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Note

Capillary gas chromatographic separation of monosaccharides as their alditol acetates

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During organic geochemical research dealing with the occurrence and composition of polysaccharides in recent marine sediments, a gas–liquid chromatographic (GLC) separation is required by which mixtures of monosaccharide derivatives can be baseline separated.

To avoid complex mixtures of anomeric monosaccharides, the hydrolytically released monosaccharides can be reduced to the corresponding alditols. The alditol mixture is subsequently derivatized into the alditol acetates. The GLC separation of alditol acetates on columns packed with OV-275[1] and ECNSS-M[2–5] has been reported. In the latter case the column is usually operated under conditions close to the maximum operating temperature, which limits column life[6]. Moreover, a baseline separation of some of the alditols used in these studies is not achieved. Holzer *et al.*[7] applied a glass capillary column coated with a chiral phase. Their results show that the separation of rhamnitol/fucitol and ribitol/arabitol is not compiete. This report describes the baseline separation of ten alditol acetates using a glass capillary column coated with OV-275.

EXPERIMENTAL

The ten alditols used as standards in this study are listed in Table I. They are commercially available from various companies. The standard mixture of the alditol acetates was prepared by acetylation of a mixture of the alditols, containing equal amounts (by weight) of the individual alditols. The acetylation was performed in a

TABLE I

THE ALDITOLS USED AS STANDARDS IN THIS STUDY

Alditol	*No.*	*Alditol*	*No.*
Erythritol	1	Xylitol	6
Rhamnitol	2	Mannitol	7
Fucitol	3	Galactitol	8
Ribitol	4	Sorbitol	9
Arabitol	5	*m*-Inositol	10

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sealed vial with pyridine–acetic anhydride (1:1) at 100°C during 2 h. After evaporation of the acetylation reagent the alditol acetate mixture was dissolved in dichloromethane.

The natural mixture of monosaccharides was obtained from a diatomaceous ooze sample from the Namibian Shelf (S.W. Africa, 22°51.5′ S, 14°14.5′ E)[8]. The sample was lyophilized and hydrolysed with 1 *M* H_2SO_4 during 3 h at 100°C. The hydrolysate was neutralized with $BaCO_3$ and reduced with $NaBH_4$. Subsequent acetylation was performed as described above.

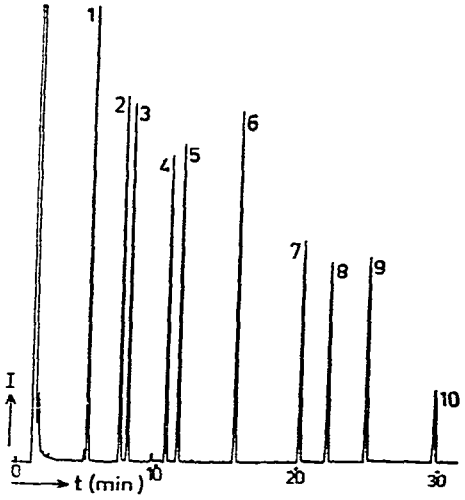
GLC was carried out on a Varian 3700 gas chromatograph equipped with a glass capillary column (25 m × 0.25 mm I.D.) coated with OV-275 (Chrompack, Middelburg, The Netherlands). The temperature was programmed from 190 to 215°C at 1°C/min. Further GLC conditions: injector, 250°C; flame ionization detector, 250°C; carrier gas, helium at a flow-rate *ca.* 1.5 ml/min; helium pressure, 18 p.s.i.; splitter, 30 ml/min; attenuator, $1 \cdot 10^{-11}$ mA.

Identification of the acetates was based on the retention times of the individual alditol acetates.

RESULTS AND DISCUSSION

Fig. 1 shows the gas chromatogram of the standard alditol acetate mixture. The peak numbers correspond to the alditols listed in Table I. All components are baseline separated, thus allowing a complete qualitative and quantitative analysis of monosaccharides as their alditol acetates.

Fig. 2 shows the gas chromatogram of the mixture obtained from the diatomaceous ooze sediment after hydrolysis, reduction and derivatization. *m*-Inositol was added as an internal standard. The relative retention times of the main peaks corre-



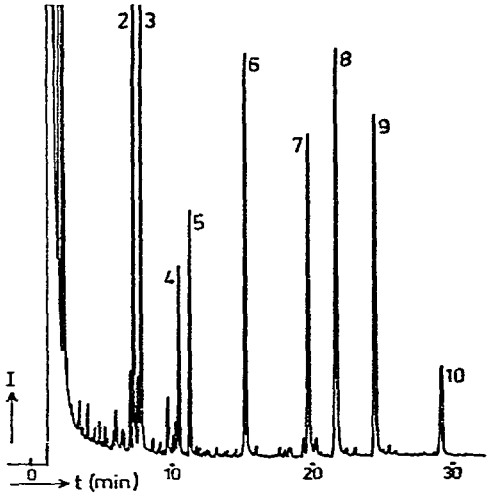


Fig. 1. Gas chromatogram of a standard mixture of ten alditol acetates. The peak numbers correspond to the alditols listed in Table I.

Fig. 2. Gas chromatogram of the alditol acetates obtained from a recent sediment. *m*-Inositol (10) was added as an internal standard. The peak numbers correspond to the alditols listed in Table I.

spond exactly to those of the alditol acetates in Fig. 1. Ultimate identification of both major and minor peaks has to be achieved by GLC–mass spectrometry.

The abundance of rhamnose and fucose in the ooze sample is not unexpected since these monosaccharides are major building blocks of algal polysaccharides[9–12].

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