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A Note on the Synthesis of 2,7-Dibenzoyl-4,5-benztropone

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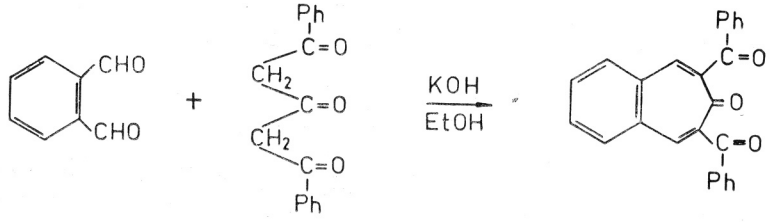
The synthesis of the 4,5-benztropone ring system was first described by Thiele *et al.*[1] Recently, Davey and Gottfried[2] prepared 2-acetyl-4,5-benztropone by condensation of o-phthalaldehyde with acetylacetone. In our endeavour to prepare a number of 4,5-benztropone derivatives the present note reports the synthesis of 2,7-dibenzoyl-4,5-benztropone (III) according to the sheme:



I II III

The addition of potassium hydroxide to an equimolar ethanolic solution of phthalaldehyde (I) and dibenzoyl-acetone (II) afforded III in a fairly high yield (59%). According to elemental analysis, UV, and IR spectra we assume that the new compound has the proposed structure.

EXPERIMENTAL

All melting points are uncorrected. IR absorption spectra were measured on a Perkin-Elmer Infracord 137 spectrophotometer, and the UV-absorption spectra on a Zeiss Universal spectrophotometer type VSU 1.

Phthalaldehyde (I)

The substance was prepared from o-xylene following the method of Bill and Tarbell[3]. The intermediate α, α, α′, α′ - tetrabromo-o-xylene was obtained in 86.4% yield and the product in 81.5% yield (calc'd. on tetrabromo-o-xylene).

2,7-Dibenzoyl-4,5-benztropone (III)

A solution of phthalaldehyde (1.5 g. 11.2 mmoles) in absolute ethanol (20 ml.) was added to the warm mixture of dibenzoylacetone[4] (2.7 g., 11.2 mmoles) in absolute ethanol (40 ml.). The flask-content was brought to boil on the water bath and freshly prepared 5 *N* potassium hydroxide in methanol (1.2 ml.) was dropwise added. Immediately white needlelike crystals appeared and the refluxing was

continued for 15 minutes. After cooling at room temperature for 2 hrs., the crystals were collected on a Büchner funnel and washed with small portions of ethanol. The crude product (2.4 g., 69%) melted after drying at 239.5—240.5°. Recrystallization from optically pure chloroform gave a product melting at 240.5 — 241°. Resublimation at 200—210°/0.05 mm Hg gave colourless needles of the same m.p. The ultraviolet absorption spectrum in absolute ethanol (c 2.87×10^{-5}): λ 280 mμ, ε 2.23×10^{4} and inflexion λ 255 mμ ε 1.41×10^{4}.

The infrared spectrum in potassium bromide plates showed the bands at 1681 cm^{-1}, 1631 cm^{-1}, and 1609 cm^{-1} which are attributed to carbonyl stretching of seven membered aromatic ring,[2,5] and the —C=C— streching bands at 1591 cm^{-1} and 1565 cm^{-1}.

Anal. $C_{25}H_{16}O_3$ (364.41) calc'd.: C 82.40; H 4.43%
found: C 82.69; H 4.59%

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IZVOD

Sinteza 2,7-dibenzoil-4,5-benztropona

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Opisana je sinteza 2,7-dibenzoil-4,5-benztropona (III) iz o-ftalaldehida i dibenzoilacetona. Na osnovu elementarne analize, UV i IR spektra pretpostavlja se da dobiveni produkt imade predloženu konstituciju.

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