RESTEK
Chromatography Products

Fast Screening of Recalled Tylenol® for Tribromoanisole and Related Adulterants

Using QuEChERS and GC-TOFMS

- Rapid sample preparation with QuEChERS improves turnaround time for emergency response analysis situations.
- Prepackaged QuEChERS extraction salts and snap-and-shoot standards reduce human error and save time.
- Rugged, inert, thermally stable Rxi®-5Sil MS column extends applicability to acids, bases, and higher molecular weight adulterants.

Introduction

The recent recall of Tylenol® pain reliever and other related products highlights the need for simple, quick sample preparation and a comprehensive analytical method for adulterants in consumer products. The rush to examine a multitude of samples in a short period of time is a common scenario for potential recalls, especially when a contaminant is found in a given product and rapid determinations need to be made to assess how widespread the problem may be.

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) sample preparation approach, originally developed to prepare fruit and vegetable samples for pesticide residue analysis, is being adopted for other applications and may be useful when rapid screening methods are required. QuEChERS employs a simple solvent shake and centrifugation step, with an optional dispersive solid phase extraction (dSPE) cleanup. In addition to being quick and easy, the use of acetonitrile in QuEChERS allows compounds containing a wide variety of chemical functionalities to be extracted, which is very important when trying to isolate an unknown adulterant. The resulting extract is appropriate for both GC/MS and LC/MS work.

The utility of the QuEChERS method is illustrated here using the aforementioned Tylenol® example, showing the applicability to this problem and, by extension, to others like it. This particular recall was due to the presence of 2,4,6-tribromoanisole (TBA) causing a musty smell in the product and, in some cases, nausea in the consumer [1]. TBA is a known breakdown product of 2,4,6-tribromophenol (TBP), which is a fumigant used on shipping pallets; TBA production occurs through a process actuated by a fungus, *Paecilomyces variotii* [2]. TBA is a common and undesirable odorant in the winemaking industry where it and similar compounds (e.g. trichloroanisole) create a situation known as cork taint [3].

This work demonstrates the potential applicability of QuEChERS sample preparation and GC-TOFMS analysis to screening methods for anisole contaminants. Advantages of methods developed based on QuEChERS and GC-TOFMS may include rapid sample screening and definitive identifications in the presence of significant amounts of matrix.

Procedure

TBA, TBP, 2,3,4,5-tetrachloroanisole, and pentachloroanisole were spiked into ground up Tylenol® caplets at two different concentrations and extracted using QuEChERS. Several cleanup procedures were performed for comparison and GC analysis was conducted using a sensitive, full mass-range time-of-flight MS.

Sample Wetting and Fortification

A bottle of recalled Tylenol® Extra Strength caplets was used for this work, although no odor of TBA was detected. Multiple caplets were ground to a fine powder using a Bamix® Mono Hand Mixer with dry grinder attachment. 1.2 g of powder, equivalent to 2 caplets (500 mg acetaminophen each) was wetted with 9 mL organic-free water for each sample for extraction. After shaking to mix well, wetted powders were fortified as follows; note that spike levels are expressed relative to approximated amount of active ingredient, not formulated product.

- Unspiked Tylenol® 100 µL of QuEChERS Internal Standard Mix for GC/MS Analysis (cat.# 33267)containing PCBs 18, 28, and 52 (50 µg/mL each); triphenylphosphate (20 µg/mL); tris-(1,3-dichloroisopropyl)phosphate (50 µg/mL); and triphenylmethane (10 µg/mL).
- ~1,000 ng/g spiked Tylenol® (2 samples) 5 µL of Custom Anisoles Standard #1 (cat.# 564667) containing 2,4,6-tribromoanisole, 2,3,4,5-tetrachloroanisole, and pentachloroanisole at 200 µg/mL each in methanol. 5 µL of Acid Surrogate Mix (cat.# 31025) containing 2,4,6-tribromophenol, 2-fluorophenol, and phenol-d6, diluted to 200 µg/mL in methanol. 100 µL of QuEChERS Internal Standard Mix for GC/MS Analysis.
- ~100 ng/g spiked Tylenol® 5 µL of Custom Anisoles Standard #1; 5 µL of Acid Surrogate Mix diluted to 20 µg/mL; 100 µL of QuEChERS Internal Standard Mix for GC/MS Analysis.

After fortification, each sample was allowed to soak for 1 hour prior to QuEChERS extraction. Originally the QuEChERS method was developed for high aqueous content fruits and vegetables. Here we used a reduced amount of material and sample wetting in order to increase extraction efficiency for a dry powder.

QuEChERS Extraction

The EN 15662 QuEChERS method was used for sample extraction [4]. 10 mL of acetonitrile was added to a wet sample. After a 1 minute shake, Q-sep™ Q110 buffering extraction salts (cat.# 26213, 4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate dihydrate, 0.5 g disodium hydrogen citrate sesquihydrate) were added. Following another 1 minute shake, the sample was centrifuged for 5 minutes at 3,000 U/min. with a Q-sep™ 3000 centrifuge (cat.# 26230).

Extract Cleanup

Four dispersive solid phase extraction methods (dSPE) were compared. For each, 1 mL portions of QuEChERS extracts were added to tubes containing drying agent and different sorbents such as primary secondary amine (PSA), C18, and graphitized carbon black (GCB) as shown below. The tubes were shaken for 2 minutes and then centrifuged for 5 minutes in the Q-sep™ 3000 centrifuge. The resulting final extracts were then analyzed with GC-TOFMS.

- Q210 (cat.# 26215): 150 mg $MgSO_4$, 25 mg PSA
- Q251 (cat.# 26125): 150 mg $MgSO_4$, 50 mg PSA, 50 mg C18
- Q252 (cat.# 26219): 150 mg $MgSO_4$, 50 mg PSA, 50 mg C18, 50 mg GCB
- Custom dSPE tube: 150 mg $MgSO_4$, 50 mg PSA, 50 mg C18, 7.5 mg GCB

GC-TOFMS

A LECO Pegasus® III GC-TOFMS instrument was used and all data were processed with LECO ChromaTOF® software. Gas chromatography was performed using an Rxi®-5Sil MS column (30 m x 0.25 mm x 0.25 µm, cat.# 13623) with a constant flow of helium at 1.2 mL/min. (40 cm/sec. at 90°C). 1 µL fast autosampler splitless injections were made into a 4mm single gooseneck liner with wool (cat.# 22405) at 250°C. The purge valve time was 60 seconds.

The GC oven program was 90 °C (1 minute), 4 °C/min. to 310 °C (2 minutes). Total run time was 58 minutes.

Electron ionization at 70 eV was used with a source temperature of 225°C. Data acquisition was from 45 to 550 amu at a rate of 5 spectra/sec.

Calibration and Quantification with Matrix-Matched Standards

Matrix-matched standards were prepared at 100 pg/µL and 10 pg/µL, as these are the expected final concentrations in extracts for Tylenol® spikes (assuming 100% recoveries for the 1,000 and 100 ng/g spikes, respectively). Matrix-matched standards were prepared by adding standard solution to the final extract from a control sample, which had no measurable amounts of the compounds of interest. Actual recoveries were calculated after quantification from one-point calibration in ChromaTOF®. The internal standard method of quantification was employed using PCB 52.

Results

The concentrations used for spikes in this case were 1,000 and 100 ng/g relative to active ingredient in the starting caplet material (estimated using labeled value). Using QuEChERS combined with GC-TOFMS, modest recoveries of all compounds were realized as can be seen in Table I. In addition, results for duplicate extracts and cleanups for 1,000 ng/g spikes, using either Q210 dSPE tubes or the custom dSPE tubes, were relatively close for each analyte. Although the spiked concentrations are higher than the odor threshold expected for an end product such as Tylenol® (TBA's odor threshold is extremely low, 0.008-0.03 ppt in water and 2-6 ppt in wine [5]), the QuEChERS approach with GC-TOFMS provides a useful technique for screening of contamination at potential levels of health concern, moderate adulteration, and for analyzing source materials such as wood pallets, for contaminants. QuEChERS can produce extracts for up to 24 samples, ready for GC or LC analysis, in less than 60 minutes, a speed conducive to the pressure of responding to a consumer product adulteration issue. In addition, the multi-compound extraction capability of the QuEChERS acetonitrile solvent offers a better chance of isolating potential adulterants from any matrix.

Table I Percent recoveries of potential adulterants from QuEChERS extractions: comparison of various dSPE cleanup procedures. (All samples are 1,000 ng/g, unless otherwise noted.)

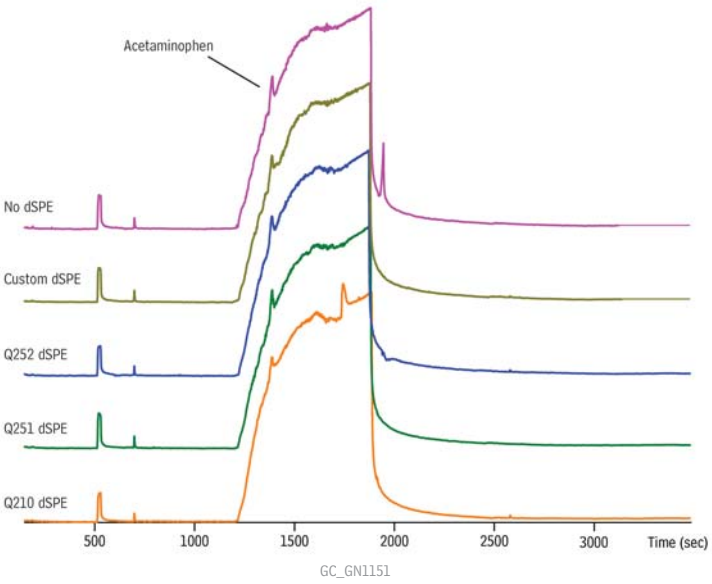
		Q210		Q251		Q252		CustomdSPE	Extract
Compound	RT (sec.)	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	1,000 ng/g	100 ng/g
2,4,6-Tribromoanisole	1097.82	82	56	62	68	73	68	59	51
2,4,6-Tribromophenol	1133.62	55	60	40	49	66	53	63	110
2,3,4,5-Tetrachloroanisole	1162.22	71	63	64	64	75	63	67	70
Pentachloroanisole	1256.82	70	67	64	70	71	61	65	60
PCB 52 (IS)	1611.02								

Q210 = 150 mg $MgSO_4$, 25 mg PSA
Q251 = 150 mg $MgSO_4$, 50 mg PSA, 50 mg C18
Q252 = 150 mg $MgSO_4$, 50 mg PSA, 50 mg C18, 50 mg GCB
Custom = 150 mg $MgSO_4$, 50 mg PSA, 50 mg C18, 7.5 mg GCB
Extract = extraction only, no clean up step was performed

The original QuEChERS approach for fruits and vegetables was developed with a novel dSPE cleanup procedure where an extract is shaken with loose sorbent material (e.g. primary secondary amine, C18, graphitized carbon black) to remove matrix coextractives like fatty acids, lipids, and pigments, that might interfere with targeted residues during instrumental analysis. Although we tried dSPE here, it is less appropriate in this application for two reasons: (1) In a true unknown adulterant situation, sorbents, especially PSA and GCB, might actually remove the adulterant from the extract, in addition to matrix interferences, leaving the adulterant undetected during instrumental analysis. (2) The gross amount of acetaminophen in the extract greatly exceeds the capacity of the dSPE sorbent, which is typically on the order of the 25-50 mg per mL extract.

Due to the overwhelming concentration of acetaminophen in the caplet powder extracts, dSPE cleanup was largely ineffective (Figure 1), but as the acetaminophen was volatile enough to chromatograph, it was not critical to remove it to prevent deposition in the injector and column. Elimination of the dSPE step did not noticeably improve, or degrade, the recovery results for TBA and TBP, or other components (Table I).

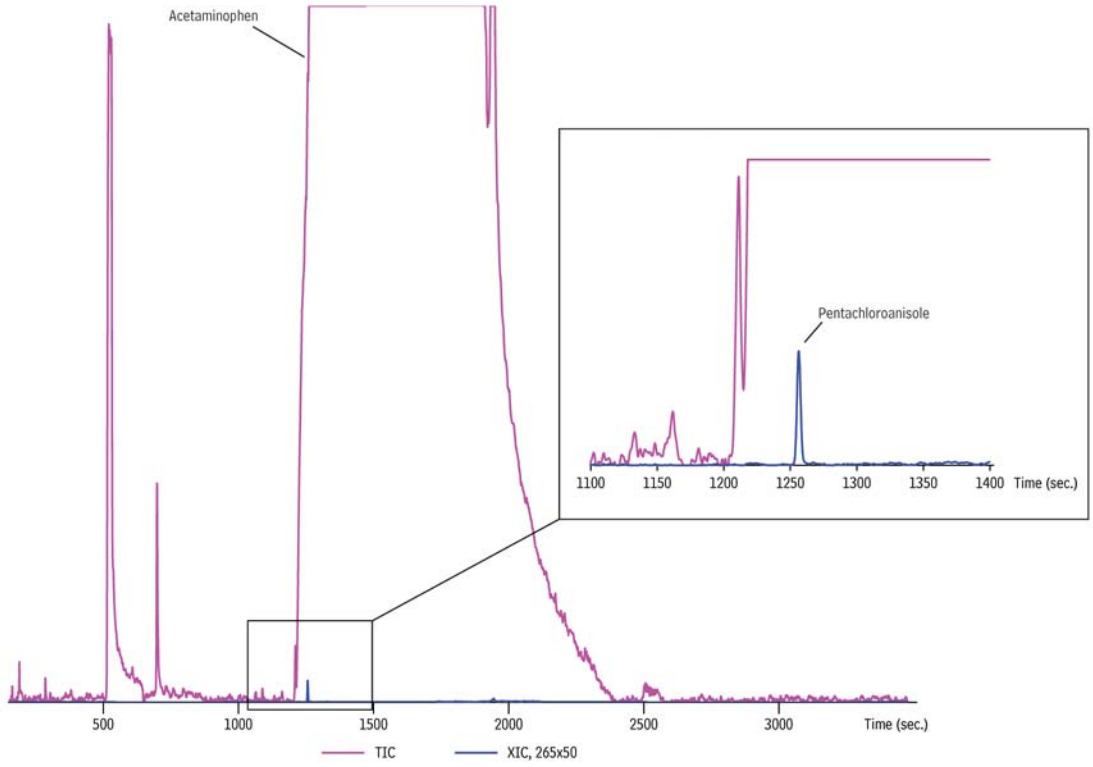
Figure 1 Chromatograms of QuEChERS extracts of Tylenol® with different dSPE cleanups.



Column	Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623)
Sample	QuEChERS extracts of Tylenol® spiked with TBA and anisoles
Diluent:	Acetonitrile
Injection	
Inj. Vol.:	1 µL splitless (hold 1 min.)
Liner:	Gooseneck Splitless (4mm) with Wool (cat.# 22405)
Inj. Temp.:	250 °C
Purge Flow:	40 mL/min.
Oven	
Oven Temp:	90 °C (hold 1 min.) to 310 °C at 4 °C/min. (hold 2 min.)
Carrier Gas	He, constant flow
Flow Rate:	1.2 mL/min.
Linear Velocity:	40 cm/sec.
Detector	TOFMS
Transfer Line Temp.:	290 °C
Analyzer Type:	TOF
Source Temp.:	225 °C
Electron Energy:	70 eV
Mass Defect:	-20 mu/100 u
Solvent Delay Time:	2 min.
Tune Type:	PFTBA
Ionization Mode:	EI
Acquisition Range:	45 to 550 amu
Spectral Acquisition Rate:	5 spectra/sec
Instrument Notes	LECO Pegasus 4D GCxGC-TOFMS Q210 = PSA Q251 = PSA and C18 Q252 = PSA, C18, and GCB Custom = PSA, C18, and less GCB

One reason to employ dSPE, or another cleanup step, is to remove matrix interferences that can prevent detection of potential adulterants. However, we relied on automated peak find and spectral deconvolution to detect analytes of interest among the overwhelming acetaminophen response. This is particularly evident for pentachloroanisole in the 1,000 ppb spike extract, which eluted well underneath the large acetaminophen peak (Figure 2). The disparity in concentrations is so large that the 265 m/z ion was only visible by magnifying it by 50, yet ChromaTOF® automatically located the peak and produced a deconvoluted spectrum that matched very well with the pentachloroanisole reference spectrum (Figure 3). Although this part of the application was a targeted analysis of TBA, TBP, and other anisoles, to help evaluate QuEChERS extract recoveries for these compounds in a difficult matrix, the peak find and spectral deconvolution algorithms employed here are very useful when looking for unknown contaminants. Pure sample mass spectra lead to better library searching and identification of components.

Figure 2 Pentachloroanisole was located under the large acetaminophen peak using an automated peak find routine. (1,000 ng/g; QuEChERS extraction only, no dSPE).



Column	Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623)
Sample	QuEChERS extracts of Tylenol® spiked with TBA and anisoles
Diluent:	Acetonitrile
Injection	
Inj. Vol.:	1 µL splitless (hold 1 min.)
Liner:	Gooseneck Splitless (4mm) with Wool (cat.# 22405)
Inj. Temp.:	250 °C
Purge Flow:	40 mL/min.
Oven	
Oven Temp:	90 °C (hold 1 min.) to 310 °C at 4 °C/min. (hold 2 min.)
Carrier Gas	He, constant flow
Flow Rate:	1.2 mL/min.
Linear Velocity:	40 cm/sec.

Detector	TOFMS
Transfer Line Temp.:	290 °C
Analyzer Type:	TOF
Source Temp.:	225 °C
Electron Energy:	70 eV
Mass Defect:	-20 mu/100 u
Solvent Delay Time:	2 min.
Tune Type:	PFTBA
Ionization Mode:	EI
Acquisition Range:	45 to 550 amu
Spectral Acquisition Rate:	5 spectra/sec
Instrument	LECO Pegasus 4D GCxGC-TOFMS

Conclusions

Shown here is a QuEChERS multi-compound extraction method that rapidly produces samples for GC or LC analysis in consumer product adulteration cases. QuEChERS is simple, efficient, and uses little solvent compared to other extraction methods. QuEChERS and GC with a sensitive, full mass-range TOFMS is a powerful approach to identifying potential adulterants in consumer products.

References

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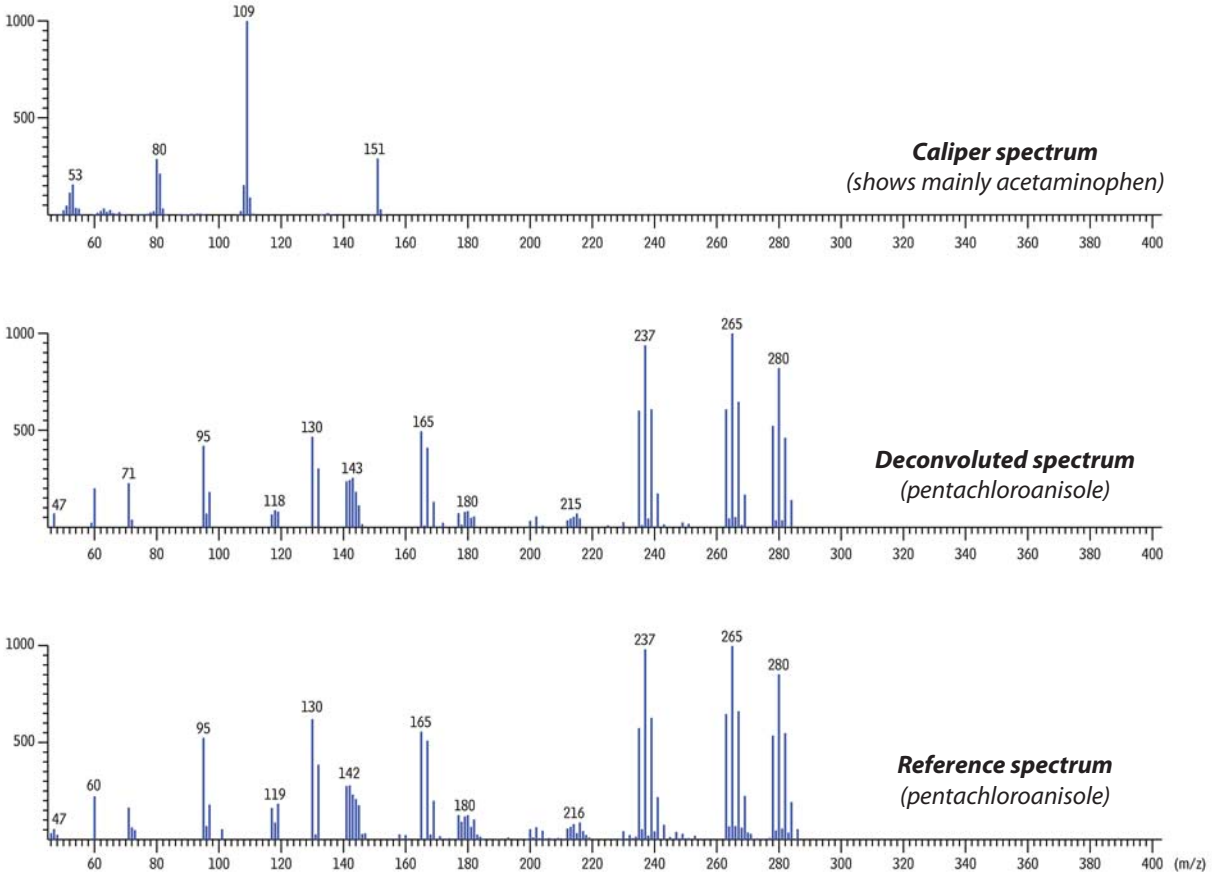
2. R. Tracy, B. Skaalen, Practical Winery and Vineyard (2008) http://www.practicalwinery.com/novdec08/page2.htm (accessed April 19, 2010).
3. F.B. Whitfield, J.L. Hill, K.J. Shaw, J. Agric. Food Chem. 45 (1997) 889.
4. Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and Clean-up by Dispersive SPE (QuEChERS-method). (EN 15662 Version 2008.).
5. P. Chatonnet, S. Bonnet, S. Boutou, J. Agric. Food Chem. 52 (2004) 1255.

Figure 3 The caliper spectrum taken at the peak apex of pentachloroanisole is representative of the overwhelming acetaminophen peak, but TOFMS allows spectral deconvolution to produce a sample spectrum that matches well with the reference spectrum.



GC_GN1153

Column	Rxi®-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623)
Sample	QuEChERS extracts of Tylenol® spiked with TBA and anisoles
Diluent:	Acetonitrile
Injection	
Inj. Vol.:	1 µL splitless (hold 1 min.)
Liner:	Gooseneck Splitless (4mm) with Wool (cat.# 22405)
Inj. Temp.:	250 °C
Purge Flow:	40 mL/min.
Oven	
Oven Temp:	90 °C (hold 1 min.) to 310 °C at 4 °C/min. (hold 2 min.)
Carrier Gas	He, constant flow
Flow Rate:	1.2 mL/min.
Linear Velocity:	40 cm/sec.

Detector	TOFMS
Transfer Line Temp.:	290 °C
Analyzer Type:	TOF
Source Temp.:	225 °C
Electron Energy:	70 eV
Mass Defect:	-20 mu/100 u
Solvent Delay Time:	2 min.
Tune Type:	PFTBA
Ionization Mode:	EI
Acquisition Range:	45 to 550 amu
Spectral Acquisition Rate:	5
Instrument	LECO Pegasus 4D GCxGC-TOFMS

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Rxi®-5Sil MS Columns (fused silica)

(low polarity Crossbond® silarylene phase; selectivity close to 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	qty.	cat. #
0.10mm	0.10µm	-60 to 330/350°C	10m	ea.	43601
0.18mm	0.18µm	-60 to 330/350°C	20m	ea.	43602
0.18mm	0.36µm	-60 to 330/350°C	20m	ea.	43604
0.25mm	0.10µm	-60 to 330/350°C	15m	ea.	13605
0.25mm	0.10µm	-60 to 330/350°C	30m	ea.	13608
0.25mm	0.25µm	-60 to 330/350°C	15m	ea.	13620
0.25mm	0.25µm	-60 to 330/350°C	30m	ea.	13623
0.25mm	0.25µm	-60 to 330/350°C	30m	6-pk.	13623-600
0.25mm	0.25µm	-60 to 330/350°C	60m	ea.	13626
0.25mm	0.50µm	-60 to 330/350°C	15m	ea.	13635
0.25mm	0.50µm	-60 to 330/350°C	30m	ea.	13638
0.25mm	1.00µm	-60 to 325/350°C	15m	ea.	13650
0.25mm	1.00µm	-60 to 325/350°C	30m	ea.	13653
0.25mm	1.00µm	-60 to 330/350°C	60m	ea.	13697
0.32mm	0.25µm	-60 to 330/350°C	15m	ea.	13621
0.32mm	0.25µm	-60 to 330/350°C	30m	ea.	13624
0.32mm	0.50µm	-60 to 330/350°C	30m	ea.	13639
0.32mm	1.00µm	-60 to 325/350°C	30m	ea.	13654
0.53mm	1.50µm	-60 to 310/330°C	30m	ea.	13670

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- Inertness verified by isothermal testing.

Description	qty.	cat.#
15m, 0.25mm ID, 0.25µmµm Rxi-5Sil MS	ea.	13620-127
30m, 0.25mm ID, 0.25µmµm Rxi-5Sil MS	ea.	13623-124
30m, 0.25mm ID, 0.25µmµm Rxi-5Sil MS	ea.	13623-127
15m, 0.25mm ID, 0.50µmµm Rxi-5Sil MS	ea.	13635-124
30m, 0.25mm ID, 0.50µmµm Rxi-5Sil MS	ea.	13638-124
30m, 0.25mm ID, 0.50µmµm Rxi-5Sil MS	ea.	13638-127
30m, 0.32mm ID, 0.50µmµm Rxi-5Sil MS	ea.	13639-125
30m, 0.32mm ID, 1.00µmµm Rxi-5Sil MS	ea.	13654-125

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- Small footprint requires less bench space.
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Priced to fit your laboratory's budget, the Q-sep™ 3000 Centrifuge is the first centrifuge specifically designed for QuEChERS methodology. This compact, quiet, yet powerful, unit spins at the 3,000 g-force required by the European method.

Centrifuge includes 50 mL tube carriers (6), 50 mL conical tube inserts (6), 4-place 15 mL tube carriers (6), and 2 mL tube adaptors (24).

Description	qty.	cat.#
Q-sep 3000 Centrifuge, 110V	ea.	26230
Q-sep 3000 Centrifuge, 220V	ea.	26231
Replacement Accessories		
50mL Tube Carrier for Q-sep 3000 Centrifuge	2-pk.	26232
50mL Conical Tube Insert for Q-sep 3000 Centrifuge	6-pk.	26249
4-Place Tube Carrier for Q-sep 3000 Centrifuge	2-pk.	26233
2mL Tube Adaptors for Q-sep 3000 Centrifuge	4-pk.	26234

Q-sep™ QuEChERS Products (cont.)

Q-sep™ QuEChERS Tubes

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- Fast, simple sample extraction and cleanup using dSPE.
- Fourfold increases in sample throughput.
- Fourfold decreases in material cost.
- Convenient, ready to use centrifuge tubes with ultra pure, preweighed adsorbent mixes.

Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS ("catchers") method, developed by the USDA Eastern Regional Research Center[1], has become very popular for extraction and cleanup of pesticide residue samples. Our products are available in three centrifuge tube sizes to meet the needs of both extraction and cleanup of a wide variety of sample matrices following various methods.

The researchers developed a simple two-step procedure. First, the homogenized samples are extracted and partitioned, using an organic solvent and salt solution. Then, the supernatant is further extracted and cleaned, using a dispersive SPE technique. Multiple adsorbents are placed in a centrifuge tube, along with the 1 mL of organic solvent and the extracted residues partitioned from step 1. The contents are thoroughly mixed, then centrifuged, producing a clean extract ready for a variety of GC or HPLC analytical techniques.[2] Validation and proficiency data for the QuEChERS method are available for a wide variety of pesticides in several common food matrices at www.quechers.com

Description	Material	Methods	qty.	cat#
Q110 Kit	4g $MgSO_4$, 1g NaCl, 1g TSCD, 0.5g DHS with 50mL Centrifuge Tube	European EN 15662	50 packets & 50 tubes	26235
Q110 Packets	4g $MgSO_4$, 1g NaCl, 1g TSCD, 0.5g DHS	European EN 15662	50 packets	26236
Q150 Kit	6g $MgSO_4$, 1.5g NaOAc with 50mL Centrifuge Tube	AOAC 2007.01	50 packets & 50 tubes	26237
Q150 Packets	6g $MgSO_4$, 1.5g NaOAc	AOAC 2007.01	50 packets	26238
Empty 50mL Centrifuge Tube			50-pk.	26239
2mL Micro-Centrifuge Tubes for dSPE (clean-up of 1mL extract)				
Q210	150mg $MgSO_4$, 25mg PSA	European EN 15662	100-pk.	26215
Q211	150mg $MgSO_4$, 25mg PSA, 25mg C18		100-pk.	26216
Q212	150mg $MgSO_4$, 25mg PSA, 2.5mg GCB	European EN 15662	100-pk.	26217
Q213	150mg $MgSO_4$, 25mg PSA, 7.5mg GCB	European EN 15662	100-pk.	26218
Q250	150mg $MgSO_4$, 50mg PSA	AOAC 2007.1	100-pk.	26124
Q251	150mg $MgSO_4$, 50mg PSA, 50mg C18	AOAC 2007.1	100-pk.	26125
Q253	150mg $MgSO_4$, 50mg PSA, 50mg GCB		100-pk.	26123
Q252	150mg $MgSO_4$, 50mg PSA, 50mg C18, 50mg GCB	AOAC 2007.1	100-pk.	26219
15mL Centrifuge Tubes for dSPE (clean-up of 6mL extract)				
Q350	1200mg $MgSO_4$, 400mg PSA	AOAC 2007.1	50-pk.	26220
Q351	1200mg $MgSO_4$, 400mg PSA, 400mg C18	AOAC 2007.1	50-pk.	26221
Q352	1200mg $MgSO_4$, 400mg PSA, 400mg C18, 400mg GCB	AOAC 2007.1	50-pk.	26222
Q370	900mg $MgSO_4$, 150mg PSA	European EN 15662	50-pk.	26223
Q371	900mg $MgSO_4$, 150mg PSA, 15mg GCB	European EN 15662	50-pk.	26224
Q372	900mg $MgSO_4$, 150mg PSA, 45mg GCB	European EN 15662	50-pk.	26225
Q373	900mg $MgSO_4$, 150mg PSA, 150mg C18		50-pk.	26226
Q374	900mg $MgSO_4$, 300mg PSA, 150mg GCB		50-pk.	26126

PSA—primary and secondary amine exchange material
GCB—graphitized carbon black

Sorbent Guide

Sorbent	Removes
PSA*	sugars, fatty acids, organic acids, anthocyanine pigments
C18	lipids, nonpolar interferences
GCB**	pigments, sterols, nonpolar interferences

*PSA—primary and secondary amine exchange material
**GCB—graphitized carbon black

Free sample packs of SPE cartridges are available by adding -248 to the catalog number when you call to place your order.

References (not available from Restek)

1. Anastassiades, M., S.J. Lehotay, D. Stajnbaher, F.J. Schenck, Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce, J AOAC International, 2003, vol 86 no 22, pp 412-431.
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Reference Standards

QuEChERS Standards

- Ready to use for QuEChERS extractions—no dilutions necessary.
- Support for GC and HPLC with MS, MS/MS, and selective detectors.

Pesticide analysis is fast and simple using QuEChERS methods. Use these cost-effective QuEChERS standards for even greater lab efficiency. Standards are compatible with all major methods, including mini-multiresidue, AOAC, and European procedures. Save time with convenient mixes or make your own blend using our full line of single component solutions.

QuEChERS Quality Control Standards for GC/MS Analysis

Cat.# 33268:	Cat.# 33264:
PCB 138	anthracene
PCB 153	

50µg/mL each in acetonitrile, 5mL/ampul
cat. # 33268 (ea.)

100µg/mL in acetonitrile, 5mL/ampul
cat. # 33264 (ea.)

QuEChERS Internal Standard Mix for GC/MS Analysis (6 components)

PCB 18	50µg/mL
PCB 28	50
PCB 52	50
triphenyl phosphate	20
tris-(1,3-dichloroisopropyl)phosphate	50
triphenylmethane	10

In acetonitrile, 5mL/ampul
cat. # 33267 (ea.)

QuEChERS Single-Component Reference Standards

Concentration is µg/mL. ACN=acetonitrile

Compound	Solvent	Conc.	cat.# (ea.)
PCB 18 (5mL)	ACN	50	33255
PCB 28 (5mL)	ACN	50	33256
PCB 52 (5mL)	ACN	50	33257
PCB 138 (5mL)	ACN	50	33262
PCB 153 (5mL)	ACN	50	33263
triphenylmethane (5mL)	ACN	10	33260
triphenylphosphate (5mL)	ACN	20	33258
tris(1,3-dichloroisopropyl) phosphate (5mL)	ACN	50	33259

Acid Surrogate Mix (4/89 SOW) (3 components)

- Highest concentrations commercially available.
- Convenient 1mL, 5mL, and 10mL package sizes.
- Reduces laboratory cost per sample extract.

2-fluorophenol
phenol-d6
2,4,6-tribromophenol

Each	15-pk.	25-pk.
2,000µg/mL each in methanol, 1mL/ampul		
31025	31025.15	31025.25
10,000µg/mL each in methanol, 1mL/ampul		
31063	31063.15	31063.25
10,000µg/mL each in methanol, 5mL/ampul		
31087	31087.15	31087.25
10,000µg/mL each in methanol, 10mL/ampul		
33029	33029.15	33029.25

Lit. Cat.# GNAN1263

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