Application Note

Analysis of Nitrosamines in Waste Water with Semi-Automated Solid Phase Extraction (EZSpe®) Using EPA Method 607

Introduction

Nitrosamines are a group of chemical compounds regulated due to their carcinogenic characteristics. They come from a variety of sources including widespread use in the manufacturing of rubber products, as well as cosmetics. Nitrosamines have also been demonstrated to react with food preservatives. Their analysis is described in US EPA 607.

To meet demands for a low cost method that requires less financial investment than 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
- Thermo Trace GC with Trace DSQ MS

Consumables

- FMS, Inc. 2 g coconut charcoal cartridges
- FMS, Inc. florisil cartridges
- FMS sodium sulfate column
- Ultra pure DI water
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Dichloromethane
- Fisher Pesticide Grade Pentane
- Fisher Pesticide Grade Diethyl Ether
- Relevant Nitrosamines Spiking Solutions

Procedure

- 6 samples (1 L water each) are prepared, acidified to a pH ~2, ands spiked with relevant standards
- Put sample bottles in place and fill automated rinse bottles with 100 mL 5% acetone in diethyl ether
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 40 mL of 15% diethyl ether in pentane
- Samples are loaded across cartridges under vacuum
- Cartridges are washed with 90 mL of 15% diethyl ether in pentane and sent to organic waste
- Sample bottles are automatically rinsed from the rinse bottles with 100 mL of 5% acetone in diethyl ether

Stage 2:

- Eluent solution from sample bottles is now loaded across the coconut charcoal cartridges and sodium sulfate cartridges and the eluent is collected for analysis into Direct to GC Vial Collection Vessels

FMS SuperVap®

- Pre-heat temp: 25 ºC
- Pre-heat time: 10 minutes
- Heat in Sensor mode at 25 ºC under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

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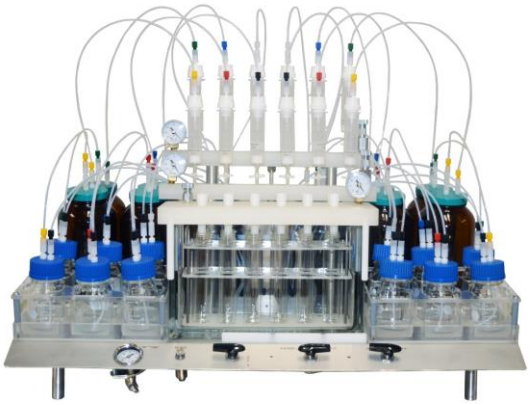
Table 1 with recoveries for Nitrosamines at 20-25 ng/L

Compound name	Average (%)
NDMA	116
NDPA	104
NDnPA	92

Conclusions

The results of these wastewater samples demonstrate the ability of the FMS EZSpe® system to deliver accurate results with excellent reproducibility. The semi-automated EZSpe® is superior to traditional, time-consuming, inconsistent and expensive liquid/liquid extractions.



FMS EZSpe® System

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