THE GAS-CHROMATOGRAPHIC DETERMINATION OF SUGARS AND GLUCURONIC ACID

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Continuing a study of the gas-chromatographic behavior of the sugars encountered in steroid and triterpene glycosides [1] we have analyzed a mixture of six sugars (D-glucose, D-galactose, D-xylose, D-fucose, L-rhamnose, and L-arabinose) and D-glucuronic acid. To perform such an analysis it has been proposed [2] to determine the acid and the sugars separately. We have found a possibility for the direct qualitative and quantitative analysis of such a mixture. The compounds were chromatographed in the form of silylated methyl glycosides [1, 2].

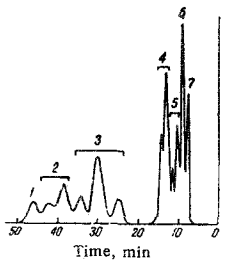
After methanolysis and silylation, glucuronic acid gives several peaks on the chromatogram, some of which are superposed on the peaks of galactose and glucose, but the main peak, relating to the α-D-pyranoside of glucuronic acid [3] is fairly well separated from the peaks of the other sugars, so that it is possible to determine glucuronic acid and the sugars mentioned in natural compounds qualitatively and also, with correction factors, quantitatively.

The correction factors were determined on synthetic mixtures of sugars and glucuronic acid [4]. For the quantitative evaluation of the chromatogram we used the method described by Caroll [5].

The relative retention times of the silylated derivatives of the methyl glycosides of the sugars recalculated, as in the preceding communication [1], in relation to silylated methyl β-D-glucopyranoside [6] are given below:

Sugar	Relative retention time			
L-Arabinose	0.19	0.20	0.22	—
L-Rhamnose	0.22	—	—	
D-Fucose	0.23	0.26	0.29	—
D-Xylose	0.32	0.35	—	
D-Galactose	0.60	0.70	0.81	—
D-Glucose	0.92	1.00	—	
D-Glucuronic acid	0.62	0.71	1.06	1.14

The figure gives a chromatogram of the silylated methyl glycosides of glucuronic acid and the six sugars. Chromatography was carried out on a UKh-1 chromatograph in a copper column (1 m × 4 mm) filled with 5% of silicone phase g=30M on Diaphorite (0.2-0.315 mm) at a column temperature of 170° C with hydrogen as the carrier gas (55 ml/min).



Chromatogram of the TMS ethers of the methyl glycosides of: 1) D-glucuronic acid; 2) D-glucose; 3) D-galactose; 4) D-xylose; 5) D-fucose; 6) L-rhamnose; and 7) L-arabinose.

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