

Effects of Crystallinity on the Infra-red Absorption Spectra of Lactose and Dried Milk

In an earlier communication¹, it was shown that the infra-red absorption spectra of lactose-casein mixtures showed very marked changes after freeze-drying from aqueous solution. This was interpreted as indicating the presence of an interaction between the lactose and the casein. During the course of attempts to elucidate further the nature of this interaction, it has now been shown that these changes are due largely to the state of crystallinity of the lactose.

Fig. 1a shows the spectrum of crystalline lactose, obtained from a sample of the crystalline α -monohydrate. Fig. 1b shows the spectrum of amorphous lactose, obtained by freeze-drying a dilute aqueous solution, or by heat-drying a thin film of lactose solution on the surface of a barium fluoride plate. Examination with a polarizing microscope of the samples used for these spectra confirmed the presence of crystalline and amorphous material in the respective samples.

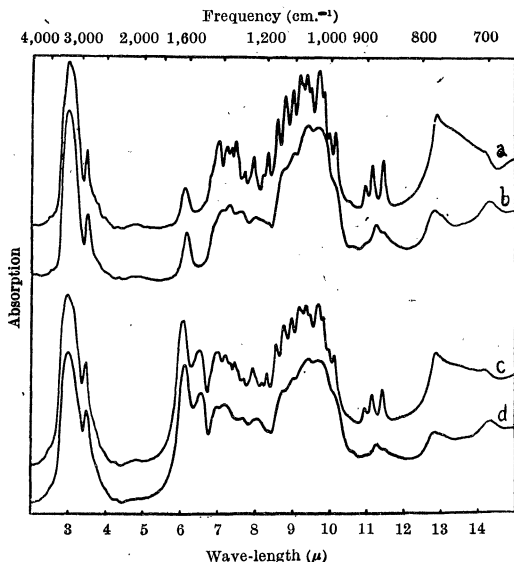


Fig. 1. Infra-red spectra of (a) crystalline lactose, (b) amorphous lactose, (c) 'soluble' dried separated milk, (d) dried separated milk

monohydrate of α -lactose crystallizes easily from solution at temperatures below 95° C., and the anhydrous sugar can be prepared by heating the monohydrate at 120–130° C.

The importance of the conditions of drying is indicated by the published spectra of heat-dried full-cream milk and freeze-dried separated milk, which show bands typical of α -lactose monohydrate and the glass form respectively.

We wish to thank Mr. S. J. Lovett for making the sample of β -lactose; this had $[\alpha]_D^{25} = +10.2$.

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When saturated lactose solutions were used for freeze-drying, the lactose appeared in the crystalline form, if the initial freezing stage was carried out too slowly. The effects of this can be seen in the spectrum 1A of ref. 1.

These results are of particular interest in the interpretation of the infra-red spectra of dried-milk samples, since the spectra of samples of dried separated milk made 'soluble' by the 'Instant' process² show that the lactose has been converted into the crystalline form (Fig. 1c). The spectra of samples which have been dried in the normal way show that the lactose exists in the amorphous form (Fig. 1d). Further confirmation of this change in the state of the lactose is afforded by freeze-drying solutions of 'soluble' milk powder, when the spectra are similar to Fig. 1d, showing that the lactose has reverted to the amorphous form.

The conversion of dry amorphous lactose to the crystalline α -monohydrate on exposure to atmospheric water vapour can be shown by the infra-red spectra, and similar changes can also be seen in the spectra of dried-milk samples under similar conditions.

The spectra were obtained with a double-beam Grubb Parsons S3A spectrometer fitted with a rock-salt prism, and the samples were examined using the potassium bromide pressed-disk technique. Similar results were obtained when the samples were examined by the 'Nujol' mull technique.

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