

Глигор Јовановски

POLYMORPHISM IN THIOSACCHARIN

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It was found that thiosaccharin (*o*-sulfothiobenzimide) crystallizes in two polymorphic modifications designated α and β . The β phase is less stable and after a relatively short period transforms into a more stable, α phase.

Introduction

In the course of our investigations of the structural characteristics of some cyclic imides and thioimides, we have observed that one of the cyclic imides - thiosaccharin (*o*-sulfothiobenzimide) at different conditions (solvent, concentration, temperature) crystallizes in two morphologically rather different types, which we designated α and β . The crystals of the α grow into a very long yellow needles while those of the β phase are prismatic. Since no evidence was found in the literature about the existence of more than one phase of thiosaccharin¹, we decided to characterize those two phases, analyzing their infrared spectra as well as their X-ray powder diagrams.

Discussion

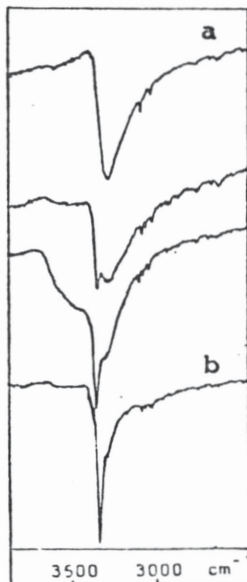


Fig. 1. The ν N-H region in a) β phase, b) α phase

The most noticeable differences between the spectra ($4000-200\text{ cm}^{-1}$) of those two phases appear in the region of the bands originating from the CSNHSO_2 fragment of the thiosaccharin. This fact is probably related to the existence of different types of hydrogen bonds between the thiosaccharin molecules in the crystals of two phases. The higher frequency of the band originating from the N-H stretching vibrations in the α phase (3340 cm^{-1}) compared with the frequency of the analogous vibrations in the β phase (3280 cm^{-1}) can be interpreted as an evidence of existence of weaker intermolecular hydrogen bonds in the α phase, (see Fig. 1). This is not in agreement with the stability of those two phases.

Namely, it was found that the β phase is less stable and after a relatively short period transforms into more stable α phase (this transformation is illustrated by the four infrared curves in Fig. 1). On lowering the temperature, the

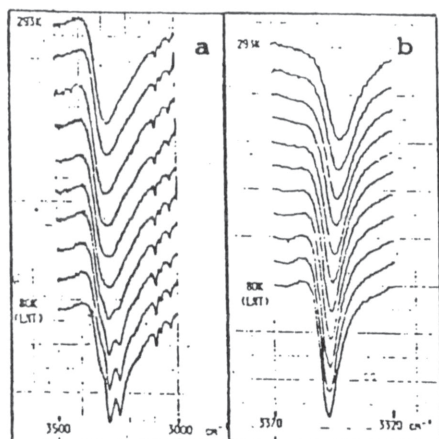


Fig. 2. Behaviour of the VN-H region by lowering the temperature in
a) β phase, b) α phase

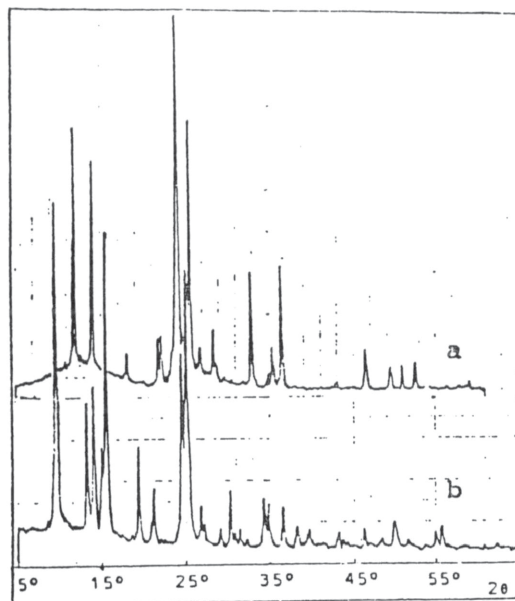


Fig. 3. X-ray diagrams of
a) β phase, b) α phase

band corresponding to the N-H stretching mode of the α phase shifts to higher frequency, while the frequency of the adequate mode in β phase remains unchanged (Fig. 2).

The appearance of the X-ray diagrams of the two morphologically different types of crystals of thiosaccharin (see Fig. 3) confirms the conclusions made by the infrared spectroscopy about the existence of polymorphism in thiosaccharin.

References

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- 2 O. Knop, W. Westerhouse, M. Falk, *Can. J. Chem.*, **58**, 867 (1980)