Quantitaion of Low Levels of Methyl Methacrylate in a Copolymer

Application Note
Rubber & Adhesive
Paint
Plastics

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When a material is made using several different monomers, like a styrene/ butadiene rubber, ethylene/propylene copolymer or a latex paint, pyrolysis-GC/ MS can be a valuable tool in identifying not only the monomers used, but the relative amounts of each. Whether mixtures, blends, laminates, random or block copolymers, compounds will be produced relating to each of the monomers present, and the peaks they make can be used to study the molecular formula. This is frequently done using copolymers with just a few monomers, each of which represents a significant part of the copolymer. But the same techniques may be used to determine relatively small concentrations of monomers, and even traces of contaminants.

The polymers shown here are primarily styrene, but each contained a small amount of methyl methacrylate - specifically 0.1%, 0.2%, 0.3% and 0.4%. Figure 1 shows a pyrogram of the 0.4% product, showing the styrene monomer, dimer and trimer as well as peaks for alpha-methyl styrene and toluene, also pyrolysis products of polystyrene, and a small, but still measurable peak for the MMA monomer.

Figure 2 contrasts the 0.1% and 0.4% samples. As the amount of MMA increases, the MMA peak becomes larger, relative to other more constant peaks. The ratio of the MMA peak area to the toluene peak area shows a linear relationship to the concentration of MMA, as shown in Figure 3. Using a peak area ratio of two peaks produced by pyrolysis of the sample makes the assay independent of the sample size, so it is not necessary to weigh each sample before pyrolysis. Producing a graph from polymers of known content then makes it simple to determine the amount of MMA contained in an unknown polymer.

Instrument Conditions

Pyroprobe

Interface:	325°C 4 minutes
Pyrolysis	750°C 15 seconds
Valve Oven:	325°C
Transfer Line:	325°C

GC/MS

Column:	5% phenyl (30m x 0.25mm)
Carrier:	Helium, 50:1 split
Injector:	350°C
Oven:	40°C for 2 minutes
	10°C/min to 325°C
Mass Range:	35-600 amu

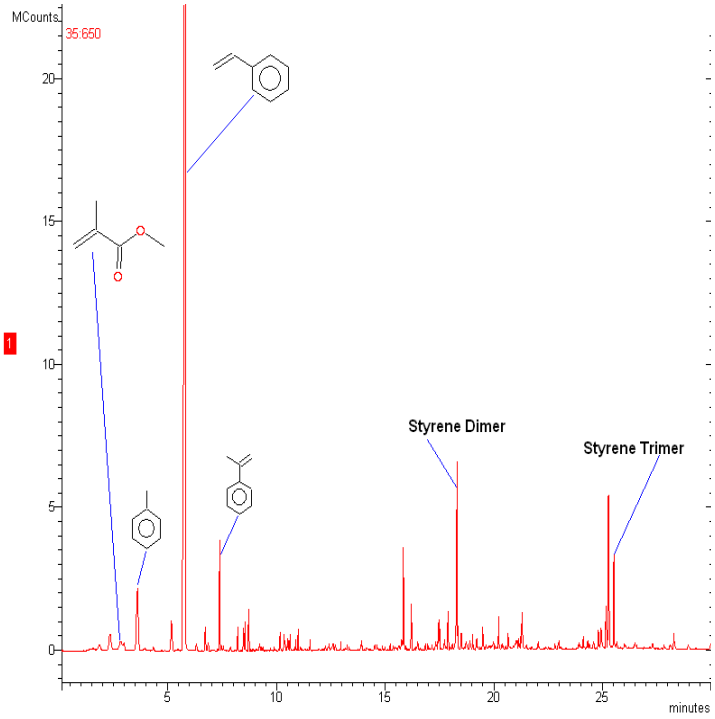

Figure 1. Copolymer with trace of MMA.

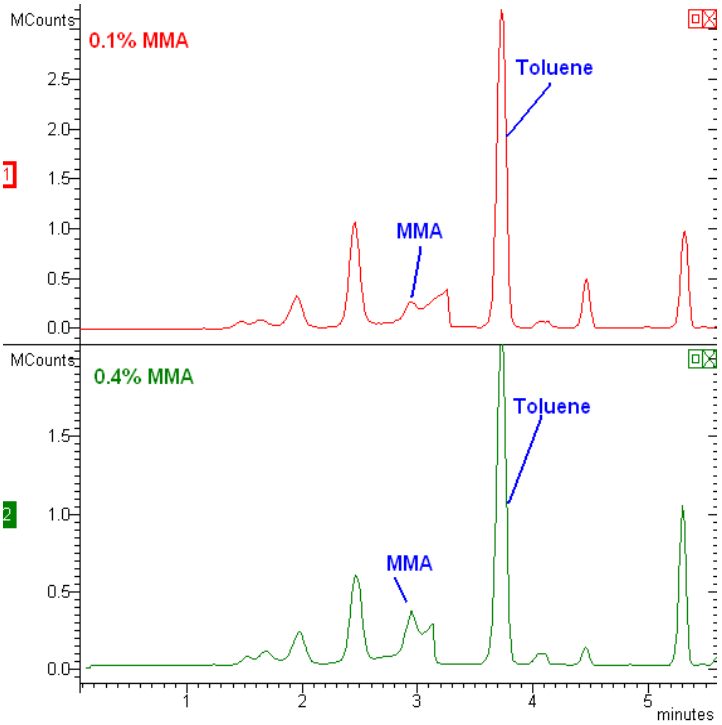

Figure 2. 0.1% and 0.4% MMA in copolymer.

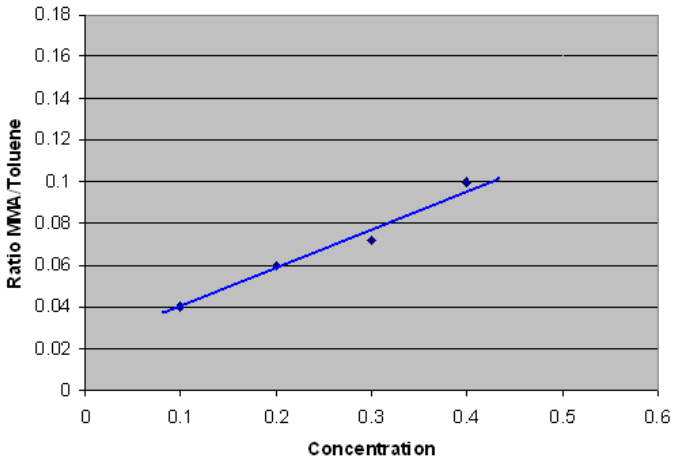

Figure 3. MMA/Toluene peak area ratios vs. MMA concentration.

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

F. Wang, Composition and Microstructure Determination of a Latex System by Pyrolysis Gas Ghromatography, Anal. Chem. 71 (1999) 4776-4780.