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Separation and identification of methylol derivatives of cardanol

Various investigators have used different methods and techniques to separate and identify the reaction products of various phenols with aldehydes under alkaline conditions¹. With a substituted phenol, such as cardanol (the chief component of commercial cashewnut shell liquid), the identification of the intermediate products is even more difficult, and available methods do not enable easy separation for identification of these products.

Preliminary results indicate that it is possible to separate the intermediate products formed in the cardanol-formaldehyde reaction under alkaline conditions by using the technique of thin-layer chromatography².

Materials and method

(1) Cardanol (1 mol.) was treated with formaldehyde (1 mol.) in presence of 40% KOH solution (1 mol.) and the reaction mixture was kept at room temperature (25°) in a stoppered bottle. After a period of 24 hours, 1 g of the reaction mixture was removed from the bottle and diluted ten times with ethanol. The reaction mixture was kept for 11 days and at intervals of 24 hours, samples were taken out as indicated above.

(2) Finely divided silica gel (200 mesh) was mixed with twice its weight of water and a little gypsum. The paste was then uniformly spread on glass plates, 20 cm in length, and dried in an oven for 2 hours at 100°.

(3) The samples collected at intervals of 24 hours were taken for spotting. After spotting these solutions on the glass plates, the chromatogram was run for 55 minutes in ethyl acetate-toluene (60:40) mixture. Four spots were obtained from the samples taken on the fifth day and there was no change up to the eleventh day. The chromatograms were developed with diazotised sulphanic acid.

Three methylol derivatives with R_F values of 0.738, 0.586 and 0.448 respectively were detected. The fourth spot was identified and confirmed to be that of unreacted cardanol.

One of the derivatives was crystalline and melted sharply at 60°. It had an R_F value of 0.738. This derivative was separated and identified after hydrogenating the

alkyl side chain. The resulting product melted at 91° , which corresponds to that of the product isolated by JONES AND ROBSON³ and designated as the 6-methylol of tetrahydro-anacardol. The crystalline methylol derivative obtained would therefore be 6-methylol-cardanol.

Thus, thin-layer chromatography shows signs of usefulness in these investigations. Further work on this subject is in progress.

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BOOK REVIEWS

Progress in Industrial Gas Chromatography, edited by H. A. SZYMANSKI, Plenum Press, Inc., New York, 1961, price \$ 10.00.

The title of this volume is very misleading, because this book does not deal with the developments and applications of gas chromatography for industrial purposes, but is a collection of the proceedings of the advanced sessions of the annual Gas Chromatography Institute held at Canisius College, Buffalo, N.Y., in 1961, where some aspects of gas chromatography have been discussed from a practical point of view.

The volume consists of twelve reports and a summary of the panel discussion dealing with instrumentation and operating technics of gas chromatography. Most of the contributions are not original, but are reviews on topics that are already available in specialized books. The majority of the authors, who are connected with firms manufacturing gas-chromatography apparatus, treat the subjects with competence and clarity. Some contributions are, however, only short recommendations on certain subjects. For instance, one paper on the use of chromatography for analysis of odors, flavors and air pollution consists of only four pages and does not contain a single reference. Since this book, according to the advertising flap, should keep "a scientist working in this field abreast of the latest developments", it seems that, at least in this field, the aim of the editors has not been reached.

The reviewer is very doubtful about the aims of publishers who issue volumes of the proceedings of a meeting as progress in a certain field. Unless these meetings have been well organized and the papers carefully selected overlapping of subjects usually occurs and the scientific and technical level of the various papers will not be the same; these remarks certainly apply to this volume.

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