

SELECTIVE REDUCTION OF DIETHYL HOMOPHTHALATE  
WITH DIISOBUTYLALUMINIUM HYDRIDE

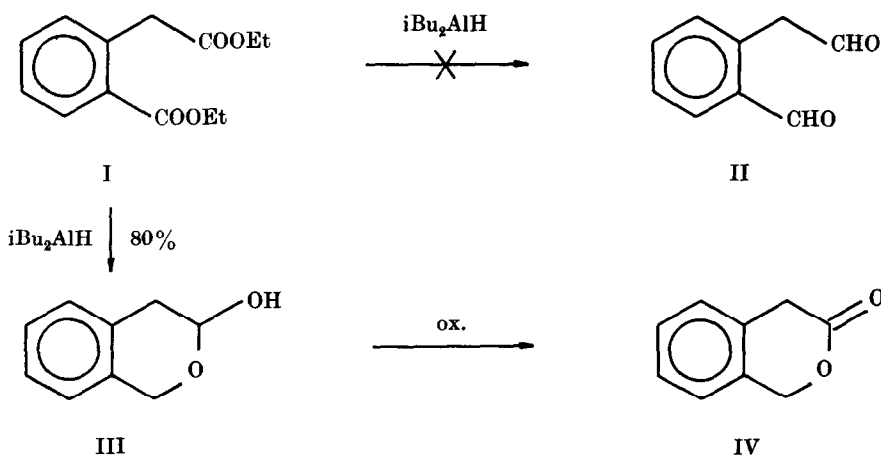
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We wish to report a novel selective reduction of diethyl homophthalate (I) with diisobutylaluminium hydride. As part of another project, we wished to prepare the homophthalaldehyde (II) with reduction of I. Although diisobutylaluminium hydride has been used successfully for reducing aliphatic and aromatic carboxylic acids and esters resulting in both aldehydes and alcohols (1—3), reduction of I under various conditions did not give the desired product.

Diethyl homophthalate (I) was treated in extra dry toluene at  $-60^\circ$  with three mol of diisobutylaluminium hydride in toluene (1 : 1) for two hours. After hydrolysis with sodium potassium tartarate solution, the reaction mixture was concentrated at reduced pressure to give a colourless or pale yellow crystalline material, 3-hydroxyisochromane (III) in 80% yield, m.p.  $71-71.5^\circ$  (from diisopropyl ether) (6).





Compound III showed strong ir absorption bands at  $3350\text{ cm}^{-1}$  (O—H),  $1140$ ,  $1060\text{ cm}^{-1}$  (C—O—C), and  $750\text{ cm}^{-1}$  (aromatic  $\gamma$  C—H), however, no carbonyl absorption was observed. In the nmr spectrum (60 MHz, in  $\text{CDCl}_3$ ) there were five separate line groups at  $\delta$  7.10, 5.25, 4.78, 4.12 and 2.87 ppm characterising the aromatic, methyne, 1-methylene, hydroxyl, and 4-methylene protons, respectively. With adding  $\text{D}_2\text{O}$ , the OH signal was absent, showing that exchange had occurred.

The structure of III was confirmed by conversion with chromic acid solution, in ether at  $0^\circ$  (4) to the known lacton IV, m.p.  $80\text{--}82^\circ$ ; lit. m.p.  $80\text{--}82^\circ$  (5). IV gave a very simple nmr spectrum ( $\delta$  7.20, 5.22 and 3.44 ppm, corresponding to aromatic, 1-methylene, and 4-methylene protons, respectively).

Treatment of III with triethyl orthoformate at room temperature gave the 3-ethoxyisochroman (V) in good yield, as a colourless liquid, b.p.  $79\text{--}82^\circ/0.15\text{ mm}$ ,  $n_D^{25}$  1.5018 (6).

Further work is in progress attempting to extend this selective reduction to include other dicarboxylic acid esters.

#### REFERENCES

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