

# QuEChERS Methodology: Mini-Multiresidue Method

## Q-sep™ Q110, cat.# 26213

**Quick, Easy, Cheap, Effective, Rugged, and Safe**, the QuEChERS (“catchers”) method is based on work done and published by the US Department of Agriculture Eastern Regional Research Center in Wyndmoor, PA.<sup>1</sup> Researchers developed a new extraction method for pesticides in fruits and vegetables, coupled with a clean-up method that removes sugars, lipids, organic acids, sterols, proteins, pigments and excess water. This technique offers a user-friendly alternative to traditional liquid-liquid and solid phase extractions.

The process involves two simple steps. 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 solid phase extraction (dSPE) technique**.

Restek products make this approach even simpler. We offer QuEChERS extraction and dSPE products in a variety of standard sizes and formats. The dSPE centrifuge tube format (available in 2mL and 15mL sizes) contains magnesium sulfate (to partition water from organic solvent) and primary secondary amine (PSA) adsorbent (to remove sugars and fatty acids). These tubes are available with or without graphitized carbon (to remove pigments and sterols) and/or C18 packing (to remove nonpolar interferences such as lipids).

Several detailed QuEChERS methods have been published and are listed below. Restek dSPE tubes, listed in Table I (back page), are formulated according to these methods.

- **Mini-Multiresidue Method**

QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products<sup>2</sup>

- **AOAC Official 2007.01 Method**

Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate<sup>3</sup>

- **European EN 15662 Method**

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<sup>4</sup>

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QuEChERS-based methods have several basic steps in common, which are described below. Specific procedures for sample extraction and dSPE sample clean-up according to *QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*<sup>2</sup> are given in the following sections.

### General Procedures (common to all 3 QuEChERS-based methods listed above)

#### Step 1: Sample preparation and extraction

Commodities are uniformly ground. Internal standards are also added at this point. Various salts, acids and buffers may then be added to enhance extraction efficiency and protect sensitive analytes.

#### Step 2: Sample extract cleanup

A subsample of the modified solvent extract from Step 1 is cleaned up using dSPE. Small polypropylene centrifuge tubes are pre-filled with precise weights and proportions of bulk drying salts and SPE adsorbent packings to remove excess water and unwanted contaminants from the sample extracts. After a brief agitation and centrifugation, the cleaned extracts are then prepared for analysis.

#### Step 3: Sample analysis

Samples may be pH adjusted, solvent-exchanged, or treated with additional agents, to protect sensitive analytes or improve analysis by either GC/MS or LC/MS.

## Multiresidue QuEChERS 2007 Procedure

The procedures below are based on *QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*<sup>2</sup>. For complete information, refer to the original source method at [www.quechers.de](http://www.quechers.de) or [www.quechers.com](http://www.quechers.com).

### Sample Extraction

1. Homogenize the frozen commodity to generate a uniform sample representative of the product (Figure 1). For products with more than 5% fat (w/w), see the specialized sample preparation procedure in the source method, *QuEChERS-A Mini Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products*.<sup>2</sup>
2. Weigh 10g of homogenized product into a clean 50mL tube (cat.#26227) as shown in Figure 2.
3. Add 10mL acetonitrile and an appropriate amount of an internal standard solution. See suggestions of internal standards and volume in the source method.<sup>2</sup>
4. Shake vigorously for 1 minute by hand (Figure 3).



### Sample Drying and Buffering

1. Add the contents (listed below) of a Q-sep™ Q110 tube (cat.# 26213) to each extracted sample (Figure 4).

4.0g ± 0.2g	magnesium sulfate, anhydrous
1.0g ± 0.05	sodium chloride
1.0g ± 0.05g	trisodium citrate dihydrate
0.5g ± 0.03g	disodium hydrogencitrate sesquihydrate

#### Note:

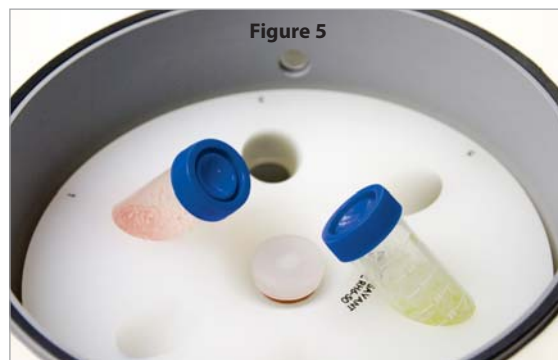
If pH <3, sample should be adjusted with addition of 600µL of 5N NaOH (lemons, limes, currants).  
If pH >3 and <5, sample should be adjusted with 200µL of 5N NaOH.

2. Shake immediately and vigorously 1 minute (Figure 3).



### Phase Separation

Centrifuge for 5 minutes at 3,000 U/min. to separate the solid material (Figure 5). Proceed with dSPE sample clean-up or analyze extract directly without clean-up.



## dSPE Sample Clean-up

The sample can be analyzed directly from the raw extract, especially if pesticides with acidic groups (e.g. phenoxyacid herbicides) are of interest. Alternatively, sample clean-up methods can be applied. Specifically, dispersive solid phase extraction is discussed here. Restek dSPE tubes are formulated in accordance with published methods and are listed in Table 1. Select tubes based on the method and sample type; general guidelines for different sample types include:

### For samples with co-extracted fats or waxes:

Before or after clean-up, samples are put in freezer (>1hr. to overnight). Cold samples are then re-centrifuged and fats or waxes are removed. If fat remains, clean up with 25mg PSA, 150mg MgSO<sub>4</sub>, and 25mg C18 **per mL** of extract. If no fat remains, clean up with 25mg PSA and 150mg MgSO<sub>4</sub> **per mL** of extract.

### For samples with remaining fats:

Clean up with 25mg PSA, 150mg MgSO<sub>4</sub>, and 25mg C18 **per mL** of extract (see above).

### For samples with intensely colored extracts:

Clean up with 25mg PSA, 150mg MgSO<sub>4</sub>, and 7.5mg graphitized carbon **per mL** of extract.

### For samples with less intensely colored extracts, or high carotinoid or chlorophyll levels:

Clean up with 25mg PSA, 150mg MgSO<sub>4</sub>, and 2.5mg graphitized carbon **per mL** of extract.

### For all other samples:

Clean up with 25mg PSA and 150mg MgSO<sub>4</sub> **per mL** of extract. Once tubes are selected, dSPE sample clean-up can be accomplished according to the procedure shown below.



1. Using the centrifuged extracts resulting from the **phase separation** stage of sample extract preparation, transfer the supernatant to the dSPE tube as shown in Figure 6. Use Table I to determine the volume of sample that should be transferred.
2. Shake vigorously for 30 seconds or 2 minutes (Figure 7). Use Table I to determine the suggested shake time.
3. Centrifuge for 5 minutes at 3,000U/min. to separate the solid material (Figure 8).
4. Immediately adjust the supernatant pH using a 5% formic acid in acetonitrile solution. Use 10µL **per mL** of supernatant. For sulfonyl urea herbicides, carbosulfan, and benfuracarb, analyze the supernatant without any pH adjustment.
5. Transfer sample to an autosampler vial and test using GC or LC methods (Figure 9).

**Note:** To determine the amount (mg) of PSA, MgSO<sub>4</sub>, and graphitized carbon, use the suggested number of milligrams and multiply by the number of mLs you want to extract.

## References

1. M. Anastassiades, S.J. Lehotay, D. Stajnbaher, F.J. Schenck, J. AOAC International 86, p. 412-431 (2003).
2. QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products. <http://www.quechers.com> (accessed July 15, 2008).
3. AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.
4. EN 15662, Foods of Plant Origin—Determination of Pesticide Residues Using GC-MS and/or LC-MS/MS Following Acetonitrile Extraction/Partitioning and by Dispersive SPE—QuEChERS method.

Clean-up

**Table I** Restek Q-sep™ dSPE tubes (organized by published method).

Cat.#	Name	Centrifuge Tube		Method	Sample Volume (mL)	Shake Time (min.)	Centrifuge Speed	Centrifuge Time (min.)
		Size (mL)	Contains					
26216	Q-sep Q211	2	150mg MgSO <sub>4</sub> , 25mg PSA, 25mg C18	Mini-Multiresidue	1	0.5	3,000 U/min.	5
26215	Q-sep Q210	2	150mg MgSO <sub>4</sub> , 25mg PSA	Mini-Multiresidue, European EN 15662	1	0.5	3,000 U/min.	5
26217	Q-sep Q212	2	150mg MgSO <sub>4</sub> , 25mg PSA, 2.5mg GCB	Mini-Multiresidue, European EN 15662	1	2	3,000 U/min.	5
26218	Q-sep Q213	2	150mg MgSO <sub>4</sub> , 25mg PSA, 7.5mg GCB	Mini-Multiresidue, European EN 15662	1	2	3,000 U/min.	5
26223	Q-sep Q370	15	900mg MgSO <sub>4</sub> , 150mg PSA	European EN 15662	6	0.5	3,000 U/min.	5
26224	Q-sep Q371	15	900mg MgSO <sub>4</sub> , 150mg PSA, 15mg GCB	European EN 15662	6	2	3,000 U/min.	5
26225	Q-sep Q372	15	900mg MgSO <sub>4</sub> , 150mg PSA, 45mg GCB	European EN 15662	6	2	3,000 U/min.	5
26124	Q-sep Q250	2	150mg MgSO <sub>4</sub> , 50mg PSA	AOAC 2007.01	1	0.5	>1,500 rcf	1
26125	Q-sep Q251	2	150mg MgSO <sub>4</sub> , 50mg PSA, 50mg C18	AOAC 2007.01	1	0.5	>1,500 rcf	1
26219	Q-sep Q252	2	150mg MgSO <sub>4</sub> , 50mg PSA, 50mg C18, 50mg GCB	AOAC 2007.01	1	0.5	>1,500 rcf	1
26220	Q-sep Q350	15	1,200mg MgSO <sub>4</sub> , 400mg PSA	AOAC 2007.01	8	0.5	>1,500 rcf	1
26221	Q-sep Q351	15	1,200mg MgSO <sub>4</sub> , 400mg PSA, 400mg C18	AOAC 2007.01	8	0.5	>1,500 rcf	1
26222	Q-sep Q352	15	1,200mg MgSO <sub>4</sub> , 400mg PSA, 400mg C18, 400mg GCB	AOAC 2007.01	8	0.5	>1,500 rcf	1
26123	Q-sep Q253	2	150mg MgSO <sub>4</sub> , 50mg PSA, 50mg GCB	AOAC 2007.01	1	2	>1,500 rcf	1
26226	Q-sep Q373	15	900mg MgSO <sub>4</sub> , 150mg PSA, 150mg C18	similar to European EN 15662	6	0.5	3,000 U/min.	5
26126	Q-sep Q374	15	900mg MgSO <sub>4</sub> , 300mg PSA, 150mg GCB	NA	6	2	3,000 U/min.	5

PSA = primary and secondary exchange material  
GCB = graphitized carbon black

Notes:  
U/min.= Undrehungen pro minute and is the German unit of revolutions per minute (RPM)  
rcf = relative centrifugal force and can be converted to RPM using  $rcf = 1.12r \left( \frac{RPM}{1000} \right)^2$   
r = the radius of the centrifuge rotation.



Description	Material	Methods	qty.	cat#
<b>50mL Centrifuge Tubes for Sample Extraction</b>				
Q110	4g MgSO <sub>4</sub> , 1g NaCl, 1g trisodium citrate dihydrate, .5g disodium hydrogencitrate sesquihydrate	European EN 15662	50-pk.	26213
Q150	6g MgSO <sub>4</sub> , 1.5g NaOAc	AOAC 2007.1	50-pk.	26214
Empty 50mL Centrifuge Tube	—	European EN 15662, AOAC 2007.1	25-pk.	26227
<b>2mL Micro-Centrifuge Tubes for dSPE (clean-up of 1mL extract)</b>				
Q210	150mg MgSO <sub>4</sub> , 25mg PSA	European EN 15662	100-pk.	26215
Q211	150mg MgSO <sub>4</sub> , 25mg PSA, 25mg C18	—	100-pk.	26216
Q212	150mg MgSO <sub>4</sub> , 25mg PSA, 2.5mg GCB	European EN 15662	100-pk.	26217
Q213	150mg MgSO <sub>4</sub> , 25mg PSA, 7.5mg GCB	European EN 15662	100-pk.	26218
Q250	150mg MgSO <sub>4</sub> , 50mg PSA	AOAC 2007.1	100-pk.	26124
Q251	150mg MgSO <sub>4</sub> , 50mg PSA, 50mg C18	AOAC 2007.1	100-pk.	26125
Q253	150mg MgSO <sub>4</sub> , 50mg PSA, 50mg GCB	—	100-pk.	26123
Q252	150mg MgSO <sub>4</sub> , 50mg PSA, 50mg C18, 50mg GCB	AOAC 2007.1	100-pk.	26219
<b>15mL Centrifuge Tubes for dSPE (clean-up of 6mL extract)</b>				
Q350	1200mg MgSO <sub>4</sub> , 400mg PSA	AOAC 2007.1	50-pk.	26220
Q351	1200mg MgSO <sub>4</sub> , 400mg PSA, 400mg C18	AOAC 2007.1	50-pk.	26221
Q352	1200mg MgSO <sub>4</sub> , 400mg PSA, 400mg C18, 400mg GCB	AOAC 2007.1	50-pk.	26222
Q370	900mg MgSO <sub>4</sub> , 150mg PSA	European EN 15662	50-pk.	26223
Q371	900mg MgSO <sub>4</sub> , 150mg PSA, 15mg GCB	European EN 15662	50-pk.	26224
Q372	900mg MgSO <sub>4</sub> , 150mg PSA, 45mg GCB	European EN 15662	50-pk.	26225
Q373	900mg MgSO <sub>4</sub> , 150mg PSA, 150mg C18	—	50-pk.	26226
Q374	900mg MgSO <sub>4</sub> , 300mg PSA, 150mg GCB	—	50-pk.	26126

**Call Technical Service at 800-356-1688 or 814-353-1300, ext. 4 (or your Restek representative) if you have any questions about this product or any other Restek product.**



**Restek U.S.** • 110 Benner Circle • Bellefonte, PA 16823 • 814-353-1300 • 800-356-1688 • fax: 814-353-1309  
**Restek France** • phone: +33 (0)1 60 78 32 10 • fax: +33 (0)1 60 78 70 90 • e-mail: restek@restekfrance.fr  
**Restek GmbH** • phone: +49 (0)6172 2797 0 • fax: +49 (0)6172 2797 77 • e-mail: info@restekgmbh.de  
**Restek Ireland** • phone: +44 (0)2890 814576 • fax: +44 (0)2890 814576 • e-mail: restekurope@aol.com  
**Restek Japan** • phone: +81 (3)6459 0025 • fax: +81 (3)6459 0025 • e-mail: ryosei.kanaguchi@restek.com  
**Thames Restek U.K. LTD** • phone: +44 (0)1494 563377 • fax: +44 (0)1494 564990 • e-mail: sales@thamesrestek.co.uk