ICP-MS : The Ultimate GC Detector

Mina Tanoshima, Steve Wilbur, and Emmett Soffey   Agilent Technologies Inc.

Agilent Technologies

Introduction

Gas chromatography (GC) is widely used for the speciation and detection of volatile organic compounds in many applications and industries. There are many detectors for GC, such as PFPD/FPD ((pulsed) flame photometric detector) or SCD (Sulfur chemiluminescence detector) for sulfur, NPD (nitrogen/phosphorus detector) for nitrogen and phosphorus and ECD (electron capture detector) for the halogens. GC is also combined with a mass spectrometer or mass selective detector (MS or MSD), both with single quadrupole or triple-quad (QQQ). However, none of these detectors is capable of providing universal, element specific quantification in the manner of ICP-MS. ICP-MS provides a unique combination of rapid multi-element/isotope analysis, with very high sensitivity.

Experimental

Instrumentation

Agilent's 7890A GC and Agilent's 7700x ICP-MS were connected using the new GC interface kit provided by Agilent (As shown in Figure 1.) to evaluate these three analyses; 1)Organo-tin, 2) Polybrominated Diphenyl Ether (PBDE), and 3) Sulfur species in diesel. This GC interface connects the two instruments via a passivated, heated Sulfinert ® tube between the GC column and the tip of the ICP injector using a special torch with a heated injector to maintain constant high temperature and inertness. The schematic is shown in Figure 2.

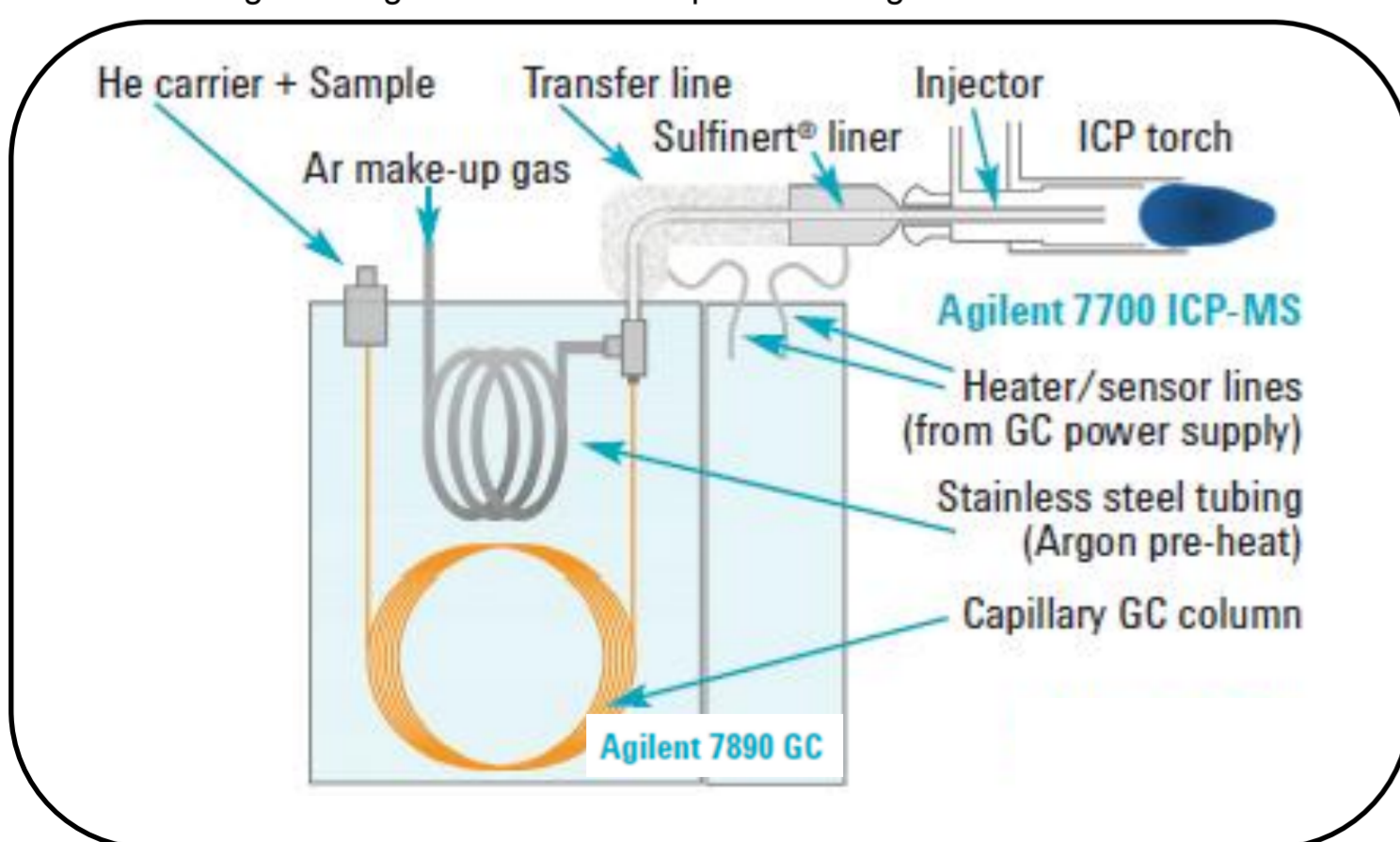Figure 1. Agilent's ICP-MS coupled to the Agilent's 7890A GC.

Figure 2. Schematic diagram for Agilent's GC-MS interface for the 7890GC/7700 ICP-MS

Results and Discussion

1) Organo-tin analysis

The toxic effects of organo-tin compounds in the environment are well known and research has begun to include matrices with human health implications, such as seafood, manufactured products, and human blood samples. In this study, a mixed organo-tin standard was analyzed. The analysis was completed in less than 12 minutes achieving detection limit of 5.9ppt for tributyl tin (TBT).
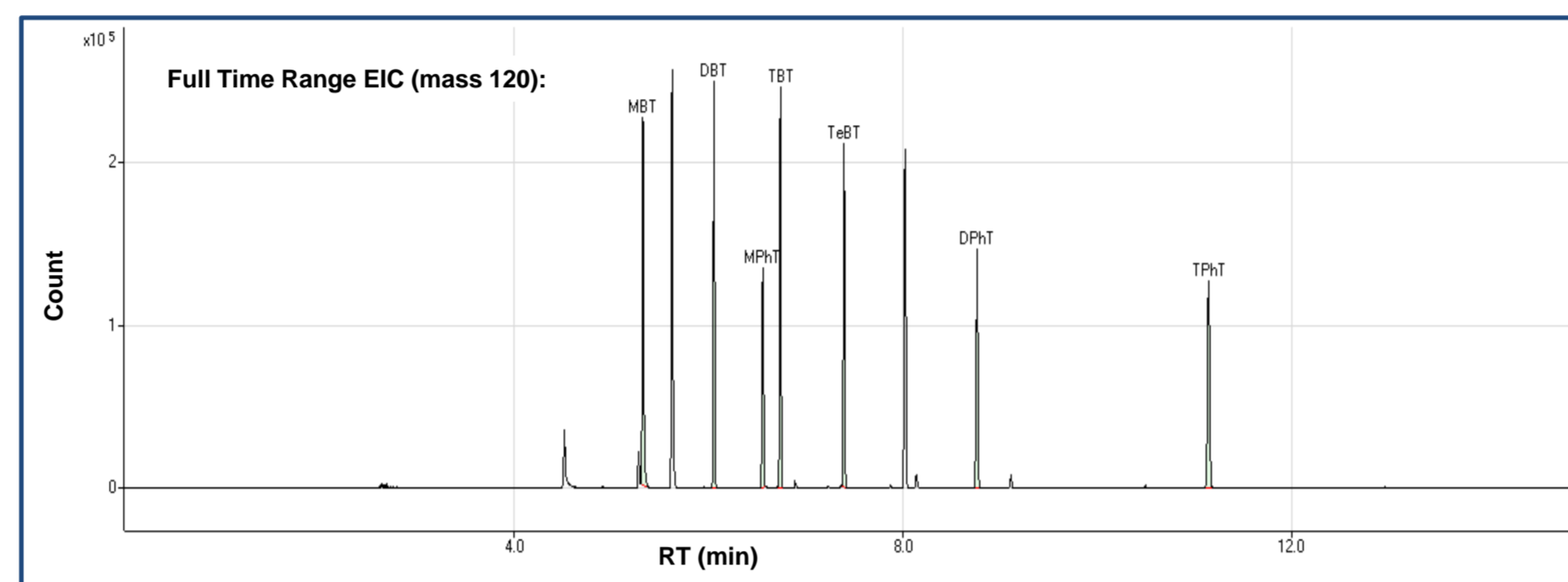

Figure 3. Chromatogram of 10ppb Organo-tin standard. The samples was delivaized beforehand.

Table 1. Method parameters for the separation of Organo-tin using the GC with ICP-MS detection.

GC		ICP-MS	
Injection	Splitless 1ul	ICP-MS model	7700x
Column	HP-5 (30m x 0.32mm i.d. x 0.25 um film thickness)	RF Power	1200 W
Oven Program	70 deg(1min) ~ 30deg/min ~190deg (0min) ~ 15deg/min ~ 270deg (4min)	Sample Depth	8 mm
		Carrier gas	0.80 L/min
Carrier gas	He at 2mL/min	Aux Gas	1.5 L/min
Inlet Temp	290 deg C	Additional Gas	No
Transfer line temp	250 deg C		
ICP injector temp	250 deg C	Monitored mass (m/z)	118,120

2) Polybrominated Diphenyl Ether (PBDE) analysis

PBDEs are widely used flame retardants added to many common household products, such as mattresses and furniture, and electronic devices. However their similarity in structure to PCBs and dioxins has raised concerns about health risks associated with their use. For this study, a PBDE mix(CIL Predominant Congener Mixture EO-5103) containing 14 different PBDEs were analyzed. (Chromatogram is shown in Figure 4.) All the PBDEs were separated successfully with good sensitivity, even for the difficult #209 (decaBDE) congener, which is thermally labile and decomposes easily during GC separation. The detection limit was150ppt for Deca BDE .
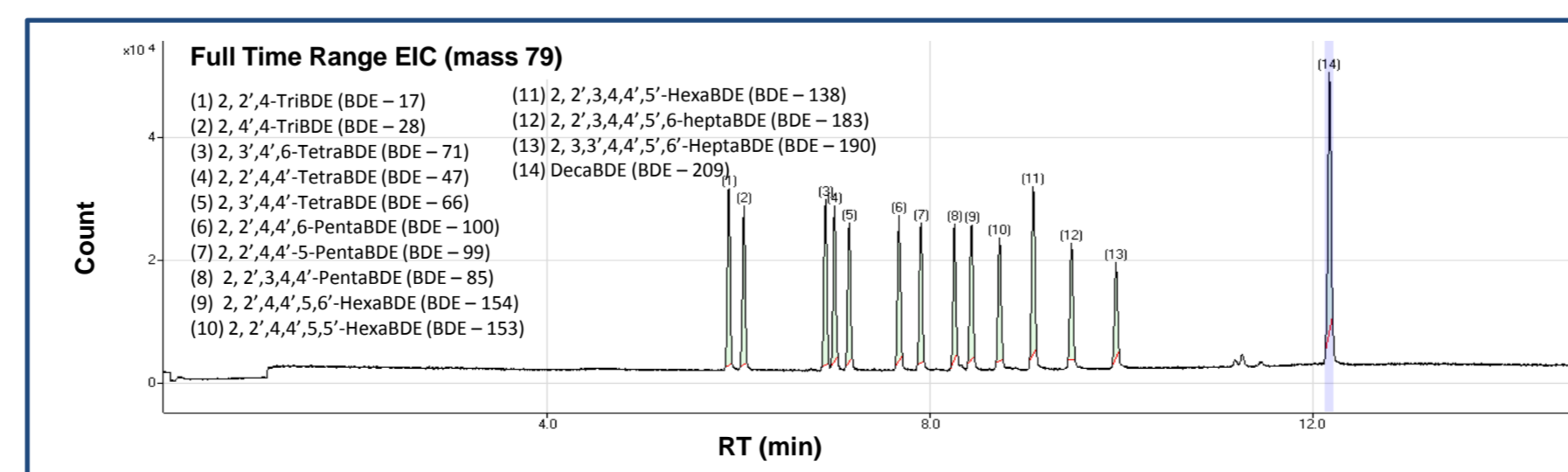

Figure 4. Chromatogram of mixed PBDE standard: 25ppb each compound except #138 (37.5ppb) and #209 (100ppb)

Table 2. Method parameters for the separation of PBDEs using the GC with ICP-MS detection.

GC		ICP-MS	
Injection	Splitless 1ul	ICP-MS model	7700x
Column	DB-5MS (5m x 0.25 mm x 0.25um film thickness)	RF Power	1250 W
Oven Program	80℃ (1min), 20℃/min -> 320℃ (5min)	Sample Depth	7 mm
Carrier gas	He at 7mL/min	Carrier gas	0.61 L/min
Inlet Temp	260 deg C	Aux Gas	1.5 L/min
Transfer line temp	250 deg C	Additional Gas	No
ICP injector temp	280 deg C	Monitored mass (m/z)	79, 81

3) Sulfur analysis in diesel fuel

Sulfur in motor fuels has been implicated in global warming and acid rain. It is also a catalyst poison for automobile catalytic converters and refinery catalytic crackers. Reducing total sulfur in motor fuels has become a critical air pollution control goal worldwide. Figure5 show the chromatogram for low sulfur in diesel samples (NIST 2724B). GC and ICP-MS operating parameters are shown in Table 3.
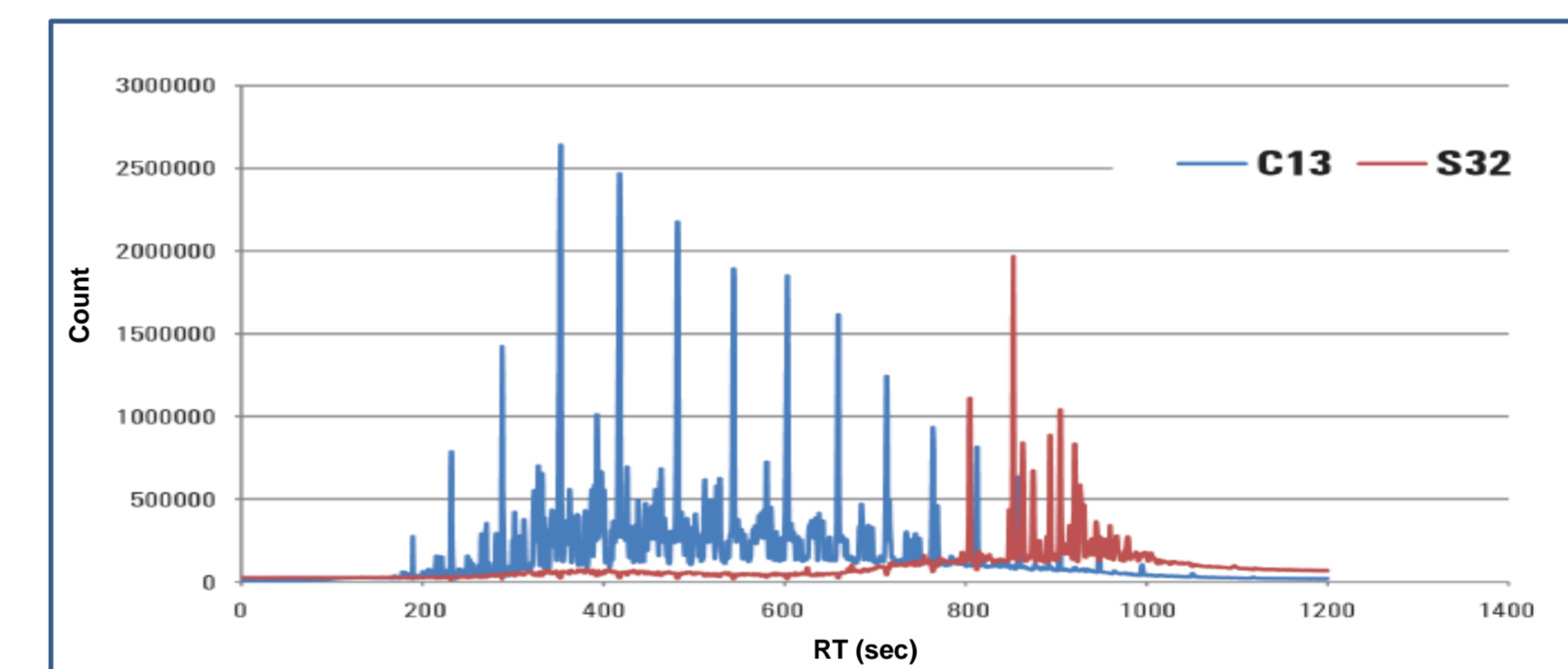

Figure 5. Carbon and sulfur extracted ion chromatograms for NIST 2724B low sulfur.

Table 3. GC and ICP-MS operating parameters.

GC		ICP-MS	
Injection	Split 1:50 1ul	ICP-MS model	7700x
Column	HP-5 (30m x 0.32mm i.d. x 0.25 um film thickness)	RF Power	1550 W
Oven Program	40deG(4min) ~ 20deG/min ~250deG (1min)	Sample Depth	8 mm
Carrier gas	He at 2.5mL/min	Carrier gas	0.80 L/min
Inlet Temp	250 degC	Aux Gas	1.5 L/min
Transfer line temp	250 degC	Additional Gas	No
ICP injector temp	260 degC	Monitored mass (m/z)	118,120

Conclusions

The use of ICP-MS as a GC detector significantly extends GC capability and expands its application. With a well-designed GC / ICP-MS interface featuring high temperature and inertness, Organo-tin , PBDE and sulfur compounds in diesel were successfully analyzed.

Those results demonstrated benefit of ICP-MS' ultimate detection limit and multi element analysis capability.