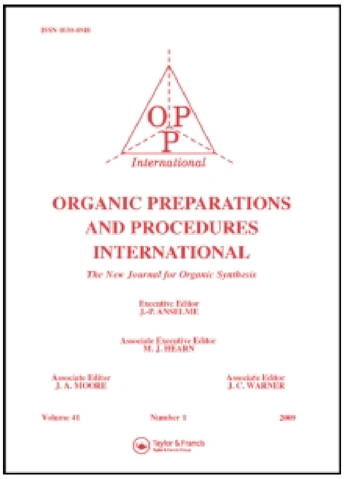

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THE MONOALKYLATION OF DIBENZOYLMETHANE

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THE MONOALKYLATION OF DIBENZOYLMETHANE

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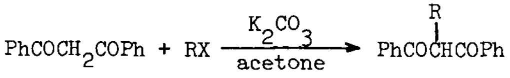
The alkylation of dibenzoylmethane using potassium carbonate[1,2] gives excellent yields (60-90%) of the alkylated products(I).



$$\text{PhCOCH}_2\text{COPh} + \text{RX} \xrightarrow[\text{acetone}]{K_2CO_3} \text{PhCO}\overset{\overset{\text{R}}{|}}{\text{C}}\text{HCOPh}$$

I

a) R = CH_3 b) R = C_2H_5 c) R = $C_6H_5CH_2$ d) R - *p*-$NO_2C_6H_4CH_2$

e) R = *p*-$BrC_6H_4CH_2$ f) R = *p*-$Me_3CC_6H_4CH_2$ g) R = *p*-$CH_3C_6H_4CH_2$

Crude product yields were nearly quantitative (except in the case of Id) and were pure enough for further reactions but only the yields of recrystallized products are reported. Analytical and spectral data are reported for the new compounds (Ie-g).

Alkyl Halide Used	Yield of Alkyl-dibenzoylmethane(%)
Methyl Iodide	90 (Ia)
Ethyl Iodide	80 (Ib)
Benzyl Bromide	85 (Ic)
p-Nitrobenzyl Chloride	60 (Id)
p-Bromobenzyl Bromide	89 (Ie)
p-*tert*-Butylbenzyl Bromide	73 (If)
p-Methylbenzyl Bromide	88 (Ig)

EXPERIMENTAL

All melting points are corrected. IR spectra were recorded on a Perkin-Elmer 599 or 337 spectrometer, and NMR spectra on Perkin-Elmer R-12B or R24B spectrometers. The analysis of new compounds was performed by Chemalytics, Inc., Tempe, Arizona and Galbraith Laboratories, Knoxville, Tennessee.

p-(t-Butyl)benzyldibenzoylmethane. Typical Procedure.- In a 25 ml round bottom flask dibenzoylmethane (1.0 g, 4.5 mmol), p-tert-butylbenzylbromide (1.04 g, 4.6 mmol), and anhydrous potassium carbonate (0.63 g, 4.56 mmol) were mixed in 20 ml of acetone (reagent grade distilled once); the mixture was heated to reflux for 18 hrs. with magnetic stirring. The reaction mixture was cooled, filtered and the solid residue was thoroughly washed with acetone. Acetone was removed from the combined washings at the rotary evaporator and the resulting solid residue was recrystallized from absolute ethanol to yield pure p-(t-butyl)benzyldibenzoylmethane, mp. 116-117°. Anal. Calcd for $C_{26}H_{26}O_2$: C, 84.27; H, 7.09. Found: C, 84.06; H, 7.10. IR (nujol mull): 1698 cm^{-1}. ^1H nmr: (δ values, $CDCl_3$): 1.87 (s, 9H, $C(CH_3)_3$), 3.38 (d, J = 6.5 Hz, 2H, $-CH_2$), 5.48 (t, J = 6.5 Hz, 1H, C-H), 6.55-8.55 (m, 14H, aromatic).

p-Bromobenzyldibenzoylmethane, mp. 123-124°, IR (nujol mull): 1675 cm^{-1}. ^1H nmr (δ values, $CDCl_3$): 3.35 (d, J = 6.5 Hz, 2H, $-CH_2$), 5.42 (t, J = 6.5 Hz, 1H, C-H), 6.52-8.65 (m, 14H, aromatic).

Anal. Calcd. for $C_{22}H_{17}BrO_2$: C, 67.18; H, 4.37. Found: C, 67.12; H, 4.17.

p-Methylbenzyldibenzoylmethane, mp. 86-87°, IR (KBr): 1680 cm^{-1}. ^1H nmr (δ values, $CDCl_3$): 2.23 (s, 3H, $-CH_3$), 3.40 (d, J = 7.0 Hz, 2H, $-CH_2$), 5.50 (t, J = 7.0 Hz, 1H, C-H), 7.50 (m, 14H, aromatic).

Anal. Calcd for $C_{23}H_{20}O_2$: C, 84.08; H, 5.73. Found: C, 84.46; H, 6.16.

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