

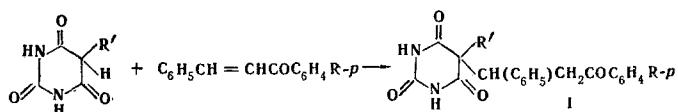
REACTION OF BARBITURIC ACID WITH
 α, β -UNSATURATED KETONES

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The addition of barbituric acid to α, β -unsaturated ketones has been accomplished for the first time.

The physiological activity of barbituric acid derivatives is well known. In order to study the effect on this activity of a substituent (R) far removed from the barbituric acid residue,* we have synthesized compounds of the general formula I (R' = H) (see Table 1).



The frequencies of the C=C bond are absent in the IR spectra of the compounds obtained, and the presence of an NH band (3380 cm^{-1}) was found in the case of the product of the addition of both barbituric and N-phenylbarbituric acids. (The intensity in the second case was half that in the first.) This indicates that the addition proceeds at the expense of the carbon atom of the methylene group.

Similar addition of 5-nitrobarbituric acid occurs in the presence of piperidine to give I (R' = NO₂), which is also obtained by the nitration of the product of the addition of barbituric acid to chalcone [1].

EXPERIMENTAL

The IR spectra of chloroform solutions were recorded with an IKS-14A spectrometer.

* This will be the subject of a separate communication.

TABLE 1. 5-(α -Phenyl- β -aryloylethyl)barbituric Acids (I, R' = H)

R	Mp, °C	Empirical formula	N, %		Yield, %
			found	calc.	
H	186-187	C ₁₉ H ₁₆ N ₂ O ₄	8.6	8.3	90
CH ₃	198-199	C ₂₀ H ₁₈ N ₂ O ₄	8.0	8.0	78
C ₂ H ₅	177-178	C ₂₁ H ₂₀ N ₂ O ₄	7.8	7.7	60
n-C ₃ H ₇	105-107	C ₂₂ H ₂₂ N ₂ O ₄	7.6	7.4	51
i-C ₃ H ₇	85-87	C ₂₂ H ₂₂ N ₂ O ₄	7.4	7.4	83
n-C ₄ H ₉	64-65	C ₂₃ H ₂₄ N ₂ O ₄	7.4	7.1	62
t-C ₄ H ₉	193-195	C ₂₃ H ₂₄ N ₂ O ₄	7.3	7.1	80
OCH ₃	98-100	C ₂₀ H ₁₈ N ₂ O ₅	7.4	7.6	96
Br	204-205,5	C ₁₉ H ₁₅ BrN ₂ O ₄	6.7	6.7	81
Cl	203-204	C ₁₉ H ₁₅ ClN ₂ O ₄	7.7	7.6	77
OH	200-201	C ₁₉ H ₁₆ N ₂ O ₅	8.0	7.9	64
NO ₂	199-200	C ₁₉ H ₁₅ N ₂ O ₆	11.2	11.0	76
C ₆ H ₅	198-199	C ₂₅ H ₂₀ N ₂ O ₄	7.1	6.8	42
CH ₂ C ₆ H ₅	187-189	C ₂₆ H ₂₂ N ₂ O ₄	6.7	6.6	58
CH ₂ CH ₂ C ₆ H ₅	191-193	C ₂₇ H ₂₄ N ₂ O ₄	6.4	6.4	60

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5-(α -Phenyl- β -aroylethyl)barbituric Acids (I, R' = H) [2]. A mixture of 0.1 mole of anhydrous barbituric acid and 0.1 mole of p-substituted chalcone was refluxed for 5 h in 70 ml of 75% methanol. The hot solution was filtered, and the precipitate that formed on cooling was crystallized from alcohol. The yields and constants of the compounds obtained are presented in Table 1.

1-Phenyl-5-(α -phenyl- β -benzoylethyl)barbituric acid was similarly obtained from 2.04 g (0.01 mole) of 1-phenylbarbituric acid and 2.08 g (0.01 mole) of chalcone. The yield of product with mp 182-183° (from alcohol) was 3.5 g (85%). Found %: N 7.0. C₂₅H₂₀N₂O₄. Calculated %: N 6.8.

5-Nitro-5-(α -phenyl- β -benzoylethyl)barbituric Acid (I, R' = NO₂). A. A mixture of 15 ml of HNO₃ (sp. gr. 1.52) and 15 ml of glacial acetic acid was added to a solution of 1 g (0.003 mole) of I (R' = H) in 15 ml of glacial acetic acid. The solution was then poured into water, and the resulting precipitate was filtered and washed with water to give 0.75 g (66%) of a product with mp 184-185° (from alcohol). Found %: N 10.9. C₁₉H₁₅N₃O₆. Calculated %: N 11.0.

B. A mixture of 1.73 g (0.01 mole) of chalcone was refluxed for 6-7 h in 20 ml of dioxane containing one to two drops of piperidine. The major portion of the solvent was removed, and the precipitate was washed with water and ether to remove the unchanged starting materials to give 0.72 g (19%) of a product with mp 184-185° (from alcohol). This preparation did not depress the melting point of the compound obtained by method A.

LITERATURE CITED

1. L. P. Zalukaev, *Zh. Obshch. Khim.*, 26, 3125 (1956).
2. M. Camlett, *J. Am. Chem. Soc.*, 77, 4898 (1955).