

Quantitative analysis of bisphenol A by direct gas chromatography*

Polycarbonate is one of several polymers which eliminate bisphenol A (bis(4-hydroxyphenyl)-2,2-propane), on degradation^{1,2}. Estimation of bisphenol A by gas chromatography can be accomplished by acetylation of the hydroxy groups³. During a study of the thermal degradation of polycarbonate we have now shown that bisphenol A itself can be detected and quantitatively analysed by gas chromatography.

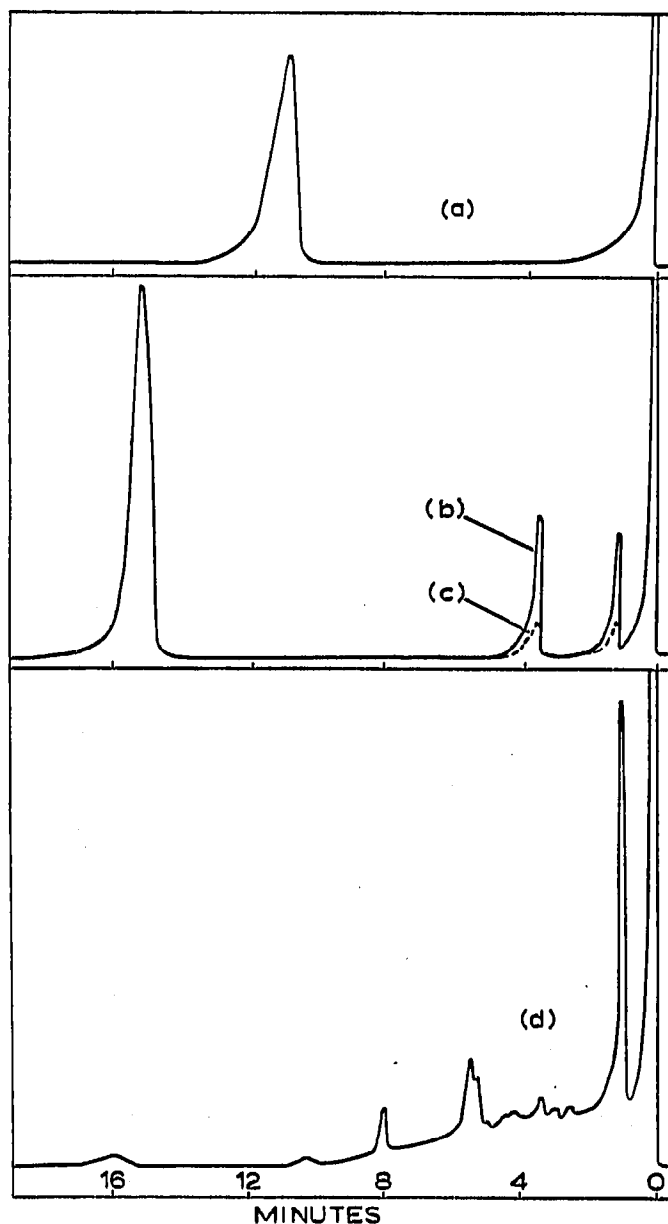


Fig. 1. (a) Isothermal chromatogram of bisphenol A at 250°. (b) Programmed chromatogram (150–250° at 24°/min) of bisphenol A after 30 min heating in the presence of nichrome wire. (c) Duplicate of (b) without nichrome wire. (d) Programmed chromatogram (150–250° at 24°/min) of bisphenol A after heating for 5 min at 250° in vacuum.

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An 801 Perkin-Elmer Gas Chromatograph was used. This is a dual-column temperature-programmed instrument with glass inlet and glass columns and a flame ionisation detector. The column packing was 20% Apiezon K on Kieselguhr ("Embacel") and the flow rate of the carrier gas, nitrogen, was 38.5 c.c./min. The inlet and detector were maintained at 250°.

Under these conditions an isothermal chromatogram of an acetone solution of bisphenol A at 250° (Fig. 1a) shows a retention time for bisphenol of 10.8 min.

One possible reason for the failure of previous attempts at direct gas chromatography of bisphenol A is the use of a metal injector and/or metal columns. Bisphenol A when heated in the presence and absence of nichrome wire for 30 min at 250° is partially degraded (Fig. 1b and c, respectively). The formation of phenol and isopropylphenol under these conditions is accelerated by metal. Another possible reason for previous lack of success is acid or base catalysis of the degradation of bisphenol A⁴. Thus, incorporation of phosphoric acid into the liquid phase, which has been done when analysing for bisphenol A³, could also accelerate the degradation. Fig. 1d shows that when bisphenol A is heated with a trace of phosphoric acid in vacuum at 250° for 5 min it is completely destroyed.

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Separation of mercapturic acids and related compounds by gas chromatography and a method for the determination of hippuric acid

Mercapturic acids (N-acetyl S-substituted cysteines) are of interest because they are formed *in vivo* as metabolites of certain foreign organic compounds. Many halogeno-alkanes are precursors, in rabbits and rats, of alkyl mercapturic acids which are excreted in the urine^{1,2}. In addition to mercapturic acids some halogeno-alkanes form further sulphur-containing metabolites. BARNSLEY³, for example, found that bromopropane formed N-acetyl-S-2-hydroxypropylcysteine and JAMES AND JEFFERY⁴ thought a similar compound from bromobutane was a hydroxy derivative of butylmercapturic acid. As the isolation of these compounds and their identification by paper chromatography was difficult the possibility of separating mercapturic acids and their derivatives by vapour phase chromatography has been investigated. The behaviour of hippuric acid, a normal urinary constituent, has also been examined on the gas chromatogram and a method worked out for its determination.

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