

Robust Analysis of Nitrosamine Using Strata™ Activated Carbon Extraction

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Overview

EPA method 521 is a popular testing method for nitrosamine compounds by GC-MS/MS. Here, a method that employs activated carbon for the extraction of polar analytes such as N-Nitrosodimethylamine is used. While dealing with polar molecules like nitrosamines, traditional sample preparation using Solid Phase Extraction (SPE) might not be efficient. Since the analyte is polar, a specialized extraction technique is needed. Activated carbon contains porous carbon with defined pore volume and high surface area that is an appropriate choice for polar analytes. In this application note, we have extracted several nitrosamines from water using Strata Activated Carbon cartridges. In addition, multiple batches of Strata Activated Carbon cartridges were evaluated to prove the consistency of the newly developed porous activated carbon material.

Sample Preparation

Condition:	Strata Activated Carbon, 2 g/6 mL cartridge (Part No. CSO-9209) with 2 washes of 3 mL Methylene Chloride, soak for 2 min, 2 washes of 3 mL Methanol, soak 2 min
Equilibrate:	Cartridges with 3 washes of 3 mL Water
Load:	500 mL water sample spiked with internal standard onto cartridges
Dry:	Cartridges for 10 min
Elute:	With 3 mL Methylene Chloride and repeat elution 3 more times (12 mL total volume)
Water Removal:	Pass eluent through Sodium Sulfate Giga™ tubes, 5 g/20 mL (Part No. 8B-S124-LEG), pre-wetted with Methylene Chloride and wash with 3 mL Methylene Chloride
Evaporate:	Solvent to approximately 0.9 mL in a water bath at 20-25 °C under a gentle stream of Nitrogen
Adjust:	Final volume to 1 mL with Methylene Chloride

GC-MS Conditions

Column: Zebtron™ ZB-5MSPLUS™

Dimension: 30 meter x 0.25 mm x 1.00 µm

Part No.: [7HG-G030-22](#)

Injection Program:	Ramp(°C/min)	Temp (°C)	Time(min)
-	-	40	0.10
200	200	250	17.0

Recommended Liner: Zebtron PLUS Z-Liner™ (Compatible with Agilent® & Thermo Scientific® GC instrument)

Liner Part No.: [AG2-0A03-05](#)

Carrier Gas: Helium @ 1 mL/min (Constant Flow)

Oven Program:	Ramp(°C/min)	Temp (°C)	Time(min)
-	-	40	2.5
10	10	105	0.0
10	10	200	0.0
50	50	280	1.5

Detector: MSD

Detector Temperature: 300 °C

Table 1. Mass Spec Transitions

Peak No.	Analyte Name	m/z
1	N-Nitrosodimethylamine-d6 (NDMA-d6) (SS)	81, 59
2	N-Nitrosodimethylamine (NDMA)	75, 56
3	N-Nitrosomethylethylamine (NMEA)	90, 61
4	N-Nitrosodiethylamine-d10 (NDEA-d10) (IS)	114, 81
5	N-Nitrosodiethylamine (NDEA)	85, 75
6	N-Nitrosopropylamine-d14 (NDPA-d14) (IS)	146, 97
7	N-Nitrosodipropylamine (NDPA)	132, 89
8	N-Nitrosopyrrolidine (NPYR)	102, 55
9	N-Nitrosomorpholine (NMOR)	87, 86
10	N-Nitrosopiperidine (NPIP)	116, 69
11	N-Nitrosodibutylamine (NDBA)	159, 103



Figure 1. Strata™ Activated Carbon Extraction Cartridges

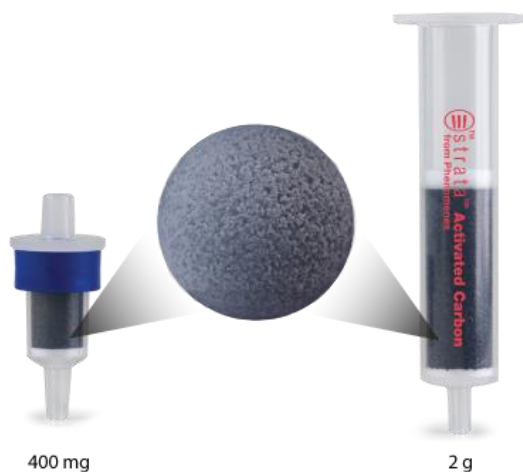
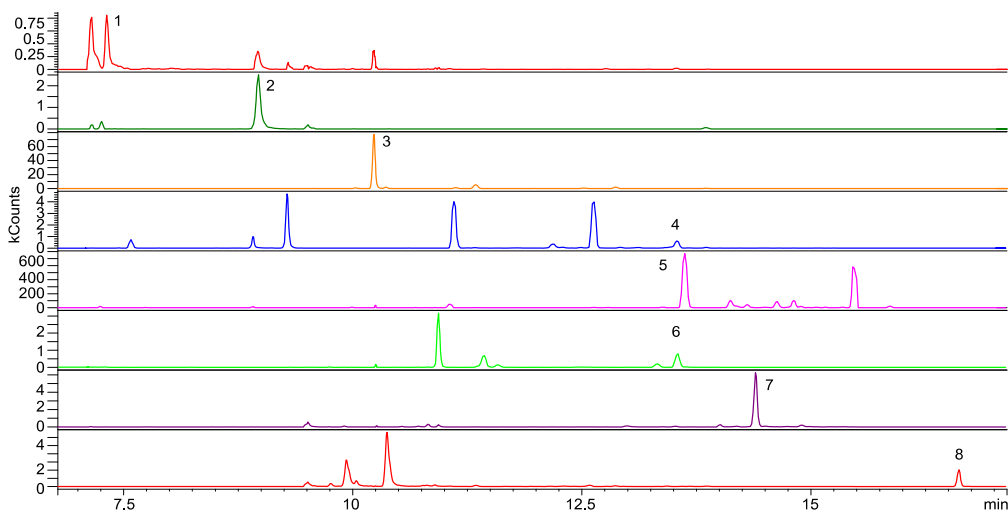


Figure 2. Nitrosamine Standards in Water after Strata Activated Carbon Extraction

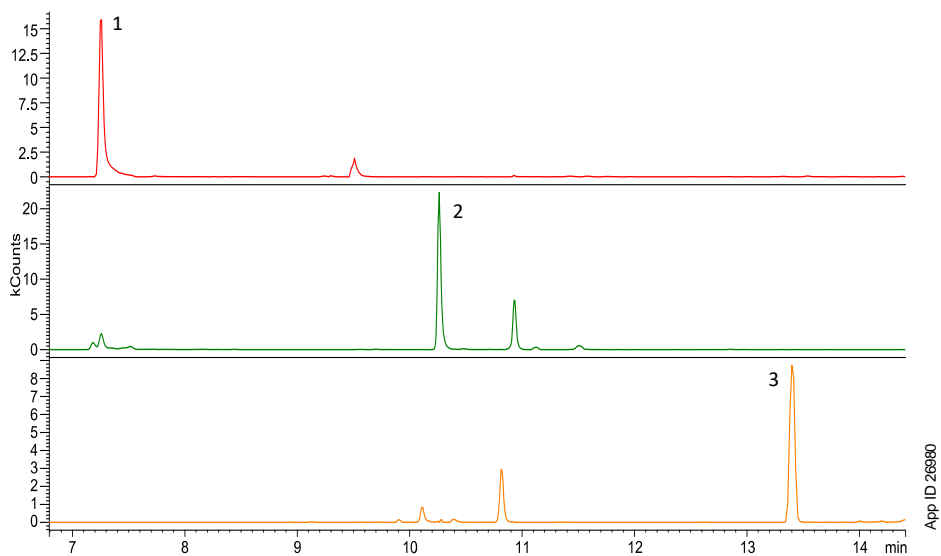


Peak No.	Analyte	RT (min)
1	NDMA	7.299
2	NMEA	8.967
3	NDEA	10.367
4	NDPA	13.503
5	NPYR	13.551
6	NMOR	13.631
7	NPIP	14.407
8	NDBA	16.623

App ID 26881



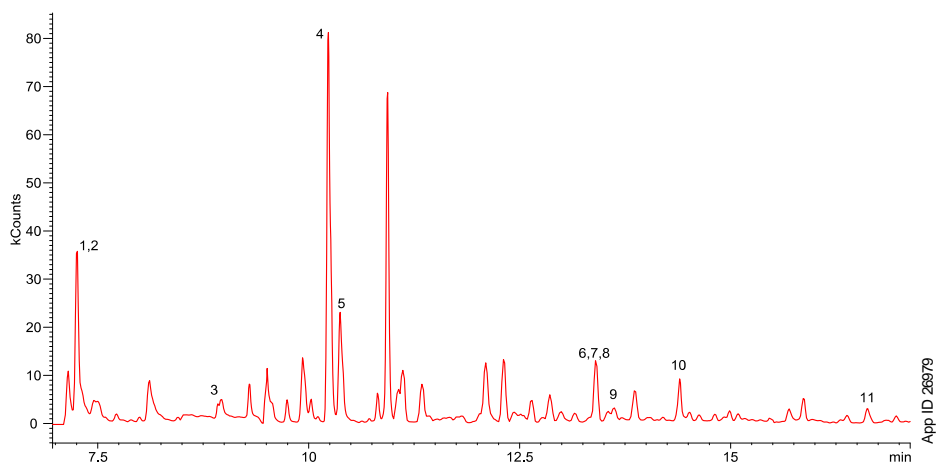
Figure 3. Nitrosamine Internal Standard and Surrogate Standard Spike in Water after Strata™ Activated Carbon Extraction



Peak No.	Analyte Name	RT (min)
1	NDMA-d6 (SS)	7.236
2	NDEA-d10 (IS)	10.268
3	NDPA-d14 (IS)	13.413

App ID 26980

Figure 4. Water Spiked with 2 ppb Nitrosamine Standards, Surrogate Standard, and Internal Standard after Strata Activated Carbon Extraction



Peak No.	Analyte	RT (min)
1	NDMA-d6 (SS)	7.236
2	NDMA	7.299
3	NMEA	8.967
4	NDEA-d10 (IS)	10.268
5	NDEA	10.367
6	NDPA-d14 (IS)	13.413
7	NDPA	13.503
8	NPYR	13.551
9	NMOR	13.631
10	NPIP	14.407
11	NDBA	16.623

App ID 26979



Figure 5. % Recovery of Surrogate Standard After Strata™ Activated Carbon Extraction

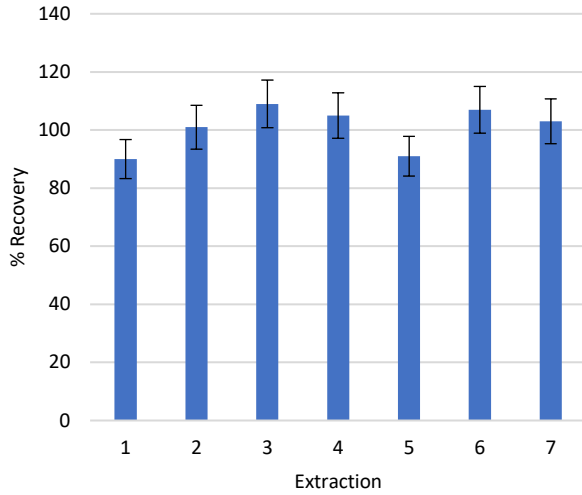


Figure 6. % Recovery of Nitrosamines from a Water Sample After Strata Activated Carbon Extraction

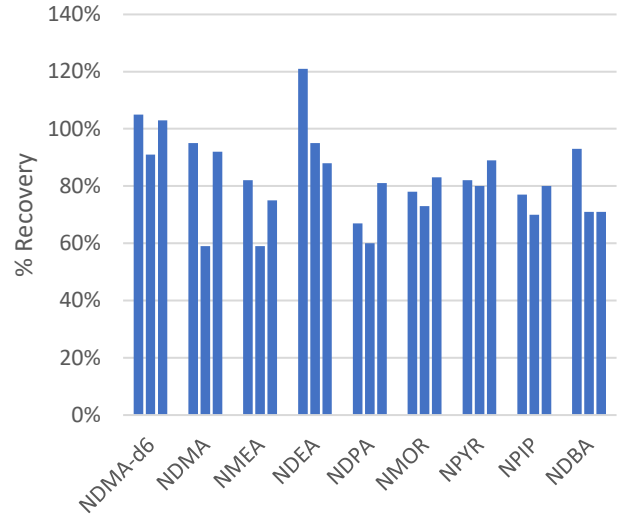
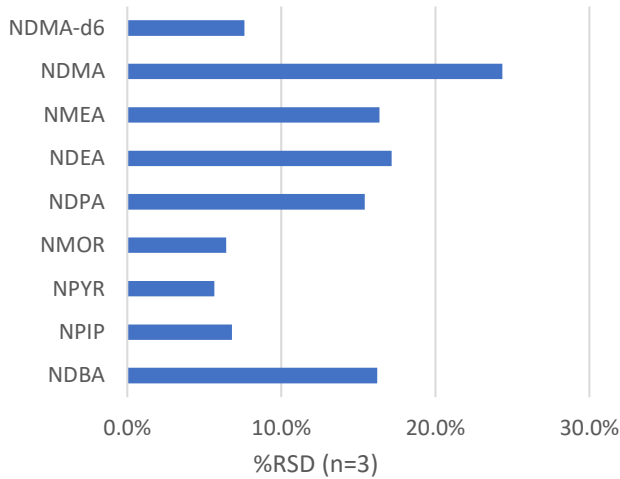


Figure 7. Nitrosamine Recovery Variability After Strata Activated Carbon Extraction



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