Fingerprinting of Polymer Manufacturers by Syringeless Injection GC/MS

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Introduction

During the production of polymers a number of residual volatile organic compound become trapped within the material[1]. Once isolated and separated, the volatile organics can act as a fingerprint to a specific manufacturer or production location.

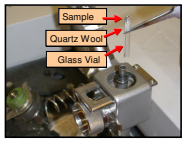
Syringeless injection technique allows polymers to be thermally desorbed within the injection port of a gas chromatograph. The thermally extracted volatiles are continually swept into a gas chromatography column for collection and separation[2]. The close proximity of sample to the GC column allows for very efficient transfer of compounds at trace levels, which in turn results in highly sensitive and reproducible results.

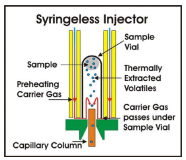
Installation

The thermal desorption device is inserted in place of an injection port liner. Carrier and vent lines attach to the device

Load Sample

Samples are placed into a glass vial with a small amount of quartz wool to prevent the sample from falling out.



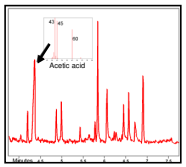


Inject Sample

Switching the Injection port between split and splitless operation allows the sample to be injected into or expelled out of the injection port

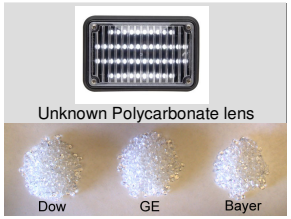
Analyze Sample

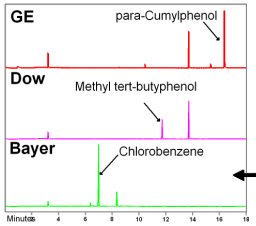
Excellent replication, resolution and sensitivity due to a short (~ 2 cm) volatiles path length from sample to GC column inlet.



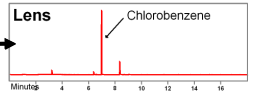
Case study #1

Identify source of polycarbonate used to make lens





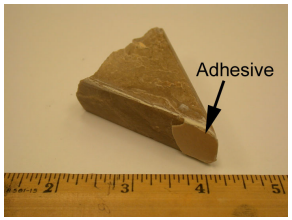
The lenses were manufactured using Bayer Polycarbonate.
A fingerprint of each manufacture is composed of residual trace organic compounds from ~0.5mg of polymer extracted at 300C for 2 minutes.



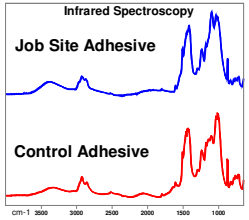
Case Study #2

Product identification of adhesive material

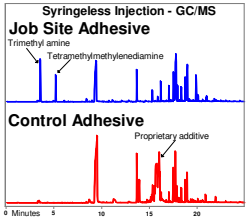
Unknown Job Site adhesive



Infrared analysis inconclusive.
High filler content prevents match.



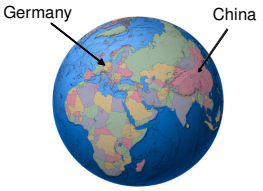
Control adhesive was not used at job site. Propriety additive missing.



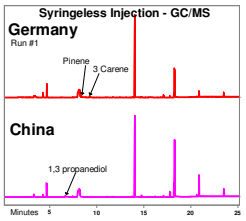
Case Study #3

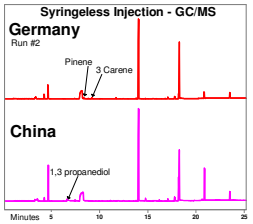
Location of (thermoplastic polymer) production

Manufacturing plant location



Polymer manufactured in Germany can be distinguished from the same manufacturers plant in China. Highly reproducible results, run 1 and 2





Conclusions

Successful polymer fingerprinting requires reproducible thermal extraction time, temperatures, and efficient transfer of volatiles into a gas chromatography column. Utilizing the Injection port to heat the sample, allows for the shortest possible distance between the sample and the GC column. As volatiles are thermally desorbed from the polymer they are swept by carrier gas into the GC column for separation and identification by mass spectrometry.

Given adequate control samples, the manufacturer of a specific polymer maybe identified by analyzing the residual volatiles trapped within the polymer during production. In one example we were able to differentiate between the same thermoplastic polymer produced at two different locations from the same manufacturer.

In addition to analyzing residual volatiles trapped during polymerization, proprietary additives within compounded polymers can also be used, as an indicator to fingerprint a specific product and or manufacturer.

This thermal extraction technique has also been used for failure analysis, surface analysis, odor analysis in static or dynamic collection modes and high temperature GC applications.

Cited Literature

1 "Polymer Devolatilization" Edited by Raymon J. Albalak, 1996, Marcel Dekker, Inc. Intro. Pg#1

2 M. Ezrin and G. Lavigne, "Failure Analysis Using Gas Chromatography/ Mass Spectroscopy, " Society of Plastic Engineers. Annual Technical Conference Proceedings, (1991) 2230-2233

Acknowledgments

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