with dilute sulfuric acid, and the xylene layer was separated. The aqueous layer was extracted again with ether, and the combined xylene-ether extracts were washed with water and dilute sodium bicarbonate solution and dried over anhydrous magnesium sulfate. Distillation of the solvents through a Vigreaux column afforded a residue, the infrared spectrum of which was identical to that of the starting trans-dimethyl ester 70. Analysis of the residue by GLC revealed the presence of a small amount of xylene, 70, and possibly a third component with a retention time slightly greater than that of 70. The third component detected by GLC was isolated by column chromatography on florex.

Column chromatography of products from acyloin reactions II and III. The combined residues from acyloin reactions II and III were placed on a 2 x 25 cm. column of florex in petroleum ether (b.p. 20-40°C.). Elution with this solvent was continued until no residue was obtained upon evaporation of the eluate. The column was then stripped with ether, and the solvent was removed to give 1.26 g. of a yellow oil. This material was oxidized with chromium trioxide by the method of Bowden and co-workers.54 Reaction of the compound in 3 ml. of acetone with a solution of 0.8 g. (0.008 mole) of chromium trioxide and 1 g. of sulfuric acid in 3 ml. of water gave