

APPLICATION NOTE

Determination of flame retardants by Gas Chromatography – Mass Spectrometry



AN035v1; SCION Instruments

Introduction

The Restriction of Hazardous Substances Directive (RoHS) regulate hazardous substances found in electrical and electronic equipment. Polybrominated biphenyls (PBBS) and polybrominated diphenyl ethers (PBDEs) are flame retardants which are commonly regulated under RoHS. Said flame retardants are easily released into the environment during their production and during disposal of products containing these contaminants. Therefore, it is vital that controlled conditions are maintained to ensure contamination is kept to a minimum with released contaminants regulated.

This application note details the determination of PBBS and PBDEs using a Scion 8500 gas chromatograph coupled with the Scion 8700 Single Quad Mass Spectrometer. The instrument could be visualized in the Figure below (Figure 1).



Figure 1. SCION Instruments 8500 GC coupled with the 8700 MS(SQ).

Experimental

Table 1 details the analytical conditions of the GC-MS instrumentation used throughout this application.

Table 1. Analytical conditions of the GC-MS.

Part	Settings
S/SL	280°C
Column	Scion-5HT 15m x 0.25mm x 0.10µm
Oven Programme	110°C (2 mins), 40°C/min to 200°C, 10°C/min to 260°C, 20°C/min to 340°C (2 min)
Carrier Gas	Helium 1mL/min constant
Transfer Line	300°C
Source	230°C
MS	Full Scan, 100-1000Da

1mL of each PBBs and PBDE standard mixtures were prepared in 5mL volumetric flasks. Toluene was used to adjust the standard stock solution to 20µg/mL. Table 2 details the preparation method for the working solution from the standard stock solution mentioned above, with toluene as the solvent.

Table 2. Working standard preparation method.

Level	Target Conc (µg/mL)	Stock Conc (µg/mL)	Volume (µL)	Constant Volume (µL)
1	0.05	20	2.5	1
2	0.15	20	7.5	1
3	0.25	20	12.5	1
4	0.35	20	17.5	1
5	0.45	20	22.5	1

Results

The total ion chromatogram (TIC) for a 20µg/mL stock standard of PBBS and PBDEs are shown in Figures 2a and 2b, respectively with peak identification found in Table 3.

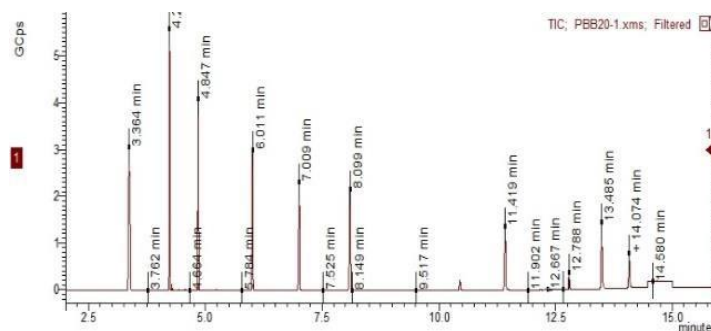


Figure 2a. TIC of 20µg/mL PBBS stock standard

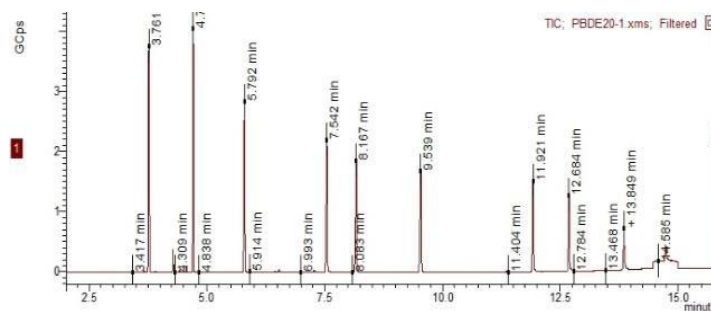


Figure 2b. TIC of 20µg/mL PBDE stock standard

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Table 3. Retention time, linear coefficients, and repeatability data of the PBBs and PBDEs. (n=10, 0.05µg/mL for repeatability).

Peak ID	Retention Time (min)	RSD %	Linear Coefficient (R ²)
PBBs	-	-	-
2-bromobiphenyl	3.36	0.02	0.99992
2,5-dobromobiphenyl	4.23	0.01	0.99992
2,4,6-tribromobiphenyl	4.84	0.01	0.99963
2,2',5,5'-tetrabromobiphenyl	6.01	0.01	0.99943
2,2',4,5',6-pentabromobiphenyl	7.01	0.006	0.99823
2,2',4,4',6,6'-hexabromobiphenyl	8.10	0.01	0.99782
2,2',3,4,4',5,5'-heptabromobiphenyl	11.42	0.007	0.99714
Octabromobiphenyl	12.80	0.006	0.99525
2,2',3,3',4,4',5,5',6-nonabiphenyl	13.49	0.005	0.99829
Decabromobiphenyl	14.06	0.005	0.99510
PBDE	-	-	-
4-monobromobiphenyl ether	3.77	0.006	0.99958
4,4'-dibromodiphenyl ether	4.71	0.03	0.99930
3,3',4-tribromodiphenyl ether	5.79	0.007	0.99855
3,3',4,4'-tetrabromodiphenyl ether	7.54	0.02	0.99724
2,2',4,4',6-pentabromodiphenyl ether	8.16	0.01	0.99588
2,2',4,4',5,6'-hexabromodiphenyl ether	9.53	0.07	0.99619
2,2',3,4,4',5,6;-heptaBDE	11.91	0.01	0.99957
2,2',3,4,4',5,5',6'-octaBDE	12.67	0.07	0.99889
2,2',3,3',4,4',5,5',6-nonbrominated diphenyl ether	13.84	0.009	0.99963
Decabromodiphenyl ether	14.73	0.0082	0.99903

Peak retention times were compared with the certificate analysis provided with the analytical standard.

The five working standards were analysed in both full scan and SIM mode, with quantification using SIM mode. Table 3 also details the linear coefficient values of the calibration curves, ranging from 0.05µg/mL to 0.45µg/mL. All coefficients were equal to or greater than 0.995. Repeatability of the system was determined through 10 replicates of each component at 0.05µg/mL with all RSD% values obtained between 0.005% an 0.03%, highlights the excellent capability of the Scion system.

Conclusions

Optimisation of the Scion GC-MS allowed excellent separation, quantification, and repeatability of polybrominated biphenyls and polybrominated diphenyl ethers, common flame retardants. Using a quantitative SIM method, it was possible to identify and quantify twenty components in fifteen minutes.

Order Information

Ordering Information for the 8300 GC	
Part	Part Number
SCION 8500 GC + SCION 8700 SQ PREMIUM EI ONLY (120V)	SCIONSQ85PRE531
SCION 8500 GC + SCION 8700 SQ PREMIUM EI ONLY (230V)	SCIONSQ85PRE532
SCION 8500 GC + SCION 8700 SQ PREMIUM EI+CI (120V)	SCIONSQ85PRC531
SCION 8500 GC + SCION 8700 SQ PREMIUM EI+CI (230V)	SCIONSQ85PRC531

Suggested Consumables	
Part	Part number
15% Graphite/85% Vespel Ferrule 1/16" with 0.4 mm hole pk/10	41312148
BTO Septa 9 mm, pk/50	CR298713
Scion-5HT 15m x 0.25mm x 0.10µm	SC32090

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