CHROM. 3615

Gas chromatography of long-chain 1,2-diols from the biological oxidation of 1-alkenes

Growth of the yeast, *Candida lipolytica*, on long-chain 1-alkenes leads to an accumulation of oxidation products in the culture medium which correspond in carbon number to that of the 1-alkene substrate on which the organism is grown. Thin-layer chromatographic analyses of ether extracts of culture fluid indicated the presence of several classes of compounds corresponding in polarity to long-chain primary and secondary alcohols, monocarboxylic acids, 2-hydroxy acids, 1,2-diols and two relatively non-polar classes.

These various classes of compounds were separated by preparative thin-layer chromatography on silica gel and were further analyzed by gas–liquid chromatography. All classes proved amenable to analysis by this method with the exception

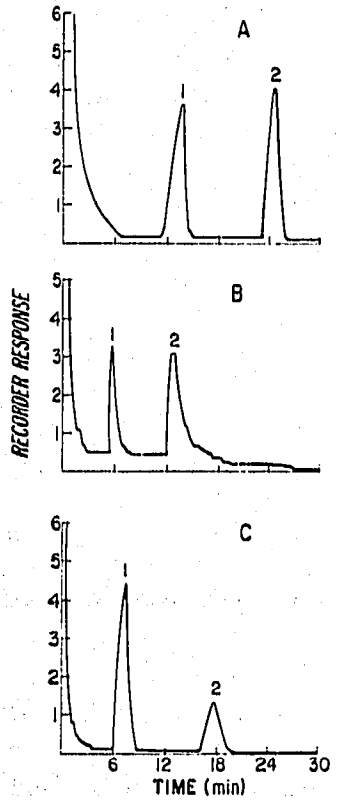

Fig. 1. Chromatograms of (1) 1,2-tetradecanediol and (2) 1,2-hexadecanediol on several columns.

of the class corresponding to 1,2-diols. WEATHERALL[1] reported that short-chain diols could be analyzed on silanized celite coated with LAC-2R-446. In our hands, it was found that long-chain 1,2-diols could not be adequately analyzed by this method. DANIELS[2] separated long-chain α,ω-diols and their derivatives by GLC but no reference was made to analysis of 1,2-diols.

After testing various liquid phases and solid supports we obtained three columns and conditions which adequately analyzed long-chain 1,2-diols. Fig. 1 shows chro-

matographic separation of the 14 and 16 carbon 1,2-diol standards synthesized by the method of SWERN, BILLEN AND SCANLON[3]. (A) represents a scan obtained using an 1/8 in. × 6 ft. copper column packed with 7 % Zonyl E-7 coated on Chromosorb W H.P., 80–100 mesh (Varian-Aerograph, Walnut Creek, Calif.). Scan (B) was from a copper column, 1/8 in. × 3 ft. packed with 2 % Versamid 900 on Chromosorb AW-DMCS, 80–100 mesh (Hewlett-Packard-F&M, Avondale, Pa.). Chromatogram (C) was from an 1/8 in. × 3 ft. copper column packed with 2 % OV-17 on Anakrom Q, 60–70 mesh (Analabs, Hamden, Conn.). Analyses were obtained using a HyFi 600 gas chromatograph (Varian-Aerograph, Walnut Creek, Calif.) equipped with a hydrogen-flame ionization detector with helium as the carrier gas at a flow rate of 30 ml/min in all cases. Column temperature conditions were: (A) 195°; (B) 195°; (C) 160°.

Use of these columns allowed us to identify 1,2-diols of 14 through 18 carbon atoms obtained from the biological oxidation of 1-alkenes of 14 through 18 carbons by *C. lipolytica*.

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The identification of glycerol, ribitol and anhydroribitol by gas–liquid and thin-layer chromatography

The difficulty of identifying the ribitol and glycerol components of cell wall hydrolysates has been outlined by IKAWA, MORROW AND HARVEY[1]. We have now found that the application of accepted gas chromatographic methods, using trimethylsilyl derivatives[2], to this problem yields excellent separations, as also does the use of the same derivatives in thin–layer chromatography. Similar TLC techniques in the analysis of other sugars have been reported by LEHRFELD[3].

Methods and results

A mixture of ribitol and anhydroribitol was prepared by hydrolysing 5 mg of ribitol (Lights) with 2 ml of 2 *N* HCl in a sealed tube at 110° for 3 h. The products were dried by rotary evaporation followed by the addition and evaporation of chloroform (B.D.H. redistilled and stabilised by the addition of 1 % absolute ethyl alcohol).