

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 is becoming increasingly 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 pre-determined amounts of salts and SPE sorbents to support the most common method configurations used today. Supelco also offers two unique sorbents (Z-Sep and Z-Sep+) when using LC-MS analysis for challenging matrices (fat-ty or lipid containing). Z-Sep/C18 and Z-Sep+ will provide improved sample clean-up over traditional PSA/C18 which can extend analytical column life and instrument throughput.

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.

Procedure 1 – as described in the following references:

- Official Method EN15662:2008
- 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., phenoxyacetic 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.
2. Transfer 6 mL of the acetonitrile layer to Supel™ QuE PSA (EN) Tube (**55228-U**).

For samples of higher fat content, use Supel™ QuE PSA/C18 (EN) Tube (**55229-U**) or transfer 1 mL of the acetonitrile layer into the Supel™ QuE Z-Sep/C18 Tube (**55284-U**) or 3 mL into the Supel™ QuE Z-Sep+ Tube (**55296-U**). The Z-Sep materials are more efficient alternatives to the material described in the EN15662:2008.

Procedure 1 (contd.)

For samples with moderate levels of chlorophyll and carotenoids (e.g., carrots, romaine lettuce, head lettuce, etc.), use Supel™ QuE PSA/ENVI-Carb (EN) Tube 1 (**55230-U**).

For samples with higher levels of chlorophyll and carotenoids (e.g., red sweet pepper, spinach, lamb's lettuce, rucicola, etc.), use Supel™ QuE PSA/ENVI-Carb (EN) Tube 2 (**55233-U**).

3. 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, carbo-sulfan, and benfuracarb.
4. 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 (**55282-U**) or Supel™ QuE PSA/C18 (AC) Tube (**55283-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 the Supel™ QuE Z-Sep/C18 Tube (**55284-U**) or transfer 3 mL of the acetonitrile layer and use Supel™ QuE Z-Sep+ Tube (**55296-U**).

For samples with higher level of chlorophyll or carotenoids, use Supel™ QuE PSA/C18/ENVI-Carb (AC) Tube (**55286-U**) or Supel™ QuE PSA/C18/ENVI-Carb (AC) Tube, 2 mL (**55289-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.
EN15662:2008 (12 mL centrifuge tubes)		
Supel™ QuE Citrate (EN) Tube 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 Biocarbonate (EN) Tube 4 g MgSO ₄ , 5 g NaBicarbonate, 1 g NaCl, 0.5 g NaCitrate dibasic sesquihydrate, 1 g NaCitrate tribasic dehydrate	50	55237-U
Supel™ QuE PSA (EN) Tube 150 mg Supelclean PSA, 900 mg MgSO ₄	50	55228-U
Supel™ QuE PSA/C18 (EN) Tube 150 mg Supelclean PSA, 150 mg Discovery DSC-18, 900 mg MgSO ₄	50	55229-U
Supel™ QuE PSA/ENVI-Carb (EN) Tube 1 150 mg Supelclean PSA, 15 mg Supelclean ENVI-Carb, 900 mg MgSO ₄	50	55230-U
Supel™ QuE PSA/ENVI-Carb (EN) Tube 2 150 mg Supelclean PSA, 45 mg Supelclean ENVI-Carb, 900 mg MgSO ₄	50	55233-U
AOAC 2007.01 (12 mL centrifuge tubes)		
Supel™ QuE Acetate (AC) Tube 6 g MgSO ₄ , 1.5 g NaAcetate	50	55234-U
Supel™ QuE PSA (AC) Tube 400 mg Supelclean PSA, 1200 mg MgSO ₄	50	55282-U
Supel™ QuE PSA/C18 (AC) Tube 400 mg Supelclean PSA, 1200 mg MgSO ₄ , 400 mg Discovery DSC-18	50	55283-U
Supel™ QuE PSA/C18/ENVI-Carb (AC) Tube 400 mg Supelclean PSA, 1200 mg MgSO ₄ , 400 mg Discovery DSC-18, 400 mg ENVI-Carb	50	55286-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 50 mg Supelclean PSA, 150 mg MgSO ₄ , 50 mg ENVI-Carb	100	Custom
Specialty Products for Challenging (Fatty/Lipid containing) Matrices (LC-MS only)		
Supel™ QuE Z-Sep/C18 Tube (2 mL centrifuge tubes) 20 mg Z-Sep, 50 mg Discovery DSC-C18	100	55284-U
Supel™ QuE Z-Sep+ (12 mL centrifuge tubes) 500 mg Z-Sep+, 12 mL centrifuge tube	50	55296-U
Non-buffered extraction tubes (12 mL centrifuge tubes)		
Supel™ QuE Non-Buffered Tube 1 4 g MgSO ₄ , 1 g NaCl	50	55294-U
Supel™ QuE Non-Buffered Tube 2 6 g MgSO ₄ , 1.5 g NaCl	50	55295-U

Description	Qty.	Cat. No.
Empty Extraction Tubes (50 mL) 50 mL empty Extraction Centrifuge Tubes	50	55248-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
Z-Sep+	20 g	55299-U
MgSO ₄ (as cited in EN15662:2008)	var.	63135
Sodium citrate dibasic sesquihydrate	var.	71635
Sodium citrate tribasic dihydrate	var.	32320
Sodium chloride	var.	71379
Sodium acetate	var.	241245

Custom Supel™ QuE Products

Supelco has introduced a custom service for these tubes that allows users to easily design their own dispersive SPE tube(s). Five different tube dimensions and types can be selected as well as adsorbents and salts from the vast range of Sigma-Aldrich including Supelco® adsorbents and Fluka and Aldrich salts. This service provides users with quick access to customized tubes for introduction into routine applications. Please visit us under sigma-aldrich.com/custom-quechers and follow the Custom Services link to get to more detailed information on this service and download of the quote request form.

Trademarks

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