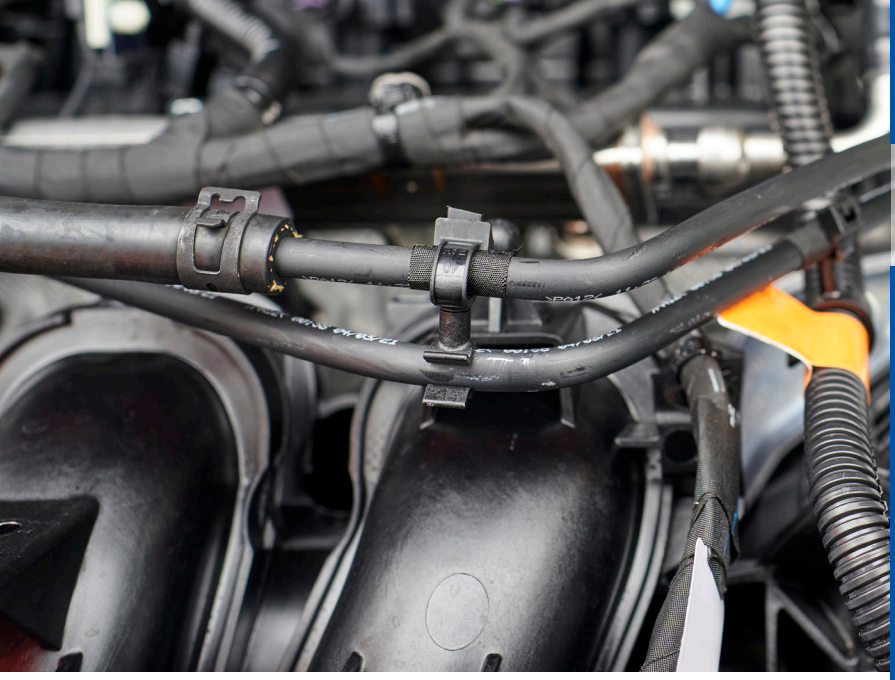

APPLICATION BRIEF

Hyphenation

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TG-IR Analysis of a Cable Used in the Automotive Industry

Introduction

Polymers are used throughout the automotive industry in areas such as the fuel and braking systems, electrical components, and upholstery on the interior of a vehicle. This work describes the analysis of the inner sheath of an insulating cable used in the automotive industry using thermogravimetric analysis coupled with infrared spectroscopy (TG-IR).

Experimental

The only sample preparation required was the removal of the outer sheath of the cable for analysis. Approximately 17 mg of sample was analyzed, thus ensuring the release of a sufficient quantity of gas for infrared analysis. The measurement was carried out using a PerkinElmer TGA 8000™ hyphenated to PerkinElmer Spectrum 3™ FTIR via a TL8000e transfer line (Figure 1). The experimental parameters are shown in Table 1. Spectra were collected every 10 seconds.

Figure 1: PerkinElmer TG-IR system coupled using a TL8000e transfer line.

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Table 1: Data collection parameters used for analysis of cable sheath.

Parameter	Value
Thermogravimetric Analysis	
Purge Gas	Nitrogen (Hold in Air for 10 min at 850 °C)
Total Purge Gas Flow (mL/min)	90
Temperature Range (°C)	35 – 850
Scan Rate (°C/min)	15
Infrared Spectroscopy	
Spectral Range (cm^{-1})	4000 – 600
Spectral Resolution (cm^{-1})	4
Number of Scans Per Spectrum	2
Transfer Line	
Transfer Line Temperature (°C)	280
Pump Flow Rate (mL/min)	70

Results and Discussion

The sample measured in this experiment demonstrated four weight loss events at 308, 496, 627 and 721 °C. The weight loss and derivative weight loss curves for the polymer sheath are shown in Figure 2, clearly indicating the four-step weight loss profile. In addition, the Gram-Schmidt (GS) profile of absorbance vs temperature is shown, demonstrating the correlation between the weight loss steps and the corresponding FTIR data.

The gas evolved during the first, and largest weight loss (40.7%) produced the spectrum shown in Figure 3. The fine band structure between 3150 and 2600 cm^{-1} corresponds to the presence of HCl gas which provides evidence that the sample is a chlorinated polymer, such as poly(vinyl chloride).

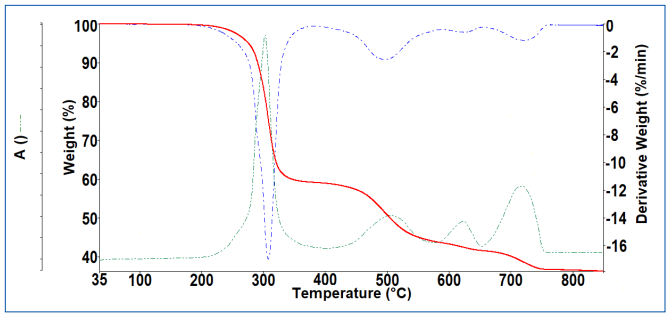

Figure 2: Weight loss, derivative weight loss and Gram-Schmidt curves for the polymer sheath determined by TG-IR.

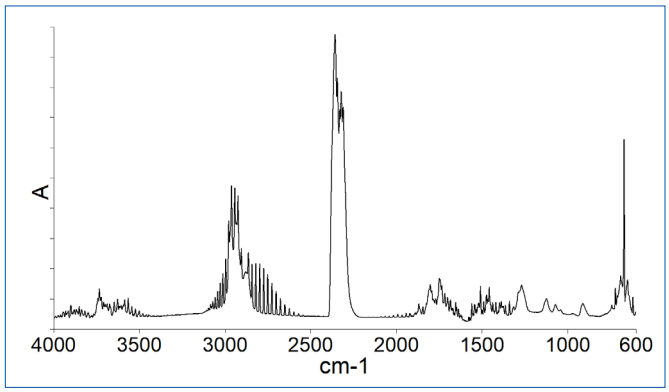

Figure 3: IR spectrum collected during the first weight loss of the cable sample.

In order to identify minor components of the evolved gas, multivariate curve resolution (MCR) was used. MCR, similarly to principal component analysis (PCA) aims to produce a series of factors that best describe the variation in the data. Unlike PCA, MCR also attempts to uncover the spectra of the components causing this variation. The result of applying the MCR algorithm in Spectrum 10™ are shown in Figure 3.

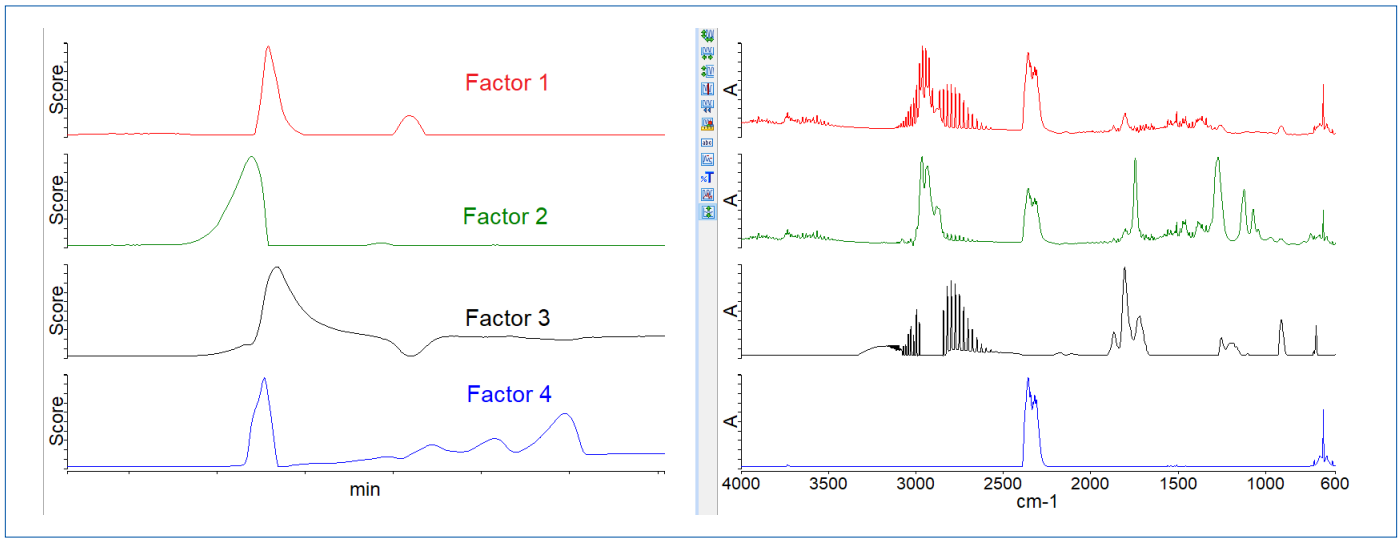

Figure 4: Results from applying an MCR algorithm to the TG-IR data obtained from the cable insulating layer.

The first factor corresponds to the release of HCl and CO_2, both of which are expected during the degradation of plasticized PVC. Likewise, factor 4 accounts for the release of CO_2 throughout the degradation, following the Gram-Schmidt profile seen in Figure 2. Factors 2 and 3 were searched against a vapour phase library. Factor 2 was identified as dioctyl phthalate (DOP), a plasticizer commonly used in PVC, with a search score of 0.975. Factor 3 has two aspects which must be considered. First, the fine band structure around 3000 cm^{-1} which corresponds to HCl, showing that factor 3 is also accounting for some of the variation caused by this component. The peaks seen around 1700 cm^{-1} correspond to an anhydride. This is supported by the search result, phthalic anhydride (0.65), a known degradation product of DOP.[1]

Summary

TG-IR is a technique which allows users to measure the thermal degradation of a material while simultaneously measuring the evolved gases. This provides in depth information about the sample which would not be achievable with other techniques or would require extensive sample preparation. In this instance, the identification of DOP, a plasticizer which is banned in certain settings.[2] Furthermore, PerkinElmer's Spectrum 10™ and Pyris™ software packages facilitate streamlined and powerful data analysis.

References

1. K. Saido, S. Motohasi, T. Kuroki, T. Ikemura, M. Satomi, M. Kirisawa, Chem. Pharm. Bull., 1979, 27, 3140-44.
2. https://cen.acs.org/articles/93/i25/Regulators-Retailers-Raise-Pressure-Phthalates.html (accessed 15/02/2023).

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