A Modified Method of Preparing of ω, p-Dihydroxyacetophenone

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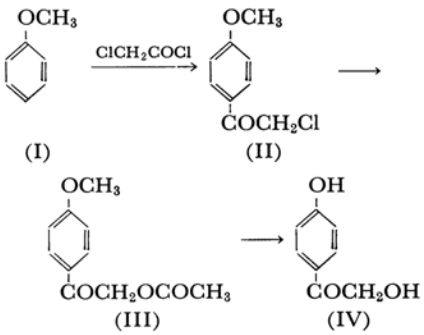
The earlier method for the preparation of ω, p-dihydroxyacetophenone (IV)[1,2] needed an improvement because of difficulties encountered in the isolation of the key intermediate, ω-chloro-p-hydroxyacetophenone,[1,3] which in its crude form is extremely lachrymatory. In the present investigation, a modified procedure has been employed to eliminate the use of ω-chloro-p-hydroxyacetophenone.



Chloroacetyl chloride and anisole (I) are condensed in cold in the presence of aluminium chloride[4] to give ω-chloro-p-methoxyacetophenone (II). This compound II, on treatment with anhydrous potassium acetate in absolute ethanol, yields ω-acetoxy-p-methoxyacetophenone[5] (III). This III is then demethylated with aluminium chloride. In the last reaction, deacetylation takes place simultaneously, and thus the final product, ω, p-dihydroxyacetophenone (IV), is directly obtained.

Experimental

ω, p-Dihydroxyacetophenone (IV, obtained by the demethylation and deacetylation of III): ω-Acetoxy-p-methoxyacetophenone (1.30 g.) was dissolved in dry benzene (30 ml.), and anhydrous aluminium chloride (4.0 g.) was added to the solution. The reaction mixture was refluxed for 3 hr., and the solvent removed under reduced pressure. The residual mass was hydrolysed with ice and hydrochloric acid to give a solid, which was extracted with ethyl acetate. The ethyl acetate solution was evaporated and the residual mass crystallised from hot water to yield colourless prisms of the final product, ω, p-dihydroxyacetophenone (0.76 g., 80%); m. p. 177—178°C (sinters at 170°C). (Found: C, 63.2; H, 5.2. Calcd. for $C_8H_8O_3$: C, 63.2; H, 5.2%).

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