

EPA 542 One-Step Extraction and Concentration for Identifying Pharmaceuticals and Personal Care Products in Drinking Water



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 effects of PPCPs on the environment are not fully understood and there is concern about the potential threat they pose to the food chain. 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 following EPA method 542.

Instrumentation and Consumables

FMS, Inc. TurboTrace[®] 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: Extraction and Concentration TurboTrace SPE

1. Prepare 5 water samples 1 L each and spike with standards
2. Condition the cartridges twice with 5 mL of 1:1 acetone: methanol
3. Repeat with 2.5 mL twice of same mix (keep wet)
4. Condition twice with 2.5 mL DI water
5. Load the 1 liter water sample at 10 mL/min
6. Rinse sample bottles with 10 mL DI water and load onto cartridges
7. Dry the cartridges with nitrogen and vacuum till no water is visible (no complete dryness necessary)
8. Elute the cartridges with 2 x 2.5 mL 1:1 acetone: methanol and collect (soak 1 min for first aliquot)
9. 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: 40 °C

Nitrogen pressure: 7-10 PSI

Sensor 1 mL Direct to GC vial



Figure 1: TurboTrace SPE and SuperVap Concentrator systems.



Results

Table 1 shows the mean recoveries from the five extracts after analysis from several types of PPCPs in Water (spiked at 5-50 ng/L).

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 TurboTrace 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 was necessary, which allowed 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 eliminated human error, saved time and increased efficiency while yielding reproducible and consistent recoveries.

For more information contact FMS:
 FMS Inc.
 580 pleasant Street
 Watertown, MA 02472
 Phone: (617) 393-2396
 Fax: (617) 393-0194
 Email: onlineinfo@fms-inc.com
 Web site: www.fms-inc.com