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Solid Phase Extraction of 200 Pesticides from Fruits and Vegetables, for Analysis by GC or HPLC

L. Nolan, Sample Handling, Supelco, Bellefonte, PA, USA

Recovery rates for polar analytes are higher, and less variable, with ENVI-Carb SPE tubes than with silica-based packings. The unique carbon-based packing in ENVI-Carb tubes is another product of our carbons program.

Investigators at Agriculture and Agri-Food Canada (Ottawa, Ontario) have developed a mult-residue cleanup and analysis for monitoring more than 200 organochlorine, organophosphorus, nitrogen-containing, and carbamate pesticides in fruits and vegetables. Their system allows quick screening of "rush" samples and thorough cleanup of complex samples. Initially, the extraction procedure required preparing and using minicolumns containing a mixture of charcoal and Celite® (1), but ENVI™-Carb carbon-based SPE tubes enabled these investigators to improve recovery rates and reproducibility for several pesticides. Subsequently, they replaced the charcoal/Celite minicolumns with ENVI-Carb tubes, and eliminated the labor-intensive minicolumn preparation process. Typical results are summarized in Table 1 – data for many additional pesticides are presented in free technical Bulletin 900, available on request.

For additional cleanup of polar and nonpolar interferences in complex samples, the investigators routinely include a C18 tube and an amino (NH_2) - phase tube in the procedure. In the Canadian group's sample-screening applications, the analysis for organochlorine, organophosphorus, and nitrogen-containing pesticides (approximately 210 compounds) is by capillary gas chromatography with mass-specific detection; residues are identified by retention time and ion ratios. Analysis for 10 carbamates is by HPLC with UV detection. Analytes monitored in each GC run (approximately 120 compounds and 90 compounds, respectively) are listed, along with recovery rates and limits of detection, in (1). Postcolumn derivatization and HPLC columns and conditions are described in (2).

Relative to liquid-liquid extraction or SPE on silica-based packings with octyl (C8) or octadecyl (C18) phases, ENVI-Carb packing provides more uniform recovery of a wide variety of polar nonvolatile analytes, while maintaining comparable results for less polar compounds. Because ENVI-Carb carbon is nonporous, samples can be processed rapidly – adsorption does not require dispersion of the analytes into porous regions. And, although the surface area of the carbon is smaller than that of porous silica ($100 m^2/g$, versus 400-$600 m^2/g$), the bed weight required for pesticide extractions typically is only half that needed with silica-based packings. The primary surface interaction mechanism is similar for ENVI-Carb packing and silica-based packings, but ENVI-Carb carbon also acts as a weak ion exchanger (3). This expands the range of analytes these tubes can be used to extract.

For details about the pesticide extraction procedure, request Bulletin 900.

Table 1. High Recovery of Pesticides from Fruits and Vegetables, Using ENVI-Carb SPE Tubes

Analyte	Mean % Recov.	Std. Dev.	% C.V.	Commodity / % Recovery Kiwi Fruit	Beet	Squash	Green Peas
Organochlorine Pesticides							
p,p'-DDT	96.6	9.1	9.4	102	99	102	79
Endosulfan sulfate	97.4	9.1	9.4	106	93	101	84
Endrin	95.0	5.5	5.8	88	98	100	91
Heptachlor	92.6	5.0	5.4	90	92	96	86
Lindane (γ-BHC)	96.1	5.0	5.2	98	90	99	92
Mirex	96.4	8.9	9.2	102	98	102	78
Methoxychlor	99.1	5.9	5.9	104	98	103	91
Nitrogen-Containing Pesticides / Triazines							
Atrazine	94.7	14.7	15.5	67	103	104	100
Cyanazine	96.7	6.5	6.7*	—	93	98	89
Cyprazine	95.7	6.8	7.1*	—	87	94	101
Prometon	89.6	10.4	11.6	75	87	96	99
Simazine	95.1	15.8	16.6	67	102	107	103
Organophosphorus Pesticides							
Chlorpyrifos	97.9	5.9	6.0	99	94	104	93
Diazinon	96.9	5.6	5.8	90	99	99	96
Dimethoate	99.3	4.6	4.6	105	96	99	100
Ethoprophos	92.0	9.8	10.7	85	83	91	91
Malathion	97.6	6.3	6.5	99	92	98	93
Methamidophos	64.9	8.3	12.7	57	59	57	65
Terbufos	89.0	8.7	9.8	76	84	97	89

*n = 6 extractions, all others n = 7.

Data provided by J. Fillion and J.C. Selwyn, Agriculture and Agri-Food Canada, Ottawa, Ontario, Canada.

Ordering Information:

Description	Cat. No.
ENVI-Carb Solid Phase Extraction Tubes	
3mL/250mg packing, pk. of 54	**57088**
6mL/250mg packing, pk. of 30	**57092**
6mL/500mg packing, pk. of 30	**57094**

References

1. Fillion, J, R. Hindle, M. Lacroix, and J. Selwyn, *Journal AOAC International*, **78:** 1252-1266 (1995).
2. Chaput, D., *J. Assoc. Off. Anal. Chem.*, **71:** 542-546 (1988).
3. Andreolini, F., *et al., Anal. Chem.* **59:** 1720 (1987).

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