Featured Application: *Nitrosamines on LPGC Rxi-624Sil MS*

2.3x Faster Nitrosamines Analysis with LPGC-MS

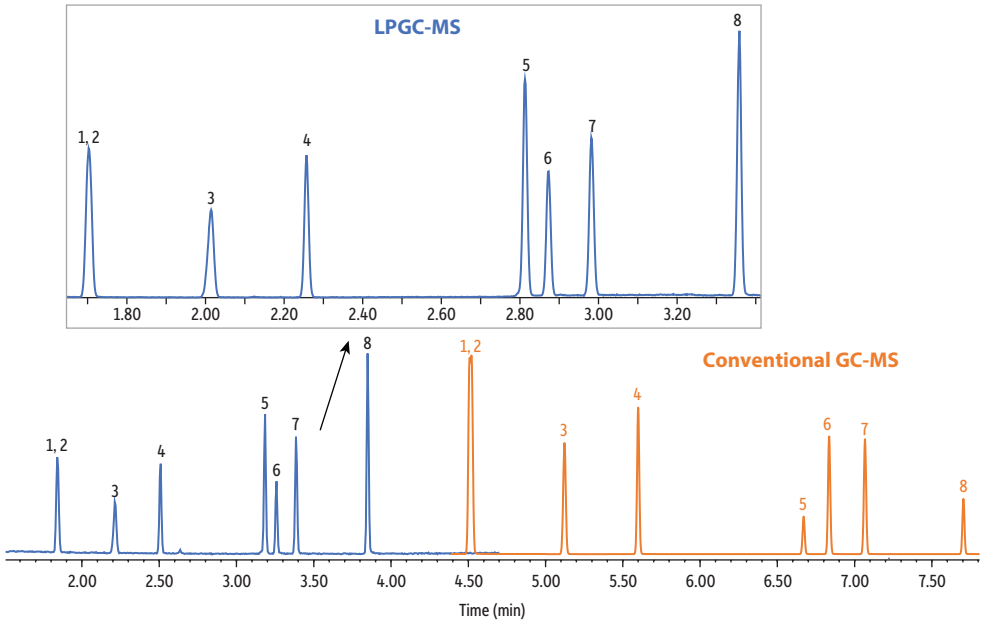
- Up to 2.3x faster than conventional GC-MS analysis.
- Reduces helium usage by up to 67% compared to conventional GC-MS methods.
- Rxi-624Sil MS kits are factory connected with a proven leak-free connector making LPGC as simple as a column change.

Nitrosamines are widespread impurities that are prevalent in food products, including processed foods, cured meats, and vegetables, along with cosmetics, drinking water, and the air. Due to the carcinogenic potential of these compounds at low levels, the FDA, in collaboration with other regulatory authorities, provides guidance on the acceptable daily limit (ADL) for nitrosamines. Accurate and efficient methods are needed to monitor nitrosamine levels across food and consumable products and the environment. GC methodologies for nitrosamines are frequently performed using 30 m 624 type capillary columns and require high amounts of helium, a costly and nonrenewable resource. To reduce helium usage and analysis times, shorter or thinner-film columns are an attractive alternative; however, these column types are not recommended for use with ion-trap GC-MS/MS systems because the chromatographic peak widths generated are insufficient to obtain a necessary number of scans during peak elution.

Our low-pressure GC (LPGC) column kits provide an advantage in nitrosamine analysis by reducing both analysis time and helium usage and are available in two different configurations to accommodate the unique testing requirements of each laboratory. The LPGC-MS method shown in Figure 1 below, run on our 10 m x 0.32 mm ID LPGC Rxi-624 Sil MS kit (cat.# 11804), increased the speed of analysis by 2.3x and reduced helium consumption by 67% compared to conventional GC-MS methods. Our 15 m x 0.53 mm ID LPGC Rxi-624 Sil MS kit (cat.# 11803) is the recommended kit for laboratories performing large volume injections and can be used out of the box without the hassle of attaching a guard column. Utilizing LPGC kit, cat.# 11803, the LPGC method was 1.8x faster with a 29% reduction in helium consumption (shown in Figure 2). LPGC is an opportunity for laboratories to increase the efficiency of current methodologies and reduce laboratory costs and is as simple as a column change. Additionally, all our LPGC column kits are factory coupled and individually tested, so you can have the assurance of a leak-free connection. Learn more about LPGC at **www.restek.com/lpgc**

Figure 1: Comparison of Conventional and LPGC-MS (cat.# 11804) Analysis of Nitrosamines

- **LPGC-MS is 2.3x faster and uses 67% less helium compared to conventional GC-MS.**



GC_FS0598

Peaks	t_R (30 m)	t_R (LPGC)	Conc. (ppm)	Ions
1. *N*-Nitrosodimethylamine (NDMA)	4.59	1.70	100	42, 74
2. *N*-Nitrosodimethylamine-d6 (NDMA-d6)	4.60	1.71	100	46, 80
3. *N*-Nitrosomethylethylamine (NMEA)	5.18	2.02	100	42, 88
4. *N*-Nitrosodiethylamine (NDEA)	5.65	2.26	100	42, 57, 102
5. *N*-Nitroso-di-*n*-propylamine (NDPA)	6.71	2.81	100	70, 113, 130
6. *N*-Nitrosopyrrolidine (NPYR)	6.87	2.87	100	41, 68, 100
7. *N*-Nitrosopiperidine (NPIP)	7.10	2.98	100	42, 55, 114
8. *N*-Nitrosodi-*n*-butylamine (NDBA)	7.74	3.36	100	84, 116, 158

Column	See notes
Standard/Sample	Nitrosamine calibration mix, method 521 (cat.# 31898) *N*-Nitrosodimethylamine-d6 (cat.# 33910)
Diluent:	Dichloromethane
Conc.:	100 µg/mL
Injection	
Inj. Vol.:	1 µL split (split ratio 100:1)
Liner:	Topaz, Precision inlet liner, 4.0 mm x 6.3 x 78.5 (cat.# 23305)
Inj. Temp.:	250 °C
Carrier Gas	He
Detector	MS
Mode:	SIM
Transfer Line Temp.:	280 °C
Analyzer Type:	Quadrupole
Source Temp.:	230 °C
Quad Temp.:	150 °C
Tune Type:	PFTBA
Ionization Mode:	EI
Instrument	Agilent 7890B GC & 5977A MSD
Sample Preparation	100 µL aliquots of each standard were diluted in 800 µL of dichloromethane for a final volume of 1 mL. The sample was mixed in a 2 mL, short-cap, screw-thread vial (cat.# 21143) and capped with a short-cap, screw-vial closure (cat.# 24495).

Notes

Conventional (30 m) Analysis:
Column: Rxi-624Sil MS, 30 m, 0.25 mm ID, 1.4 µm (cat.# 13868)
Temp. program: 40 °C (hold 0.5 min) to 320 °C at 30 °C/min (hold 7 min)
Flow: 1.4 mL/min

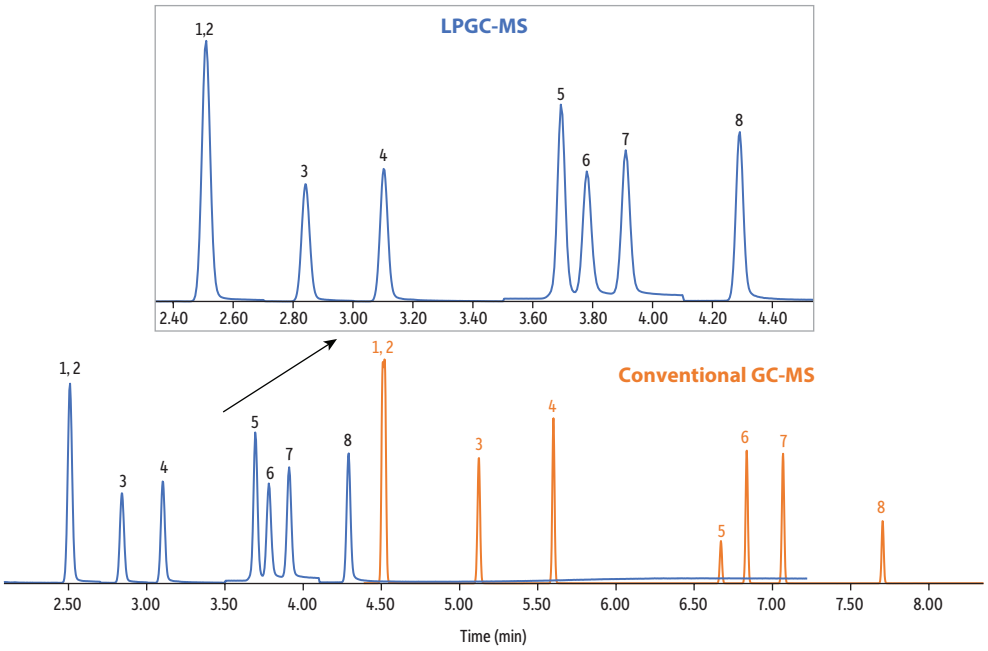
LPGC-MS Analysis:
Column: LPGC Rxi-624Sil MS column kit, includes 10 m x 0.32 mm ID x 1.8 µm Rxi-624Sil MS analytical column and 5 m x 0.15 mm ID Rxi restrictor factory connected via SilTite connector (cat.# 11804)
Temp. program: 40 °C (hold 0.5 min) to 300 °C at 55 °C/min (hold 3 min)
Flow: 0.9 mL/min

The injections were performed on different instruments under different head pressures, resulting in different analyte responses.

For nitrosamine analysis, we also recommend Resprep SPE cartridges (cat.# 26032) for sample preparation.

Figure 2: Comparison of Conventional and LPGC-MS (cat.# 11803) Analysis of Nitrosamines

- **LPGC-MS is 1.8x faster and uses 29% less helium compared to conventional GC-MS.**



GC_FS0599

Peaks	t_R (30 m)	t_R (LPGC)	Conc. (ppm)	Ions
1. *N*-Nitrosodimethylamine (NDMA)	4.587	2.51	100	42, 74
2. *N*-Nitrosodimethylamine-d6 (NDMA-d6)	4.6	2.51	100	46, 80
3. *N*-Nitrosomethylethylamine (NMEA)	5.18	2.843	100	42, 88
4. *N*-Nitrosodiethylamine (NDEA)	5.646	3.105	100	42, 57, 102
5. *N*-Nitroso-di-*n*-propylamine (NDPA)	6.705	3.697	100	70, 113, 130
6. *N*-Nitrosopyrrolidine (NPYR)	6.873	3.783	100	41, 68, 100
7. *N*-Nitrosopiperidine (NPIP)	7.104	3.912	100	42, 55, 114
8. *N*-Nitrosodi-*n*-butylamine (NDBA)	7.736	4.292	100	84, 116, 158

Column See notes
Standard/Sample Nitrosamine calibration mix, method 521 (cat.# 31898)
N-Nitrosodimethylamine-d6 (cat.# 33910)
Diluent: Dichloromethane
Conc.: 100 µg/mL
Injection
Inj. Vol.: 1 µL split (split ratio 100:1)
Liner: Topaz, Precision inlet liner, 4.0 mm x 6.3 x 78.5 (cat.# 23305)
Inj. Temp.: 250 °C
Carrier Gas He
Detector MS
Mode: SIM
Transfer Line Temp.: 280 °C
Analyzer Type: Quadrupole
Source Temp.: 230 °C
Quad Temp.: 150 °C
Tune Type: PFTBA
Ionization Mode: EI
Instrument Agilent 7890B GC & 5977A MSD
Sample Preparation 100 µL aliquots of each standard were diluted in 800 µL of dichloromethane for a final volume of 1 mL. The sample was mixed in a 2 mL, short-cap, screw-thread vial (cat.# 21143) and capped with a short-cap, screw-vial closure (cat.# 24495).

Notes

Conventional (30 m) Analysis:
Column: Rxi-624Sil MS, 30 m, 0.25 mm ID, 1.4 µm (cat.# 13868)
Temp. program: 40 °C (hold 0.5 min) to 320 °C at 30 °C/min (hold 7 min)
Flow: 1.4 mL/min

LPGC-MS Analysis:
Column: LPGC Rxi-624Sil MS column kit, includes 15 m x 0.53 mm ID x 3.0 µm Rxi-624Sil MS analytical column and 5 m x 0.18 mm ID Rxi restrictor factory connected via SilTite connector (cat.# 11803)
Temp. program: 40 °C (hold 0.5 min) to 300 °C at 55 °C/min (hold 3 min)
Flow: 1.5 mL/min

The injections were performed on different instruments under different head pressures, resulting in different analyte responses.

For nitrosamine analysis, we also recommend Resprep SPE cartridges (cat.# 26032) for sample preparation.

Featured Products

Analytical Column	Reference Standards	Maintenance & Accessories
		
LPGC Rxi-624Sil MS Column Kits (cat.# 11804 and 11803)	**Nitrosamine Calibration Mix, Method 521** (cat.# 31898) ***N*-Nitrosodimethylamine-d6** (cat.# 33910)	**Topaz Precision Inlet Liner,** 4.0 mm x 6.3 x 78.5, for Agilent GCs, w/Quartz Wool, Premium Deactivation, 5-pk. (cat.# 23305) **GC Accelerator Oven Insert Kit,** for Agilent 5890, 6890, 7890, and 8890 GCs (cat.# 23849)

LPGC Rxi-624 Sil MS Column Kit

- Up to 2.3x faster analysis of nitrosamines with up to 67% less helium consumption.
- Factory-coupled, leak-free kit makes setup as simple as a column change.
- Ideal for speeding up GC-MS and GC-MS/MS methods.

Temp. Limits	Description	qty.	cat.#
-20 to 280/310°C	LPGC Rxi-624Sil MS column kit, includes 15 m x 0.53 mm ID x 3.0 µm Rxi-624Sil MS analytical column and 5 m x 0.18 mm ID Rxi restrictor factory connected via SilTite connector	kit	11803
-20 to 300/320°C	LPGC Rxi-624Sil MS column kit, includes 10 m x 0.32 mm ID x 1.8 µm Rxi-624Sil MS analytical column and 5 m x 0.15 mm ID Rxi restrictor factory connected via SilTite connector	kit	11804

Nitrosamine Calibration Mix, Method 521

N-Nitrosodiethylamine (55-18-5)
N-Nitrosodimethylamine (62-75-9)
N-Nitrosodi-*n*-butylamine (924-16-3)
N-Nitroso-di-*n*-propylamine (621-64-7)
N-Nitrosomethylethylamine (10595-95-6)
N-Nitrosopiperidine (100-75-4)
N-Nitrosopyrrolidine (930-55-2)

Cat.#	Concentration	Solvent	Volume	Units
31898	1000 µg/mL	Methylene Chloride	1 mL/ampul	ea.

N-Nitrosodimethylamine-d6 Standard

Cat.#	Concentration	Solvent	Volume	Units
33910	1000 µg/mL	Methylene Chloride	1 mL/ampul	ea.

Topaz Precision Inlet Liner

Topaz GC inlet liners feature revolutionary technology and inertness to deliver you the next level of True Blue Performance:

- **Deactivation**—unbelievably low breakdown for accurate and precise low-level GC analyses.
- **Reproducibility**—unbeatable manufacturing controls and QC testing for superior reliability across compound classes.
- **Productivity**—unparalleled cleanliness for maximized GC uptime and lab throughput.
- **100% Satisfaction**—if a liner doesn't perform to your expectations, we will replace it or credit your account.

Patented

Cat.#	Product Name	Units
23305	Topaz, Precision inlet liner, 4.0 mm x 6.3 x 78.5, for Agilent GCs, w/quartz wool, premium deactivation	5-pk.

GC Accelerator Oven Insert Kit

for Agilent 5890, 6890, 7890, and 8890 instruments

- GC Accelerator kit installs easily without damaging the GC column or interfering with the MS interface.

Description	Instrument	qty.	cat.#
GC Accelerator Oven Insert Kit	for Agilent 5890, 6890, 7890, and 8890 instruments	kit	23849

If using a 120 V GC oven, a GC Accelerator oven insert kit (cat.# 23849) may be needed to meet aggressive ramp rates.

Related Products

Vials and Caps

Cat.#	Product Name	Units
21143	Short-Cap Vial with Grad Marking Spot, 9-425 Screw-Thread, 2.0 mL, 9 mm, 12 x 32 (vial only), Amber	1000-pk.
24495	Short Screw Caps, Polypropylene, Screw-Thread, PTFE/Silicone/PTFE Septa, Black, Preassembled, 2.0 mL, 9 mm	100-pk.

SPE Cartridge, EPA Methods 521 and 522

- Designed to provide consistent and reproducible results.
- Activated charcoal sorbent phase.
- May be processed with any one or all of these techniques: positive pressure, sidearm flask, centrifuge, or vacuum manifold.

Cat.#	Includes	Units
26032	SPE Cartridge, EPA Methods 521 and 522, 6 mL/2g	30-pk.

RESTEK
Pure Chromatography

Questions? Contact us or your local Restek representative (www.restek.com/contact-us).

www.restek.com

Lit. Cat.# GNFA4240A-UNV