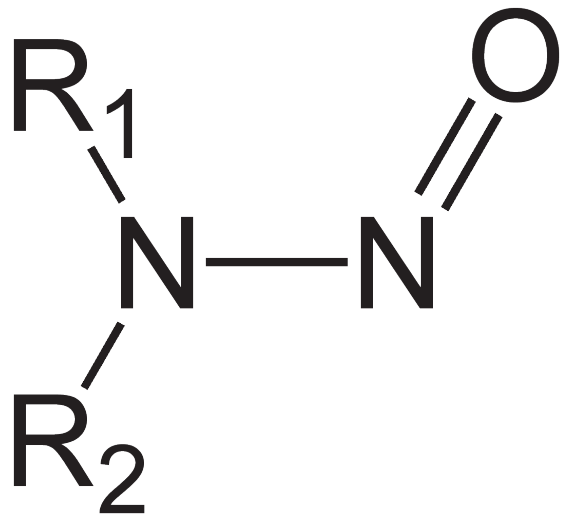
# Determination of Nitrosamines in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography with Large Volume Injection and Chemical Ionization Tandem Mass Spectrometry (MS/MS)*




## UCT Part Numbers

**EU52112M6**
2000 mg activated coconut carbon, 6 mL

**ECSS156**
6 mL Drying Cartridge with 5 grams anhydrous sodium sulfate

## Summary:

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

| 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

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## 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.

**Note:** 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.**

**Note:** 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 database.

**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 |

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**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 |

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