

QuEChERS Methodology: AOAC Approach

Resprep™ Q150, cat.# 26214

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.¹ 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, are formulated according to these methods.

- **Mini-Multiresidue Method:** QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products²
- **AOAC Official 2007.01 Method:** Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate³
- **European prEN_15662 Method, Version 2007-10-24:** 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-method⁴

General Procedures

Several common steps to all 3 QuEChERS-based methods above. 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*² are given inside this instruction guide.

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.

References

1. M. Anastassiades, S.J. Lehotay, D. Stajnbauer, 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. 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. prEN 15662 Version 2007-10-24, 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).

Table I Restek 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	Resprep Q211	2	150mg MgSO ₄ , 25mg PSA, 25mg C18	Mini-Multiresidue	1	0.5	3,000 U/min.	5
26215	Resprep Q210	2	150mg MgSO ₄ , 25mg PSA	Mini-Multiresidue, European prEN-15662	1	0.5	3,000 U/min.	5
26217	Resprep Q212	2	150mg MgSO ₄ , 25mg PSA, 2.5mg GCB	Mini-Multiresidue, European prEN-15662	1	2	3,000 U/min.	5
26218	Resprep Q213	2	150mg MgSO ₄ , 25mg PSA, 7.5mg GCB	Mini-Multiresidue, European prEN-15662	1	2	3,000 U/min.	5
26223	Resprep Q370	15	900mg MgSO ₄ , 150mg PSA	European prEN-15662	6	0.5	3,000 U/min.	5
26224	Resprep Q371	15	900mg MgSO ₄ , 150mg PSA, 15mg GCB	European prEN-15662	6	2	3,000 U/min.	5
26225	Resprep Q372	15	900mg MgSO ₄ , 150mg PSA, 45mg GCB	European prEN-15662	6	2	3,000 U/min.	5
26124	Resprep Q250	2	150mg MgSO ₄ , 50mg PSA	AOAC 2007.01	1	0.5	>1,500 rcf	1
26125	Resprep Q251	2	150mg MgSO ₄ , 50mg PSA, 50mg C18	AOAC 2007.01	1	0.5	>1,500 rcf	1
26219	Resprep Q252	2	150mg MgSO ₄ , 50mg PSA, 50mg C18, 50mg GCB	AOAC 2007.01	1	0.5	>1,500 rcf	1
26220	Resprep Q350	15	1,200mg MgSO ₄ , 400mg PSA	AOAC 2007.01	8	0.5	>1,500 rcf	1
26221	Resprep Q351	15	1,200mg MgSO ₄ , 400mg PSA, 400mg C18	AOAC 2007.01	8	0.5	>1,500 rcf	1
26222	Resprep Q352	15	1,200mg MgSO ₄ , 400mg PSA, 400mg C18, 400mg GCB	AOAC 2007.01	8	0.5	>1,500 rcf	1
26123	Resprep Q253	2	150mg MgSO ₄ , 50mg PSA, 50mg GCB	AOAC 2007.01	1	2	>1,500 rcf	1
26226	Resprep Q373	15	900mg MgSO ₄ , 150mg PSA, 150mg C18	similar to European prEN-15662	6	0.5	3,000 U/min.	5
26126	Resprep Q374	15	900mg MgSO ₄ , 300mg PSA, 150mg GCB	NA	6	2	3,000 U/min.	5

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

U/min. = Umdrehungen 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.

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AOAC Approach

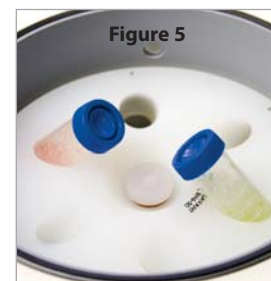
The procedures below are based on AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.³ For complete information, refer to the original source method.



Sample Extraction

1. Homogenize the frozen commodity to generate a uniform sample representative of the product (Figure 1).
2. Weigh 15g of homogenized product into a clean 50mL tube (cat.# 26227) (Figure 2).
3. Add 15mL of 1% acetic acid in acetonitrile (v/v) and an appropriate amount of an internal standard solution. Add the contents of a Resprep™ Q150 tube (cat.# 26214) to each extracted sample (Table II, Figure 3).
4. Shake vigorously for 1 minute by hand (Figure 4).
5. Centrifuge for 1 minute at >1,500 rcf to separate the solid material (Figure 5). Proceed with sample clean-up.

Table II
Resprep™ Q150 Tube Contents
6.0g magnesium sulfate, anhydrous



dSPE Sample Clean-up

Restek dSPE tubes are formulated in accordance with published methods and are listed in Table I. Select tubes based on the method used and sample type; general guidelines for different sample types include:

For samples with greater than 1% fat:

Clean up with 50mg PSA, 150mg MgSO₄, and 50mg C18 **per mL** of extract.

For samples with colored extracts:

Clean up with 50mg PSA, 150mg MgSO₄, and 50mg graphitized carbon **per mL** of extract.

For samples with colored extracts containing greater than 1% fat:

Clean up with 50mg PSA, 150mg MgSO₄, 50mg C18, and 50mg graphitized carbon **per mL** of extract.

For all other samples:

Clean up with 50mg PSA and 150mg MgSO₄ **per mL** of extract.

Note: To determine the amount (mg) of PSA, MgSO₄, and graphitized carbon, use the suggested number of milligrams and multiply by the number of mLs you want to extract.



Once tubes are selected, dSPE sample clean-up can be accomplished according to the following procedure:

1. Using the centrifuged sample extracts, transfer the supernatant to the dSPE tube as shown in Figures 6A & 6B. Use Table 1 to determine the volume of sample that should be transferred.
2. Shake vigorously for 30 seconds (Figure 7).
3. Centrifuge for 1 minute at >1,500 rcf to separate the solid material (Figure 8).
4. Transfer sample to an autosampler vial and test using GC or LC methods. Additional steps to prepare the sample for specific types of analysis are addressed in AOAC Official Method 2007.01.

