Chromatography Technical Note No AS114

Automated Pyrolysis of Polystyrene and Poly (bisphenol A carbonate)

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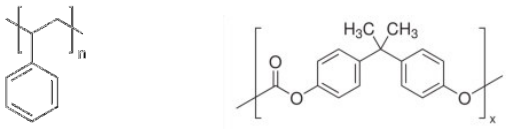
Introduction

Pyrolysis uses high temperatures at 350 °C and above to decompose organic material with a limited supply of oxygen. Structural information of a number of polymers can be obtained using this technique.

Polystyrene and Poly (bisphenol A Carbonate) are check-out materials provided to ensure the Pyro, which is made by GERSTEL, is working correctly.

If you are interested in automating pyrolysis and would like to run some samples, please contact either Paul Roberts or Dan Carrier at Anatune. The repeating chemical structure units for Polystyrene and Poly (bisphenol A Carbonate) are shown in Figure 1.



Polystyrene Poly (bisphenol A Carbonate)

Figure 1 Repeated chemical structure units for Polystyrene and Poly (bisphenol A Carbonate)

The Pyro is housed within the Thermal Desorption Unit. Pulsed pyrolysis can be performed using a range of temperatures between 350 °C and 1000 °C. The temperature can be varied from sample to sample in order to analyze different kinds of samples in one batch. In addition to pulsed pyrolysis, programmable heating can be specified at rates ranging from 0.02 to 100 °C/s. A thermal desorption temperature range from ambient to 350 °C can be set prior to pyrolysis in order to purge VOCs from the sample or for solvent venting. Figure 2 shows a schematic of how the Pyro is connected to the Agilent 7890 GC. The Pyrolysis breakdown products are transferred to the GC/MS system using the GERSTEL Cooled Injection System (CIS) PTV – inlet. The CIS can be used either simply as a heated split interface as described within this application note or as an intermediate cryofocusing trap for trace analytes.

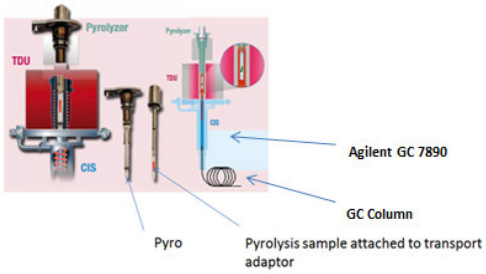
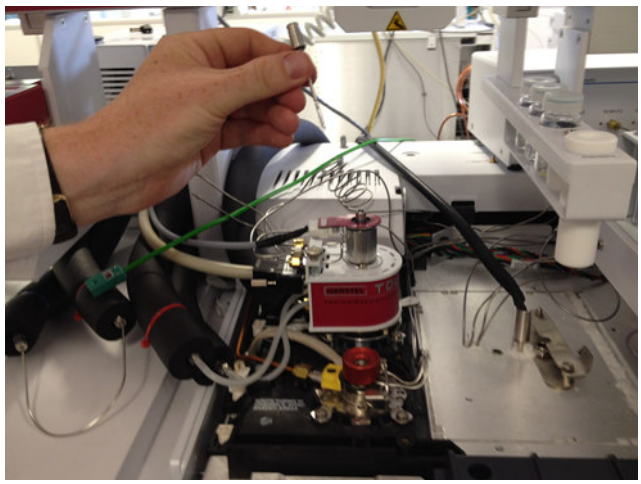


Figure 2 A schematic of how the Pyro is connected to the Agilent 7890 GC.

Figure 3 shows two photographs of the Pyrolysis unit at our new site in Girton.



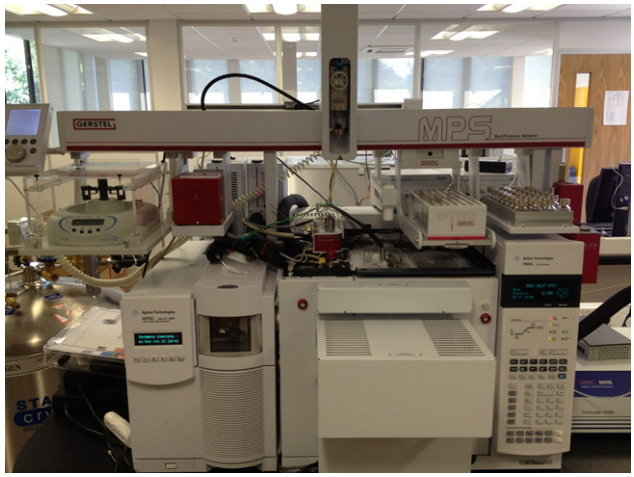


Figure 3 Pyro photographs at our new site in Girton.

Each sample is transported using the Multi Purpose Sampler (MPS2) into the Pyro for Pyrolysis. Each Pryo tray can hold up to 98 samples.

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Instrumentation

GERSTEL Multipurpose Sampler MPS 2 XL
Maestro Version 1.4.8.14/3.5
GERSTEL Pyro
GERSTELThermal Desorption Unit
GERSTEL Cooled Injection System (CIS) 4
Agilent 5975C Inert XL MSD
Agilent GC 7890A

Method

Pulsed Pyrolysis was performed at three different temperatures: 500 °C, 800 °C, and 1000 °C using Poly (bisphenol A Carbonate) and Polystyrene with a standard thermal GC gradient from 50 °C to 240 °C using a VF1 60 M x 0.32 mm id column (film thickness 1 micron) with a split of 50:1.

Results

Figure 4 shows a TIC chromatogram comparison of Poly (bisphenol A Carbonate) with blank. Pyrolysis products Toluene and Benzene appear to increase in intensity with the higher Pyrolysis temperatures.

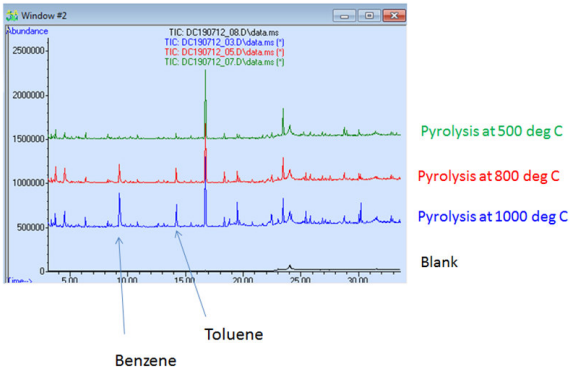

Figure 4 TIC chromatogram comparison of Poly (bisphenol A Carbonate) with blank.

The following characteristic pyrolysis products for Poly (bisphenol A Carbonate) were also detected at the three different temperatures: 4-methylphenol, Naphthalene, and Styrene.

Figure 5 shows a TIC comparison of Polystyrene. Pyrolysis products 1-Heptene and 1-Hexene appear to decrease in intensity with the higher Pyrolysis temperatures.

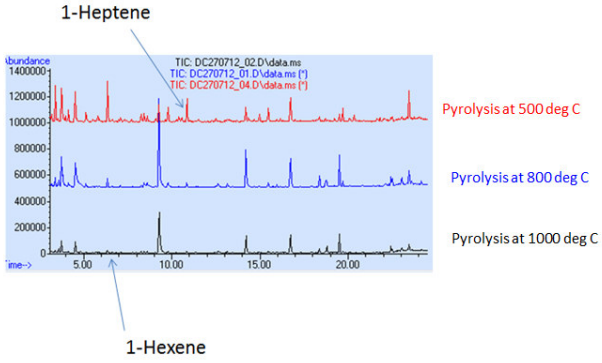

Figure 5 TIC chromatogram comparison of Polystyrene.

The following characteristic pyrolysis products for Polystyrene were also detected at the three different temperatures: α-methylstyrene, Ethyl benzene, and Toluene.

Figure 6 shows TIC chromatogram comparison of Pyrolysis for Poly (bisphenol A Carbonate) at 500 °C to give an initial impression of the pyrolysis reproducibility.

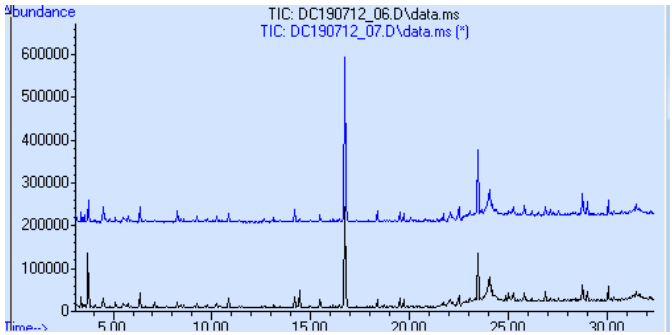

Figure 6 duplicate analysis of Pyrolysis at 500 °C for Poly (bisphenol A Carbonate)

Discussion

This application note shows how pyrolysis can be easily automated. As mentioned above, please feel free to contact either Paul Roberts or Dan Carrier at Anatune to discuss a potential demonstration of the Pyro for your samples.

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