COMMENTS ON PCB METHODS

Method I. Gas Chromatographic Determination of Higher Boiling Homologues in Aroclor 1016 and Aroclor 1242.

1. There should be a clearer definition of how the chromatogram is divided and over what range the elution is recorded.

   Recommendations:

   a) Describe the division of the chromatogram in terms of a retention index (ASTM's preferred retention parameter E 355-68, p. 1128); e.g., all components eluting with a retention index greater than 2400 will be calculated as a "High Boiler."

   b) In a similar manner the range over which compounds are measured could be described using a retention index window: e.g., all compounds eluting with retention index 100 $<I_x<2400$ are "Low Boilers," those with $2400 <I_x <3600$ are "High Boilers."

2. Although pure known materials are generally not available, Dr. Ronald G. Webb, Southeast Environmental Research Laboratory, National Environmental Research Center - Corvallis, Environmental Protection Agency, Athens, Georgia 30601 can provide samples of materials whose compositions have been determined by a combination of elemental analysis, gas chromatography with a Coulson conductivity detector and combined gas chromatography - mass spectrometry. Using these as standards, response factors can readily be determined.

3. The method should provide information on sensitivity and minimum detectable amounts. Refer to ASTM E260-69, p. 748.

4. For precision, five rather than two measurements should be made.

Method II. Polychlorinated Biphenyls in Petrochemicals.

1. In Note 1, there appears to be some confusion of the terms "sensitivity" and "minimum detectable limit."

2. Method Summary:

   a) Is documentation available to show that compounds elute in boiling point order? It is generally accepted that the free energy of solution determines elution order in gas-liquid partition chromatography, rather than the boiling points.

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b) Many hydrocarbons with functional groups show strong electron capture response.

3. It seems that 100 µl is an awfully large sample to inject on to a 3.5% loaded column. That much material would probably kill the response of a Ni^{63} electron capture cell.

4. Retention parameters should probably be expressed in terms of retention indices. Refer to ASTM E 355-68.

5. Provide some information on precision, accuracy, and sensitivity measurements as per ASTM E 260-69.

6. We have found that PCBs are adsorbed readily on untreated glass surfaces. Serial dilutions should not be made in glass containers unless the glass is properly pre-treated.

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