

QuEChERS Sample Preparation Procedures

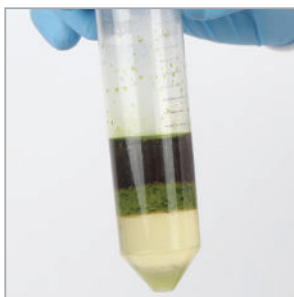
cat.# 25847, 25848, 25849, 25850, 25851, 25852, 26123, 26124, 26125, 26126, 26215, 26216, 26217, 26218, 26219, 26220, 26221, 26222, 26223, 26224, 26225, 26226, 26242, 26243, 26244, 26245

Welcome to the **Quick, Easy, Cheap, Effective, Rugged, and Safe** sample preparation technique: QuEChERS (“catchers”)! Starting with a properly homogenized sample, QuEChERS is a two-stage process, as outlined below. These QuEChERS instructions cover both Stage 1 (extraction) and Stage 2 (cleanup). They include a general overview of the stages, as well as method-specific information for each of the three major QuEChERS methods. Simply find the section pertaining to the specific catalog part number (cat.#) that you purchased and follow the instructions provided.

NOTE: These instructions are intended to supplement the originating method documentation cited in the References section. Depending on the QuEChERS methodology you have chosen, you should also obtain the documentation associated with that method to help with certain method-specific choices.

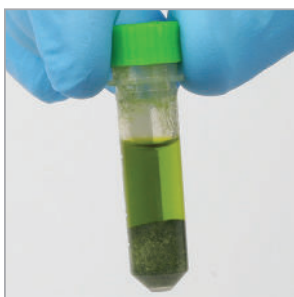
General overview of the two stages of the QuEChERS technique

NOTE: The process of obtaining a properly homogenized sample is not covered in these QuEChERS instructions. Please consult either the official methods or other resources to determine the best way of preparing your sample for the QuEChERS extraction technique.



Stage 1: Sample extraction

Analytes of interest are extracted from the sample through the addition of an organic solvent and a blend of salts. The salts enhance extraction efficiency and allow the normally miscible organic solvent to separate from the water in the sample. The mixture is shaken to assist extraction. Centrifugation is then used to separate the organic phase from the aqueous phase and the sample solids, allowing for easy subsampling of the extract. Internal standards are typically added during this stage. Note that these instructions provide only general guidelines; optimal results may be obtained by setting parameters such as pH, extraction time, and centrifugation conditions during method development for your specific analytes and sample matrices.



Stage 2: Sample cleanup

A subsample of the organic solvent extract from Stage 1 is cleaned up through the use of a dispersive solid phase extraction media (dSPE). Sugars, fatty acids, organic acids, pigments, lipids, and other potential interferences can also be extracted in Stage 1, depending on the commodity being tested. Stage 2 offers a variety of cleanup options that can be selected to meet the demands of your particular sample. Visit www.restek.com/quenchers to determine which dSPE product works best for your sample type.



Stage 1: Sample Extraction

ORIGINAL UNBUFFERED METHOD INSTRUCTIONS (cat.# 25847 and 25848)

Extraction Salt Packet Contents:

- 4 g anhydrous magnesium sulfate
- 1 g of sodium chloride



1. Homogenize the food matrix to generate a uniform sample that is representative of the product.



2. Open the cap of a 50 mL centrifuge tube and weigh in 10 g of homogenized sample. Note that the 10 g sample size is for matrices >80% water. For drier materials, consult the method or other resources.



3. Add 10 mL of acetonitrile.



4. Immediately recap the 50 mL tube and vortex or shake it vigorously by hand for 1 minute.



5. Open the extraction salt packet by tearing straight across the precut slit.



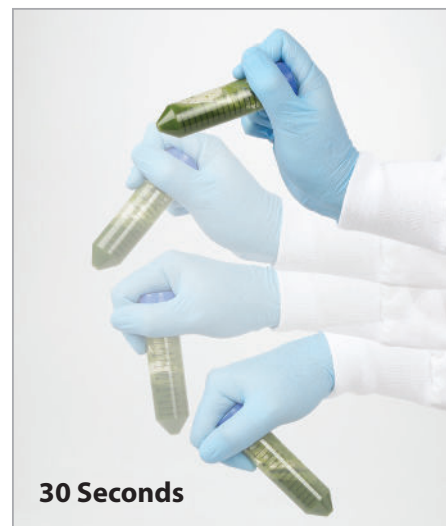
6. Open the cap of the 50 mL tube from step 4 and pour in the entire contents of one packet of extraction salts.



7. Immediately recap the 50 mL tube and vortex or shake it vigorously by hand for 1 minute.



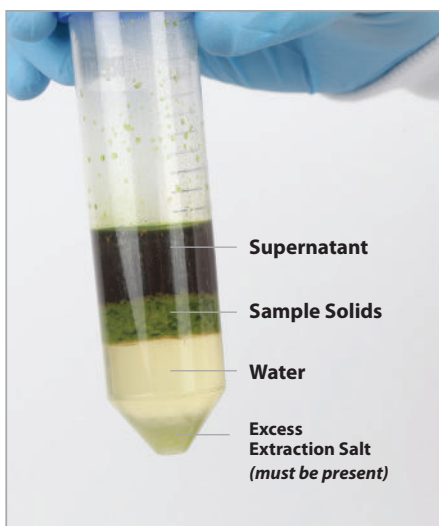
8. Open the cap of the 50 mL tube and add internal standards per reference [1]. Standards are available from Restek.



9. Immediately recap the 50 mL tube and vortex or shake it vigorously by hand for 30 seconds.



10. Centrifuge the 50 mL tube per reference [1] to separate the layers.



11. Confirm that four layers are present to ensure the extraction occurred correctly, and then proceed to instructions for Stage 2: Sample Cleanup [Page 8] or analyze the sample without cleanup.

Stage 1: Sample Extraction

EN 15662 & MINI-MULTIRESIDUE METHOD INSTRUCTIONS (cat.# 25849 and 25850)

Extraction Salt Packet Contents:

- 4 g anhydrous magnesium sulfate
- 1 g trisodium citrate dihydrate
- 1 g of sodium chloride
- 0.5 g disodium hydrogencitrate sesquihydrate



1. Homogenize the food matrix to generate a uniform sample that is representative of the product. For commodities with more than 5% fat (w/w), see the specialized sample preparation procedure in the source method [2, 3].



2. Open the cap of a 50 mL centrifuge tube and weigh in 10 g of homogenized sample. Note that the 10 g sample size is for matrices >80% water. For drier materials, consult the method or other resources.



3. Add 10 mL of acetonitrile.



4. Add internal standards per reference [2] or [3]. Standards are available from Restek.



5. Immediately recap the 50 mL tube and vortex or shake it vigorously by hand for 1 minute.

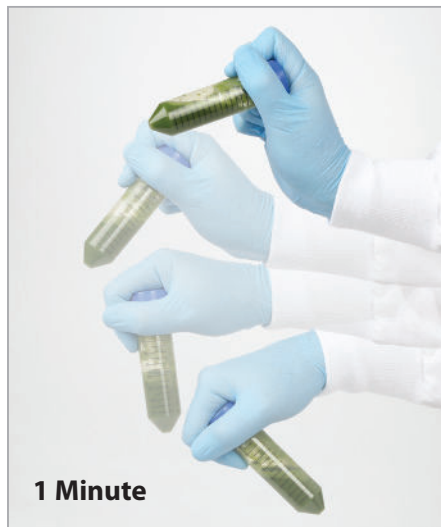
Note: If pH < 3, sample should be adjusted with addition of 600 µL of 5 N NaOH. If pH > 3 and < 5, sample should be adjusted with 200 µL of 5 N NaOH.



6. Open the extraction salt packet by tearing straight across the pre-cut slit.



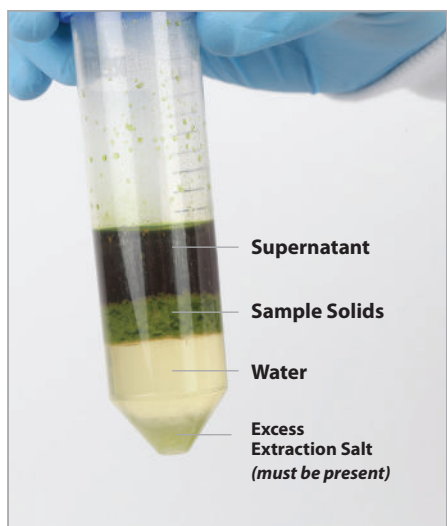
7. Open the cap of the 50 mL tube from step 5 and pour in the entire contents of one packet of extraction salts.



8. Immediately recap the 50 mL tube and vortex or shake it vigorously by hand for 1 minute.



9. Centrifuge the 50 mL tube per reference [2] or [3] to separate the layers.



10. Confirm that four layers are present to ensure the extraction occurred correctly, and then proceed to instructions for Stage 2: Sample Cleanup [Page 8] or analyze the sample without cleanup.

Stage 1: Sample Extraction

AOAC 2007.01 METHOD INSTRUCTIONS (cat.# 25851 and 25852)

Extraction Salt Packet Contents:

- 6 g anhydrous magnesium sulfate
- 1.5 g anhydrous sodium acetate



1. Homogenize the food matrix to generate a uniform sample that is representative of the product.



15 g

2. Open the cap of a 50 mL centrifuge tube and weigh in 15 g of homogenized sample. Note that the 15 g sample size is for matrices >80% water. For drier materials, consult the method or other resources.



3. Add 15 mL of a 1% acetic acid solution in acetonitrile (v/v).



4. Add internal standards per reference [4]. Standards are available from Restek.



5. Open the extraction salt packet by tearing straight across the precut slit.



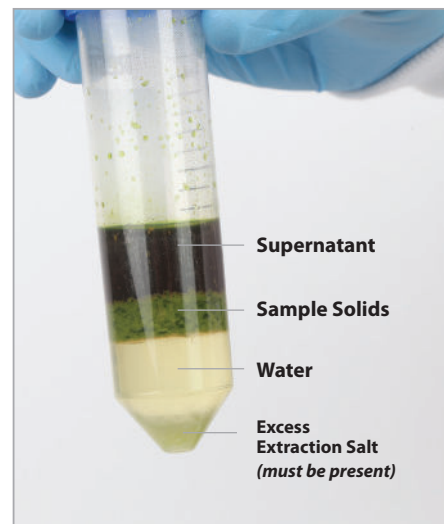
6. Open the cap of the 50 mL tube from step 4 and pour in the entire contents of one packet of extraction salts.



7. Immediately recap the 50 mL tube and vortex or shake it vigorously by hand for 1 minute.



8. Centrifuge the 50 mL tube per reference [4] to separate the layers.



9. Confirm that four layers are present to ensure the extraction occurred correctly, and then proceed to instructions for Stage 2: Sample Cleanup [Page 8] or analyze the sample without cleanup.

Stage 2: dSPE Cleanup of Sample Extracts

The same general dSPE cleanup steps are followed regardless of extraction technique and cleanup product. However, the specific quantities of sorbent, sample volume, and shake time used may differ based on the cleanup product being used. Use the catalog number for your dSPE product to identify the correct values for your cleanup process.



1. Open the cap of a dSPE tube and transfer in an aliquot of the supernatant (organic solvent layer) of the centrifuged extracts from Stage 1 (see table for transfer volume).



2. Immediately recap the dSPE tube and vortex or shake it vigorously by hand (see table for shake time).



3. Centrifuge following the analytical method to separate the solid material.

Note: Consult your specific method for additional sample acidification or stabilization steps that may be required for your particular analytes of interest and matrix.



4. After centrifugation is complete, transfer the supernatant from the dSPE tube to an autosampler vial, make any final adjustments if required, and test using an appropriate GC or LC method.

Part numbers are arranged in ascending numerical order

dSPE (cat.#)	Volume (mL)	Shake Time (min)
26123	1	2
26124	1	0.5
26125	1	0.5
26126	6	2
26215	1	0.5
26216	1	0.5
26217	1	2
26218	1	2
26219	1	0.5
26220	8	0.5
26221	8	0.5
26222	8	0.5
26223	6	2
26224	6	2
26225	6	2
26226	6	0.5
26242	1	2
26243	1	2
26244	8	0.5
26245	6	2

References (Restek is not able to provide copies of these documents.)

- [1] M. Anastassiades, 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 Int. 86 (2003) 412-431. <http://pubag.nal.usda.gov/pubag/download-PDF.xhtml?id=555&content=PDF>
- [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, 2008.
- [3] QuEChERS-A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products, 2004. <http://quechers.cvua-stuttgart.de/pdf/reality.pdf>
- [4] AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate, 2007.

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