DIONEX

Application Note 810

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US EPA Method 527 — Determination of Pesticides and Flame Retardants in Drinking Water

INTRODUCTION

This method was developed for the automated extraction of pesticides and flame retardants as described in EPA Method 527. The analytes are determined by capillary column gas chromatography/mass spectrometry (GC/MS).

METHOD SUMMARY

5 mL of methanol is added to one liter of sample and spiked with 10 µL of 500 µg/mL surrogate solution. The samples are passed through a 47 mm Polystyrenedivinylbenzene (SDVB) SPE disk. After elution with ethyl acetate and dichloromethane, the extract is concentrated down to a final volume of 1.0 mL. Extracts were fortified with 10 µL of 500 µg/L internal standard solution and analyzed using a GC/MS in accordance with EPA Method 527.

INSTRUMENTATION USED FOR SAMPLE PREPARATION

Dionex AutoTrace® instrument

EQUIPMENT SPECIFICATIONS AND OPERATING CONDITIONS

Sample evaporated as follows:
- Solvent starting volume: 25 mL
- Solvent end volume: 1.0 mL
- Solvent: Ethyl acetate/dichloromethane
- Bath temperature: 30 ºC

CG/MS or MSD:
- Column: Restek Rxi-5MS 30 M column, 0.25 mm ID, 0.25 µm df
- Initial Temp: 80 ºC for 1.0 min
- Ramp: 10.0 ºC/min
- Final Temp: 320 ºC for 2.0 min
- Inlet Temp: 250 ºC

SPE Disk Used:
- 47 mm Polystyrenedivinylbenzene (SDVB) SPE disk

AutoTrace Instrument Procedure

Process the six samples using the following procedure:
Step 1: Condition disk with 5.0 mL of 1:1 EtAC/$MeCl_2$ into solvent waste.
Step 2: Pause for 1 min.
Step 3: Dry disk with gas for 1 min.
Step 4: Condition disk with 10.0 mL of CH_3OH into solvent waste.
Step 5: Pause for 1 min.
Step 6: Condition disk twice with 10.0 mL of reagent water into aqueous waste.
Step 7: Load 1000 mL of sample onto disk.
Step 8: Dry disk with gas for 9.0 min.
Step 9: Manually rinse sample container twice with 5.0 mL to collect (EtAc).
Step 10: Manually rinse sample container with 5.0 mL to collect ($MeCl_2$).
Step 11: Soak and collect 5.0 mL fraction to a second tube using 1:1 EtAc/$MeCl_2$.
Step 12: Collect 5.0 mL fraction into sample tube using 1:1 EtAc/$MeCl_2$.

Solvent 1:	1:1 EtAc/$MeCl_2$	Cond Flow:	10 mL/min
Solvent 2:	CH_3OH	Load Flow:	15 mL/min
Solvent 3:	Water	Rinse Flow:	20.0 mL/min
Solvent 4:	$MeCl_2$ (Dichloromethane)	Elute Flow:	5.0 mL/min
Solvent 5:	EtAc (Ethyl acetate)	Cond Air Push:	15.0 mL/min
		Rinse Air Push:	20.0 mL/min
		Elute Air Push:	5.0 mL/min

RESULTS

The following table shows recoveries of selected compounds.

Compound	Mean (ug/L) n=4	Accuracy Mean Rec (%)	Precision/% RSD
Dimethoate	2.11	105.3	7.07
Terbufos-Sulfone	1.94	97.0	5.94
BDE-47	0.81	80.8	3.83
BDE-100	0.81	81.3	5.06
BDE-99	0.88	88.3	1.94
HBB	0.85	86.0	4.55
DBE-153	0.92	92.3	3.58

BDE – Bromodiphenyl Ether
HBB – Hexabromobiphenyl

CONCLUSION

Sample bottles were chilled during extraction to ensure the high recovery of dimethoate. Due to the nature of the BDEs and some surrogates, the sample bottle rinsing steps after sample loading are very important. Automation of the extraction with the AutoTrace instrument ensures consistency throughout the extraction batches.

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