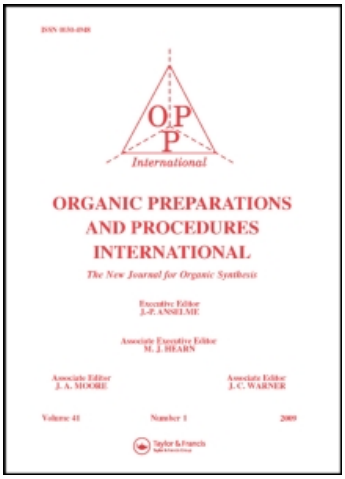

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3-ACETYL-2,4,6-TRIMETHYLBENZOPHENONE

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3-ACETYL-2,4,6-TRIMETHYLBENZOPHENONE

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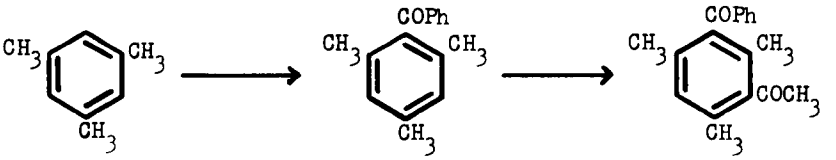

Previous attempts to prepare 'mixed' diacylmesitylenes by the Friedel-Crafts acylation reaction were unsuccessful.[1] Synthesis of the title compound becomes straightforward, provided the benzoylation stage is carried out first, and the intermediate complex is treated in situ with acetyl chloride and aluminum chloride.[2]

Experimental

3-Acetyl-2,4,6-trimethylbenzophenone. To a stirred mixture of mesitylene (12 g.) and powdered anhydrous aluminum chloride (26.7 g.) in dry carbon disulfide (20 ml.), benzoyl chloride (15.5 g.) was added dropwise over 10 min. The mixture was gently boiled under reflux for ½ hr., and cooled to room temperature. More aluminum chloride (20 g.) was then added, followed by dropwise addition over 5 min. of a solution of acetyl chloride (7.9 g.) in carbon disulfide (10 ml.). The mixture was again gently boiled for ½ hr.[3]

The reaction mixture was cooled in ice, and treated with concentrated hydrochloric acid and crushed ice, added alternatively in portions and in large excess, and stirred for 1 hr. The organic layer

was separated, and the aqueous phase was extracted with chloroform. The combined organic extracts were washed with 2 N sodium hydroxide solution, 3 N hydrochloric acid, and with water, dried ($MgSO_4$) and evaporated to give an oil. Distillation gave a pale yellow viscous oil (14.8 g.), b.p. 150-168°/0.5 mm., which slowly solidified. Analysis by gas-liquid chromatography (Apiezon L, 20%, on Chromosorb P, with N_2 as carrier gas, flow rate 20 ml./min., at 200°) revealed the components to be: 3-acetyl-2,4,6-trimethylbenzophenone (93%), 2,4,6-trimethylbenzophenone (3.3%), 1,3-dibenzoyl-2,4,6-trimethylbenzene (2.2%), and 1,3-diacetyl-2,4,6-trimethylbenzene (1.3%). The residue from the distillation was dissolved in a minimum volume of benzene, and filtered through a short column of chromatographic alumina, and the eluate evaporated to give a pale yellow glass (9.8 g.). The glass comprised: 3-acetyl-2,4,6-trimethylbenzophenone (69%) and 1,3-dibenzoyl-2,4,6-trimethylbenzene (31%). Crystallisation of both fractions separately from acetone, and methanol, afforded 3-acetyl-2,4,6-trimethylbenzophenone (18.6 g., 70%) as colorless crystals, m.p. 81-81.5°.

Anal. Calcd. for $C_{18}H_{18}O_2$: C, 81.16; H, 6.82. Found: C, 80.82; H, 6.74.

I.R. bands (CCl_4): 1706 (acetyl C=O), 1677 (benzoyl C=O) $cm.^{-1}$;

U.V. spectrum (MeOH): 194 nm (47,000), 200 (49,000), 251.5 (18,200) and 278 (sh) (3000); N.m.r. (CCl_4) in p.p.m.: τ 8.07 (s,2-CH_3), 798 (s,4-CH_3), 7.79 (s,6-CH_3), 7.64 (s,$COCH_3$), 3.16 (s,5-H) and 2.76-2.20 (m,C_6H_5).

References

1. H. Weil, Ber., 30. 1285 (1897).
2. P.H. Gore and J.A. Hoskins, J. Chem. Soc.,(C), 517 (1970).
3. Constant stirring is essential, as the reaction is partially heterogeneous.

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