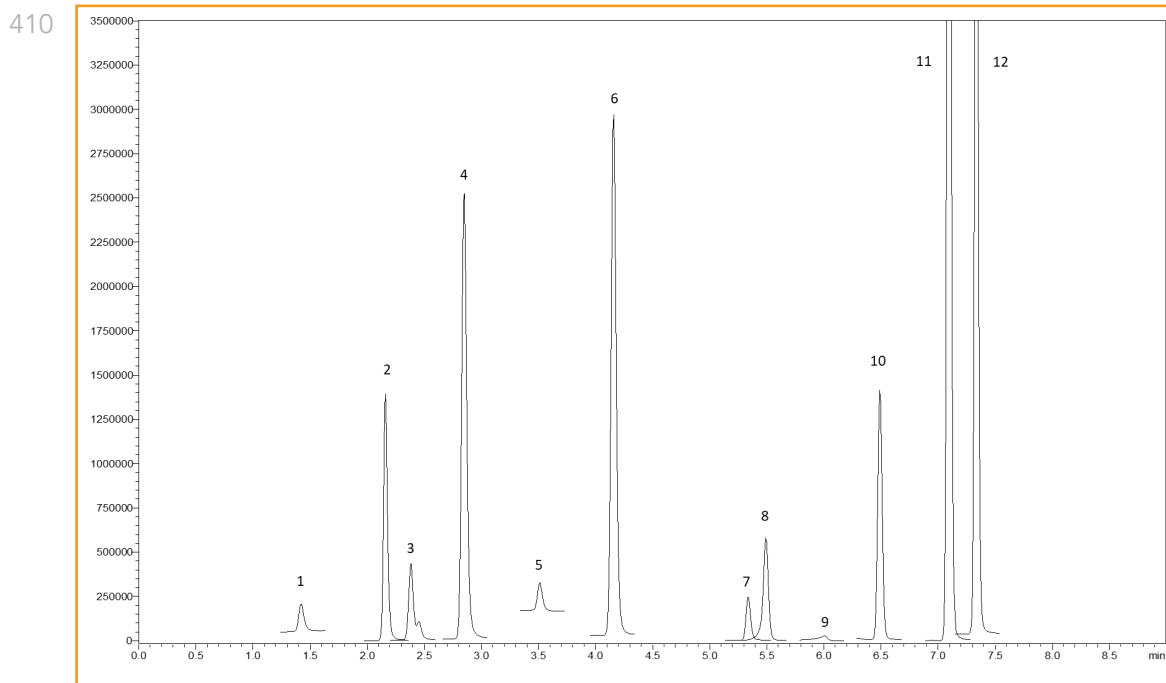




LC-MS Analysis of Nitrosamines on HALO® Biphenyl



TEST CONDITIONS:

Column: HALO 90 Å Biphenyl, 2.7 µm, 2.1 x 100 mm
Part Number: 92812-611

Mobile Phase A: Water/0.1% formic acid

Mobile Phase B: Methanol/0.1% formic acid

Gradient:	Time	%B
	0.00	5
	8.00	100
	9.00	100
	9.01	5
	12.00	5

Flow Rate: 0.4 mL/min

Pressure: 180 bar

Temperature: 45 °C

Injection Volume: 0.6 µL

Sample: GB/T 24153-2009 Nitrosamines Mixture 137
100 µg/mL in Methanol (DRE-A50000137ME) diluted
to 10 µg/mL

Sample Solvent: 90/10 water/methanol

LC System: Shimadzu Nexera X2

MS CONDITIONS:

System: Shimadzu 8060

Detection Mode: DUIS ESI + 1 kV;

Corona Needle 3.5 kV

Nebulizer Gas Flow: 3 L/min

Interface Temperature: 300 °C

DL Temperature: 200 °C

Heat Block Temperature: 200 °C

Drying Gas Flow: 5 L/min

Peak #	Compound	m/z Transition	Retention Time (min)
1	N-nitrosodimethylamine	75.10>43.25	1.42
2	N-nitrosomorpholine	117.10>87.10	2.16
3	N-nitrosomethylethylamine	89.10>61.10	2.38
4	N-nitrosopyrrolidine	101.10>55.10	2.85
5	N-nitrosodiethylamine	103.10>75.05	3.51
6	N-nitrosopiperidine	115.10>69.05	4.16
7	N-nitroso-n-propylamine	131.20>43.10	5.34
8	N-nitroso-N-methylaniline	137.00>107.00	5.49
9	N-nitroso-N-ethylaniline	151.00>121.00	6.01
10	N-nitrosodi-n-butylamine	159.20>57.15	6.49
11	N-nitrosodiphenylamine	199.22>169.05	7.10
12	N-nitrosodibenzylamine	227.00>91.00	7.34

Nitrosamines (most of which are carcinogenic) are a class of compounds formed from reactions between a nitrosating agent, such as nitrites or nitrates, and a nitrogenated precursor, such as a secondary or tertiary amine. They can be found in food, medical devices, industrial products, and the environment and can also be formed as drug substance related impurities in pharmaceuticals. After screening multiple HALO® stationary phases with a standard mix of 12 nitrosamines, a HALO® Biphenyl column was selected since it showed maximum retention and symmetrical peak shape.