Influence of Storage Temperature of Florisil on Analysis of Polychlorinated Biphenyls[1,2]

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The observation that certain polychlorinated biphenyl (PCB) gas chromatographic (GC) peaks disappear or are reduced in size when PCB's are isolated from various species has been interpreted by some investigators to be indicative of metabolism of those PCB components. Recently in a PCB feeding study with trout, we found the disappearance of some PCB peaks was an artifact of the analytical procedure and could be traced to the method of storing Florisil.

Column chromatography with Florisil is a common method for separating pesticide and PCB residues from extracted lipid. Methods using Florisil for column packing are similiar with variations in the pretreatment of Florisil by heating in various ways with or without the addition of water and in the use of different eluting solvents.

The Florisil column chromatography technique of Bills and Sloan (1967) was employed with slight modification to separate PCB's from the lipid material extracted from trout. A glass column 2.2 cm x 50 cm equipped with a Teflon stopcock was tightly packed with 3 cm glass wool, followed by 11.5 cm of Florisil and finally 2.5 cm of anhydrous Na_2SO_4. Pretreatment of the packed column consisted of eluting with 50 ml methylene chloride followed by 50 ml of petroleum ether. A lipid sample of 0.5 g or less was placed on top of the column, washed into the column with three 5 ml portions of solvent, and eluted with a total of 250 ml of petroleum ether. The eluate was collected and evaporated prior to GC separation.

Florisil (60/100 mesh, Fisher Scientific Co.) was heated in unstoppered glass Erlenmeyer flasks overnight at 130° C. Different adsorption characteristics of the Florisil were noted depending on the method of storing the Florisil after the initial overnight heating. Chromatogram A is of PCB residues separated from lipid extracts on

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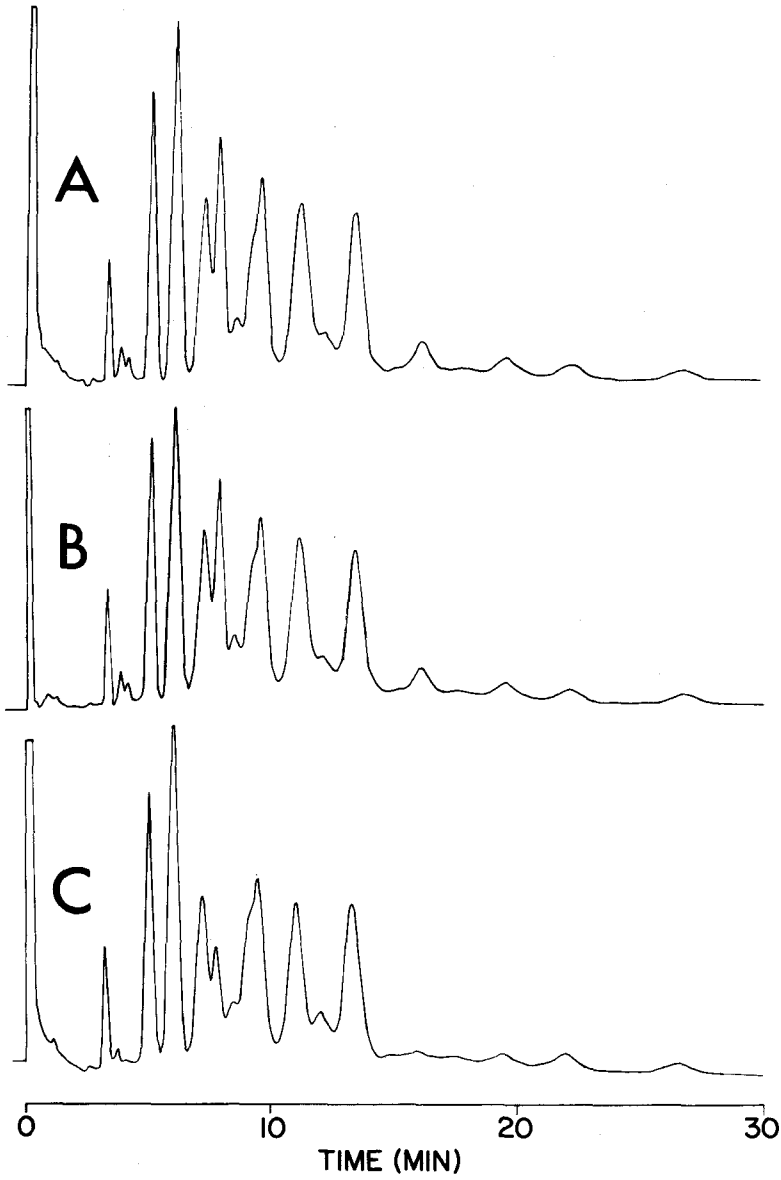

Figure 1. A. Aroclor-1254 separated from lipid on Florisil heated overnight at 130° C and stored at room temperature; B. Aroclor 1254 in hexane; C. Aroclor 1254 separated from lipid on Florisil heated overnight and stored at 130° C.

Separations obtained with a Varian Aerograph 1400 series GC equipped with an electron capture detector and a 6' x ¼" O.D. glass column packed with 2% SE-30 and 2% QF-1 on 70-80 mesh Anakrom ABS with a gas flow of 25 ml/min of nitrogen. Injection port, detector and column temperatures were 240° C, 220° C, and 180° C, respectively.

Florisil heated overnight and then stored in a glass stoppered Erlenmeyer flask at room temperature. The peak pattern is identical to that of Aroclor 1254 in hexane as shown in Figure B. Chromatogram C is of PCB residues from the same source separated on Florisil that was continually stored at 130° C in a glass stoppered Erlenmeyer flask after the initial overnight heating as recommended by Mills et al. (1972) for pesticide analysis.

Note that some peaks in chromatogram C are greatly reduced in size and others are completely absent. The reduction of size of some peaks was erroneously interpreted at first as selective absorption or metabolism of PCB components by the trout. Evidently some PCB components were selectively adsorbed by the Florisil that was stored at 130° C. This was verified by noting that a similar pattern was obtained when authentic Aroclor 1254 in hexane was chromatographed on Florisil stored at 130° C. Therefore, to avoid erroneous PCB analyses, Florisil should be stored at room temperature after the initial overnight heating.

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