DETERMINATION OF ORGANOCHLORINE PESTICIDES IN WASTEWATER USING EMPORE DISKS

Merlin K. L. Bicking, Fred L. DeRoos, Jean A. Osterheim, and Erin M. Dooley
TCT
737 Pelham Blvd.
St. Paul, MN, USA 55114

U.S. EPA Method 608 is a standard analytical procedure used for monitoring organochlorine pesticide concentrations in wastewater effluents. The extraction portion of the method utilizes either sequential or continuous liquid-liquid extraction with an organic solvent. Increasing concern about the use of large volumes of organic solvents, however, has resulted in efforts to develop alternative extraction approaches. Solid phase extraction (SPE) is one such alternative approach which has received considerable attention. Recently, a modification was introduced in which reverse phase (i.e., C_{18}-bonded silica) particles are supported by an inert Teflon™ membrane matrix. These membrane disks, marketed under the trade name "Empore", have been evaluated for their effectiveness in extracting a wide range of analyte classes from drinking water. The classes of analytes studied include phthalates, dioxins, semivolatile organic compound, and polycyclic aromatic hydrocarbons. The EPA has approved the use of the Empore disks for Methods 506, 513, 525, and 550.1.

The objective of this study was to evaluate Empore disks as an alternative to liquid-liquid extraction for determining organochlorine pesticides and PCBs in wastewater. To date, the disks have only been used for relatively "clean" samples containing few particulates or interferences (e.g., groundwater, drinking water). Wastewater samples represent a more significant challenge for the disks.

This presentation will summarize our efforts to develop this modification of EPA Method 608. Representative wastewater samples from several industries (POTW, pulp/paper, pesticide, and petroleum) were extracted using Empore extraction disks as well as liquid-liquid extraction. Analytes were then removed from the disks using a small volume of solvent. This design allowed a rigorous evaluation of two different approaches. The Empore extraction approach provided comparable results compared to conventional procedures, with a considerable reduction in solvent use (see Table 1).

TABLE 1. RECOVERIES FROM SPIKED POTW SAMPLES

Analyte	Spike Level, g/L	Recovery, percent (std. dev.) LLE[a]	Empore-Solvent[b]
gamma-BHC	1	86.3 (8.9)	84.3 (2.6)
Heptachlor	1	81.6 (8.8)	72.6 (8.9)
Aldrin	1	63.6 (6.6)	55.8 (5.9)
Heptachlor Epoxide	1	86.7 (6.1)	78.9 (4.7)
Endosulfan I	1	96.0 (6.9)	87.8 (4.9)
Dieldrin	2	93.1 (6.4)	82.5 (5.0)
Endosulfan II	2	82.1 (1.1)	74.3 (3.0)
Endrin Aldehyde	2	105 (5.8)	81.1 (5.8)
4,4'-DDT	2	82.9 (9.3)	80.6 (4.3)
alpha-BHC	1	83.6 (4.3)	82.9 (3.6)
beta-BHC	1	105 (9.0)	103 (6.8)
delta-BHC	1	114 (7.3)	116 (7.6)
alpha-Chlordane	2	69.9 (6.7)	85.9 (5.7)
DDE	2	83.1 (6.0)	81.8 (5.3)
Endrin	2	131 (7.2)	131 (6.1)
DDD	2	103 (15.4)	112 (12.5)
Endosulfan Sulfate	2	79.3 (6.5)	85.0 (4.0)
Endrin Ketone	2	78.8 (6.2)	82.6 (5.0)
Aroclor 1016-1	4	78.4 (3.0)	52.6 (4.2)
Aroclor 1016-2	4	93.8 (8.3)	79.6 (3.6)
Aroclor 1016-3	4	129 (3.9)	76.8 (4.8)
Aroclor 1016-4	4	101 (6.0)	90.5 (7.9)
Aroclor 1260-1	4	81.6 (1.5)	63.8 (12.8)
Aroclor 1260-2	4	97.5 (6.1)	65.0 (7.0)
Aroclor 1260-3	4	88.5 (11.3)	74.5 (14.9)
Aroclor 1260-4	4	95.4 (3.1)	84.8 (22.7)
Toxaphene-1	24	129 (8.6)	113 (5.9)
Toxaphene-2	24	126 (16.2)	116 (14.1)
Toxaphene-3	24	111 (11.1)	103 (8.1)
Toxaphene-4	24	102 (10.1)	95.2 (7.8)

[a] Liquid-liquid extraction (EPA Method 608): recovery values corrected for levels in blanks, n = 5.

[b] Method 608 using Empore disks, methylene chloride elution, recovery values corrected for levels in blanks, n = 5.