

Using FT-IR Spectroscopy with Gel Permeation Chromatography

Technical Overview

Introduction

Combining gel permeation chromatography (GPC) with Fourier transform infrared (FT-IR) spectroscopy is a powerful technique for the analysis of copolymers and polymer blends. However, for FT-IR to be successfully employed in GPC, a linear concentration response is required, allowing chromatograms to be generated from the FT-IR data for GPC calculations.

In GPC, polymer molecules are separated on the basis of molecular size in solution. In the simplest form of the experiment, a detector is employed to determine the concentration of material eluting from the GPC column. FT-IR spectroscopy is a widespread technique used for investigating the functional-group properties of materials. The coupling of these techniques allows the composition of copolymers and blends to be investigated, providing a wealth of information.



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To investigate the use for FT-IR as a concentration detector in GPC, a system was set up with both differential refractive index detector and FT-IR using the Agilent RTGPC-FTIR interface flow cell. A series of polystyrene and polymethylmethacrylate (PMMA) standards were injected at varying concentrations and time-resolved spectra were obtained across the elution profile of the material, see Figure 1. The overall spectral response was then summed for each material and from these data calibrations of spectral response versus concentration were determined. Similar data were generated from the differential refractive index detector peak area response for comparison. Figure 2 shows the PMMA calibration plots for both DRI and FT-IR detectors, and Figure 3 the plots for polystyrene.

Columns: 2 x PolyPore,
300 x 7.5 mm
(p/n PL1113-6500)
Eluent: Dichloromethane
Flow Rate: 1 mL/min
Injection Volume: 500 µL
Sample Concentration: 2 mg/mL
Temperature: Ambient
Detection: Agilent PL-GPC 220
(DRI and Agilent RTGPC-
FTIR Interface fitted in an
FT-IR Spectrometer)

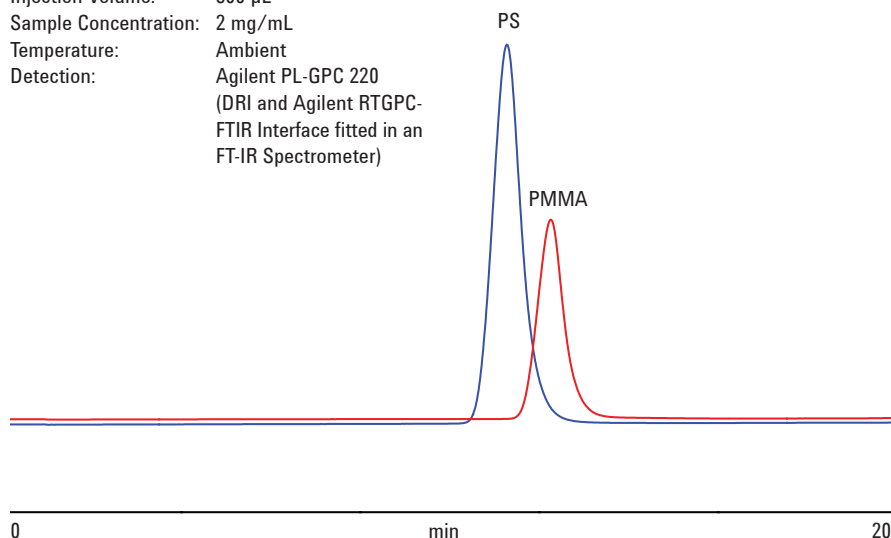


Figure 1. Overlaid chromatograms of polystyrene and polymethylmethacrylate standards

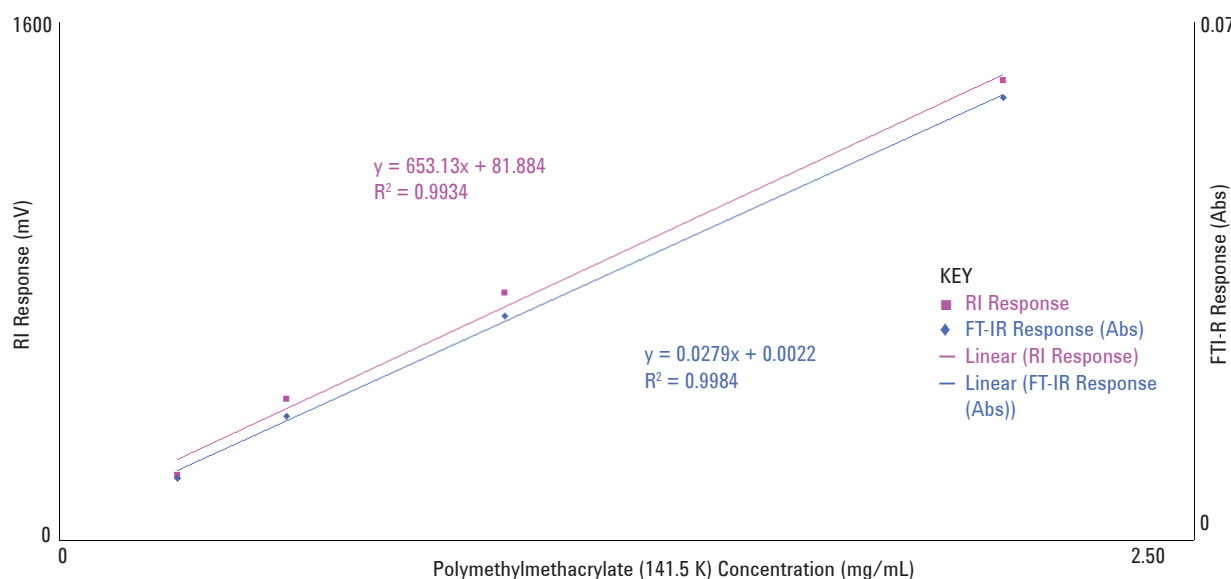


Figure 2. Calibration curves of polymethylmethacrylate for DRI and FT-IR detectors

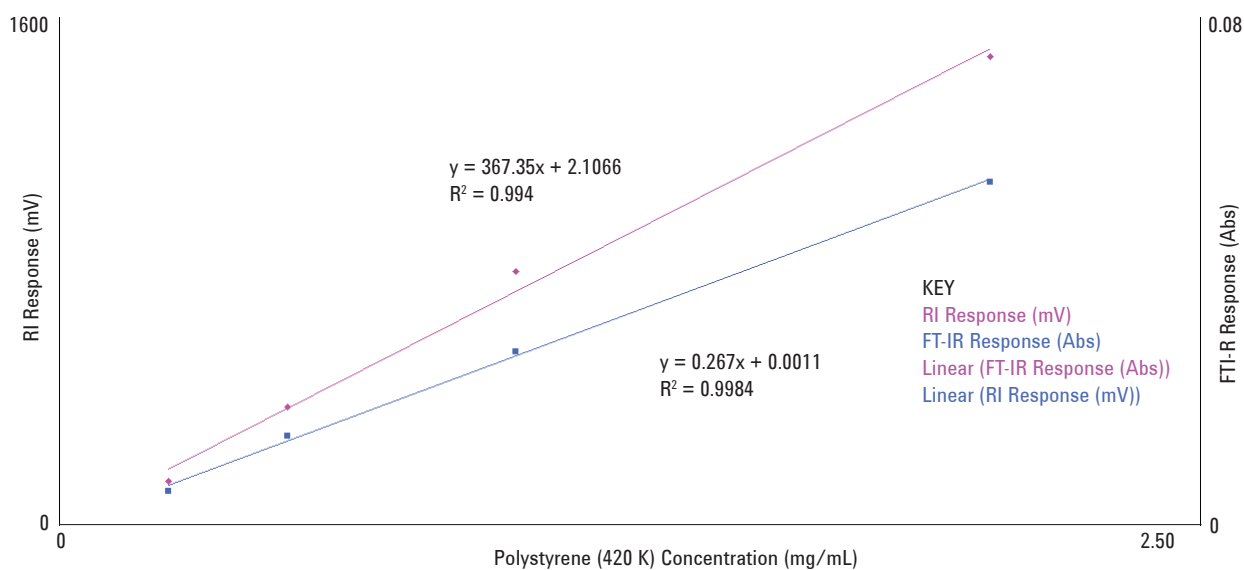


Figure 3. Calibration curves of polystyrene for DRI and FT-IR detectors

Conclusions

The Agilent RTGPC-FTIR interface permits FT-IR to be successfully and routinely coupled to gel permeation chromatography, allowing time-resolved spectra to be obtained of materials eluting from a GPC column set. The spectral data can then be used to generate chromatograms to enable full GPC analysis of materials.

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