Gas Chromatography

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The Determination of Polychlorinated Biphenyls (PCB) Using EPA Method 8082

Introduction

Polychlorinated biphenyls (PCB) are considered persistent organic pollutants under the Stockholm Convention of Persistent Organic Pollutants and have not been manufactured in the U.S. since they were banned in 1979. They are relatively chemically inert and do not decompose readily, being resistant to oxidation, and are only very slightly soluble in water. Prior to being banned, PCBs were widely used in closed systems such as electrical transformers, capacitors and as heat transfer fluids. They were also used in adhesives, paint, plasticizers and machining oils.

Production in the U.S. was wholly from the Monsanto company, who manufactured PCBs under the name Aroclor from 1930 to 1977, using a naming scheme that generally followed a four number convention with the first two digits the number of carbons and the last two digits the percentage of chlorine by mass. Aroclor 1260 (12 carbons and 60% chlorine by mass) was used in electrical equipment up until 1950, when it was replaced by Aroclor 1242 through the next 20 years, until Aroclor 1016 (12 carbons and 42% chlorine) was produced.

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In all cases the Aroclor sold was a mixture of 209 congeners that can result in a challenging separation and quantitation. There are two main options for detection at the required limits of quantitation; typically Mass Spectrometry (MS) or Electron Capture Detection (ECD). This paper describes the application of an ECD for the quantitation and identification of the PCBs found in Aroclor 1260 and Aroclor 1016.

The Environmental Protection Agency (EPA) has released method 8082 for the analysis of PCBs as Aroclors, having tested the method against a select group of 19 congeners common to many Aroclor mixtures. The analysis of Aroclors and PCBs is complicated by matrix effects and the method allows the analysis of Aroclors, using those selected congeners, and also enables the analysis of other PCBs if appropriate quality controls are in place.

Experimental

Table 1. GC conditions: Clarus® 580 GC.

Capillary Injector	225 °C	Electron Capture Detector	300 °C	60 ml/min Nitrogen make up
Oven Program	150 °C (Hold 1 min)	Ramp 25 °C/min		
	280 °C (Hold 0.6 min)	Ramp 35 °C/min		
	315 °C (Hold 0 min)			
Analytical column	Elite CLP 15 m x 0.530 mm x 0.5 µm			

A splitless injection was performed with 1 µL injected into a 4 mm SGE deactivated single taper liner without glass wool.

Results

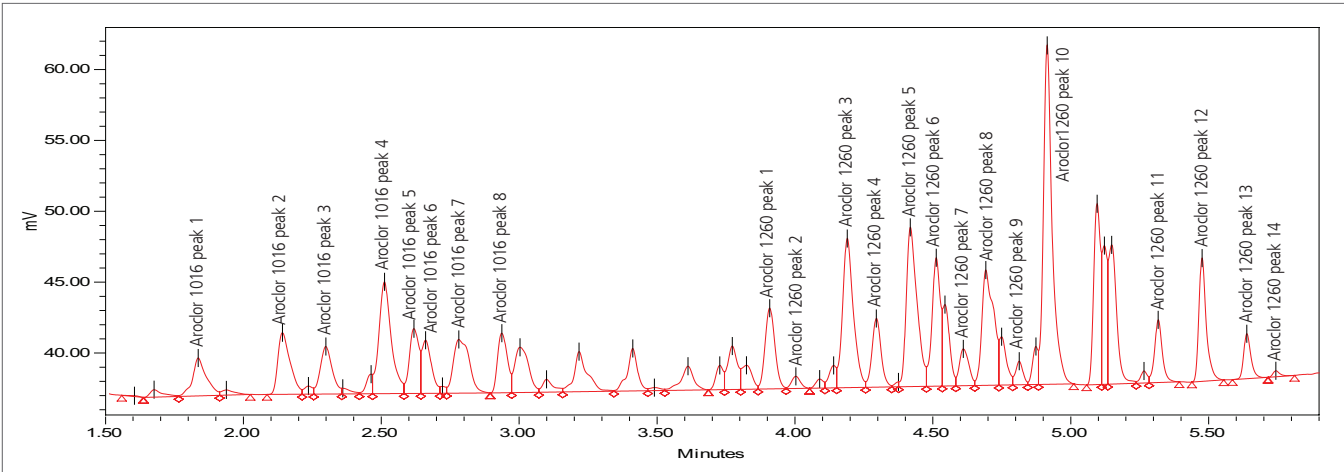A standard typical Aroclor chromatogram is shown in Figure 1.

Figure 1. Standard chromatogram of mixed Aroclor 1016 and Aroclor 1260.

A standard containing Aroclor 1016 and Aroclor 1260 will contain many of the peaks that are present in other Aroclor samples. The analyst therefore calibrates on EPA Method 8082 to cover the broad range of available congeners and it should be noted that it is also possible to use EPA Method 8082 for other congeners if appropriate resolution and reporting steps are completed.

The method requires that a five point calibration is obtained for linearity and to establish the response factor (RF) for a minimum of 5 peaks from each Aroclor. The calibration curve generated was from standards of 0.025, 0.05, 0.2, 0.4, and 0.8 µg/mL prepared from a commercial stock standard diluted in iso-octane.

The RF for each analyte is automatically calculated for each injection and it is possible to create a custom calculation using the Waters® Empower® 3 software that will average the RF for each analyte based on the calibration curve.

An additional custom field will then calculate the amount from the peak area and Average RF as described in Figure 3.
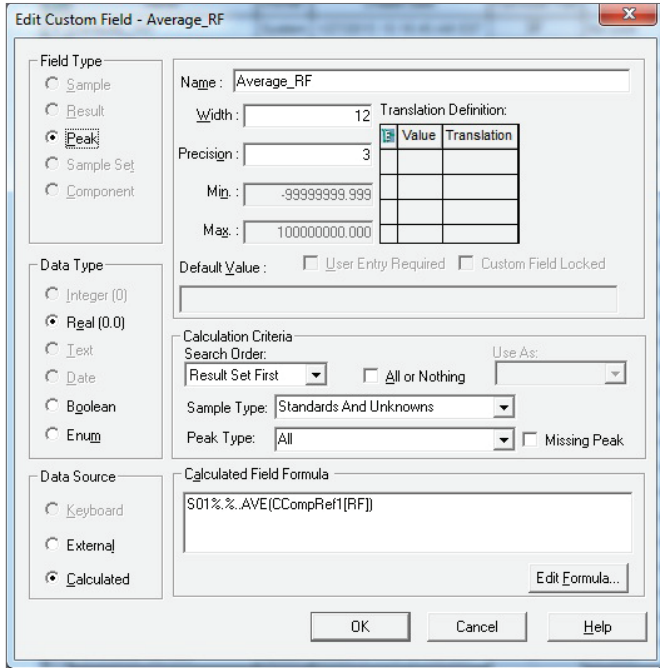

Figure 2. Custom calculation to establish the Average RF for the detailed reference component.

| | Sample Sets | Injections | Channels | Methods | Result Sets | Results | Peaks | Fractions | Sign Offs | Curves | View Filters | **Custom Fields** | Audit Trails |

🗉	Name	Field Type	Type	Width	Precision	Minimum	Maximum	Default	Source	Required	Formula
1	Average_RF	Peak	Real (0.0)	12	3	-100000000	100000000		Calculated	☐	S01%.%..AVE(CCompRef1[RF])
2	Calculated_Amount	Peak	Real (0.0)	12	3	-100000000	100000000		Calculated	☐	Area/Average_RF
3	Column Name	Sample	Text	32	0	0	0		Keyboard	☐	
4	Column Serial Number	Sample	Text	32	0	0	0		Keyboard	☐	
5	Dilution	Sample	Real (0.0)	11	4	0	1000000	1.0	Keyboard	☐	
6	Level Values	Sample	Enum	32	0	0	0		Keyboard	☐	
7	Sample Matrix	Sample	Enum	32	0	0	0		Keyboard	☐	
8	SampleName	Sample	Text	32	0	0	0		Keyboard	☐	
9	SampleWeight	Sample	Real (0.0)	11	4	0	1000000	1.0	Keyboard	☐	

Figure 3. Calculated_Amount now entered as a custom calculation.

The requirements of EPA Method 8082 are to quantify on three to five selected peaks. The Empower® 3 software integrates all of the standard peaks, therefore only one integration method is required. The analyst can then filter the Result table for those peaks that are not compromised by other Aroclors or matrix effects to obtain the correct result.

Filtering is achieved in the report result table by simply selecting the analytes of interest in the data filtering option shown in Figure 5.

Initially all of the analytes are present in the report before the analyst filters the peaks of interest.

Processed Channel: Channel B

	Peak Name	Processed Channel	Retention Time (min)	Area	Height	Calculated Amount (ng)
1	Aroclor 1016 peak 1	Channel B	1.836	9352	2675	0.106
2	Aroclor 1016 peak 2	Channel B	2.141	13335	4345	0.106
3	Aroclor 1016 peak 3	Channel B	2.299	10121	3358	0.105
4	Aroclor 1016 peak 4	Channel B	2.512	24688	7893	0.107
5	Aroclor 1016 peak 5	Channel B	2.618	11046	4572	0.109
6	Aroclor 1016 peak 6	Channel B	2.661	9142	3752	0.107
7	Aroclor 1016 peak 7	Channel B	2.781	15257	3790	0.109
8	Aroclor 1016 peak 8	Channel B	2.937	10372	4207	0.109
9	Aroclor 1016 peak 9	Channel B	3.003	11165	3195	0.108
10	Aroclor 1260 peak 1	Channel B	3.908	14081	5699	0.108
11	Aroclor 1260 peak 2	Channel B	4.003	1925	858	0.014
12	Aroclor 1260 peak 3	Channel B	4.190	28715	10528	0.109
13	Aroclor 1260 peak 4	Channel B	4.295	11063	4851	0.109
14	Aroclor 1260 peak 5	Channel B	4.418	30541	11245	0.109
15	Aroclor 1260 peak 6	Channel B	4.512	18900	9057	0.110
16	Aroclor 1260 peak 7	Channel B	4.611	6202	2596	0.106
17	Aroclor 1260 peak 8	Channel B	4.692	24172	8133	0.108

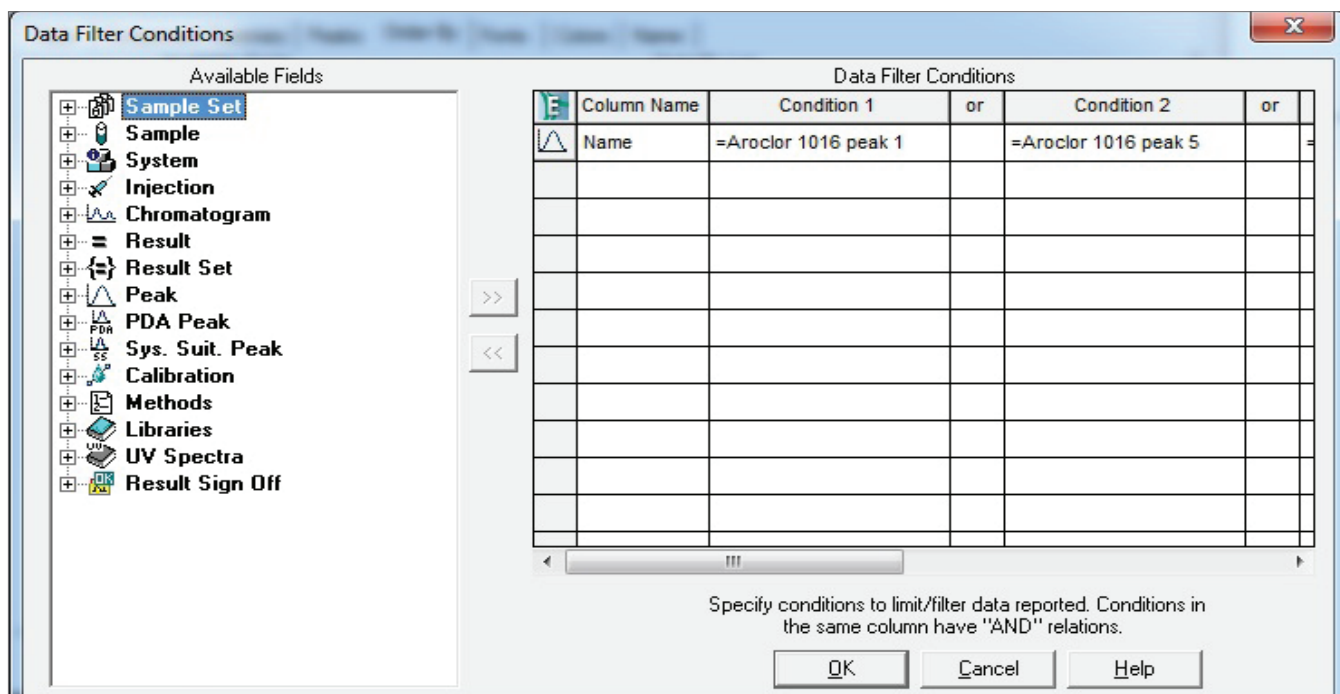*Figure 4.* All analytes are present in the initial report.

Figure 5. Entering the filter conditions, selecting the peaks by name.

Aroclor 1016 Results

Processed Channel: Channel B

	Peak Name	Processed Channel	Retention Time (min)	Area	Height	Calculated Amount (ng)
1	Aroclor 1016 peak 1	Channel B	1.836	9352	2675	0.106
2	Aroclor 1016 peak 5	Channel B	2.618	11046	4572	0.109
3	Aroclor 1016 peak 6	Channel B	2.661	9142	3752	0.107
4	Aroclor 1016 peak 7	Channel B	2.781	15257	3790	0.109
5	Aroclor 1016 peak 9	Channel B	3.003	11165	3195	0.108
Mean						0.108

Figure 6. Aroclor 1016 peaks selected and averaged.

Aroclor 1260 Results

Processed Channel: Channel B

	Peak Name	Processed Channel	Retention Time (min)	Area	Height	Calculated Amount (ng)
1	Aroclor 1260 peak 3	Channel B	4.190	28715	10528	0.109
2	Aroclor 1260 peak 4	Channel B	4.295	11063	4851	0.109
3	Aroclor 1260 peak 5	Channel B	4.418	30541	11245	0.109
4	Aroclor 1260 peak 7	Channel B	4.611	6202	2596	0.106
5	Aroclor 1260 peak 9	Channel B	4.813	3094	1661	0.106
Mean						0.107

Figure 7. Aroclor 1260 peaks selected and averaged.

Having the ability to selectively choose the best peaks that contain the least matrix interference or overlap from other Aroclors, allows for more accurate quantitation of the chosen analytes. This selection process is fast and easy and does not require further processing. The five peaks selected are then analyzed in the report for the final quantification result.

A 100 µg/mL standard was prepared and analyzed to validate the calculations and the result was determined to be within 10% of the expected value showing the applicability of the instrument and calculations.

Conclusion

The Empower® 3 chromatography data software (CDS) can easily complete the calculations of EPA Method 8082 with a rapid and simple filtering of the result table by analyte name, easily quantifying samples that are in a challenging matrix. No separate integration is required which decreases the analyst work load and increases productivity.