

Pyrolysis ramp rate comparison with Polystyrene and Polycarbonate

Key words:

OPTIC-4
Polystyrene
Polycarbonate
Polymers
Pyrolysis ramp rate

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Introduction

The hyphenation of thermal sample treatment techniques such as thermal desorption and pyrolysis with gas chromatography gives a versatile and powerful tool in the study of polymers. An inexpensive system where thermal treatment at different inlet temperature ramp rates is shown. Results obtained indicate the interesting potentials of fast ramp rates for the characterization of polymer composition. The absence of a heated transfer line and switching valves, which are often present in conventional set-ups, eliminates the risk of losses of high molecular weight components. Further advantages of the technique are the simplicity and versatility as well as its inexpensive nature.

Sample materials

Polystyrene dissolved in dichloromethane. 1 µl injected.
Polycarbonate dissolved in dichloromethane. 1 µl injected.

Instrumentation

Inlet: OPTIC-4 Multi Mode Inlet
Liner: L100011, DMI liner with taper
Auto sampler: CombiPAL
GC-MS: Shimadzu QP2010
GC column: : GL Sciences InertCap 5 MS/Sil, 0.25 m x 30 m, film 0.25 µm

Methods:
OPTIC-4

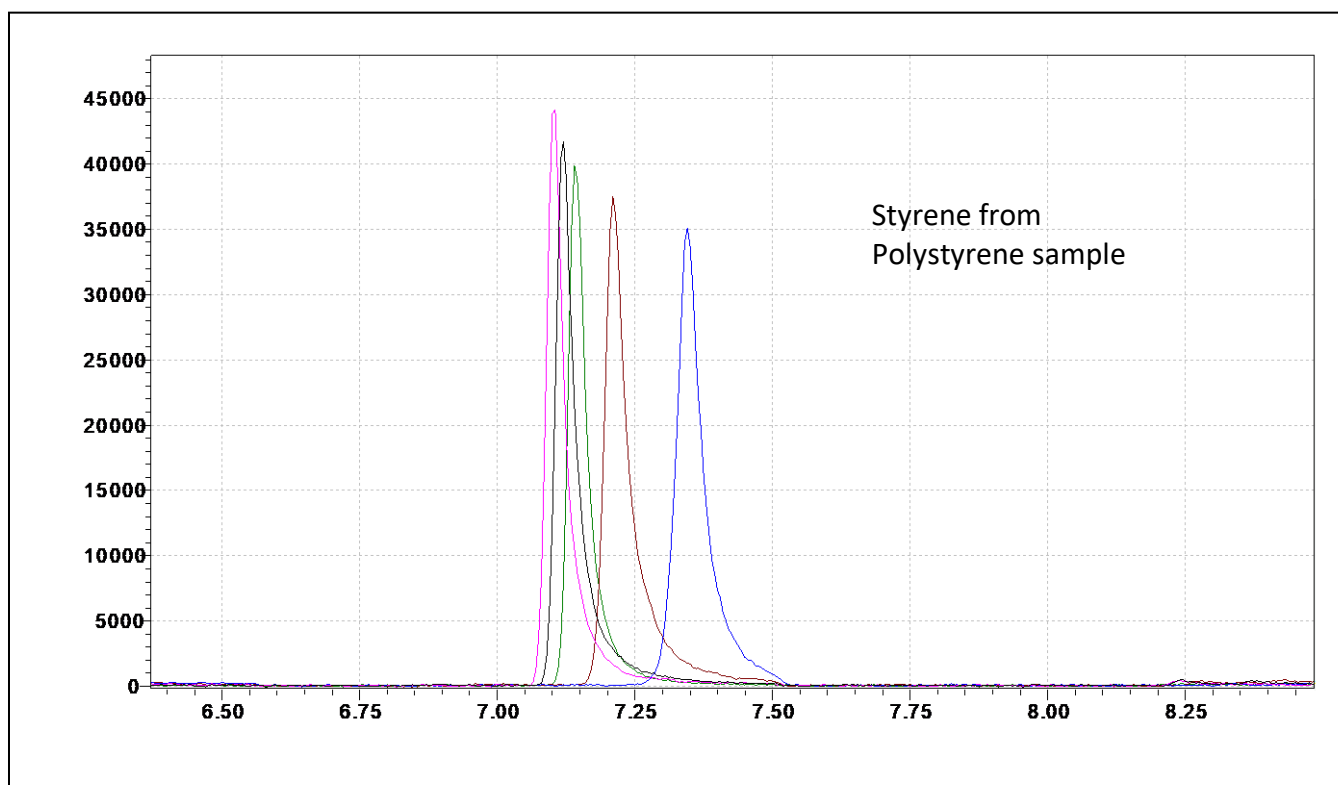
Method Type	Large Volume
Method Name	ramp_15
Equilibration Time	00:05 mm:ss
End Time	10:00 mm:ss
Initial Temperature	40 °C
Ramp Rate	5 - 60 °C/s
Final Temperature	600 °C
Temperature Control	Keep Current Temperature
Vent Mode	Fixed Time
Vent Time	00:30 mm:ss
Carrier Control Mode	Flow Control
Transfer Time	03:00 mm:ss
Sample Sweep Column Flow	1.0 ml/min
Transfer Column Flow	1.0 ml/min
Start Column Flow	1.0 ml/min
End Column Flow	1.0 ml/min
Vent Flow	10 ml/min
Split Flow	25 ml/min

GC/MS

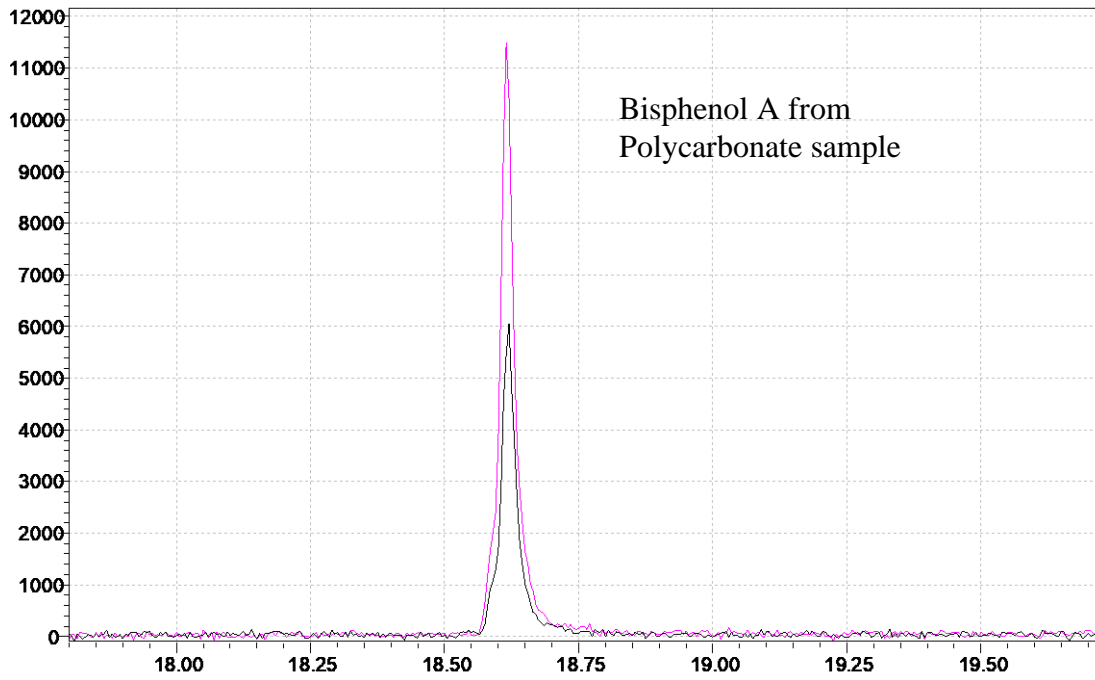
Column Oven Temp. :	45.0 °C	
Oven Temp. Program		
Rate (°C/min)	Temperature(°C)	Hold Time(min)
-	45.0	3.00
15.00	340.0	10.00
Equilibrium Time	0.0 min	
GCMS-QP2010		
Ion Source Temp	200.00 °C	
Interface Temp	250.00 °C	
Solvent Cut Time	1.00 min	
Detector Gain Mode	Relative	
Detector Gain	0.00 kV	
Threshold	0	
MS Table		
Start Time	1.00 min	
End Time	31.67 min	
ACQ Mode	Scan	
Event Time	0.30sec	
Scan Speed	1250	
Start m/z	50.00	
End m/z	400.00	
Sample Inlet Unit	GC	

CombiPAL:

Air Volume (µl)	0
Pre Clean with Solvent 1	0
Pre Clean with Solvent 2	0
Pre Clean with Sample	3
Filling Volume (µl)	3
Filling Speed (µl/s)	10
Filling Strokes	5
Pullup Delay (ms)	0
Inject to	OPTIC
Injection Speed (µl/s)	100
Pre Inject Delay (ms)	0
Post Inject Delay (ms)	0
Post Clean with Solvent 1	3
Post Clean with Solvent 2	3


Results:

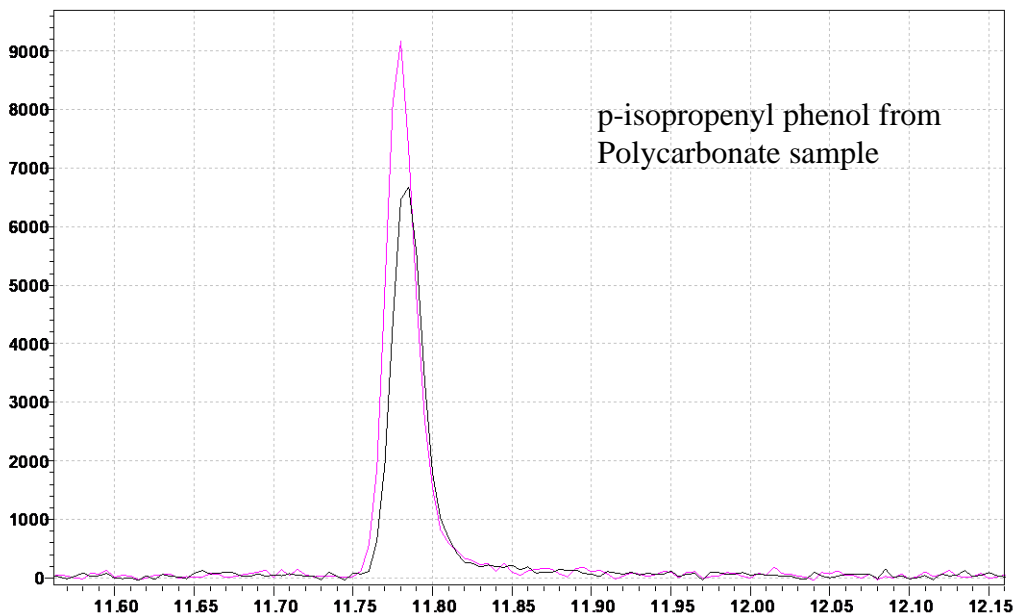
		RT	M/z	Area	Height
Blue	5°C/sec	7.345	104.00	131510	35005
Brown	10°C/sec	7.211	104.00	132088	37374
Green	20°C/sec	7.142	104.00	111031	39877
Black	30°C/sec	7.119	104.00	127139	41752
Pink	60°C/sec	7.103	104.00	120062	44120



Component shown is Bisphenol A. This is a compound used for the production of Polycarbonate.

Black: 30°C/sec

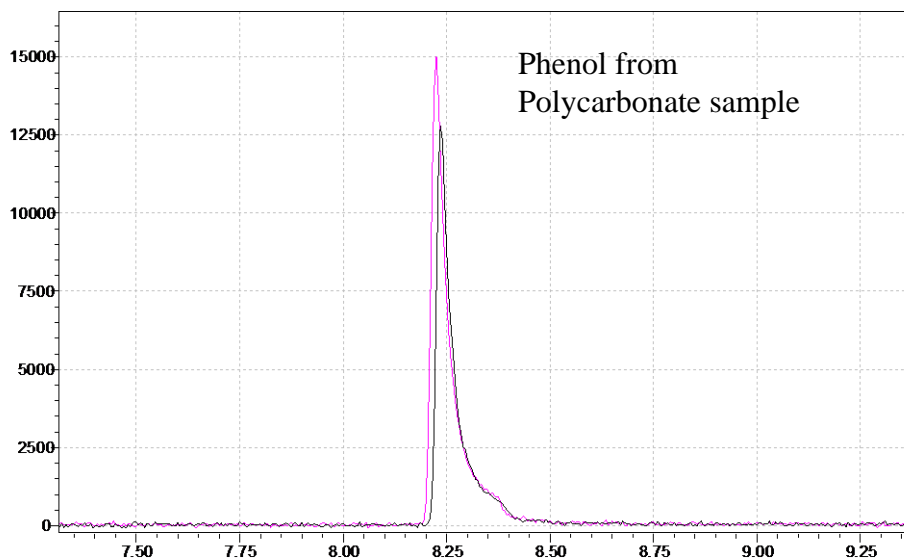
Pink: 60°C/sec



Component shown is p-isopropenyl phenol. This is a compound used for the production of Polycarbonate.

Black: 30°C/sec

Pink: 60°C/sec



Component shown is Phenol. This is a compound used for the production of Polycarbonate.

Black: 30°C/sec

Pink: 60°C/sec

Conclusion:

A fast ramp rate can improve the chromatography when the OPTIC is used as a pyrolysis instrument. For the more high molecular weight components the effect is much more as for the more volatile components.