



DiscovIR-GCTM

Deposition and Detection System

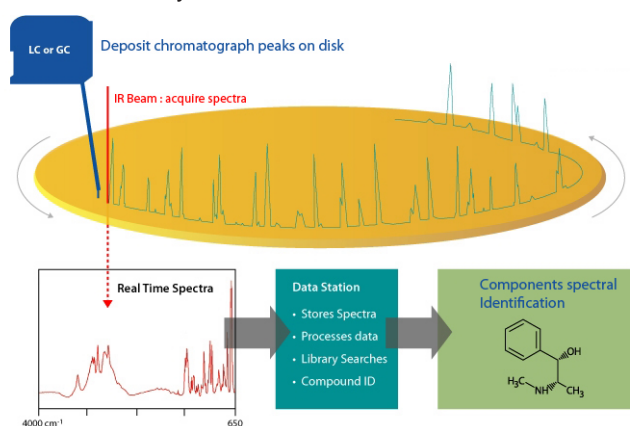
Application Note 017
December 2007

Characterization of a Hot-Melt Adhesive by LC-IR

The DiscovIR-LC is a powerful new tool for materials analysis.

When connected to the outlet of a LC column, the DiscovIR removes the mobile phase and deposits LC eluants as a continuous track on an infrared transparent substrate.

The built-in interferometer simultaneously captures a set of time-ordered infrared spectra from the deposited track.



The resultant map of molecular structure of all sample components provides for immediate identification of components.

This application note shows how this technology is applied to characterizing hot-melt adhesives. Present industrial manufacture makes use of many tons/year of hot-melts. Hot-melts have multiple diverse uses. As a result, there are numerous components that may be used to optimize properties for a given application. Polymers provide cohesive bond strength, while lower molecular weight tackifier materials promote wetting of bonding surfaces, control of melt/set characteristics, and flexibility. Hot-melt adhesive product composition determines product performance, and composition control is key to optimizing that performance.



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Experimental

A sample of a demonstration hot-melt glue stick[†] was dissolved in chloroform, and an injection was made onto a GPC column, using chloroform as the mobile phase.

Column Jordi 500 angstrom DVB column, 50 cm X 10 mm ID

Mobile Phase: Chloroform, 1 ml/min

Sample: 40 µl injection of 1mg/ml hot-melt glue stick dissolved in chloroform

Column eluant was fed to the DiscovIR. The eluant was deposited as a track, which was immediately scanned to generate a time-ordered set of infrared spectra.

Results

The figure below shows infrared chromatogram generated by the DiscovIR. The trace is a display of integrated absorbance over the whole mid-IR range.

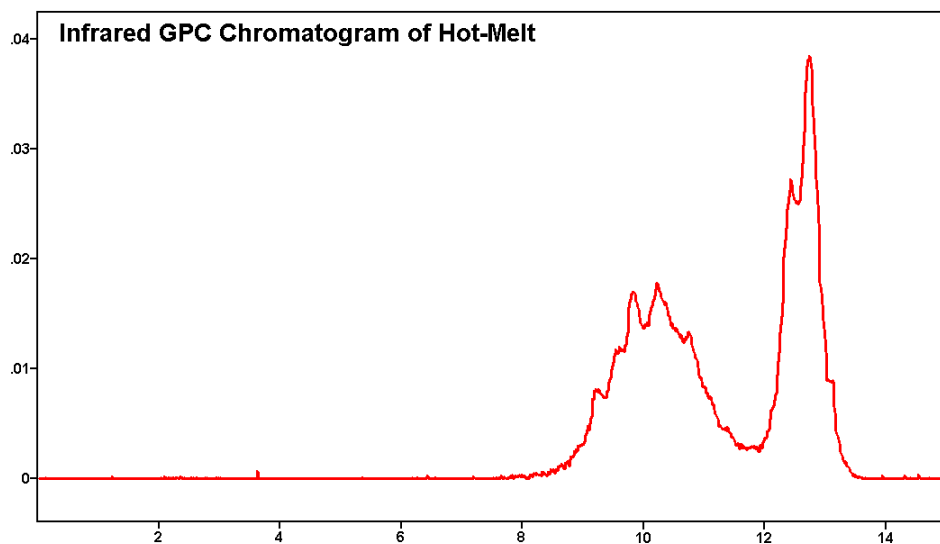


Figure 1 Hot-Melt Chromatogram

The elution profile shows a broad early elution peak, followed by what could be two co-elutants that are partially resolved.

A spectrum is displayed (Figure 2, upper spectrum) for the solute eluting at 10 minutes. This spectrum shows a good spectral library match (Figure 2, lower spectrum) for ethylene-vinyl acetate polymer.

[†] Spectra Analysis wishes to thank Janice Mayer of Forbo Adhesives, Durham NC for the demonstration sample and materials, and also for her technical advice on adhesive technology.



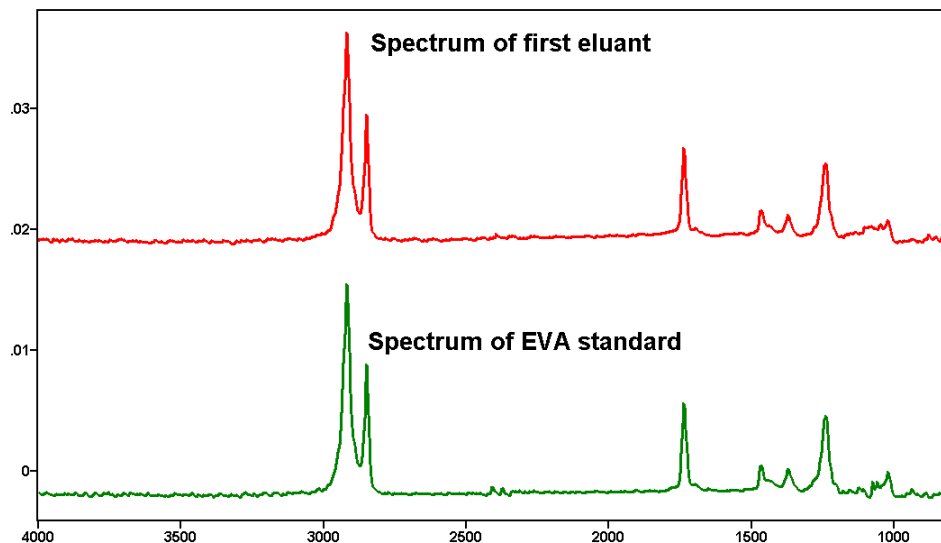


Figure 2 Spectra of polymer component and a library standard

Referring back to the Figure 1, the late elution “doublet” peak at 12 – 13 min elution time is typical of a pair of partially resolved components. Infrared functional group chromatograms can be very useful in this regard. Cursory inspection of spectra in the eluant around 12 minutes showed a probable carbonyl band. An infrared chromatogram of this wavenumber was generated. In Figure 3 one sees both the general IR chromatogram and also one that was extracted at 1724 cm^{-1} wavenumber.

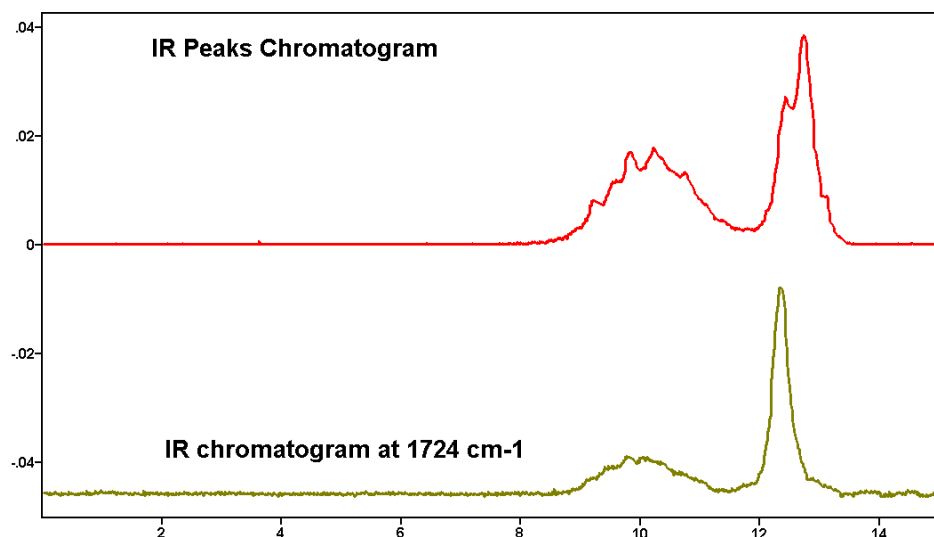


Figure 3 Specific spectral peak chromatogram

It is immediately apparent that the 1724 cm^{-1} band is not present in the late eluting fraction of the doublet. Two spectra were sampled at the leading and trailing shoulders of this double peak.

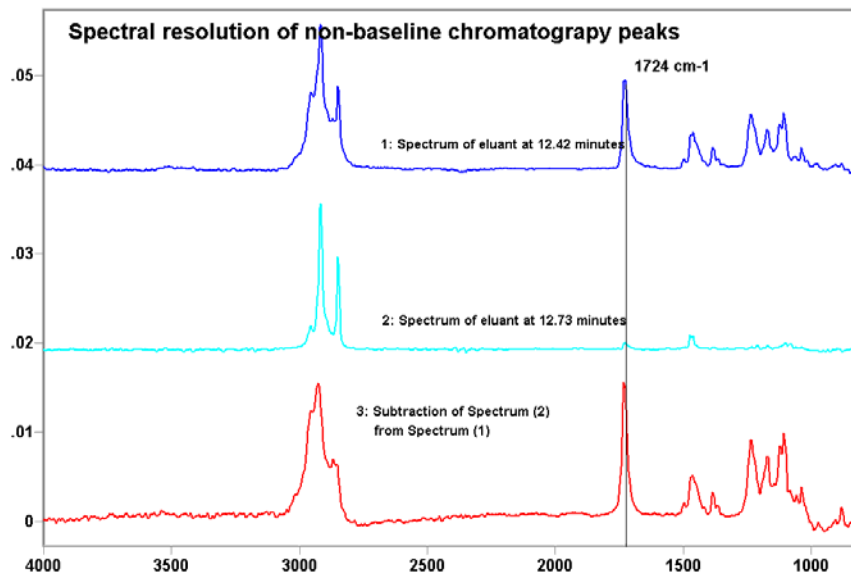


Figure 4 Spectra of the GPC doublet peak

These are two different materials, but there is some co-elution overlap. To deconvolve the two spectra, a weighted intensity of the later elution spectrum was subtracted from the earlier elution spectrum, and vice versa. The late eluting resultant spectrum contains entirely C-H bond absorption bands, typical of an alkane wax.

This spectral subtraction technique eliminated the artifacts of co-elution and yielded clean spectra of the two discrete components. Spectral matching can be done on such samples, by comparing sample spectra to known standards, or to a spectral library which will automatically search for the most similar spectra from the library data base. For specialized applications the user can enter spectra of specific materials into a personal library. Comparison of the subtracted spectrum to a spectral library provides a very good match to a Glycerol rosin ester.

As a confirmation to the spectral matches, a set of the individual identified components were independently chromatographed under the same GPC conditions. Chromatographs are shown in the figure below. It can be seen that the elution times of these pure components match the elution peaks of the mixture. The chromatogram elution times provide supporting evidence to the spectral matches obtained.



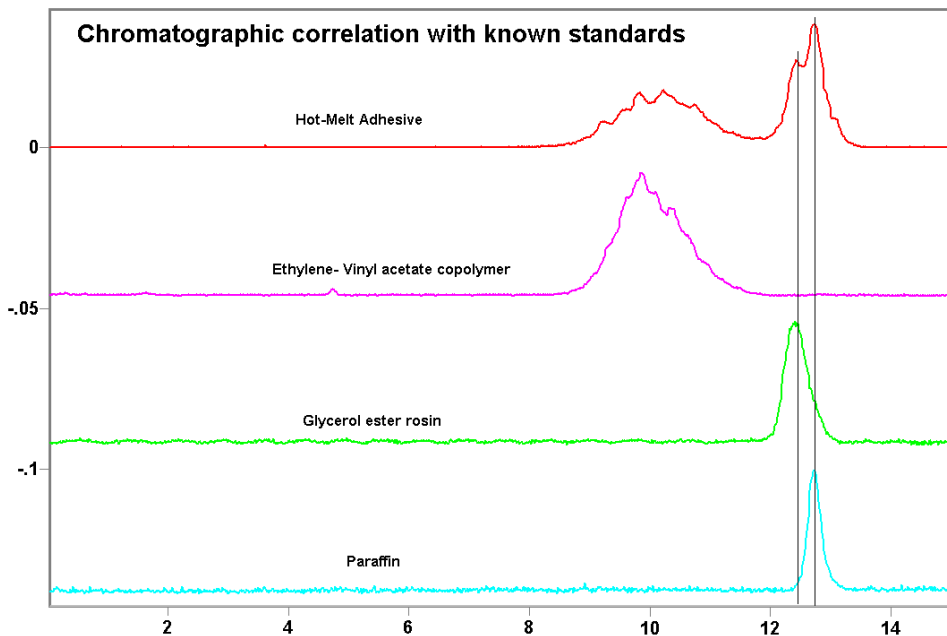


Figure 5 Spectral identification is supported by elution times of discrete standards

Conclusions

This data illustrates the ability of Chromatography + DiscovIR to rapidly obtain identification of materials in a multi-component adhesive sample. All data was collected during the course of a single GPC chromatography run. Spectra from any part of the chromatograph can be examined for material identity. There is an enormous gain in productivity when compared to the classical methods of prep-HPLC fraction collection and sample preparation of the many discrete fractions for spectral analysis.

Benefits of DiscovIR Technology

- Direct interface to HPLC/GPC instruments.
- Automated LC run-time spectral data acquisition of all portions of the LC column eluant.
- FTIR included in compact footprint (24 in. total bench space).
- DiscovIR software includes spectra interpretation/identification capability.
- FTIR spectra are solid phase transmission spectra compatible with available spectral libraries.
- Sample injection to materials deformation typically 3 hours.



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