

The proposed tetrahydroborate-2-TBA procedure is applicable to the analysis of various bacterial LPSs. The standard deviation of the results of the determinations for ODO and for 3,6-DDHs amounts to 4%. The results of an analysis of a hydrolyzate of the LPSs obtained by the tetrahydroborate-2-TBA procedure and by the preparative separation of the ODO and 3,6-DDHs on a column of Dowex 1 (CO_3^{2-} form) [2] agree within the limits of the standard deviation shown.

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A NEW METHOD FOR OBTAINING METHYL β -L-RHAMNOPYRANOSIDE

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UDC 542.91+547.455

The method for obtaining methyl β -L-rhamnopyranoside described in the literature [1], which is based on the Koenigs-Knorr reaction, is a multistage process and is characterized by a low yield of the end-product (of the order of 1%). We have proposed a method which consists in obtaining methyl β -L-rhamnopyranoside as a by-product in the synthesis of methyl α -L-rhamnopyranoside by the Fischer method [2].

Analysis by GLC and by chromatomass spectrometry showed that after a solution of 98 g of α -L-rhamnose in 1 liter of methanol containing 1% of HCl had been left for 40 h, the reaction product contained 89% of methyl α -L-rhamnopyranoside and 11% of methyl β -L-rhamnopyranoside. After the methyl α -L-rhamnopyranoside (68.5 g) had been crystallized out by the usual method, the remaining mother syrup (21.6 g) contained 38% of methyl β -L-rhamnopyranoside. This product was acetylated in pyridine with acetic anhydride, and the resulting acetate (36.0 g) was crystallized from ethanol. After two crystallizations, the acetate of methyl β -L-rhamnopyranoside (6.1 g) was chromatographically pure and contained no methyl α -L-rhamnopyranoside. It melted at 154-155°C $[\alpha]_D^{20} + 44.3^\circ$ (c 2.5; chloroform). Literature information [1]: mp 151-152°C, $[\alpha]_D^{18} + 45.73^\circ$, $\text{C}_6\text{H}_8\text{O}_4$ (OCH₃) (COCH₃)₃.

The acetate of methyl β -L-rhamnopyranoside (6.0 g) was deacetylated with sodium methoxide in methanol. After recrystallization from ethanol, the yield was 2.8 g (3.1% on the initial rhamnose), mp 140-141°C, $[\alpha]_D^{20} + 91.9^\circ$. Literature information [1]: mp 138-140°C, $[\alpha]_D^{20} + 95.39^\circ$, $\text{C}_6\text{H}_{11}\text{O}_4$ (OCH₃).

For analytical GLC we used a Tsvet-106 instrument fitted with a flame-ionization detector and two columns (100 × 0.4 cm). As the stationary phase we used 10% of BDS on Chromosorb W (60-80 mesh). The temperature of the thermostat was 190°C, the rate of flow of argon 60 ml/min, and the retention time for the acetate of methyl α -rhamnoside 6.4 min and for the acetate of the β -rhamnoside 9.4 min.

The melting points were measured on a Boetius instrument and the specific rotations on a Perkin-Elmer 141 instrument.

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Pacific Ocean Institute of Bioorganic Chemistry, Far Eastern Scientific Center of the Academy of Sciences of the USSR, Vladivostok. Translated from *Khimiya Prirodnikh Soedinenii*, No. 1, pp. 87-88, January-February, 1976. Original article submitted July 9, 1975.

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