CHROM. 6123

A simple differentiation of polychlorobiphenyls from chlorinated naphthalenes

In a recent paper[1], ARMOUR AND BURKE have drawn attention to the fact that many of the uses and properties of chlorinated naphthalenes are similar to those of the polychlorobiphenyls (PCB). They have also shown that the chlorinated naphthalenes appear in exactly the same fraction as the PCB isomers, when a column of silicic acid and Celite, on which they have been adsorbed, is eluted with petroleum ether[2].

An identical fractionation occurs when the chlorinated naphthalenes are adsorbed on a column of silica gel (containing 2.5% moisture) and the column then eluted with *n*-hexane; a technique which is used in this laboratory to separate PCB from organochlorine pesticide residues[3,4]. In addition, the behaviour of the chlorinated naphthalenes on thin-layer chromatography[4,5] is closely similar to that of the PCB isomers.

The usual procedure for the determination of PCB is electron capture gas-liquid chromatography (GLC), following extraction and columnar clean-up[4]. The nature and identity of the PCB is inferred from the multiple peak pattern given by the chromatogram. However, the chlorinated naphthalenes give similar peak patterns. Therefore, since the two groups of compounds cannot be easily separated from each other by present procedures, it is possible that there could be interference from the presence of the chlorinated naphthalenes in the estimation of the PCB content of some samples, leading to an incorrect interpretation of the nature of the compounds present with an overestimation of the PCB content. A simple method to differentiate between the PCB and the chlorinated naphthalenes would be obviously an advantage.

Following an investigation with hexane solutions of Aroclor 1254 (a commercial PCB preparation) and the Halowaxes 1005, 1013 and 2148 (commercial chlorinated naphthalenes) at concentrations of 10 μg/ml, the following method has been found to remove any interference caused by the chlorinated naphthalenes. It is based on the relative ease of oxidation of the latter compounds as opposed to the resistance to oxidation of the PCB isomers, and has been used previously to differentiate *p,p'*-DDE in the presence of PCB[4].

Experimental

Apparatus. A Kuderna–Danish type evaporator with interchangeable 10-ml graduated collection tubes; two-bubble Snyder micro-columns[6], with groundglass cones to fit the Kuderna–Danish collection tubes.

Reagents. Acetic acid, glacial, redistilled; *n*-hexane, redistilled from potassium hydroxide; sodium hydroxide, a 5 *N* solution in water; chromium trioxide, general purpose reagent.

Adjust the volume of the PCB fraction, suspected of containing chlorinated naphthalenes, to 2 ml, using a Kuderna–Danish evaporator. Remove the collection tube and add 2 ml of glacial acetic acid. Fit a Snyder column to the tube and heat it carefully in a steam bath until all the original petroleum ether or *n*-hexane has evaporated, as judged by the reduction in volume. Introduce 100 mg of chromium trioxide and place the tube in boiling water for 20 min. Cool the mixture and shake it vigorously with 2.0 ml of *n*-hexane in the stoppered tube. Neutralise the acid with approximately 7 ml of 5 *N* sodium hydroxide solution. Shake the tube again and allow the two phases to separate. Examine an aliquot portion of the upper hexane layer by GLC, using electron capture detection[4]. The peaks which appear on the GLC chart are those due to the PCB isomers only; these compounds are unaffected by the oxidation, whereas the chlorinated naphthalenes are oxidised completely and do not show any GLC peaks when similarly treated.

It must be stressed, however, that the method is designed only to eliminate the possibility of interference by any chlorinated naphthalenes which may be present in the sample fraction. It must not be considered as a positive means of identification of either of the two groups of compounds involved.

We would like to thank the Government Chemist for permission to publish this note.

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Received April 27th, 1972