RAPID PASS-THROUGH SPE CLEANUP FOR MULTI-RESIDUE ANALYSIS OF FOOD MATRICES WITH HIGH LIPID AND PIGMENT CONTENT

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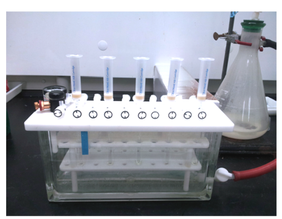
INTRODUCTION

Food safety laboratories have adopted new and simplified sample preparation methods designed to reduce analysis time and related costs, as well as to increase throughput. The QuEChERS methods for fruits and vegetables require only minutes for sample preparation, are suitable for effective recovery of hundreds of pesticides, and replace prior methods that took hours or days. In a similar fashion, effective acetonitrile based sample extraction methods have been developed for determination of multi-residue veterinary drugs in foodstuffs. Fast and effective cleanup options are available for matrices that are high in fats, phospholipids and pigments, such as avocado, liver, and eggs. Excessive amounts of these substances in the injected sample can shorten LC or GC column life, contribute to ion-suppression, and contaminate the LC-MS or GC-MS. The goal of the cleanup is to remove as much of the extracted sample matrix as possible without significant effect on the recovery of the target analytes. Among the sorbents often used for dispersive SPE cleanup (dSPE)* are octadecyl silica (C18), primary/ secondary amine silica (PSA), and graphitized carbon black (GCB). The performance of these dSPE options are compared with a new rapid pass-through cleanup option, the Oasis PRiME HLB cartridge.**

Dispersive SPE (dSPE) is sometimes simply referred to as "QuEChERS Cleanup". This is misleading because other forms of cleanup are often suitable or superior to dSPE for cleanup of QuEChERS extracts.

****Pass-through SPE is an effective cleanup method that takes only minutes to perform. Cartridges can be chosen for manual syringe manipulation (bottom left) or for use with positive-pressure or vacuum manifold (bottom right).***

or



Pass-Through SPE

SAMPLE PREPARATION

Veterinary Drugs
Initial Extraction

A 2 g sample of tissue is transferred to a 15 mL centrifuge tube containing ceramic homogenizer balls

10 mL of extraction solvent is added (0.2 % formic acid in 80:20 acetonitrile/water)

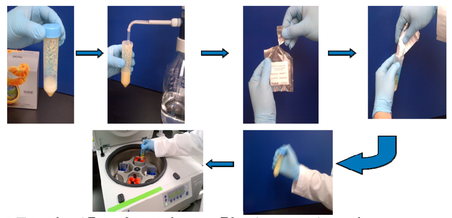
The sample is homogenized for 1.5 min (see at right)

The tubes are then centrifuged for 5 minutes @ 4000 rpm

Veterinary Drugs
Typical Cleanup Options

- *dSPE using C18 silica*
- *Pass-through SPE using Oasis PRiME HLB*

Pesticides
Initial QuEChERS Extraction



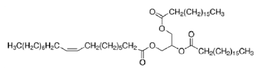
- Transfer 15 g of sample to a 50 mL extraction tube
- Add 15 mL acetonitrile. Shake for 1 minute
- Add contents of DisQuE pouch for AOAC QuEChERS
- Shake vigorously for 1 minute
- Centrifuge, take aliquot of top layer for cleanup by dSPE or pass-thru cleanup

Pesticides
Typical Cleanup Options

- *dSPE using PSA , C18 silica, and GCB*
- *Pass-through SPE using Oasis PRiME HLB*

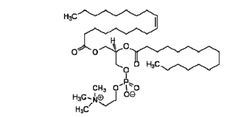
POTENTIAL MATRIX INTERFERENCES

Matrix Components for Cleanup



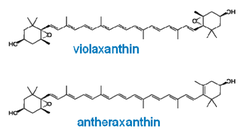
Fat (triacyl glycerol)

Can precipitate when sample is diluted for LC



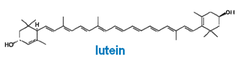
Phospholipid
(phosphatidylcholine from animal or plant lecithin)

Can build up on injector, column, mass spec

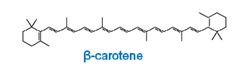


violaxanthin

antheraxanthin

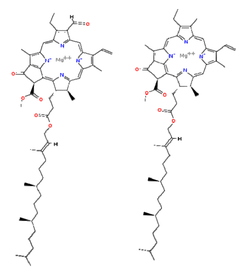


lutein



β-carotene

Pigments carotenoids (from plant and animal tissues)
Can co-elute with analytes, build up on GC injector and column



chlorophyll b chlorophyll a

Pigments chlorophyll (from plant tissues)
Can co-elute with analytes, build up on GC injector and column

Cleanup Choices

For Fat Removal:
dSPE with C18: Good to Excellent
dSPE with PSA: Poor
dSPE with GCB: Fair

Oasis PRiME HLB: Good to Excellent

For Phospholipid Removal:
dSPE with C18: Poor
dSPE with PSA: Good*
dSPE with GCB: Poor

Oasis PRiME HLB: Excellent

*** cannot use for acidic analytes**

For Carotenoid Removal:
dSPE with C18: Fair
dSPE with PSA: Poor
dSPE with GCB: Good

Oasis PRiME HLB: Good

For Chlorophyll Removal:
dSPE with C18: Fair
dSPE with PSA: Poor
dSPE with GCB: Good to Excellent**

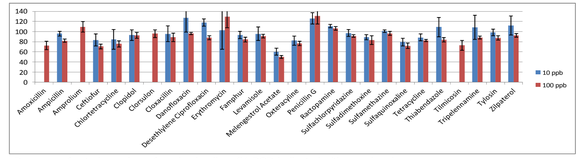
Oasis PRiME HLB: Excellent

**** Sufficient GCB for effective removal of pigments may cause significant loss of planar analytes**

GCB is only recommended for cleanup of QuEChERS extracts

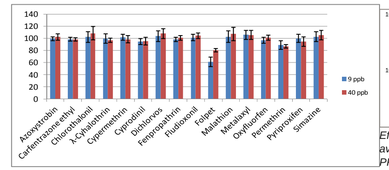
OASIS PRIME HLB CLEANUP RESULTS

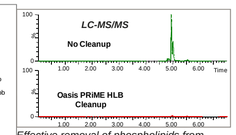
Veterinary Drugs in Beef Liver by LC-MS/MS



Recovery of veterinary drugs from bovine liver (% RSD indicated with error bars)

Pesticides in Avocado by GC-MS/MS





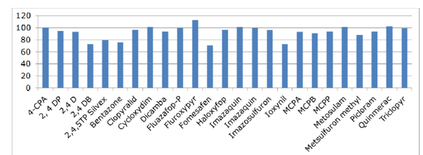
Recovery of pesticides from Avocado QuEChERS extracts

Effective removal of phospholipids from avocado QuEChERS extract with Oasis PRiME HLB clean-up.

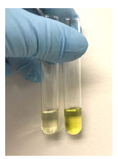
Pass-through cleanup with Oasis PRiME HLB effectively removes fats and phospholipids from extracts of beef liver, salmon, shrimp, and infant formula obtained for veterinary drug analysis. Also, similar cleanup was obtained for avocado and egg extracts obtained for pesticide analysis (QuEChERS).

Pass-through cleanup with Oasis PRiME HLB effectively removes chlorophyll and many other pigments from QuEChERS extracts of spinach and other green vegetables. (More information regarding chlorophyll cleanup is presented in poster **PW - 035**)

Acidic Pesticides in Edamame (Soybean Pods) by LC-MS/MS



Recovery of acidic hebicides from soybean pod QuEChERS extracts



Oasis PRiME HLB cleanup (left) and no cleanup (right)

Above are preliminary results from an on-going study. Pass-through cleanup with Oasis PRiME HLB effectively removes fats, phospholipids, chlorophyll and other pigments from the QuECHERS extract of green soy pods. Among the pesticides included in studied are the acidic herbicides shown above. **These acidic analytes show good recovery using Oasis PRiME HLB cleanup but *cannot be recovered using dSPE with PSA sorbent.***

For instrument conditions and other detailed information on the applications mentioned in this poster see Waters application notes: 720005488EN (fish), 720005730EN (infant formula), 720005816EN (avocado), 720005887EN (liver), 720005794 (egg) and 720005994 (spinach).

CONCLUSIONS

- **Pass-through cleanup with the Oasis PRiME HLB cartridge is an effective alternative to dSPE for QuEChERS pesticide analysis**
- **Pass-through cleanup with the Oasis PRiME HLB cartridge is an effective alternative to dSPE for veterinary drug analysis**
- **Fats, phospholipids and non-polar pigment are removed**
- **Oasis PRiME HLB cleanup <u>can</u> be applied to acidic pesticides; dSPE with PSA <u>cannot</u>**

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