

Automated Solid Phase Extraction (SPE) of Nitrosamines in Water

INTRODUCTION

Due to the carcinogenic nature of nitrosamines at very low levels, new regulations setting detection limits at PPT (ng/L) levels have been set for nitrosamines in drinking water, milk, beer, and food products. EPA Method 521 recommends coconut carbon SPE cartridges and this method can be easily adapted for the analysis of food and beverages.

INSTRUMENTATION USED FOR SAMPLE PREPARATION

Dionex AutoTrace® instrument

SAMPLE PREPARATION

- A 500 to 1000 mL sample of water is taken for analysis.
- Screen for NDMA, (N-nitrosodimethylamine) plus NMEA, NDEA, NDPA, NMOR, NPYR, NPIP, NDBA, and DPhA.
- The eluent can be dried using an in-line short anhydrous sodium sulphate cartridge.

AUTOTRACE METHOD AND CONCLUSION

All solvent lines are purged and primed with solvent. A maximum of five solvents can be used to run a wide range of methods. Independent lines separate the aqueous and organic waste solvents. The instrument sample rack has six positions that takes volumes from 10 to 2000 mL and six sample collection positions for the eluent. This method offers an automated rugged and reproducible solution for cleaning up the samples to concentrate and remove interferences.

No.	Method: Estimated time 2h 22 min
1	Process six samples using the following method steps:
2	Condition the column with 5.0 mL of dichloromethane into aqueous waste.
3	Condition column with 5.0 mL of MTBE into solvent waste.
4	Condition column with 5.0 mL of DI water into aqueous waste.
5	Load 1000.0 mL of sample into column.
6	Clean each sample path with 10.0 mL into aqueous waste.
7	Wash syringe with 5.0 mL of dichloromethane.
8	Dry column with gas for 20.0 min.
9	Collect 5.0 mL fraction into sample tube using dichloromethane.
10	Wash syringe with 5.0 mL of CH ₃ OH.
11	Wash syringe with 5.0 mL of DI water.
12	End

PARAMETERS:

Flow Rates

Cond Flow:	15.0 mL/min
Load Flow:	10.0 mL/min
Rinse Flow:	20.0 mL/min
Elute Flow:	5.0 mL/min
Cond Air Push:	15.0 mL/min
Rinse Air Push:	20.0 mL/min
Elute Air Push:	5.0 mL/min

SPE Parameters

Push Delay:	5 sec
Air Factor:	1.0
Autowash Vol.:	1.00 mL

Instrument Parameters

Max. Elution Vol.:	12.0 mL
Exhaust Fan On:	Yes
Beeper On:	Yes

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LPN 2215 PDF 02/09
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