Application Note

Analysis of Phenylurea Compounds in Drinking Water Using EPA Method 532 with Semi-Automated Solid Phase Extraction (EZSpe®)

Introduction

Phenyl urea compounds are used for weed control. They are notoriously toxic to amphibians and fish, when they seep into the water table. EPA Method 532 outlines the procedure for extraction and analysis of these compounds in 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 532 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 semi-automating these processes apparent.

To meet demands for a low cost method that requires less financial investment than fully automated systems, FMS developed a simple semi - automated system which is fast, inexpensive, and yields high quality data.

Instrumentation

- FMS EZSpe® System
- FMS SuperVap®
- Vacuum pump
- Waters Acquity UPLC w/ Xevo TQD MSMS

Consumables

- FMS, Inc. 500 mg C-18 cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Fisher Pesticide Grade Methanol
- Restek 532 spiking standards

Procedure

- 6 samples (500 mL water each) are prepared
- Spike with various 532 standards
- Put sample bottles in place and fill rinse bottles with 6 mL methanol
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 2 x 5 mL methanol (soak 30 seconds, keep wet) and 3 x 5 mL water (soak 30 seconds, keep wet).
- Samples are loaded across cartridges under vacuum at 20 mL/min.
- Cartridges are dried under vacuum for 15 minutes.
- Sample bottles are automatically rinsed from the rinse bottles, with 6 mL methanol.

Stage 2:

- Methanol from sample bottles is loaded across the cartridges (3 ml then 2 mL then 1 mL, soak 30 seconds per elution) and the eluent is collected for analysis into Direct to LC Vial Collection Vessels

FMS SuperVap®

- Pre-heat temp: 40 ºC
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 40 ºC under nitrogen (7-10 psi)
- Direct to LC Vial Vessel Reduce to 0.5 mL
- Samples are now ready for analysis

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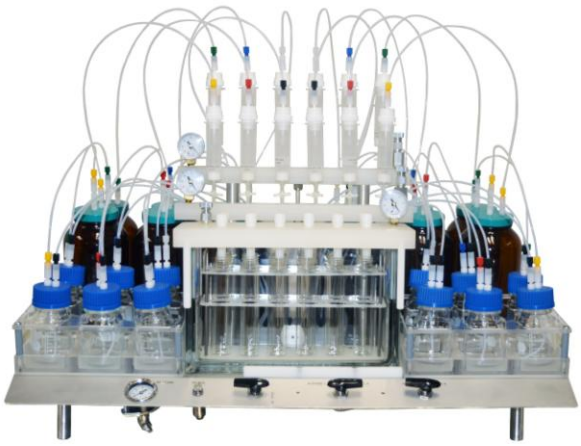
Table 1 with recoveries for the eight 532 compounds

Compound	%Recovery	Stdev
Diflubenzuron	78.9	3.2
Diuron	85.8	2.5
Fluometuron	92.4	1.9
Linuron	83.1	4.4
Propanil	104.2	3.8
Siduron	93.9	5.1
Tebuthiuron	80.2	6.3
Thidiazuron	96.5	3.1

Conclusions

Reviewing the sample data shows high recoveries and precision for several phenylurea analytes, demonstrating excellent efficiency for these compounds. Samples can be taken from collecting flasks to LC vials in one quick, consistent, reproducible process that will save laboratories both time and money.



FMS EZSpe® system

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