Identification of Aroclors: EPA 8082

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Polychlorinated biphenyls (PCB's) are a family of 209 manmade chemicals with varying numbers of chlorine atoms attached in varying positions to two connected benzene rings. PCB's are classified by the degree of substitution of the biphenyl rings. With the exception of Aroclor 1016, which contains 41% chlorine by weight, the last two digits of the numerical designation represent the weight percentage of the chlorine in the mixture.

EPA Method 8082 is used to determine the concentrations of polychlorinated biphenyls as Aroclors or as individual congeners in extracts from solid and aqueous matrices. Aroclors are not completely separated in the analysis; rather a unique fingerprint is obtained. A lab performing this analysis would be required to determine the composition of a mix of Aroclors by identifying the unique patterns within the chromatogram.

PCB analysis has become a very hot topic in the environmental industry lately. Many labs are beginning to specialize in this PCB analysis and are attempting to separate co-planar ring systems prior to analysis by HPLC. Co-planar ring identification is necessary because some of the isomers show carcinogenic character at picogram concentrations.

Experimental

Instrumentation: Analysis was performed using an HP 6890 Gas Chromatograph (Agilent Technologies, Palo Alto, California, USA) with µ-ECD detection equipped with HP Chemstation software (Version A.09.01) used for data analysis and a G2614A autosampler from Agilent. The GC columns used for analysis were Zebron (Phenomenex, Torrance, CA, USA) ZB-5, 30m x 0.32mm x 0.25µm; ZB-35, 30m x 0.32mm x 0.25µm, and a ZB-5, 15m x 0.32mm x 0.25µm. Carrier Gas was UHP grade Helium. Make-up gas for micro ECD was a P-5 mixture, 5% methane in 95% argon. Standards were ordered as Aroclor mixtures (100µg/mL in hexane) and decachlorobiphenyl solution (1000µg/mL in toluene) from Ultra Scientific (North Kingstown, RI). All other chemicals were of HPLC grade.

Sample Preparation: Single Aroclor samples were prepared by combining 100µL of Aroclor sample and 3µL of decachlorobiphenyl solution. Aroclor mixtures were made by mixing 50µL of each Aroclor and adding 3µL of decachlorobiphenyl solution.

Chromatographic Conditions: Oven parameters for all injections with the ZB-5, 15 meter column, started at 150°C and ramped at 12°C/min to 300°C. Conditions for both ZB-5 and ZB-35, 30meter columns, started at 120°C for 1 minute and ramped to 300°C at 9°C/min, hold for 5 minutes. Other conditions remained constant for all columns. Injection volumes were 1µL and split 15:1 at 250°C. Carrier gas flow was at 1.7mL/min. Detector temperature was 350°C with makeup flow of 60.0mL/min.

Results

Aroclors and mixtures of Aroclors were analyzed using three Zebron brand columns commonly used for this application. The Aroclors that were analyzed were Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268. Chromatograms for single Aroclor mixtures are shown in *Figure 1* for a ZB-5, 15m x 0.32mm x 0.25µm, *Figure 3* for a ZB-5, 30m x 0.32mm x 0.25µm, and *Figure 5* for a ZB-35, 30m x 0.32mm x 0.25µm column. Identities of the Aroclors that are represented are listed in the legend below the figure. The first peak in all plots is the solvent (hexane) and the last peak is decachlorobiphenyl. As a general rule, the more substituted the biphenyl rings, the greater the retention of the molecule. This can be seen as the percent of chlorine increases, the grouping of the Aroclor shifts to longer retention times closer to the decachlorobiphenyl.

Some Aroclors were also mixed and analyzed. The chromatograms for these mixtures are presented in *Figure 2* for ZB-5 15m x 0.32mm x 0.25µm, *Figure 4* for ZB-5, 30m x 0.32mm x 0.25µm, and *Figure 6* for a ZB-35, 30m x 0.32mm x 0.25µm column. Identities of the mixtures are listed in the legend below the figure. Again, the first peak in the spectrum is the solvent (hexane) and the last peak is decachlorobiphenyl.

Ordering Information

Order Number	Description
7EM-G002-11	ZB-5 - 15m x 0.32mm x 0.25µm
7HM-G002-11	ZB-5 - 30m x 0.32mm x 0.25µm
7HM-G003-11	ZB-35 - 30m x 0.32mm x 0.25µm

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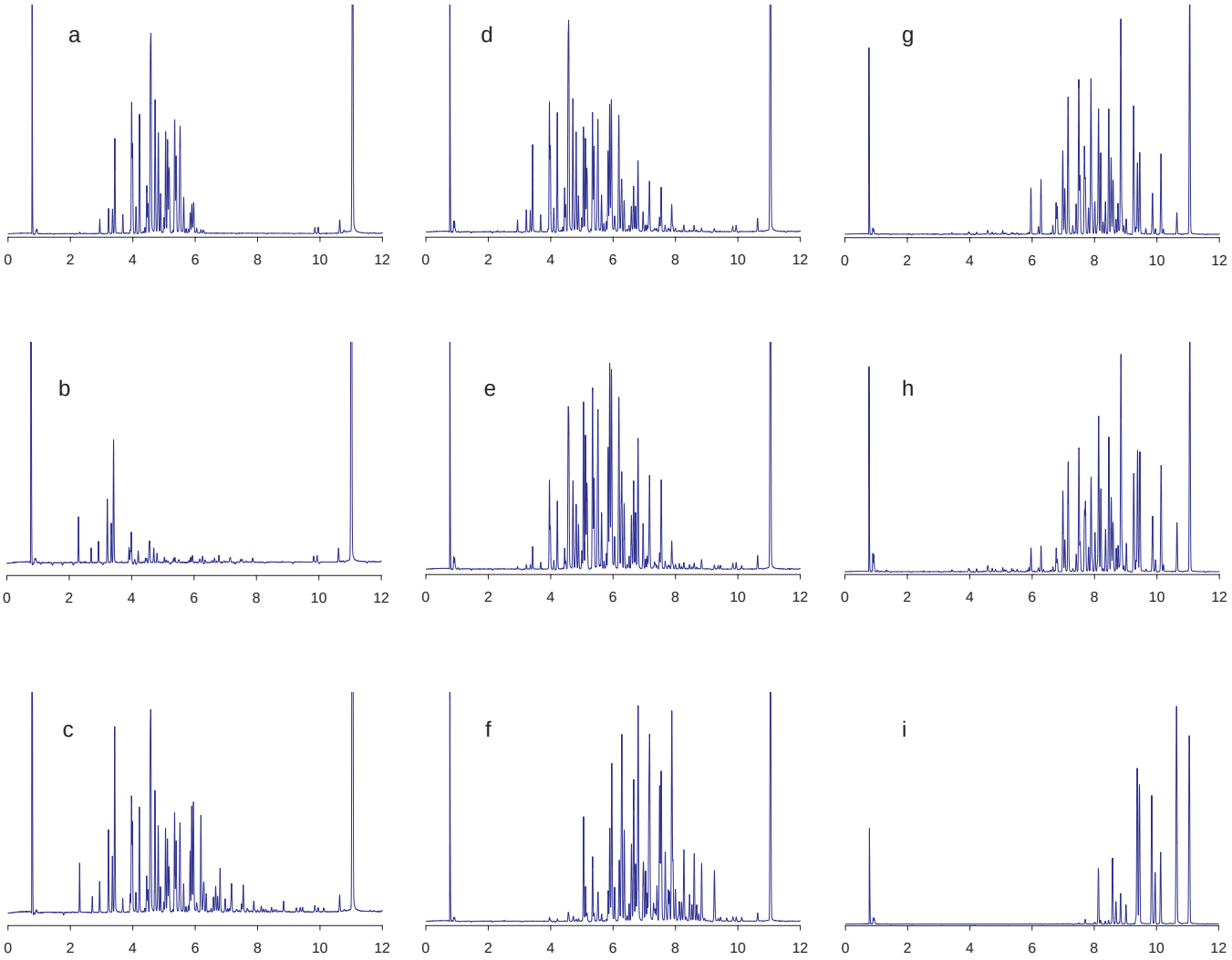

Figure 1. Single Aroclor chromatograms on ZB-5 15m x 0.32mm x 0.25µm. Identities of Aroclor mixtures are as follows: a) Aroclor 1016, b) Aroclor 1221, c) Aroclor 1232, d) Aroclor 1242, e) Aroclor 1248, f) Aroclor 1254, g) Aroclor 1260, h) Aroclor 1262, i) Aroclor 1268.

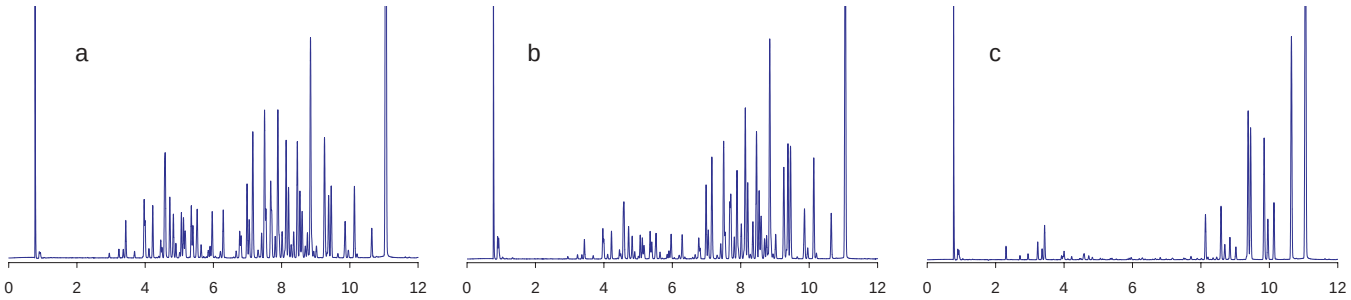

Figure 2. Mixtures of Aroclors on ZB-5 15m x 0.32mm x 0.25µm: a) Aroclors 1016 and 1260, b) Aroclors 1016 and 1262, c) Aroclors 1268 and 1221.

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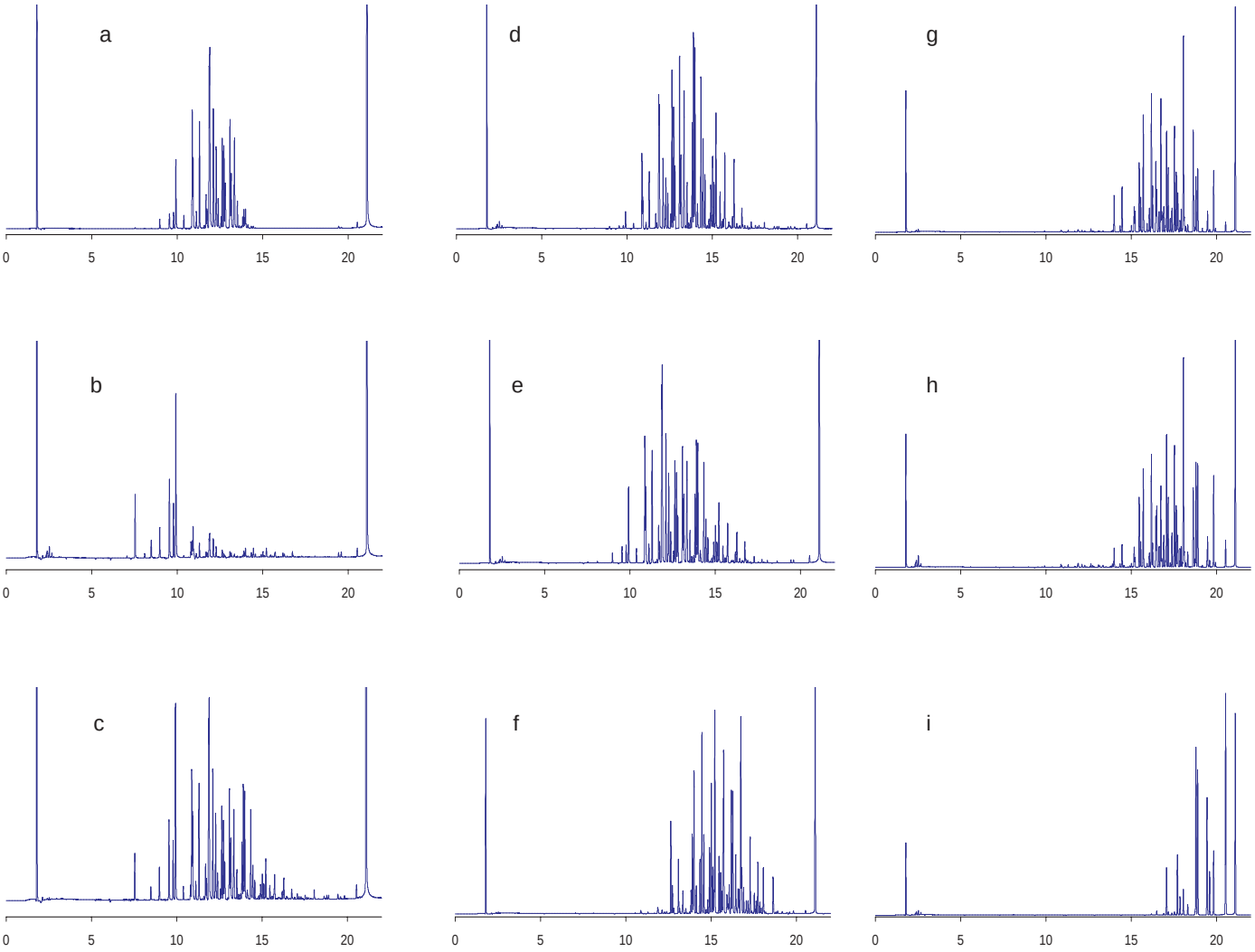

Figure 3. Single Aroclor chromatograms on ZB-5 30m x 0.32mm x 0.25µm. Identities of Aroclor mixtures are as follows: a) Aroclor 1016, b) Aroclor 1221, c) Aroclor 1232, d) Aroclor 1242, e) Aroclor 1248, f) Aroclor 1254, g) Aroclor 1260, h) Aroclor 1262, i) Aroclor 1268.

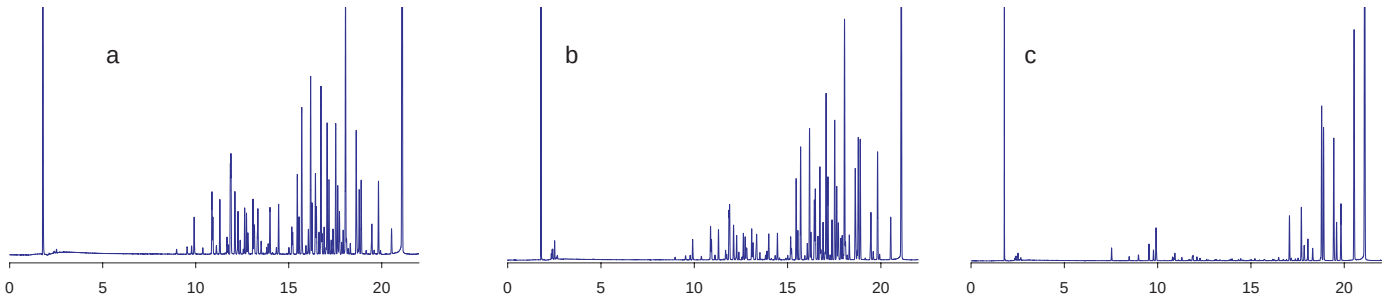

Figure 4. Aroclor Mixtures on a ZB-5 30m x 0.32mm x 0.25µm: a) Aroclors 1016 and 1260, b) Aroclors 1016 and 1262, c) Aroclors 1268 and 1221.

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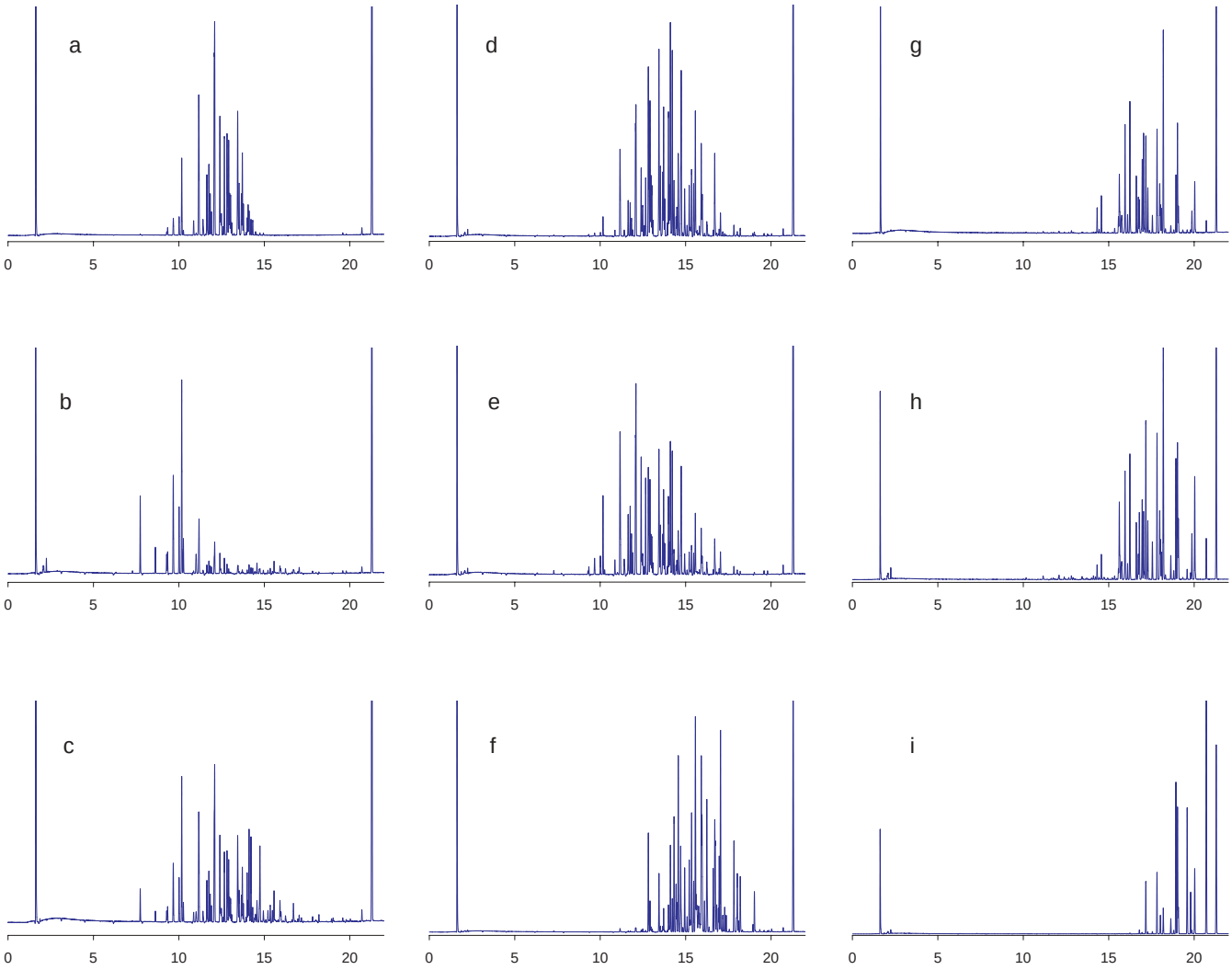

Figure 5. Single Aroclor chromatograms on ZB-35 30m x 0.32mm x 0.25µm. Identities of Aroclor mixtures are as follows: a) Aroclor 1016, b) Aroclor 1221, c) Aroclor 1232, d) Aroclor 1242, e) Aroclor 1248, f) Aroclor 1254, g) Aroclor 1260, h) Aroclor 1262, i) Aroclor 1268.

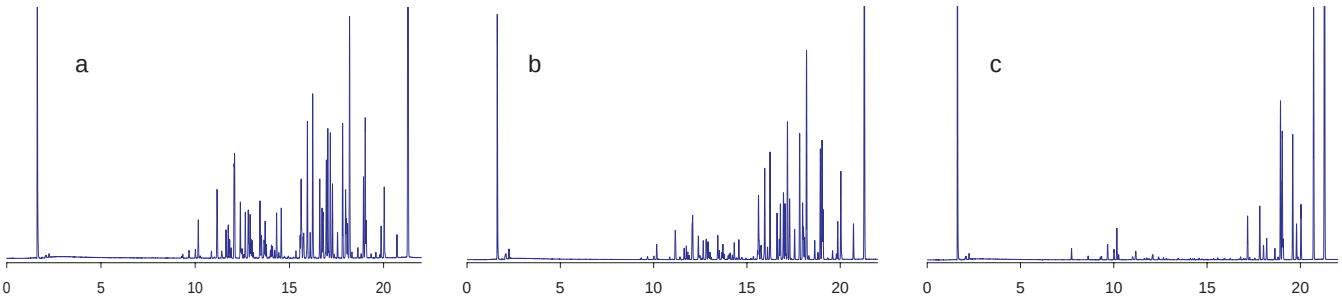

Figure 6. Aroclor Mixtures on a ZB-35 30m x 0.32mm x 0.25µm: a) Aroclors 1016 and 1260, b) Aroclors 1016 and 1262, c) Aroclors 1268 and 1221.