ICP-MS: The Ultimate GC Detector

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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

An Agilent 7890A GC and Agilent 7700x ICP-MS were connected using the new Agilent GC-ICP-MS interface (Figure 1) to evaluate these three analyses; 1)Organo-tin, 2) Polybrominated Diphenyl Ether (PBDE), and 3) Sulfur species in diesel. The 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 7700x ICP-MS coupled to an Agilent 7890A GC

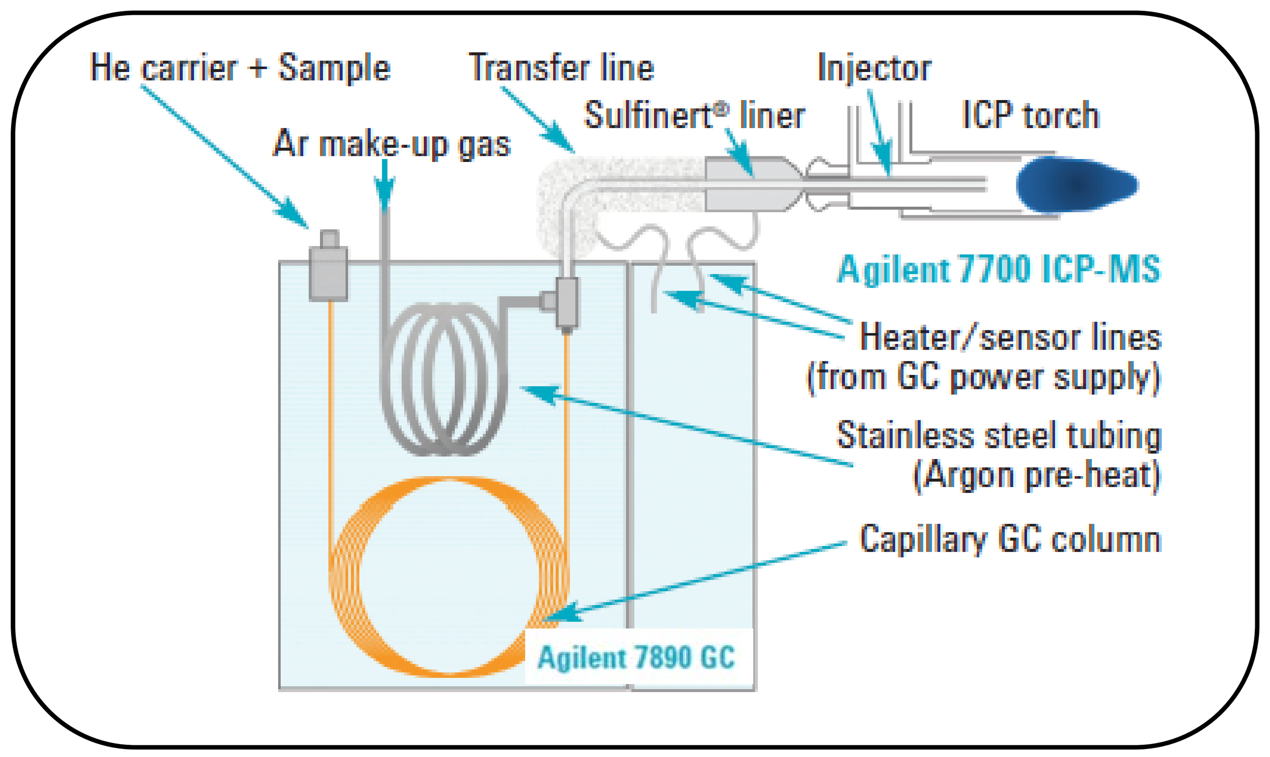

Figure 2. Schematic diagram for the Agilent GC-ICP-MS interface for the 7890GC/7700 ICP-MS

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 5.9 ppt for tributyl tin (TBT).

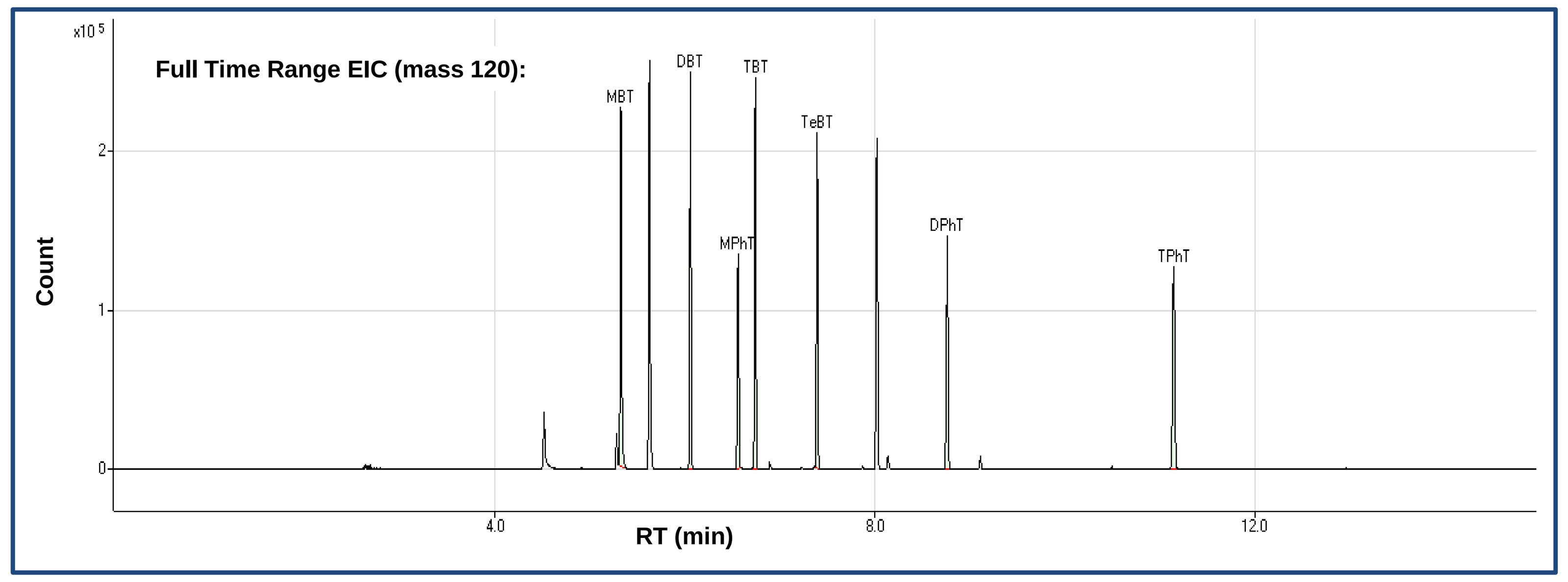

Figure 3. Chromatogram of 10 ppb organo-tin standard. The samples was derivatized beforehand

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

GC	
Injection	Splitless 1ul
Column	HP-5 (30m x 0.32mm i.d. x 0.25 um film thickness)
Oven program	70 deg(1min) ~ 30deg/min ~190deg (0min) ~ 15deg/min ~ 270deg (4min)
Carrier gas	He at 2mL/min
Inlet temp	290 deg C
Transfer line temp	250 deg C
ICP injector temp	250 deg C

ICP-MS	
ICP-MS model	7700x
RF power	1200 W
Sample depth	8 mm
Carrier gas	0.80 L/min
Aux gas	1.5 L/min
Additional gas	No
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 was150 ppt for Deca BDE .

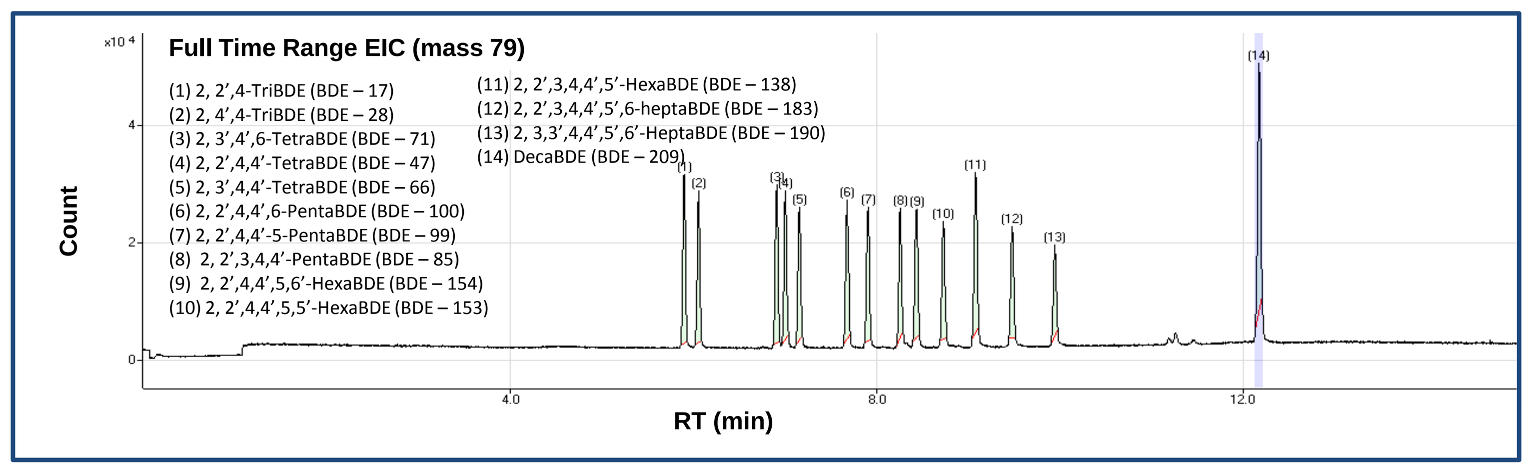

Figure 4. Chromatogram of mixed PBDE standard: 25ppb each compound except #138 (37.5 ppb) and #209 (100 ppb)

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

GC	
Injection	Splitless 1ul
Column	DB-5MS (5m x 0.25 mm x 0.25um film thickness)
Oven program	80℃ (1min), 20℃/min -> 320℃ (5min)
Carrier gas	He at 7mL/min
Inlet temp	260 deg C
Transfer line temp	250 deg C
ICP injector temp	280 deg C

ICP-MS	
ICP-MS model	7700x
RF power	1250 W
Sample depth	7 mm
Carrier gas	0.61 L/min
Aux gas	1.5 L/min
Additional gas	No
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. Figure 5 show the chromatogram for low sulfur in diesel samples (NIST 2724B). GC and ICP-MS operating parameters are shown in Table 3.

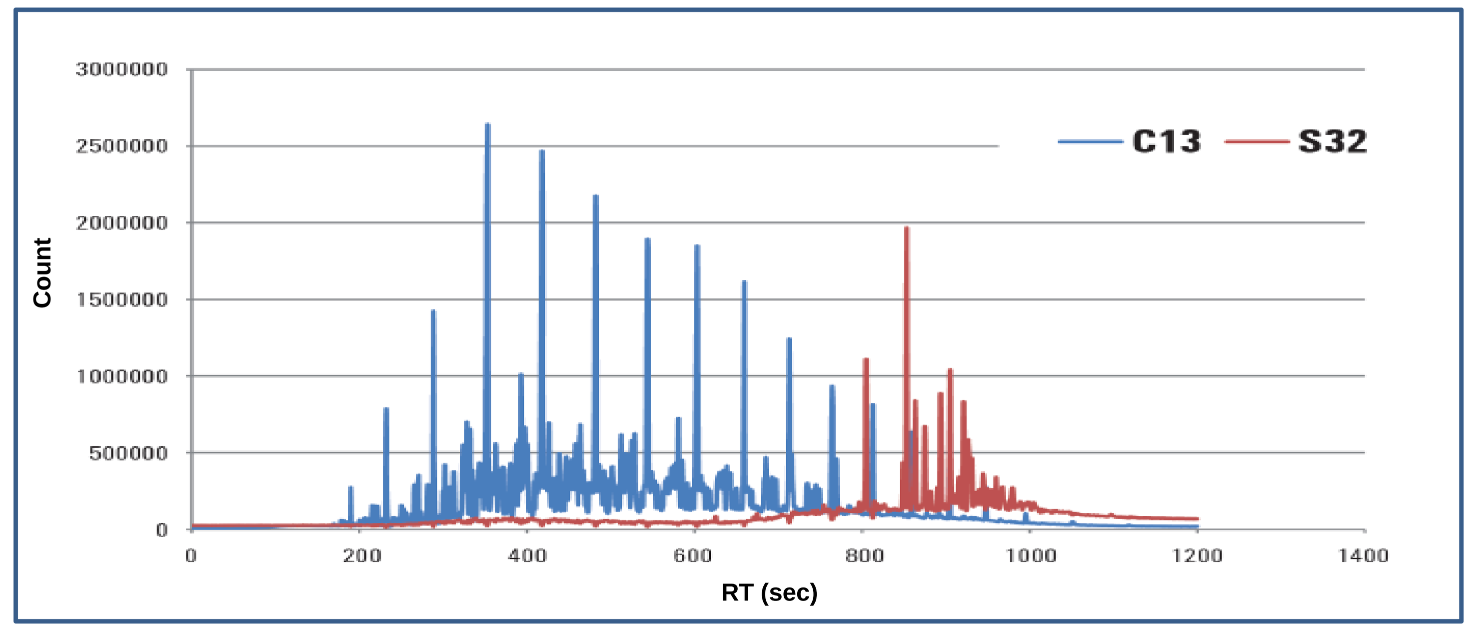

Figure 5. Carbon and sulfur extracted ion chromatograms for NIST 2724B low sulfur

Table 3. GC and ICP-MS operating parameters

GC	
Injection	Split 1:50 1ul
Column	HP-5 (30m x 0.32mm i.d. x 0.25 um film thickness)
Oven program	40deG(4min) ~ 20deG/min ~250deG (1min)
Carrier gas	He at 2.5mL/min
Inlet temp	250 degC
Transfer line temp	250 degC
ICP injector temp	260 degC

ICP-MS	
ICP-MS model	7700x
RF power	1550 W
Sample depth	8 mm
Carrier gas	0.80 L/min
Aux gas	1.5 L/min
Additional gas	No
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. These results demonstrate the ultimate detection limits and multi element analysis capability of ICP-MS.