Automated Solid Phase Extraction and Concentration for the Determination of Nitrosamines in Drinking Water (EPA Method 521)

Introduction

Nitrosamines are a group of chemical compounds regulated due to their carcino-genic characteristics. Nitrosamines come from a variety of sources including widespread use in the manufacturing of rubber products, as well as cosmetics. They have also been demonstrated to form reactions between food preservatives and amines. In drinking water, established limits are low (<1 ng/L), requiring detection by Mass Spectrometer in the chemical ionization mode.

The use for the FMS PowerPrep™ SPE system and integrated SuperVap concentrator enables labs to fully automate the extraction and concentration process of EPA Method 521 as a single turnkey solution. Samples can be taken from the bottle and processed directly to GC vial for a hands free extraction and concentration. This capability frees up extraction chemists to perform other tasks and eliminates operator error.

Reagents and Consumables

- Supelco Supelclean™ Coconut Charcoal SPE (2 grams)
- Pesticide grade Methylene Chloride
- Pesticide grade Methanol
- HPLC grade or DI water
- Restek activated sodium sulfate cartridges (2 grams)
- Ultra Scientific Nitrosamines Mixture (US-113N)
- Ultra Scientific N-Nitrosodimethylamine-d6 Solution (IST-760)
- Ultra Scientific N-Nitrosodi-n-propylamine-d14 Solution (IST-770)
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Instrumentation

- FMS, Inc. PowerPrep™ SPE (Solid Phase Extraction) System
- FMS, Inc. SuperVap Concentrator
- FMS, Direct-to-Vial concentrator tubes
- Thermo Trace GC with Polaris Q MS

PowerPrep SPE procedure

1. The Cartridge is conditioned with 5 mL methylene chloride
2. The Cartridge is purged dried with Nitrogen
3. The Cartridge is conditioned with 20 mL methanol
4. The Cartridge is conditioned with 10 mL DI H_2O
5. The Sample is loaded across the Cartridge via vacuum (~10mL/min)
6. The is Cartridge dried under vacuum for an additional 10 minutes
7. The is Cartridge eluted with methylene chloride in three, 10 mL cycles (1 minute pause between each).
8. The lines are purged with Nitrogen

SuperVap Concentrator settings

Pre-heat temp: 35 ºC

Pre-heat time: 15 minutes

Heat in Sensor mode: 35 ºC

Nitrogen Pressure: 10 PSI

Figure 1: PowerPrep SPE and SuperVap Concentrator systems.

FMS
Fluid Management Systems

Results

Table 1 shows the calculated recoveries and RPD of duplicate samples.

	ng/L Run #1	Percent Recovery	ng/L Run #2	Percent Recovery	RPD
NDMA-d6	25	125.0%	22.8	114.0%	9.2%
NDMA	23.6	118.0%	23	115.0%	2.6%
NMEA	24.8	124.0%	25.4	127.0%	-2.4%
NDEA	22.8	114.0%	24	120.0%	-5.1%
NPYR	22.2	111.0%	22	110.0%	0.9%
NMOR	23	115.0%	19.8	99.0%	15.0%
NDPA	22.4	112.0%	19	95.0%	16.4%
NPIP	21.6	108.0%	22	110.0%	-1.8%
NDBA	22	110.0%	18.6	93.0%	16.7%
NDFA	18.6	93.0%	18.4	92.0%	1.1%

Conclusions

Review of both the recovered spike concentrations and sample RPDs demonstrate the FMS PowerPrep™ SPE system along with the integrated SuperVap Concentrator system can fully automate sample preparation for EPA Method 521 with a high level of proficiency. EPA 521 historically being a timely and delicate analytical method for sample prep labs, reliable automation is an almost necessity. FMS systems make it possible to take samples from the collection bottle directly to the GC vial, freeing up prep chemists to perform other tasks, thus increasing lab efficiency and productivity.

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