

FIG. 1. Time curve of anhydride formation by the reaction of palmitic and oleic acids with DCC. The reaction was started by the addition of 1 mmole of DCC in 5 ml of CCl_4 to 2 mmoles of fatty acid in 15 ml of CCl_4 . Aliquots were taken at intervals for neutral hydroxamate determination (12). Zero-time values were obtained by subsequent addition of fatty acid and DCC to hydroxylamine.

The influence of different solvents on the course of the reaction between DCC and several fatty acids is summarized in Table 1. Carbon tetrachloride was found to be the solvent in which the highest yield of anhydride was produced. From the kinetics of the formation of oleic and palmitic anhydrides at 25° it is concluded that the reaction is complete after 40 min (Fig. 1).

The acid anhydride can be freed from the small amounts of DCAU and residual free acid by crystallization. This purification step can be omitted when the anhydride is used for acylation reactions, since the small amounts of DCAU and free acid do not interfere with these reactions.¹

Synthesis of Palmitic Anhydride. A solution of DCC (10 mmoles) in dry CCl_4 (50 ml) was added to a solution of palmitic acid (20 mmoles) in dry CCl_4 (150 ml). The reaction mixture was kept at room temperature. After 5 hr the DCU precipitate was removed by filtration and the solvent was removed by evaporation under

¹ Lapidot, Y., and Z. Selinger, manuscript submitted for publication.

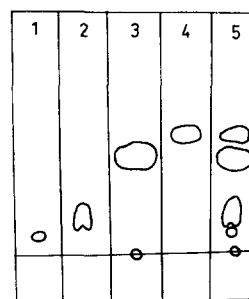


FIG. 2. Thin-layer chromatography on microchromatoplate coated with silica gel (without CaSO_4) by dipping (13). Solvent: petroleum ether (bp $40-60^\circ$)-diethyl ether 8:2 (v/v). Indicator: Charring with aqueous sulfuric acid 1:1 (v/v). 1, DCAU; 2, palmitic acid; 3, DCC with small spot of DCCU at the origin; 4, palmitic anhydride; 5, mixture 1-4.

reduced pressure. The solid residue was recrystallized from acetone (125 ml) yielding 4.22 g of pure palmitic anhydride (85.4%), mp 64° . A thin-layer chromatogram of starting materials and reaction products is shown in Fig. 2.

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WARNING

Several laboratories have reported that skin sensitivity and, later, acute respiratory difficulties may result from continual use of DCC.

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