Preparation and Photolysis of Acyloins: A New Acylation of Pyrroles and Indole

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Summary Photolysis of the acyloins obtained by reaction of biacetyl with pyrroles and indole readily affords, in high yield, 2-acetylpyrroles and 3-acetylindole, respectively.

WE now report a novel acylation of pyrroles and indole by photolysis of acyloins.

The condensation of pyrroles with ketones and aldehydes is a useful method for the synthesis of dipyrrylmethanes. 1-3 Accordingly, we prepared the acyloins by reaction of biacetyl with pyrroles and indole under mild conditions. A solution of the pyrrole (I) and excess of biacetyl (neat or in benzene) in the presence of formic acid was set aside at room temperature. After 1 day, distillation under reduced pressure afforded (50-60%) pure 1:1 adducts[†] (IIa), b.p. 102° at 4 mmHg, and (IIb), b.p. 90° at 5 mmHg. Similar treatment of indole (IV) gave 3-(1-acetyl-1-hydroxyethyl)indole (V) (70-77%), m.p. 136.5-137.5°

The acyloins were photolysed using a 350 W highpressure mercury lamp (Pyrex filter) at room temperature under N₂. In every case, the starting acyloin was completely consumed after 48 h irradiation. Photolysis of (IIa) and (IIb) in benzene followed by distillation of photolysate gave pure (IIIa) (80%), b.p. 95° at 15 mmHg, m.p. 90°, and (IIIb) (67%), b.p. 90° at 23 mmHg, respectively. The spectra of (IIIa) and (IIIb) were in accord with those of authentic samples.^{4,5} Photolysis of (V) in MeOH followed by sublimation under reduced pressure gave pure (VI) (77%), m.p. 188-189°, identified by comparison with an authentic sample.⁶



Photolysis of the corresponding acyloins is potentially a valuable synthetic route to acylated pyrroles and indoles. The scope and mechanism of this photolysis are now under investigation.

(Received, November 8th, 1971; Com. 1928.)

+ Satisfactory elemental analyses were obtained for all compounds reported herein.

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