α-Indanone from β-Propiolactone

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The preparation of α-tetralone by condensation of benzene with γ-butyrolactone in the presence of excess aluminum chloride was reported several years ago.[1,2] This reaction does not appear to have been extended to the preparation of α-indanone from β-propiolactone, although a mixture of β-phenylpropionic acid (62%) and phenyl vinyl ketone (15%) was obtained in one study,[3a] while no product—β-phenylpropionic acid was expected—was isolated in another.[3b] In the present report use of excess aluminum chloride has given α-indanone in *ca.* 80% yield when the lactone was added to an excess of aluminum chloride and benzene. The reverse order of addition (aluminum chloride added to lactone and benzene)[1,2] gave only a 30% yield.

The present method has the usual advantage of a one-step synthesis from available starting material and presumably can be extended to the preparation of substituted and polycyclic compounds. Previous one-step procedures employed the much more expensive β-chloropropionyl chloride (55% yield)[4] and acrylyl chloride (3% yield).[5] α-Indanone also has been prepared from indene[6] and indane[7,8] by oxidative procedures; from β-phenylpropionic acid by cyclization with polyphosphoric acid,[9,10] with fluosulfonic acid,[11] with trifluoroacetic anhydride,[12] and with aluminum chloride-sodium chloride;[13] and from β-phenylpropionyl chloride by cyclization with aluminum chloride.[14]

EXPERIMENTAL

A solution of 43.5 g. (0.605 mole) of β-propiolactone in 100 ml. of benzene was added dropwise during 45 min. to a stirred mixture of 300 g. (2.25 moles) of anhydrous aluminum chloride and 400 ml. of sodium-dried, thiophene-free benzene. The mixture was heated for 18 hr. under reflux, cooled, and poured over ice and hydrochloric acid. The aqueous layer was extracted with ether and the combined organic phases were washed with water, 0.1*N* potassium hydroxide solution and water, then dried over sodium sulfate. Solvent was removed by flash distillation and the residue was distilled through a 30-cm. spiral-wound column with total reflux head. Indanone (61.5 g., 77%) boiled at 84–85°/1.5 mm., m.p. 40° (lit.[9] m.p. 40–42°); its 2,4-dinitrophenylhydrazone melted at 256.5–257.5° (lit.[15] m.p. 258°).

The distillation residue (46.5 g.) was extracted with petroleum ether (b.p. 60–80°) in a Soxhlet extractor. Concentration of the extract yielded 10.1 g. of yellow oil, whose infrared spectrum showed it to be mainly indanone. The oil was dissolved in a minimum of ethanol and added to a solution of 15 g. of 2,4-dinitrophenylhydrazine in ethanol. The immediate precipitate of indanone-2,4-dinitrophenylhydrazone was filtered and crystallized from ethyl acetate, then from glacial acetic acid; wt., 7.4 g. (4%), m.p. 256–257°. The total yield of indanone was thus 81%.

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