



Using Thermal Desorption Technique for Characterization of the Polymer Emission

Abstract

This study examines the emission of polymers (nitrile butadiene rubber, polyethylene, silicone rubber). Samples were placed in glass tubes (1/4" OD, 3.5" length) and analyzed using a thermal desorber Chromatec TDA. The tubes were heated and the emissions were collected on a cooled focusing trap Tenax TA. Identification of the released compounds was carried out on a Chromatec Crystal GC/MS.

Introduction

Plastics are commonplace in everyday life. Emissions of volatile organic compounds from plastic materials effect on human health. Therefore, the study of polymer emissions is an actual problem.

The direct thermal desorption technique is simple to use and allows for the rapid analysis of emissions. This method requires a small sample amount (10–40 mg).

A piece of polymer sample is placed in a glass tube and fixed with glass wool. To increase the emission rate, the tube heats up and the inert gas flow transfers volatile compounds into the focusing trap. On the next stage, the trap is heated and components of interest are injected into the chromatographic column.

Direct thermal desorption presents important advantages: it can be easily coupled to GC/MS; the analytes are released directly in the thermal desorber and prior extraction is not required; the ability to analyze a number of samples automatically.

Equipment

- Chromatec Crystal 9000 GC/MSD
- Capillary column CR-5ms (30 m × 0.25 mm × 0.25 μm)
- Thermal Desorber Chromatec TDA
- Empty glass tubes (1/4" OD, 3.5" length)
- Focusing trap Tenax TA

Analysis mode

GC			
Run time	32 min		
Column			
Carrier gas	Helium		
Constant flow	1 mL/min		
Column temperature	50 °C	2 min	10 °C/min
	280 °C	7 min	
Inlet			
Temperature	250 °C		
Split	1:10		
MSD			
Scan mode	TIC		
Mass range	35–450 a.e.m.		
Ion source temperature	250 °C		
Transfer line temperature	280 °C		
Thermal Desorber			
Valve temperature	250 °C		
Transfer line temperature	250 °C		
Purge gas	Helium		
Desorption temperature	60 °C		
Desorption flow	20 mL/min		
Desorption time	10 min		
Trap low temperature	-20 °C		
Trap high temperature	280 °C		
Trap heating rate	2000 °C/min		
Trap heating time	2 min		

Experimental

Samples were cut into small pieces. One piece of sample (about 20 mg) was introduced into a glass tube (1/4" OD, 3.5" length). Two glass wool plugs were installed on both sides of the sample (Figure 1). The tube was placed in the Chromatec TDA thermal desorber. Volatile compounds were desorbed at 60 °C for 10 min and focused on a Tenax TA trap at -20 °C. Then the trap was heated to 280 °C and volatiles were transferred to the chromatographic column.

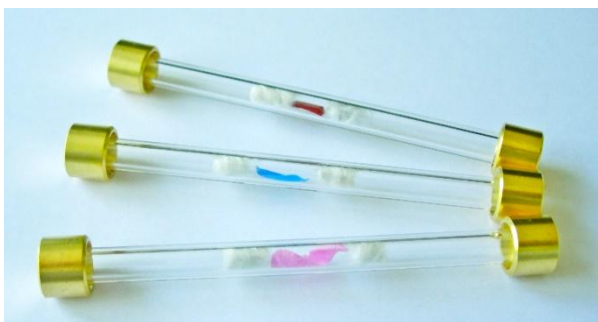


Figure 1 – Polymer samples in the glass tubes

Results and discussion

Nitrile butadiene rubber

A piece of nitrile glove was used as a sample for study. Figure 2 shows the chromatogram of the emission data. 4-Cyanocyclohexene was dominated in the nitrile butadiene rubber sample. In addition tetradecane, pentadecane, hexadecane, heptadecane, (1-Propyloctyl)benzene, (1-Ethylnonyl)benzene were detected.

Polyethylene

Polyethylene bag for containing and transporting foods was taken as an object of analysis. As shown on Figure 3, a number of alkanes and alkenes were found in the sample. 2,6-Di-tert-butylphenol was also detected that is used as a UV stabilizer.

Silicone rubber

A GC inlet septum was chosen as a silicone rubber sample. In contrast to previous experiments, volatiles from silicone rubber were emitted at a temperature of 250 °C. This temperature is close to the actual operating conditions of the septum. Figure 4 illustrates the emission profile. As expected, cyclic siloxanes were major compounds in the thermal degradation of silicone septum.

Conclusion

Direct thermal desorption with GC/MS demonstrates some advantages for study of plastic emission like the simple and fast sample preparation, the short analysis time and the small sample amount required.

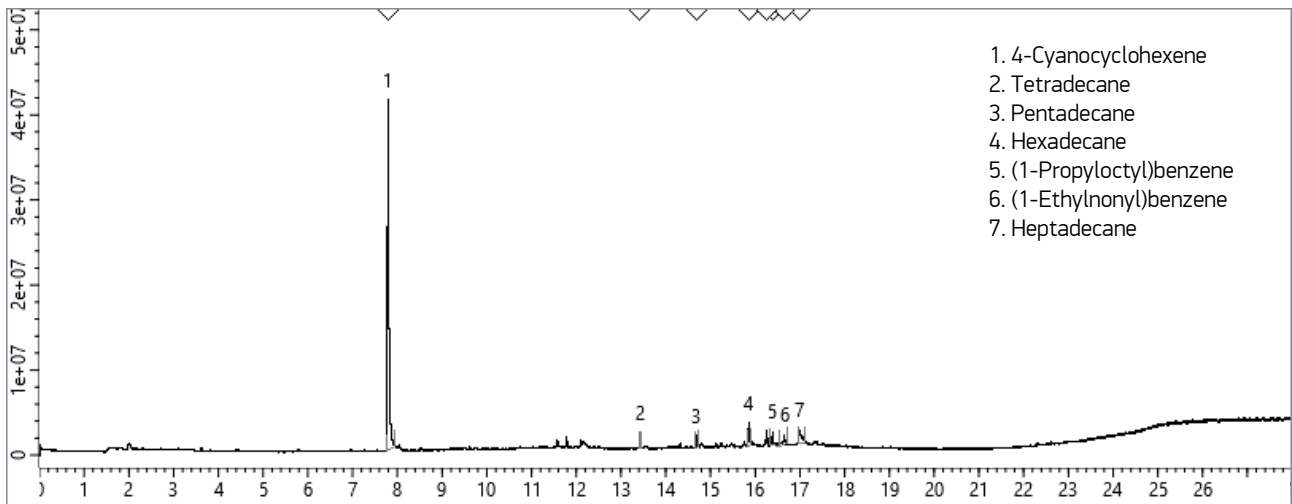


Figure 2 – Chromatogram obtained by emission from nitrile butadiene rubber

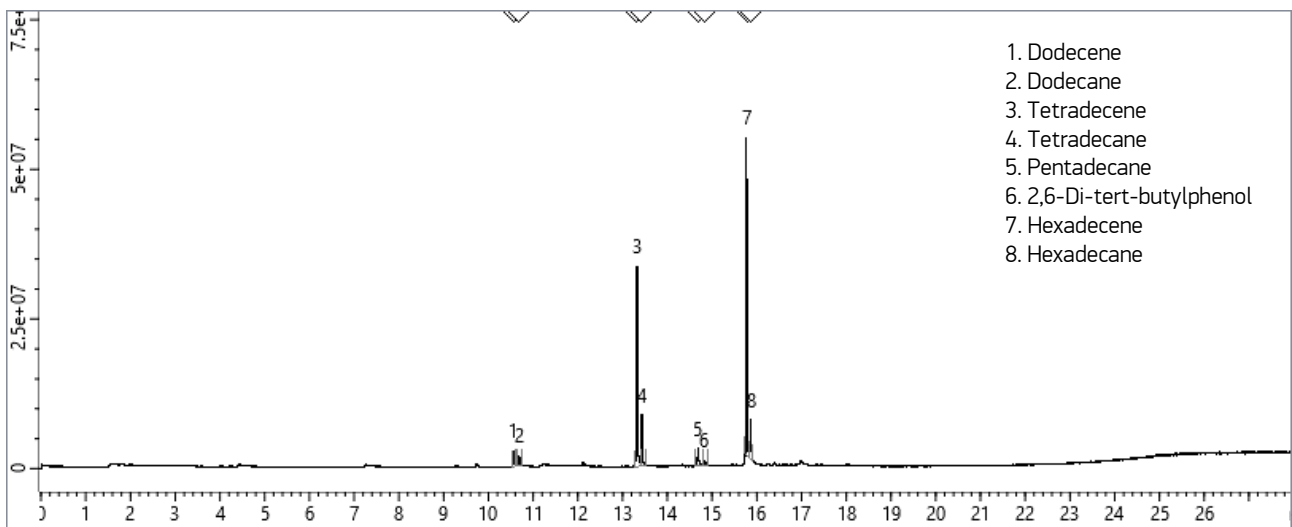


Figure 3 – Chromatogram obtained by emission from polyethylene

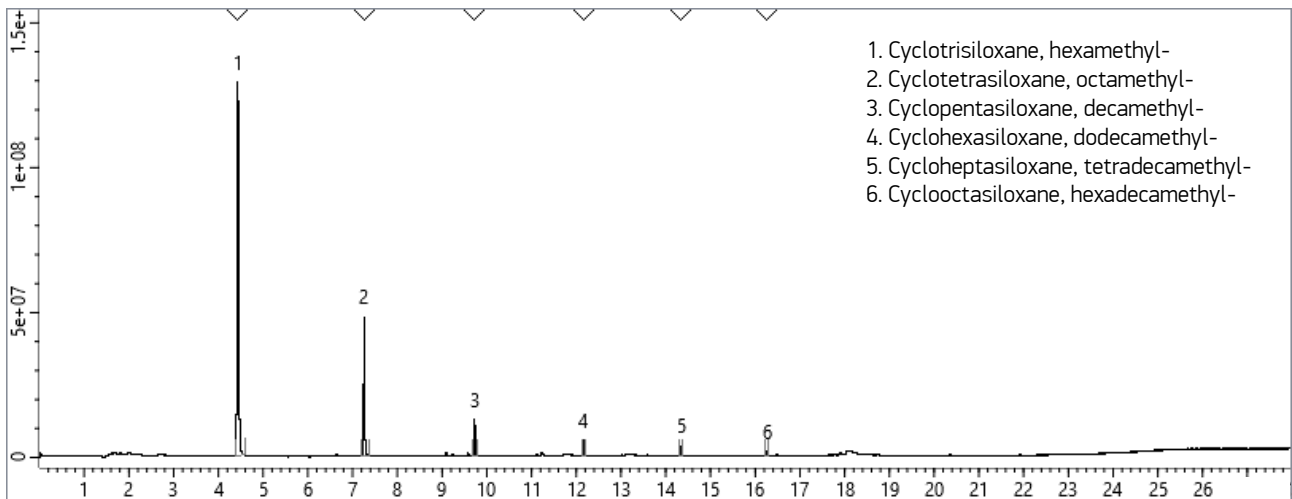


Figure 4 – Chromatogram obtained by emission from silicone rubber

