



Determination of Pesticides in Drinking Water via LC-MS/MS Using Polymeric Solid Phase Extraction Cartridges

Application Note ENV0113

Keywords

SPE (Solid Phase Extraction), Pesticide, Herbicide, Fungicide, Pharmaceuticals, Drinking Water, WHO (World Health Organization), National Standards

Introduction

WHO issues guidelines to assist with developing national standards for safe drinking water that does not pose a health or disease risk in developed or developing countries.¹ Drinking water safety is of primary concern in reducing the harmful impact of residues, such as pesticides, herbicides, fungicides, and pharmaceuticals on both the human and animal populations. This application studied a wide range of residues in drinking water using two different SPE polymeric cartridges. Recovery results were compared.

Materials & Methods

Materials

- Solid Phase Extraction Cartridges:
 - ASPEC™ HLB 6 mL/200 mg
 - Gilson PN: 54350564

Solid Phase Extraction Steps

1. **Condition 1:** 6 mL of MeOH@ 10 mL/min
2. **Condition 2:** 5 mL of H₂O @ 5 mL/min
3. **Load:** 100 mL of drinking water @ 5 mL/min
4. **Wash:** 6 mL of H₂O @ 10 mL/min
5. **Elute 1:** 6 mL of MeOH@ 5 mL/min
6. **Elute 2:** 6 mL of MeOH@ 10 mL/min



Sample Reconstitution

- Sample fractions were evaporated at 50°C for 10 minutes with nitrogen
- Each evaporated fraction was reconstituted with 3 mL of MeOH

Chromatographic Conditions

- **Mobile Phase:** 0.800 mL/min
 - **A:** 1mM ammonium formate in (10/90) MeOH/H₂O, 0.1% formic acid (v/v)
 - **B:** 1mM ammonium formate in (90/10) MeOH/H₂O, 0.1% formic acid (v/v)

GRADIENT & MRM TRANSITION		
Time (min)	MPA (%)	MPB (%)
0.00	100	0
10.00	0	100
10.01	100	0
12.00	100	0
Analyte	MRM Transition	
Atrazine	216.1 -> 174.1	
Benalaxyl	326.2 -> 148.2	
Carbendazim	192.1 -> 160.2	
Chloroxuron	291.1 -> 72.0	
Imazalil	297.0 -> 159.1	
Methalaxyl	280.2 -> 220.3	
Myclobutanil	289.1 -> 70.0	
Propoxur	210.1 -> 111.2	
Simazine	202.1 -> 174.2	
Thiambadazole	202.2 -> 175.1	

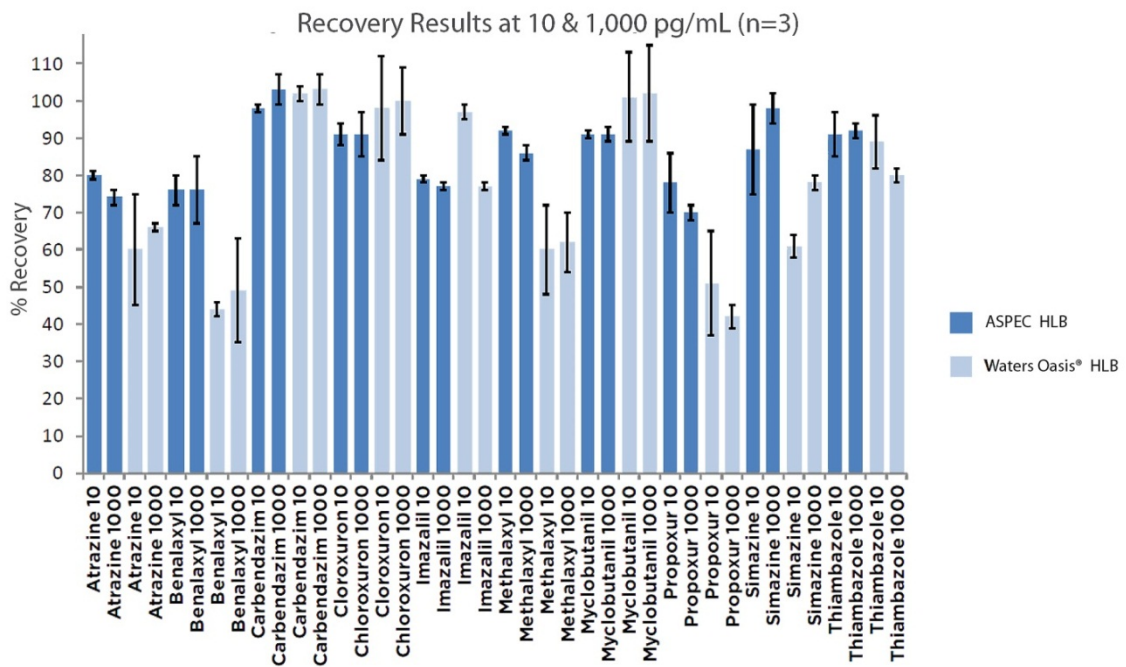
- **Column:** 3.0 x 30 mm C18, 2.5 µm @ 23 °C
- **MS Splitting Flow:** 0.30 mL/min
- **Detector:** Sciex API 3000
 - **Turbo Ion Spray Heater Gas Flow:** 8000 cc/min
 - **Turbo Ion Spray Heater Temperature:** 375 °C, ESI⁺, MRM
- **Injection Volume:** 5 µL



Results

LC-MS/MS recovery results from this multi-pesticide drinking water application show that recoveries for each of the pesticides vary with both SPE cartridges used; however, more consistent results and less recovery variation were obtained from samples that used the ASPEC HLB cartridges for the SPE clean-up and concentration. Detection at 10 and 1,000 pg/mL was more consistent with the ASPEC HPLC cartridges (Figure 1).

Figure 1: Pesticide Recovery Results from Drinking Water Samples



Conclusion

LC-MS/MS analysis provided high selectivity with a large class of pesticides in drinking water. Recovery values are most consistent and show less variation at 10 and 1,000 pg/mL when the Gilson ASPEC HLB cartridges are used for the SPE clean-up and concentration.



References

1. World Health Organization (2006). Guidelines for Drinking-water Quality.
http://www.google.com/url?sa=t&rct=j&q=spe%20drinking%20water&source=web&cd=5&ved=0CEUQFjAE&url=http%3A%2F%2Fwww.who.int%2Fwater_sanitation_health%2Fdwq%2Fgdwq0506.pdf&ei=U-XyUZ_0HITW9QSI74DoAw&usg=AFQjCNENElu7NUkCm3ZktNaunwBXLx9a-w