Agilent Model 255 Nitrogen Chemiluminescence Detector (NCD) Simultaneous Hydrocarbon Analysis with the NCD and an FID

Technical Overview

Introduction

The nitrogen specificity of the Agilent Model 255 NCD and the universal detection of the flame ionization detector (FID) can provide a detailed analysis of a sample matrix. Chemiluminescence detection enables isolation of nitrogen-containing compounds, while the FID provides universal response for major components in many sample matrices.

Agilent has developed a detector interface to allow simultaneous universal and nitrogen specific detection for gas chromatography. An FID is used for universal detection and the Model 255 NCD is used for the specific detection of nitrogen compounds.

To perform simultaneous NCD and FID analysis, the capillary column is connected directly to the FID following the gas chromatograph manufacturer's installation instruction. The column effluent flows into the FID and then immediately continues through to the stainless steel burner of the NCD. The FID uses oxygen instead of air and the makeup gas is helium rather than nitrogen. Use of oxygen and helium avoids the formation of background nitric oxide in the flame of the detector.

The simultaneous mode is useful when the concentration of nitrogen in individual compounds is greater than 1 ppm. The detection scheme is also useful when the matrix of interest is unknown and there is a need for hydrocarbon data on the sample.

When necessary, the stainless steel burner is easily converted to Direct Analysis mode for better nitrogen sensitivity. The stainless steel burner was designed for use for either simultaneous NCD and FID analysis or specific nitrogen analysis only.

Figure 1 shows a chromatogram with nitrobenzene, 3-methylindole, and 9-methylcarbazole in toluene that demonstrates both the equimolar response and specificity of the NCD. The concentration of nitrogen is approximately 25 ppm for each compound. Also notice the lack of a solvent peak at the beginning of the NCD analysis. A benefit of the NCD is that non-nitrogen containing hydrocarbons in the sample are transparent to the NCD. The sample compounds flow from the column into the FID and the FID measures the hydrocarbon response. A portion of the FID effluent flows directly into the burner of the NCD.

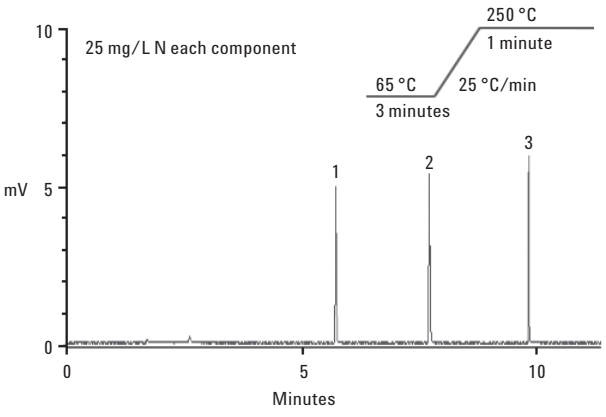

Figure 1. NCD analysis.

Table 1. Precision of Simultaneous NCD and FID Analysis

Number of runs	Compound concentration (ppm)	N concentration (ppm)	Model 255 NCD (% RSD)	FID (% RSD)
Nitrobenzene	218	25	2.3	1.5
3-methylindole	234	25	2.2	1.3
9-methylcarbazole	326	25	2.5	1.5

Notice the number of other small impurity peaks present in the FID chromatogram (Figure 2). The NCD did not detect these peaks since the NCD is specific only for nitrogen. If any of the small compounds contained nitrogen, the NCD would have detected them. Also, with the equimolar response of the NCD, it is possible to determine the concentration of nitrogen impurities in the sample.

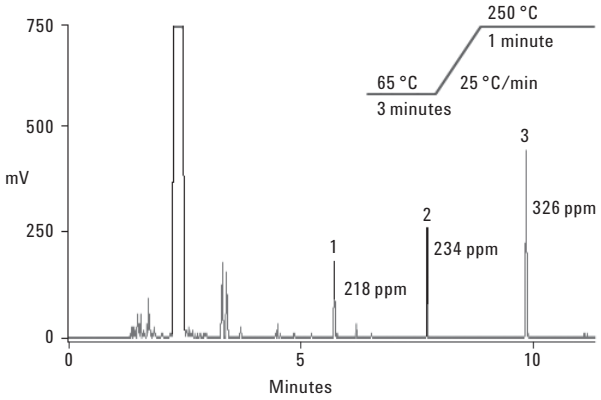

Figure 2. FID analysis.

GC Operating Conditions
(Agilent 6890 with EPC)

Initial temperature:	65 °C for 3 min
Temperature ramp:	25 °C/min
Final temperature:	250 °C for 1 min
Helium carrier:	2.2 mL/min
Split injection:	50:1 split
	250 °C
	2 µL injection volume

NCD Burner Condition

Temperature:	800 °C
Hydrogen flow rate:	25 mL/min
Oxygen flow rate:	10 mL/min
Column:	30 m HP-5, 0.32 mm id
	0.25 µm film thickness

Components

Peak 1:	Nitrobenzene
Peak 2:	Methylindole
Peak 3:	9-Methylcarbazole

The toluene sample with nitrobenzene, 3-methylindole, and 9-methylcarbazole was analyzed 143 times over a 3-day period to demonstrate the stability of the simultaneous NCD and FID analysis. The results of 2.5% relative standard deviation and less demonstrate the stability of the NCD when operated in tandem with the FID (see Table 1). The results also demonstrate that the tandem NCD/FID operation does not affect the performance of the FID. Furthermore, the results also demonstrate the consistency in transferring a fraction of the FID exhaust gases to the NCD.

The nitrogen specificity of the NCD and the universal detection of the FID can provide a detailed analysis. Chemiluminescence detection enables isolation of nitrogen-containing compounds in the sample, while the FID provides universal response for the major compounds of many sample matrices.

For More Information

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Printed in the USA
May 17, 2007
5989-6772EN