A Note on *sym*-Dibenzoyl Acetone. Polyoxo Compounds. VII*

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In the 3rd communication of this series[1], Balenović and Munk described a compound obtained by alkaline hydrolysis of 2,6-diphenyl pyrone, which, after a molecule of water was split off, yielded the original pyrone. They came to the conclusion that this compound is identical with the hitherto undescribed *sym*-dibenzoyl acetone. The ketonic form of this compound should contain very reactive methylene groups in 1,3-positions, and should, therefore, readily react with sodium nitromalondialdehyde[2]. In fact, a compound was obtained by this condensation which corresponded to the undescribed 2,6-dibenzoyl-4-nitrophenol, giving further evidence for the proposed structure of *sym*-dibenzoyl acetone.

EXPERIMENTAL

2,6-Dibenzoyl-4-nitrophenol.

Sym-Dibenzoyl acetone (5 g., 0.02 mole) was dissolved at room temperature in ethanol (25 ml.) to which *N*-sodium hydroxide (10 ml.) had been previously added. To the resulting clear solution, a solution of finely powdered sodium nitromalondialdehyde (2.95 g., 0.03 mole) prepared following Fanta's procedure[3] in *N*-sodium hydroxide (25 ml.) and water (25 ml.) was added. This mixture was shaken at room temperature until it became clear (2 hours). After standing for 48 hours 4.4 g. (63.5%) of the yellow sodium salt of 2,6-dibenzoyl-4-nitrophenol were collected, which after recrystallization from ethanol had the m. p. 300° C. A stream of carbon dioxide was passed through the filtrate until saturation, and 0.75 g. of *sym*-dibenzoyl acetone (m. p. 107°) separated. The finely powdered sodium salt of 2,6-dibenzoyl-4-nitrophenol was suspended in a mixture of water and chloroform, acidified with hydrochloric acid to pH = 3 ,and extracted in a continuous extractor with chloroform for 12 hours. From chloroform-petroleum ether yellow crystals of *2,6-dibenzoyl-4-nitrophenol* separated, which after sublimation at 170°/0.02 mm. had the m. p. 163°. This compound gave an orange-red color with an ethanolic ferric chloride solution.

Anal. 12.778 mg. subst.: 32.36 mg. CO_2, 4.32 mg. H_2O
$C_{20}H_{13}O_5N$ (347.31) calc'd: C 69.16; H 3.77%
found: C 69.10; H 3.78%

REFERENCES

1. K. Balenović and R. Munk, *Arhiv kem.* **18** (1946) 41.
2. Hill and Torrey, *Am. Chem. J.* **22** (1899) 89.
3. P. E. Fanta, *Organic Syntheses* **32** (1952) 95.

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IZVOD

Bilješka o *sim*-dibenzoilacetonu
Poliokso spojevi. VII

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Kondenzacijom *sim*-dibenzoilacetona, opisanog u trećem saopćenju ovoga niza[1], s natrijevom soli nitromalondialdehida, dobiven je neopisani 2,6-dibenzoil-4-nitrofenol, što je novi dokaz, da hidroliza 2,6-difenilpirona, opisana u trećem saopćenju, daje *sim*-dibenzoilaceton.

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