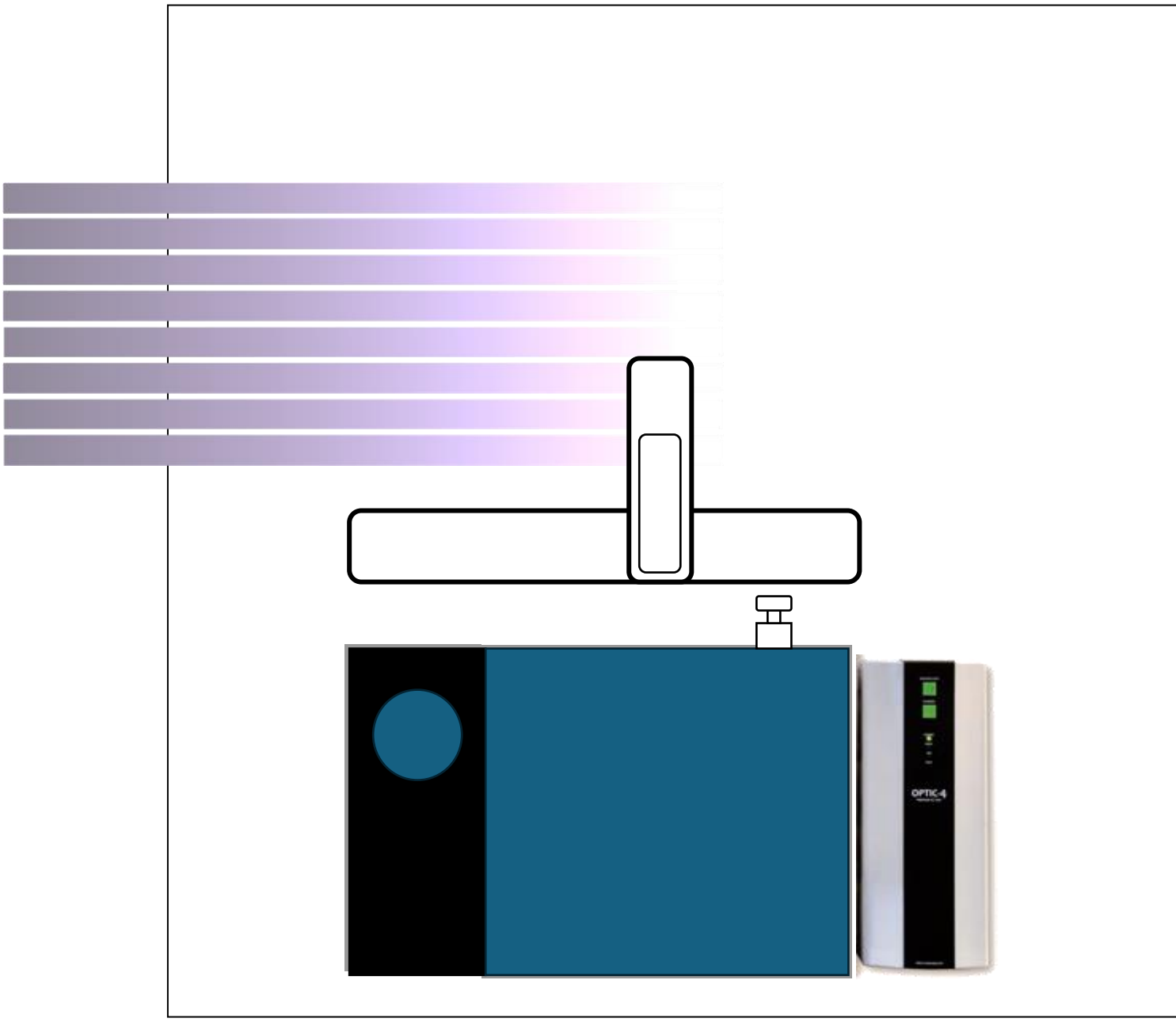
GL Sciences

Application note: 134

Polyethylene Pyrolysis with an OPTIC-4 Pyrolysis System



Keywords: OPTIC, Pyrolysis, Polyethylene

Introduction:

The OPTIC-4 patented low thermal mass design of the inlet body together with direct resistive heating provides fast linear temperature programming up to 700 °C at rates as high as 60 °C/s. In addition to standard sampling modes, the programmable inlet can operate effectively with Pyrolysis, Large Volume Injections, Cold Injections or Thermal Desorption sample introduction and combination of the mentioned techniques. With the options for sub-ambient cooling, cryogenic trapping and automated liner exchange, OPTIC-4 is the world's most versatile inlet for Gas Chromatography

Cryogenic cold trapping is frequently used for narrowing the chromatographic band and improving the detection limit. When trapped, analytes must be released from the trap using a highly accurate and very fast heating ensuring that they are introduced onto the column in a very sharp band. This results in better detection limits and better resolution.

This application note will show that pyrolysis with OPTIC in combination with automatic liner exchange is easy and simple.

Equipment:

OPTIC-4PS Pyrolysis system (2403-3301, Full automated Pyrolysis System with PAL3, Cold trap and LINEX)
Gas chromatograph with detector (for example a GC-MS)
Column: InertCap 5HT with Proguard, I.D.0.25 mm, length 5+30m, Film 0.25µm (1010-90926)
Pyrolysis preparation kit (2406-4602)
Pyrolysis checkout sample (2403-4501)
Liner: Quartz DMI Liner (2414-1018)
Inserts: DMI Quartz Sample Insert 30ul, 5 mm liner, conditioned, pk/100 (2406-1030)

Analytical conditions:

OPTIC:

Method Type	Pyrolysis
Method Name	Pyro
Equilibration Time	00:05 min:sec
End Time	35:00 min:sec
Initial Temperature	40 °C
Delay Time	00:10 min:sec
Ramp Rate 1	60.0 °C/s
Hold Temperature 1	700 °C
Hold Time 1	01:00 min:sec
Hold Temperature 2	200 °C
Septum Purge Flow	5 mL/min
Vent Mode	Fixed Time

Vent Time	01:00 min:sec
Carrier Control Mode	Flow Control
Zero LINEX Head Pressure	No
Sample Sweep Column Flow	1.0 mL/min
Transfer Column Flow	1.0 mL/min
Transfer Time	02:00 min:sec
Start Column Flow	1.0 mL/min
End Column Flow	1.0 mL/min
Vent Flow	10.0 mL/min
Split Flow	10.0 mL/min
Save Split Flow	25.0 mL/min

GC:

Rate (°C/min)	Temp (°C)	hold time (min)
-	40.0	0.00
15.00	400.0	11.00

MS:

Ion Source Temperature: 260°C
Interface Temperature: 250°C

Start	End time	acq mode	speed	scan speed	Start m/z	end m/z
0.00	31.00	Scan	0.30	2500	23.00	725.00

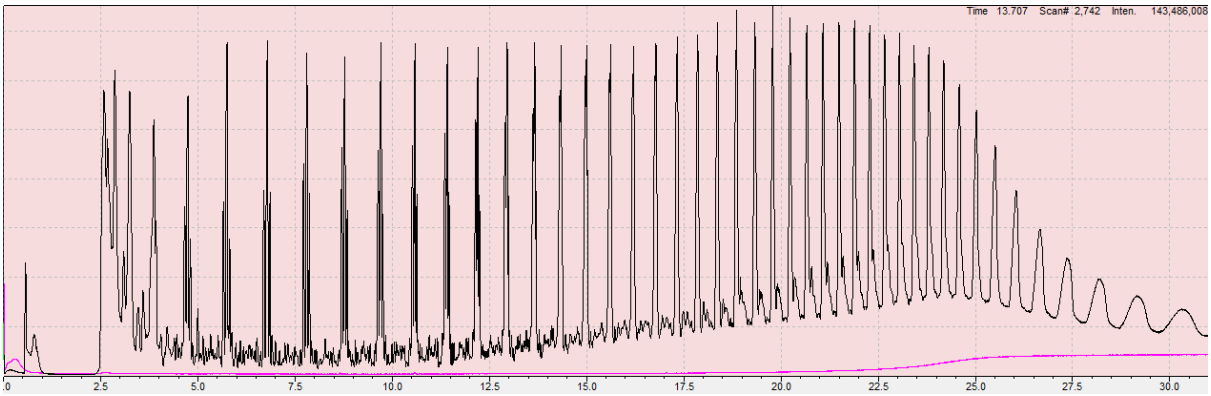
Sample Preparation:

As a sample, a plastic grip bag was used. The bag is made from the material PE.
With a small punch, 2 pieces were removed and placed in a DMI insert.

Analysis:

The DMI insert was placed inside the liner which was placed in the pyrolysis inlet of OPTIC-4.
The OPTIC-4 and GC both used their program to run.
After the run, the sample (DMI insert) was removed, the liner which was used remained in the inlet and a Carry-over check was performed.

Results:



Black line: PE pyrolysis temperature of 700°C
Pink Line: Check to see if Carry-over is there.

Conclusion:

OPTIC-4 is very suitable to do pyrolysis analysis with ease and simple operation.
It doesn't show any carry-over.