LABCONNECTIONS

LC-TRANSFORM®

INTERFACING CHROMATOGRAPHY WITH SPECTROSCOPY

Analysis Of Urethane Polymers
AN-3

Urethane formulations are complex mixtures of several oligomeric components. A complete characterization of such systems is a time consuming procedure involving preparative chromatographic isolation of individual components, which can then be identified or characterized by spectroscopy. The separation and isolation of the individual components of a formulation greatly helps in unequivocal identification by IR, NMR, MS, etc. Thus individual polymers can be functionally and structurally characterized (e.g., polyethers, polyesters, other homo or copolymers.

Additives such as UV stabilizers, antioxidants, and diluents can also be isolated and characterized with chromatography. In the case of ethylene oxide (EO) and propylene oxide (PO) based polyethers, information about PO/EO ratio, primary and total hydroxyl content of the components can be ascertained together with molecular weights (GPC calculated) and functionality.

ANALYSIS OF AN UNKNOWN URETHANE POLYMER FORMULATION

A sample was solubilized in tetrahydrofuran (THF), and run on a room temperature GPC column. The outlet of the GPC column UV detector was connected to the Flow Divider of the LC-Transform. Sample collection conditions were as follows:

SAMPLE COLLECTION CONDITIONS

Sample concentration	0.4%
GPC flow rate	1ml/min
Mobile phase	THF(w BHT)
Flow split ratio to LC-Transform	35:1
Sheath gas temp	49°C
Sheath gas flow	20
Nebulizer gas flow	25

Disc rotation	6.5°/min

SPECTRAL ANALYSIS

The sample disc was placed in the LC-Transform Scanning Module. The FT-IR instrument was a Nicolet 20SXB. Using GC-IR software, 750 spectral data sets (16 scans/set) were obtained at 8 cm-1 resolution. With the use of continuous collection software, there was no need to search the disc for individual component peaks; they were all collected in one scan run.

Because of the polymeric nature of the sample the chromatographic deposit on the disc appears as a smeared track, with individual solute components not visually distinguishable. The chromatograph detector output is shown in figure 1., and indicates four components.

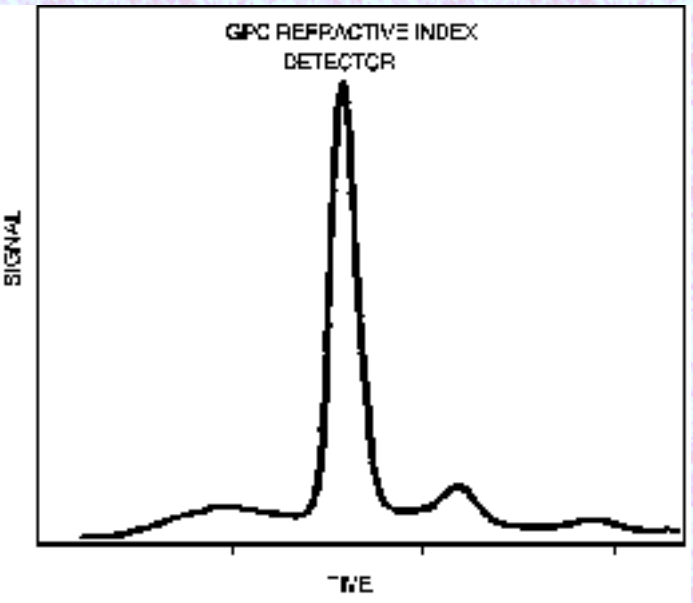
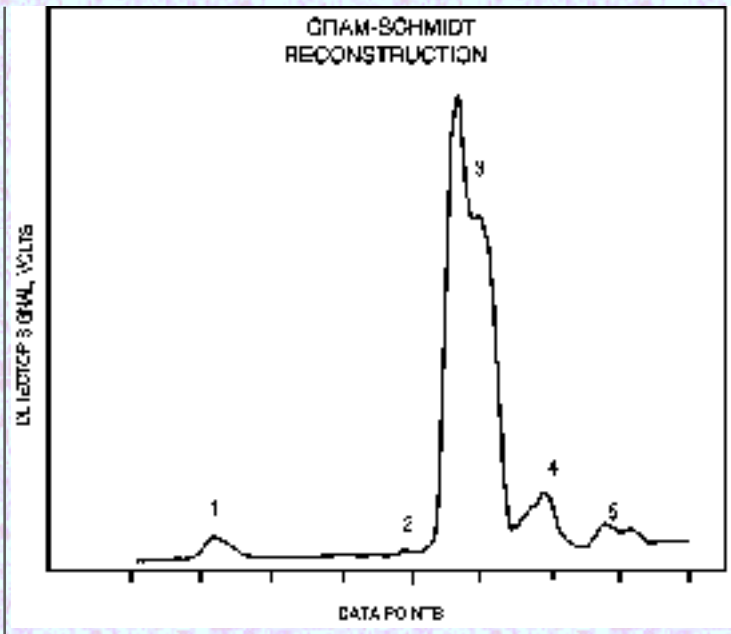
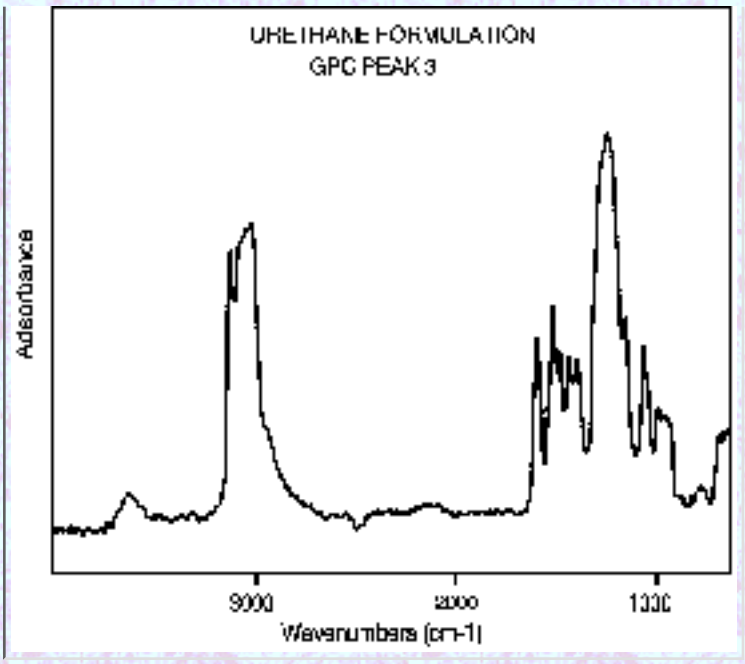
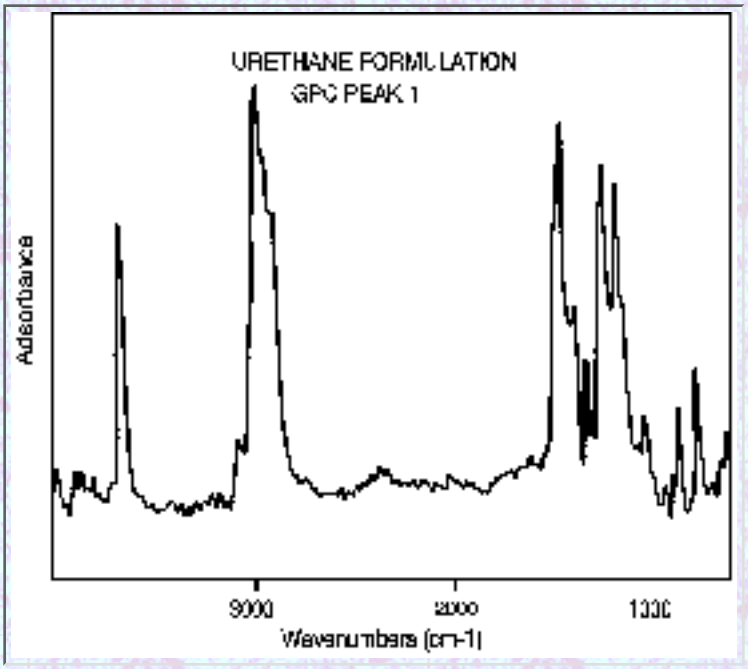


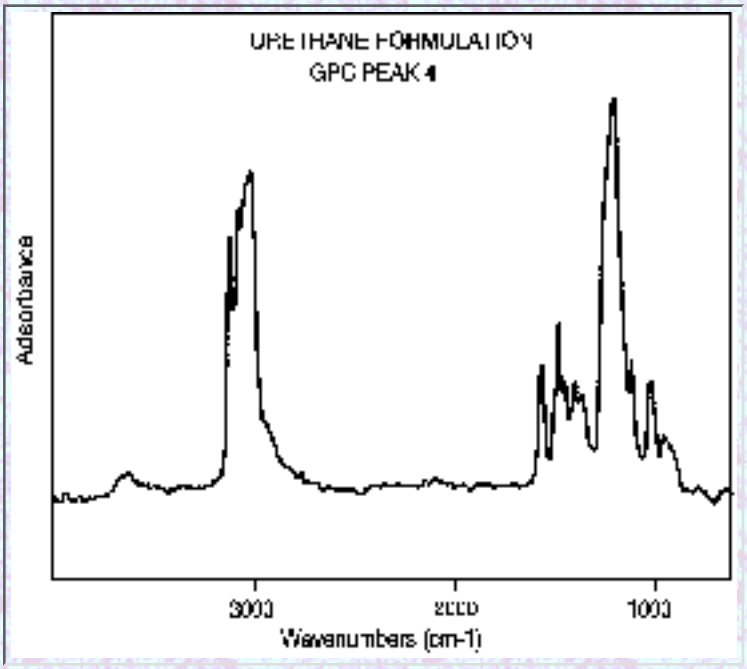
Figure 1. GPC detector response

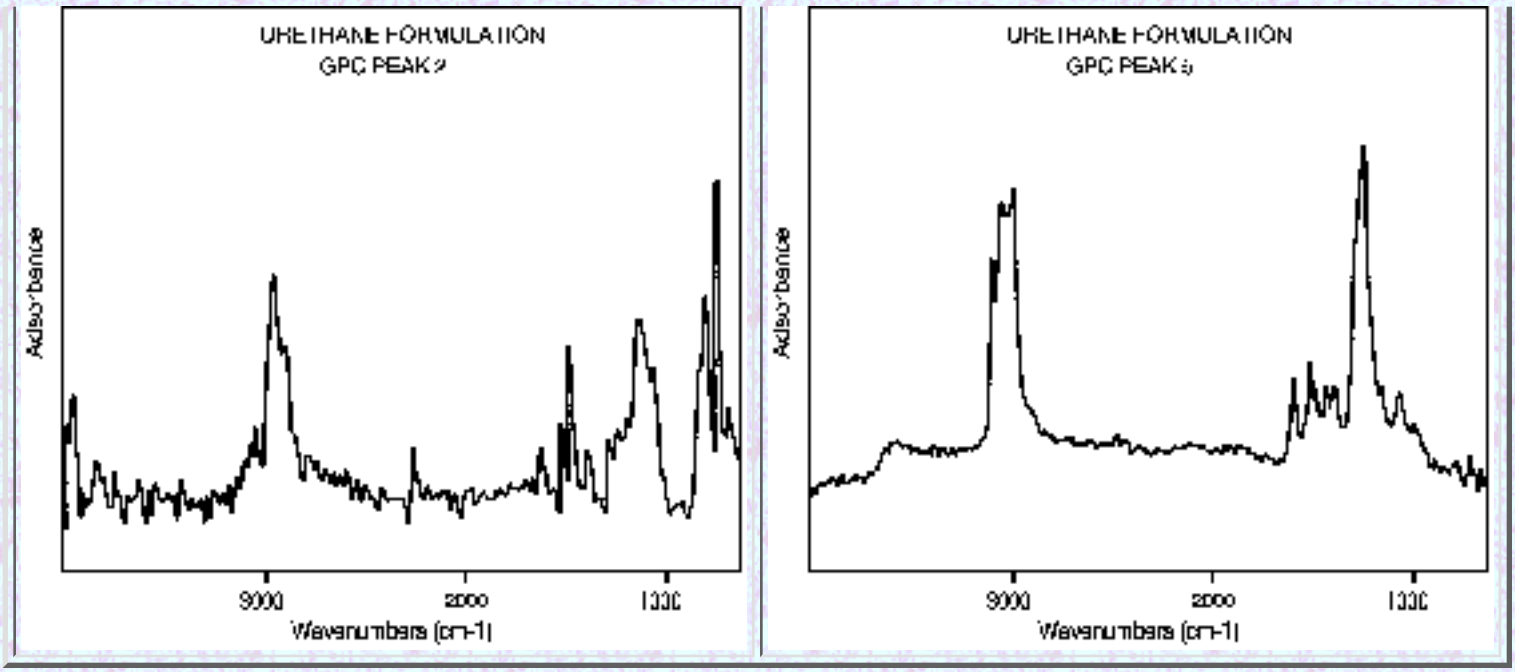
Figure 2. is a Gram-Schmidt reconstruction of the collected scan sets. This is the virtual equivalent of using the LC-Transform as a chromatograph detector. Note that an additional peak was observed which was not picked up by the UV detector. (The apparent double peak #3 was an artifact of an instrument adjustment made during the data collection.) The spectra of the five solute peaks are shown.


GRAM-SCHMIDT
RECONSTRUCTION
1
2
3
4
5
DATA POINTS


URETHANE FORMULATION
GPC PEAK 3
Absorbance
3000
2000
1000
Wavenumbers (cm-1)


URETHANE FORMULATION
GPC PEAK 1
Absorbance
3000
2000
1000
Wavenumbers (cm-1)


URETHANE FORMULATION
GPC PEAK 4
Absorbance
3000
2000
1000
Wavenumbers (cm-1)



CONCLUSIONS

Based on the interpretation of spectral data the 5 peaks of the Gram-Schmidt chromatogram were identified as:

- Peak #1 BHT
- Peak #2 alkylisocyanate
- Peak #3 polyglycol(mw 12000) *
- Peak #4 polyol(mw 3500) *
- Peak #5 polyol(mw 1000) *

* molecular weights were from the chromatography elution volumes.

It should be noted that this information is obtainable with less than one day of effort. By contrast, weeks of project effort may be required to obtain this array of data from the conventional means of preparative chromatography and analysis of individual collected and prepared fractions.

The LC-Transform makes possible ready identification and analysis of samples that pose a difficult and costly analytical problem.

Our thanks to Dr. Patricia Turley and Dr. George Polson of Olin Corporation, who performed the experiments presented in this application note.

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