Application Note

Analysis of Benzidines and Nitrogen-Containing Pesticides in Drinking Water Using EPA Method 553 with Automated Solid Phase Extraction (FMS TurboTrace®)

Introduction

Nitrogenous pesticides and benzidines are easily leached into the water table, due to their high solubility. They have been linked to incidences of bladder and pancreatic cancer.

EPA Method 553 outlines the procedure for the extraction and analysis of these compounds in drinking water. The extraction method outlines the use of solid phase extraction for water matrix samples employing both cartridges and disks. Consistent with other EPA 500 series methods, EPA 553 incorporates a rigid set of QC and acceptance criteria requiring precise and reproducible analytical practices. The potential for error and the variability associated with manual extractions makes the benefits of automating these processes apparent. Hence, FMS developed a simple automated system which is fast, inexpensive and yields high quality data.

Instrumentation

- FMS TurboTrace® System
- FMS SuperVap®
- Vacuum pump
- Waters Alliance 2695 HPLC, UV254

Consumables

- FMS, Inc. 500 mg C-18 cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Fisher 6 N Hydrochloric Acid or Sodium Hydroxide
- Fisher pesticide-grade methanol
- Ammonium acetate buffer
- Restek 553 spiking standards

Procedure

- 6 samples (1 L water each) are prepared and adjusted to a pH of 7.0
- Ammonium acetate buffer (0.01 M) is added
- Spike with various 553 standards
- Put sample bottles in place and fill rinse bottles with 7.5 mL methanol
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 2 x 10 mL methanol then 2 x10 mL water (kept wet after each solvent)
- Samples are loaded across cartridges under vacuum
- Cartridges are dried under vacuum for 10 min (no nitrogen)
- Sample bottles are automatically rinsed from the rinse bottles with 7.5 mL methanol
- Rinse bottles are refilled and the above step is repeated

Stage 2:

- Methanol from sample bottles is loaded across the cartridges (15 mL, total) and the eluent is collected for analysis

FMS SuperVap®

- Pre-heat temp: 45 ºC
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 45 ºC under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

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Table 1 with recoveries for the 553 compounds

Compound	%Recovery	Stdev
Benzidine	93.6	2.94
Benzoyl prop ethyl	101.5	1.36
Caffeine	84.5	1.85
Carbaryl	77.2	3.84
o-chlorophenyl thiourea	94.9	2.37
3,3'-dichlorobenzidine	103.7	1.46
3,3'-dimethoxybenzidine	100.4	4.70
3,3'-dimethylbenzidine	85.3	2.65
Diuron	90.1	1.99
Ethylene thiourea	86.2	1.05
Linuron (Lorox)	96.8	3.74
Monuron	79.5	2.17
Rotenone	102.6	3.11
Siduron	83.3	3.58

Conclusions

Reviewing the sample data shows high recoveries for fourteen spiked analytes, demonstrating excellent efficiency for these compounds. Samples can be taken from collection bottle to LC vial in one quick, consistent, reproducible process that will save laboratories both time and money.

FMS TurboTrace® System

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