Thermal Extraction of Oligomers in a Plastic

Application Note
Plastics

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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 thermally labile 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 Pyroprobe at sub-pyrolysis temperatures, we were able to combine extraction and analysis into one step.

The Pyroprobe 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 of Figure 1. 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.

Instrument Conditions

Pyroprobe

Py Chamber:	300°C 2 minutes
Valve Oven:	300°C
Transfer Line:	325°C

GC/MS

Column:	5% phenyl (30m x 0.25mm)
Carrier:	Helium, 100:1 split
Injector:	350°C
Oven:	40°C for 2 minutes
	10°C/min to 350°C, hold 5 min
Mass Range:	35-550

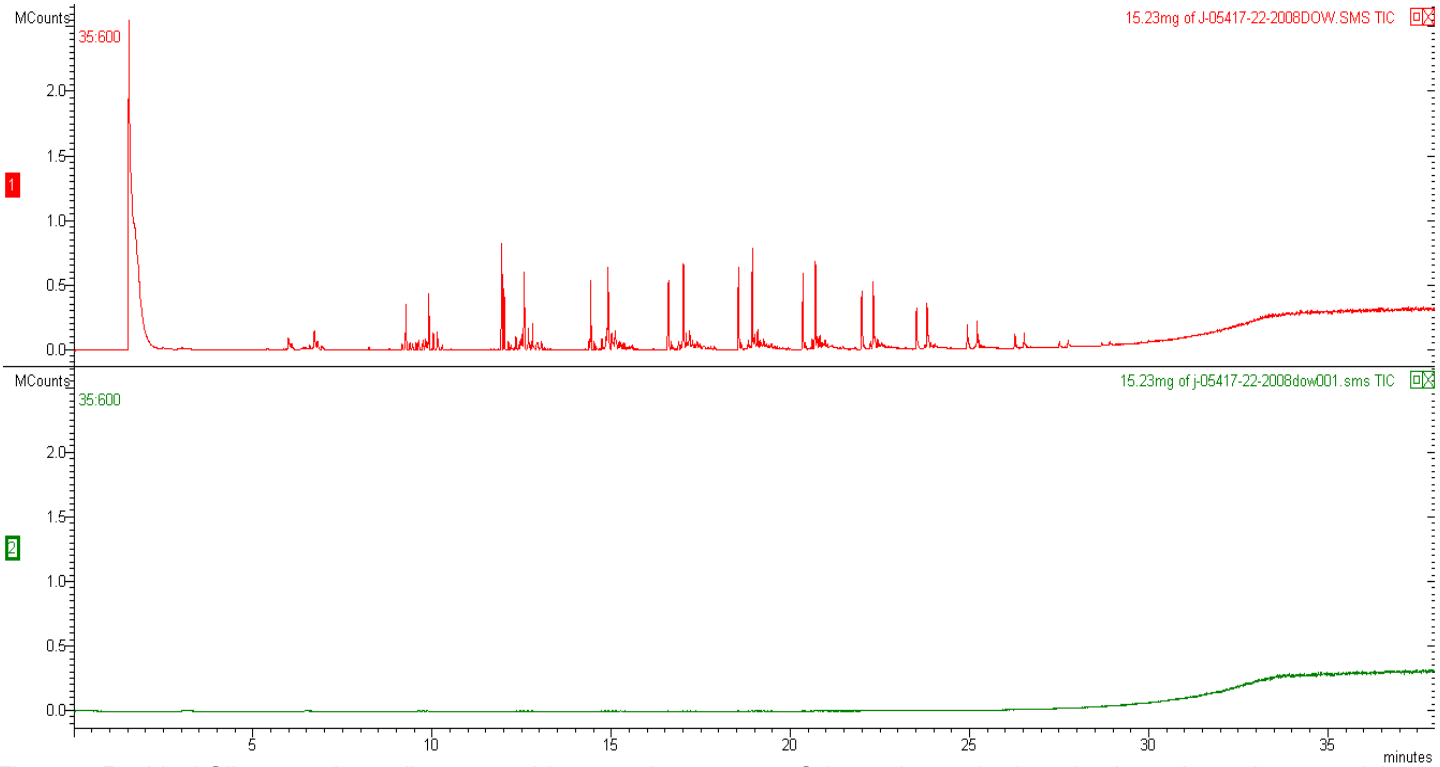

Figure 1: Residual Oligomers thermally extracted from a polymer at 300°C for 2 minutes (top), and polymer heated a second time at 300°C for 2 minutes (bottom), all residual oligomers had been extracted during the first thermal extraction.