NEW COMPOUNDS

An Improved Preparation of 1-Naphthylpropiolic Acid

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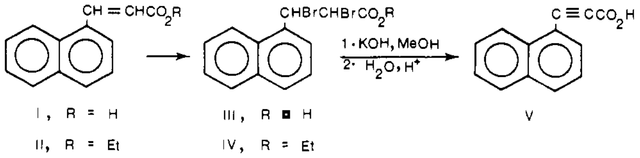
The preparation of 1-naphthylpropiolic acid can be facilitated by an improvement in the preparation of 1-naphthylpropanoic acid dibromide. A recrystallized sample of 1-naphthylpropenoic acid can be brominated quickly by the use of boiling carbon tetrachloride. Although, a priori two diastereomeric dibromides are possible, bromination under these conditions proceeded to supply only one isomer.

1-Naphthylpropiolic acid (V) has been prepared (*1*) by dehydrobromination of the dibromide IV obtained from the photoinduced bromination of ethyl 1-naphthylpropenoate (II). Esterification prior to bromination was necessary since direct bromination of the acid I in ice-cold carbon tetrachloride was found extremely slow. We now report that 1-naphthylpropenoic acid (I) may be brominated directly and in an excellent yield by using boiling carbon tetrachloride to yield only one isomer, mp 181–182 °C. Dehydrobromination of III was accomplished smoothly with 25% methanolic KOH solution instead of the 98% solution used in the older procedure. The overall yield of acid V from the starting acid I was 82%.



Experimental Section

Melting points were measured in an electrothermal melting point apparatus and are uncorrected. Infrared spectra were recorded on Pye-Unicam SP 300 spectrophotometer. NMR spectra were recorded in $CDCl_3$ on Bruker WP 80-SY spectrometer with Me_4Si as an internal standard.

3-(1-Naphthyl)propanoic Acid Dibromide (III). To a suspension of 3-(1-naphthyl)propenoic acid (*2*) (89.5 g, 0.25 mol) and carbon tetrachloride (750 mL) in a two-necked round-bottomed flask fitted with a reflux condenser and a separatory funnel, heated to boiling with continuous stirring, was added bromine (40.0 g, 0.25 mol) in carbon tetrachloride (100 mL). The bromine color disappeared slowly at first, then rapidly so that all bromine was added in the course of 30 min. The solution was poured into a large dish, and all the carbon tetrachloride and unused bromine allowed to evaporate. The dibromide III separated as fine crystals which formed a solid cake in the bottom of the dish. Crystallization from CCl_4 afforded 144 g (89%) of white crystals of III, mp 181–182 °C; NMR ($CDCl_3$) δ 7.52 (m, 7 H, Ar*H*), 6.33 (d, 1 H, C*H*, J = 12 Hz), 5.34 (d, 1 H, C*H*, J = 12 Hz), and 11.72 (s, 1 H, COO*H*). Elemental analysis (C, H, Br) in agreement with theoretical values was obtained and submitted for review.

3-(1-Naphthyl)propiolic Acid (V). A mixture of 2-(1-naphthyl)propanoic acid dibromide (118 g, 0.33 mol) in 25% potassium hydroxide/methanol (400 mL) was heated for 30 min over a steam bath. The mixture was poured into a large dish and the solvent allowed to evaporate. The dry residue was dissolved in water and acidified. The precipitated acid was collected and recrystallized from water to give white needles, 59.4 g (92%), mp 137–139 °C (lit. (*3*) 137 °C) ν_{max} $CHCl_3$ 2210, 1695 cm^{-1}; NMR ($CDCl_3$) δ 7.41 (m, 7 H, Ar*H*) and 11.37 (s, 1 H, COO*H*). Elemental analysis (C, H) in agreement with theoretical values was obtained and submitted for review.

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Synthesis and Properties of Some New Hydroxamic Acids

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Five new hydroxamic acids are synthesized for their plausible analytical applications. These acids are characterized by elemental analysis, melting points, UV, IR, NMR, and mass spectra. Their physicochemical properties have been determined.

Introduction

Hydroxamic acids are weak acids widely used for organic and inorganic analysis including pharmaceuticals, as food additives, and in nuclear fuel processing (*1–7*). In the present communication five new hydroxamic acids have been reported and

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