Featured Application: *Arylamines on LPGC Rxi-35Sil MS*

Rapid LPGC-MS Analysis of Arylamines Increases Throughput and Reduces Helium Use

- Analyze arylamines 3.3x faster than conventional GC-MS.
- Cut costs by reducing helium consumption 81%.
- Pre-connected LPGC column kit prevents leaks, simplifies installation, and allows large-volume injection.

Azo dyes are synthetically produced colorants that are commonly used in the food, cosmetic, pharmaceutical, and textile industries. They are present in a wide variety of consumer products, and some can produce carcinogenic or mutagenic arylamines as breakdown products. Due to concerns about human health and exposure, effective methods are needed for the analysis of arylamines. Testing is usually performed by conventional GC-MS or LC-MS, but long analysis times limit sample throughput.

A much faster alternative, low-pressure GC-MS (LPGC-MS) is a paired-column technique that can bring significant speed gains to labs analyzing arylamines. This approach uses the MS vacuum to lower pressure within the analytical column, thus increasing speed. For example, in the analysis of arylamines (22 of which are regulated by EU REACH legislation [1]) shown in Figure 1, the run time was 3.3x faster than the conventional method, and it also used 81% less helium, which provides a substantial cost savings.

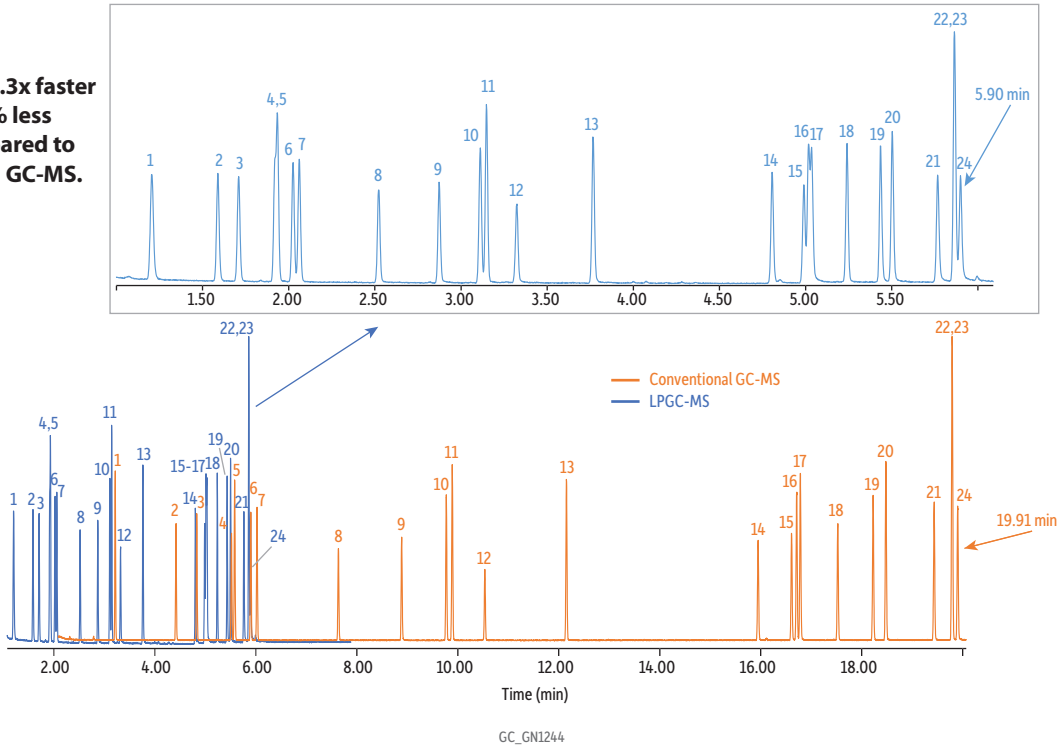
LPGC-MS only works because the inlet pressure is maintained by pairing the analytical column to a narrow restrictor column. However, due to the difference in tubing diameters, it can be difficult to make a robust connection manually, which has limited adoption of the technique. LPGC column kits from Restek solve this problem because they are factory connected and individually leak checked, which ensures a leak-free connection and simplifies setup. Visit **www.restek.com/lpgc** to learn more about this powerful technique.

References

1. Regulation (EC) No. 1907/2006 of the European Parliament and of the Council of 18 December 2006 Concerning the Registration, Evaluation, Authorization and Restriction of Chemicals (REACH). https://osha.europa.eu/en/legislation/directives/regulation-ec-no-1907-2006-of-the-european-parliament-and-of-the-council.

Figure 1: Arylamines on LPGC Rxi-35Sil MS Compared to Conventional GC-MS Analysis

- **LPGC-MS is 3.3x faster and uses 81% less helium compared to conventional GC-MS.**



GC_GN1244

	Peaks	tR (30 m)	tR (LPGC)	Conc. (ppm)	Quant Ion
1.	*o*-Toluidine	3.217	1.207	2	107
2.	*o*-Anisidine	4.42	1.589	2	123
3.	4-Chloroaniline	4.832	1.71	2	127
4.	*p*-Cresidine	5.519	1.92	2	137
5.	2,4,5-Trimethylaniline	5.582	1.935	2	135
6.	3-Chloro-*o*-toluidine	5.902	2.026	2	141
7.	4-Chloro-*o*-toluidine	6.025	2.061	2	141
8.	2,4-Diaminotoluene	7.637	2.523	2	122
9.	2,4-Diaminoanisole	8.89	2.872	2	138
10.	2-Naphthylamine	9.773	3.112	2	143
11.	2-Aminobiphenyl	9.892	3.148	2	169
12.	2-Amino-4-nitrotoluene	10.537	3.325	2	152
13.	4-Aminobiphenyl	12.154	3.767	2	169
14.	*p*-Aminoazobenzene	15.95	4.805	2	197
15.	4,4'-Oxydianiline	16.613	4.99	2	200
16.	4,4'-Diaminodiphenylmethane	16.714	5.016	2	198
17.	Benzidine	16.787	5.034	2	184
18.	*o*-Aminoazotoluene	17.53	5.24	2	225
19.	3,3'-Dimethyl-4,4'-diaminodiphenylmethane	18.23	5.436	2	226
20.	3,3'-Dimethylbenzidine	18.483	5.504	2	212
21.	4,4'-Thiodianiline	19.439	5.765	2	216
22.	3,3'-Dichlorobenzidine	19.795	5.864	2	252
23.	4,4'-Methylenebis(2-chloroaniline)	19.795	5.864	2	266
24.	3,3'-Dimethoxybenzidine	19.905	5.898	2	244

Column	See notes
Standard/Sample	AccuStandard carcinogenic aryl amine mix (AE-00049-R1-10X) AccuStandard 2,4-diaminoanisole (ALR-070S-R2)
Diluent:	Ethyl acetate
Conc.:	20 µg/mL
Injection	
Inj. Vol.:	1 µL split (split ratio 10:1)
Liner:	Topaz, splitless, single taper inlet liner, 4.0 mm x 6.5 x 78.5 (cat.# 23303)
Inj. Temp.:	280 °C
Carrier Gas	He
Detector	MS
Mode:	Scan
Transfer Line Temp.:	300 °C
Analyzer Type:	Quadrupole
Source Temp.:	230 °C
Quad Temp.:	150 °C
Electron Energy:	70 eV
Tune Type:	PFTBA
Ionization Mode:	EI
Instrument	Agilent 7890B GC & 5977A MSD
Sample Preparation	The standards were diluted with ethyl acetate to 20 ppm; analyzed 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-35Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13823)
Temp. program: 200 °C (hold 0.5 min) to 320 °C at 9.5 °C/min (hold 5 min)
Flow: 2.0 mL/min
Scan start time: 2 min
Scan range: 30-300 amu
Scan rate: 10 scans/sec

LPGC-MS Analysis:
Column: LPGC Rxi-35Sil MS column kit, includes 10 m x 0.32 mm ID x 0.25 µm Rxi-35Sil MS analytical column and 5 m x 0.15 mm ID Rxi restrictor factory connected via SilTite connector (cat.# 11806)
Temp. program: 100 °C (hold 0.5 min) to 300 °C at 35 °C/min (hold 5 min)
Flow: 0.9 mL/min
Scan start time: 1 min
Scan range: 35-300 amu
Scan rate: 9.7 scans/sec

Compound list based on EU legislation of Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) Appendix 8 (https://reachonline.eu/reach/en/appendix-8.html). The 3-chloro-*o*-toluidine and 2-aminibiphenyl compounds are not part of the list.

Featured Products

Analytical Column	Maintenance & Accessories	
		
LPGC Rxi-35Sil MS column cat.# 11806	**GC Accelerator** cat.# 23849	**Topaz single taper splitless inlet liner** cat.# 23303

Related Products

Maintenance & Accessories	
	
2 mL, short-cap, screw-thread vial cat.# 21143	**Short-cap, screw-vial closure** cat.# 24495

LPGC Rxi-35Sil MS column kit

- 1.4x faster analysis of phthalates with 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.

Catalog No.	Includes	Units
11806	10 m x 0.32 mm ID x 0.25 µm Rxi-35Sil MS analytical column and 5 m x 0.15 mm ID Rxi restrictor factory connected via SilTite connector	kit

Topaz Single Taper Splitless 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

Catalog No.	Product Name	Units
23303	Topaz, single taper, splitless inlet liner, 4.0 mm x 6.5 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 GCs

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

Catalog No.	Product Name	Instrument	Units
23849	GC Accelerator oven insert kit	for Agilent 5890, 6890, 7890, and 8890 GCs	kit

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

Vials and Caps

Catalog No.	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.

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

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Lit. Cat.# GNFA4235-UNV