Application Note No. 044

Nitrile Rubber Analysis Using the Optic 2 Programmable Injector

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Introduction

PVC plastic is blended with nitrile rubber to produce a compound, which is softer and more durable than pure PVC. Nitrile rubber is a polymeric mixture of butadiene and acrylonitrile in the respective ratio 2:1. The aim of this experiment was to quantify the amount of nitrile rubber in a PVC plastic sample. Various weights of nitrile rubber were pyrolysed in the liner of the Optic Injector and the products analysed by gas chromatography. A peak characteristic of the chromatograms was isolated and a calibration of peak area against sample weight constructed. A known weight of PVC plastic was then pyrolysed under the same conditions and using this calibration the amount of nitrile rubber in the pyrolysed PVC sample was determined.

Experimental

The Optic injector conditions were as follows:

- Mode: Desorption
- Equilibration Time: 0 mins
- Sweep Pressure: 0.00 Bar
- Sweep Time: 0 Minutes
- Initial Temperature: 50 ^{o}C
- Ramp Rate: 16 ^{o}C/Sec
- Final Temperature: 600 ^{o}C
- End Time: 37 mins
- Desorption Pressure: 0.00 Bar
- Desorption Time: 1.5 mins
- Transfer Pressure: 0.65 Bar
- Transfer Time: 2 mins
- Final Pressure: 0.55 Bar

A 0.5 mg sample of nitrile rubber was placed in a clean, empty injector liner. Once the liner was sealed, run cycles on the GC and injector were started. The analysis was then repeated for a similar sized sample of PVC. A peak common to both chromatograms was then identified on which to base a calibration. Chromatograms of pyrolysed nitrile rubber and the PVC sample can be seen in Figures 1 & 2.

The peak selected had a retention time of 20.6 minutes. The next stage of the analysis was to identify the species responsible for this peak. Judging from the composition of nitrile rubber it is unlikely that a peak with such a long retention time and relative abundance is due to a low molecular weight hydrocarbon fragment. It was therefore suspected this peak was a cyano containing species from the acrylonitrile section of the polymer, as cyano containing compounds are usually quite polar and are likely to be more strongly retained on the CPS1119 capillary column.

Samples of nitrile rubber ranging from 0.16 mg to 1.18 mg were pyrolysed as described and the peak areas of the peaks with a retention time of 20.6 minutes were recorded.

Results and Discussion

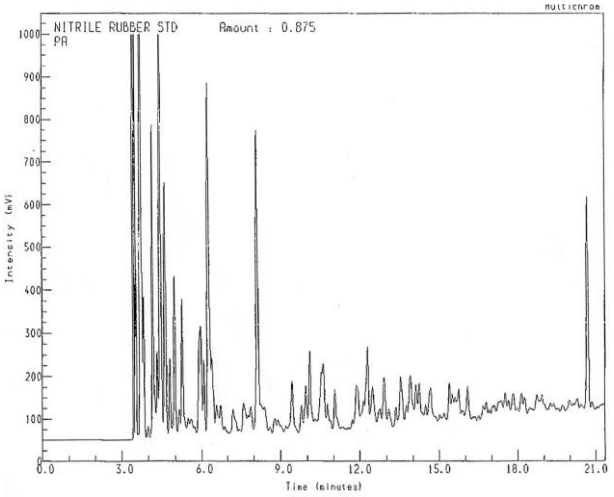

Figure 1: *Pyrolysed nitrile rubber standard*

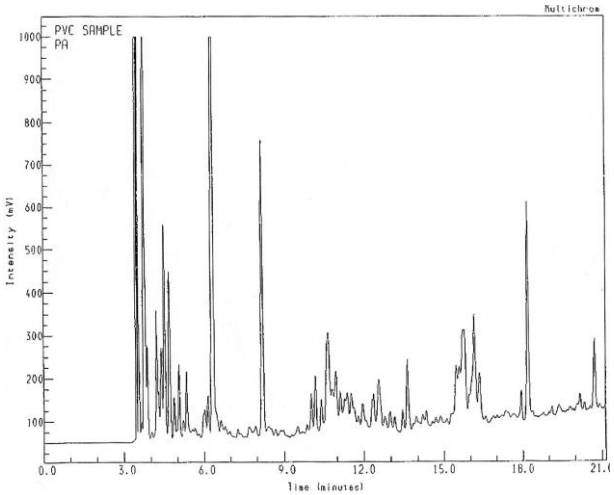

Figure 2: *Pyrolysed PVC sample*

A calibration graph for the characteristic nitrile rubber peak (Rt = 20.6 minutes) was constructed and can be seen in Figure 3. This was used to quantify the amount of nitrile rubber in thirteen PVC replicate samples.

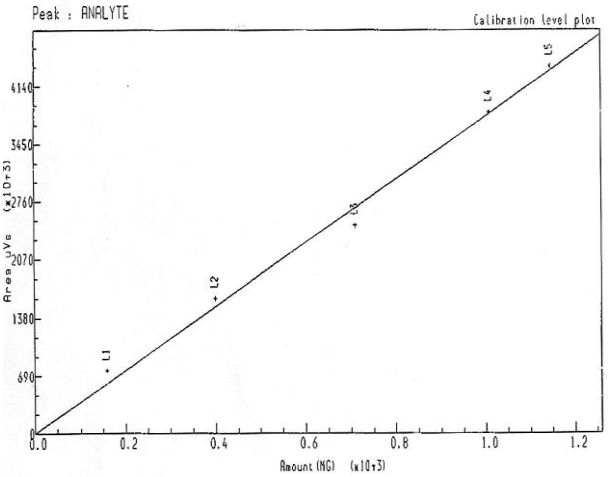

Figure 3: *Calibration graph for characteristic nitrile rubber peak*

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The quantification results are shown in the table below:

Sample Number	Weight of PVC (mg)	% w/w Nitrile Rubber
1	1.09	30
2	1.34	30
3	2.34	23
4	0.78	32
5	1.01	28
6	0.72	28
7	0.33	27
8	1.63	22
9	2.95	25
10	0.74	30
11	1.22	30
12	0.75	28
13	0.73	30

Figure 4: *Quantification results for 13 samples using Optic pyrolysis*

At 95% confidence:

- Mean % nitrile rubber in PVC = 28 ± 6% w/w
- Standard deviation = 3%
- Relative standard deviation = 11%

The percentage weight of Nitrile rubber in the PVC samples determined by "Khjeldal" total nitrogen gave a value of 29% w/w.

Conclusion

The Optic programmable injector has shown to be suitable for the quantification of nitrile rubber in PVC by this method involving in-liner pyrolysis. The results are accurate, 28 ± 6% w/w using the Optic compared to 29% w/w as determined by the Khjeldal total nitrogen method. However, the RSD is slightly high at 11%, although for this analysis this is acceptable.