Application Note No. 078

The Thermal Desorption / Pyrolysis of Glass Fibre Air Filters

Diane Nicholas

- ***Direct desorption of analytes from sample matrix to column***
- ***Multi-step thermal desorption followed by pyrolysis of same sample in injector***
- ***Can be automated using the CombiPAL and LINEX***

Keywords:
Pyrolysis, thermal desorbtion, in-injector pyrolysis, multi mode inlet pyrolysis

Instrumentation

- ATAS GL Optic 2-200 programmable injector
- Agilent 5890 gas chromatograph with 5971 mass selective detector
- SGE CO_2 cryotrap

Sample analysed

Borosilicate glass air filters impregnated with 5 % phenolic and epoxy resins and some fluorocarbons.

Principles

- Place 2-4 mg of filter paper in a fritted liner, place in injector and start run
- Liner is firstly swept and the cryotrap turned-on
- The analytes are thermally desorbed under static flow conditions
- The analytes are swept onto the column with a small split flow and trapped
- The cryotrap is turned off and the separation and analysis is performed
- At the end of the run the injector and oven are cooled and the sample is pyrolysed, following the same principles

Chromatograms

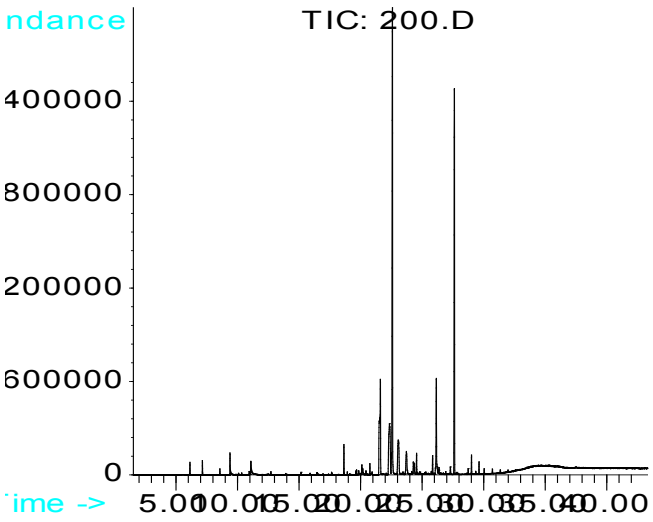

Figure 1: *Filter A: Thermal desorption at 200 °C*

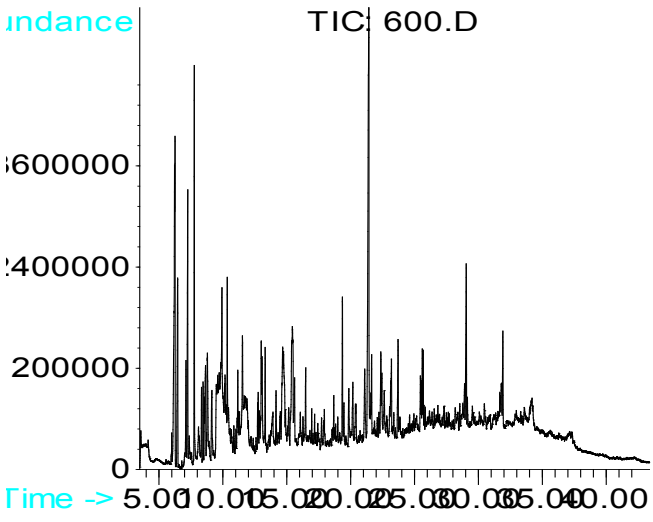

Figure 2: *Filter A: Pyrolysis at 600 °C*

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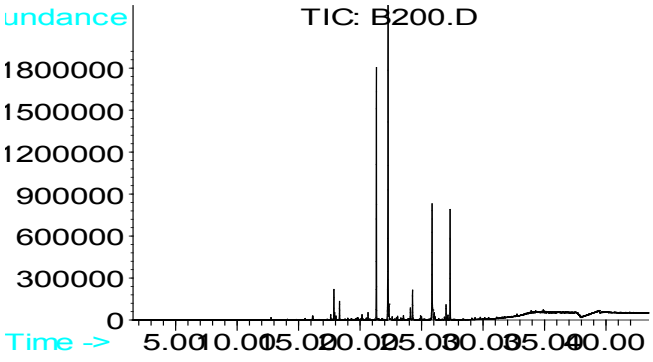

Figure 3: *Filter B: Thermal desorption at 200 °C*

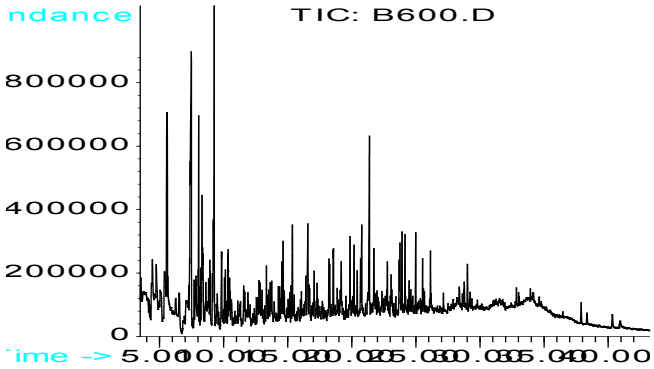

Figure 4: *Filter B: Pyrolysis at 600 °C*

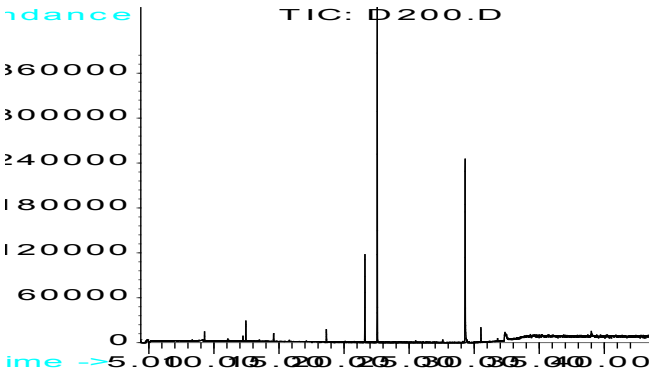

Figure 5: *Filter D: Thermal desorption at 200 °C*

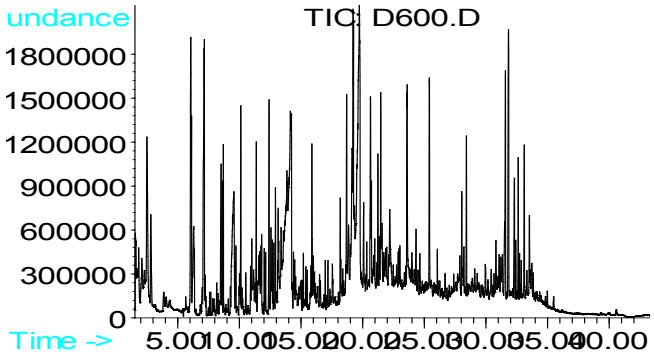

Figure 6: *Filter D: Pyrolysis at 600 °C*

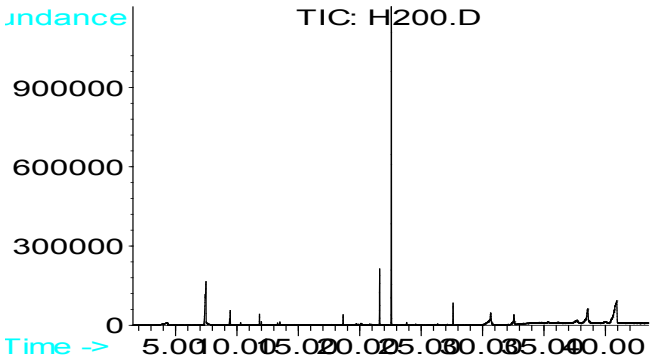

Figure 7: *Filter H: Thermal desorption at 200 °C*

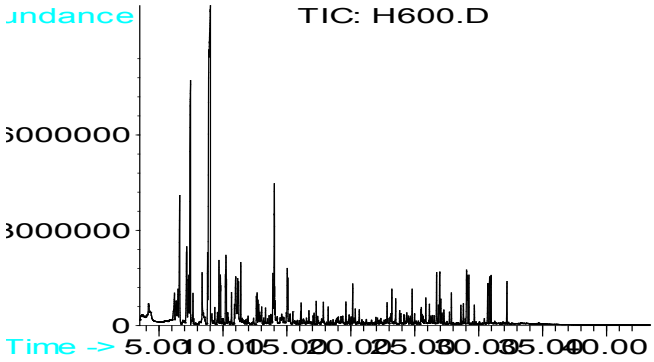

Figure 8: *Filter H: Pyrolysis at 600 °C*

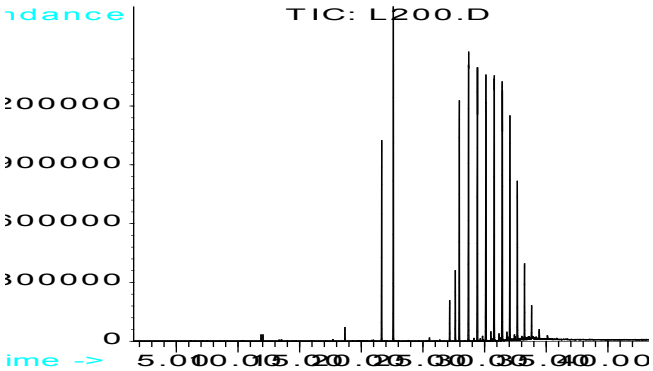

Figure 9: *Filter L: Thermal desorption at 200 °C*

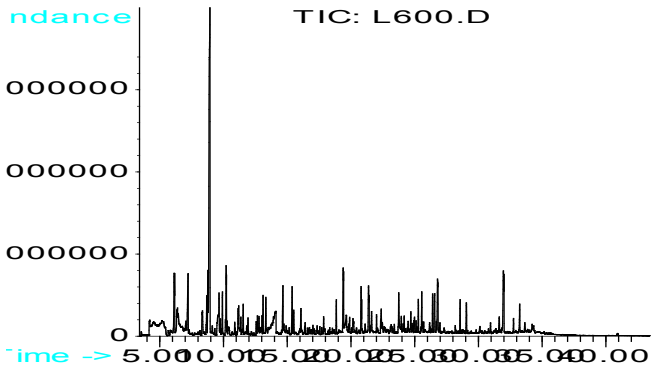

Figure 10: *Filter L: Pyrolysis at 600 °*

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Appendix

Optic Parameters:

Liner:		ATASGL Fritted
Mode:		Expert
Gas Flows:	Vent:	75 ml/min
	Split:	10 ml/min
Initial temperature:		35 °C
Isothermal time:		1 min
Ramp rate:		16 °C/s
Final temperature:		200 °C (thermal desorption)
Final temperature:		600 °C (pyrolysis)
End time:		43.5 mins
Sweep pressure:		8 psi
Sweep time:		0.5 mins
Split open time:		0.5 mins
Desorption pressure:		0 psi
Desorption time:		2.5 mins
Transfer pressure:		7.1 psi
Transfer time:		2 mins
Initial pressure:		7.1 psi
Final pressure:		24.8 psi

Cryotrap Parameters:

Cryo on:	0.25 mins
Cryo off:	4 mins

GC Parameters:

Column: HP5-MS 30m x 0.25mm i.d. x 0.25um film

Initial temperature:	45 °C
Initial time:	5 mins
Ramp rate:	10 °C/min
Final temperature:	330 °C
Final time:	10 mins

MS Parameters:

Acquisition mode:	Scan
Low mass:	50 m/z
High mass:	300 m/z
Sampling number:	2
Threshold:	500
Transfer line:	330 °C
Solvent delay:	2 mins

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