



ENVIRO-CLEAN[®] Carbon Cartridges

Part Number: EU52112M6

EPA Method 521

August 25, 2009

The UCT activated carbon cartridge is designed to provide a high level of performance in solid-phase extraction for the analysis of **nitrosamines** in finished drinking water.

Product Benefits

- Designed to meet US EPA specifications
- Free from interferences that cause false positives
- Double ringed to prevent fines
- No Lot to Lot variability
- Excellent analytical reproducibility
- Packaged in metalized, sealed pouches to maintain product cleanliness

Product Features

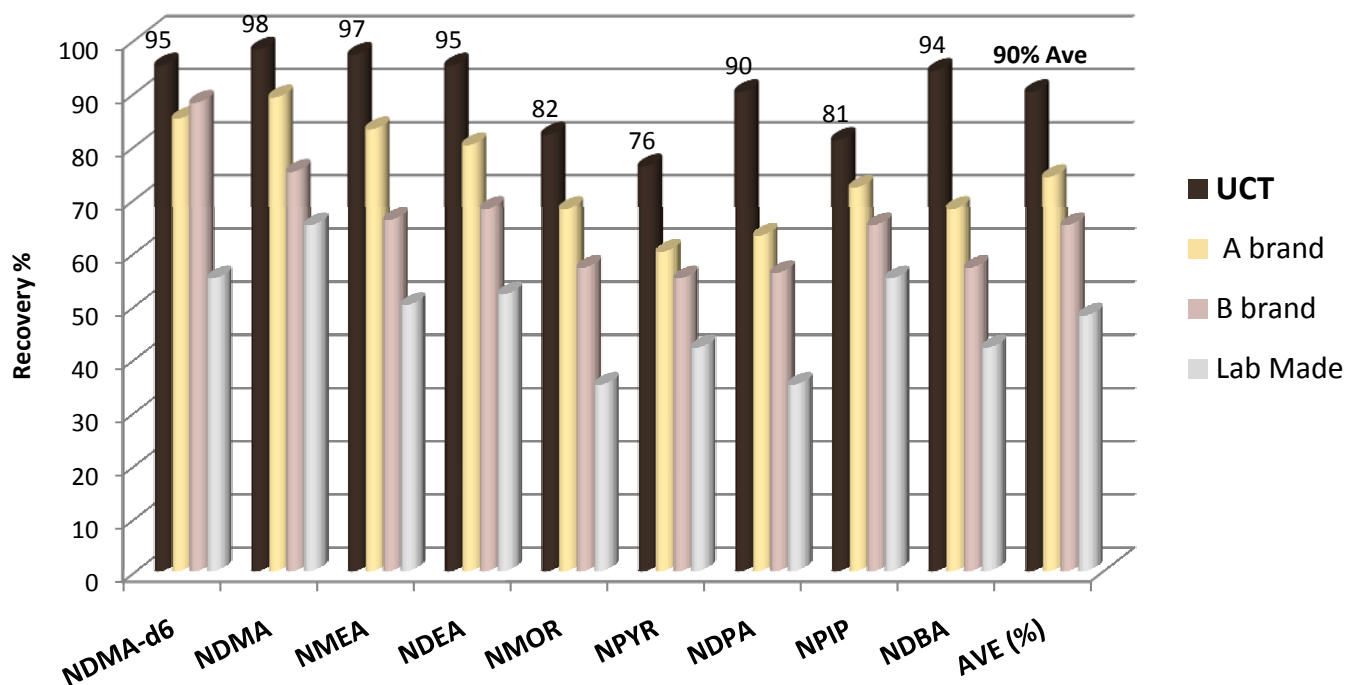
- Each 6 mL cartridge contains 2000 mg of activated coconut carbon sorbent
- Can be used on manual single or multi-station manifold systems
- Cartridges may be used with automated extraction systems

Nitroaromatics, Nitramines and Nitrate Ester Analytes with CAS

UCT Product Number: ENVIRO-CLEAN® EU52112M6

Analyte	Abbreviation	CAS	% Recovery n=3
N-Nitrosodimethylamine	NDMA	62-75-9	95
N-Nitrosomethyldiethylamine	NMEA	10595-95-6	98
N-Nitrosodiethylamine	NDEA	55-18-5	95
N-Nitrosodi-n-propylamine	NDPA	621-64-7	90
N-Nitrosodi-n-butylamine	NDBA	924-16-3	94
N-Nitrosopyrrolidine	NPYR	930-55-2	76
N-Nitrosopiperidine	NPIP	100-75-4	81

Comparison of UCT Cartridges to Competitive & Lab Prepared Cartridges



Data indicate the performance of **UCT** brand cartridges exceeds competitive brands **A** and **B**, and cartridges prepared in the laboratory

EPA Method 521* Summary

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)

Scope and Application

Method 521 is a procedure using activated carbon for the determination of various nitrosamines in finished drinking water. The method can also be used for untreated source waters but has not been evaluated for these sources. Nitrosamines are sufficiently thermally stable and volatile for direct analysis by gas chromatography. Single laboratory LCMRL for the analytes in this method range from 1.2-2.1 ng/L.

Method Summary

Analytes and surrogates are readily extracted when a 500 mL water sample is drawn through a solid-phase extraction cartridge containing 2 grams of coconut activated carbon. The organic compounds eluted from the solid-phase with a small quantity of methylene chloride. The solvent is concentrated and an internal standard added. The sample components are identified after injection on a fused silica capillary column of a GC/MS/MS equipped with a large volume injection injector.

Interferences

- Major contaminant sources are reagents and water
- Nitrosamines may be present in trace amount in rubber products such as gloves and water systems. NDMA can leach from rubber products. These products must be avoided in the reagent water system. Analysis of a laboratory blank can provide information about the source of contamination
- Water stored in glass bottles with PTFE caps is recommended
- Rubber coated septa on injection vial may also introduce method analytes into the sample extracts giving false high readings

Sample Collection

- Field sampling equipment must be free of plastic or rubber tubing
- All field samples must be dechlorinated with 80-100 mg of sodium thiosulfate per liter at time of collection
- Samples must be iced during shipment and not exceed 10 °C
- Sample stored in the lab must be held at 6°C
- Analyze within 14 days after collection
- Sample extracts can be stored up to 28 days in amber vials at -15°C or less and protected from light

Safety

The analytes in this method are classified as known human and mammalian carcinogens. Standard and stock solutions should be handled using suitable protection to skin and eyes.

Notes

- GC systems must be capable of temperature programming
- Deactivated post liners should be used
- Tandem mass spectrometers may be either triple quadrupole or ion trap

Procedure (manual or automated)

1) Cartridge Conditioning

- a) Add 3 mL of methylene chloride to the cartridge, then turn on the vacuum and slowly draw completely through the cartridge
- b) Add 3mL 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 layer of carbon.

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

- d) Add 3 mL of reagent water to the cartridge 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 step 3)

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 then turn vacuum off and allow cartridge to soak for 1 minute
- d) Draw the remaining methylene chloride through in dropwise fashion
- e) Continue to add methylene chloride to the cartridge as it is being drawn through until a total of 12-13 mL have been added

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 **UCT 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.

- f) 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

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

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 internal std	14.63	113	81(81)
NDPA-d6 internal std	22.07	145	97(97)

Retention times were obtained on a Varian Saturn 4 GC/MS/MS using the following conditions:

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

*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