

Analysis of Nitrosamines in Drinking Water with Semi-Automated Solid Phase Extraction (EZSpe[®]) Using EPA Method 521

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 521.

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 Polaris Q MS

Consumables

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

Procedure

- 6 samples (500 mL water each) are prepared and spiked with relevant Standards
- Put sample bottles in place and fill automated rinse bottles with 10 mL dichloromethane
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 3 mL dichloromethane twice
- Cartridges are conditioned with 3 mL methanol twice (keep wet)
- Cartridges are conditioned with 3 mL water six times (keep wet)
- Samples are loaded across cartridges under vacuum (50 min)
- Cartridges are dried under vacuum for 10 min
- Sample bottles are automatically rinsed from the rinse bottles with 10 mL dichloromethane

Stage 2:

- Fill empty part of cartridges with dichloromethane and soak 1 min, pull through cartridges
- Dichloromethane 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
- Remove coconut charcoal cartridges and rinse sodium sulfate cartridges with 3 mL dichloromethane and collect

FMS SuperVap[®]

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



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

Compound name	Average (%)
NDMA d6	120
NDMA	116
NMEA	125
NDEA	117
NPYR	110
NMOR	107
NDPA	104
NPIP	109
NDBA	103
NDFA	92

Conclusions

The results of these drinking water 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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