PRESENT STATE OF POLYCHLORINATED BIPHENYL RESIDUE ANALYSIS

When extracts of environmental samples are examined by electron capture gas chromatography, peaks corresponding to widely disseminated chlorinated hydrocarbon (CHC) pesticides and their metabolites and break down products appear on the resultant chromatograms. In addition to these peaks pesticide researchers have been aware for some time that it was not unusual for an additional series of as many as 10 to 20 peaks, corresponding to unidentified compounds, to also appear on these chromatograms. Little is known of the structure and origin of these compounds except that they possessed electron capture gas chromatographic properties similar to the CHC pesticides.

In about 1965, it was established that these unidentified materials were organochlorine in nature, with substantial chlorine content and consequently possessed electron-capturing properties.

Early in 1967, Swedish researchers at the University of Stockholm (Jensen and Widmark) announced that they had succeeded in identifying a series of such peaks as higher chlorinated biphenyls using gas chromatographic/mass spectrometric techniques.

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Based on Jensen and Widmark's unpublished mass spectrometric identification of some similar unknown contaminants in a relatively small number of Swedish fish and bird extracts, some researchers in the United States and other parts of Europe precipitously began to report unidentified organochlorine materials as polychlorinated biphenyls with no supporting data other than relative retention times from electron capture gas chromatograms.

This would seem to indicate, in view of the well-known limitations of electron capture gas chromatographic analysis, that it would be scientifically prudent to continue the established practice of reporting these materials as organochlorine contaminants when positive structural confirmation techniques such as mass spectrometry are not employed. This is especially true when one considers that any number of technical grade chlorinated materials (toxaphene, chlordane, chlorinated naphthalenes, etc.) produce electron capture gas chromatographic peaks with retention times corresponding to those produced by commercial PCB.

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Furthermore, the response of an electron capture detector to closely related isomers can vary considerably. Theoretically, there are 210 possible PCB isomers. So it should be clearly stated, at this point in time, that PCB residue analysis using only electron capture gas chromatography is neither positive nor quantitative.

In attempts to make the less sophisticated and more readily accessible technique of electron capture gas chromatography more selective (positive), several laboratories have introduced prior chemical treatments (saponification, extraction with sulfuric acid, nitration, etc.). Unfortunately the use of the nitration procedure has caused considerable confusion. This treatment is essentially that used to remove DDT and its metabolites from toxaphene.

In our laboratories we have found that this treatment results in the disappearance of the early eluding possible PCB isomer peaks in the production of peaks with longer retention times (nitrochlorobiphenyls). Additionally, the treatment is not easily reproduced nor specific for PCB.

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Is is apparent that our primary concern at this time should be to develop more specific and systematic analytical procedures which will allow us to accumulate enough unequivocal data to permit the ecologist to conscientiously assess the situation.

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