

Nitrosamine Analysis by Gas Chromatography and Agilent 255 Nitrogen Chemiluminescence Detector (NCD)

Technical Overview

Introduction

Nitrosamines are known or suspected carcinogens. These compounds potentially appear in cured meat products, tobacco, rubber products, cosmetics, other consumer products, and environmental matrices. Gas chromatography combined with nitrogen chemiluminescence detection offers a reliable, sensitive, and specific means for nitrosamine analysis.

For many years, concern has grown regarding the health effects of nitrosamines, formed from naturally occurring or added nitrates, nitrites, and amines. Several of these N-nitroso compounds are classified as carcinogens by the U.S. Environmental Protection Agency and the Food and Drug Administration. Other studies have indicated that certain nitrosamines are mutagens and can cause birth defects, even at very low concentrations. Clearly, effective analytical techniques are needed by many industries to measure these toxic compounds.

Capillary gas chromatography combined with the Agilent 255 Nitrogen Chemiluminescence Detector (NCD) is a rapid and selective means to perform nitrosamine analysis. Figure 1 illustrates this, and Table 1 lists the compound name and retention times for typical nitrosamines of interest. The Agilent Dual Plasma NCD has been specifically designed with an integral nitrosamines mode. The NCD is highly specific, exhibiting little or no response to hydrocarbons. The NCD also is not subject to quenching, often observed with other

nitrogen specific detectors, such as a nitrogen phosphorus detector (NPD) and a flame photometric detector (FPD). The nitrosamine mode allows users to optimize burner conditions to selectively measure nitrosamines.

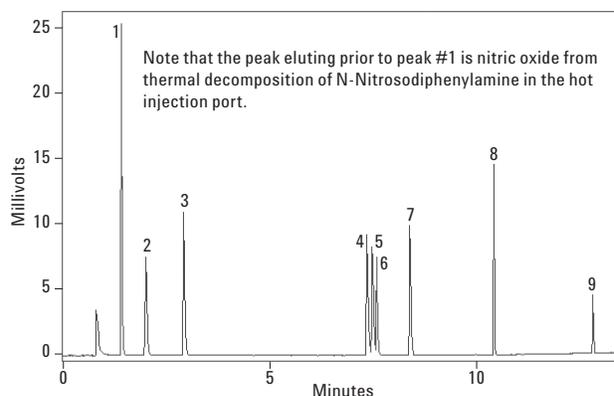


Figure 1. Selected nitrosamines (2 µg/mL levels) by GC/NCD.

Table 1. Selected Nitrosamines Graphed in Figure 1 (2 µg/mL Levels)

Peak number	Compound	Retention time (min)	% RSD
1	N-Nitrosodimethylamine	1.45	4.6
2	N-Nitrosomethylethylamine	2.04	5.1
3	N-Nitrosodiethylamine	2.96	5.6
4	N-Nitrosopyrrolidine	7.39	5.2
5	N-Nitrosomorpholine	7.52	5.4
6	N-Nitrosodi-n-propylamine	7.63	5.2
7	N-Nitrosopiperidine	8.42	4.1
8	N-Nitrosodi-n-butylamine	10.45	4.4
9	N-Nitrosodiphenylamine	12.84	15.4



Another advantage offered by the Agilent NCD is that the response to nitrogen compounds is equimolar, unlike the NPD. The response from NPDs can vary by as much as 30-fold depending on how the nitrogen is bound in the compound. This can complicate calibrations and make quantitation of unknowns highly unreliable. However, the NCD overcomes these issues. In the nitrosamine mode, the NCD response is proportional only to the presence of the thermally unstable nitroso moiety. Excellent linearity and repeatability are also characteristics that are important in a GC detector. The NCD is linear over at least four orders of magnitude and offers good stability over time. An example of the reproducibility obtained from the Agilent NCD is shown in Table 1. Over a period of

4.5 days, 33 injections of a 9-component nitrosamine mix at 100 ppb were performed. The percent relative standard deviation (% RSD) shows exceptional precision over time. Note that the higher deviation in N-Nitrosodiphenylamine is likely due to variability in the well-documented injection port decomposition of that compound. Table 2 summarizes the chromatographic conditions for this analysis.

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Table 2. Chromatographic Conditions for Analysis– HP-5

Injector temperature	200 °C	Column type	HP-5
Initial temp, time	60 °C, 5 min	Column length	30 m
Ramp rate (1), temp (1)	8 °C/min, 85 °C	Internal diameter	0.32 mm
Ramp rate (2), temp (2)	25 °C/min, 180 °C, 3 min	Film thickness	0.25 µm
Injection volume, type	1 µL, 10:1 split	Column flow	1.7 mL/min

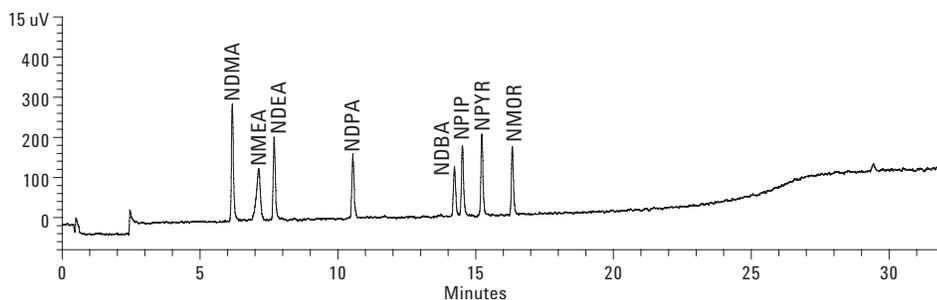


Figure 2. Chromatographic results of analysis–DB-WAX¹.

Table 3. Chromatographic Conditions for Analysis–DB-WAX

Injector temperature	53 °C (oven track)	Column type	DB-WAX
Initial temp, time	50 °C, 1 min	Column length	30 m
Ramp rate (1), temp (1)	6 °C/min, 200 °C	Internal diameter	0.53 mm
		Film thickness	1 µm
Injection volume, type	2 µL, on-column	Column flow	3.5 mL/min, constant

¹ A 0.53-mm id carbowax type phase is sometimes recommended for the analysis of nitrosamines because of its polar selectivity and large sample capacity. The chromatogram shown in Figure 2 is from a nominally 100 ng/mL (customer supplied) standard solution of nitrosamines. The injection volume was 2 µL on-column. This chromatogram demonstrates excellent sensitivity. Table 3 displays the chromatographic conditions for this analysis.

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