



Analysis of Aroclor Using a Chromatec Crystal GC with μ -ECD

Abstract

The Chromatec Crystal GC with micro-electron capture detector (μ -ECD) was used to analyze polychlorinated biphenyl (PCB) congeners. Separation of Aroclor mixture on a HT8 column was demonstrated.

Introduction

Since the middle of the last century, Aroclors have been used in transformers as a dielectric. However, their toxicity was later shown, as well as high resistance in the environment. In 2001, a Stockholm Convention on Persistent Organic Pollutants was signed, that aims to eliminate or restrict the production and use of persistent organic pollutants (POPs), including PCBs.

PCBs contain chlorine and the electron capture detector (ECD) is the most optimal for sensitive and selective determination. At the same time the μ -ECD, in contrast to the traditional ECD, has a smaller flow cell. It is optimized for capillary columns and shows better sensitivity.

Equipment

- Chromatec Crystal 9000 GC
- Autosampler AS-2M
- Liner Splitless Single Taper wool packed
- Detector μ -ECD
- Capillary column HT8 (50 m \times 0.22 mm \times 0.25 μ m)

Operating mode

GC			
Run time	40 min		
Column			
Carrier gas	Helium		
Constant velocity	30 cm/s		
Column temperature			
	80 °C	2 min	30 °C/min
	180 °C	0 min	5 °C/min
	300 °C	10 min	
Inlet			
Temperature	280 °C		
Mode	Splitless		
μ-ECD			
Temperature	350 °C		
Makeup gas	Nitrogen		
Makeup flow (total)	10 mL/min		
Autosampler AS-2M			
Syringe volume	10 μ L		
Aspiration speed	3		
Number of washes	5		
Viscosity delay	2 s		
Sample volume	2 μ L		
Pre injection delay	1 s		
Post injection delay	1 s		
Injection speed	9		

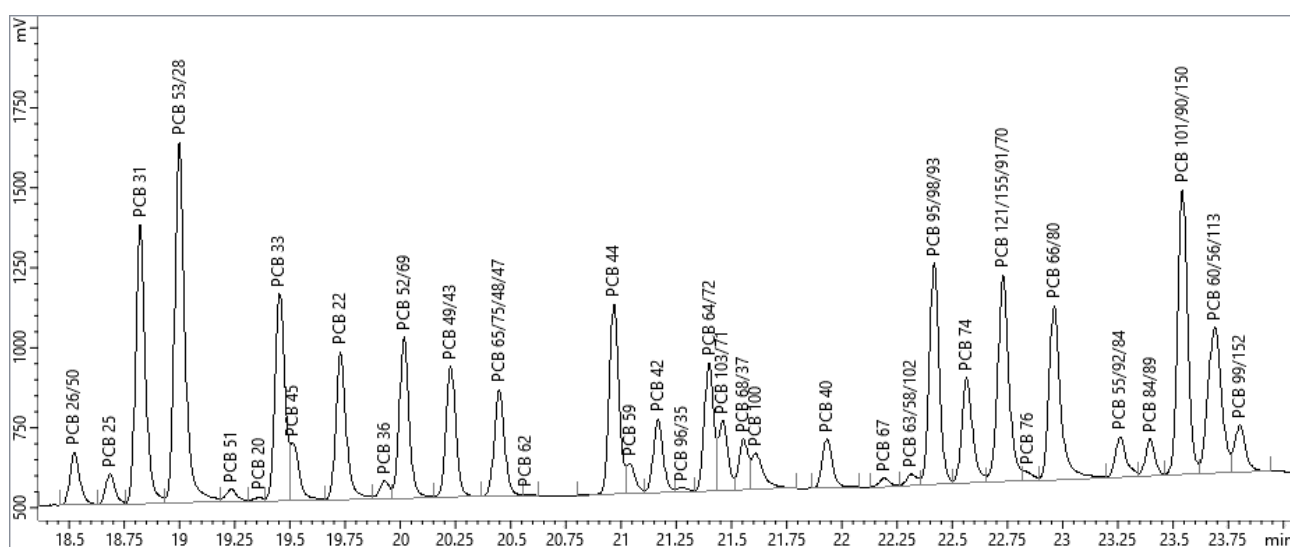
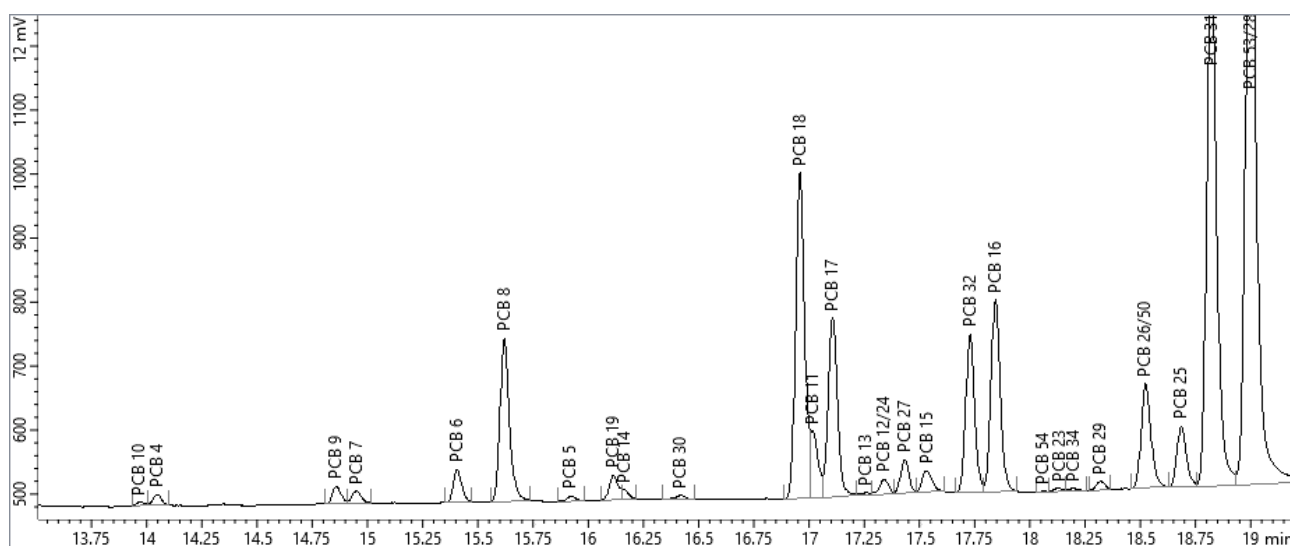
Experimental

All Aroclor standards were purchased from Supelco. Mixtures of Aroclor 1242, 1254, 1260 (1:1:1) were prepared from stock solutions by diluting with *n*-Hexane. Five working solutions were made – 10 ppb, 50 ppb, 100 ppb, 500 ppb, 1000 ppb. The concentrations are indicated for each Aroclor in the solution.

Results and discussion

Initially, the method was optimized and the PCB congeners were identified on the HT8 column. Chromatograms with congener retention order are shown in Figure 1. The data demonstrates acceptable separation of critical pairs: 31, 28 and 163, 138, 158.

Chromatograms



Aroclor working standards from 10 to 1000 ppb respectively were run for sensitivity evaluation. The chromatograms of each solution are illustrated in Figures 2-6. Within the specified range, the μ -ECD provides the determination of the analyzed mixture congeners.

Conclusion

The Chromatec Crystal GC with μ -ECD shows good sensitivity and in combination with the HT8 column ensures separation of PCB congeners.

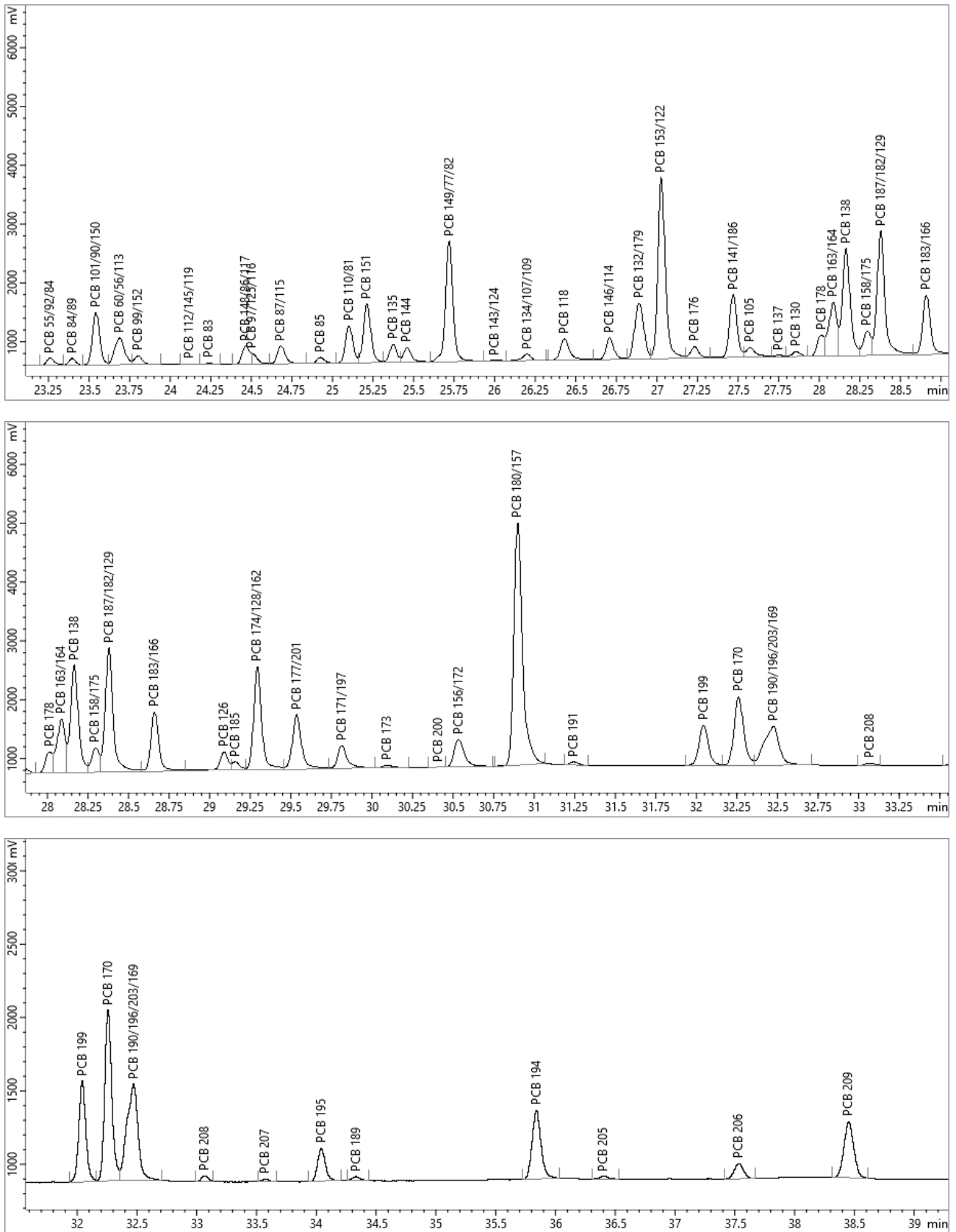


Figure 1 – PCB congener identification on HT8 column

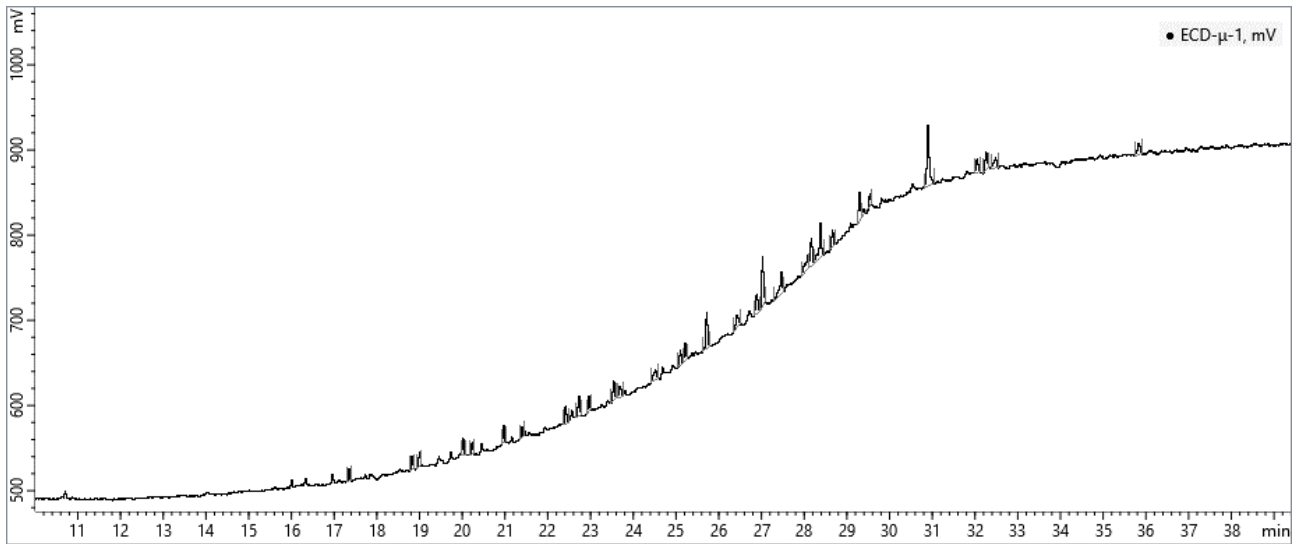


Figure 2 – Chromatogram of Aroclors 1242 + 1254 +1260, 10 ppb each

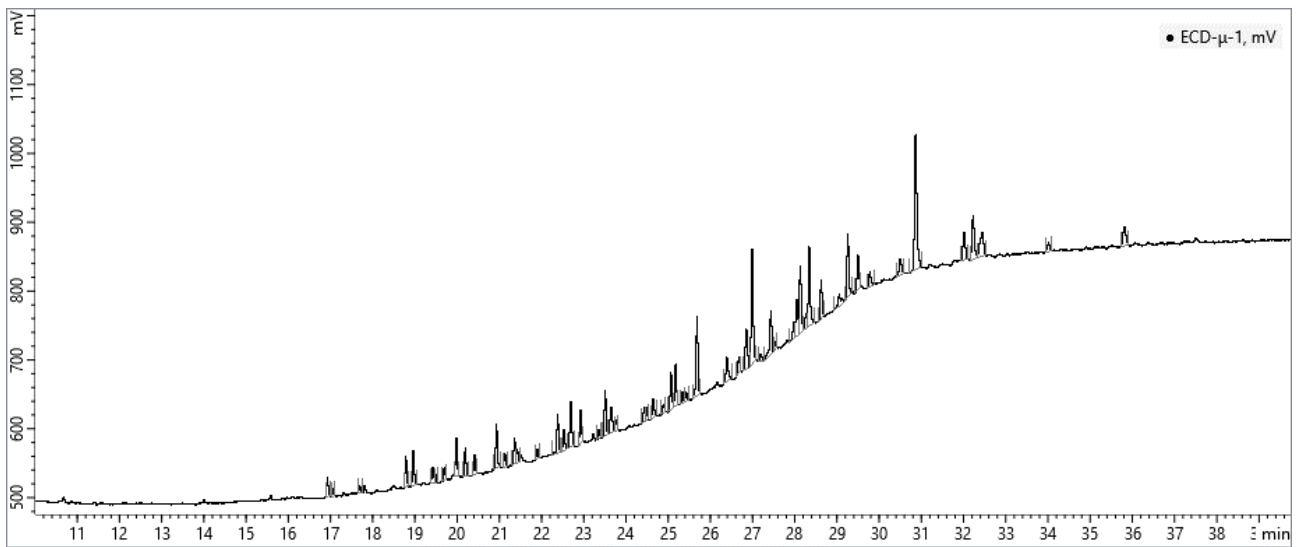


Figure 3 – Chromatogram of Aroclors 1242 + 1254 +1260, 50 ppb each

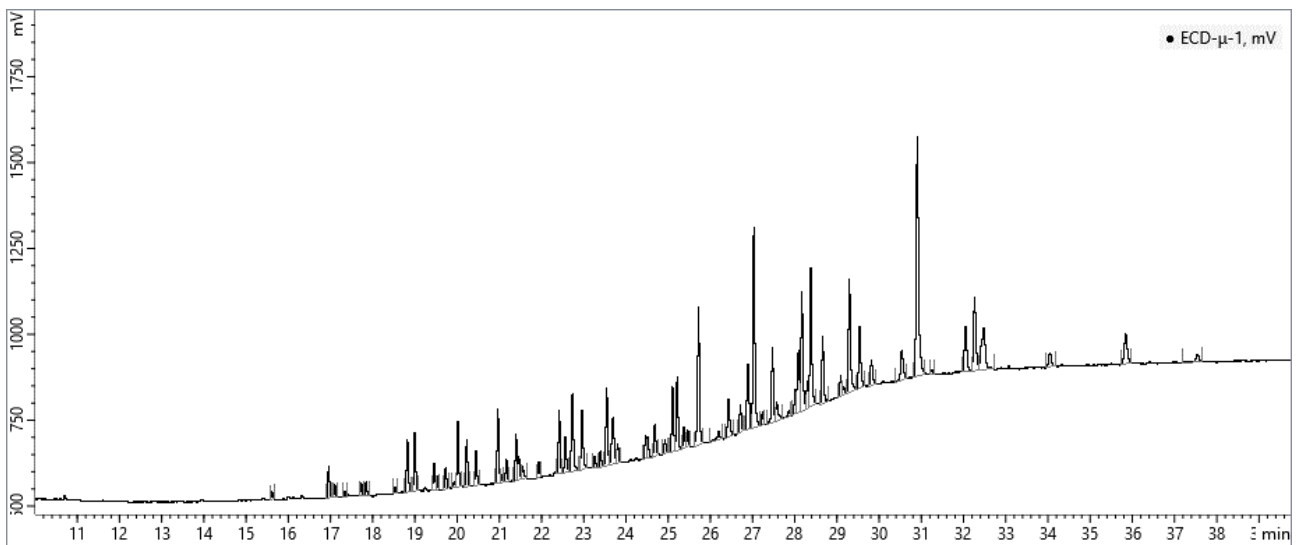


Figure 4 – Chromatogram of Aroclors 1242 + 1254 +1260, 100 ppb each

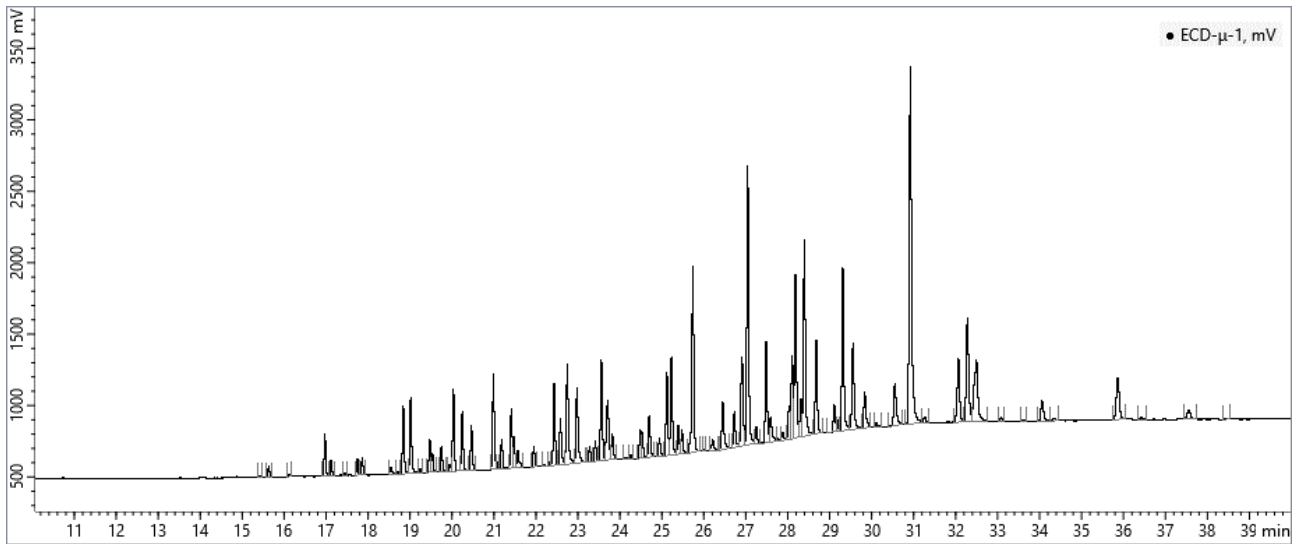


Figure 5 – Chromatogram of Aroclors 1242 + 1254 +1260, 500 ppb each

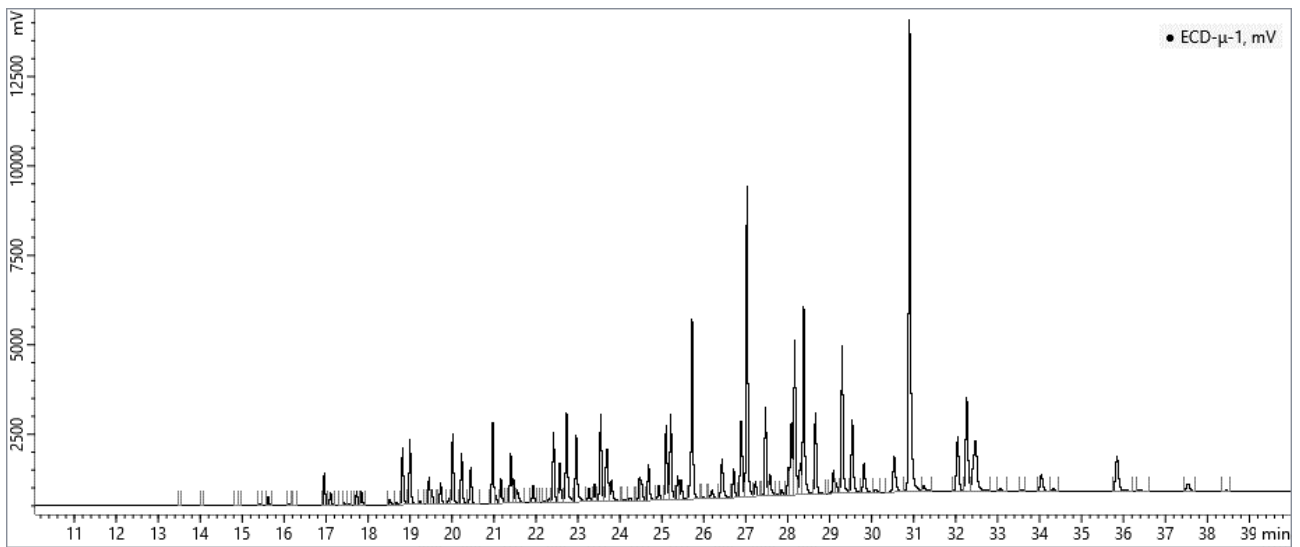


Figure 6 – Chromatogram of Aroclors 1242 + 1254 +1260, 1000 ppb each