Application Note

One-Step Extraction and Concentration for Identifying Pharmaceuticals and Personal Care Products in Water, Biosolids and Solids

Introduction

Over the last decade the use of Pharmaceuticals and Personal Care Products (PPCPs) has doubled in the United States. As a result, PPCPs have entered the environment through both human activity and as by-products from manufacturing, agricultural activities, medical use and veterinarian facilities. PPCPs are usually introduced into the environment through the disposal of unused medications into sewer systems and trash. PPCPs tend to be water soluble and do not evaporate under normal temperatures, which is why they end up in soil and water. The full effect of PPCPs on the environment is not fully understood and there is concern about the potential threat they pose to the food chain. Because of the high solubility of most PPCPs, aquatic organisms are most vulnerable. The classes of pharmaceuticals found in these organisms have been linked to slow growth in frogs and the increased feminization of exposed fish. The scope of human exposure to PPCPs from the environment is complicated and increased monitoring is occurring to determine the effect on humans of long-term, low-level exposure to PPCPs.

Due to their persistent nature and toxicity, monitoring water sources for PPCPs is a growing priority for both government agencies and consumers. The following procedure outlines the fully automated, sample-to-vial extraction and concentration of water matrices for the detection of these compounds in one rapid and efficient process.

Instrumentation and Consumables

FMS, Inc. PowerPrep™ SPE system (Solid Phase Extraction)

FMS, Inc. SuperVap™ Concentrator

FMS, Direct-to-Vial concentrator tubes

1 gram Waters Oasis™ HLB cartridge

UPLC, LC/MS.

Procedure: Sample Prep Extraction and Concentration

PowerPrep SPE

1. Condition the cartridge with 10 mL of methanol
2. Condition the cartridge with 10 mL of water
3. Load the 1 liter water sample at 100 mL/min
4. Rinse the cartridge
5. Dry the cartridge with nitrogen and vacuum for 20 minutes
6. Elute the cartridge with 15 mL of methanol base fraction
7. Elute the cartridge with 15 mL of methanol 2% formic acid
8. The fractions are directly eluted to the SuperVap Concentrator system

SuperVap Direct-to-Vial Concentration

Pre-heat temp: 40 °C.

Pre-heat time: 10 minutes

Heat in sensor mode: 50 °C

Nitrogen pressure: 15 PSI

Sensor 1 mL Direct to GC vial

Figure 1: PowerPrep SPE and SuperVap Concentrator systems.

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Fluid Management Systems

Results

Table 1 shows the mean recoveries from the five extracts after analysis from several types of PPCPs in water.

Compound	Average Recovery
Atenolol	88%
Atorvastatin	81%
Avobenzone - A	97%
Avobenzone - B	92%
Ciprofloxacin	99%
Benzophenone-1	98%
Benzophenone-3	94%
DEET	90%
4,4-Dihydroxybenzophenone	86%
Estradiol	81%
Estrone	84%
Naproxen	95%
Methylparaben	85%
Propanolol	80%
Ranitidine	99%
Sulfamethoxazole	98%
Sucralose	97%
TCEP	86%
Trimethoprim	83%
Thiabendazole	92%
Warfarin	87%
Xanthine	92%

Conclusions

Analysis of the LC/MS data demonstrates excellent recoveries and reproducibility from a traditionally difficult sample matrix. Adding to the efficiency was the use of nitrogen and vacuum to dry the cartridge and a water free extract that enables a fast concentration step with no loss of analytes. The extract takes 45 minutes to concentrate using the PowerPrep™ SPE system compared to all other drying methods. Using the automated, one-step SPE and Direct-to-Vial Concentration tubes from FMS, Inc. eliminates error-prone manual or semi-automated steps from the sample prep process. No sample transfer is necessary, which allows the sample to be extracted and automatically sent to the SuperVap Concentrator where the final extract is concentrated directly to a vial for LC/MS analysis. This capability eliminates human error, saves time and increases efficiency while producing reproducible, consistent recoveries.

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