

Automated Multi-Cartridge Solid Phase Extraction of Phosphorus Containing Pesticides in Drinking Water

Introduction

Organophosphate pesticides (OPPs) are widely used globally for agricultural pest control. Unlike chlorinated pesticides (OCPs), OPPs are less persistent in the environment but offer greater acute toxicity. Additionally, OPPs have a higher water solubility which makes them of particular concern in municipal drinking water supplies.

Analytically, OPPs can pose some challenges due to the wide diversity of physical structures and behavioral characteristics of individual analytes. This often results in individual extraction protocols being implemented for labs to meet regulatory compliances. The FMS Inc TurboTrace® ABN Multi Cartridge SPE system, and its ability to incorporate dual cartridge extractions, it is possible to pair analytes with dissimilar characteristics into a single, fully automated extraction process.

- FMS, Inc. TurboTrace® ABN SPE system
- FMS, Inc. SuperVap® 12 Concentrator
- FMS, Inc. 50ml direct to vial concentrator tubes
- Thermo Trace Ultra GC with Polaris Q
- Agilent 7890 GC with FPD detector

Consumables

- FMS, Inc. 1 gram C18 cartridges
- Restek Resprep 2 gram coconut charcoal cartridges (Cat# 26032)

Sample Prep

1. 1 Liter drinking Water Samples were spiked with target analytes of interest.
2. 1 ml of 6N HCl is added to each sample to bring the pH <2.
3. Sample Bottles are fitted onto SPE extraction system
4. 1 gram DVB cartridges (position #1) and coconut charcoal cartridges (position #2) fitted on SPE system.

SPE Procedure

1. SPE cartridges are pre-conditioned with 5 mls MeCL each
2. SPE Cartridges are pre-conditioned with 10 mls MeOH each
3. SPE Cartridges are pre-conditioned with 20 mls H₂O each
4. Samples are loaded across cartridge at a 20 ml/min rate via vacuum
5. SPE cartridges are Nitrogen dried for 10 minutes to remove excess water.
6. DVB cartridge eluted with 5 mls EtAC.
7. Sample containers are rinsed with 15mls MeCL and eluted through DVB cartridge (10ml/min).
8. 5 mls additional MeCL eluted through DVB Cartridge.
9. Coconut charcoal cartridges eluted with 15mls MeCL.
10. Residual water removed by NaSO₄ in-line cartridge filtration and eluate emptied directly into Super Vap concentrator tubes.

SuperVap

1. Preheat temp: 20 minutes at 60 °C
2. Evap mode w/Sensor temp: 60 °C
3. Nitrogen Pressure: 10 PSI
4. Samples reduced to 1ml
5. Samples reduced to 1ml final volume



Results

<u>Analyte</u>	<u>Mean Recoveries</u>		<u>Dev</u>
	<u>FPD</u>	<u>MS-MS</u>	
Methamidophos	63	67	2.7
Monocrotophos	96	91	3.8
Diazinon	112	97	14.6
Parathion	99	113	7.3
EPN	85	103	16.8

Results run in triplicate by both GC/Ion Trap and GC/FPD

Conclusions

Extraction of the drinking water samples on the Turbo Trace ABN system enabled a fully automated extraction process due to its dual cartridge capabilities. Samples were loaded simultaneously across the C18 and coconut charcoal cartridges in succession. Cartridges were then able to be eluted independently with no manual transferring or handling of cartridges. Elutions then could be either independently fractionated for individual analysis, or combined for a single GC run by utilizing the systems 3 fraction lines.

Analysis of water extracts showed consistent instrumental performance by both GC/MS-MS and GC/FPD detection. Recoveries for C18 bonding pesticides displayed excellent precision with all falling between 80—120%. Methamidophos consistently recovered >60% from the coconut charcoal cartridge when using 1 liter samples. Higher recoveries were observed when using reduced sample volumes indicating 2 gram coconut charcoal cartridges were better suited for samples ≤500mls.



FMS Inc. Turbo Trace ABN Multi cartridge system

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