Agilent Model 255 Nitrogen Chemiluminescence Detector (NCD) Analysis of Adhesive Samples Using the NCD

Technical Overview

Introduction

The Agilent Model 255 NCD can easily replace a NPD for the analysis of 2-pyrrolidone and 1-vinyl-2-pyrrolidinone in adhesive samples.

A short-term evaluation was performed for the analysis of adhesive samples for two of the starting materials, 2-pyrrolidone and 1-vinyl-2-pyrrolidinone. According to government regulations, the concentration of 1-vinyl-2-pyrrolidinone cannot exceed 800 ppm in the final product.

The primary objective for the analysis of adhesive was to demonstrate the repeatability and stability of the Model 255 NCD. The secondary objective of the study was to prove the NCD could replace the nitrogen-phosphorus detector (NPD) being used for the application. Table 1 compares the NCD and NPD.

The results from the evaluation demonstrated the Model 255 NCD could easily replace a NPD to monitor 2-pyrrolidone and 1-vinyl-2-pyrrolidinone. The gas chromatograph and the Model 255 NCD were calibrated once and not recalibrated again during the 3-day demonstration. Representative precision data are shown in Tables 2 and 3. The gas chromatograph made over 160 injections of the adhesive samples during the 3-day evaluation with the percent relative standard deviation for the Model 255 NCD of less than 4%. The Model 255 was also linear from 20 to 3000 ppm. Figures 1 and 2 illustrate chromatographic response at high and low levels, respectively. Unlike an NPD, the sample matrix did not affect the detector performance.

For the 2-pyrrolidone and 1-vinyl-2-pyrrolidinone analysis, the NCD provides very good short-term and long-term precision. The NCD is unaffected by high levels of the sample matrix, and its use would require less day-to-day maintenance than an NPD. Use of the Model 255 NCD versus an NPD would result in more accurate and precise results and would reduce the level of instrument maintenance required.

Table 1. Comparison of Agilent NCD to NPD

	Agilent 255 NCD	NPD
Response	Equimolar	Non-equimolar
Quenching	No	Yes
Selectivity	> 107 gN/gC	105 gN/gC
Sensitivity	< 5 pg/sec	0.4 pg/sec
Ease of Use	Straightforward	Daily maintenance required

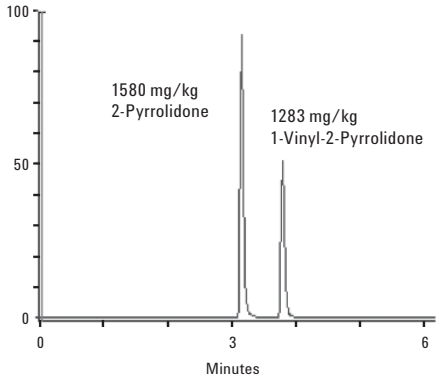

Figure 1. Agilent Model 255 analysis of adhesive standard.

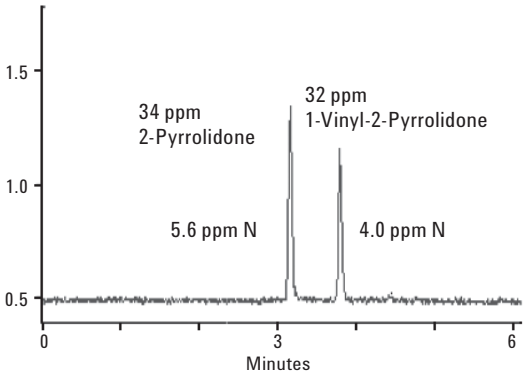

Figure 2. Agilent Model 255 NCD analysis of adhesive sample diluted in tetrahydrofuran.

GC Operating Conditions

(Agilent 6890 with EPC)

Temperature:	160 °C isothermal
Helium carrier:	2.2 mL/min
Split injection:	45.5:1 split
	200 °C
	1 µL injection volume

NCD Burner Conditions

Temperature:	800 °C
Hydrogen flow rate:	25 mL/min
Oxygen flow rate:	10 mL/min
Column:	20 Rtx-5, 0.32 mm id
	3 µm film thickness

Sample Preparation

Samples diluted in toluene
Dilution factors of 1:25 to 1:50

Table 2. Summary of 2-Pyrrolidone in Adhesive Results

Sample number	Number of runs	Dilution factor	Diluted concentration (ppm)	Sample concentration (ppm)	RSD (%)
Adhesive 1	43	1:44	25.0	1094	2.2
Adhesive 2	43	1:41	28.1	1163	2.2
Adhesive 3	43	1:28	34.0	1405	2.2
Adhesive 4	43	1:45	20.3	838	1.7

Table 3. Summary of 1-Vinyl-2-Pyrrolidinone in Adhesive Results

Sample number	Number of runs	Dilution factor	Diluted concentration (ppm)	Sample concentration (ppm)	RSD (%)
Adhesive 1	43	1:44	27.1	1188	2.2
Adhesive 2	43	1:41	15.1	624	2.2
Adhesive 3	43	1:28	32.5	1342	2.2
Adhesive 4	43	1:45	Not detected	Not detected	

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