CDS olutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Using a Thermal Technique to Extract Residual Oligomers

Many analytical labs use solvents as a way to extract key analytes from their matrix. These methods can be labor intensive, time consuming, and use harmful chemicals that require fume hoods. Also, solvent extraction provides less than optimal sample recoveries. Furthermore, the extract may contain non volatile components which can dirty the column and injection port, causing more GC downtime for maintenance. A simpler way to extract and analyze volatiles and semivolatiles is by using a thermal technique. This technique will only extract relatively volatile compounds, keeping non volatiles out of the GC injection port, as well as remove a solvent peak.

One company came to us looking for an alternative way to extract and analyze residual oligomers from the polymers they manufacture. Using the 5250 Pyrolysis Autosampler at sub-pyrolysis temperatures, we were able to combine extraction and analysis into one step.

The 5250 Autosampler was programmed to drop the sample into the pyrolysis chamber, put the chamber online with the GC, and heat the chamber to 300°C for 2 minutes. Resulting volatiles were swept through a heated transfer line to the GC/MS for analysis. The chromatogram is shown in the top picture in Figure 1 below. The sample was heated a 2nd time, to determine if the polymer was degrading, and to be sure that it had been fully extracted. A degrading polymer would show peaks with increased abundances, different from the residue peaks. On the second heating, there was very little residual left (bottom picture, Figure 1), demonstrating that heating the sample chamber to 300°C for 2 minutes one time is not too intense to degrade the polymer, and enough time to extract the original polymer almost entirely. This method compared to sovent extraction techniques increases sensitivity and simplifies sample preparation.

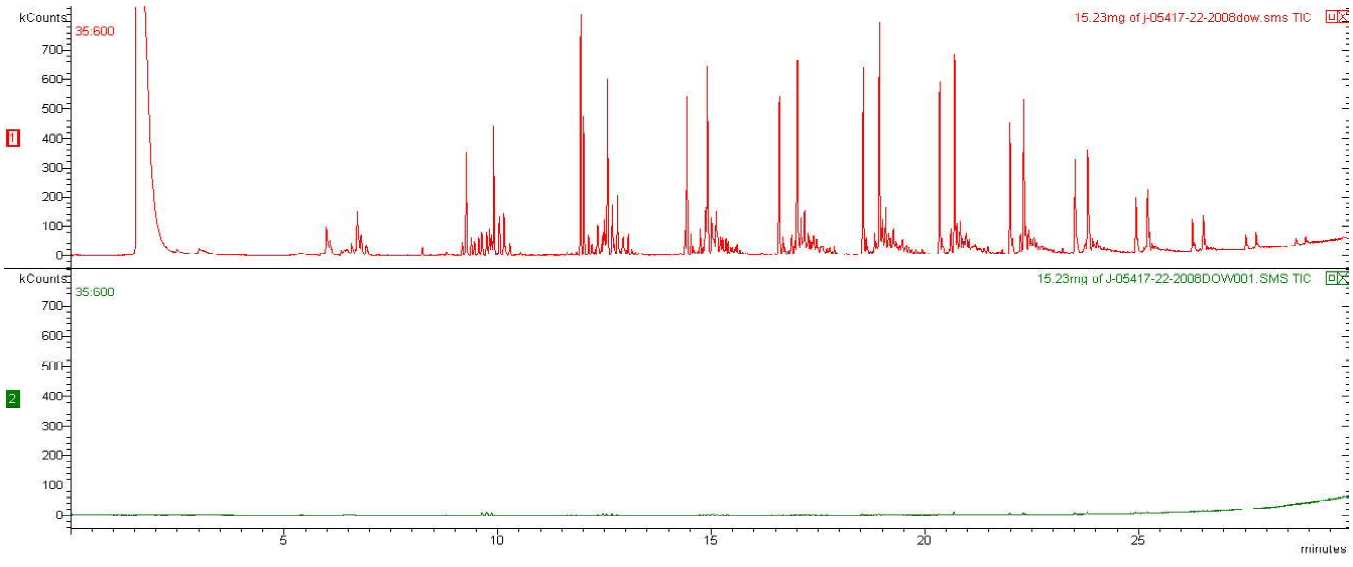

Figure 1: Residual Oligomers thermally extracted from a polymer at 300°C for 2 minutes.

Equipment

All samples were heated using a CDS Model 5250 Pyrolysis Autosampler interfaced to a Varian CP3800 gas chromatograph with an ion trap detector.

Thermal Desorption

Chamber Temperature:	300°C
Chamber Time:	2 minutes

Chromatography

Carrier:	He
Column:	VF5-HT
	30 m x 0.25 mm
Split:	100:1
Initial temperature:	40°C for 2 minutes
Ramp:	10°C/minute
Final temperature:	350°C for 5 minutes

FOR MORE INFORMATION CONCERNING THIS APPLICATION, WE RECOMMEND THE FOLLOWING READING:

Temperature as a Sample Preparation Tool in the Analysis of Materials by GC-MS, T. P. Wampler, LC-GC Vol 17, 9, 14-17 (1999).

Additional literature on this and related applications may be obtained by contacting your local CDS Analytical representative, or directly from CDS at the address below.

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