

## Dispersive SPE (dSPE) Product Line

### Description

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

In dSPE, 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 centrifuge tubes containing pre-determined amounts of salts and SPE sorbents to support the most common method configurations used today.

### 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 Dispersive SPE (dSPE) 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.

### Dispersive SPE

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 Dispersive SPE (dSPE) PSA SPE Cleanup Tube 1 (**55228-U**).

For samples with *higher fat content*, use Dispersive SPE (dSPE) PSA/C18 SPE Cleanup Tube 1 (**55229-U**).

### Procedure 1 (contd.)

For samples with *moderate levels of chlorophyll and carotenoids* (e.g., carrots, romaine lettuce, head lettuce, etc.), use Dispersive SPE (dSPE) PSA/ENVI-Carb SPE Cleanup 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 Dispersive SPE (dSPE) PSA/ENVI-Carb SPE Cleanup 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 Dispersive SPE (dSPE) MgSO<sub>4</sub> Extraction Tube (**55234-U**) + 75 µL I.S. solution.
3. Shake vigorously 1 min.; Centrifuge > 1500 rcf for 1 min.

### Dispersive SPE

1. Transfer **8 mL** of acetonitrile layer to Dispersive SPE (dSPE) PSA Cleanup Tube 2 (**55282-U**) or Dispersive SPE (dSPE) PSA/C18 Cleanup Tube 2 (**55283-U**). Or transfer 1 mL of acetonitrile layer to Dispersive SPE (dSPE) 2 mL PSA Cleanup Tube (**55287-U**) or Dispersive SPE (dSPE) 2 mL PSA/C18 Cleanup Tube (**55288-U**)

For samples with higher level of chlorophyll or carotenoids, use (dSPE) PSA/C18/ENVI-Carb Cleanup Tube (**55286-U**) or 2 mL PSA/C18/ENVI-Carb Cleanup Tube (**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.
<b>EN15662:2008 (12 mL centrifuge tubes)</b>		
<b>Citrate Extraction Tube</b> 4 g MgSO <sub>4</sub> , 1 g NaCl, 0.5 g NaCitrate dibasic sesquihydrate, 1 g NaCitrate tribasic dehydrate	50	<b>55227-U</b>
<b>PSA SPE CleanUp Tube 1</b> 150 mg Supelclean PSA, 900 mg MgSO <sub>4</sub>	50	<b>55228-U</b>
<b>PSA/C18 SPE CleanUp Tube 1</b> 150 mg Supelclean PSA, 150 mg Discovery DSC-18, 900 mg MgSO <sub>4</sub>	50	<b>55229-U</b>
<b>PSA/ENVI-Carb SPE CleanUp Tube 1</b> 150 mg Supelclean PSA, 15 mg Supelclean ENVI-Carb, 900 mg MgSO <sub>4</sub>	50	<b>55230-U</b>
<b>PSA/ENVI-Carb SPE CleanUp Tube 2</b> 150 mg Supelclean PSA, 45 mg Supelclean ENVI-Carb, 900 mg MgSO <sub>4</sub>	50	<b>55233-U</b>
<b>AOAC 2007.01 (12 mL centrifuge tubes)</b>		
<b>MgSO<sub>4</sub>/Acetate Extraction Tube</b> 6 g MgSO <sub>4</sub> , 1.5 g NaAcetate	50	<b>55234-U</b>
<b>PSA SPE CleanUp Tube 2</b> 400 mg Supelclean PSA, 1200 mg MgSO <sub>4</sub>	50	<b>55282-U</b>
<b>PSA/C18 SPE CleanUp Tube 2</b> 400 mg Supelclean PSA, 1200 mg MgSO <sub>4</sub> , 400 mg Discovery DSC-18	50	<b>55283-U</b>
<b>PSA/C18/ENVI-Carb SPE CleanUp Tube</b> 400 mg Supelclean PSA, 1200 mg MgSO <sub>4</sub> , 400 mg Discovery DSC-18, 400 mg ENVI-Carb	50	<b>55286-U</b>
<b>AOAC 2007.01 (2 mL centrifuge tubes)</b>		
<b>PSA SPE CleanUp Tube 2 mL</b> 50 mg Supelclean PSA, 150 mg MgSO <sub>4</sub>	100	<b>55287-U</b>
<b>PSA/C18 SPE CleanUp Tube 2 mL</b> 50 mg Supelclean PSA, 150 mg MgSO <sub>4</sub> , 50 mg Discovery DSC-18	100	<b>55288-U</b>
<b>PSA/C18/ENVI-Carb SPE CleanUp Tube 2 mL</b> 50 mg Supelclean PSA, 150 mg MgSO <sub>4</sub> , 50 mg Discovery DSC-18, 50 mg ENVI-Carb	100	<b>55289-U</b>
<b>PSA/ENVI-Carb SPE CleanUp Tube</b> 50 mg Supelclean PSA, 150 mg MgSO <sub>4</sub> , 50 mg ENVI-Carb	100	<b>Custom</b>
<b>Empty Extraction Tubes (50 mL)</b>		
dSPE 50 mL empty Extraction Centrifuge Tubes	50	<b>55248-U</b>
<b>Non-buffered extraction tubes (12 mL centrifuge tubes)</b>		
<b>MgSO<sub>4</sub>/NaCl Extraction, Tube 1</b> 4 g MgSO <sub>4</sub> , 1 g NaCl	50	<b>55294-U</b>
<b>MgSO<sub>4</sub>/NaCl Extraction, Tube 2</b> 6 g MgSO <sub>4</sub> , 1.5 g NaCl	50	<b>55295-U</b>

## Bulk Adsorbents and Salts

Description	Qty.	Cat. No.
Supelclean PSA, bulk sorbent	100 g	<b>52738-U</b>
Supelclean ENVI-Carb, bulk sorbent	50 g	<b>57210-U</b>
Discovery DSC18, bulk sorbent	100 g	<b>52600-U</b>
MgSO <sub>4</sub> (as cited in EN15662:2008)	var.	<b>63135</b>
Sodium citrate dibasic sesquihydrate	var.	<b>71635</b>
Sodium citrate tribasic dihydrate	var.	<b>32320</b>
Sodium chloride	var.	<b>71379</b>
Sodium acetate	var.	<b>241245</b>

## Custom Dispersive SPE 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](http://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

Discovery, ENVI-Carb, Supelclean, Supelco — Sigma-Aldrich Biotechnology LP