/min at 0.25 min, gas saver on at 2 min 20 mL/min |
| Carrier: | Helium, constant pressure 43.5 psi at 95 °C |
| Oven: | 95 °C (1.3 min), 15 °C/min to 125 °C, 5 °C/min to 165 °C, 2.5 °C/min to 195 °C, 20 °C/min to 280 °C (3.75 min) |
| Postrun backflush: | 5 min at 280 °C, PCM 1 pressure 70 psi during backflush, 2 psi inlet pressure during backflush |
| MSD: | 310 °C transfer line, 310 °C source, 150 °C quad |

Flow path supplies

| | |
|----------------|---|
| Vials: | Amber crimp top glass vials (Part No. 5183-4496) |
| Vial caps: | Crimp caps (Part No. 5181-1210) |
| Vial inserts: | 250 μL glass/polymer feet (Part No. 5181-8872) |
| Syringe: | 5 μL (Part No. 5181-1273) |
| Septum: | Advanced Green (Part No. 5183-4759) |
| Inlet liner: | Deactivated dual-taper helix liner (Part No. 5188-5398) |
| Ferrules: | 0.4 mm id short; 85/15 Vespel/graphite (Part No. 5181-3323) |
| PCT fittings: | Internal nut (Part No. G2855-20530) |
| PCT ferrules: | SilTite ferrules, 0.25 mm id (Part No. 5188-5361) |
| 20x Magnifier: | 20x Magnifier loop (Part No. 430-1020) |

Results

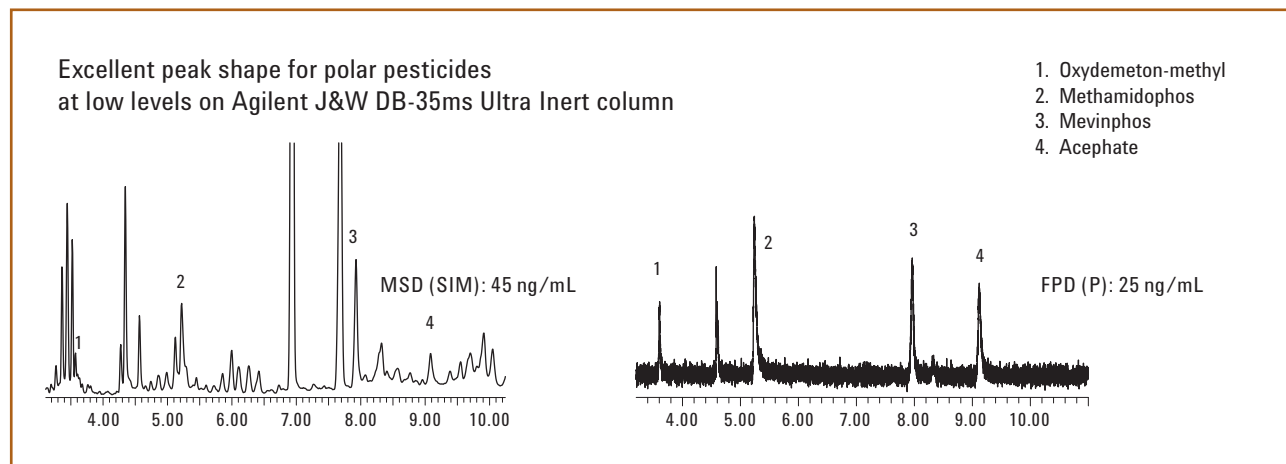


Figure 2. Enlarged section of GC/MS/SIM and FPD chromatograms of the more problematic polar pesticides analyzed on an Agilent J&W DB-35ms Ultra Inert capillary column. Chromatographic conditions are listed in Table 1 of the app note. The effluent split ratio was MSD:FPD = 3:1.



Products used in the above application

Agilent Bond Elut QuEChERS Extraction Salt Packet. Part No. 5982-5555.

Agilent Bond Elut QuEChERS General Fruits and Vegetables Dispersive SPE, 15 mL Tube. Part No. 5982-5058.

Agilent J&W DB-35ms Ultra Inert Column, 20 m x 0.18 mm, 0.18 μ m. Part No. 121-3822UI.

To review this Application Note in its entirety, please search for 5990-7165EN at agilent.com/chem

Analysis of Pesticide Residues in Rice using Agilent Bond Elut QuEChERS AOAC Kit by LC/MS/MS Detection (Publication 5990-8034EN)

Introduction

This application describes the use of the QuEChERS AOAC sample preparation approach for the extraction and cleanup of 12 pesticide residues representing various pesticides classes in rice. The analysis was performed on an Agilent 1200 Infinity Series and an Agilent 6460 Triple Quadrupole LC/MS with electrospray ionization. The mean recoveries ranged between 76% and 108% (average of 97.8%).

Instrument conditions

HPLC conditions

| | | |
|---------------------|--|----|
| Column: | Agilent Poroshell 120 EC-C18, 2.1 × 100 mm, 2.7 μm (Part No. 695775-902) | |
| Flow rate: | 0.4 mL/min | |
| Column temperature: | 30 °C | |
| Injection volume: | 5 μL | |
| Mobile phase: | A: 0.1% FA in water B: 0.1% FA in ACN | |
| Gradient: | Time (min) | %B |
| | 0 | 5 |
| | 1 | 5 |
| | 3 | 50 |
| | 7 | 90 |
| | 8 | 90 |
| | 8.2 | 5 |
| | 9 | 5 |
| Post run: | 2 min | |
| Total cycle time: | 11 min | |

MS conditions

Positive mode

| | |
|------------------|----------|
| Gas temperature: | 350 °C |
| Gas flow: | 10 L/min |
| Nebulizer: | 40 psi |
| Capillary: | 3,500 V |

QuEChERS Procedure

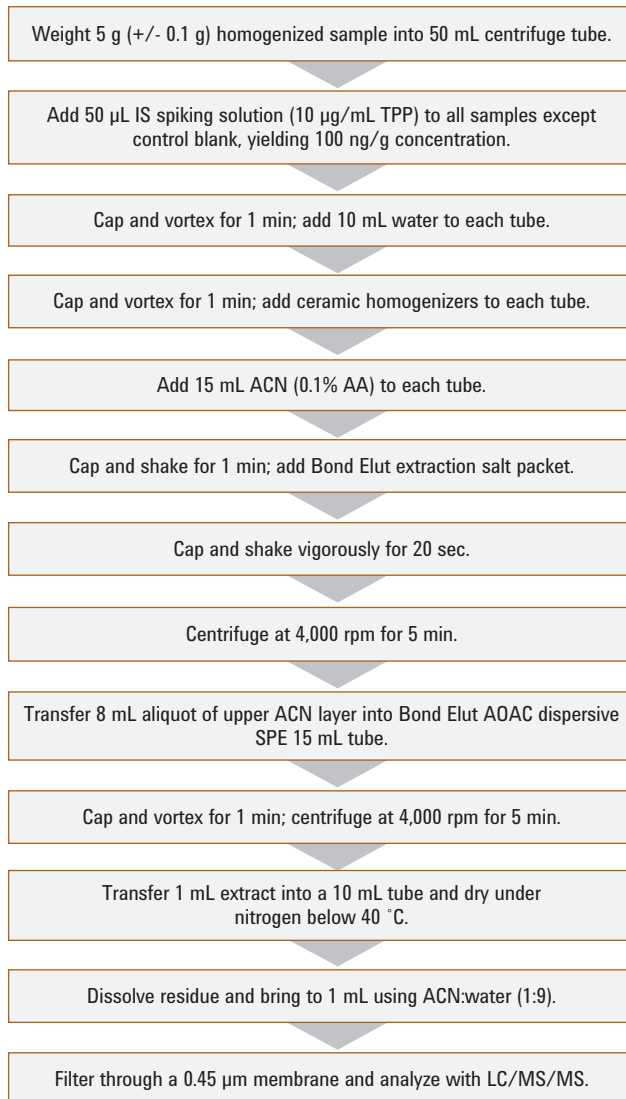


Figure 1. Agilent QuEChERS extraction procedure for rice samples.

Results

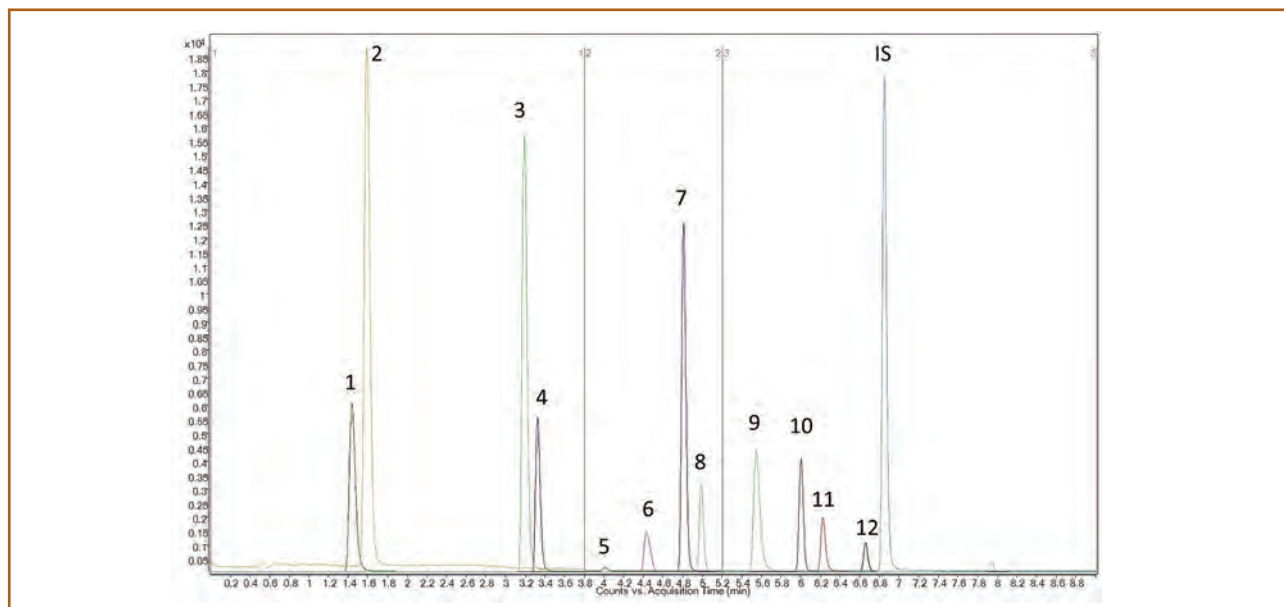


Figure 2. MRM chromatograms of 10 ng/g fortified sample processed by AOAC method. Peak identification: 1. Pymetrozine, 2. Acephate, 3. Carbendazim, 4. Thiabendazole, 5. Imidacloprid, 6. Imazalil, 7. Propoxur, 8. Carbaryl, 9. Cyprodinil, 10. Ethoprophos, 11. Penconazole, 12. Kresoxim-methyl, IS: TPP.

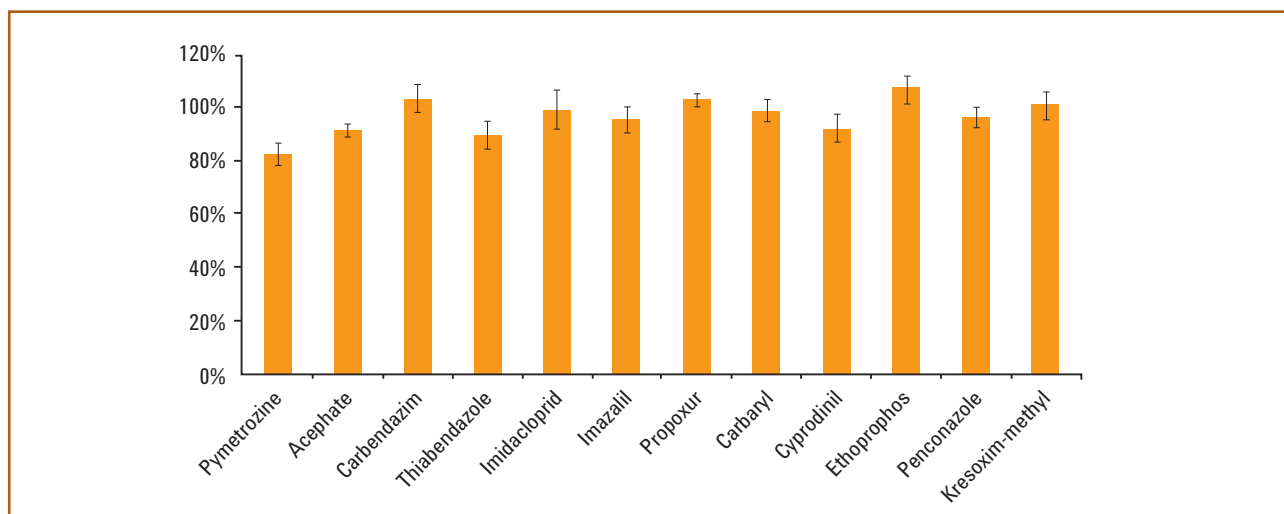


Figure 3. The recovery and precision results of 12 pesticides in rice.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kits for Fruits and Vegetables with Fats and Waxes. Part No. 5982-5158.

Agilent ceramic homogenizers, 50 mL tubes. Part No. 5982-9313.

Agilent Poroshell 120 EC-C18 Column, 2.1 × 100 mm, 2.7 μm. Part No. 695775-902.

To review this Application Note in its entirety, please search for 5990-8034EN at agilent.com/chem

Quantitative and Repeatability Analysis of Trace Level Pesticides in Produce and Grains by GC/MS/MS (Publication 5990-9317EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach for the extraction of 33 representative pesticides in six different types of produce and grains, including pear, orange, strawberry, flour, pepper, and spinach. This analysis was performed on the Agilent 7890 GC and the Agilent 7000 Triple Quadrupole GC/MS/MS Analyzer, which has a Pesticides and Environmental Pollutants MRM database.

QuEChERS Procedure

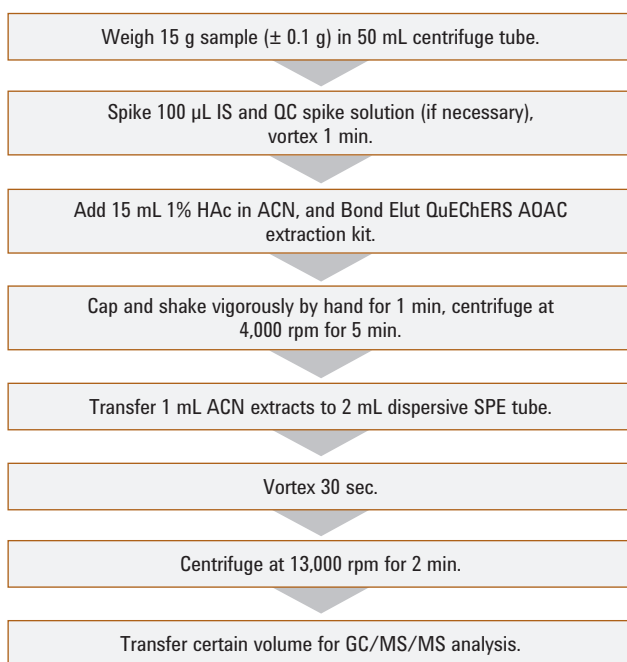


Figure 1. QuEChERS AOAC extraction procedure for produce and grains.

Instrument conditions

GC conditions

| | |
|----------------------------|---|
| Analytical column: | Agilent J&W HP-5ms Ultra Inert, 15 m x 0.25 mm, 0.25 µm (Part No. 19091-431UI) |
| Column connections: | Between inlet and Purged Ultimate Union (Part No. G3182-61580) |
| GC: | Agilent 7890 Series GC |
| Autosampler: | Agilent 7693 Autosampler and sample tray 5 µL syringe (Part No. 5181-5246), 1 µL injection volume Postinj solvent A (acetone) washes: 3 Postinj solvent B (acetonitrile) washes: 3 Sample pumps: 3 |
| Carrier gas: | Helium, constant pressure |
| Inlet: | Multimode Inlet (MMI) |
| Inlet temperature: | 280 °C |
| Injection mode: | Pulsed splitless mode |
| Injection pulse pressure: | 36 psi until 1 min |
| Purge flow to split vent: | 50 mL/min at 1 min |
| Inlet pressure: | 18.35 psi (RT locked) during run, and 1.0 psi during backflush |
| RT locking: | Chlorpyrifos methyl at 8.298 min |
| Oven profile: | 100 °C for 2 min, to 150 °C at 50 °C/min, to 200 °C at 6 °C/min, to 280 °C at 16 °C/min and hold for 6 min |
| Post run: | 2 min at 280 °C |
| Capillary flow technology: | Purged Ultimate Union (Part No. G3182-61580) – used for backflushing the analytical column and inlet. Aux EPC gas: helium plumbed to Purged Ultimate Union |
| Bleed line: | 0.0625 in od x 0.010 in id x 100 cm, 316 SS tubing, on top of the oven |
| Aux pressure: | 4 psi during run, 75 psi during backflushing |
| Restrictor: | Inert fused silica tubing, 0.65 m x 0.15 mm (Part No. 160-7625-5) |
| Restrictor connections: | Between Purged Ultimate Union and the MS |
| MS: | Agilent 7000 Triple Quadrupole GC/MS |
| Mode: | MRM |
| Database: | Agilent Pesticides and Environmental Pollutants database (Part No. G9250AA) |
| Transfer line temperature: | 280 °C |
| Source temperature: | 300 °C |
| Quad temperature: | Q1 and Q2 = 150 °C |
| Solvent delay: | 2.3 min |
| Collision gas flows: | Helium quench gas at 2.35 mL/min, N ₂ collision gas at 1.5 mL/min |
| MS resolution: | MS1 and MS2 = 1.2 amu (Low resolution or Wide setting) |

Results

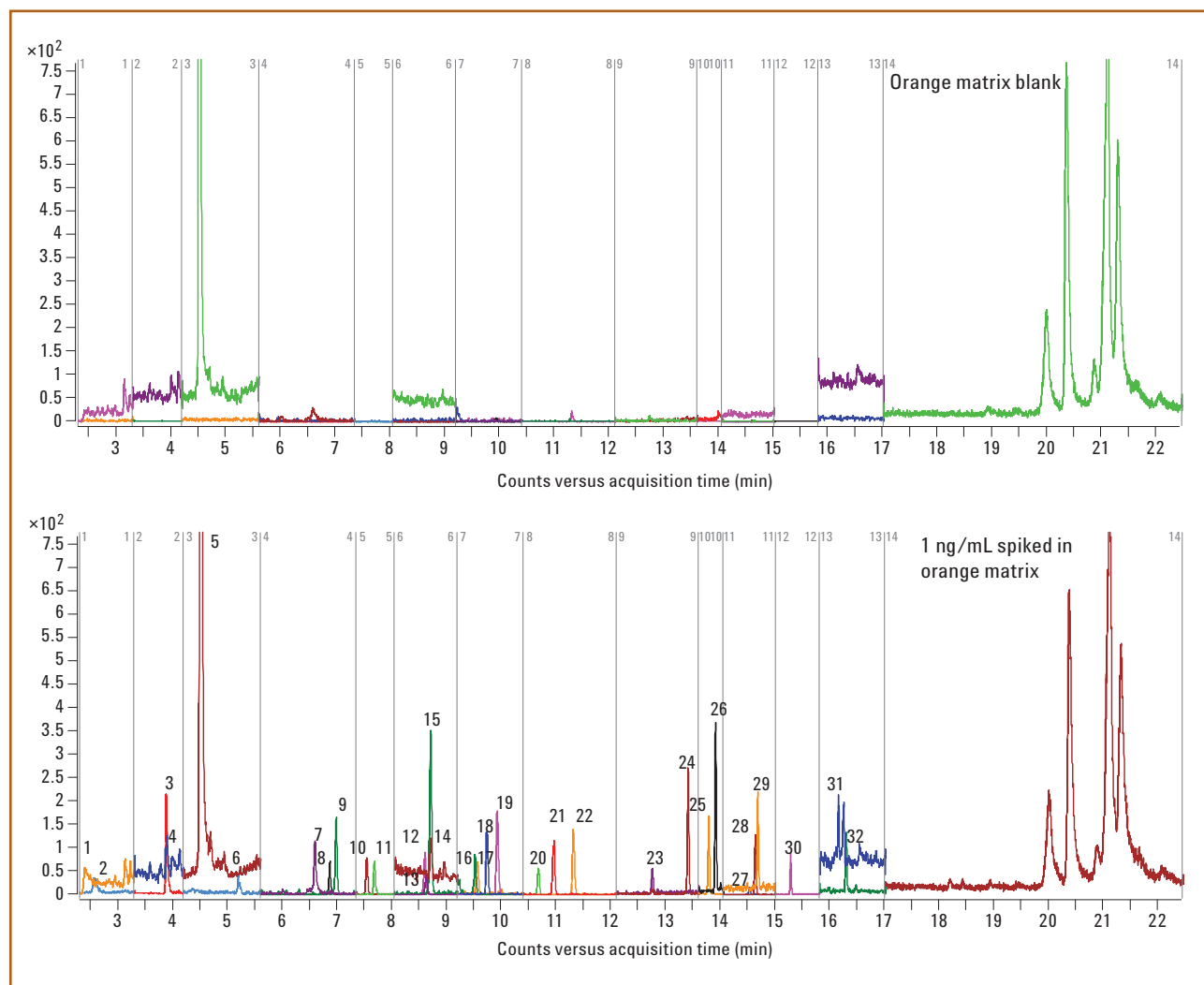


Figure 2. GC/QQQ MRM chromatograms for orange matrix blank and orange matrix spiked with 1 ng/mL pesticides. Refer to Table 3 in the app note for peak identification. Deltamethrin (33) was not identified at 1 ng/mL in orange matrix due to low responses.

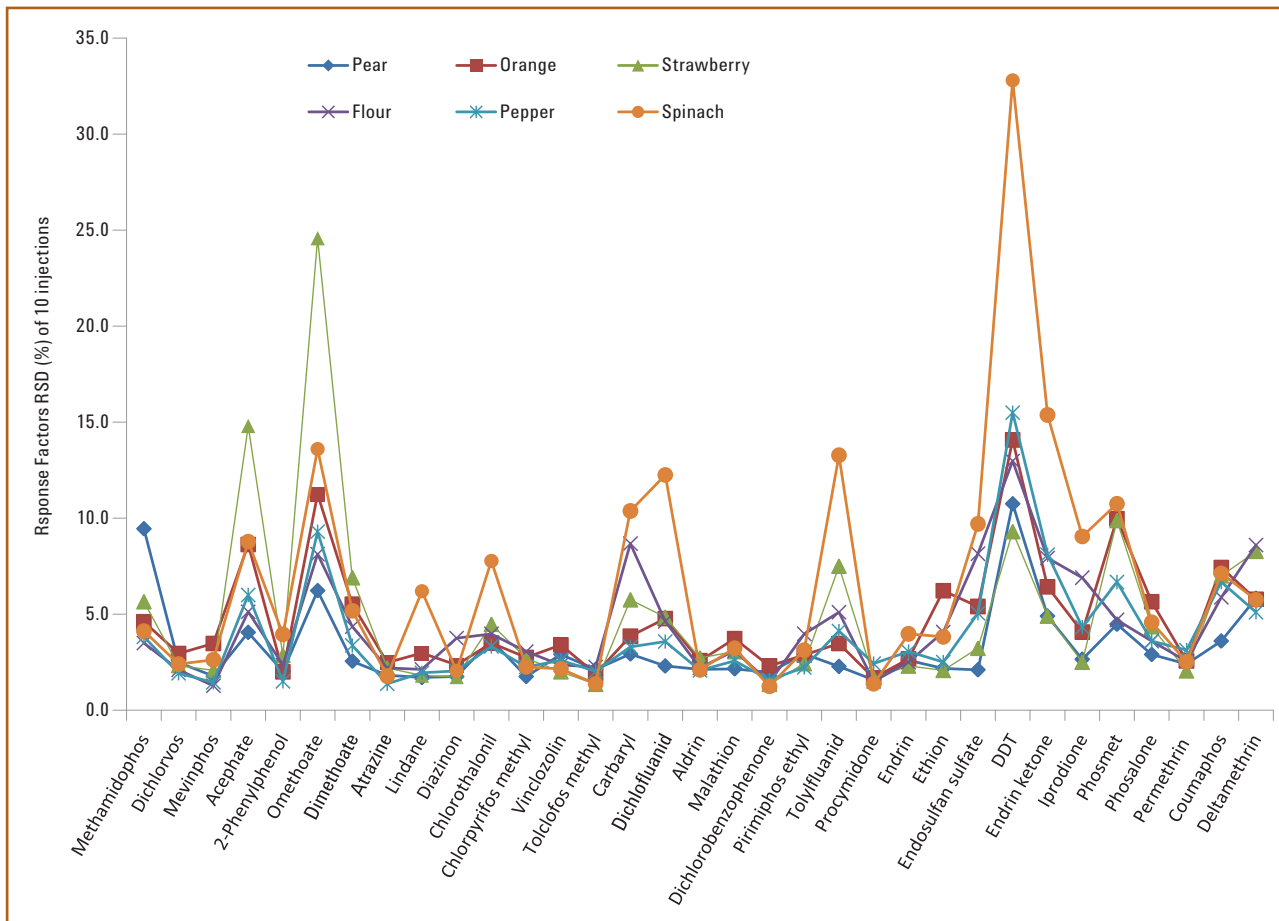


Figure 3. Repeatability (% RSD in response factors) of 10 injections in different matrices.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS General Dispersive SPE Kit. Part No. 5982-5022.

Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm, 0.25 µm. Part No. 19091-431UI.

Agilent Pesticides and Environmental Pollutants MRM Database. Part No. G9250AA.

To review this Application Note in its entirety, please search for 5990-9317EN at agilent.com/chem

Comprehensive Pesticide Analysis in Juice using a Combination of GC/MS and LC/MS Methods (Publication 5990-9924EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach for the effective use of three mass spectral techniques – GC/MS, GC/MS/MS, and LC/MS/MS – for the comprehensive analysis of 39 pesticide residues in vegetable juice. The GC/MS experiments were performed on an Agilent 7890A Series GC coupled to an Agilent 5975C Series GC/MS inert XL MSD with triple axis detector and operated in electron ionization (EI) mode. The GC/MS/MS experiments were performed on an Agilent 7890A Series GC coupled to an Agilent 7000B Triple Quadrupole GC/MS operated in EI mode. The LC/MS/MS experiments were performed on an Agilent 1200 Infinity Series coupled to an Agilent 6460 Series Triple Quadrupole LC/MS System with Jet Stream technology. This comprehensive approach covers a wide range of pesticide classes for confident screening and confirmation of 39 pesticides in vegetable juice.

Note: The procedure below represents a general approach to the extraction of beverages using QuEChERS extraction and dSPE cleanup steps.

QuEChERS Procedure

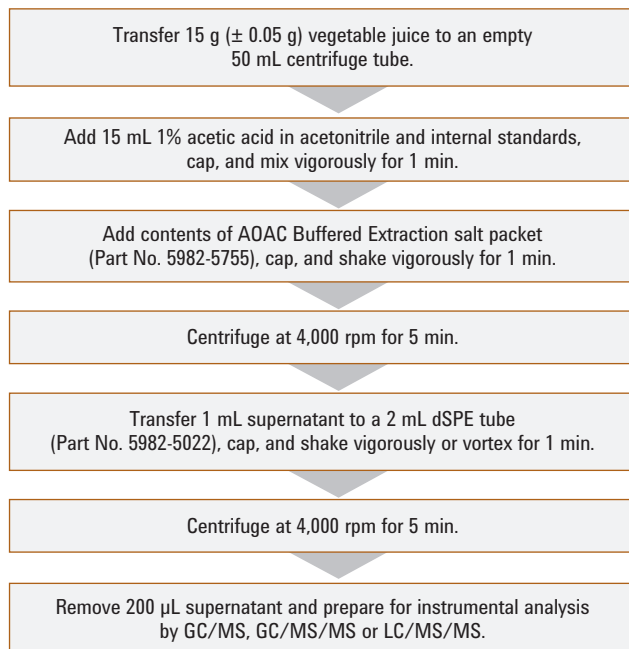


Figure 1. The Agilent QuEChERS extraction procedure. Note: This represents a general approach to the extraction of beverages using QuEChERS extraction and dispersive SPE clean-up steps.

GC/MS conditions

GC conditions

| | |
|-----------------------------|--|
| Columns: | Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 µm (Part No. 19091S-431UI) |
| Injection volume: | 1 µL |
| Injection mode: | Cold splitless using a Multimode Inlet |
| Inlet temperature program: | 60 °C (0.35 min hold); 900 °C/min to 280 °C (15 min hold); 900 °C/min at 300 °C. |
| Oven program: | Scan mode 70 °C for 1 min 70 °C to 150 °C at 50 °C/min 150 °C to 200 °C at 6 °C/min 200 °C to 280 °C at 16 °C/min, 5 min hold 4 min added on to the run at 290 °C for column backflush SIM mode 60 °C for 1.5 min 60 °C to 150 °C at 50 °C/min 150 °C to 240 °C at 8 °C/min 240 °C to 280 °C at 50 °C/min, 2.5 min hold 280 °C to 290 °C at 100 °C/min, 2.05 min hold 4 min added on to the run at 290 °C for column backflush |
| Flow rate for SIM: | 1 mL/min constant flow mode |
| Initial flow rate for scan: | 2.7 mL/min (nominal, constant pressure mode) |
| Retention time locking: | Chlorpyrifos-methyl locked to 8.298 min for scan runs |
| Restrictor: | 0.7 m × 0.15 mm deactivated, (Part No. 160-7625-5) |
| Transfer line temperature: | 280 °C |
| Backflush configuration: | The analytical column was connected between the Multimode Inlet and a Purged Ultimate Union. The 0.7 m restrictor was connected between the Purged Ultimate Union and the MSD. Pressure at the Purged Ultimate Union was set to 4 psig using an auxiliary EPC module. |

MSD conditions

| | |
|-------------------------|-----------------------------|
| Scan mode: | Scan and SIM run separately |
| Mode: | Electron ionization (EI) |
| Source temperature: | 300 °C |
| Quadrupole temperature: | 200 °C |

GC/MS/MS conditions

GC conditions

| | |
|----------------------------|--|
| Columns: | Two Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 µm (Part No. 19091S-431U) columns joined by a Purged Ultimate Union |
| Injection volume: | 1 µL |
| Injection mode: | Cold splitless using a Multimode Inlet |
| Inlet temperature program: | 60 °C (0.35 min hold); 600 °C/min to 270 °C |
| Oven program: | 60 °C for 1 min 60 °C to 170 °C at 40 °C/min 170 °C to 310 °C at 10 °C/min, 1.25 min hold |
| Flow rate: | 1.224 mL/min (constant flow) |
| Solvent delay: | 2.3 min |
| Flow mode: | Constant flow, chlorpyrifos methyl retention time locked to 9.143 min |
| Transfer line temperature: | 300 °C |
| Run time: | 19 min |
| Backflush configuration: | A Purged Ultimate Union (PUU) was connected between the two 15 m analytical columns. Column 1 was backflushed for 4 min at the end of the run with the GC oven at 310 °C, the inlet pressure at 1 psi, and the pressure at the PUU held at 60 psi. |

Triple Quadrupole MS conditions

| | |
|--------------------------|-------------------------------|
| Mode: | Electron ionization (EI), MRM |
| Source temperature: | 300 °C |
| Quadrupole temperatures: | Both at 180 °C |

LC/MS/MS conditions

LC conditions

| | |
|---------------------|--|
| Column: | Agilent ZORBAX Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm (Part No. 959758-902) |
| Column temperature: | 40 °C |
| Injection volume: | 5 µL |
| Mobile phase: | A: 0.1% Formic acid in H ₂ O B: 0.1% Formic acid in acetonitrile |
| Run time: | 15 min |
| Flow rate: | 0.3 mL/min |
| Gradient: | Initial 5% B; 10 min gradient to 95% B, then step to 100% B for 5 min |

Triple Quadrupole MS conditions

| | |
|---------------------|--------------------|
| Mode: | ESI, positive, MRM |
| Sheath gas: | 350 °C, 11 L/min |
| Drying gas flow: | 11 L/min |
| Nebulizer pressure: | 40 psi |
| Capillary voltage: | 4,000 V |
| Nozzle voltage: | 1,000 V |



Results

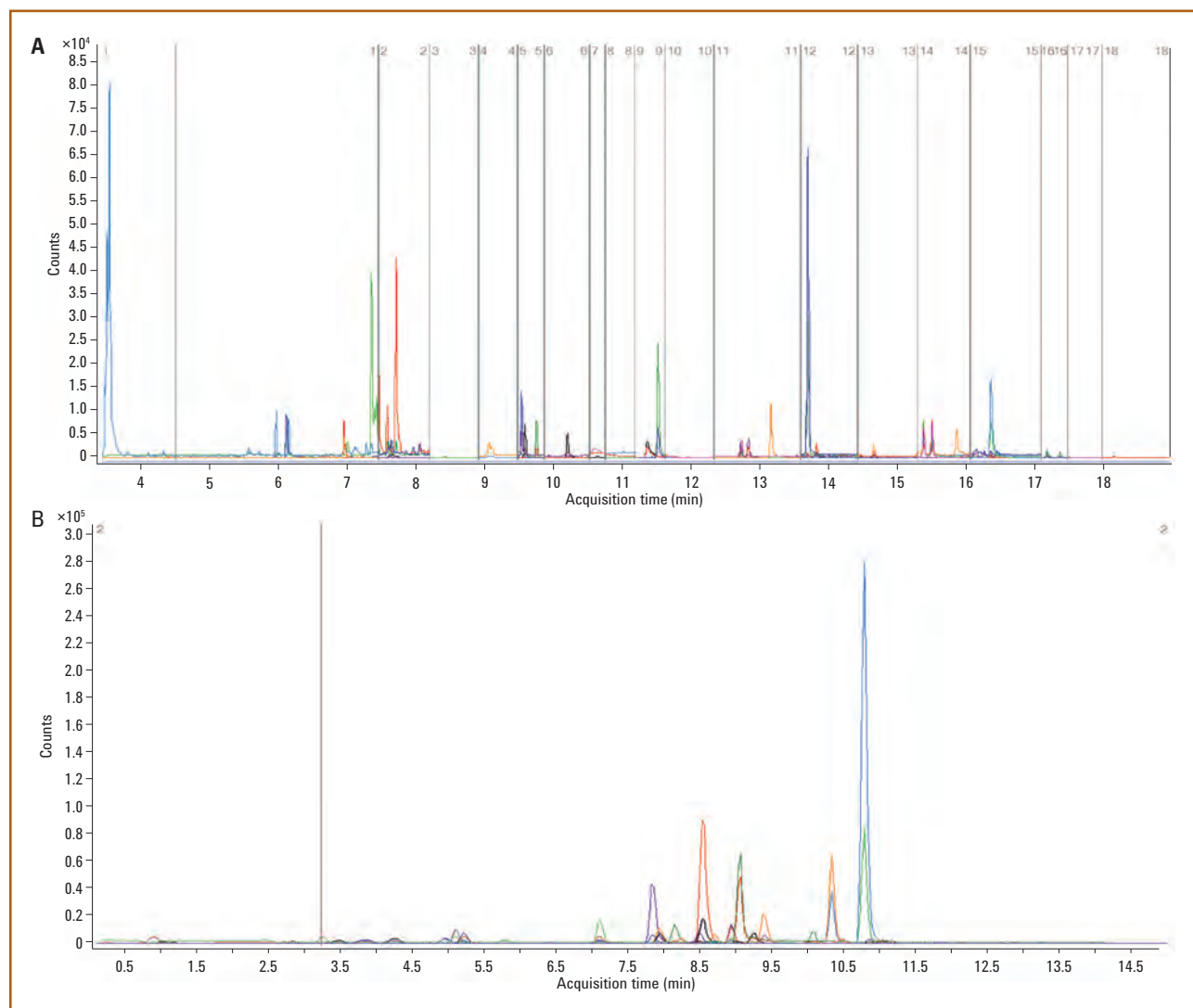


Figure 2. The MRM transitions (in overlay) of the pesticides spiked at 10 ppb (ng/mL) and detected in the vegetable juice blend matrix with GC/MS/MS (Figure 2A) and LC/MS/MS (Figure 2B).

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit for Fruits and Vegetables. Part No. 5982-5022.

Agilent ZORBAX Eclipse Plus C18 Column, 2.1 x 100 mm, 1.8 μm . Part No. 959758-902.

Agilent J&W HP-5ms Ultra Inert Column, 15 m x 0.25 mm, 0.25 μm . Part No. 19091S-431UI.

To review this Application Note in its entirety, please search for 5990-9924EN at agilent.com/chem

Other Food Methods

Determination of Quinolone Antibiotics in Bovine Liver using Agilent Bond Elut QuEChERS Kits by LC/MS/MS (Publication 5990-5085EN)

Introduction

A method for the determination of 11 quinolone antibiotics in bovine liver was established:

- Analytes were extracted and cleaned up from bovine liver with Agilent Bond Elut QuEChERS kits
- Extraction was performed using Bond Elut EN extraction kits and 5% formic acid (FA) in acetonitrile
- Cleanup was performed using Bond Elut dispersive SPE kits Part No. 5982-4921 (25 mg C18 and 150 mg MgSO_4)
- Extracted samples were then analyzed by LC/MS/MS
- Limits of quantitation (LOQ) were 5.0 ng/g
- Calibration curves were linear over the range of 5.0 to 400 ng/g
- The sample pre-fortified recoveries were between 62.0% and 113.1% with RSD (n=6) values between 2.2% and 13.4%



Instrument conditions

HPLC conditions

Column: Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl, 3.0 x 150 mm, 3.5 µm (Part No. 959963-312)

Flow rate: 0.3 mL/min

Column temperature: 30 °C

Injection volume: 10 µL

Mobile phase: A: 5 mM Ammonium acetate in H₂O, pH 3.0
B: 1:1 Methanol:acetonitrile

Post time: 4 min

Gradient:

| Time (min) | % B | Flow Rate (mL/min) |
|------------|------|--------------------|
| 0 | 15 | 0.3 |
| 0.2 | 15 | 0.3 |
| 8.0 | 75 | 0.3 |
| 9.0 | 100 | 0.3 |
| 11.5 | STOP | |

MS conditions

Polarity: Positive

Gas temperature: 325 °C

Gas flow: 8 L/min

Nebulizer: 50 psi

Capillary: 4,000 V

QuEChERS Procedure

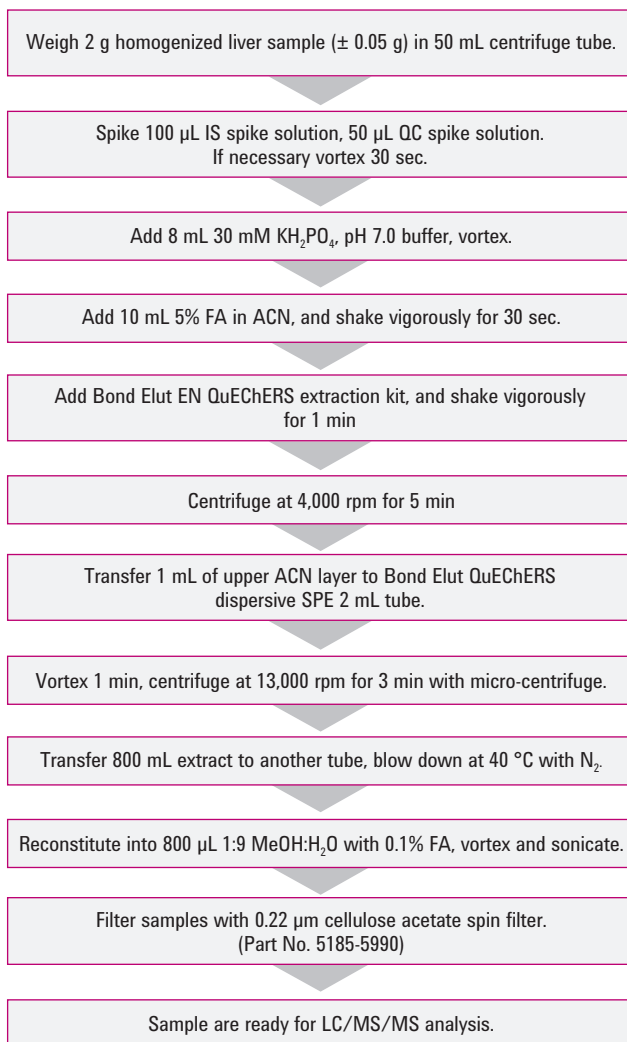


Figure 1: Agilent QuEChERS flow chart procedure for antibiotics.

Results

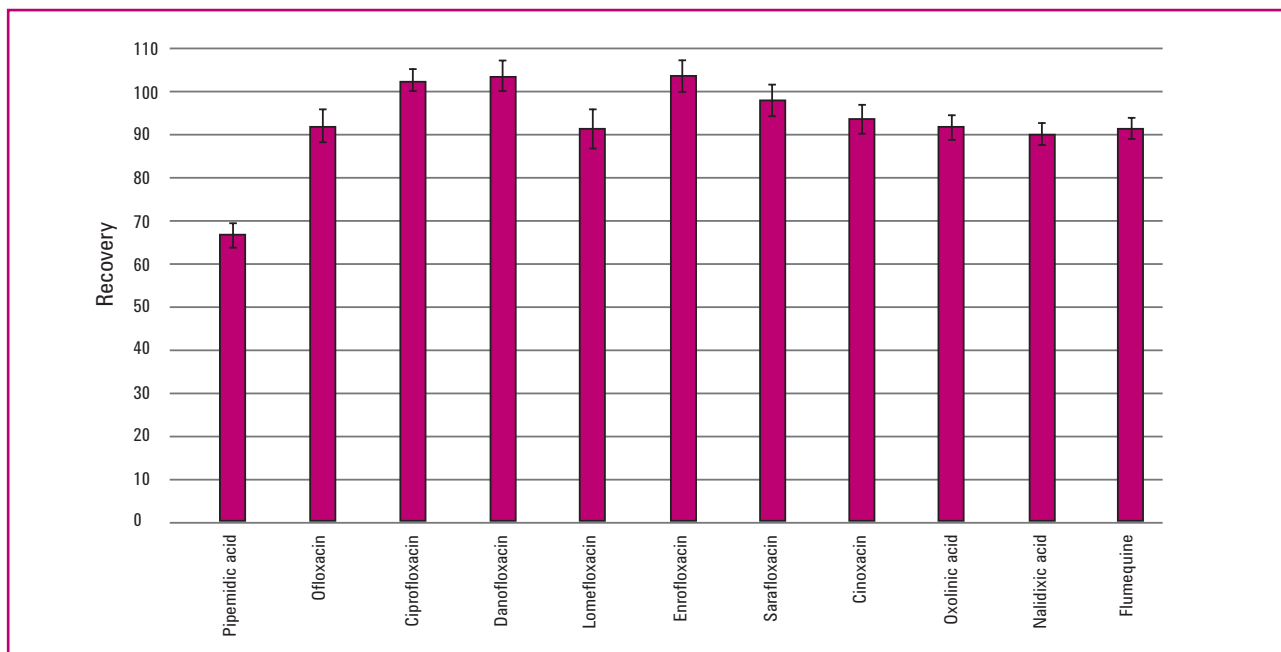


Figure 2: Recovery and reproducibility of eleven quinolone antibiotics from bovine liver.

Products used in the above application

Agilent Bond Elut QuEChERS EN Extraction Kit. Part No. 5982-5650.

Agilent Bond Elut QuEChERS Dispersive SPE Kit, 2 mL. Part No. 5982-4921 or 15 mL. Part No. 5982-4956

Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 μ m. Part No. 959963-312.

To review this Application Note in its entirety, please search for 5990-5085EN at agilent.com/chem

Determination of Sulfonamide Antibiotics in Bovine Liver using Bond Elut QuEChERS EN Kits by LC/MS/MS (Publication 5990-5086EN)

Introduction

A method for the determination of nine sulfonamide antibiotics in bovine liver was established:

- Analytes were extracted and cleaned up from bovine liver with Agilent Bond Elut QuEChERS kits
- Extraction was performed using Bond Elut EN extraction kits and 1% acetic acid (AA) in acetonitrile
- Cleanup was performed using Bond Elut EN fatty dispersive SPE kits, 6 mL (150 mg PSA, 150 mg C18 and 900 mg MgSO₄)
- Extracted samples were then analyzed by LC/MS/MS
- Limits of quantitation (LOQ) were 2.0 ng/g
- Calibration curves were linear over the range of 2.0 to 400 ng/g
- The sample pre-fortified recoveries were between 53.0% and 92.8% with RSD (n=6) values between 2.1% and 16.8%

Instrument conditions

HPLC conditions

| | | |
|---------------------|--|------|
| Column: | Agilent ZORBAX Eclipse Rapid Resolution HT Plus C18, 3.0 x 50 mm, 1.8 µm (Part No. 959941-302) | |
| Flow rate: | 0.3 mL/min | |
| Column temperature: | 30 °C | |
| Injection volume: | 10 µL | |
| Mobile phase: | A: 5 mM Ammonium acetate in H ₂ O, pH 3.0 B: 1:1 Methanol:acetonitrile | |
| Post time: | 3.5 min | |
| Gradient: | Time (min) | % B |
| | 0 | 15 |
| | 0.2 | 15 |
| | 6.0 | 60 |
| | 6.01 | 100 |
| | 7.0 | STOP |

MS conditions

| | |
|------------------|----------|
| Polarity: | Positive |
| Gas temperature: | 325 °C |
| Gas flow: | 8 L/min |
| Nebulizer: | 50 psi |
| Capillary : | 4,000 V |

QuEChERS Procedure

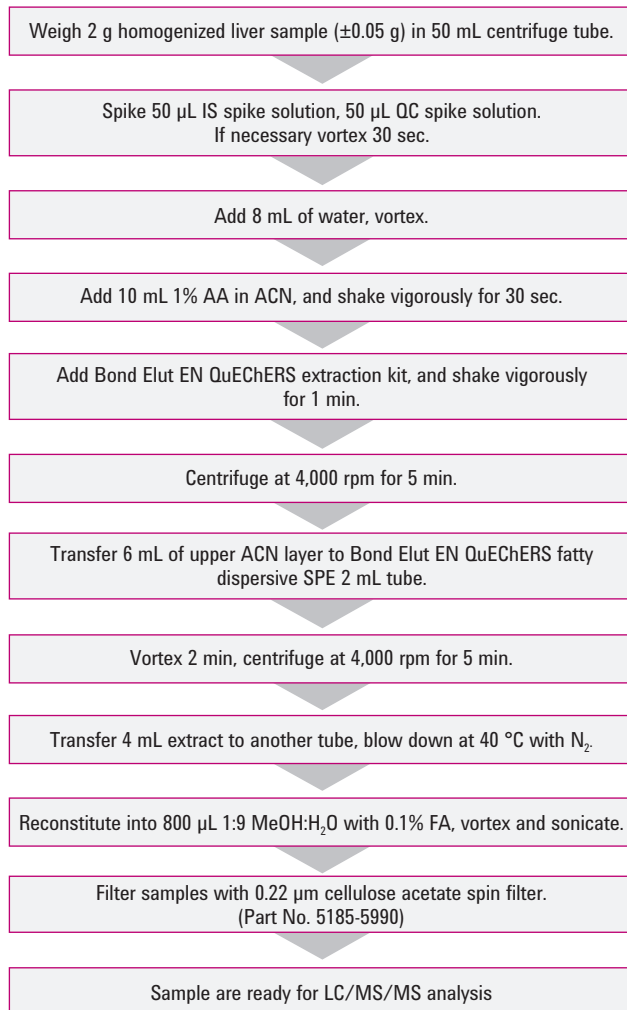


Figure 1. Agilent QuEChERS procedure for sulfonamide antibiotics in bovine liver.

Products used in the above application

Agilent Bond Elut QuEChERS EN Extraction Kit.

Part No. 5982-5650.

Agilent Bond Elut QuEChERS EN Fatty Dispersive SPE Kit.

Part No. 5982-5156.

Agilent ZORBAX Eclipse Rapid Resolution HT Plus C18 LC Column, 3.0 x 50 mm, 1.8 µm. Part No. 959941-302.

Agilent Spin Filters, 0.22 µm Cellulose Acetate.

Part No. 5185-5990.

Results

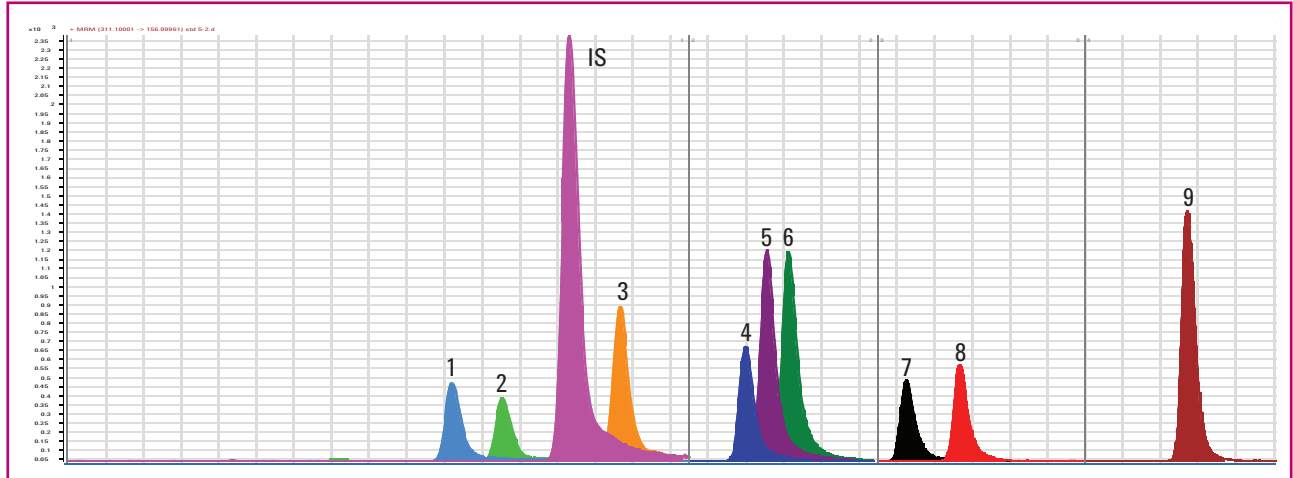


Figure 2. LC/MS/MS chromatogram of 100 ng/g fortified liver extract. Peaks identification: 1. Sulfadizine, 2. Sulfathiazole, 3. Sulfamerazine, 4. Sulfamethizole, 5. Sulfamethazine, 6. Sulfamethoxyipyridazine, 7. Sulfachloropyridazine, 8. Sulfamethoxazole, 9. Sulfadimethoxin, IS (internal standard)

Table 1. Quantitation results – recovery and reproducibility (n=6)

| Compound | Low QC (5 ng/g) | | Mid QC (100 ng/g) | | High QC (400 ng/g) | |
|-------------------------|-----------------|------|-------------------|------|--------------------|-----|
| | Recovery | RSD | Recovery | RSD | Recovery | RSD |
| Sulfadizine | 73.9 | 15.6 | 90.0 | 13.7 | 81.9 | 5.3 |
| Sulfathiazole | 62.9 | 16.8 | 75.3 | 8.4 | 67.9 | 5.8 |
| Sulfamerazine | 77.6 | 11.5 | 92.8 | 6.6 | 82.0 | 4.2 |
| Sulfamethizole | 62.8 | 4.7 | 60.7 | 6.5 | 53.0 | 2.1 |
| Sulfamethazine | 87.4 | 6.9 | 90.0 | 10.7 | 83.4 | 3.4 |
| Sulfamethoxyipyridazine | 81.8 | 9.4 | 84.8 | 8.1 | 76.4 | 2.9 |
| Sulfachloropyridazine | 84.2 | 10.0 | 78.6 | 6.3 | 73.8 | 3.6 |
| Sulfamethoxazole | 85.9 | 7.6 | 82.3 | 5.9 | 78.1 | 3.3 |
| Sulfadimethoxin | 77.8 | 8.4 | 80.9 | 4.9 | 75.6 | 3.3 |

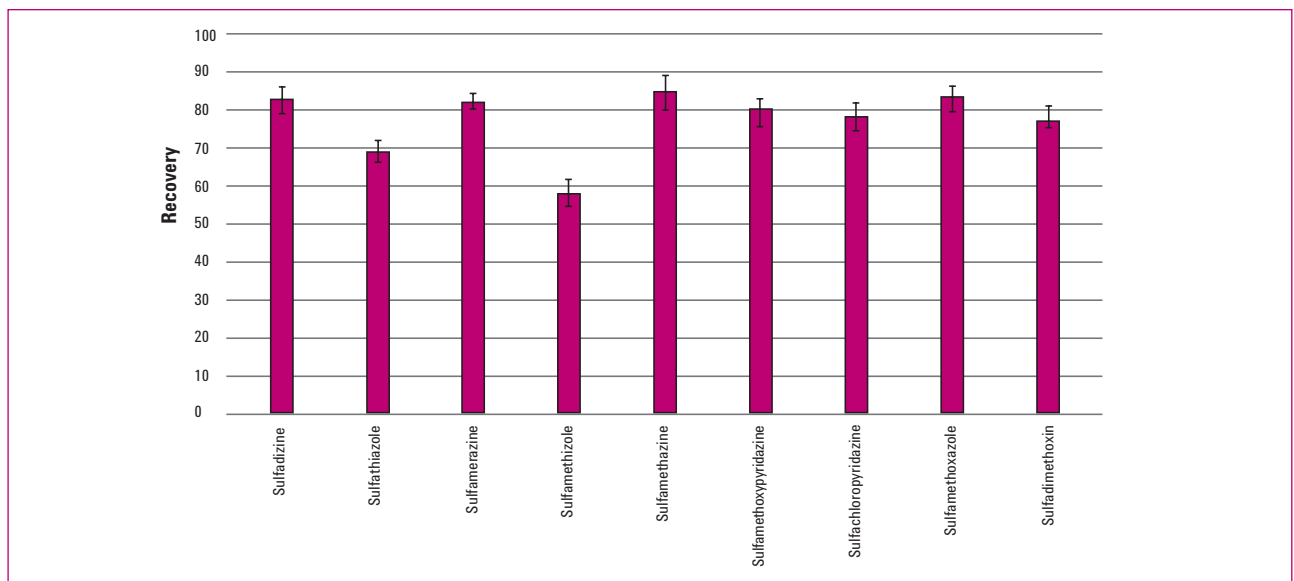


Figure 3. Recovery and reproducibility of nine sulfonamides in bovine liver.

To review this Application Note in its entirety, please search for 5990-5086EN at agilent.com/chem

GC- μ ECD Analysis and Confirmation of Contract Laboratory Protocol Pesticides in Olive Oil (Publication 5990-5553EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach to analyze an olive oil sample from a local grocery store for 20 contract laboratory protocol (CLP) pesticides. A dual μ ECD and dual capillary GC column approach accomplished simultaneous primary and confirmatory analysis where endosulfan sulfate and endosulfan 1 were present in the oil sample. This note successfully shows a robust, inexpensive analytical method to monitor CLP pesticides in olive oil, to address food safety concerns. The method demonstrates the feasibility of using a dual column μ ECD approach for routine olive oil screening as an alternative to GC/MS.

QuEChERS Procedure

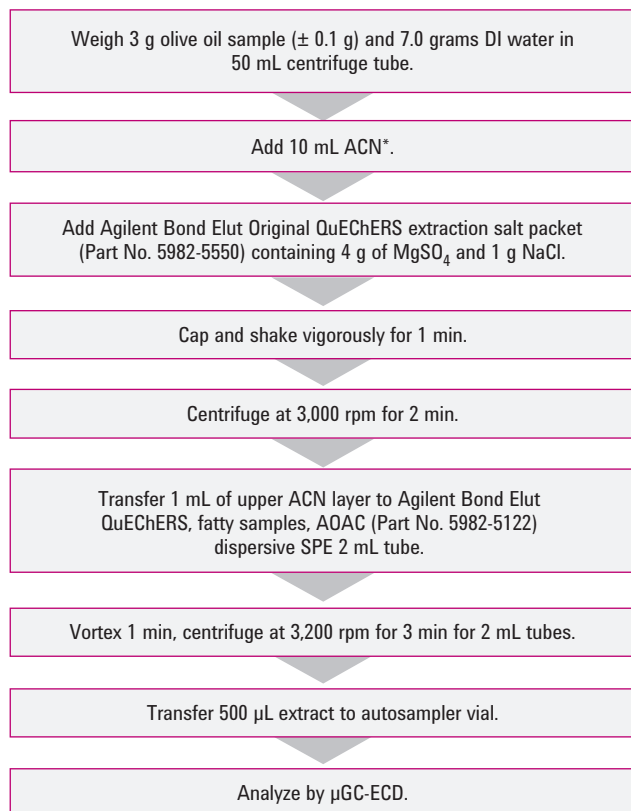


Figure 1. Agilent QuEChERS procedure for pesticides in olive oil.

*Spike sample addition.

Instrument conditions

GC conditions

| | |
|--------------------|--|
| Column 1: | Agilent J&W DB-35 ms 30 m × 0.25 mm, 0.25 μ m (Part No. 122-3832) |
| Column 2: | Agilent J&W DB-XLB 30 m × 0.25 mm, 0.50 μ m (Part No. 122-1236) |
| GC/dual μ ECD: | 7890A equipped with dual μ ECD detection and a 7873B auto sampler |
| CFT device: | 2-way unpurged splitter capillary flow technology (Part No. G3181B) |
| Carrier gas: | Hydrogen 56 cm/sec |
| Oven: | 110 °C (1.4 min), 21 °C/min to 285 °C (1 min), 30 °C/min to 300 °C (2 min) |
| Injection: | 1 μ L, 250 °C splitless, purge 50 mL/min at 0.3 min, gas saver 50 mL/min on at 2 min |
| Dual μ -ECD: | 350 °C, N ₂ makeup; constant column + makeup = 30 mL/min |

Flow path supplies

| | |
|----------------|--|
| Vials: | Amber screw top glass vials (Part No. 5183-2072) |
| Vial caps: | Screw caps (Part No. 5182-0723) |
| Vial inserts: | 100 μ L glass/polymer feet (Part No. 5181-8872) |
| Syringe: | 5 μ L (Part No. 5183-4729) |
| Septum: | Advanced Green (Part No. 5183-4759) |
| Inlet seal: | Gold-plated inlet seal (Part No. 5188-5367) |
| Inlet liners: | Dual-taper direct connect liner (Part No. G1544-80700) |
| Ferrules: | 0.4 mm id short, 85/15 Vespel/graphite (Part No. 5181-3323) |
| CFT fittings: | Internal nut (Part No. G2855-20530) |
| CFT ferrules: | SilTite ferrules, 0.25 mm id (Part No. 5188-5361) |
| 20x Magnifier: | 20x Magnifier loop (Part No. 430-1020) |

Results

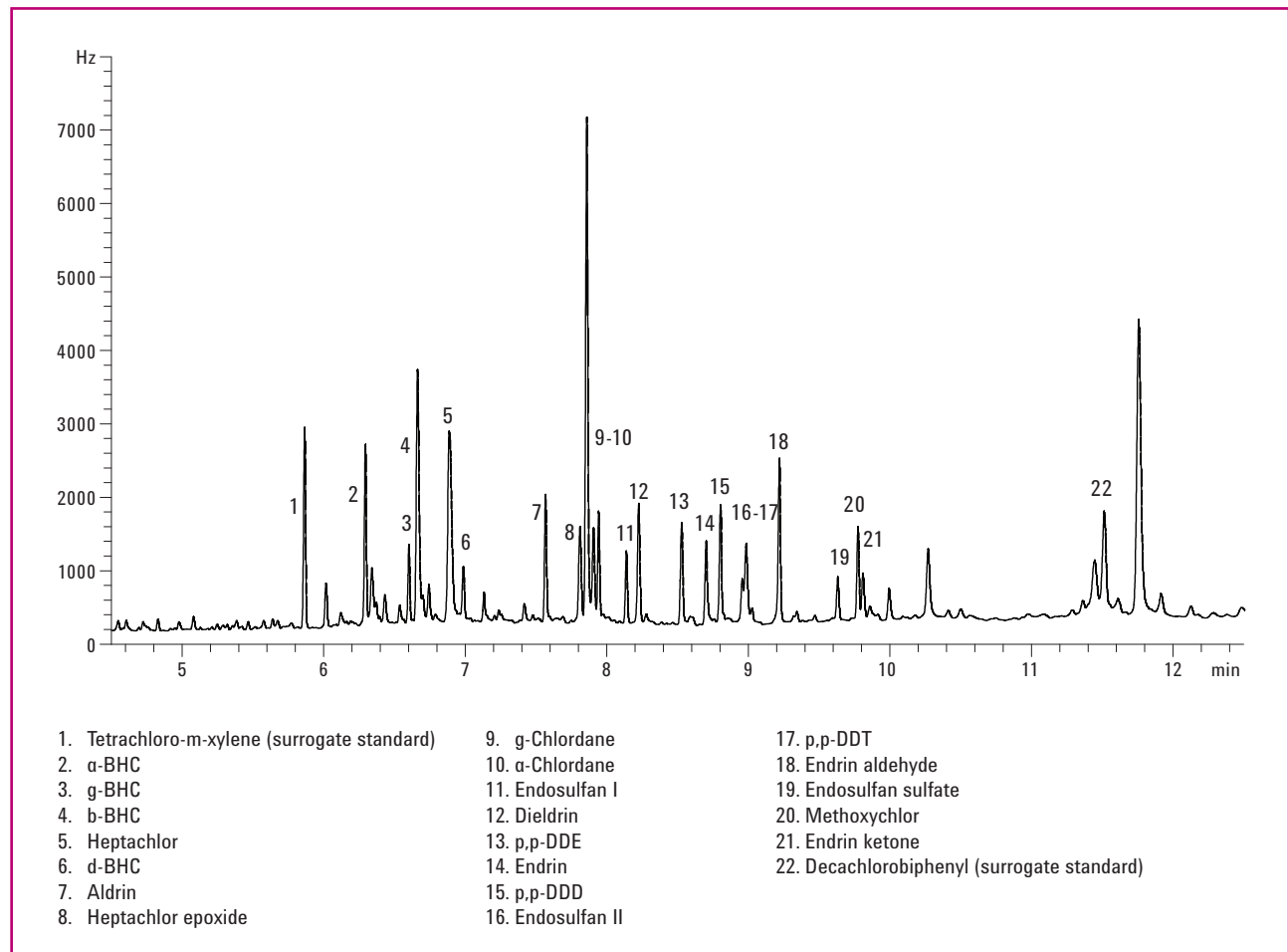


Figure 2. GC- μ ECD chromatogram of olive oil sample spiked with 80 ng/mL of CLP pesticides and 20 ng/mL surrogates standard.



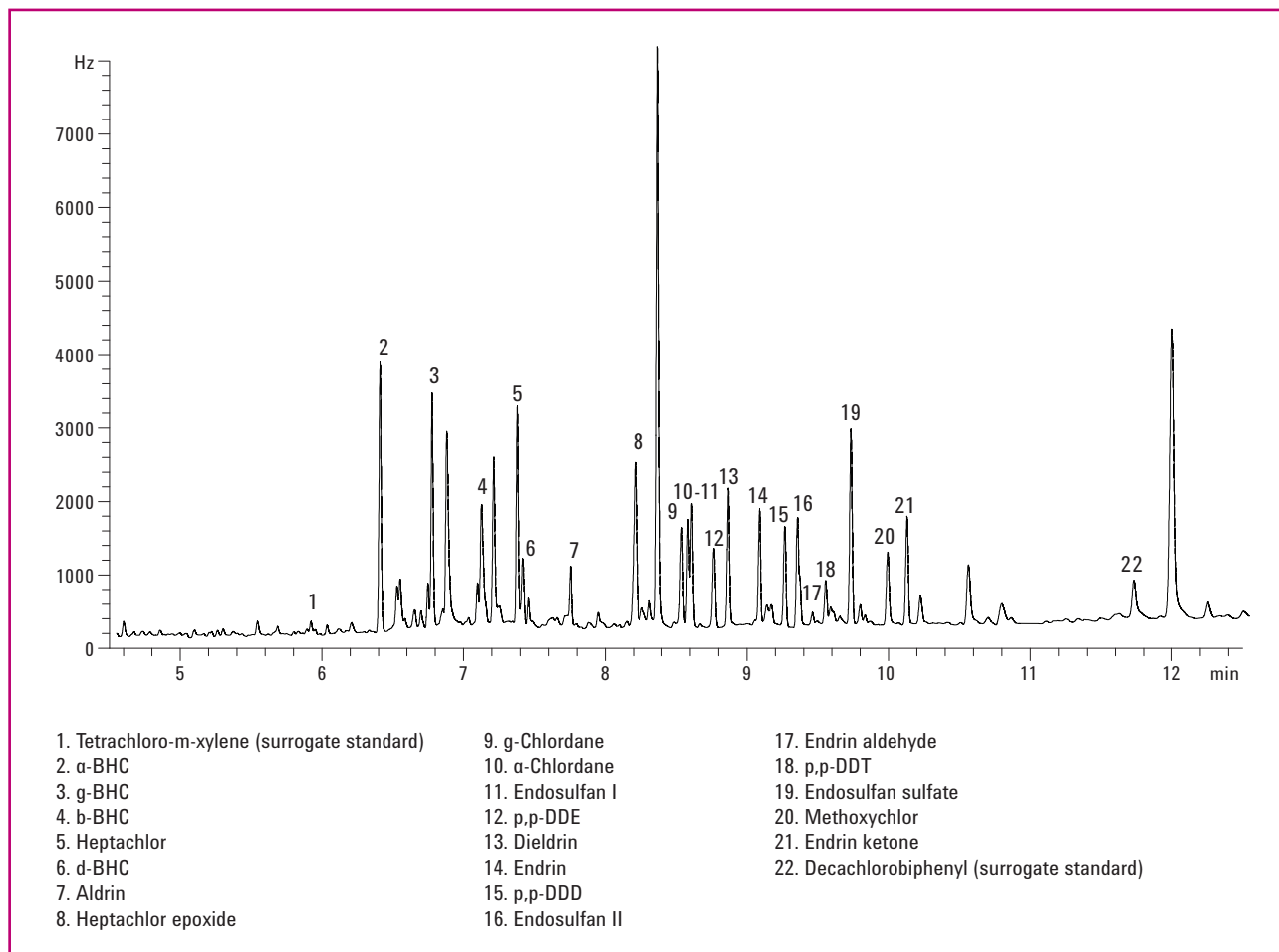


Figure 3. GC- μ ECD chromatogram of olive oil sample spiked with 80 ng/mL of CLP pesticides and 20 ng/mL surrogates standard.

Products used in the above application

Agilent Bond Elut Original QuEChERS Extraction Salt Packet. Part No. 5982-5550.

Agilent Bond Elut QuEChERS, Fatty Samples, AOAC Dispersive SPE, 2 mL Tube. Part No. 5982-5122.

Agilent J&W DB-35ms Column, 30 m x 0.25 mm, 0.25 μ m. Part No. 122-3832.

Agilent J&W DB-XLB Column, 30 m x 0.25 mm, 0.50 μ m. Part No. 122-1236.

To review this Application Note in its entirety, please search for 5990-5553EN at agilent.com/chem

Determination of Pesticides in Lemon Oil via Modified Agilent Bond Elut QuEChERS Method (Publication 5990-6432EN)

Introduction

This application describes a simple and rapid extraction procedure for pesticide residues in lemon essential oils using Agilent Bond Elut QuEChERS extraction and modified dispersive SPE coupled with Agilent gas chromatography and Agilent 5975C Series GC/MSD. Pesticide extraction in essential oil is considered difficult due to the complicated oily matrix. The approach presented here uses multiple dispersive SPE steps along with hexane in the initial extraction step, which results in cleaner extracts amenable with GC/MS analysis using selected ion monitoring (SIM) in electron ionization mode.

Instrument conditions

GC conditions

Column: Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 μm
Inlet: Splitless
Inlet liner: Helix double-taper, deactivated (Part No. 5188-5398)
Carrier gas: Helium
Inlet pressure: 19.6 psi (constant pressure mode)
Inlet temperature: 250 °C
Injection volume: 1.0 μL
Purge flow to split vent: 30 mL/min at 0.75 min
Over temperature program: 70 °C (1 min)
50 °C/min to 150 °C (0 min)
6 °C /min to 200 °C (0 min)
16 °C/min to 280 °C (6 min)

MS conditions

Tune file: Atune.u
Mode: SIM (refer to Table 3 in app note for settings in detail)
Source, quad, transfer: 230 °C, 150 °C, and 280 °C, respectively line temperature
Solvent delay: 2.30 min
Multiplier voltage: Autotune voltage

QuEChERS Procedure

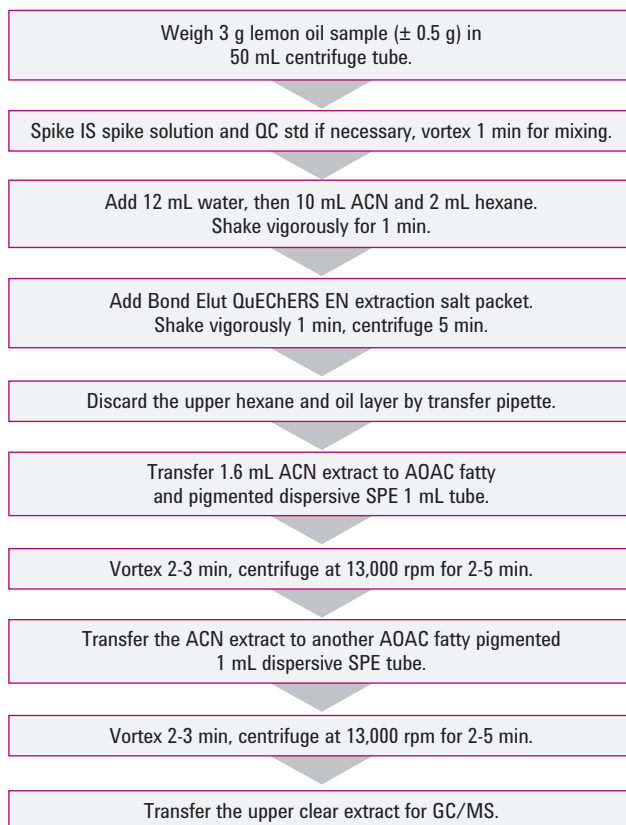


Figure 1. Agilent modified QuEChERS EN extraction procedure for pesticides in lemon oil.

Results

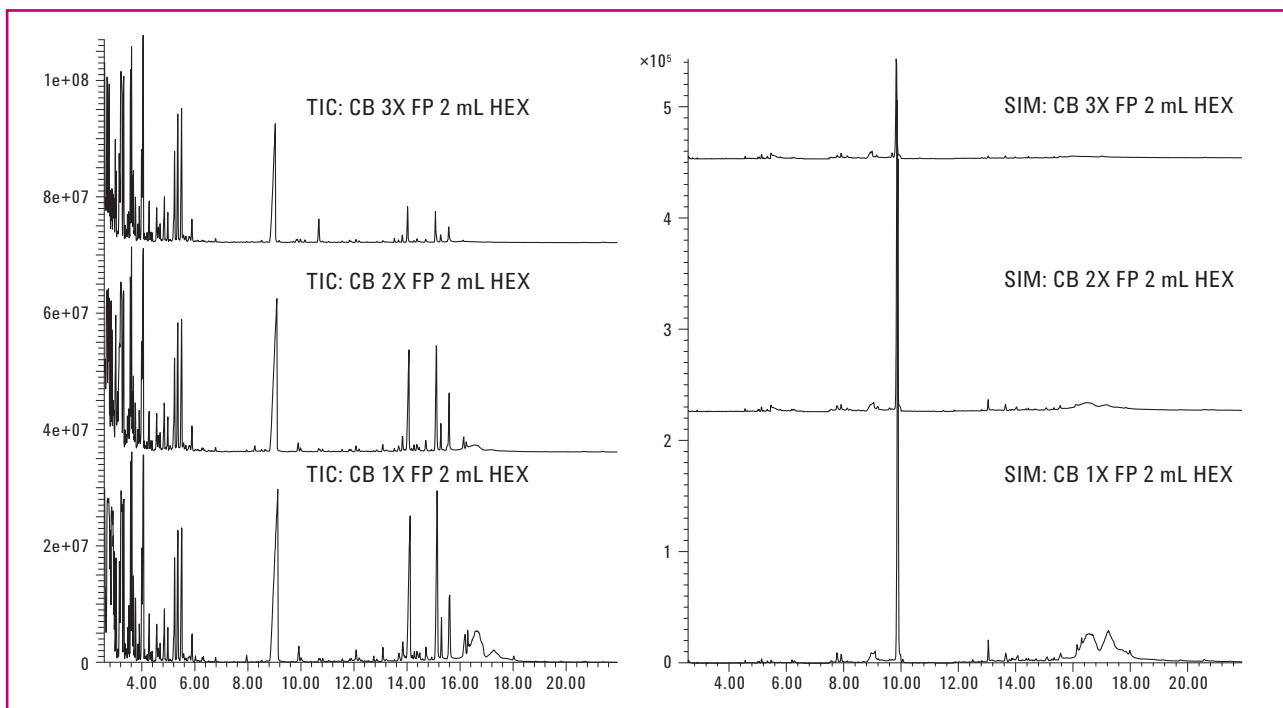


Figure 2. Optimized extraction from the method development experiments involving the addition of hexane and multiple dispersive SPE steps. CB (control blank), FP (AOAC fatty pigmented dispersive SPE), HEX (hexane).



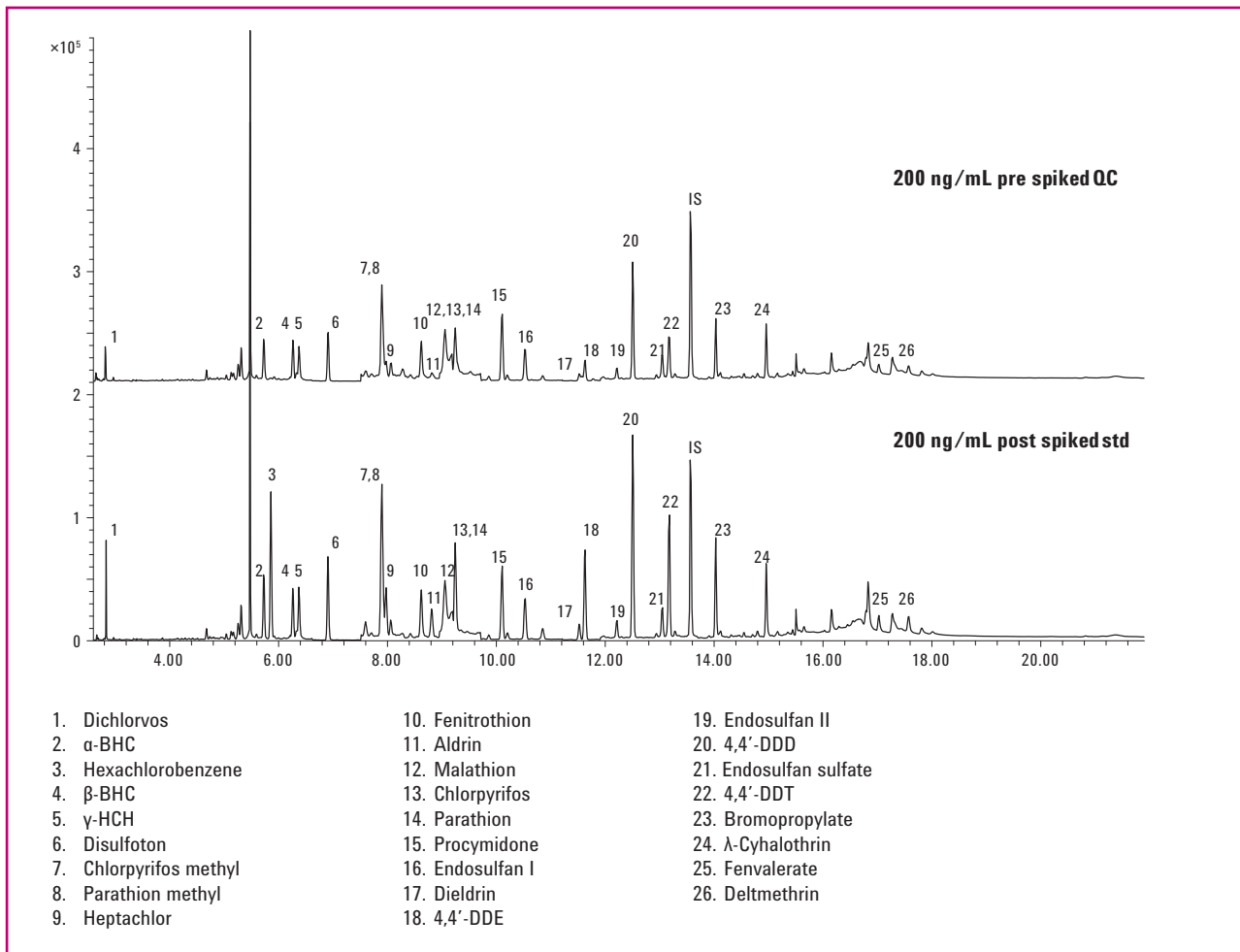


Figure 3. GC/MS chromatograms of 200 ng/mL pre and postmatrix fortified samples. This shows the chromatograms for a prespiked and postspiked lemon oil extract. Twenty-five of the twenty-six pesticides gave repeatable results with RSD < 5% on average.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered EN Extraction Kit. Part No. 5982-5650.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit for Fatty and Pigmented Produce. Part No. 5982-5421.

Agilent J&W HP-5MS Ultra Inert Column, 15 m x 0.25 mm, 0.25 μ m. Part No. 19091S-431UI.

To review this Application Note in its entirety, please search for 5990-6432EN at agilent.com/chem

Polycyclic Aromatic Hydrocarbon (PAH) Analysis in Fish by GC/MS using Agilent Bond Elut QuEChERS dSPE Sample Preparation and a High Efficiency DB-5ms Ultra Inert GC Column (Publication 5990-6668EN)

Introduction

This application describes the use of the QuEChERS dispersive solid phase extraction (dSPE) sample preparation approach for the determination of low and trace level polycyclic aromatic hydrocarbons (PAHs) in fish. The analysis was performed on an Agilent 7890 GC and Agilent 5975B GC/MS system equipped with a Multimode Inlet (MMI) and Agilent 7693 Automatic Liquid Sampler. The GC was also fitted with a pressure controlled tee (PCT) post-column for automated backflush. Recoveries ranged from 80% to 139%.

QuEChERS Procedure

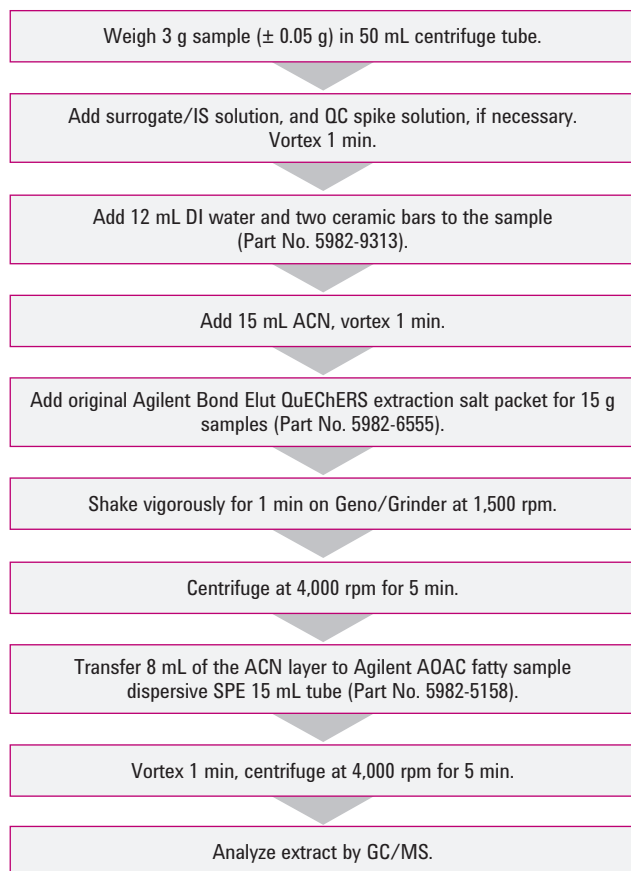


Figure 1. Agilent Bond Elut QuEChERS modified extraction procedure for fish samples.

Instrument conditions

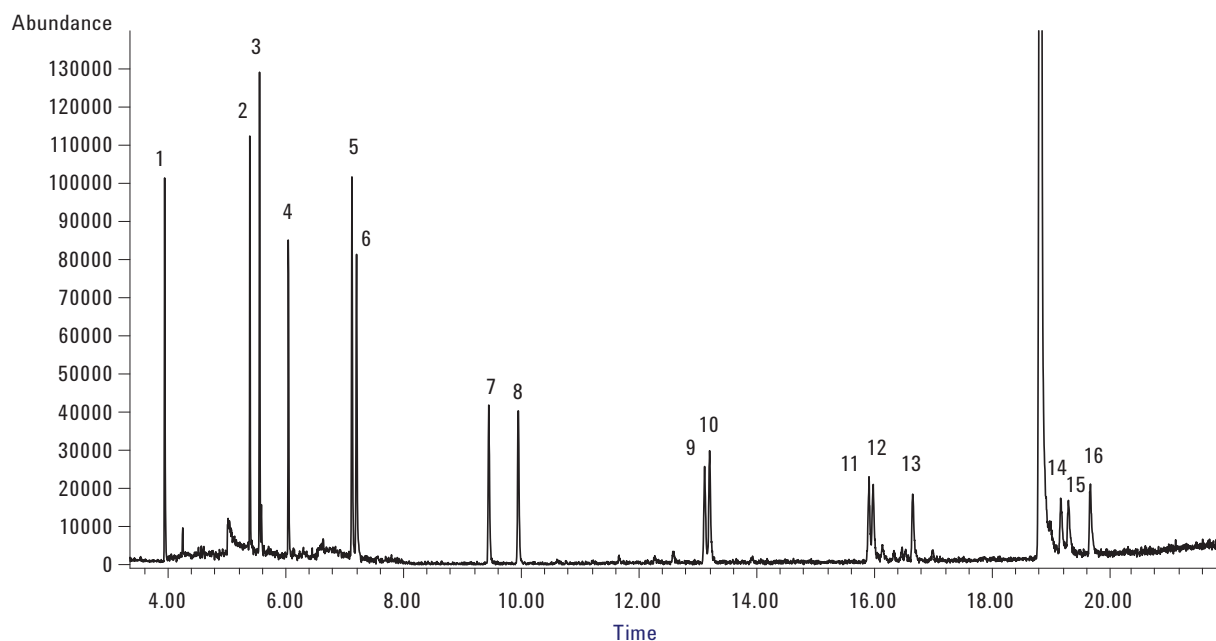
GC conditions

| | |
|--------------------|--|
| Column: | Agilent J&W DB-5ms UI 20 m × 0.18 mm, 0.18 μm (Part No. 122-5522UI) |
| GC/MSD: | Agilent 7890 GC/Agilent 5975B GC/MS System |
| Sampler: | Agilent 7693 Automatic Liquid Sampler, 5.0 μL syringe (Part No. 5181-1273) |
| PCT device: | Purged Ultimate Union (Part No. G3186-60580) |
| Carrier: | Helium, constant flow 1.7 mL/min |
| Restrictor: | 0.7 m × 0.15 mm id deactivated silica tubing |
| PCM 1: | 3.8 psi constant pressure |
| MMI: | 0.5 μL splitless; 320 °C, purge flow 50 mL/min at 0.8 min; Gas saver 30 mL/min at 2 min |
| Oven: | 50 °C (0.4 min), 25 °C/min to 195 °C (1.5 min), 8 °C/min to 265 °C, 20 °C/min to 315 °C (1.25 min) |
| Postrun backflush: | 7 min at 315 °C, backflush pressure 70 psi, 2 psi inlet pressure during backflush |
| MSD: | 340 °C transfer line, 340 °C source, 150 °C quad |

Flow path supplies

| | |
|----------------|--|
| Vials: | Amber screw top glass vials (Part No. 5183-2072) |
| Vial caps: | Blue screw caps (Part No. 5182-0717) |
| Vial inserts: | 100 μL glass/polymer feet (Part No. 5181-8872) |
| Syringe: | 5 μL (Part No. 5181-1273) |
| Septum: | Advanced Green (Part No. 5183-4759) |
| Inlet liners: | Deactivated dual-taper helix liner (Part No. G5188-5398) |
| Ferrules: | 0.4 mm id short, 85/15 Vespel/graphite (Part No. 5181-3323) |
| PCT fittings: | Internal nut (Part No. G2855-20530) |
| PCT ferrules: | SilTite ferrules, 0.25 mm id (Part No. 5188-5361) |
| 20x Magnifier: | 20x Magnifier loop (Part No. 430-1020) |

Separation of 16 PAHs on an Agilent J&W DB-5ms Ultra Inert column



- | | | |
|-------------------|--------------------------|-----------------------------|
| 1. Naphthalene | 7. Fluoranthene | 13. Benz[a]pyrene |
| 2. Acenaphthylene | 8. Pyrene | 14. Indeno[1,2,3-c,d]pyrene |
| 3. Acenaphthene | 9. Benz[a]anthracene | 15. Dibenzo[a,h]anthracene |
| 4. Fluorene | 10. Chrysene | 16. Benzo[g,h,i]perylene |
| 5. Phenanthrene | 11. Benzo[b]fluoranthene | |
| 6. Anthracene | 12. Benzo[k]fluoranthene | |

Figure 2. GC/MS chromatogram of the 500 ng/mL PAH standard prepared in sample matrix analyzed on an Agilent J&W DB-5ms Ultra Inert GC capillary column (Part No. 122-5522U). Chromatographic conditions are listed in Table 1 of the app note.



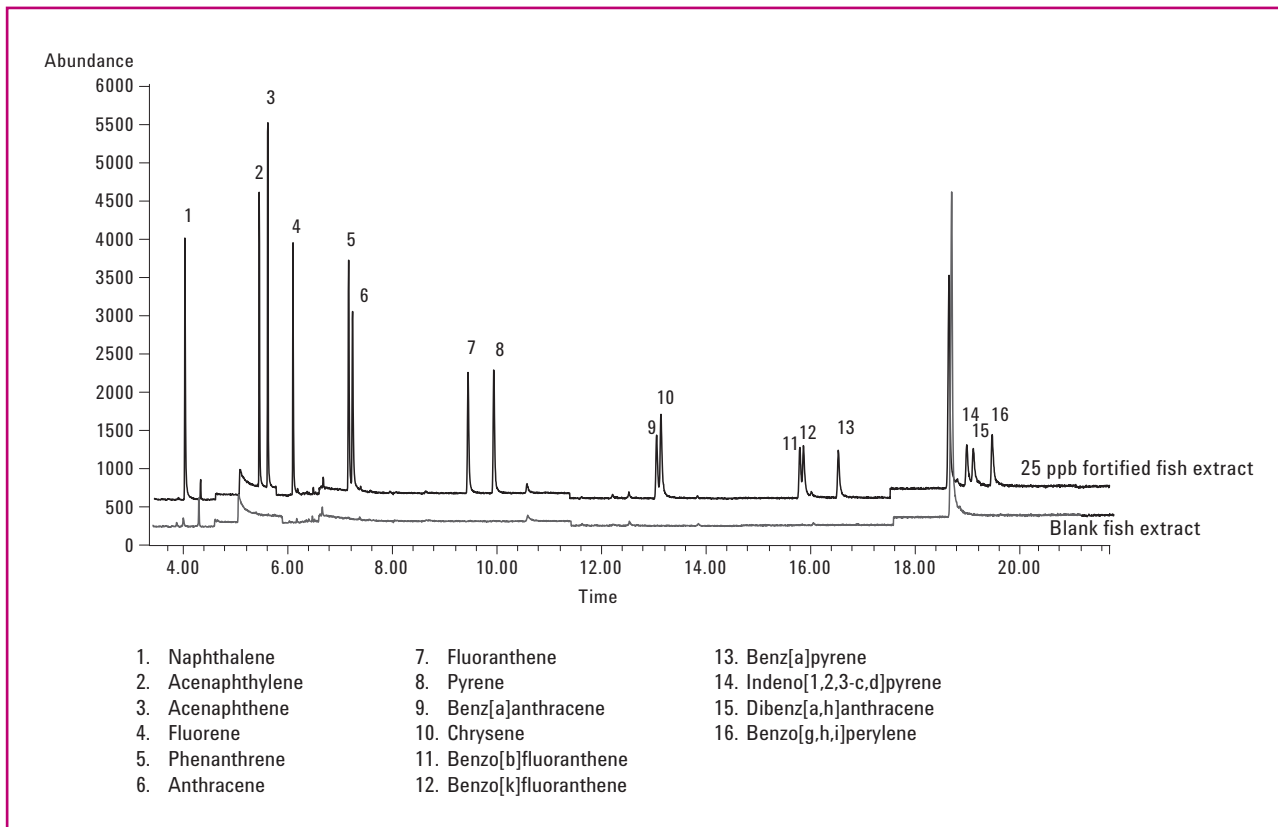


Figure 3. GC/MS SIM chromatogram of the fish extract blank and the 25 ng/mL spiked fish extract analyzed on Agilent J&W DB-5ms Ultra Inert GC capillary column. Chromatographic conditions are listed in Table 1 of the app note.

Products used in the above application

Agilent Bond Elut QuEChERS Extraction Salt Packet. Part No. 5982-6555.

Agilent Bond Elut QuEChERS Fatty Sample Dispersive SPE, 15 mL Tubes. Part No. 5982-5158.

Agilent Bond Elut QuEChERS Ceramic Homogenizers, 50 mL Tubes. Part No. 5982-9313.

Agilent J&W DB-5ms Ultra Inert Column, 20 m × 0.18 mm, 0.18 μm. Part No. 122-5522UI.

To review this Application Note in its entirety, please search for 5990-6668EN at agilent.com/chem

Analysis of Pesticides in Food by GC/MS/MS using the Ultra Inert Liners with Wool (Publication 5990-7706EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach for analyzing multi-residue pesticides. Five kinds of fruits and vegetables were selected: white flowers, banana, strawberry, pear, and lettuce. A representative group of 33 difficult pesticides were selected to evaluate the Agilent Ultra Inert liner. The pesticides standard was spiked in fruit and vegetables matrix blank samples extracted by QuEChERS AOAC method. All testing was done on an Agilent 7890A GC equipped with a 7693B Autosampler and 7000 Series Triple Quadrupole MSD system. The extracted samples were used to demonstrate the performance advantages of Agilent Ultra Inert liners with wool.

QuEChERS Procedure

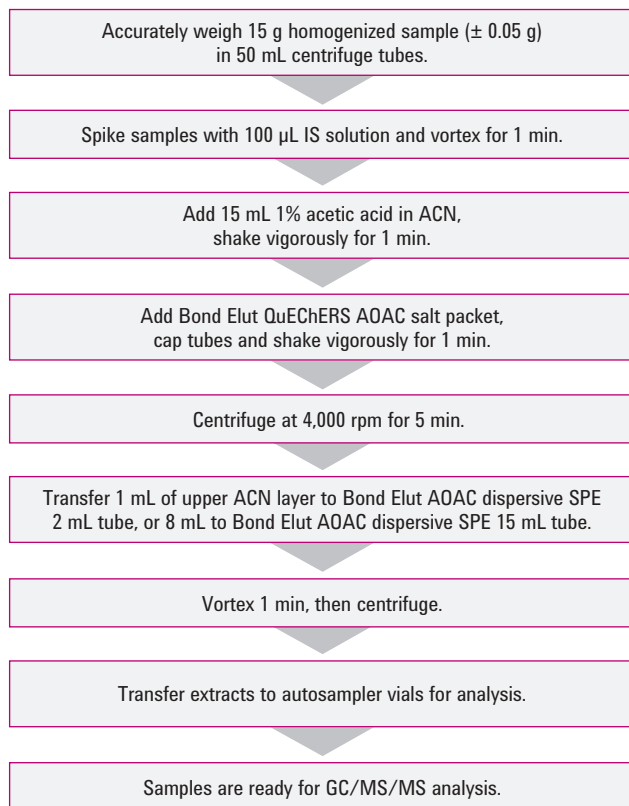


Figure 1. QuEChERS AOAC sample preparation procedures for pesticides in food.

Instrument conditions

GC conditions

| | |
|----------------------------|--|
| Analytical column: | Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 µm (Part No. 19091-431UI) |
| GC: | Agilent 7890A Series |
| Autosampler: | Agilent 7693 Autosampler and sample tray, 5 µL syringe (Part No. 5181-5246), 1 µL injection volume. Postinj solvent A (acetone) washes: 3 Sample pumps: 3 Postinj solvent B (acetonitrile) washes: 3 |
| Carrier gas: | Helium, constant pressure |
| Inlet: | MMI inlet at pulsed splitless mode: 280 °C, |
| Injection pulse pressure: | 36 psi until 1 min |
| Purge flow to split vent: | 50 mL/min at 1 min |
| Inlet pressure: | 18.35 psi (RT locked) during run, and 1.0 psi during back flushing |
| RT locking: | Chlorpyrifos methyl at 8.298 min |
| Oven profile: | 100 °C for 2 min, then to 150 °C at 50 °C/min, to 200 °C at 6 °C/min, to 280 °C at 16 °C/min and hold for 6 min (for sample run); 100 °C for 1 min, then to 280 °C at 100 °C/min and hold for 5.2 min (for matrix blank run) |
| Post run: | 2 min at 280 °C |
| Capillary flow technology: | Purged Ultimate Union (Part No. G3182-61580) – used for backflushing the analytical column and inlet |
| Aux EPC gas: | Helium plumbed to Purge Ultimate Union |
| Bleed line: | 0.0625 in od × 0.010 in id × 100 cm, 316 SS tubing, on top of the oven |
| Aux pressure: | 4 psi during run, 75 psi during backflushing |
| Connections: | Between inlet and Purged Ultimate Union (Part No. G3182-61580) |
| Restrictor: | Inert fused silica tubing, 0.65 m × 0.15 mm (Part No. 160-7625-5) |
| Connections: | Between Purged Ultimate Union and the MSD |
| MSD: | Agilent 7000 Triple Quadrupole Inert with performance electronics |
| Vacuum pump: | Performance turbo |
| Mode: | MRM |
| Tune file: | Atune.u |
| Transfer line temperature: | 280 °C |
| Source temperature: | 300 °C |
| Quad temperature: | Q1 and Q2, 150 °C |
| Solvent delay: | 2.3 min |
| Collision gas flows: | He quench gas at 2.35 mL/min, N ₂ collision gas at 1.5 mL/min |
| MS resolution: | MS1 and MS2, 1.2 u |

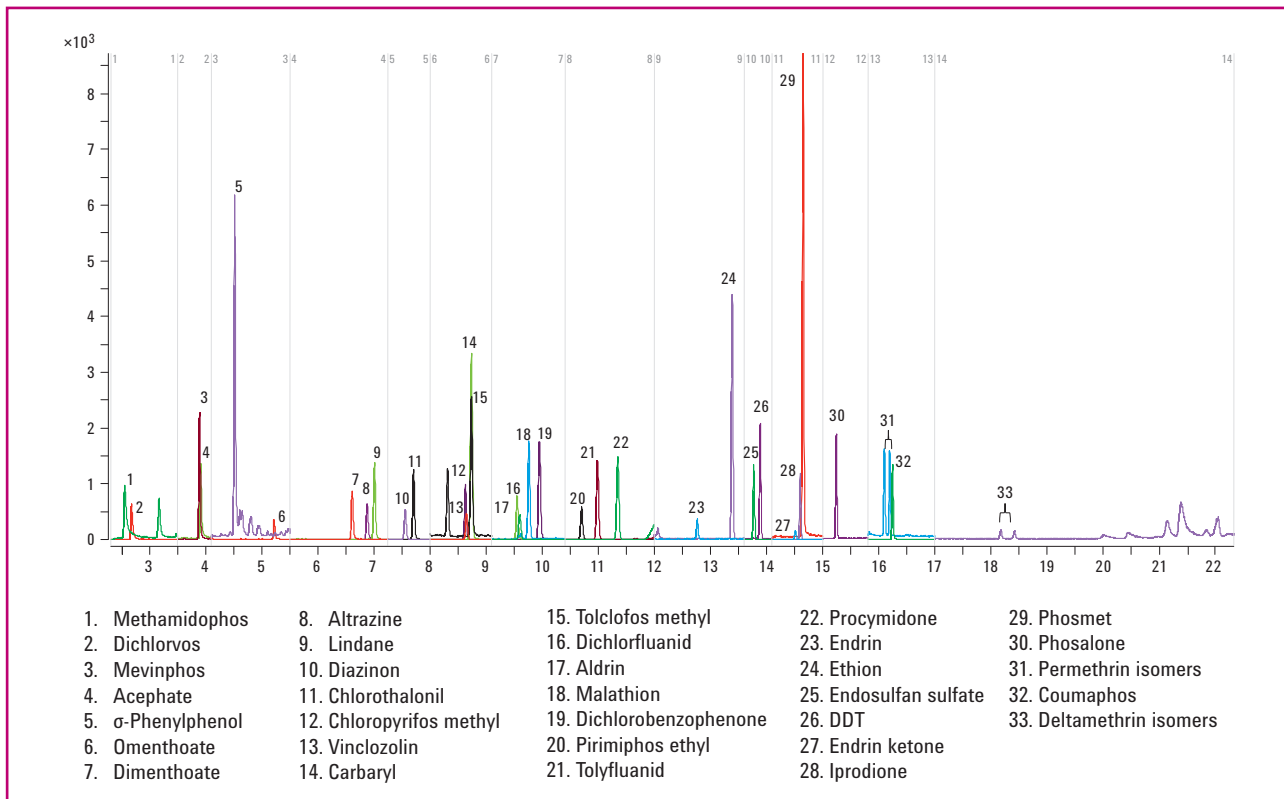


Figure 2. GC/MS/MS chromatogram (MRM) for 10 ppb spiked QuEChERS sample using Ultra Inert liner with wool.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent Bond Elut QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 2 mL. Part No. 5982-5022.

Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm, 0.25 μ m. Part No. 19091-431UI.

To review this Application Note in its entirety, please search for 5990-7706EN at agilent.com/chem

Determination of Chemical Contaminants in Marine Shellfish using the Agilent 7000 Triple Quadrupole GC/MS System (Publication 5990-7714EN)

Introduction

This application describes the use of the QuEChERS sample preparation approach for the determination of selected organochlorine pesticides, polyaromatic hydrocarbons, and polychlorinated biphenyl congeners in marine mussel tissue. The analysis was performed on an Agilent 7890 GC configured with a carbon-dioxide-cooled Multimode Inlet (MMI) with an Agilent 7693A Automatic Liquid Sampler and an Agilent 7000 Triple Quadrupole GC/MS system. In this application, a QuEChERS extraction is coupled with solvent and tube-based SPE clean-up steps to result in final extracts that are suitable for use with GC/QQQ confirmation of the target analytes.

Instrument conditions

GC conditions

| | |
|----------------------------|---|
| Column (1): | Agilent J&W DB-5ms Ultra Inert, 15 m × 0.25 mm id, 0.25 μm (Part No. 122-5512UI) |
| Column (2): | Agilent J&W DB-5ms Ultra Inert, 0.65 m × 0.15 mm id, 0.15 μm (cut from Part No. 165-6626) |
| Injection mode (1): | 1 μL cold pulsed splitless using CO ₂ cooled Multimode Inlet (MMI) and a 10 μL syringe |
| Inlet temperature program: | 50 °C (0.05 min), 600 °C/min to 325 °C |
| Inlet pressure pulse: | 13.0 psig for 0.75 min |
| Purge flow to split vent: | 50 mL/min at 1.0 min |
| Injection port liner: | 2 mm id, multi-baffled (Part No. 5190-2296) |
| Injection mode (2): | 10 μL solvent vent using CO ₂ -cooled Multimode Inlet (MMI) and a 25 μL syringe |
| Inlet temperature program: | 40 °C (0.31 min), 600 °C/min to 325 °C |
| Inlet vent pressure: | 5.0 psig |
| Inlet vent flow: | 100 mL/min |
| Inlet vent time: | 0.31 min |
| Outlet pressure: | 0 psig |
| Injection speed: | 100 μL/min |
| Purge flow to split vent: | 50 mL/min at 1.0 min |
| Injection port liner: | 2 mm id, multi-baffled (Part No. 5190-2296) |
| Carrier gas: | Helium, constant flow 1.2 mL/min |
| Oven temperature program: | 50 °C (1 min), 20-200 °C/min (0 min), 10 °C/min, 300 °C (1.5 min) |
| RTL compound: | PCB 118, locked at 12.370 min |
| Pressure controlled tee: | G3186B, operated at 2.0 psig constant pressure |
| Back flush conditions: | Inlet pressure |

MS conditions

| | |
|------------------------|---|
| MS transfer line temp: | 325 °C |
| MS source: | 300 °C |
| MS quad 1, 2 temp: | 150 °C, 150 °C |
| Collision cell gases: | Nitrogen 1.5 mL/min, helium 2.25 mL/min |
| MS1/MS2 resolution: | Wide/wide |
| MRM settings: | See Table 3 in the app note |
| Electron energy: | -70 eV |
| Ionization mode: | Electron impact (EI) |
| EI Autotune: | Gain normalized |
| Gain factor: | 5 |



QuEChERS Procedure

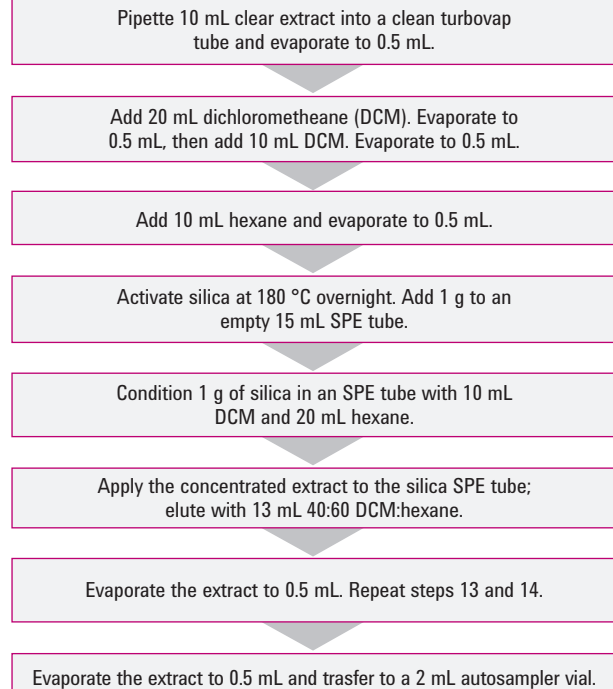
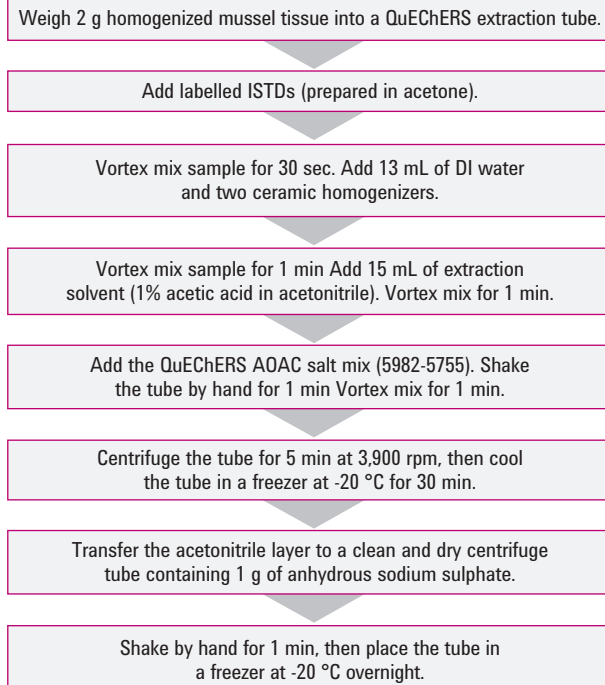


Figure 1. QuEChERS sample extraction and clean-up procedure for contaminants in shellfish.

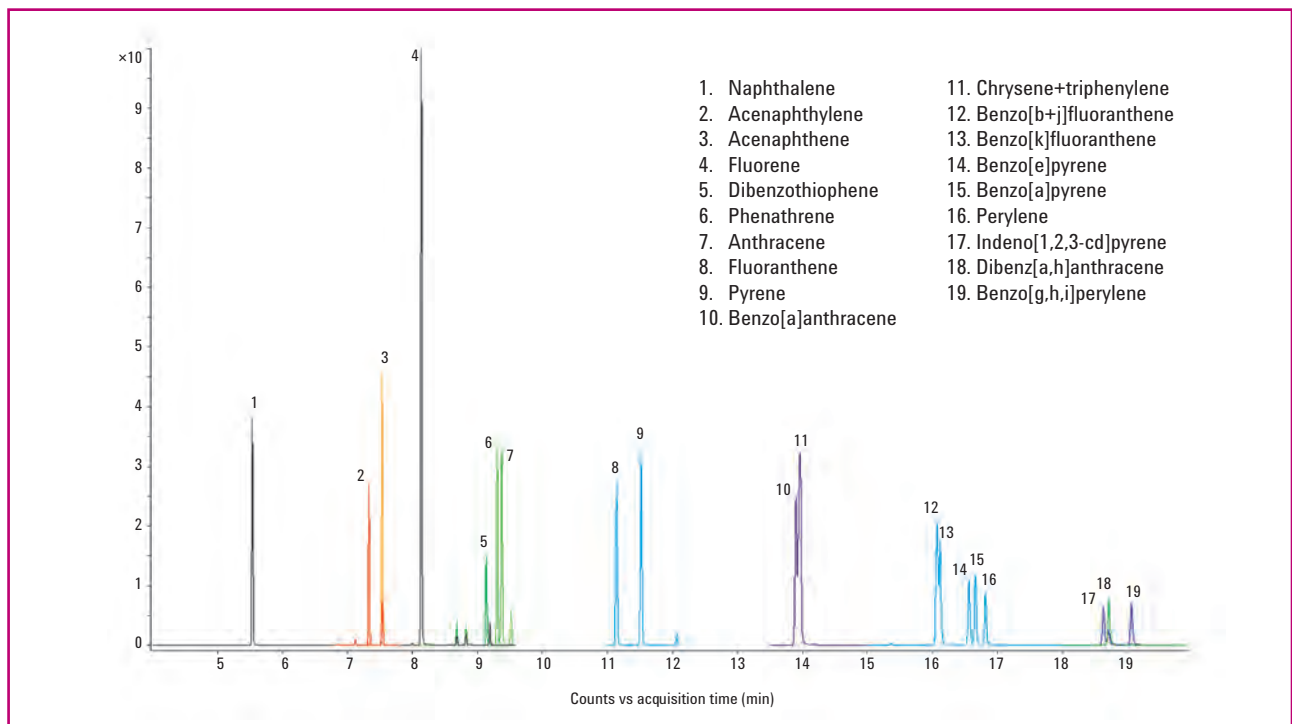


Figure 2. TIC MRM chromatogram for PAH analytes.



Figure 3. Graphical representation of analyte percent recovery values for (a) OCPs, (b) PAHs and (c) PCB congeners in spiked mussel tissue.

Products used in the above application

Agilent Bond Elut QuEChERS Buffered AOAC Extraction Kit. Part No. 5982-5755.

Agilent J&W DB-5ms Ultra Inert Column, 15 m x 0.25 mm, 0.25 μ m. Part No. 122-5512UI.

Agilent J&W DB-5ms Ultra Inert Column, 0.65 m x 0.15 mm, 0.15 μ m. Cut from Part No. 165-6626.

To review this Application Note in its entirety, please search for 5990-7714EN at agilent.com/chem

LC/MS/MS of Trichothecenes and Zearalenone in Wheat using Different Sample Prep Methods (Publication 5990-9107EN)

Introduction

This application describes a modified QuEChERS method and Agilent Bond Elut Mycotoxin SPE for sample preparation and then subsequent detection of trichothecenes and zearalenone by LC/MS/MS. An Agilent 6460 Triple Quadrupole LC/MS with MassHunter Optimizer software was used for this analysis. Recoveries were within 72 to 105% relative to immuno-affinity extractions. Both the QuEChERS approach and the traditional SPE cleanup using the Mycotoxin columns provided good recoveries. Samples prepared using SPE cleanup were cleaner and resulted in lower detection limits, while those prepared using the QuEChERS approach were processed more quickly.

Instrument conditions

HPLC conditions

| | |
|---------------------|---|
| Columns: | Agilent ZORBAX Rapid Resolution HT Eclipse Plus C18, 2.1 × 100 mm, 1.8 μm (Part No. 959764-902) |
| Instrument: | Agilent 6460 Triple Quadrupole LC/MS, Agilent 1290 Infinity LC |
| Flow rate: | 0.25 mL/min |
| Column temperature: | 30 °C |
| Injection volume: | 10 μL |
| Mobile phase: | A: Water + 0.2% acetic acid, 5 mM ammonium acetate B: Methanol + 0.2% acetic acid, 5 mM ammonium acetate |

MS/MS conditions

| | |
|--|------------|
| ESI with Agilent Jet Stream parameters, pos/neg fast polarity switching. | |
| Drying gas temperature: | 200 °C |
| Drying gas flow: | 8 L/min |
| Nebulizer pressure: | 45 psi |
| Sheath gas temperature: | 400 °C |
| Sheath gas flow: | 12 L/min |
| Capillary voltage: | ± 3,000 V |
| Nozzle voltage: | ± 500 V |
| Delta EMV: | 500 V |
| Resolution: | Unit, unit |

QuEChERS Procedure

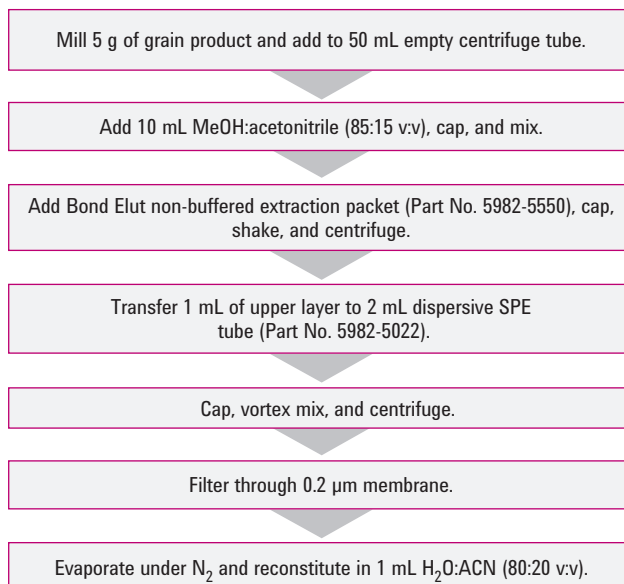


Figure 1. QuEChERS AOAC sample preparation procedures for grains.

Bond Elut Mycotoxin Method

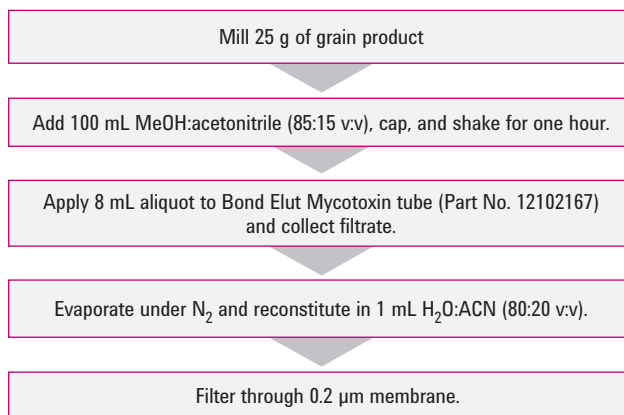


Figure 2. Bond Elut Mycotoxin SPE sample preparation method for grain mycotoxins.

Table 1. MS conditions for the analysis of trichothecenes and zearalenone

| Mycotoxin | Precursor ion | Product ion | Fragmentor | Collision energy | Polarity |
|--------------------------|---------------|-------------|------------|------------------|----------|
| 15-acetyl Deoxynivalenol | 356 | 321 | 95 | 5 | Positive |
| 15-acetyl Deoxynivalenol | 356 | 137 | 95 | 8 | Positive |
| 15-acetyl Deoxynivalenol | 339 | 137 | 105 | 12 | Positive |
| 3-acetyl Deoxynivalenol | 397 | 337 | 95 | 4 | Negative |
| 3-acetyl Deoxynivalenol | 397 | 59 | 95 | 20 | Negative |
| Diacetoxyscirpenol (DAS) | 384 | 307 | 105 | 4 | Positive |
| Diacetoxyscirpenol (DAS) | 384 | 247 | 105 | 6 | Positive |
| Deoxynivalenol (DON) | 355 | 265 | 95 | 4 | Negative |
| Deoxynivalenol (DON) | 355 | 59 | 95 | 20 | Negative |
| Fusarenone-X | 413 | 263 | 95 | 8 | Negative |
| Fusarenone-X | 413 | 59 | 95 | 28 | Negative |
| HT-2 toxin | 442 | 263 | 105 | 4 | Positive |
| HT-2 toxin | 442 | 215 | 105 | 4 | Positive |
| Neosolaniol | 400 | 215 | 95 | 16 | Positive |
| Neosolaniol | 400 | 185 | 95 | 16 | Positive |
| Nivalenol | 371 | 311 | 108 | 4 | Negative |
| Nivalenol | 371 | 281 | 108 | 8 | Negative |
| Nivalenol | 371 | 59 | 108 | 24 | Negative |
| T-2 toxin | 484 | 215 | 120 | 16 | Positive |
| T-2 toxin | 484 | 185 | 120 | 14 | Positive |
| Zearalenone | 317 | 175 | 190 | 16 | Negative |
| Zearalenone | 317 | 131 | 190 | 24 | Negative |
| Zearalanone | 319 | 275 | 185 | 16 | Negative |
| Zearalanone | 319 | 205 | 185 | 16 | Negative |

Table 2. Comparison of modified QuEChERS and cartridge SPE recovery data spiked in wheat samples at 50 µg/kg (n=9)

| Analyte | Analyte retention time (min) | Modified QuEChERS recovery (%) | Modified QuEChERS RSD (%) | BE Mycotoxin SPE recovery (%) | BE Mycotoxin SPE RSD (%) |
|--------------------------|------------------------------|--------------------------------|---------------------------|-------------------------------|--------------------------|
| Nivalenol | 5.6 | 73 | 7 | 93 | 11 |
| Deoxynivalenol | 6.4 | 85 | 8 | 84 | 11 |
| Fusarenon-X | 6.8 | 81 | 9 | 89 | 9 |
| Neosolaniol | 6.8 | 94 | 9 | 77 | 9 |
| 15-acetyl Deoxynivalenol | 7.2 | 88 | 9 | 72 | 10 |
| 3-acetyl Deoxynivalenol | 7.2 | 100 | 9 | 92 | 11 |
| Diacetoxyscirpenol | 7.7 | 105 | 2 | 104 | 3 |
| HT-2 | 8.0 | 83 | 8 | 99 | 4 |
| T-2 toxin | 8.2 | 83 | 8 | 100 | 4 |
| Zearalenone | 8.4 | 87 | 8 | 79 | 9 |

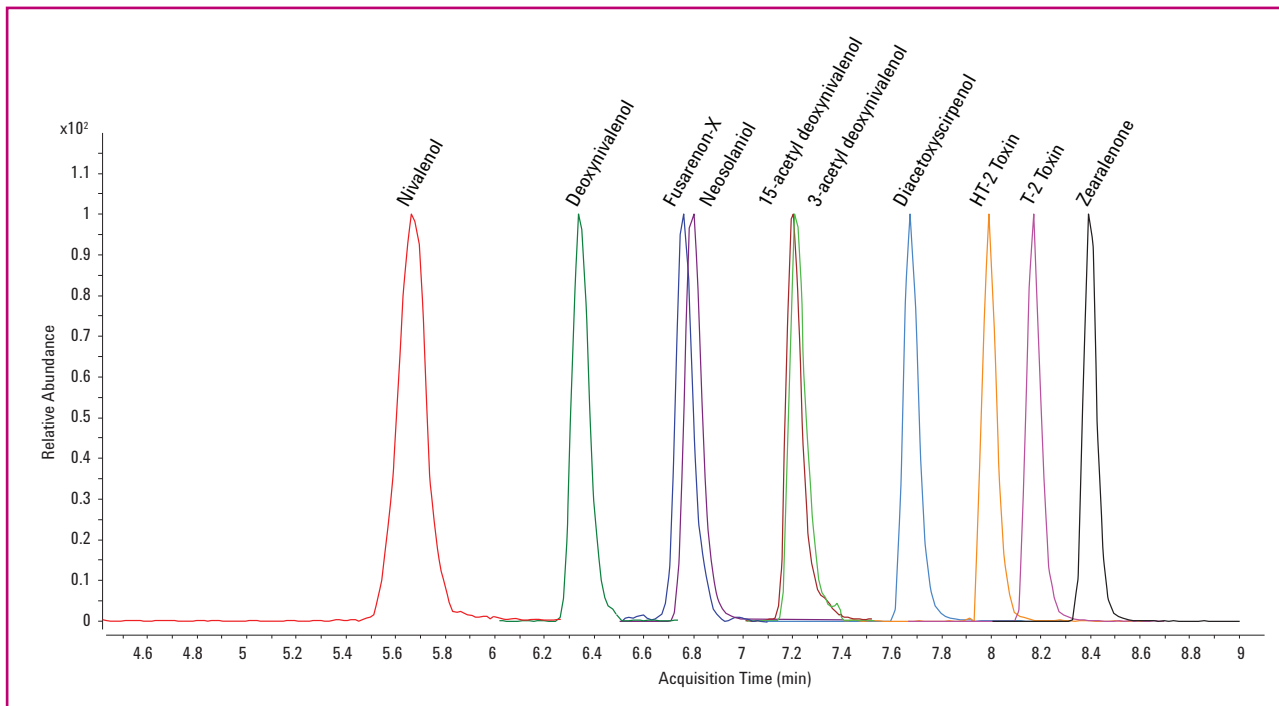


Figure 3. 50 ppb wheat matrix standard: normalized chromatogram for primary transitions. Please note that this method does not require chromatographic resolution between the isomers 15-acetyl DON and 3-acetyl DON because they are distinguished through measurement under different mass spec polarities.

Products used in the above application

Agilent Bond Elut QuEChERS Nonbuffered Extraction Packet. Part No. 5982-5550.

Agilent Bond Elut QuEChERS Dispersive SPE Kit. Part No. 5982-5022.

Agilent Bond Elut Mycotoxin SPE Cartridge, 500 mg, 3 mL. Part No. 12102167.

Agilent ZORBAX Rapid Resolution HT Eclipse Plus C18 Column, 2.1 x 100 mm, 1.8 μ m. Part No. 959764-902.



To review this Application Note in its entirety, please search for 5990-9107EN at agilent.com/chem

Determination of Multi-Pesticide Residues in Dried Tea Samples using an Optimized Extraction/Cleanup Regime and the Agilent 7000 Series Triple Quadrupole GC/MS System (Publication 5990-9865EN)

Introduction

This application describes the use of an optimized QuEChERS sample preparation approach to effectively extract pesticides from tea, while at the same time minimizing the extraction of caffeine and other co-extractives, which can cause degenerative effects on chromatographic peak shape, analyte retention time shifts and loss of sensitivity. The tea extracts were analyzed by GC/MS/MS using MRM mode on an Agilent 7890 GC with an Agilent 7000B Triple Quadrupole GC/MS system. This novel approach uses a QuEChERS extraction followed by clean-up using liquid extraction for consistent results.

QuEChERS Procedure

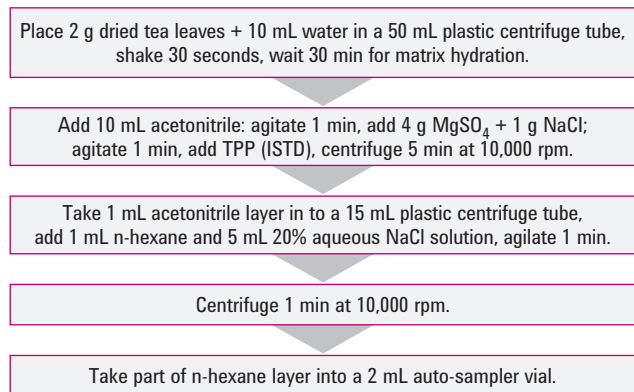


Figure 1. QuEChERS sample extraction and clean-up regime for tea.

Instrument conditions

GC conditions

| | |
|-------------------------------|--|
| Column (1): | Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm, 0.25 μm (Part No. 19091S-431UI) |
| Column (2): | Agilent J&W DB-5ms Ultra Inert, 0.50 m × 0.15 mm, 0.15 μm (cut from Part No. 165-6626) |
| Capillary flow device: | Pressure controlled tee (PCT) with pneumatics control module (PCM) |
| Auto-sampler: | Agilent 7693A Automatic Liquid Sampler |
| Injection: | 2 μL cold splitless using CO ₂ -cooled Multimode Inlet (MMI) |
| Splitless period: | 1 min |
| Injection: | Port liner 2 mm id dimpled deactivated liner (Part No. 5190-2296) |
| Inlet temperature program: | 50 °C (0.1 min), 600 °C/min to 300 °C |
| Purge flow to split vent: | 50 mL/min at 1.0 min |
| RTL compound: | Trifluralin, locked to 6.219 min |
| Carrier gas: | Helium |
| Inlet pressure: | 17.460 psig constant pressure mode (during run) |
| PCM pressure: | 2.0 psig constant pressure mode (during run) |
| Oven program: | 50 °C (1.0 min), 50 °C/min to 150 °C, 6 °C/min to 200 °C, 16 °C/min to 280 °C (4.07 min) |
| Post-run time: | 2.0 min |
| Post-run temperature: | 280 °C |
| Post-run pressures: | Inlet 1.0 psig, PCM 60.0 psig |
| MS transfer line temperature: | 280 °C |

MS conditions

| | |
|---------------------------|--|
| Ionization mode: | Electron ionization |
| Electron energy: | -70 eV |
| Tune: | El autotune |
| EM gain: | 10 |
| MS1 resolution: | 1.2 amu full width at half maximum |
| MS2 resolution: | 1.2 amu full width at half maximum |
| Transitions: | See reference [2] on app note |
| Collision energies: | See reference [2] on app note |
| Dwell times: | 2-28 ms depending on the number of transitions per time window to achieve 5 cycles/sec |
| Collision cell gas flows: | Nitrogen at 1.5 mL/min, helium at 2.25 mL/min |
| MS temperature zones: | Ion source 280 °C, Q1 150 °C, Q2 150 °C |

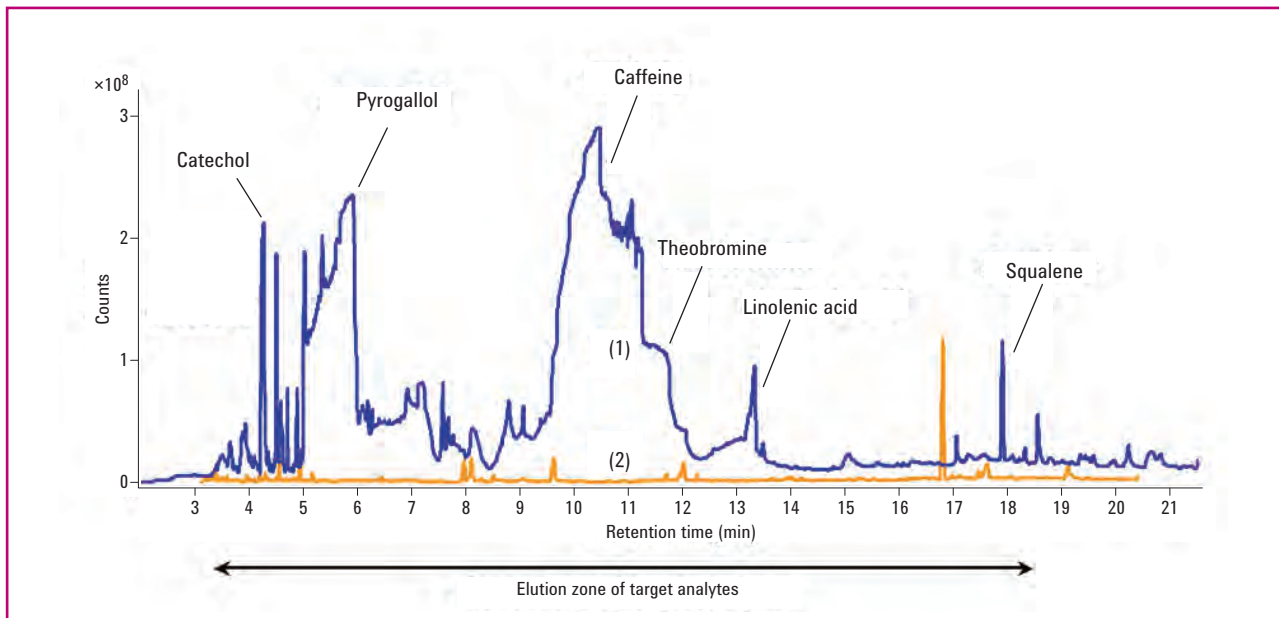


Figure 2. Full scan MS chromatograms showing very high abundance of caffeine and other co-extractives remaining in the acetonitrile extract after the QuEChERS extraction (1 blue trace) compared to their significant reduction when subsequent liquid-liquid extraction is employed (2 yellow trace).

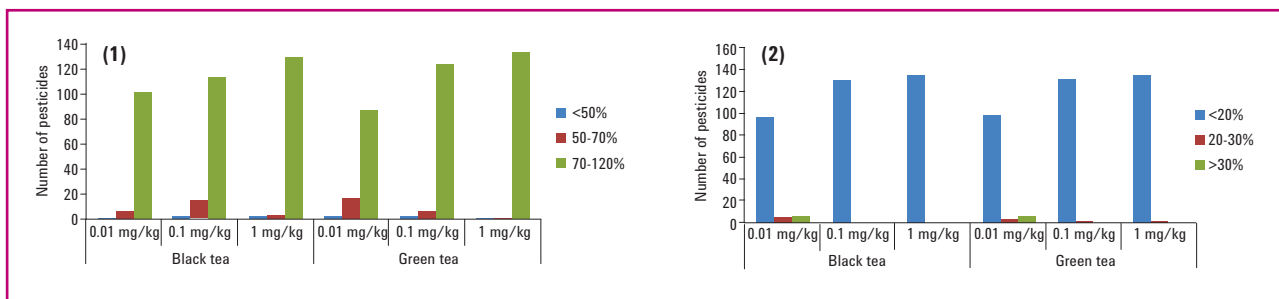


Figure 3. Distribution of (1) overall recoveries and (2) RSDs for the pesticide residues in green and black tea at spiking levels of 0.01, 0.1 and 1 mg/kg.

Products used in the above application

Agilent Bond Elut QuEChERS Nonbuffered Extraction Packet. Part No. 5982-5550.

Agilent J&W HP-5ms Ultra Inert Column, 15 m x 0.25 mm, 0.25 μ m. Part No. 19091S-431UI.

Agilent J&W DB-5ms Ultra Inert Column, 0.50 m x 0.15 mm, 0.15 μ m. Cut from Part No. 165-6626.

To review this Application Note in its entirety, please search for 5990-9865EN at agilent.com/chem

Screening 36 Veterinary Drugs in Animal Origin Food by LC/MS/MS Combined with Modified QuEChERS Method (Publication 5991-0013EN)

Introduction

This application introduces a modified QuEChERS method that screens food for four classes of veterinary drugs: sulfanilamides, macrocyclic lactones, quinolones, and clolidols. Satisfactory recoveries were achieved by this method for all four classes of veterinary drugs. Instrumental analysis was performed using the Agilent 1260 Infinity LC with Diode Array Detector and the Agilent 6460 Triple Quadrupole LC/MS with the Agilent Jet Stream technology electrospray ionization source. In addition to the meat matrix, the method was successfully used on egg, milk, and honey samples.

QuEChERS Procedure

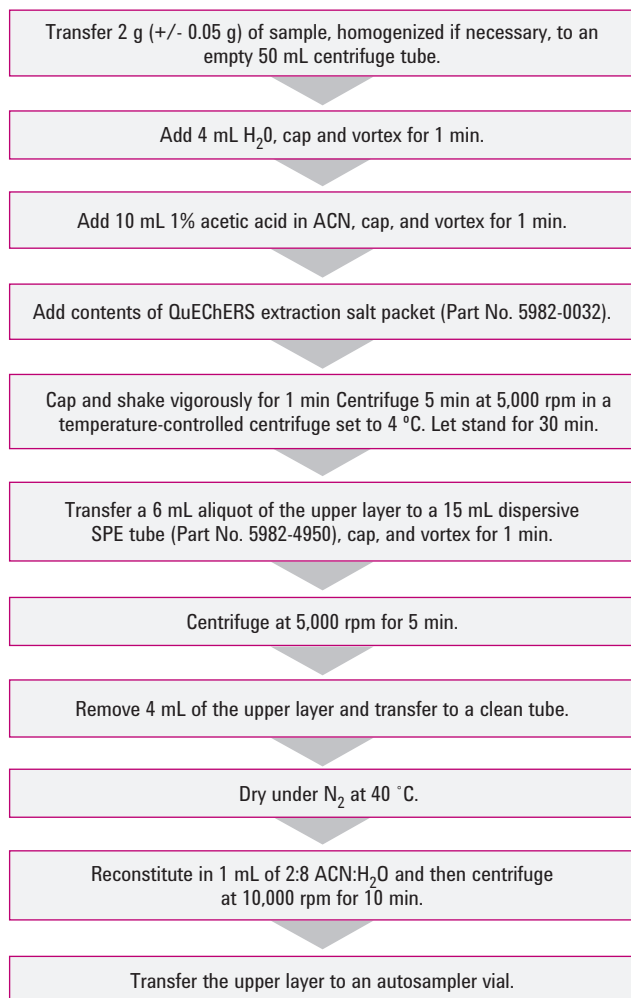


Figure 1. QuEChERS AOAC sample preparation procedures for veterinary drugs in foods of animal origin.

Instrument conditions

HPLC conditions

| | | | |
|---------------------|--|----|----|
| Column: | Agilent ZORBAX Solvent Saver HD Eclipse Plus C18, 3.0 × 100 mm, 1.8 μm | | |
| Flow rate: | 0.5 mL/min | | |
| Column temperature: | 30 °C | | |
| Injection volume: | 5 μL | | |
| Mobile phase: | A: H ₂ O 0.1% formic acid B: ACN | | |
| Gradient: | Time (min) | %A | %B |
| | 0.0 | 90 | 10 |
| | 0.5 | 90 | 10 |
| | 1.0 | 80 | 20 |
| | 4.0 | 75 | 25 |
| | 8.0 | 40 | 60 |
| | 9.0 | 5 | 95 |
| | 12.0 | 5 | 95 |
| | 12.1 | 90 | 10 |
| | 15.0 | 90 | 10 |

MS conditions

| | |
|-------------------------|----------|
| Polarity: | Positive |
| Gas temperature: | 300 °C |
| Gas flow: | 7 L/min |
| Nebulizer: | 50 psi |
| Capillary: | 3,000 V |
| Sheath gas temperature: | 350 °C |
| Sheath gas flow: | 10 L/min |
| Scan mode: | DMRM |



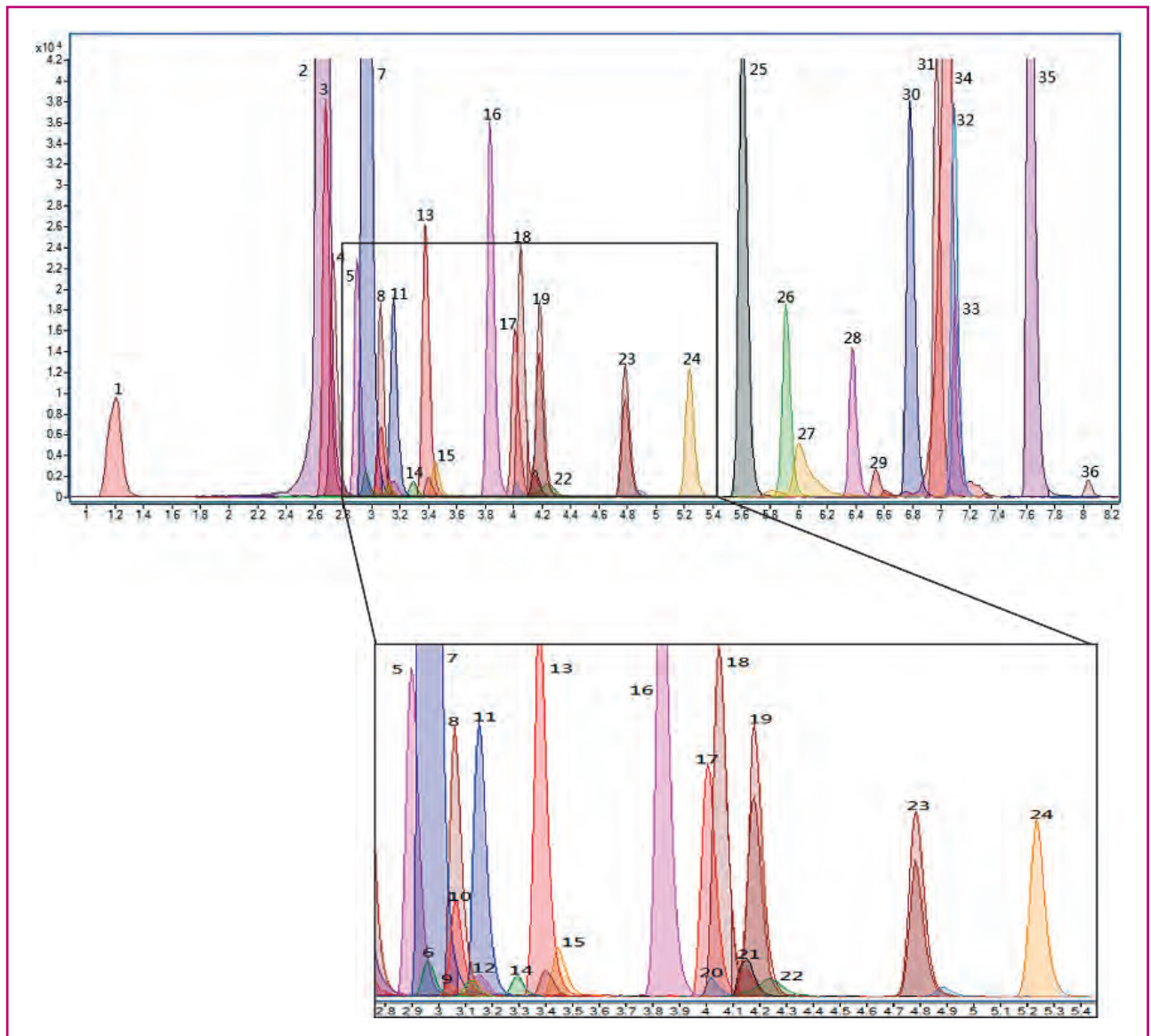


Figure 2. MRM extracted chromatogram for veterinary drugs.

Table 1. Results of optimization of the extraction step. An extraction salt mixture including Na₂SO₄ was selected based on better recoveries

| Method | 1 | 2 | 3 | 4 |
|--|--------------------------------|---------------------------------------|---------------------------------------|---------------------------------------|
| Extract salt | MgSO ₄ +NaCl | Na ₂ SO ₄ +NaCl | Na ₂ SO ₄ +NaCl | Na ₂ SO ₄ +NaCl |
| Dispersive-SPE mix | C18EC+MgSO ₄ | C18EC+Na ₂ SO ₄ | C18EC+Na ₂ SO ₄ | C18EC+Na ₂ SO ₄ |
| Extract solvent | 1% formic acid acetonitrile | 1% formic acid acetonitrile | 1% formic acid acetonitrile | 1% formic acid acetonitrile |
| Water | 8 mL | 8 mL | 4 mL | 4 mL |
| Average recovery of macrocyclic lactones | 22.95% | 43.66% | 45.12% | 70.46% |
| Average recovery of sulfanilamides | 10.86% | 25.96% | 33.25% | 54.35% |
| Average recovery of quinolones | 86.79% | 47.69% | 62.69% | 64.44% |
| Average recovery of clopidols | 55.12% | 38.02% | 49.89% | 65.37% |
| Zearalenone | 8.4 | 87 | 8 | 79 |

Table 2. Results of dispersive SPE parameter optimization. A dSPE mixture including PSA was selected for greater versatility.

| Method | 1 | 2 | 3 | 4 | 5 |
|---|---|--|---|--|---|
| Extract salt | Na ₂ SO ₄ +NaCl | Na ₂ SO ₄ +NaCl | Na ₂ SO ₄ +NaCl | Na ₂ SO ₄ +NaCl | Na ₂ SO ₄ +NaCl |
| Dispersive-SPE mix | 50 mg PSA+ 150 mg C18EC+ 900 mg Na ₂ SO ₄ | 100 mg PSA+ 150 mg C18EC+ 900 mg Na ₂ SO ₄ | 50 mg SAX+ 150 mg C18EC+ 900 mg Na ₂ SO ₄ | 100 mg NH2+ 150 mg C18EC+ 900 mg Na ₂ SO ₄ | 300 mg C18EC+ 900 mg Na ₂ SO ₄ |
| Extract solvent | 1% acetic acid acetonitrile | 1% acetic acid acetonitrile | 1% acetic acid acetonitrile | 1% acetic acid acetonitrile | 1% acetic acid acetonitrile |
| Water | 4 mL | 4 mL | 4 mL | 4 mL | 4 mL |
| Average recovery of macrocyclic lactone | 54.57% | 42.23% | 59.87% | 33.70% | 66.10% |
| Average recovery of sulfanilamide | 64.37 % | 63.27 % | 77.35% | 51.71% | 71.80% |
| Average recovery of quinolone | 73.88% | 88.34 % | 76.82% | 97.03% | 84.66% |
| Average recovery of clopidol | 85.12% | 100.11% | 71.57% | 70.27% | 91.17% |

Products used in the above application

Agilent Bond Elut Extraction Tubes. Part No. 5982-0032.

Agilent Bond Elut QuEChERS Dispersive SPE Kits for Drug Residues. Part No. 5982-4950.

Agilent ZORBAX Solvent Saver HD Eclipse Plus C18 Column, 3.0 x 100 mm, 1.8 μm. Part No. 959757-302.

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