gram (0.05 mole) quantity of N-carbethoxy-β-aminopropionic acid was dissilved in 15 ml. of thionyl chloride. The mixture was allowed to stand with occasional ice-bath cooling when gas evolution became too rapid. After a short time gas evolution ceased and the mixture was allowed to stand overnight at room temperature. The excess thionyl chloride was removed under reduced pressure and a solution of 7.3 grams (0.05 mole) of β-naphthol in 175 ml. of benzene was added. Pyridine (10 ml.) was added and a white ppt. formed, which was removed by filtration. The filtrate was extracted with 5% HCl, 5% $NaHCO_3$, 1% NaOH, and, finally, with water. It was dried over $MgSO_4$, and the solvent was removed in vacuo. The remaining solid was recrystallized first from 95% ethanol and then from benzene-petroluem ether. Recovered were 6.2 grams (44%) of crystals melting at 91.5–93°. An analytical sample was prepared from a similar run, m.p. 91–93.5°.

On dissolving the β-naphthyl ester in aqueous sulfuric acid and treating with sodium nitrite, no product separated. When the addition was complete and the reaction mixture was diluted with water, only a tarry mass separated from which no crystalline product was obtained by any means attempted.

Attempted Preparation of Diphenylmethyl N-Carbethoxy-β-aminopropionate. The preparation of this ester was attempted by means of treating diphenylmethyl bromide with N-carbethoxy-β-aminopropionic acid in the presence of alkali (*3*). A thick oil was obtained which was resistant to crystallization.

When this oil was treated with sulfuric acid preliminary to nitrosation, solid benzhydryl ether was formed, as shown by mixed m.p. with an authentic sample. The formation of this ether is not surprising in view of its ready formation from diphenylmethyl bromide in water (*2*).

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The Swamping Catalyst Effect VII. Synthesis of 1,6-Diacetonaphthalene

D. E. PEARSON and C. R. McINTOSH
Vanderbilt University, Nashville, Tenn.

MANY ATTEMPTS were made in this Laboratory to acylate acetophenone under swamping conditions. They, like previous attempts (*1*), failed, and it was concluded that the acylating agent was not active enough to substitute into a deactivated nucleus. Even trifluoroacetyl chloride failed to substitute into acetophenone but instead was converted to carbon. Apparently, acetophenones cannot be acylated except if an alkyl group is situated ortho to the acetyl group such as in acetomesitylene (*4*).

Although diacylation of a benzene ring was not possible, diacylation of a naphthalene ring was brought about by acetylation of β-acetonaphthalene to yield the new compound, 1,6-diacetonaphthalene. Characterization of this compound is given in the Experimental and described more fully in the thesis by McIntosh (*6*).

EXPERIMENTAL

1,6-Diacetonaphthalene. The apparatus, conditions and isolation have been described (*8*). β-Acetonaphthalene (85 grams, 0.5 mole) was complexed with anhydrous aluminum chloride (1.6 mole) while the internal temperature was maintained at 70–80°. The complex was a viscous, reddish black fluid. Acetyl chloride (0.66 mole) was added dropwise to the stirred mixture over a period of about an hour and the mixture heated at 70–80° for an additional hour. The crude product, b.p. 147–160° at 0.1 mm. Hg, weighed 82 grams, 77%, m.p. 65–70°. It was recrystallized from a mixture of methylcyclohexane and isopropyl alcohol to give 53 grams (50%) of slightly off white fine crystals, m.p. 80.8–82°. A small sample, sublimed at 0.1 mm. Hg, melted at 82–82.5°.

Calcd. for $C_{14}H_{12}O_2$: C, 79.23: H, 5.70. Found: C, 79.13; H, 5.85.

1,6-Dicarboxynaphthalene. The diketone was oxidized with aqueous sodium hypobromite using a Morton high speed stirrer to give intimate contact between diketone and reagent (*7*). The diacid was obtained in 76% yield, m.p. 322–325°, reported m.p. 310° (*2*); neutral equivalent calcd. 108, found 108.8; dimethyl ester made from the acid chloride and methanol, m.p. 97.5–98°, reported 99° (*2*).

1,6-Diacetaminonaphthalene. The dioxime, m.p. 189–191°, made from the diketone, was rearranged to the titled compound by the method of Horning (*5*). The yield was 80% of white needles, m.p. 262.5–263.5°, reported m.p. 263.5° (*3*).

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