

LETTERS TO THE EDITOR

Synthesis of 2-(3-Phenoxybenzoyl)cyclohexanone

Yu. V. Popov, T. K. Korchagina, G. V. Kalmykova, and E. A. Shinkarenko

Volgograd State Technical University, ul. Lenina 28, Volgograd, 400005 Russia

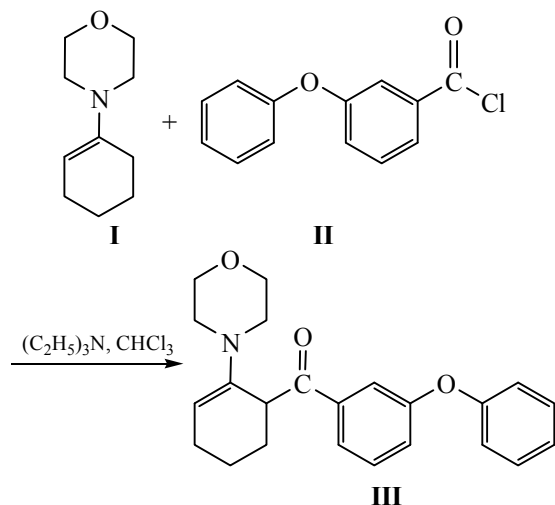
e-mail: shinkarenko.ek@gmail.com

Received February 20, 2012

DOI: 10.1134/S1070363212070262

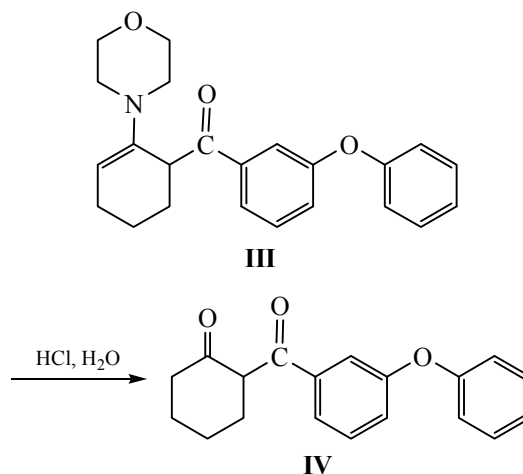
Dicarbonyl compounds and their derivatives are widely used both in industry and in laboratory syntheses. The presence of dicarbonyl groups in these compounds leads to their high reactivity with respect to many types of reagents. The diketones are used as photosensitive materials, monomers for polymers and intermediates in organic synthesis. They are the lanthanide and actinide extractants and also components of some catalysts and luminescent systems. Many dicarbonyl-containing compounds show biological activity of various types. The introducing of diphenyl oxide fragment into the molecule leads to the new aspects of the use of such substances.

To construct the dicarbonyl system we used the acylation of 4-(cyclohexene-1-yl)morpholine by 3-phenoxybenzoyl chloride.



The synthesis was carried out in the presence of triethylamine in chloroform at 35°C using the molar ratio **I:II** = 1:0.5.

The reaction of compound **III** with hydrochloric acid results in a 2-(3-phenoxybenzoyl)cyclohexanone in 70% yield.



The reaction was carried out in the temperature range of 50–60°C for 3–5 h.

The synthesized 2-(3-phenoxybenzoyl)cyclohexanone is an oily substance that slowly crystallizes upon cooling.

The structure of the resulting 1,3-diketone was confirmed by the ¹H NMR spectroscopy.

2-(3-Phenoxybenzoyl)cyclohexanone (IV). A solution of 10.4 g (62.5 mmol) of 4-(cyclohexen-1-yl)morpholine and 6.3 g (4.7 ml, 62.5 mmol) of anhydrous triethylamine in anhydrous chloroform (32 ml) was placed in a round-bottom three-neck flask equipped with a dropping funnel, reflux condenser, and mechanical stirrer. The reaction flask was placed in a glycerol bath heated to 35°C. To a solution in the reaction flask was added dropwise a solution of 7.3 g (31.25 mmol) of 3-phenoxybenzoyl chloride in an-

hydrous chloroform (12.5 ml). The reaction mixture gradually becomes orange, and the precipitation occurs. After the addition of a solution of acid chloride, the reaction mixture was stirred at 35°C for 3 h and diluted with 32 ml of 20% hydrochloric acid. The resulting mixture was refluxed with vigorous stirring for 3–5 h and cooled to room temperature. The aqueous layer was separated. The organic phase was washed with water (6×10 ml). The organic layer was dried with magnesium sulfate, filtered, evaporated and evacuated at 10–50 mm Hg to give oil, which slowly crystallizes upon cooling. Yield 6.4 g (21 mmol, 70%). ^1H NMR spectrum, δ , ppm: 1.81–2.27 m (4H, CH_2), 3.725 m (H, CH), 7.14–7.79 m (9H, $\text{C}_6\text{H}_5\text{OC}_6\text{H}_4$).

The ^1H NMR spectrum was recorded on a Varian Mercury 300VV instrument relative to internal HMDS in CDCl_3 .

REFERENCES

1. *Comprehensive Organic Chemistry*, Barton, D. and Ollis, W.D., Ed., Oxford: Pergamon Press, 1979, vol. 2.
2. Popov, yu.V., Korchagina, T.K., Kalmykova, G.V., and Shinkarenko, E.A., Abstract of Papers, *XIX Mendeleevskii s'yezd po obshchei i prikladnoi khimii* (XIX Mendeleev Conf. on General Applied Chemistry), Volgograd, 2011, vol. 1, p. 339.
3. Hauptmann, Z., Graefe, J., and Remane, H., *Lehrbuch der organischen Chemie* (Handbook of Organic Chemistry), Leipzig: Grundstoffindustrie, 1976.