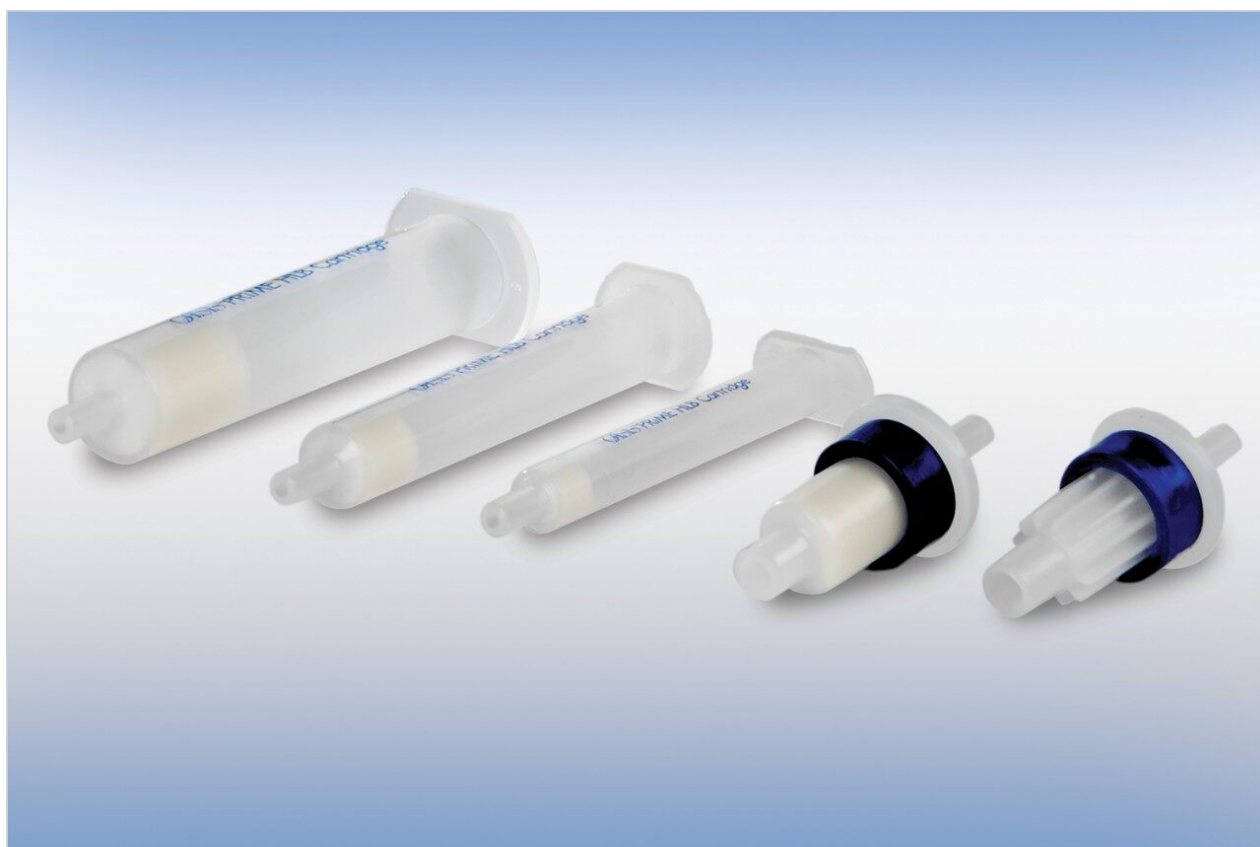


Application Note

Oasis PRiME HLB Cartridges Now Available in Syringe Compatible Plus Format

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This is an Application Brief and does not contain a detailed Experimental section.

Abstract

This application brief demonstrates Oasis PRiME HLB Cartridges in formats suitable for processing samples with or without the need for a vacuum/positive pressure manifold.

Benefits

Oasis PRiME HLB Cartridges provide rapid sample cleanup of many types of food matrices in food safety analysis. Now, Oasis PRiME HLB cartridges are available in "Plus" format with luer fittings. These versatile cartridge formats are compatible with standard syringes or can be fitted with appropriate reservoirs for use with vacuum or positive pressure manifolds.

Introduction

Oasis PRiME HLB Cartridges are effective for rapid pass-through cleanup of various food matrices in food safety analyses. These include pesticides in fruits and vegetables, as well as antibiotic residues in meats and fish. For these cleanups, the traditional "Vac" style cartridges are most conveniently used with vacuum manifolds. These cartridges are available in many sizes; the choice is made based on the volume of extract required by the analyst. In addition, the sample cleanup is performed without the cumbersome centrifugation steps required with dispersive cleanup procedures. However, there is also a need for cartridges that can be used for pass-through cleanup without a processing manifold.

The Oasis PRiME HLB Sorbent is now available in "Plus" type cartridges. These cartridges are easily connected to a syringe (in a manner similar to a syringe filter). Alternatively, when fitted with an appropriate reservoir, they can be used with vacuum manifolds. Figure 1 shows an Oasis PRiME HLB Cartridge used for pass-through cleanup of QuEChERS spinach extract in the manual mode with a syringe.



Figure 1. Oasis PRiME HLB Cartridge used in pass-through cleanup of a QuEChERS spinach extract.

Experimental

In previous studies, good recoveries were shown for a wide variety of pesticides in avocado¹ and in spinach² after cleanup using Oasis PRiME HLB Cartridges in "Vac" formats. In this application brief, similar cleanups were performed using Oasis PRiME HLB Cartridges in the "Plus" formats.

QuEChERS Extraction. A 15 g homogenized sample was weighed into a 50 mL centrifuge tube. 15 mL 1:99 acetic acid/acetonitrile were added and the sample was manually shaken for 1 minute. Then, QuEChERS salts (contents of DisQuE Pouch for AOAC QuEChERS, p/n 186006812) were added and the tube was shaken vigorously by hand for 1 minute. After centrifugation (3200 rcf for 5 minutes), portions of the supernatant were taken for cleanup with Oasis PRiME HLB Cartridges.

Cleanup. No cartridge conditioning was performed. A 3 mL syringe was connected for cleanup using Oasis

PRiME HLB in the Plus Light format and a 6 mL syringe was connected for cleanup using Oasis PRiME HLB in the Plus Short format. The extract was delivered by syringe in a manner to obtain a dropwise flow through the cartridge. A vacuum manifold was used for the “Vac” style cartridge formats. For all cartridge types, an initial portion of the QuEChERS extract (supernatant) was sent to waste after passing through the cartridge and a second portion was passed through the cartridge and collected. The volumes used for each type of cartridge are presented in Table 1.

Cartridge	Discard volume	Collect volume
3 cc 60 mg “Vac”	0.4 mL	0.6 mL
6 cc 150 mg “Vac”	0.8 mL	1.5 mL
Plus Light	0.6 mL	1 mL
Plus Short	2 mL	3 mL

Table 1. Volumes used for pass-through cleanup for each type of cartridge.

Instrumental analysis

Chlorophyll removal from spinach extracts was monitored using UPLC coupled to a photodiode array detector (PDA). Phospholipid removal from avocado extracts was monitored using UPLC-MS/MS. Pesticide analysis was accomplished using APGC-MS/MS. Details for these analyses are given in references 1 and 2.

Results and Discussion

No significant difference was seen among all cartridge types tested for any of the relevant cleanup or recovery parameters measured in this study. Phospholipid removal (see Figure 2), chlorophyll removal (see Figure 3), and pesticide recovery (see Figure 4) was virtually the same for “Vac” type cartridges processed using a vacuum manifold, or for “Plus” type cartridges processed by hand via syringe.

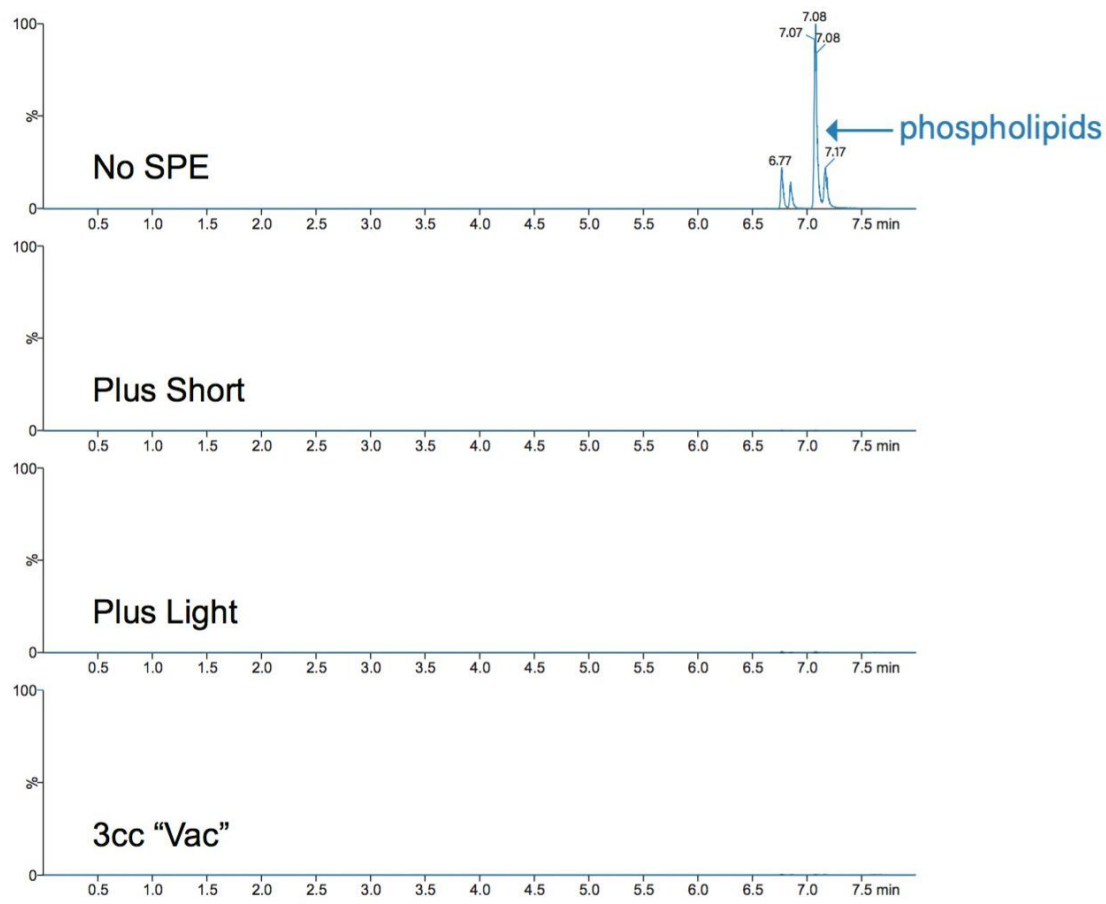


Figure 2. Equivalent removal of phospholipids from avocado QuEChERS extract using various Oasis PRiME HLB Cartridge formats (UPLC-MS/MS).

- Compounds:**
1. Violaxanthin
 2. Antheraxanthin
 3. Lutein
 4. Chlorophyll b
 5. Chlorophyll a
 6. Carotene

UPLC-PDA Conditions:

Detector: ACQUITY® PDA®
 Monitored wavelength: 450 nm
 UPLC system: ACQUITY UPLC I-Class FTN
 Column: CORTECS® UPLC T3 (2.1 x 100 mm)
 Temp.: 30 deg
 Injection Vol.: 5 µL (QuEChERS extract diluted 1:4 with water)
 Mobile phase A: 5 mM ammon. formate in water
 Mobile phase B: acetonitrile/methanol 75:25
 Gradient:

Time (min)	Flow (mL/min)	%A	%B	Curve
Initial	0.5	25	75	Initial
5.0	0.5	1	99	6
20.0	0.5	1	99	6
20.2	0.5	25	75	6
21.0	0.5	25	75	6

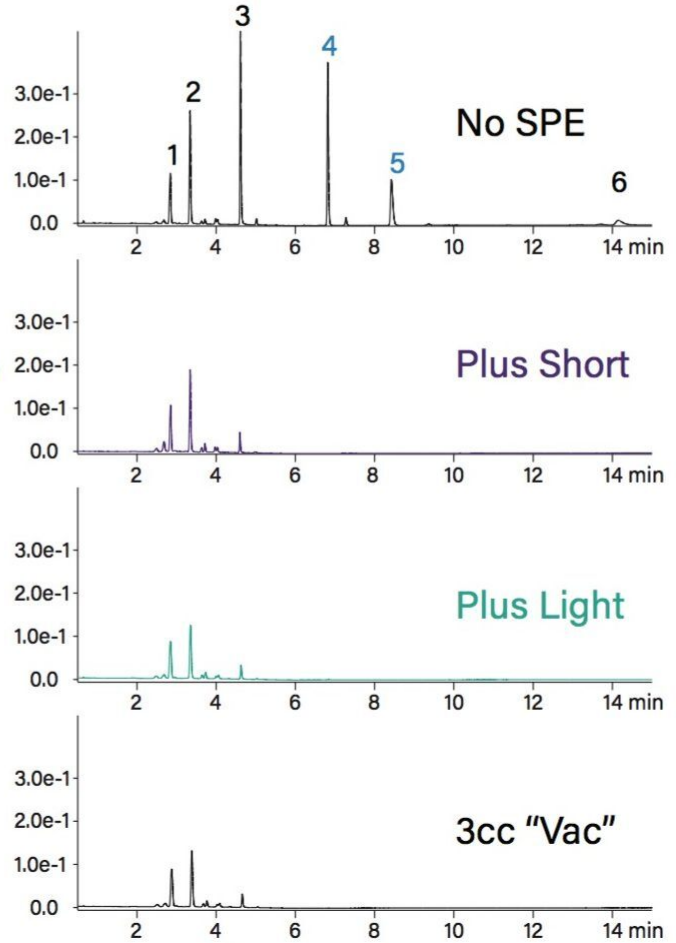
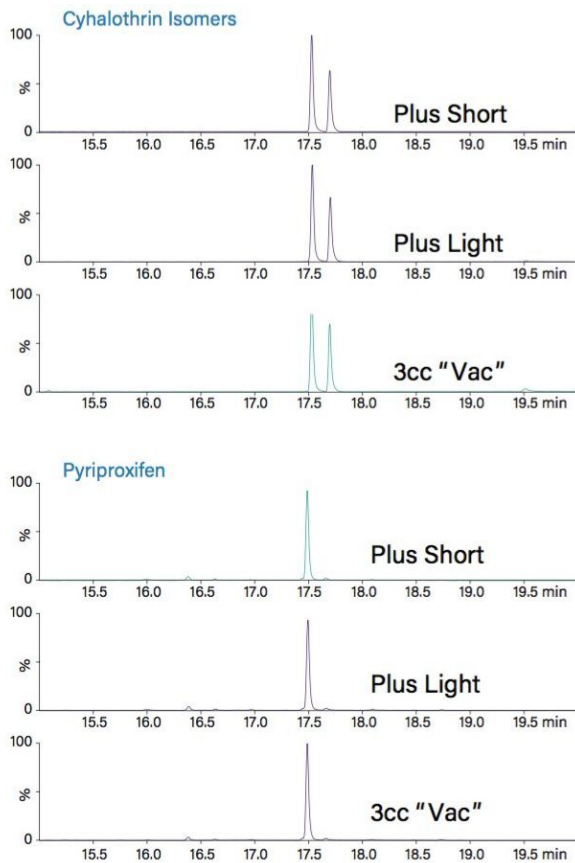


Figure 3. Effective and equivalent removal of chlorophyll from spinach QuEChERS extract using various Oasis PRiME HLB Cartridge formats.



APGC conditions:
 GC system: Agilent 7890
 Column: Restek Rxi-5ms, 30 m x 0.25 mm x 0.25 μ m
 Flow rate: 1.0 mL/min Helium
 Injection vol.: 1 μ L (15:1 split)
 Temperature program: 80 $^{\circ}$ C initial, hold for 0.5 min, 12 $^{\circ}$ C /min to 320 $^{\circ}$ C and hold for 8 min

MS conditions:
 Mass spectrometer: Xevo[®] TQ-S
 Ion mode: API+ (MRM mode)
 Corona: 2.8 μ A
 Source temp.: 150 $^{\circ}$ C
 Probe temp.: 450 $^{\circ}$ C
 Cone gas: 170 L/Hr
 Auxiliary gas: 170 L/Hr
 Collision gas: 0.15 mL/min (Ar)
 Nebulizer: 4.0 Bar
 Data management: MassLynx[®] v4.1

MRM transitions:
 Cyhalothrin: 449.0>181.2 (20 V cone, 20 eV collision)
 449.0>197.3 (20 V cone, 14 eV collision)
 Pyriproxifen: 136.1>78.0 (20 V cone, 20 eV collision)
 136.1>96.0 (20 V cone, 20 eV collision)

Figure 4. Equivalent recovery of pesticides from spinach QuEChERS extract using various Oasis PRiME HLB Cartridge formats.

The choice of cartridge size is made based on the volume of extract required by the analyst. Figure 5 illustrates this cartridge choice.

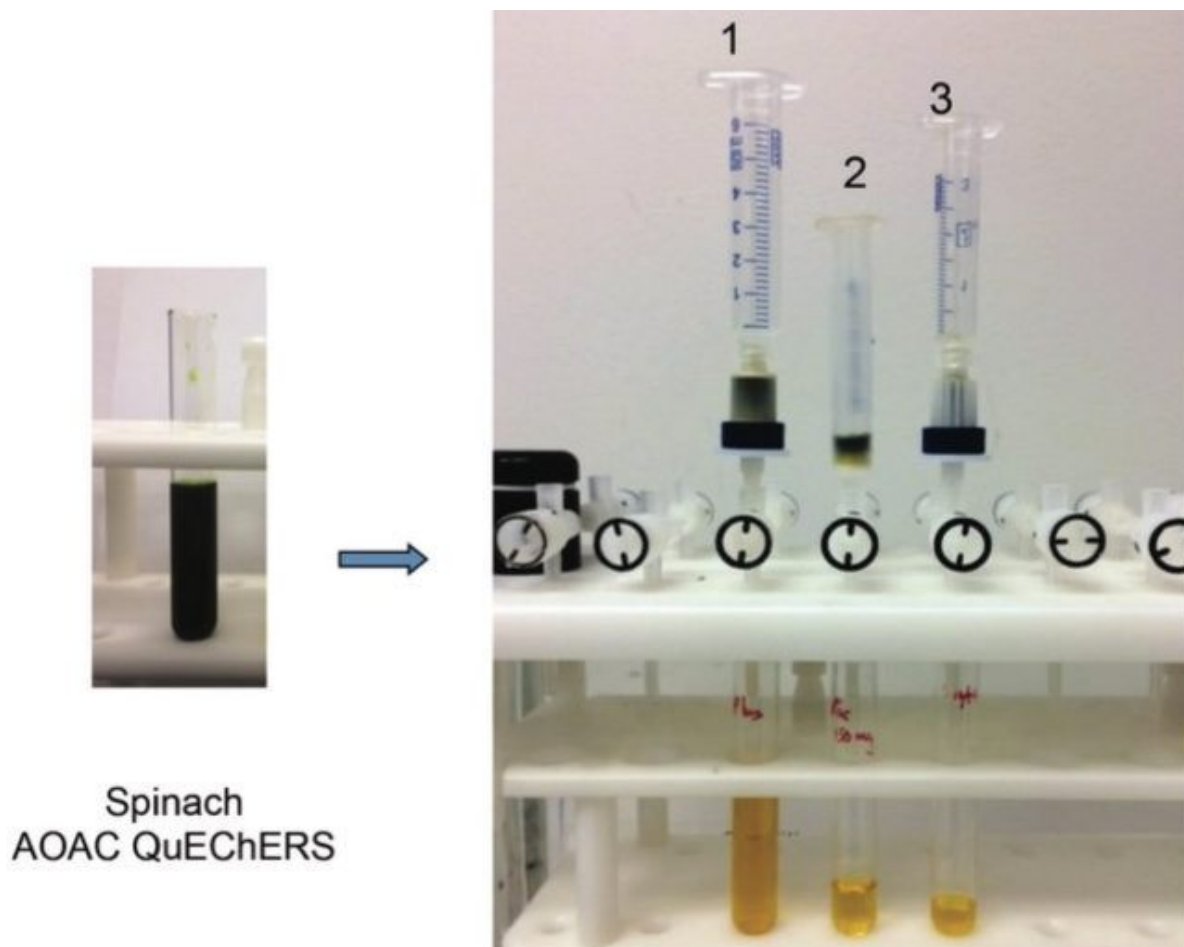


Figure 5. Oasis PRiME HLB in plus type cartridges provide identical cleanup compared with the traditional vac style cartridge.

Conclusion

- In addition to traditional "Vac" formats, Oasis PRiME HLB Cartridges are available in two "Plus" formats, Plus Light/100 mg and Plus Short/335 mg, suitable for manual syringe cleanup.
- No difference was seen for cleanups using either cartridge format.
- Pass-through cleanup with an Oasis PRiME HLB Cartridge effectively removes greater than 99% of chlorophyll and 95% of phospholipids from QuEChERS extracts.
- Pass-through cleanup with an Oasis PRiME HLB Cartridge is an effective alternative cleanup for

QuEChERS and similar acetonitrile based extraction methods.

References

1. Oasis PRiME HLB Cartridge for Rapid and Effective Cleanup of Avocado, A High Fat Matrix, Prior to APGC-MS/MS Analysis, Waters Application Note 720005816EN, 2016.
 2. Oasis PRiME HLB Cartridges for Rapid and Effective Removal of Chlorophyll From QuEChERS Spinach Extracts, Waters Technology Brief 720005994EN, 2017.
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