

EXPERIMENTAL

Dehydration of carbohydrate solutions was studied using the method of IR-Fourier's spectrometry in the regime of impaired total internal reflection (ITIR), and the products formed in this case were investigated using the method of thermal analysis. The IR spectra were recorded on a FT-IR Nicolet 6700 spectrometer from Thermo Electron Corporation at room temperature, with a resolution of 4 cm^{-1} , an accuracy of $\pm 0.5\text{ cm}^{-1}$, and a scan number of 32.

An ITIR prefix with a ZnSe single crystal (45°) was used, which made it possible to record the spectra at a 12-fold reflection of an infrared laser beam with a beam penetration depth of about $2\text{ }\mu\text{m}$ into the sample. The solution was applied in a thin layer on the surface of the single crystal, so that the contact area of the liquid with the solid surface was maximal. As the solution dried out, the spectra were recorded at intervals of about 0.5 hours.

Thermal analysis was performed on a combined thermal analysis instrument STA-449 F1 Jupiter from Netzsch. Samples weighing 3 - 4 mg. in the form of pieces of film formed during the drying of aqueous solutions in air at room temperature were heated in open alumina crucibles with a volume of $85\text{ }\mu\text{l}$ at a speed of 5 degrees/min in a dry nitrogen atmosphere.

There were used commercial carbohydrates supplied by Sigma-Aldrich: maltotriose No. M8378, lot 017K0679; maltose No.M5885; maltotetraoz No.M8253, lot 109K1271; maltopentaose No.M8128, Lot 040M1774; maltohexaose No.M9153; maltoheptaose No.M7753, Lot 079K0987. Solutions of saccharides in distilled water with a concentration of about 10 % were prepared at room temperature.

RESULTS AND DISCUSSION

The infrared spectrum of a solution of maltotriose differs very little from the same spectrum of water (Fig. 1). Only in the low-frequency region, where the saccharides show the strongest spectral lines related to the stretching (valence) vibrations of the chemical bonds C-O and C-C [14 - 16], weak absorption is observed. In the high-frequency region of the spectrum, where the stretching vibrations of OH bonds in $\nu(\text{OH})$ water molecules are recorded, there are also slight differences, manifested in the mismatch of the line profile for the solution and for pure water, as well as $17 - 20\text{ cm}^{-1}$ wider at half-height at the strip of solution. In addition, the peak of deformation oscillations $\delta(\text{OH})$ in OH bonds in water

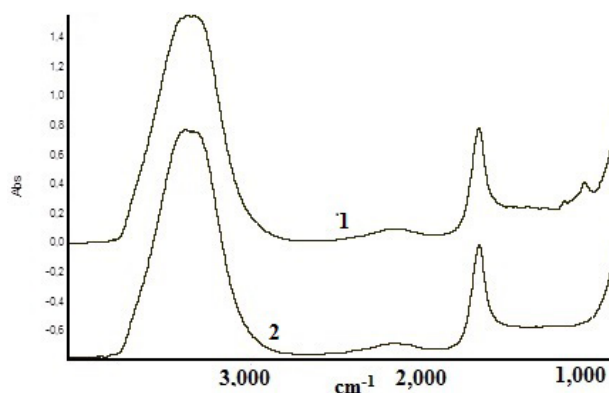


Fig. 1. IR-Fourier spectra of an aqueous solution of maltotriose (1) and water (2).

molecules (range $1,560 - 1,800\text{ cm}^{-1}$) for the solution is slightly shifted to the high-frequency region. These differences, although not significant, are quite distinct and reproducible, and they indicate the effect of saccharide on water at the molecular level, as already reported, for example, in [17, 18].

As the maltotriose solution dries out due to an increase in its concentration, the spectral lines related to the stretching vibrations of the C-O and C-C chemical bonds become more pronounced, and the water bands, on the contrary, gradually weaken. Therefore, the ratio of their intensities can serve as a measure of the depth of dehydration for a saccharide solution.

From Fig. 2, it can be seen that the relation $I_{\nu(\text{CO+CC})}/I_{\nu(\text{OH})}$ is initially small, since the sample is enriched with water, it increases slightly with time, and after two hours of drying it increases stepwise. After 3 - 3.5 hours

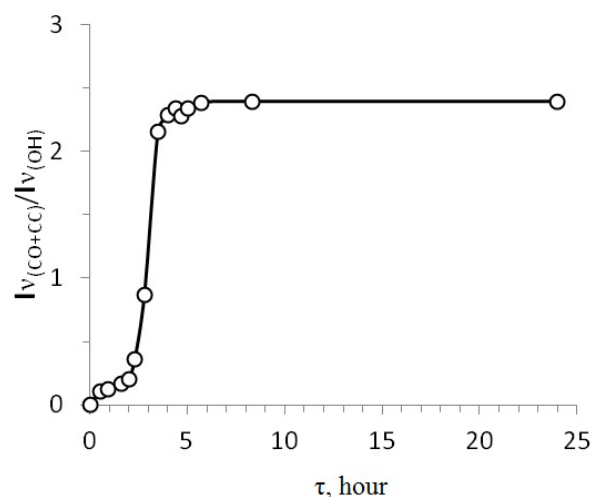


Fig. 2. Relative intensity of the $\nu(\text{CO} + \text{CC})$ and $\nu(\text{OH})$ lines in the IR spectra: dehydration products of an aqueous solution of maltotriose and reactive maltotriose.