Application Note

Automated, One-Step SPE and Concentration of Nitrogen and Phosphorus Containing Pesticides in Water

Introduction

Nitrogen and phosphorus containing pesticides are being widely used as replacements for organochlorine pesticides. Initially considered more desirable than organochlorine pesticides because of the way they rapidly break down in the environment, they inhibit the enzyme acetylcholine-sterase and disrupt nerve function. Nitrogen and phosphorus containing pesticides, particularly organophosphates, have proven to be extremely toxic to vertebrates.

Despite their ability to breakdown rapidly, nitrogen and phosphorus containing pesticides constitute a major health concern due to their extreme toxicity and their tendency to persist in drinking water. The following procedure outlines the use of Solid Phase Extraction (SPE) to extract phosphorus and nitrogen containing pesticides for analysis by GC via EPA Methods 507, 614, 1657 and 8141.

Instrumentation & Consumables

FMS, Inc. PowerPrep™ SPE System

FMS, Inc. SuperVap™ Concentrator

FMS, Direct-to-Vial concentrator tubes

Waters, 1 gm Oasis HLB® Cartridge

Varian Chem Elut Hydromatrix® cartridge

Thermo Trace GC w/Polaris QMS

PowerPrep SPE

The HLB Cartridge is conditioned with 10 mL methanol

The HLB Cartridge is conditioned with 10 mL DI H_2O

Sample is loaded across HLB Cartridge via vacuum

The sample bottle is auto-rinsed and rinse loaded on HLB

The cartridge is dried with nitrogen

The cartridge is eluted with ethyl acetate followed by methylene chloride, passed across a Chem Elut Hydromatrix® cartridge and eluted directly into the Direct-to-Vial FMS SuperVap concentrator.

SuperVap Concentrator

Pre-heat temp: 55 °C
Pre-heat time: 30 minutes
Heat in Sensor mode: 55 °C
Nitrogen Pressure: 15 PSI

Procedure: Sample Prep and Extraction

1. Two, 1 liter water samples spiked using Ultra NPM-525C & NPM 525B Nitrogen/Phosphorus Pesticide Mix (Diluted).
2. The samples are acidified to PH <2.
3. Samples are equilibrated for 15 minutes.
4. Two samples are loaded onto to corresponding sample positions on the FMS PowerPrep SPE system.
5. The Program is initiated to run each sample sequentially.
6. The sample is extracted and automatically transferred to the FMS SuperVap Concentrator system with Direct-to-Vial tubes.
7. Extracts are concentrated with the SuperVap system to <1 mL.
8. Extracts are removed from the SuperVap system, volume adjusted to 1 mL, internal standard added (Ultra ISM-510) and transferred to Agilent GC for analysis.

Figure 1: PowerPrep SPE and SuperVap Concentrator systems.

FMS
Fluid Management Systems

Results

Compound	Position #1	Position #2	Mean Rec	RSD
Alachlor	4.72	4.61	93.3%	2.4%
Ametryn	4.46	4.5	89.6%	0.9%
Atraton	4.59	4.26	88.5%	7.5%
Atrazine	4.49	4.42	89.1%	1.6%
Bromacil	4.16	5.07	92.5%	19.2%
Butachlor	4.08	4.09	81.7%	0.2%
Butylate	3.62	3.54	71.6%	2.2%
Carboxin	4.3	3.64	79.4%	16.6%
Chlorophram	4.61	4.17	87.8%	10.0%
Cycloate	4.11	3.9	80.1%	5.2%
Dichlorvos	4.23	3.91	81.4%	7.9%
Diphenamid	4.13	4.55	86.8%	-9.7%
Disulfoton*	4.03	3.32	73.5%	19.3%
EPTC	3.74	3.41	71.5%	9.2%
Ethoprop	4.64	4.44	90.8%	4.4%
Fenamiphos	4.21	5.07	92.8%	18.5%
Fenarimol	5.27	4.82	100.9%	8.9%
Fluridone	5.51	5.06	105.7%	8.5%
Hexazinone	4.86	4.69	95.5%	3.6%
Methyl Paraoxon	5.35	5.56	109.1%	-3.8%
Metolachlor	4.5	4.41	89.1%	2.0%
Mevinphos	4.57	4.38	89.5%	4.2%
MGK 264	4.13	3.59	77.2%	14.0%
Molinate	4.24	3.97	82.1%	6.6%
Napropamide	4.49	4.28	87.7%	4.8%
Norflurazon	4.74	4.67	94.1%	1.5%
Pebulate	4.07	3.52	75.9%	14.5%
Prometon	4.16	4.3	84.6%	-3.3%
Prometryn	4.4	4.57	89.7%	-3.8%
Pronamide**	4.46	4.36	88.2%	2.3%
Propazine	4.72	4.55	92.7%	3.7%
Simazine	4.2	4.24	84.4%	-0.9%
Simetryn	4.79	4.73	95.2%	1.3%
Stirofos	4.33	4.4	87.3%	-1.6%
Tebuthiuron	4.36	4.18	85.4%	4.2%
Terbacil	4.81	5.33	101.4%	10.3%
Terbufos**	3.58	3.51	70.9%	2.0%
Terbutryn	4.43	4.82	92.5%	-8.4%
Triademefon	5.69	4.82	105.1%	16.6%
Tricyclazole	5.25	4.95	102.0%	5.9%
Vemolate	3.69	3.529	72.2%	4.5%

Conclusions

Analysis of duplicate samples across the automated, one-step PowerPrep SPE Direct-to-Vial Concentration system demonstrates highly efficient extractions with excellent recoveries and reproducibility. Watch the product video.

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