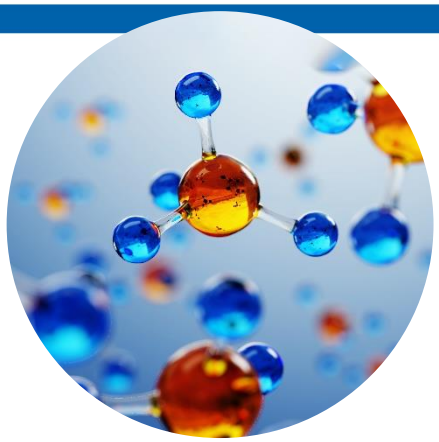


TN-0148

Robust Analysis of Nitrosamine using Strata™ Activated Carbon Extraction in Comparison to Competitor Brand

Eduardo Morales[1], Agustin Pierri, PhD[1], Ramkumar Dhandapani, PhD[2], Philip Leong[2], and Bryan Tackett, PhD[2]
[1]**Weck Laboratories, Inc., 14859 Clark Ave, Hacienda Heights, CA 91745 USA**
[2]**Phenomenex, Inc., 411 Madrid Ave., Torrance, CA 90501 USA**

Introduction

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 technical 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. CS0-9209) or Enviro-Clean® 521 Activated Carbon, 2000 mg/6 mL 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 Method Parameters

Column: Zebron™ ZB-5MS*PLUS*™
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	250	17.0

Recommended Liner: Zebron 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	105	0.0
	10	200	0.0
	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

Ramkumar Dhandapani, PhD
Ramkumar has earned a PhD in Analytical Chemistry and has over 17 years troubleshooting and hands-on experience in chromatography. He loves to write poems, watch, and read Shakespeare's plays.

Results and Discussion

Figure 1 is a representation of the Strata™ Activated Carbon extraction cartridges. The porous carbon is optimized for reproducible extraction of polar analytes. Although the 2 g/6 mL format was employed in this application, there is also a 400 mg format available for smaller sample sizes.

As presented in **Figure 2**, the individual nitrosamine standards were identified after extraction using the Strata Activated Carbon extraction cartridges. **Figure 3** represents the surrogate standard (SS) N-Nitrosodimethylamine-d6 (NDMA-d6) and the two internal standards (IS) N-Nitrosodiethylamine-d10 (NDEA-d10) and N-Nitrosopropylamine-d14 (NDPA-d14) recommended by EPA Method 521.

Figure 4 is a representative chromatogram of a water sample spiked with 2 parts per billion (ppb) each of the nitrosamine standards, the internal standard, and the surrogate standard after extraction using the Strata Activated Carbon extraction cartridge. For analysis, the Zebron™ ZB-5MS*PLUS*™ was utilized. The 5 % Phenyl Arylene selectivity, extensive cross-linkage in the stationary phase, and the highly deactivated fused silica in this column provide MS compatibility and an inert environment for active analytes. This results in low baseline noise and symmetric peaks for even the most challenging analytes like nitrosamines.

Surrogate standard % recovery for seven extractions is presented in **Figures 5**. Multiple extractions using the Strata Activated Carbon cartridges provided low variance of % recovery between extractions when compared to the Enviro-Clean® 521 Activated Carbon cartridges. This is evidenced by the smaller error bars seen with the Strata Activated Carbon cartridges.

Water sample recovery and variability in results are presented in **Figures 6** and **7**. These consistent results are possible because of the narrow range of pore size and surface area that provides low variability.

Figure 1. Strata Activated Carbon Extraction Cartridges

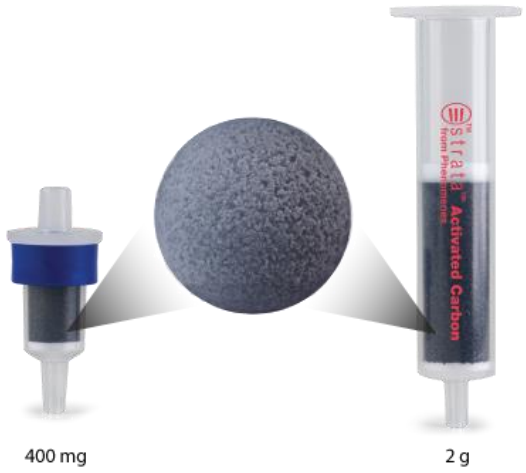
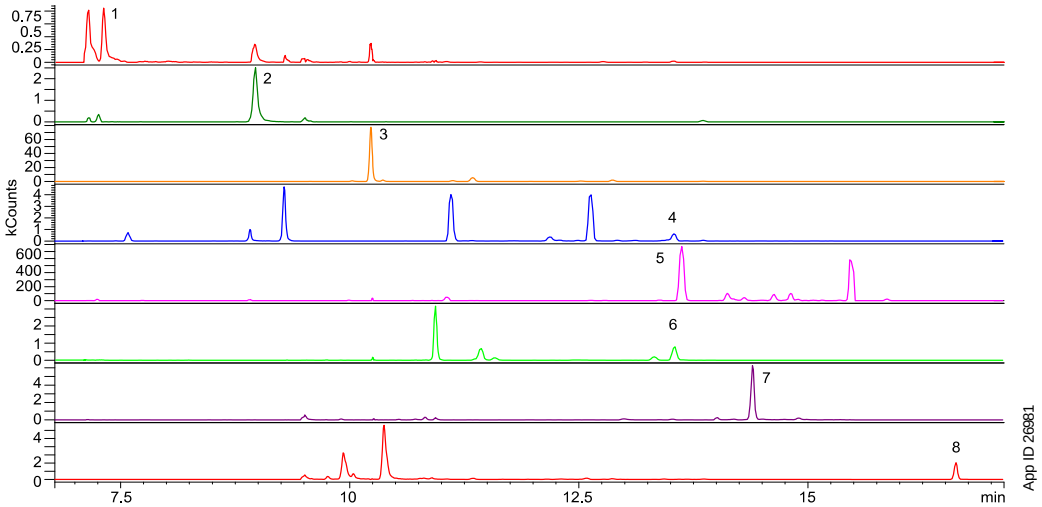
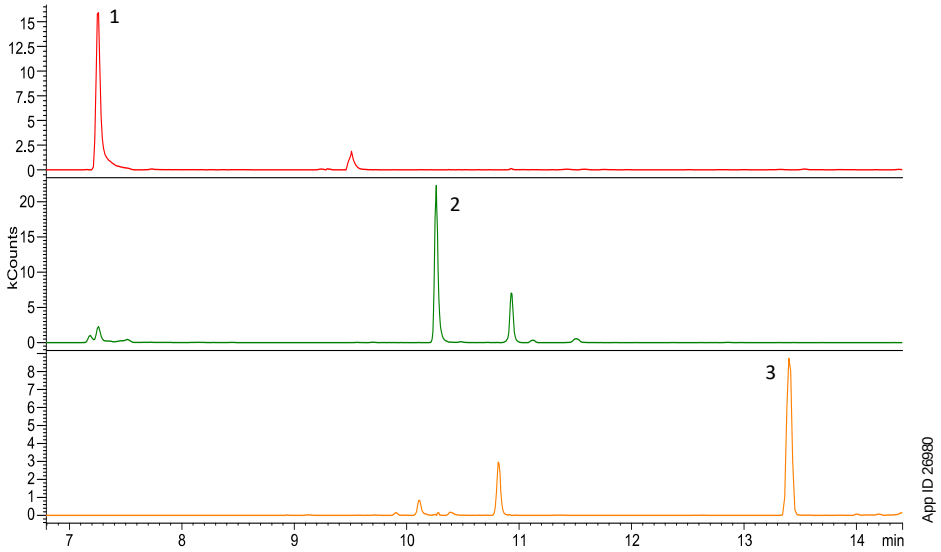


Figure 2. Nitrosamine Standards in Water after Strata Activated Carbon Extraction



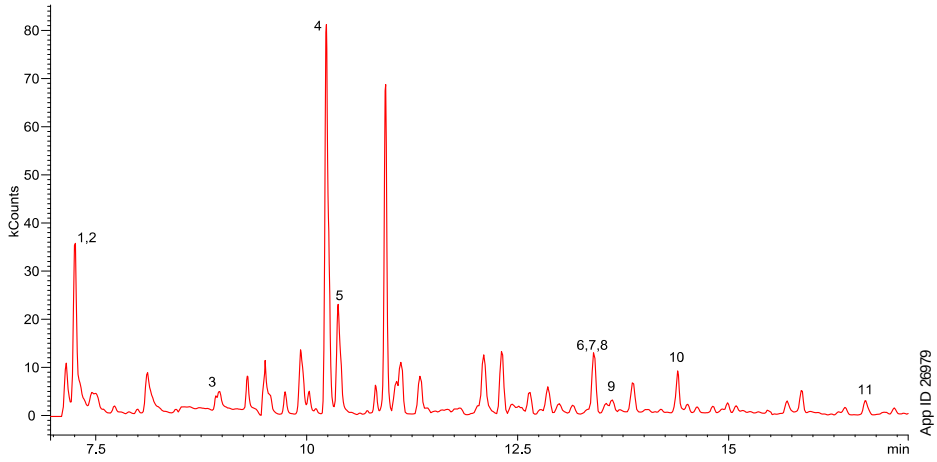
Peak No.	Analyte	RT (min)
1	NDMA	7.299
2	NMEA	8.967
3	NDEA	10.367
4	NDPA	13.503
5	NPYR	13.551
6	NMOR	13.631
7	NPIP	14.407
8	NDBA	16.623

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

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

Figure 5. Comparison of % Recovery of Surrogate Standard

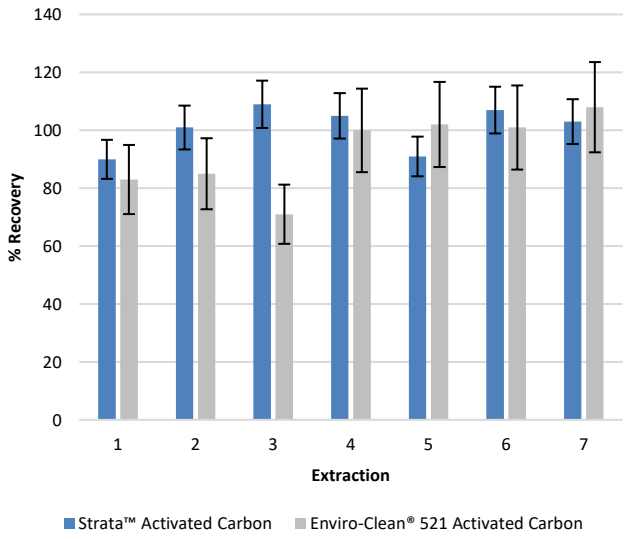


Figure 6. Comparison of % Recovery of Nitrosamines from a Water Sample

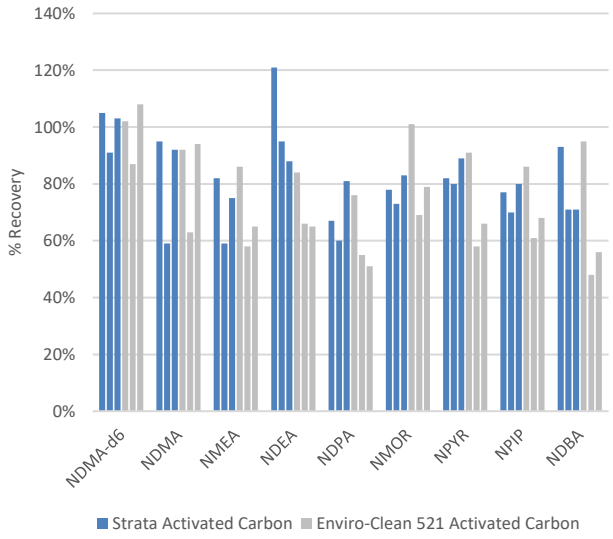
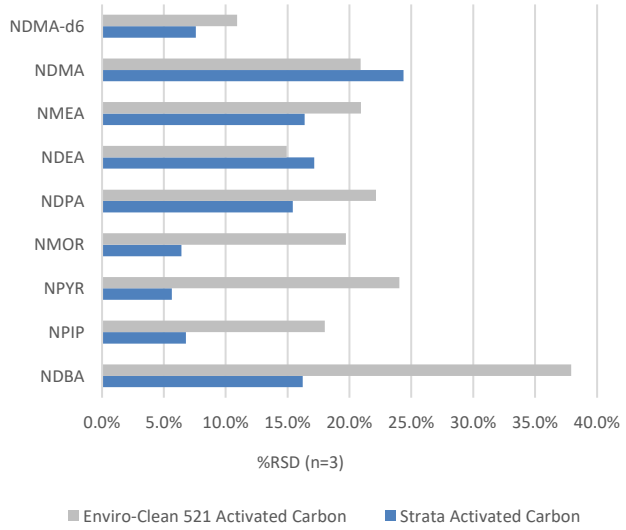


Figure 7. Comparison of Nitrosamine Recovery Variability



Conclusions

Strata Activated Carbon extraction provided reproducible extraction of several nitrosamines as per EPA Method 521. The results demonstrated low variability between batches of activated carbon extraction cartridges due to the narrow range of pore size and surface area of Strata Activated Carbon.

Strata™ Tubes and Cartridges Ordering Information

Strata Activated Carbon				
Part	**Sorbent Mass**	**Volume**	**Format**	**Units/pk**
CS0-9209	2 g	6 mL	Tube	30
CS0-9210	400 mg	Pass through	Cartridge	50

Zebron™ ZB-5MS*plus*™ GC Columns Ordering Information

ID(mm)	df(µm)	Temp. Limits °C	Part No.
1.5-Meter			
0.25	0.25	-60 to 325/350	7XG-G030-11
15-Meter			
0.25	0.25	-60 to 325/350	7EG-G030-11
0.25	0.50	-60 to 325/350	7EG-G030-17
0.25	1.00	-60 to 325/350	7EG-G030-22
20-Meter			
0.18	0.18	-60 to 325/350	7FD-G030-08
0.18	0.36	-60 to 325/350	7FD-G030-53
30-Meter			
0.25	0.25	-60 to 325/350	7HG-G030-11
0.25	0.50	-60 to 325/350	7HG-G030-17
0.25	1.00	-60 to 325/350	7HG-G030-22
0.32	0.25	-60 to 325/350	7HM-G030-11
0.32	0.50	-60 to 325/350	7HM-G030-17
0.32	1.00	-60 to 325/350	7HM-G030-22
0.32	1.50	-60 to 325/350	7HM-G030-28
0.53	1.00	-60 to 325/350	7HK-G030-22
0.53	3.00	-60 to 325/350	7HG-G030-36
30-Meter with 5-Meter Guardian™ Integrated Guard			
0.25	0.25	-60 to 325/350	7HG-G030-11-GGA
0.25	0.50	-60 to 325/350	7HG-G030-17-GGA
30-Meter with 5-Meter Guardian™ Integrated Guard			
0.25	0.25	-60 to 325/350	7HG-G030-11-GGC
0.25	0.50	-60 to 325/350	7HG-G030-17-GGC
60-Meter			
0.25	0.25	-60 to 325/350	7KG-G030-11
0.25	1.00	-60 to 325/350	7KG-G030-22
0.32	1.00	-60 to 325/350	7KM-G030-22

Note: If you need a 5 in. cage, contact Technical support via Phenomenex.com/Chat or simply reach out to your Technical consultant. Conditions may apply. Agilent 6850, some SRI and process GC systems use only 5 in. cages.

Liners Compatible with Thermo Scientific® GC Systems Ordering Information

Zebron™ PLUS Liners for For 5890, 6890 and 7890 Models						
Description	**Application**	**Inlet Style**	**Dimensions**	**Deactivation**	**Part No.**	**Unit**
Direct Connect	Trace analysis, Splitless injections	S/SL	4 x 78.5	PLUS Inert	AG2-0A50-01 AG2-0A50-05 AG2-0A50-25	ea 5/pk 25/pk
Single Taper	Pesticides	S/SL	4 x 78.5	PLUS Inert	AG2-0A10-01 AG2-0A10-05 AG2-0A10-25	ea 5/pk 25/pk
Single Taper Z-Liner™	Semi-volatiles, Dirty samples	S/SL	4 x 78.5	PLUS Inert	AG2-0A13-01 AG2-0A13-05 AG2-0A13-25	ea 5/pk 25/pk
Single Taper with Wool	Semi-volatiles	S/SL	4 x 78.5	PLUS Inert	AG2-0A11-01 AG2-0A11-05 AG2-0A11-25	ea 5/pk 25/pk
Straight	Volatiles	S/SL	4 x 78.5	PLUS Inert	AG2-0A00-01 AG2-0A00-05 AG2-0A00-25	ea 5/pk 25/pk
Straight Z-Liner	Dirty samples, Volatiles, High initial oven temperatures	S/SL	4 x 78.5	PLUS Inert	AG2-0A03-01 AG2-0A03-05 AG2-0A03-25	ea 5/pk 25/pk
Straight Single Baffle	Semi-volatiles, Pesticides	PTV	1.8 x 71	PLUS Inert	AG2-1F06-01 AG2-1F06-05 AG2-1F06-25	ea 5/pk 25/pk

Sodium Sulfate Tubes Ordering Information

Strata™			
Format	**Sorbent Mass**	**Part Number**	**Unit**
Tube	1 g	8B-S124-JCH	6 mL (30/box)
Giga™ Tube	5 g	8B-S124-LEG	20 mL (20/box)

Need a different column size or sample preparation format?

No problem! We have a majority of our available dimensions up on www.phenomenex.com, but if you can't find what you need right away, our super helpful Technical Specialists can guide you to the solution via our online chat portal www.phenomenex.com/Chat.

Australia
t: +61 (0)2-9428-6444
auinfo@phenomenex.com

Austria
t: +43 (0)1-319-1301
anfrage@phenomenex.com

Belgium
t: +32 (0)2 503 4015 (French)
t: +32 (0)2 511 8666 (Dutch)
beinfo@phenomenex.com

Canada
t: +1 (800) 543-3681
info@phenomenex.com

China
t: +86 400-606-8099
cninfo@phenomenex.com

Czech Republic
t: +420 272 017 077
cz-info@phenomenex.com

Denmark
t: +45 4824 8048
nordicinfo@phenomenex.com

Finland
t: +358 (0)9 4789 0063
nordicinfo@phenomenex.com

France
t: +33 (0)1 30 09 21 10
franceinfo@phenomenex.com

Germany
t: +49 (0)6021-58830-0
anfrage@phenomenex.com

Hong Kong
t: +852 6012 8162
hkinfo@phenomenex.com

India
t: +91 (0)40-3012 2400
indiainfo@phenomenex.com

Indonesia
t: +62 21 5010 9707
indoinfo@phenomenex.com

Ireland
t: +353 (0)1 247 5405
eireinfo@phenomenex.com

Italy
t: +39 051 6327511
italiainfo@phenomenex.com

Japan
t: +81 (0) 120-149-262
jpinfo@phenomenex.com

Luxembourg
t: +31 (0)30-2418700
nlinfo@phenomenex.com

Mexico
t: 01-800-844-5226
tecnicomx@phenomenex.com

The Netherlands
t: +31 (0)30-2418700
nlinfo@phenomenex.com

New Zealand
t: +64 (0)9-4780951
nzinfo@phenomenex.com

Norway
t: +47 810 02 005
nordicinfo@phenomenex.com

Poland
t: +48 22 104 21 72
pl-info@phenomenex.com

Portugal
t: +351 221 450 488
ptinfo@phenomenex.com

Singapore
t: +65 800-852-3944
sginfo@phenomenex.com

Slovakia
t: +420 272 017 077
sk-info@phenomenex.com

Spain
t: +34 91-413-8613
espinfo@phenomenex.com

Sweden
t: +46 (0)8 611 6950
nordicinfo@phenomenex.com

Switzerland
t: +41 (0)61 692 20 20
swissinfo@phenomenex.com

Taiwan
t: +886 (0) 0801-49-1246
twinfo@phenomenex.com

Thailand
t: +66 (0) 2 566 0287
thaiinfo@phenomenex.com

United Kingdom
t: +44 (0)1625-501367
ukinfo@phenomenex.com

USA
t: +1 (310) 212-0555
info@phenomenex.com

All other countries/regions
Corporate Office USA
t: +1 (310) 212-0555
info@phenomenex.com

www.phenomenex.com

Phenomenex products are available worldwide. For the distributor in your country/region, contact Phenomenex USA, International Department at international@phenomenex.com