

QuEChERS Methodology: AOAC Method

Q-sep™ Packets—cat.# 26237 and 26238

Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS (“catchers”) method is based on work done and published by Anastassiades *et al.* [1] QuEChERS was developed using an extraction method for pesticides in fruits and vegetables, coupled with a cleanup 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 2 mL and 15 mL sizes) contains magnesium sulfate (to remove residual water) and primary secondary amine (PSA) sorbent (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 Q-sep™ dSPE tubes, listed in Table I (last page), are formulated according to these methods.

- **Original Unbuffered Method**

Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and “Dispersive Solid-Phase Extraction” for the Determination of Pesticide Residues in Produce [1]

- **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 [2]

- **Mini-Multiresidue Method**

QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products [3]

- **AOAC Official 2007.01 Method**

Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate [4]



Choose a QuEChERS method based on the commodity and the compounds of interest. The Original Unbuffered Method is the most simple and works well for many pesticides and matrices; however, it is important to choose a properly buffered method like EN 15662, Mini-Multiresidue, or AOAC 2007.01 when analyzing pH-sensitive pesticides (e.g., folpet, chlorothalonil, captan, captafol, pymetrozine, atrazine, tolylfluand, and dichlofluanid). Buffered methods help control pH during the extraction steps and optimize recovery of acid- and base-sensitive pesticides.

For help determining the most suitable QuEChERS method for your project, please contact Restek technical service at 1-814-353-1300, ext. 4.

General Procedures (common to all 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 sorbent 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 or solvent-exchanged prior to analysis by either GC-MS or LC-MS.

Multiresidue QuEChERS Procedure

The procedures below are based on *AOAC Official 2007.01 Method* [4]. For complete information, refer to the original source method available from AOAC International.

Sample Extraction

1. Homogenize the commodity to generate a uniform sample representative of the product (Figure 1).
2. Weigh 15 g of homogenized product into a clean 50 mL tube (cat.# 26239) as shown in Figure 2.
3. Add 15 mL of 1% acetic acid in acetonitrile (v/v) and an appropriate amount of an internal standard solution.

Sample Drying and Buffering

1. Use one Q-sep™ AOAC Method packet (cat.# 26238) for each extracted sample. Contents are listed below.

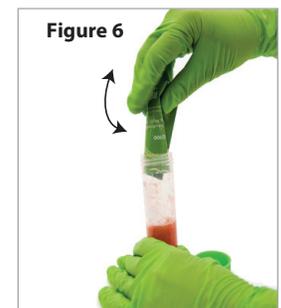
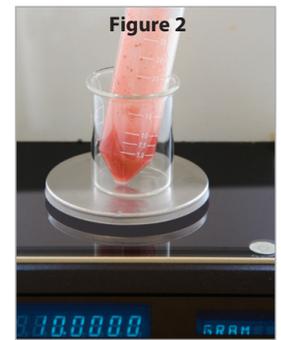
6.0g ± 0.3g magnesium sulfate, anhydrous
1.5g ± 0.1g sodium acetate, anhydrous

2. Tap the packet on a flat surface or shake the packet to ensure the salts are at the bottom of the packet (Figure 3).
3. Open the packet by tearing straight across at the precut slit (Figure 4).
4. Pinch the packet to form an opening. Tilt the packet over the tube and insert the top of the packet approximately 1 inch into the top of the tube (Figure 5).
5. Tap the bottom of the packet to ensure all of the salts have been poured into the extraction tube. (Figure 6).
6. Immediately vortex or shake vigorously by hand for 1 minute.

Phase Separation

Centrifuge for 1 minute at >1,500 rcf to separate the solid material (Figure 8). Proceed with dSPE sample cleanup or analyze extract directly without cleanup.

The Q-sep™ 3000 centrifuge shown here (110V, cat.# 26230, and 220V, cat.# 26231) meets or exceeds Original Unbuffered, AOAC, and European QuEChERS methods requirements and supports 50 mL, 15 mL, and 2 mL centrifuge tubes.



dSPE Sample Cleanup

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 cleanup methods can be applied. Specifically, dispersive solid phase extraction is discussed here. Restek Q-sep™ dSPE tubes are formulated in accordance with published methods and are listed in Table I. 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 cleanup, samples are put in freezer (>1 hr. to overnight). Cold samples are then re-centrifuged and fats or waxes are removed. If fat remains, clean up with 50 mg PSA, 150 mg MgSO₄, and 50 mg C18 **per mL** of extract. If no fat remains, clean up with 50 mg PSA and 150 mg MgSO₄ **per mL** of extract.

For samples with remaining fats:

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

For samples with intensely colored extracts:

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

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

Clean up with 25 mg PSA, 150 mg MgSO₄, and 2.5 mg graphitized carbon **per mL** of extract.

For all other samples:

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

Once tubes are selected, dSPE sample cleanup 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 9. Use Table I to determine the volume of sample that should be transferred.
2. Vortex or shake vigorously by hand for 30 seconds or 2 minutes (Figure 10). Use Table I to determine the suggested shake time.
3. Centrifuge for 1 minute at >1,500 rcf. to separate the solid material (Figure 11).
4. Transfer sample to an autosampler vial and test using GC or LC methods (Figure 12). Additional steps to prepare the sample for specific types of analysis are addressed in AOAC Official Method 2007.01.⁴

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.

References

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

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

| Cat.# | Centrifuge Tube Size (mL) | Contains | Method | Sample Volume (mL) | Shake Time (min.) | Centrifuge Speed | Centrifuge Time (min.) |
|-------|---------------------------|---|--|--------------------|-------------------|------------------|------------------------|
| 26216 | 2 | 150mg MgSO ₄ , 25mg PSA, 25mg C18 | Mini-Multiresidue | 1 | 0.5 | 3,000 U/min. | 5 |
| 26217 | 2 | 150mg MgSO ₄ , 25mg PSA, 2.5mg GCB | Mini-Multiresidue, European EN 15662 | 1 | 2 | 3,000 U/min. | 5 |
| 26218 | 2 | 150mg MgSO ₄ , 25mg PSA, 7.5mg GCB | Mini-Multiresidue, European EN 15662 | 1 | 2 | 3,000 U/min. | 5 |
| 26224 | 15 | 900mg MgSO ₄ , 150mg PSA, 15mg GCB | European EN 15662 | 6 | 2 | 3,000 U/min. | 5 |
| 26225 | 15 | 900mg MgSO ₄ , 150mg PSA, 45mg GCB | European EN 15662 | 6 | 2 | 3,000 U/min. | 5 |
| 26215 | 2 | 150mg MgSO ₄ , 25mg PSA | Original Unbuffered Mini-Multiresidue, European EN 15662 | 1 | 0.5 | 3,000 U/min. | 5 |
| 26223 | 15 | 900mg MgSO ₄ , 150mg PSA | Original Unbuffered European EN 15662 | 6 | 0.5 | 3,000 U/min. | 5 |
| 26124 | 2 | 150mg MgSO ₄ , 50mg PSA | AOAC 2007.01 | 1 | 0.5 | >1,500 rcf | 1 |
| 26125 | 2 | 150mg MgSO ₄ , 50mg PSA, 50mg C18 | AOAC 2007.01 | 1 | 0.5 | >1,500 rcf | 1 |
| 26219 | 2 | 150mg MgSO ₄ , 50mg PSA, 50mg C18, 50mg GCB | AOAC 2007.01 | 1 | 0.5 | >1,500 rcf | 1 |
| 26220 | 15 | 1,200mg MgSO ₄ , 400mg PSA | AOAC 2007.01 | 8 | 0.5 | >1,500 rcf | 1 |
| 26221 | 15 | 1,200mg MgSO ₄ , 400mg PSA, 400mg C18 | AOAC 2007.01 | 8 | 0.5 | >1,500 rcf | 1 |
| 26222 | 15 | 1,200mg MgSO ₄ , 400mg PSA, 400mg C18, 400mg GCB | AOAC 2007.01 | 8 | 0.5 | >1,500 rcf | 1 |
| 26123 | 2 | 150mg MgSO ₄ , 50mg PSA, 50mg GCB | AOAC 2007.01 | 1 | 2 | >1,500 rcf | 1 |
| 26226 | 15 | 900mg MgSO ₄ , 150mg PSA, 150mg C18 | similar to European EN 15662 | 6 | 0.5 | 3,000 U/min. | 5 |
| 26245 | 15 | 900mg MgSO ₄ , 300mg C18 | similar to European EN 15662 | 6 | 2 | 3,000 U/min. | 5 |
| 26244 | 15 | 1,200mg MgSO ₄ , 400mg C18 | similar to AOAC 2007.01 | 8 | 0.5 | >1,500 rcf | 1 |
| 26242 | 2 | 150mg MgSO ₄ , 50mg C18 | NA | 1 | 2 | 3,000 U/min. | 5 |
| 26243 | 2 | 150mg MgSO ₄ , 50mg PSA, 50mg C18, 7.5mg GCB | Universal | 1 | 2 | 3,000 U/min. | 5 |
| 26126 | 15 | 900mg MgSO ₄ , 300mg PSA, 150mg GCB | NA | 6 | 2 | 3,000 U/min. | 5 |

Notes: U/min. = Undrehungen per 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.



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

TSCD—trisodium citrate dihydrate
 DHS—disodium hydrogen citrate sesquihydrate
 NaOAc—sodium acetate

Questions?

Contact Technical Service at 1-800-356-1688, 1-814-353-1300, ext. 4, or support@restek.com (or contact your Restek representative).

Q-sep™ QuEChERS Sample Prep Packets & Tubes

| Description | Material | Methods | qty. | cat# |
|--|---|---|-----------------------|-------|
| Q-sep Kit | 4g MgSO ₄ , 1g NaCl with 50mL Centrifuge Tube | Original Unbuffered | 50 packets & 50 tubes | 23991 |
| Q-sep Packets | 4g MgSO ₄ , 1g NaCl | Original Unbuffered | 50 packets | 23992 |
| Q-sep Kit | 4g MgSO ₄ , 1g NaCl, 1g TSCD, 0.5g DHS with 50mL Centrifuge Tube | European EN 15662 | 50 packets & 50 tubes | 26235 |
| Q-sep Packets | 4g MgSO ₄ , 1g NaCl, 1g TSCD, 0.5g DHS | European EN 15662 | 50 packets | 26236 |
| Q-sep Kit | 6g MgSO ₄ , 1.5g NaOAc with 50mL Centrifuge Tube | AOAC 2007.01 | 50 packets & 50 tubes | 26237 |
| Q-sep Packets | 6g MgSO ₄ , 1.5g NaOAc | AOAC 2007.01 | 50 packets | 26238 |
| Empty 50mL Centrifuge Tube, Polypropylene | | | 50-pk. | 26239 |
| Empty 50mL Centrifuge Tube, FEP | | | 2-pk. | 23997 |
| Description | | Methods | qty. | cat# |
| 2mL Micro-Centrifuge Tubes for dSPE (cleanup of 1mL extract) | | | | |
| 150mg MgSO ₄ , 25mg PSA | | Original Unbuffered, Mini-Multiresidue, European EN 15662 | 100-pk. | 26215 |
| 150mg MgSO ₄ , 25mg PSA, 25mg C18 | | Mini-Multiresidue | 100-pk. | 26216 |
| 150mg MgSO ₄ , 25mg PSA, 2.5mg GCB | | Mini-Multiresidue, European EN 15662 | 100-pk. | 26217 |
| 150mg MgSO ₄ , 25mg PSA, 7.5mg GCB | | Mini-Multiresidue, European EN 15662 | 100-pk. | 26218 |
| 150mg MgSO ₄ , 50mg PSA | | AOAC 2007.01 | 100-pk. | 26124 |
| 150mg MgSO ₄ , 50mg PSA, 50mg C18 | | AOAC 2007.01 | 100-pk. | 26125 |
| 150mg MgSO ₄ , 50mg PSA, 50mg GCB | | AOAC 2007.01 | 100-pk. | 26123 |
| 150mg MgSO ₄ , 50mg PSA, 50mg C18, 50mg GCB | | AOAC 2007.01 | 100-pk. | 26219 |
| 150mg MgSO ₄ , 50mg C18 | | NA | 100-pk. | 26242 |
| 150mg MgSO ₄ , 50mg PSA, 50mg C18, 7.5mg GCB | | Universal | 100-pk. | 26243 |
| 15mL Centrifuge Tubes for dSPE (cleanup of 6mL and 8mL extract) | | | | |
| 1200mg MgSO ₄ , 400mg PSA | | AOAC 2007.01 | 50-pk. | 26220 |
| 1200mg MgSO ₄ , 400mg PSA, 400mg C18 | | AOAC 2007.01 | 50-pk. | 26221 |
| 1200mg MgSO ₄ , 400mg PSA, 400mg C18, 400mg GCB | | AOAC 2007.01 | 50-pk. | 26222 |
| 1200mg MgSO ₄ , 400mg C18 | | similar to AOAC 2007.01 | 50-pk. | 26244 |
| 900mg MgSO ₄ , 150mg PSA | | Original Unbuffered, European EN 15662 | 50-pk. | 26223 |
| 900mg MgSO ₄ , 150mg PSA, 15mg GCB | | European EN 15662 | 50-pk. | 26224 |
| 900mg MgSO ₄ , 150mg PSA, 45mg GCB | | European EN 15662 | 50-pk. | 26225 |
| 900mg MgSO ₄ , 150mg PSA, 150mg C18 | | similar to European EN 15662 | 50-pk. | 26226 |
| 900mg MgSO ₄ , 300mg PSA, 300mg C18, 45mg GCB | | similar to European EN 15662 | 50-pk. | 26245 |
| 900mg MgSO ₄ , 300mg PSA, 150mg GCB | | NA | 50-pk. | 26126 |



Innovative Chromatography Products

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