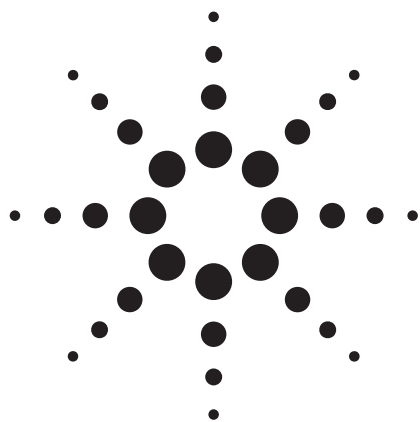




Analysis of Pesticide Residues in Spinach Using Bond Elut Carbon, Carbon/PSA and Carbon/NH₂ SPE Cartridges

Technical Overview

General Description

This technical note describes a multiresidue vegetable cleanup method for the determination of six chemicals representing various classes in spinach. Three sorbent variations: Bond Elut Carbon, Bond Elut Carbon/PSA and Bond Elut Carbon/NH₂, are evaluated with a challenging, highly pigmented matrix.

Bond Elut is the original and most trusted name in solid phase extraction. Years of use by companies worldwide have thoroughly documented its many applications and proven its properties. There are more literature references for Bond Elut than any other SPE product in the industry.

Bond Elut Carbon, Carbon/PSA, and Carbon/NH₂ Dual Phase SPE products are designed for simplicity, improved analytical performance, and ease-of-use. Additionally, time savings are realized through the simultaneous processing of the dual phase technique [1].

Bond Elut Carbon, Carbon/PSA and Carbon/NH₂ SPE products are packed with ultrapure graphitized carbon particles that have been optimized for the adsorption of pigments in food, fruits and vegetables, and small organic residues in wastewater. Therefore, regardless of the application or sample type, methods are simple, flow characteristics are superior, and analytical performance is improved, which leads to easier validation.

Simple methods deliver clean extracts and high recoveries from a wide range of compounds. The advanced dual layer design effectively eliminates the common matrix interferences that may result in lowered sensitivity and decreased data quality [1, 2].



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Key Benefits

The key benefits include:

- Reduced interference. Bond Elut Carbon provides the best removal of pigments such as chlorophyll when analyzing organic extracts of solid samples such as fruits, vegetables, grains and other foods. The laminar structure presents a highly polarizable surface to your sample. The result is fewer chromatographic or mass interferences.
- Excellent recovery. Bond Elut Carbon offers excellent retention for small organics, including those that are too polar to retain on C18 or polymeric SPE. The non-porous structure also permits easy elution, so your recoveries are higher and detection limits are better.
- Improved multiresidue analysis. Bond Elut Carbon gets broader retention and easier elution for analytes right across the polarity scale, from highly nonpolar to highly polar. The dual layer Carbon/NH₂ Bond Elut is compliant with the Japanese Positive List System for the analysis of pesticide residues in food.

Bond Elut Carbon, Carbon/NH₂ and Carbon/PSA SPE products allow you to clean and concentrate samples in complex matrices.

Individual material characteristics associated with carbon, amino, and PSA solid phase extraction include:

Bond Elut Carbon

Bond Elut Carbon is an ultra pure graphitized carbon particle that has been optimized for the adsorption of pigments in food, fruits and vegetables, and small organic residues in waste water. The powerful retention mechanism of the sorbent is appropriate for a broad range of analytes. In addition, careful manufacturing techniques result in lower carbon fines on the wall of the device.

- Very effective for cleanup of highly pigmented samples [2].
- Non-porous, allowing for rapid processing as the adsorption does not require analyte dispersion into the solid phase pores.

Bond Elut Carbon/NH₂

Bond Elut NH₂ (primary amine) is a weaker anion exchanger than sorbents such as SAX (a quaternary amine sorbent that is always charged). Therefore it is a better choice for the retention of very strong anions such as sulfonic acids, which retain irreversibly on an SAX sorbent. Similar to Diol and Si sorbents, Bond Elut NH₂ is excellent for the separation of structural isomers. Bond Elut NH₂ is an ideal sorbent for removing fatty and organic acids, as well as pigments and sugars when performing multiresidue pesticide food analysis.

- Normal phase or anion exchange sorbent
- Weaker anion exchange than SAX
- Amenable to separating structural isomers

Bond Elut Carbon/PSA

Bond Elut PSA is an alkylated amine sorbent that contains two different amino functionalities: a primary and a secondary amine. This gives a slightly higher pKa and ionic capacity compared to Bond Elut NH2. PSA has a significantly higher carbon load than most amino functional sorbents and is therefore a better choice for polar compounds that retain too strongly on Bond Elut NH2. Bond Elut PSA is an ideal sorbent for removing fatty and organic acids, as well as pigments and sugars when performing multiresidue pesticide food analysis.

- Alternative choice to Bond Elut NH2 for polar compounds
- Higher ionic capacity than NH2

Quality Controls

Bond Elut Carbon, PSA and NH2 are manufactured by Agilent under a strict ISO 9001 quality assurance program. All Bond Elut products are subject to rigorous quality control tests to ensure reproducibility of bed mass, particle size, surface area, purity and flow rate. In addition, every batch of Bond Elut Carbon is tested for its ability to decolorize chlorophyll-containing extracts.

Comparison of Bond Elut Carbon, Carbon/PSA and Carbon/NH2 with SampliQ Carbon, Carbon/PSA and Carbon/NH2

Sample preparation/SPE Method

Ten grams of spinach were spiked at 10 ppm with an aqueous, six-component analyte mixture and extracted with 20 mL of acetonitrile.

Ten milliliters of the acetonitrile spinach extract was evaporated to 1 mL, applied to the SPE cartridge, preconditioned with 5 mL of acetonitrile:toluene (3:1). The pesticides were eluted with 20 mL acetonitrile: toluene (3:1), and the resulting eluent was evaporated and reconstituted with 1 mL (85:15) H2O (0.1% Formic Acid): ACN (0.1% Formic Acid).

HPLC Conditions

Mobile phase A: H₂O 0.1% formic acid

Mobile phase B: ACN 0.1% formic acid

Gradient Program:	Time (minute)	%B
	0.0	15
	0.1	15
	5	21
	18	30
	30	67
	30.1	15

Column: Agilent ZORBAX Rapid Resolution Eclipse Plus C18;
4.6 × 150 mm, 3.5 μm (p/n 959963-902)

Detection: DAD 254 nm

Compounds	Log P	Type	pKa
Caffeine	-0.13	CNS stimulant	14
Tebuthiuron,	1.79	herbicide	0.9
Sulfadimethoxine	1.48	sulfa drug	6.1
Bromacil	2.10	pesticide	9.1
Prednisone	1.57	steroid	N/A
Warfarin	3.42	anticoagulant	4.9

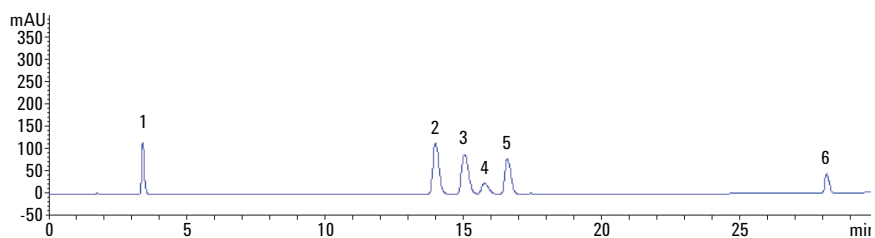


Figure 1. HPLC UV vis chromatogram of 10 ppm neat solution extract. Peak identification: 1. Caffeine, 2. Tebuthiuron, 3. Sulfadimethoxine, 4. Bromacil, 5. Prednisone, 6. Warfarin.

Experimental spinach extract results

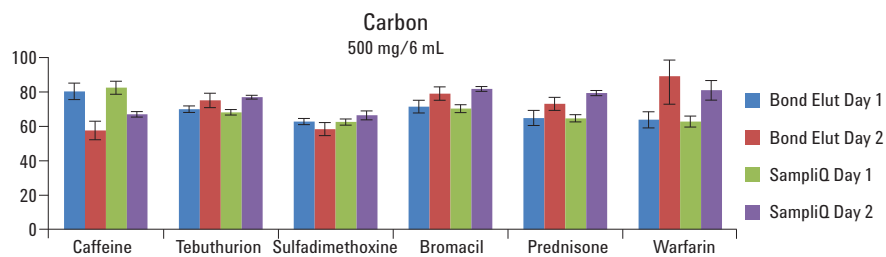


Figure 2. Recoveries and precision results for Carbon SPE comparison.

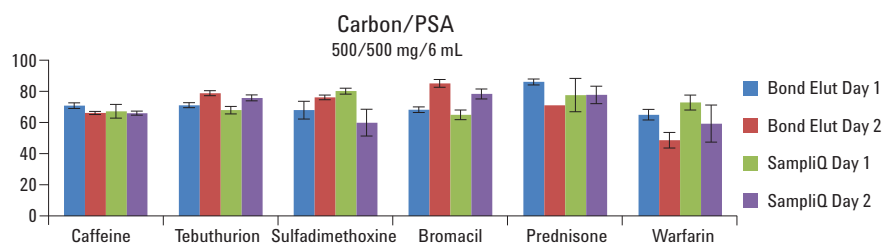


Figure 3. Recoveries and precision results for Carbon/PSA SPE comparison.

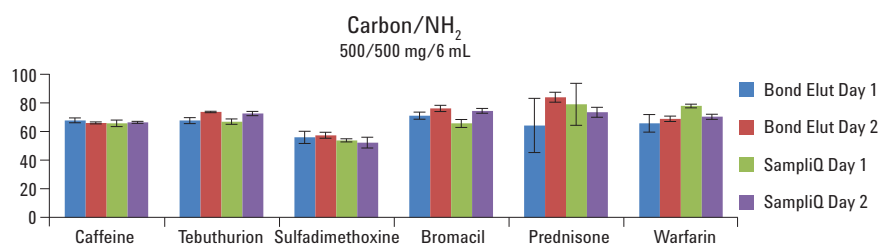


Figure 4. Recoveries and precision results for Carbon/NH₂ SPE comparison.

Spiked Spinach Matrix Cleanup

Relative comparison of matrix absorption at 254 nm.

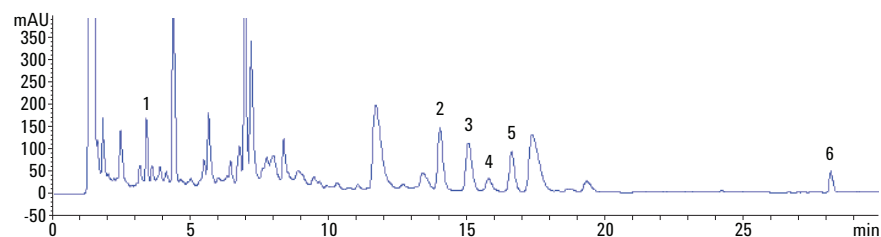


Figure 5. HPLC UV vis chromatogram of spinach sample extracts processed by Bond Elut Carbon 500 mg/6mL, p/n: 12252201.

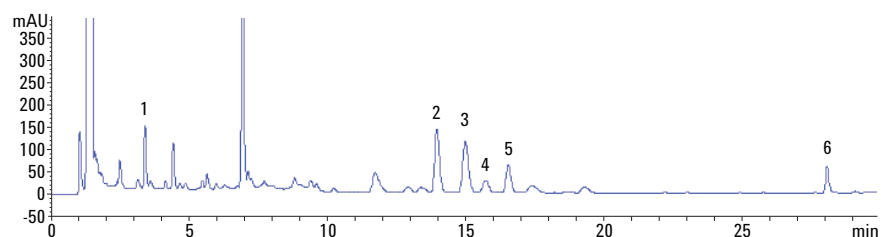


Figure 6. HPLC UV vis chromatogram of spinach sample extracts processed by Bond Elut Carbon/PSA 500/500 mg/6 mL, p/n: 12102042C500.

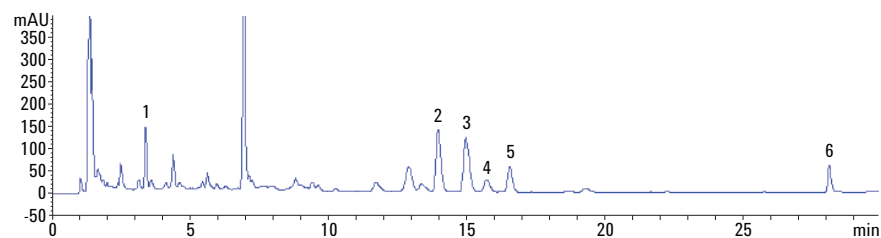


Figure 7. HPLC UV vis chromatogram of spinach sample extracts processed by Bond Elut Carbon/NH2 500/500 mg/6 mL, p/n: 12252202.

Conclusion

Exceptional recoveries and precision were obtained for the analysis of pesticides, pollutants and other organic residues. The powerful retention mechanisms of Bond Elut Carbon, Bond Elut Carbon/PSA and Bond Elut Carbon/NH₂ are the analytical cornerstone to an analyst who values better and cleaner results.

SPE is an integral sample preparation technique in the analytical workflow of complex samples. Even with the adoption of highly selective detectors, LC/MS/MS or GC/MS/MS, ion suppression from co-eluting impurities can adversely affect quantitative analyses. A clean extract directly correlates to less complicated analysis conditions, longer column life, and more accurate results. SPE is the preferred sample preparation technique compared to LLE, Protein PPT, or SPME because it offers greater flexibility, more reproducible recoveries, reduced solvent cost, substantial effectiveness as a cleanup tool, and easy automation. SPE sample preparations are used by researchers in the pharmaceutical, forensic, food safety and environmental sector.

The high quality Agilent Bond Elut Carbon, Bond Elut Carbon/PSA and Bond Elut Carbon/NH₂ product lines achieve the same or better performance as the analog SampliQ products, and should be chosen when seeking an SPE product.

References

1. J. Fillion, F. Sauve, J. Selwyn, "Multiresidue Method for the Determination of Residues of 251 Pesticides in Fruits and Vegetables by Gas Chromatography/Mass Spectrometry and Liquid Chromatography with Fluorescence Detection", J. AOAC Int. 2000,83,698-713.
2. F. J. Schenck, S. J. Lehotay, V. Vega, "Comparison of solid-phase extraction sorbents for cleanup in pesticide residue analysis of fresh fruits and vegetables", J. Sep. Sci. 2002, 25, 883-890.

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