PROMOCHROM TECHNOLOGIES

Application Note
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EPA Method 1634

AUTOMATED SOLID PHASE EXTRACTION OF 6PPD-QUINONE FOLLOWING DRAFT EPA METHOD 1634

Authors

Ian Wan, PromoChrom Technologies, Richmond, BC, Canada

Keywords

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ABSTRACT

N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD) is an additive used in tires to enhance durability by protecting against environmental degradation. Under certain conditions, including friction, ground-level ozone, and UV exposure, 6PPD transforms into 6PPD-Quinone (6PPD-Q). This transformation product has drawn attention due to its release through tire wear particles and its toxicity to aquatic species. Given its environmental persistence and potential ecological risks, 6PPD-Q has become a growing concern for scientists and regulatory agencies, emphasizing the need for further research and mitigation efforts.

INTRODUCTION

To support tribes and local governments in monitoring 6PPD-Q in water, the EPA released draft EPA Method 1634 in January 2024. This method employs reverse-phase SPE for sample extraction, followed by LC/MS/MS analysis.

In an effort to enhance testing efficiency, PromoChrom has developed its own SPE cartridge to be utilized on the SPE-03 automated extractor. This application note demonstrates how the packaged solution successfully extracts 6PPD-Q from river water samples following draft EPA Method 1633.

MATERIALS

- PromoChrom SPE-03 with MOD-005 (Minimal-Teflon configuration) and MOD-00P (Volume-Matrix Plus configuration)
- PromoChrom Polymeric Reverse Phase cartridge (Cat. No. C-6PPD-6mL-200mg)
- Reagents and standards following draft EPA Method 1634
- LCMSMS

METHOD SUMMARY

SPE Method

Solvent 1 = Acetonitrile, **Solvent 2** = H_2O, **Solvent 3** = 1:1 Methanol/ H_2O,
W1 = Aqueous waste, **W2** = Organic waste

Table 1 – 6PPD-Q extraction steps programmed on the SPE-03.

Action	Inlet 1	Flow	Volume	*Description*
Elute W2	Solvent 1	5 mL/min	5 mL	*Condition cartridges with 5mL Acetonitrile*
Elute W1	Solvent 2	10 mL/min	10 mL	*Condition cartridges with 10mL Water*
Add Sample W1	Sample	15 mL/min	285 mL	*Load samples at 15mL/min*
Rinse	Solvent 3	70 mL/min	5 mL	*Rinse bottles with 5mL 1:1 Methanol/ H_2O*
Air-Purge R	Air	70 mL/min	3 mL	*Purge rinse lines*
Add Samp S	Sample	20 mL/min	10 mL	*Oscillate rinsate to recover any trapped analytes if inline filter is used*
Add Samp W1	Sample	10 mL/min	10 mL	*Deliver all rinsate through cartridges*
Air-Purge W1	Air	10mL/min	5 mL	*Purge large water droplets out of cartridges*
Blow N2	Time based		5 mins	*Dry cartridges with nitrogen for 5 mins*
Rinse	Solvent 1	70 mL/min	5 mL	*Rinse bottles with 5mL Acetonitrile*
Add Samp S	Sample	20 mL/min	10 mL	*Oscillate rinsate to recover any trapped analytes if inline filter is used*
Collect 1	Sample	5 mL/min	5 mL	*Collect rinsate through the cartridges into fraction 1*
Rinse	Solvent 1	70mL/min	4 mL	*Rinse bottles with 4mL Acetonitrile*
Air-Purge R	Air	70 mL/min	3 mL	*Purge rinse lines*
Add Samp S	Sample	20 mL/min	10 mL	*Oscillate rinsate to recover any trapped analytes if inline filter is used*
Collect 1	Sample	5mL/min	10mL	*Collect rinsate through the cartridges into fraction 1*

The extraction takes just 75 minutes to complete for 8 x 250 mL samples.

RESULTS

8 x 250 mL river water samples, with an example shown on the right, were spiked with the native 6PPD-Q analyte at 400 ng/L. The extracted internal standard, 13C6-6PPD-QCIL, is not added to demonstrate that good recoveries can also be achieved directly.

The SPE-03 system with PromoChrom's reverse phase cartridges demonstrated excellent recoveries and consistency. Average recoveries and %RSD are well within the interim acceptance criteria of ±30% from the true value for the mean recovery and <30% for RSD.

Table 2 - Precision and Accuracy Results of 6PPD-Q

Pos 1	Pos 2	Pos 3	Pos 4	Pos 5	Pos 6	Pos 7	Pos 8	Average	%RSD
91.9%	91.3%	91.9%	90.2%	94.2%	87.3%	91.9%	89.9%	91.07%	2.21%

CONCLUSIONS

PromoChrom's SPE-03 system coupled with reverse phase cartridges offer a quick and effective solution for the extraction of 6PPD-Q from water. Aside from achieving excellent recoveries, it provides high efficiency by fully automating the extraction process for 8 samples in parallel. The results were obtained on a PFAS-configured system with MOD-005 Minimal-Teflon option, demonstrating that labs can perform EPA Method 1634 and PFAS methods on the same platform.

RECOMMENDED SOLUTION

Part No.	Description	Notes
SPE-03	8-Channel SPE-03 System	Automated SPE System https://www.promochrom.com/spe-03
MOD-00P	Volume-Matrix Plus configuration	For automatic rinsing of up to 1L sample containers and handling of samples with particulates https://www.promochrom.com/spe-03
C-6PPD-6mL-200mg	Aquaris™ SPE cartridge, 6mL, 200mg, Reverse Phase sorbent	To extract 6PPD-Q from aqueous samples https://www.promochrom.com/spe-cartridges
F-HC-30	High-Capacity Inline Filter	To enable the extraction of samples with particulates https://www.promochrom.com/inline-filters
F-T-M	Anti-clogging Tip	To handle large sample sediments https://www.promochrom.com/anti-clogging-tips

References

1. EPA Method 1634
   https://www.epa.gov/system/files/documents/2024-01/draft-method-1634-for-web-posting-1-23-24_508.pdf

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