Application Note No. 110

Key Words:
Pyrolysis
PE
PPSU

Pyrolysis of solid PE and PPSU with OPTIC 3 - GC-MS

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Subject description:

Pyrolysis of four samples named A,B,C and D. Samples A and B are PE samples and C and D are PPSU samples. The request from the customer is to show the repeatability of pyrolysis performed with the OPTIC for all the samples, especially for sample A and C.

Experimental information about methods:

The first step before the analysis was to cut a small piece of the samples and to weight it on a micro-balance. The sample-pieces were than inserted into a micro-cup. The micro-cup was put into a DMI liner [L1000011] which was present inside the PTV-injector. To get the air out of the PTV injector, a high split flow was used to flush the injector with Helium. After this step, pyrolysis was occurred by heating up the injector using a fast ramp rate to a temperature of 600 °C. After three minutes, the injector was cooled down to 320 °C during GC analysis of the Pyrolysis fragments.

Intrumentation and experimental conditions:

Hardware:

Pyrolysis unit:	OPTIC 3 PTV-injector (ATAS GL)
GC-MS	QP 2010 (Shimadzu)
GC-Column	TC-5MS 30 m x 0.25 mm x 0.25 µm (L x ID x film thickness)

Conditions:

Sample Introduction:

Sample weight: ~0.8 mg in micro-cup
Start temperature: 40°C
Split flow: 150 ml/min (flushing injector)
Column flow: 0.7 ml/min

Pyrolysis:

Final temperature: 600 °C (ramp rate 30 °C/sec)
Split flow: 10 ml/min
Column flow: 1.5 ml/min

GC-oven:

40 °C for 2 min
4 °C/min to 230 °C
10 °C/min to 320 °C for 20 min

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Results:

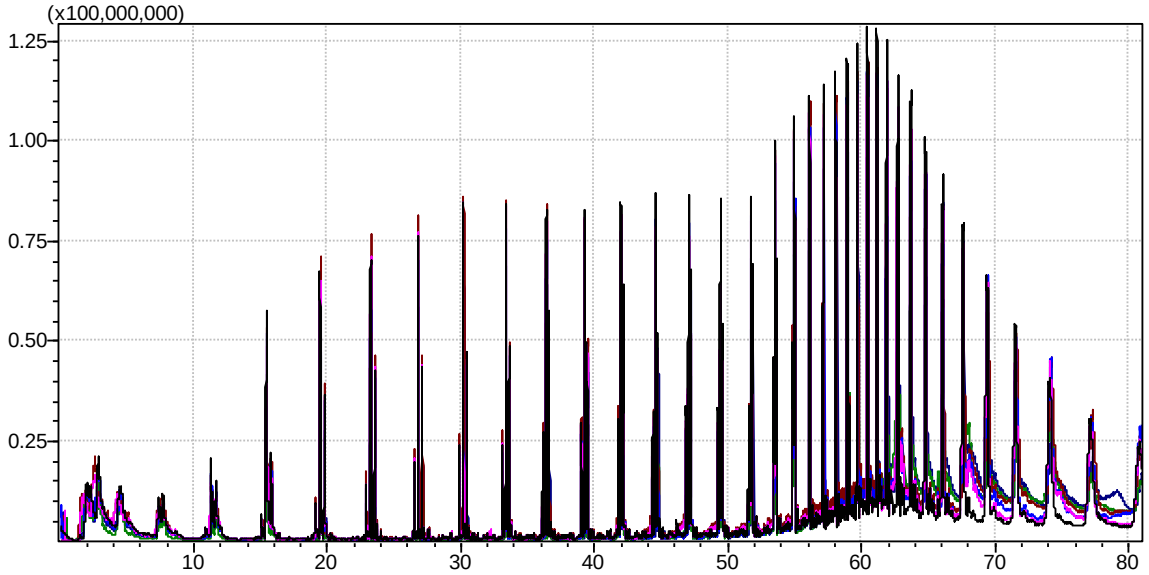

Figure 1: Overlay of six TIC-chromatograms of sample A (PE).

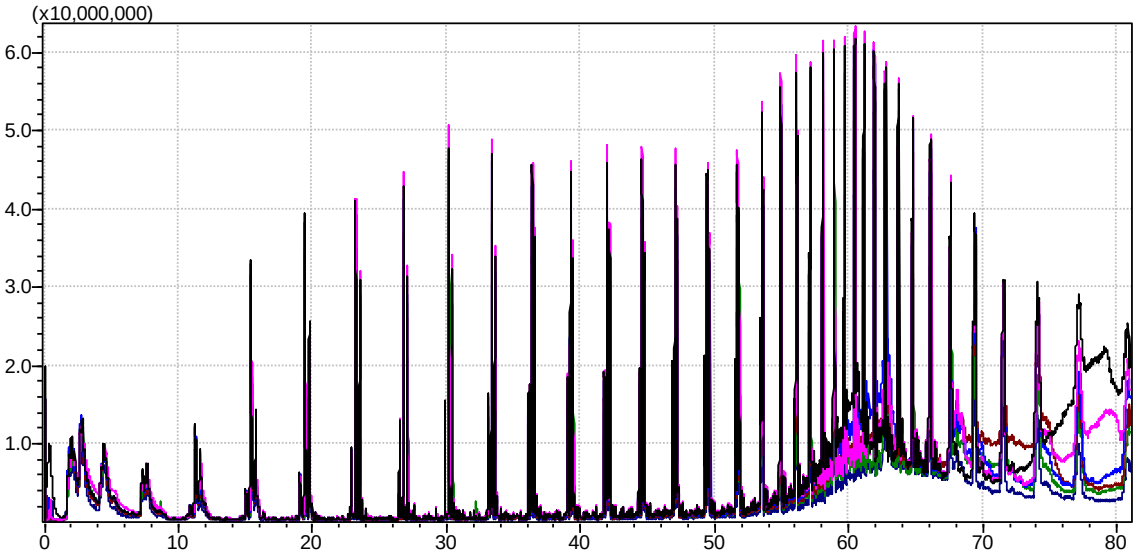

Figure 2: Overlay of six TIC-chromatograms of sample B (PE).

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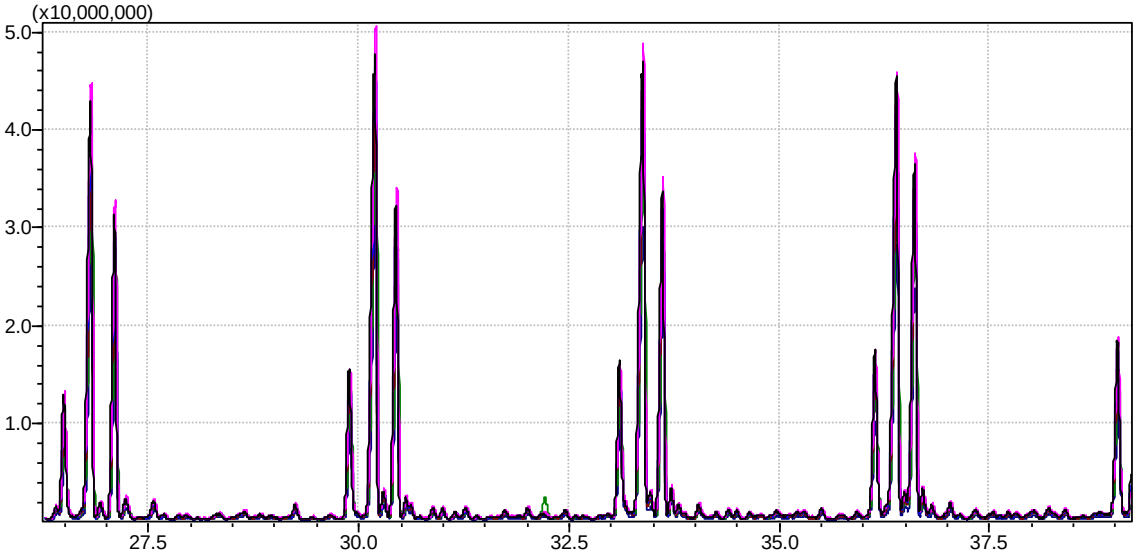

Figure 3: Detail of the six TIC-chromatograms obtained for sample B

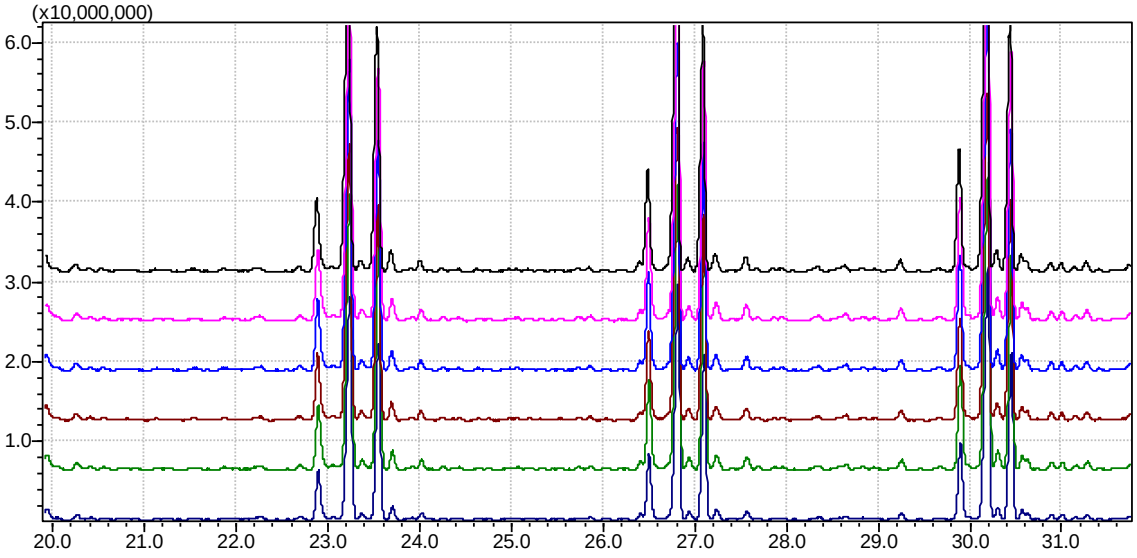

Figure 4: Detail of the six TIC-chromatograms obtained for sample B.

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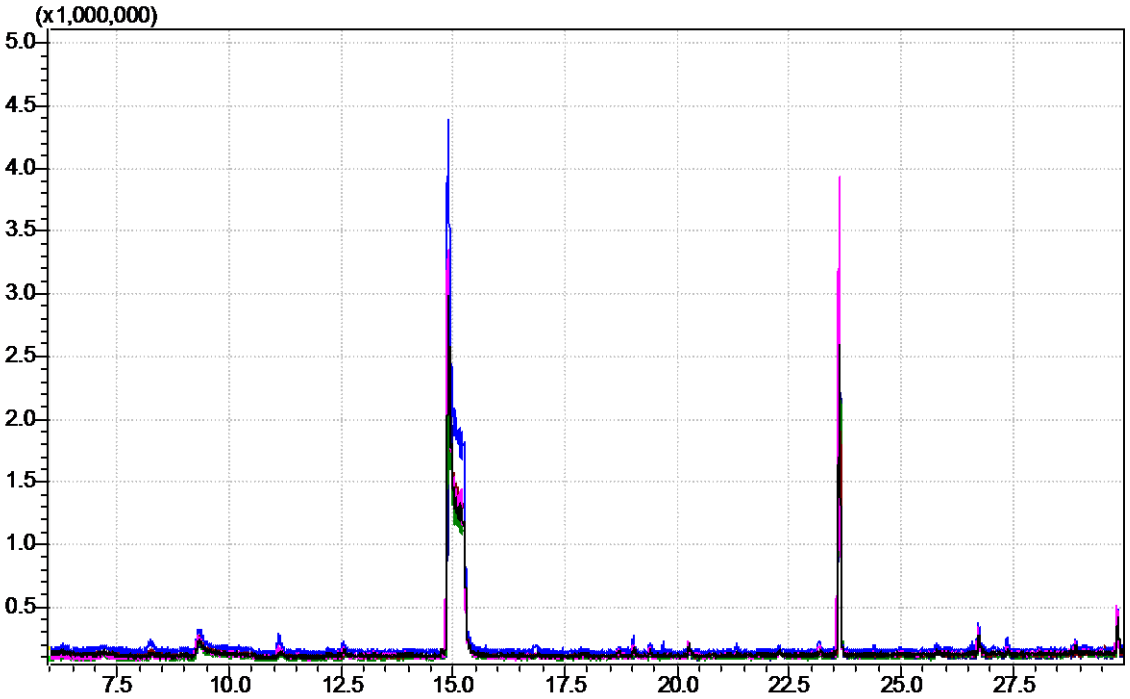

Figure 5: Overlay of six TIC chromatograms of sample C (PPSU). The first peak (Rt 15 min) is Phenol and the second peak is Tetramethyl Sulfone (Rt 23 min).

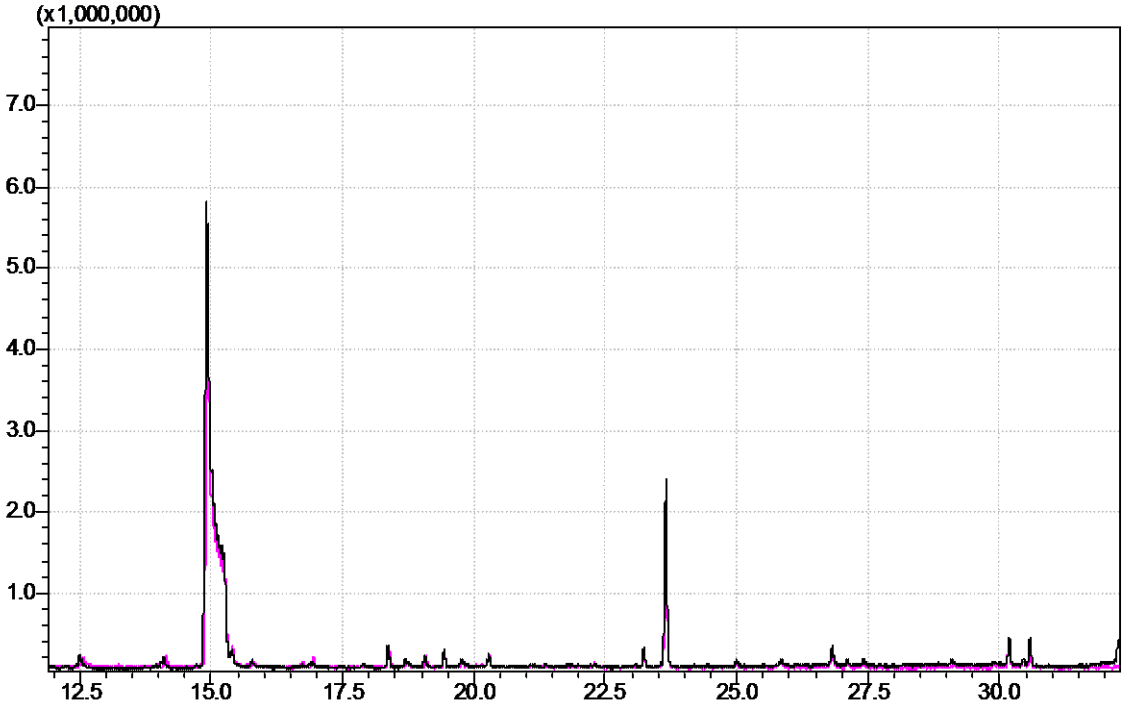

Figure 6: Overlay of two TIC chromatograms of sample D (PPSU). The first peak (Rt 15 min) is Phenol and the second peak is Tetramethyl Sulfone (Rt 23 min).

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Discussion:

The repeatability obtained with sample A and B is less than RSD 5% (for n=6) for the retention times and peak areas (corrected for the weight). However, if it is possible to dissolve these samples and to inject the extract into the OPTIC injector, we expect even lower RSD values. It is clear from the results that the pyrolysis with the OPTIC is performing well, however the chromatography is not so good. Properly, the GC-column is not performing well anymore. Especially, with sample C and D it is clear that the chromatography is not so good for the polar peak phenol. However, the performance of the OPTIC for pyrolysis for this relatively difficult polymer is very well, again a RSD of less than 5% .
Finally, Py-GC can be performed well using the OPTIC 3 injector. Quantitative results can be obtained using this novel method. The whole method is fast and easy to automate with the LINEX.

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