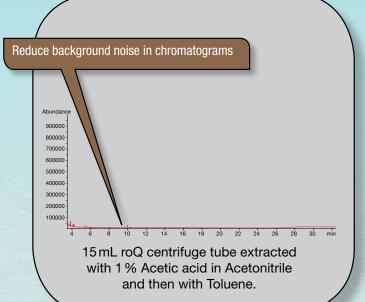


# Cleaner Extracts with Low Extractable Tubes





No Salt Spills with Easy Pour Salt Packets

Avoid Leaky Tubes with No Leak Caps



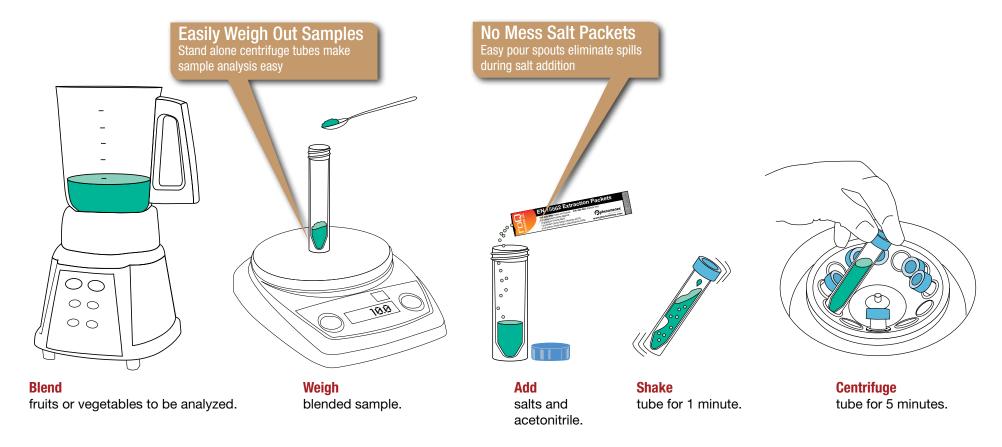
# How does it work?

The QuEChERS technique addresses shortcomings of traditional sample preparation methods, such as long extraction procedures and the use of hazardous solvents, and radically simplifies multi-residue pesticide analysis in food or environmental samples.

The QuEChERS technique consists of two steps; Extraction and Dispersive SPE (dSPE)

## **Step 1: Extraction**

Pesticides and analytes of interest must first be extracted from the sample. This process relies on the combination of organic solvent and various salts to partition the analytes from samples into an organic layer (typically acetonitrile).



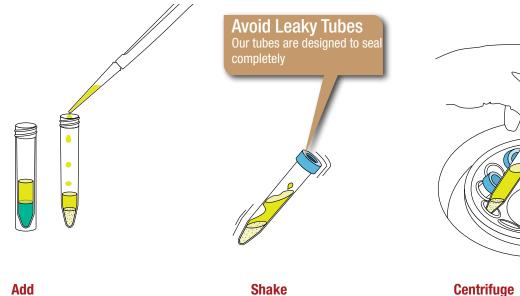


# **Step 2: Dispersive SPE (dSPE)**

supernatant from extraction procedure into

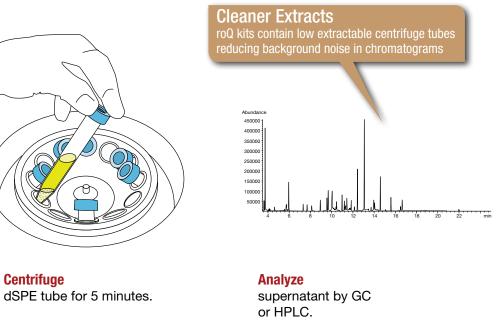
a roQ dSPE tube.

An aliquot of the organic layer from the extraction step is subjected to further clean up by dispersive SPE. This step selectively removes unwanted interferences such as lipids and pigments.



dSPE tube for

30 seconds.



## Step 1

# roQ<sup>™</sup> Extraction Kits

QuEChERS can be performed by following 3 different methods: the AOAC 2007.01 Method, the EN 15662 Method, or the Original Non-Buffered Method.

Use the chart below to select the most appropriate kits for your work and sample size/type.

#### AOAC 2007.01 Method

Salts used:

#### Magnesium Sulfate (MgSO<sub>4</sub>)

Induces phase separation between water content in sample and acetonitrile layer

#### **Sodium Acetate (NaOAc)**

Buffers the sample to stabilize pH

## **Original Non-Buffered Method**

Salts used:

#### Magnesium Sulfate (MgSO<sub>4</sub>)

Induces phase separation between water content in sample and acetonitrile layer

#### **Sodium Chloride (NaCl)**

Induces phase separation between water content in sample and acetonitrile layer

#### EN 15662 Method

Salts used:

#### Magnesium Sulfate (MgSO<sub>4</sub>)

Induces phase separation between water content in sample and acetonitrile layer

#### **Sodium Chloride (NaCl)**

Induces phase separation between water content in sample and acetonitrile layer

#### **Sodium Citrate Tribasic Dihydrate (SCTD)**

Buffers the sample to stabilize pH

#### **Sodium Citrate Dibasic Sesquihydrate (SCDS)**

Buffers the sample to stabilize pH

## Sample Size

15 g

Part No.: KS0-8911

15 g

Part No.: **KS0-8912** 

to be used with AOAC 2007.01 dSPE procedure

or

10 g

Part No.: KS0-8910

to be used with EN 15662 dSPE procedure

10 g

Part No.: **KS0-8909** 

All roQ Extraction Kits include 50 easy-pour salt packets and fifty 50 mL stand-alone centrifuge tubes.

# Step 2

# **Select Your roQ™ dSPE Kit**

		Fruit and vegetable Type									
		General		Fats and Waxes		Pigmented		Highly Pigmented		Pigments and Fats	
	Sample										
Method	Size	Contents	Part No.	Contents	Part No.	Contents	Part No.	Contents	Part No.	Contents	Part No.
AOAC	1 mL	150 mg MgSO₄ 50 mg PSA	KS0-9511	150 mg MgSO <sub>4</sub> 50 mg PSA 50 mg C18E	KS0-9512	150 mg MgSO <sub>4</sub> 50 mg PSA 50 mg GCB	KS0-9513	_	_	150 mg MgSO <sub>4</sub> 50 mg PSA 50 mg GCB 50 mg C18E	KS0-9514
2007.01	8 mL	1200 mg MgSO <sub>4</sub> 400 mg PSA	KS0-9515	1200 mg MgSO <sub>4</sub> 400 mg PSA 400 mg C18E	KS0-9516	1200 mg MgSO <sub>4</sub> 400 mg PSA 400 mg GCB	KS0-9517	_	_	1200 mg MgSO <sub>4</sub> 400 mg PSA 400 mg GCB 400 mg C18E	KS0-9518
EN 15662	1 mL	150 mg MgSO₄ 25 mg PSA	KS0-9503	150 mg MgSO <sub>4</sub> 25 mg PSA 25 mg C18E	KS0-9504	150 mg MgSO <sub>4</sub> 25 mg PSA 2.5 mg GCB	KS0-9505	150 mg MgSO <sub>4</sub> 25 mg PSA 7.5 mg GCB	KS0-9506	_	_
	6 mL	900 mg MgSO <sub>4</sub> 150 mg PSA	KS0-9507	900 mg MgSO <sub>4</sub> 150 mg PSA 150 mg C18E	KS0-9508	900 mg MgSO <sub>4</sub> 150 mg PSA 15 mg GCB	KS0-9509	900 mg MgSO <sub>4</sub> 150 mg PSA 45 mg GCB	KS0-9510	_	_

Fruit and Vegetable Type

Salts and sorbents used in roQ dSPE kits					
Magnesium Sulfate (MgSO₄)	Removes excess water from sample				
Primary/Secondary Amine (PSA)	Removes organic acids, fatty acids, sugars, and anthocyanin pigments from sample				
Endcapped C18 Sorbent (C18E)	Removes fats, sterols, and other non-polar interferences from sample				
Graphitized Carbon Black (GCB)	Removes pigments from sample NOT FOR USE WITH PLANAR PESTICIDES				

All roQ dSPE Kits include pre-weighed sorbents/salts inside 2 mL or 15 mL centrifuge tubes.

## Extraction of Pesticide Residues from Kale and Grapes Using roQ™ EN 15662 QuEChERS Kit

#### LC-MS Analysis

Over 200 pesticides were screened at concentration ranges between 0.01 ppm to 1 ppm with the majority of analytes having a recovery range of 70-130%. The method produced excellent selectivity and reproducibility for the earlier eluting polar pesticides owing to the use of QuEChERS for removing sample matrix interferences and the biphenyl bonded-phase chemistry of the Kinetex® Biphenyl LC column for the chromatographic separation across the entire range of pesticides.

#### **QuEChERS Procedure (EN 15662 Method)**

Kale and grapes samples were prepared at four concentrations: no spike (0.0 ppm), low spike (0.1 ppm), medium spike (0.5 ppm), and high spike (1 ppm)

Pretreatment: Kale and grapes were frozen at ~-80 °C overnight

#### **Step 1: Extraction**

- Homogenize sample using a blender or similar apparatus.
- Weigh 10 g of homogenized sample into a clean 50 mL tube (provided in roQ Extraction Kits).
- 3. Add 10 mL of Acetonitrile containing internal standard.
- Dispense contents of the included QuEChERS salt packet into the 50 mL tube containing homogenized sample.
- 5. Shake vigorously by hand for 1 minute.
- Centrifuge for 5 minutes @ 4000 rpm, making sure that the solid material is at the bottom of the tube and a liquid layer forms on top of the solid material.

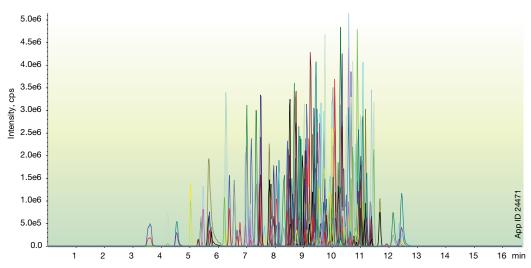
#### Step 2: Dispersive SPE (dSPE)

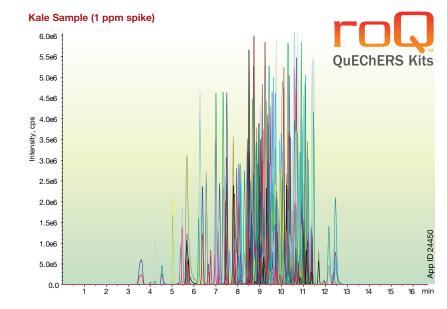
- Transfer 6 mL of supernatant from Step 6 of the extraction process into the 15 mL tube containing the QuEChERS dSPE sorbents.
- 2. Shake vigorously by hand for 30 seconds.
- Centrifuge for 5 minutes @ 4000 rpm to separate solid material from the liquid layer.
- 4. Transfer desired supernatant to an autosampler vial.
- 5. Dilute samples 1:10 in mobile phase A prior to injection or LC-MS analysis.





#### Standard Chromatogram (0.5 ppm standard)



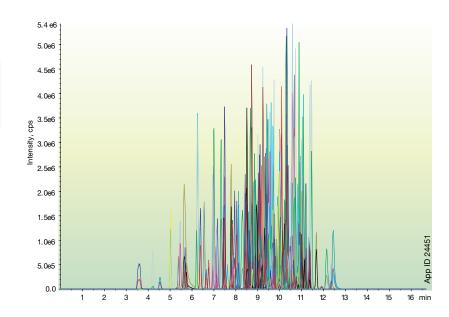


#### **LC-MS/MS Conditions**

Flow Rate: 0.5 mL/min Column: Kinetex® 5 µm Biphenyl Inj. Volume: 20 µL Dimensions: 50 x 4.6 mm Temperature: 35 °C Part No.: 00B-4627-E0 Detection: MS/MS (ESI+), scheduled MRM SecurityGuard™ ULTRA: AJ0-9207 Instrument: SCIEX 4000 QTRAP® Mobile Phase: A: 10mM Ammonium Formate in Water B: Methanol Gradient: Time (min) 0 2 2 10 100 13 100 13.1 2 www.phenomenex.com and 16

#### Grapes Sample (0.5 ppm spike)

TN-0115



# Mycotoxins from Corn Meal Products Using roQ™ EN 15662 QuEChERS Kit

#### LC-MS/MS

The use of roQ QuEChERS and Kinetex® XB-C18 Core-Shell Technology LC columns deliver a rapid and simple approach for mycotoxin screening from corn products.

### **QuEChERS Procedure (EN 15662 Method)**

#### **Step 1: Extraction from Ground Corn**

- 1. Homogenize sample using a blender or similar apparatus
- 2. Weigh and transfer 5 g of ground corn meal to a 50 mL roQ QuEChERS extraction tube
- Add 10 mL of water and 10 mL of acetonitrile with 1.0 % formic acid
- Dispense contents of the included roQ QuEChERS extraction packet (KS0-8909) into the 50 mL tube containing homogenized sample
- 5. Shake vigorously by hand for 1 minute
- Centrifuge for 5 minutes @ 4000 rpm, making sure that the solid material is at the bottom of the tube and a liquid layer forms on top of the solid material

# Step 2: Clean up using dispersive Solid Phase Extraction (dSPE)

- Transfer the supernatant from Step 6 of the extraction process into a roQ QuEChERS 15 mL centrifuge tube containing 900 mg MgSO<sub>4</sub> and 150 mg PSA (KS0-9507)
- 2. Shake vigorously by hand for 30 seconds
- Centrifuge for 5 minutes at 4000 rpm to separate solid material from the liquid layer
- 4. Transfer the supernatant to a vessel for evaporation

#### **LC-MS/MS Conditions**

Column: Kinetex® 2.6 µm XB-C18
Dimensions: 50 x 2.1 mm
Part No.: 00B-4496-AN

 $\textbf{SecurityGuard Cartridge:} \ \, \text{AJ0-8782}$ 

Mobile Phase: A: 5 mM Ammonium acetate with 0.5 % Acetic acid

B: 5 mM Ammonium acetate in Methanol with 0.5 % Acetic acid

 Gradient:
 Time (min)
 % B

 0
 5

 2
 5

 5
 80

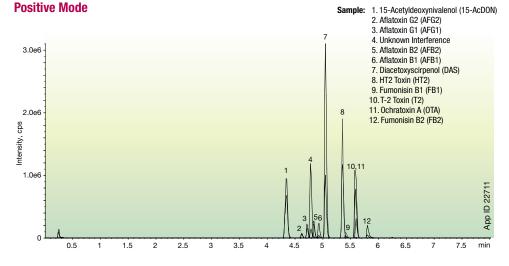
 5.2
 98

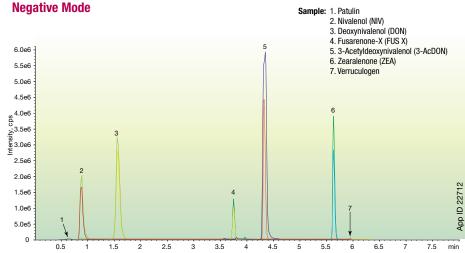
 8
 98

Flow Rate: 0.45 mL/min Temperature: Ambient Injection Volume: 25 uL

**Detection:** Tandem Mass Spec (MS/MS)

Instrument: SCIEX API 5000™







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## **Determination of PFASs in Sediments Using roQ™ AOAC 2007.01 QuECHERS Kits**

LC-MS Analysis

In order to extract Perfluoroalkyl substances (PFASs) from marine and freshwater sediment, QuEChERS was introduced to save time and offer reliable results.

#### **QuEChERS Procedure (AOAC 2007.01 Method)**

#### **Step 1: Extraction from Sediments**

- 1. Weigh 2.0 g of suitably dried sediment into a polypropylene container and spike with isotopically-labeled internal standards. PPCPs, Steroids, and Pyrethroids can be extracted concurrently with this method by adding the appropriate internal standard and spiking solutions to the samples and QCs.
- 2. Add 10 mL deionized water and vortex. Add 10 mL acidified acetonitrile (1 % Acetic acid) to the slurry and vortex.
- Add the extraction salts (1.5 g Sodium acetate and 2 g MgSO<sub>4</sub>) to the sample and vortex for 1 minute.
- Centrifuge the samples for 5 minutes at 4000 rpm.
- Place the samples in a rack and freeze at -20 °C for 30-60 minutes. This freezing step allows for easier extraction of the supernatant.

#### Step 2: Clean up Using dispersive Solid Phase Extraction (dSPE)

- Transfer 8-9 mL of the acetonitrile supernatant into a roQ QuEChERS PSA/C18 dSPE clean-up tube (Part no. KS0-9516)
- Vortex for one minute.
- Centrifuge the dSPE tubes for 10 minutes at 3000 rpm.
- Place an aliquot of the extract in a polypropylene HPLC vial and dilute 1:1 with deionized water. The sample is now ready for analysis.

#### LC-MS/MS Conditions

Column: Gemini® 3 µm C18 Dimensions: 100 x 3.0 mm Part No.: 00D-4439-Y0

Inline Filter: Phenomenex KrudKatcher™ Ultra (AFO-8497)

**Delay Column:** Luna® 5 μm C18(2) 30 x 2.0 mm

Part No.: 00A-4252-B0

Mobile Phase: A: 20 mM Ammonium acetate in Water

B: Methanol

Gradient: Time (min) % B 10 1.5 65 8 95 8.1 12 0.6 mL/min Flow Rate: Injection: 90 µL 40°C Temperature:

Detector: SCIEX 5500 QTRAP® **Detection:** MS/MS ESI Negative (sMRM) 1. PFBA 11. PFNA

Samples:

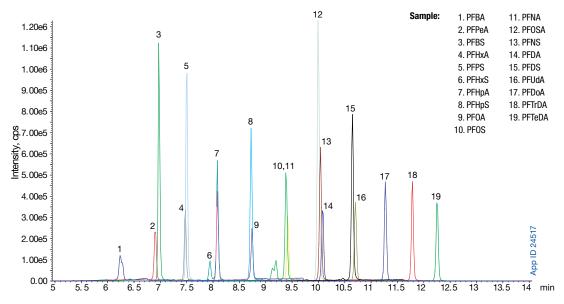
2. PFPeA 12. PFOSA 3. PFBS 13. PFNS 4. PFHxA 14. PFDA 5. PFPS 15. PFDS 6. PFHxS 16. PFUdA 7. PFHpA 17. PFDoA 8. PFHpS 18. PFTrDA 9. PFOA 19. PFTeDA

10. PF0S





#### Sediment Spiked with 1 ng/g



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# Method Performance Data for Sediments Spiked at 1 ng/g of the Target Analytes (n=4)

Compound	Average % Recovery	% RSD
PFBA	91.7	0.76
PFPeA	86.3	6
PFHxA	89.4	1.2
PFHpA	93.1	2.9
PFOA	98.3	1.5
PFNA	93	1.6
PFDA	87.7	4.5
PFUdA	92.3	2.1
PFDoA	92.5	4.1
PFTrDA	88.2	2.1
PFTeDA	87.6	2.1
PFBS	86.3	2.1
PFPeS	96.2	3.2
PFHxS	81.3	5
PFHpS	92.3	2.6
PFOS	92.1	2.6
PFOSA	104.5	6.3
PFNS	89.8	6.8
PFDS	87.3	6.7

#### Acknowledgement

Special thanks to Syljohn Estil and the Sanitation Districts of Los Angeles County - San Jose Water Quality Laboratory for contributing this method.

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- Long column lifetimes

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# **Ordering Info**

#### roQ Extraction Kits

Extraction kits contain fifty easy-pour salt packets and fifty 50 mL stand-alone centrifuge tubes

Description	Unit	Part No.
AOAC 2007.01 Method Extraction Kits		
6.0 g MgSO <sub>4</sub> , 1.5 g NaOAc	50/pk	KS0-8911
EN 15662 Method Extraction Kits		
4.0 g MgSO <sub>4</sub> , 1.0 g NaCl, 1.0 g SCTD, 0.5 g SCDS	50/pk	KS0-8909
Original Non-buffered Method Extraction Kits		
4.0 g MgSO <sub>4</sub> , 1.0 g NaCl	50/pk	KS0-8910
6.0 g MgSO <sub>4</sub> , 1.5 g NaCl	50/pk	KS0-8912

#### roQ dSPE Kits

dSPE kits contain pre-weighed sorbents/salts inside 2 mL or 15 mL centrifuge tubes

Continugo tubos		
Description	Unit	Part No.
2 mL dSPE Kits		
150 mg MgSO <sub>4</sub> , 25 mg PSA, 25 mg C18E	100/pk	KS0-9504
150 mg MgSO <sub>4</sub> , 25 mg PSA, 2.5 mg GCB	100/pk	KS0-9505
150 mg MgSO <sub>4</sub> , 25 mg PSA, 7.5 mg GCB	100/pk	KS0-9506
150 mg MgSO <sub>4</sub> , 25 mg PSA	100/pk	KS0-9503
150 mg MgSO <sub>4</sub> , 50 mg PSA, 50 mg C18E, 50 mg GCB	100/pk	KS0-9514
150 mg MgSO <sub>4</sub> , 50 mg PSA, 50 mg C18E	100/pk	KS0-9512
150 mg MgSO <sub>4</sub> , 50 mg PSA, 50 mg GCB	100/pk	KS0-9513
150 mg MgSO <sub>4</sub> , 50 mg PSA	100/pk	KS0-9511
15 mL dSPE Kits		
900 mg MgSO <sub>4</sub> , 150 mg PSA, 150 mg C18E	100/pk	KS0-9508
900 mg MgSO <sub>4</sub> , 150 mg PSA, 15 mg GCB	100/pk	KS0-9509
900 mg MgSO <sub>4</sub> , 150 mg PSA, 45 mg GCB	100/pk	KS0-9510
900 mg MgSO <sub>4</sub> , 150 mg PSA	100/pk	KS0-9507
1200 mg MgSO <sub>4</sub> , 400 mg PSA, 400 mg C18E, 400 mg GCB	100/pk	KS0-9518
1200 mg MgSO <sub>4</sub> , 400 mg PSA, 400 mg C18E	100/pk	KS0-9516
1200 mg MgSO <sub>4</sub> , 400 mg PSA, 400 mg GCB	100/pk	KS0-9517
1200 mg MgSO <sub>4</sub> , 400 mg PSA	100/pk	KS0-9515

#### **Bulk roQ QuEChERS Sorbents**

Phase	10 g	100 g
C18-E	_	04G-4348
GCB (Graphitized Carbon Black)	04D-4615	04G-4615
PSA	_	04G-4610

#### roQ Extraction Salt Packets

Salt packets only. Centrifuge tubes not included.

Description	Unit	Part No.
AOAC 2007.01 Method Extraction Packets		
6.0 g MgSO <sub>4</sub> , 1.5 g NaOAc	50/pk	KS0-9043
EN 15662 Method Extraction Packets		
4.0 g MgSO <sub>4</sub> , 1.0 g NaCl, 1.0 g SCTD, 0.5 g SCDS	50/pk	KS0-9041
Original Non-Buffered Method Extraction Packets		
4.0 g MgSO <sub>4</sub> , 1.0 g NaCl	50/pk	KS0-9042
6.0 g MgSO <sub>4</sub> , 1.5 g NaCl	50/pk	KS0-9044

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