The Thermal Desorption / Pyrolysis of Glass Fibre Air Filters

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- 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 desorption, 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 CO2 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
We would like to thank Dr Anthony Lawson from Hollingworth & Vose Co Ltd. for his kind permission to publish this data.
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 °C
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