Analysis of PCBs : a GC-ECD approach to comply with EPA method 8082 requirements

APPLICATION NOTE - AN170

Introduction

Polychlorinated biphenyls (PCBs) are a class of man-made organic compounds with no known natural source, commercially produced for a wide variety of uses in 1900s, especially as coolants and dielectric fluids, stabilizing additives in PVC and plastic products, flame retardants, sealants etc. Their chemical properties, such as non-flammability, heat resistance, insulation) and physical stability contribute to their environmental persistence. Today they are included among the substances defined as Persistent Organic Pollutants (POPs).
Moreover, exposure to PCBs can cause human health issues with acute and chronic effects, including chlorine acne, irritation of mucous membranes, cancer and a variety of damages of the immune, reproductive and endocrine systems.
During the years they have also found their way to the food chain through contaminated water, accumulating in the fatty tissues of both animals and humans.
Starting from 1960s, GC/ECD configuration has been widely used for the analysis of PCBs. Initially, with packed columns, then, from 1980s it was introduced the use of capillary columns. This last configuration became in a short amount of time very popular and routinely applied for the analysis of PCBs.
The reference method for this analytical configuration is EPA Method 8082.

EXPERIMENTAL

Sample

A standard mixture of PCB congeners, purchased from Restek, Bellefonte, PA, U.S.A. was diluted in *n*-hexane to different concentrations ranging from 5 to 500 ng/mL. As described in EPA Method 8082, the standard compounds decachlorobiphenyl was used as Internal Standard (I.S.).
Prior to injection, the internal standard was automatically added by DANI Master AS Liquid Autosampler through its standard addition capability.

System Configuration and Control

GC-ECD conditions are summarized in *Table 1*.

Instrumental Conditions	
Column	Rxi-5MS 20m - 0.18 - 0.18µm
Oven Temperature Program	120°C, 12°C/min, 300°C (4min)
SL/IN Injector	280°C
Split Ratio	1:20
Carrier Gas Flow (He)	0.8 mL in constant flow
ECD Temp.	320°C
ECD Acquisition Rate	25 Hz
ECD Flow Rate	30 mL/min
Injected Volume	1 µL

Table 1: GC-ECD Conditions

Calibration

A seven-concentration levels calibration curve covering the entire analytical range was plotted for each PCB contained in the standard mixture.
For this purpose, three repetitions of the calibration curves in the concentration range of 50-500 ng/mL was carried out.

RESULTS

The standard mixture of PCB congeners was resolved on the Restec Rxi-5MS 20m - 0.18mm - 0.18µm column in less than 15 minutes. *Figure 1* shows the separation of 500 ng/mL PCB standard solution.

The linearity defined by the R^2 values of the PCB congeners standard curve ranged from 0.9964 to 0.9999. The individual PCB congener values are shown in *Table 2*.

The repeatability was evaluated on 5 consecutive injections of 200 ng/mL PCB standard solution reported in *Table 3*.

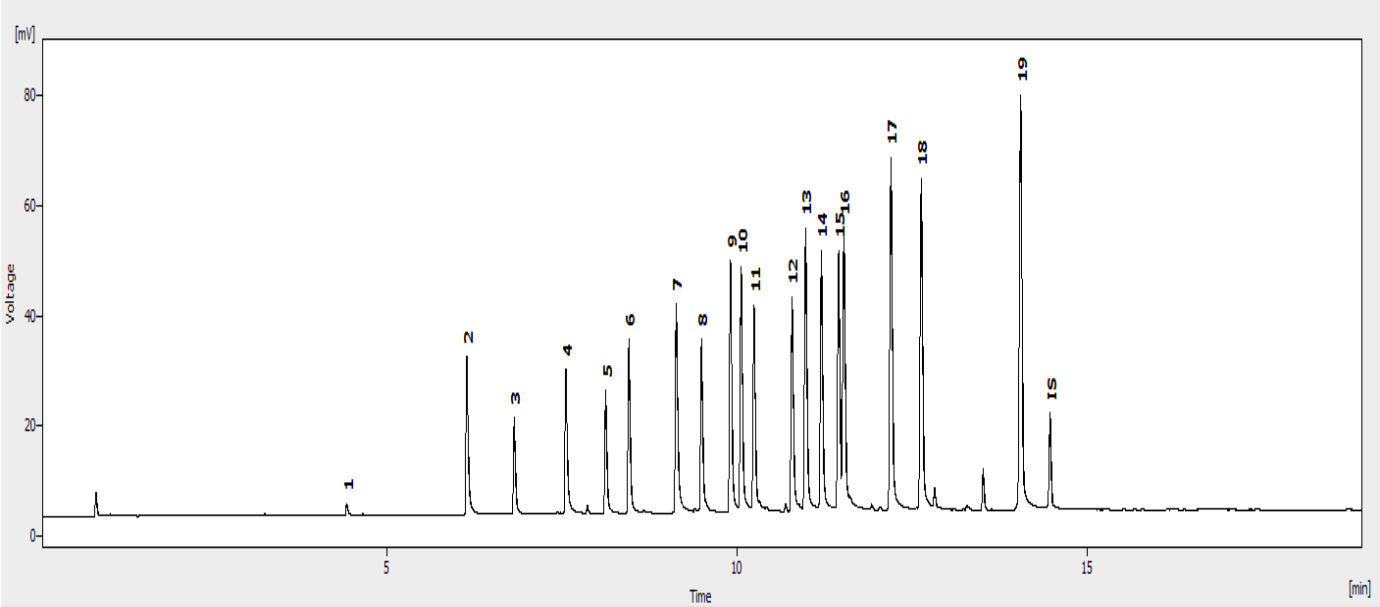

Figure 1: Separation of PCB congeners standard at 500 ng/mL

	Compounds	R^2
1	2-Chlorobiphenyl	0.9988039
2	2,3-Dichlorobiphenyl	0.9999160
3	2,2',5-Trichlorobiphenyl	0.9999330
4	2,4',5-Trichlorobiphenyl	0.9999384
5	2,2',5,5'-Tetrachlorobiphenyl	0.9997998
6	2,2',3,5'-Tetrachlorobiphenyl	0.9998669
7	2,3',4,4'-Tetrachlorobiphenyl	0.9994819
8	2,2',4,5,5'-Pentachlorobiphenyl	0.9993930
9	2,2',3,4,5'-Pentachlorobiphenyl	0.9987134
10	2,3,3',4',6-Pentachlorobiphenyl	0.9988497
11	2,2',3,5,5',6-Hexachlorobiphenyl	0.9990608
12	2,2',4,4',5,5'-Hexachlorobiphenyl	0.9977757
13	2,2',3,4,5,5'-Hexachlorobiphenyl	0.9971231
14	2,2',3,4,4',5'-Hexachlorobiphenyl	0.9980708
15	2,2',3,4',5,5',6-Heptachlorobiphenyl	0.9964768
16	2,2',3,4,4',5',6-Heptachlorobiphenyl	0.9995501
17	2,2',3,4,4',5,5'-Heptachlorobiphenyl	0.9978048
18	2,2',3,3',4,4',5-Heptachlorobiphenyl	0.9976461
19	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	0.9987217

Table 1: Individual PCB congener values

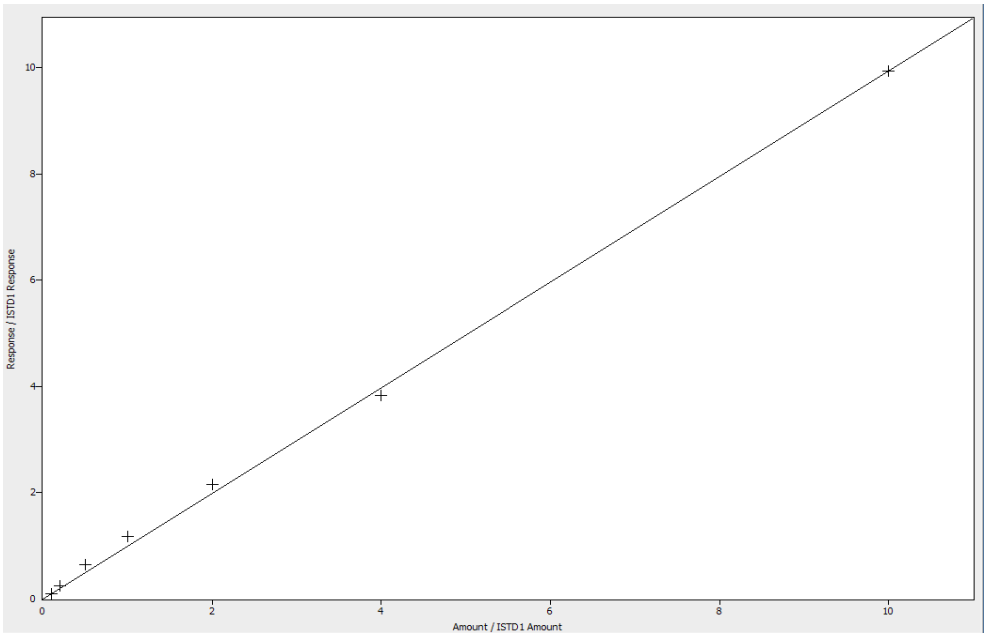

Figure 2 : 2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl calibration curve from 5 to 500 ng/mL

Compound	RSD% Retention Time	RSD% Area	RSD% Height
1	0.04	2.66	2.30
2	0.02	2.61	3.04
3	0.03	2.29	3.04
4	0.02	2.44	1.98
5	0.02	2.20	2.93
6	0.03	2.58	1.77
7	0.02	2.35	2.71
8	0.02	2.42	2.05
9	0.01	2.38	2.08
10	0.02	2.44	2.36
11	0.02	2.23	1.63
12	0.01	2.05	1.98
13	0.02	2.05	1.94
14	0.03	2.13	0.95
15	0.02	1.35	2.46
16	0.02	1.89	1.72
17	0.02	2.32	0.75
18	0.01	2.52	2.64
19	0.01	1.95	1.69
I.S.	0.01	2.15	2.15

Table 3 : Repeatability

CONCLUSION

The GC-ECD method has proven to be a well-established method for the analysis of PCBs.
Method detection limits for this type of analysis are in the order of one to fractional parts per billion range. These limits are easily reachable with the DANI Master GC equipped with an Electron Capture Detector as illustrated above.
The GC-ECD method, in fact, provides good specificity, hence sensitivity satisfying the requirements of the EPA official method.

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