

DETERMINING SELECTED PERSISTENT ORGANIC POLLUTING PESTICIDES IN WATER USING AUTOMATED SPE

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Introduction:

The purpose of this investigation/method development was to establish a new extraction method using hexane as the extraction solvent for the determination of selected persistent organic polluting pesticides in aqueous samples ranging from the low ug/L ppb range, with a minimum of organic solvent consumption, while also conforming to the European Union standards. This was accomplished by optimizing a Solid Phase Extraction (SPE) method based on the Horizon Technology SPE-DEX® 4790 Automated Extractor System using a HLB-M extraction disk. The sample preparation step is an essential element of this method development, and as such, the advancement of an automated disk extraction has resulted in less solvent use, elimination of the solvent exchange step, reduced glassware use, faster extraction time with highly particulate samples, and more consistent and reproducible results.

Materials and Methods:

Instrumentation and materials used in this study consisted of Horizon Technology's SPE-DEX® 4790 Automated Extractors with an Envision Controller and Atlantic HLB-M SPE Disks for the sample preparation. Concentration of the extracts was done with the Horizon DryVap. The analysis was conducted with an Agilent 6890 GC with Dual ECD detectors with Restek columns: Primary- Rtx-CLP1, 30 m x 0.25 mm ID x 0.25 um film. Confirmation Column Rtx-CLP2, 30 m x 0.25 mm ID x 0.25 um film.

Method for Persistent Organic Polluting Pesticides:

- 1) 1L of acidified water <2.
- 2) Spike the samples with a pesticide standard and surrogates (varying concentrations 0.5ug/L-1.25ug/L).
- 3) Place the sample bottle on the SPE-DEX® 4790 Extractor System and place the HLB-M disk, in the disk holder.
- 4) Run the SPE-DEX® 4790 using the pesticides method using hexane as the extraction solvent.
- 5) After the extraction is complete collect the final elution 30mL.
- 6) Clean the extract using florisil.
- 7) Concentrate the sample down to 5.0 mL.
- 8) Analyze by GC/ECD. (Following GC Conditions)

Results and Discussion:

To show the efficiency of the automated spe extraction the spike was added to the DryVap concentrator to find out what typical recoveries would be in an ideal situation. Two concentrator tubes were spiked with 20 ug of pesticide standard into 25mL of solvent. The results showed on Table 1 are excellent for all of the persistent organic polluting pesticides.

With the sample extracted with hexane and not dichloromethane an entire step solvent exchanging and concentrating has been totally eliminated. The results in Table 2 show that hexane as the extraction solvent resulted in high recoveries on the primary column along with the confirmation column with runtimes of 10 minutes on the GC/ECD. By using hexane as the extraction solvent it not was only more convenient with this method, but results were not sacrificed to meet the European Union's requirement as well.

Table 1: Recoveries of Persistent Organic Polluting Pesticides with the DryVap

Compounds	Average Recovery%
Aldrin	93
DDT	95
Dieldrin	95
Endrin	96
Heptachlor	87
4, 4'DDE	95
4, 4'DDD	96
Endrin Aldehyde	99
Endrin Ketone	98

Table2: Recoveries of Persistent Organic Polluting Pesticides in Water

Compound	Concentration ug/L	Primary Column CLP 1	Secondary Column CLP 2
		Recovery%	Recovery%
Aldrin	0.50	91	80
DDT	1.25	90	86
Dieldrin	1.25	95	78
Endrin	1.25	92	81
Heptachlor	0.50	91	82
4,4'DDE	1.25	97	85
4,4'DDD	1.25	97	86
Endrin Aldehyde	1.25	89	81
Endrin Ketone	1.25	97	86
Tcmx	1.00	81	76
DCB	1.00	88	76

The study performed using automated solid phase extraction with a disk achieves high recoveries of persistent organic polluting pesticides in water samples. When using the HLB-M disk and pre-filters the entire process duration is 25 to 45 minutes depending on vacuum settings and the amount of particulate matter in the sample. The concentration step at a final volume of 5mL with a typical 30mL sample extract will take 10 minutes. Coupled with the short run times on the GC/ECD samples were extracted concentrated cleaned and analyzed within 2 hours with great recoveries.