

Supel™ QuE (QuEChERS) Product Line

Description

Dispersive SPE (dSPE), often referred to as the “QuEChERS” method (Quick, Easy, Cheap, Effective, Rugged, and Safe), is a sample prep technique that has become popular in the area of multi-residue pesticide analysis in food and agricultural products.

Food/agricultural samples are first extracted with an aqueous miscible solvent (e.g., acetonitrile) in the presence of high amounts of salts (e.g., sodium chloride and magnesium sulfate) and/or buffering agents (e.g. citrate) to induce liquid phase separation and stabilize acid and base labile pesticides, respectively. Upon shaking and centrifugation, an aliquot of the organic phase is subjected to further cleanup using SPE. Unlike traditional methods using SPE tubes, dSPE cleanup is conducted by mixing bulk amounts of SPE (e.g., Supelclean™ PSA, ENVI-Carb™, and/or Discovery® DSC-18) with the extract. After sample cleanup, the mixture is centrifuged and the resulting supernatant can either be analyzed directly or can be subjected to minor further treatment before analysis.

Supelco® now carries a line of Supel™ QuE centrifuge tubes containing predetermined amounts of salts and SPE sorbents to support the most common method configurations used today. Supelco® also offers Z-Sep sorbents, unique sorbents for analysis of challenging matrices (fatty or lipid containing). The Z-Sep sorbents provide improved sample cleanup over traditional PSA/C18 which can extend analytical column life and instrument throughput. They provide an advantage especially for analysis of more hydrophobic compounds.

Procedure

The two most predominate dispersive methods were developed by Steven Lehotay and Michelangelo Anastassiades. Described below is a summary outline of the methods. For a detailed description of the protocol, please refer to the original references. For PFAS testing in food, follow QuEChERS sample prep method described in FDA method F_C.010.02.

Procedure 1—as described in the following references:

- Official Method EN15662:2018
- M. Anastassiades, QuEChERS—A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products, <http://www.quechers.com/>

Sample Extraction

1. Weigh 10 g sample into a 50 mL centrifuge tube.
2. Add 10 mL acetonitrile + 100 µL I.S. solution. Shake vigorously for 1 min.
3. Add contents of Supel™ QuE Citrate Extraction Tube (55227-U). Shake vigorously for 1 min., and centrifuge for 5 min. at 3000 U/min.

Note: For pesticides with acidic functional groups (e.g., phenoxyalcanoic acids), 200 µL of the supernatant should be aliquoted and analyzed directly (without dispersive SPE cleanup) by LC-MS/MS (negative ion mode). Acidic pesticides may retain on the dispersive SPE PSA sorbent resulting in poor recovery.

QuEChERS technique

1. For samples with high fat content and/or waxes (some cereals and citrus fruits), place the extract in the freezer for >1 hour.

Transfer 6 mL of the acetonitrile layer to Supel™ QuE PSA (EN) Tube (55228-U), or transfer 1 mL of the acetonitrile layer to Supel™ QuE PSA (EN) Tube, 2 mL (55172-U).

For samples of higher fat content, use Supel™ QuE PSA/C18 (EN) Tube (55439-U or 55173-U) or any of Z-Sep/Z-Sep+ sorbents. The Z-Sep materials are more efficient alternatives to the material described in the EN15662:2018.

Procedure 1 (continued)

For samples with moderate levels of chlorophyll and carotinoides (e.g., carrots, romaine lettuce, head lettuce, etc.), use Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 1 (55446-U or 55174-U).

For samples with higher levels of chlorophyll and carotinoides (e.g., red sweet pepper, spinach, lamb's lettuce, rucolla, etc.), use Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 2 (55464-U or 55176-U). Alternatively, for improved recovery of planar pesticides in green matrices, use Supel™ QuE Verde (55442-U or 55447-U).

2. Shake for 30 sec. (2 min. when ENVI-Carb™ is used). Centrifuge for 5 min. at 3000 U/min. Remove a small aliquot of the cleaned extract supernatant for the analysis of sulfonyl urea herbicides, carbosulfan, and benfuracarb.
3. Acidify the remaining supernatant by transferring an aliquot of the supernatant to a fresh cap vial and adding 10 µL of 5% formic acid in acetonitrile to every mL of supernatant isolated.

Procedure 2—as described in the following references:

- AOAC International Official Method 2007.01.
- S.J. Lehotay, K. Mastovska, A.R. Lightfield, Use of Buffer and Other Means to Improve Results of Problematic Pesticides in a Fast and Easy Method for Residue Analysis of Fruits and Vegetables, J-AOAC-Int., Mar-Apr 2005; 88(2):615-629
- S.J. Lehotay, Interlaboratory Validation of the QuEChERS Method to Analyze Pesticide Residues in Fruits and Vegetables, Proceedings AOAC Annual meeting, St. Louis, MO USA (2004)

Sample Extraction

1. Transfer 10–15 g homogenized food sample to 50 mL PTFE centrifuge tube.
2. Per 15 g sample, add 15 mL 1% acetic acid in acetonitrile + contents of Supel™ QuE Acetate (AC) Tube (55234-U) + 75 µL I.S. solution.
3. Shake vigorously 1 min.; Centrifuge >1500 rcf for 1 min.

QuEChERS technique

1. Transfer 8 mL of acetonitrile layer to Supel™ QuE PSA (AC) Tube (55466-U) or Supel™ QuE PSA/C18 (AC) Tube (55470-U). Or transfer 1 mL of acetonitrile layer to Supel™ QuE PSA (AC) Tube, 2 mL (55287-U) or Supel™ QuE PSA/C18 (AC) Tube, 2 mL (55288-U). As a more efficient alternative to the PSA/C18 material described in the AOAC 2007.01 use any of the Z-Sep/Z-Sep+ sorbents.

For samples with higher level of chlorophyll or carotenoids, use Supel™ QuE PSA/C18/ENVI-Carb™ (AC) Tube (55474-U) or Supel™ QuE PSA/C18/ENVI-Carb™ (AC) Tube, 2 mL (55289-U). Alternatively, for improved recovery of planar pesticides in green matrices, use Supel™ QuE Verde (55442-U or 55447-U).

2. Centrifuge >1500 rcf for 1 min.
3. Transfer supernatant to a GC or LC vial for concurrent LC-MS and GC-MS analysis. Note that further processing may be necessary prior to chromatographic analysis (e.g., addition of formic acid for LC-MS analysis; or evaporation of supernatant and reconstitute with toluene for GC-MS analysis).



Pre-Packed dSPE Tubes

Description	Qty.	Cat. No.
EN15662:2018 (15 mL centrifuge tubes, shaker compatible)		
Supel™ QuE PSA (EN) Tube, 15 mL 150 mg Supelclean™ PSA, 900 mg MgSO ₄	50	55228-U
Supel™ QuE PSA/C18 (EN) Tube, 15 mL 150 mg Supelclean™ PSA, 150 mg Discovery® DSC-18, 900 mg MgSO ₄	50	55439-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 1, 15 mL 150 mg Supelclean™ PSA, 15 mg Supelclean™ ENVI-Carb™, 900 mg MgSO ₄	50	55446-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 2, 15 mL 150 mg Supelclean™ PSA, 45 mg Supelclean™ ENVI-Carb™, 900 mg MgSO ₄	50	55464-U
Supel™ QuE PSA/ENVI-Carb™ Tube 3, 15 mL 300 mg Supelclean™ PSA, 150 mg Supelclean™ ENVI-Carb™, 900 mg MgSO ₄	50	55479-U
Supel™ QuE Citrate (EN) Tube, 15 mL 4 g MgSO ₄ , 1 g NaCl, 0.5 g NaCitrate dibasic sesquihydrate, 1 g NaCitrate tribasic dehydrate	50	55227-U
Supel™ QuE Citrate/Sodium Bicarbonate (EN) Tube, 15 mL 4 g MgSO ₄ , 5 g NaBicarbonate, 1 g NaCl, 0.5 g NaCitrate dibasic sesquihydrate, 1 g NaCitrate tribasic dehydrate	50	55237-U
EN15662:2018 (2 mL centrifuge tubes)		
Supel™ QuE PSA (EN) Tube, 2 mL 25 mg Supelclean™ PSA, 150 mg MgSO ₄	100	55172-U
Supel™ QuE PSA/C18 (EN) Tube, 2 mL 25 mg Supelclean™ PSA, 25 mg Discovery® DSC-18, 150 mg MgSO ₄	100	55173-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 1, 2 mL 25 mg Supelclean™ PSA, 2.5 mg Supelclean™ ENVI-Carb™, 150 mg MgSO ₄	100	55174-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 2, 2 mL 25 mg Supelclean™ PSA, 7.5 mg Supelclean™ ENVI-Carb™, 150 mg MgSO ₄	100	55176-U
AOAC 2007.01 (15 mL centrifuge tubes, shaker compatible)		
Supel™ QuE PSA (AC) Tube, 15 mL 400 mg Supelclean™ PSA, 1200 mg MgSO ₄	50	55466-U
Supel™ QuE PSA/C18 (AC) Tube, 15 mL 400 mg Supelclean™ PSA, 400 mg Discovery® DSC-18, 1200 mg MgSO ₄	50	55470-U
Supel™ QuE PSA/C18/ENVI-Carb™ (AC) Tube, 15 mL 400 mg Supelclean™ PSA, 400 mg Discovery® DSC-18, 400 mg Supelclean™ ENVI-Carb™, 1200 mg MgSO ₄	50	55474-U
Supel™ QuE Acetate (AC) Tube, 15 mL 6 g MgSO ₄ , 1.5 g NaAcetate	50	55234-U
AOAC 2007.01 (2 mL centrifuge tubes)		
Supel™ QuE PSA (AC) Tube, 2 mL 50 mg Supelclean™ PSA, 150 mg MgSO ₄	100	55287-U
Supel™ QuE PSA/C18 (AC) Tube, 2 mL 50 mg Supelclean™ PSA, 150 mg MgSO ₄ , 50 mg Discovery® DSC-18	100	55288-U
Supel™ QuE PSA/C18/ENVI-Carb™ (AC) Tube, 2 mL 50 mg Supelclean™ PSA, 150 mg MgSO ₄ , 50 mg Discovery® DSC-18, 50 mg ENVI-Carb™	100	55289-U
Supel™ QuE PSA/ENVI-Carb™ (AC) Tube, 2 mL 50 mg Supelclean™ PSA, 150 mg MgSO ₄ , 50 mg ENVI-Carb™	100	Custom

Description	Qty.	Cat. No.
Specialty Products for Challenging Matrices and Analytes (2 mL centrifuge tubes)		
Supel™ QuE Z-Sep Tube, 2 mL 75 mg Z-Sep	100	55411-U
Supel™ QuE Z-Sep/C18 Tube, 2 mL 20 mg Z-Sep, 50 mg Discovery® DSC-18	100	55284-U
Supel™ QuE Z-Sep+ Tube, 2 mL 75 mg Z-Sep+	100	55408-U
Supel™ QuE Verde Tube, 2 mL 60 mg Z-Sep+, 50 mg Supelclean™ PSA, 10 mg Supelclean™ ENVI-Carb™ Y, 150 mg MgSO ₄	100	55447-U
15 mL centrifuge tubes, shaker compatible		
Supel™ QuE Z-Sep Tube, 15 mL 500 mg Z-Sep	50	55491-U
Supel™ QuE Z-Sep/C18 Tube, 15 mL 120 mg Z-Sep, 300 mg Discovery® DSC-18	50	55506-U
Supel™ QuE Z-Sep+ Tube, 15 mL 500 mg Z-Sep+	50	55486-U
Supel™ QuE Z-Sep+/MgSO ₄ Tube, 15 mL 300 mg Z-Sep+, 900 mg MgSO ₄	50	55511-U
Supel™ QuE Verde Tube, 15 mL 480 mg Z-Sep+, 400 mg Supelclean™ PSA, 80 mg Supelclean™ ENVI-Carb™ Y, 1200 mg MgSO ₄	50	55442-U
Non-buffered extraction tubes (15 mL centrifuge tubes)		
Supel™ QuE Non-Buffered Tube 1, 15 mL 4 g MgSO ₄ , 1 g NaCl	50	55294-U
Supel™ QuE Non-Buffered Tube 2, 15 mL 6 g MgSO ₄ , 1.5 g NaCl	50	55295-U

Bulk Adsorbents and Salts

Description	Qty.	Cat. No.
Supelclean™ PSA, bulk sorbent	100 g	52738-U
Supelclean™ ENVI-Carb™, bulk sorbent	50 g	57210-U
Discovery® DSC18, bulk sorbent	100 g	52600-U
Supel™ QuE Z-Sep+, bulk sorbent	20 g	55299-U
Supel™ QuE Z-Sep, bulk sorbent	20 g	55418-U
MgSO ₄ (as cited in EN15662:2018)	var.	208094
Sodium citrate dibasic sesquihydrate	var.	71635
Sodium citrate tribasic dihydrate	var.	54641
Sodium chloride	var.	57653
Sodium acetate	var.	241245

Description	Qty.	Cat. No.	Comment
FDA Method C_010.02 (PFAS in Food)			
Supel™ QuE, Non-Buffered tube 2, pk of 50	50	55295-U	Extraction salts
pk of 50 Supel™ QuE PSA/ ENVI-Carb™ Tube 3, 15 mL	50	55479-U	Clean-up tube with sorbent, 900 mg MgSO ₄ , 300 mg PSA, 150 mg ENVI-Carb
BRAND® PP graduated cen- trifuge tube, screw cap volume 50 mL, without base, non-sterile	300	BR114820	Empty tube

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