



AMERICAN SOCIETY FOR TESTING AND MATERIALS

1916 Race St., Philadelphia, Pa., 19103

Reprinted from the Annual Book of ASTM Standards, Copyright ASTM

If not listed in the current combined index, will appear in the next edition

Standard Method for RAPID GAS CHROMATOGRAPHIC ESTIMATION OF HIGHER BOILING HOMOLOGUES OF CHLORINATED BIPHENYLS FOR CAPACITOR ASKARELS¹

This Standard is issued under the fixed designation D 3303; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers a rapid empirical determination of the level of higher boiling homologues in Specification D 2233, Types D and A. Higher boiling homologues are defined in 3.2. This procedure is not intended for the precise analysis of polychlorinated biphenyl products or mixtures.

2. Applicable Documents

2.1 ASTM Standards:

D 2233 Specification for Chlorinated Aromatic Hydrocarbons (Askarels) for Transformers²

E 260 Recommended Practice for General Gas Chromatography Procedures³

E 355 Recommended Practice for Gas Chromatography Terms and Relationships³

3. Summary of Method

3.1 The sample is injected into a support-coated open tubular gas chromatography column. The components are separated as they pass through the column with helium carrier gas and their presence in the effluent is measured by a flame ionization detector and recorded on a chromatogram. The method is made quantitative by internal normalization comparing the area of the higher boiling homologue peaks to the total area of all the peaks in the chromatogram.

NOTE 1—Reference should be made to Recommended Practices E 260 and E 355.

3.2 This method arbitrarily splits up the

chromatogram into two regions. The major region represents mostly mono, di-, tri-, and tetra-chlorobiphenyls. The lesser region represents the higher boiling homologues and is mostly penta- and hexa-chlorobiphenyls. However, there is some overlap of the tetra- and penta-chlorobiphenyls between the two regions. Some of the important peaks in the major region contain both tetra- and penta-chlorobiphenyls.

3.3 The only way to obtain a closer approximation to true composition of these samples is to use a longer column with a gas chromatography-mass spectrometry (GS/MS) combination to correct for the composition of these overlap peaks. Area correction factors must also be used. However, GC/MS instrumentation is not available in many laboratories and is too cumbersome and costly for routine analysis. Therefore, this empirical short method has been adopted. This method yields values that are about 40 % of the true value when the true value is in the range of 1 %.

4. Significance

4.1 Present knowledge indicates that higher boiling homologues of chlorinated biphenyls are less degradable than the lower

¹ This method is under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gases.

Current edition approved Feb. 27, 1974. Published April 1974.

² 1974 Annual Book of ASTM Standards, Part 40.

³ 1973 Annual Book of ASTM Standards, Part 30.