Determination of Nitrosamines in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (MS/MS)*

UCT Part Numbers:
EU52112M6 (2000 mg activated coconut carbon, 6 mL)
ECSS156 (6 mL Drying Cartridge with 5 grams anhydrous sodium sulfate)

EPA Method 521

Activated carbon is used for the determination of various nitrosamines in finished drinking water and untreated source waters using GC/MS/MS.

Nitroaromatics, Nitramines and Nitrate Ester Analytes

Analyte	Abbreviation	% Recovery n=3
N-Nitrosodimethylamine	NDMA	95
N-Nitrosomethyldiethylamine	NMEA	98
N-Nitrosodiethylamine	NDEA	95
N-Nitrosodi-n-propylamine	NDPA	90
N-Nitrosodi-n-butylamine	NDBA	94
N-Nitrosopyrollidine	NPYR	76
N-Nitrosopiperidine	NPIP	81

*For complete details on Method 521, September 2004, the analyst is referred to: J.W.Munch & M.V.Bassett, "Determination of Nitrosamines in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (MS/MS), National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

Procedure

1. **Cartridge Conditioning**

    a) Add 3 mL of methylene chloride to the cartridge, then slowly draw all solvent through the cartridge

    b) Add 3 mL of methanol to the cartridge, turn on vacuum and draw through

    c) Add 3 mL of methanol again and draw through so that the methanol just covers the top of the cartridge frit

    Do not let the cartridge go dry after this step otherwise repeat starting at step 1 b)

    d) Add 3 mL of reagent water and draw through

    e) Repeat water rinse, step d) **5 additional times**

Proper conditioning of the cartridge is essential for good precision and accuracy

2. **Sample Extraction**

   a) Adjust the vacuum setting so that the flow rate is 10 mL/minute

   b) After sample extraction, draw air through the cartridge for **10 minutes** at full vacuum

   c) After drying, proceed immediately to cartridge elution

3. **Cartridge Elution**

   a) Insert a clean collection tube in the manifold

   b) Fill the cartridge with methylene chloride

   c) Partially draw the methylene chloride through at low vacuum and then turn vacuum off

   d) Allow cartridge to soak for 1 minute

   e) Draw the remaining methylene chloride through in dropwise fashion

   f) Continue to add methylene chloride to the cartridge as it is being drawn through until a total of 12-13 mL have been added

   g) Concentrate the methylene chloride to about 0.9 mL in a water bath near room temperature. Do not concentrate less than 0.5 mL as loss of analyte may occur

Note: Small amounts of residual water from the sample container and SPE cartridge may form an immiscible layer with the extract. To eliminate the water a drying column packed with 5 grams of anhydrous sodium sulfate or use **ECSS15M6** for drying. Wet the cartridge with a small volume of methylene chloride before adding extract. Rinse the drying column with 3 mL of methylene chloride.

4. **Sample Analysis**

   a) Calibrate the MS in EI mode using FC-43

   b) Inject into a GC/MS/MS

   c) Identify the product ion spectrum to a reference spectrum in a user created data base

Mass Spectral Data

Analyte	Retention Time (min)	Precursor Ion (m/z)	Product/Quantitation Ion (m/z)
NDMA	8.43	75	43(56)
NMEA	11.76	89	61(61)
NDEA	14.80	103	75(75)
NPYR	22.34	101	55(55)
NDPA	22.40	131	89(89)
NPIP	24.25	115	69(69)
NDBA	30.09	159	57(103)
NDMA-d6 surrogate	8.34	81	46(59)
NMEA-d10 IS	14.63	113	81(81)
NDPA-d6 IS	22.07	145	97(97)

Injector Program

Temp (°C)	Rate (°C/min)	Time (min)
37	0	0.72
250	100	2.13
250	0	40

Injector Split Vent Program

Time (min)	Split Status
0	Open
0.70	Closed
2.00	Open

GC Oven Temperature Program

Temperature (°C)	Rate (°C/min)	Hold Time (min)
40	0	3.0
170	4.0	0
250	20.0	3.0

Limits and Lowest Concentration Minimum Reporting Levels

Analyte	DL (ng/L)	LCMRL (ng/L)
NDMA	0.28	1.6
NMEA	0.28	1.5
NDEA	0.26	2.1
NPYR	0.35	1.4
NDPA	0.32	1.2
NPIP	0.66	1.4
NDBA	0.36	1.4