Russian Journal of General Chemistry, Vol. 73, No. 4, 2003, pp. 661– 662. Translated from Zhurnal Obshchei Khimii, Vol. 73, No. 4, 2003, p. 700.

LETTERS TO THE EDITOR

Three-Component Synthesis of 5-Arylmethylene-3-benzoylmethylenepiperazine-2,6-diones

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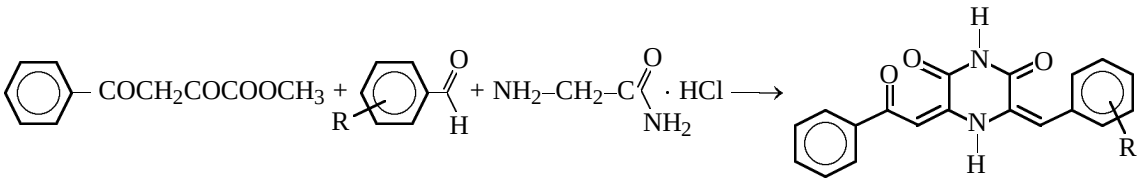
Received April 8, 2002

We have found that heating for a short time of a mixture of glycine amide hydrochloride, aromatic aldehyde, and methyl benzoylpyruvate in aqueous ethanol in the presence of an equivalent amount of sodium carbonate reasults in formation of 5-arylmethylene-3-benzoylmethylenepiperazine-2,6-diones **Ia**–**Ic**.



Ia–**Ic**

R = *p*-Cl (**a**), 3,4-$(CH_3O)_2$ (**b**), *p*-Br (**c**).

Compounds **Ia**–**Ic** are red-orange crystalline substances, which are soluble in ethanol, acetic acid, DMF, and DMSO and insoluble in water.

The IR spectra of **Ia**–**Ic** contain absorption bands belonging to imide carbonyl groups in positions 2 and 6 of the heteroring (1697–1752 cm^{-1}), ketone carbonyl group conjugated with double bond (1620–1664 cm^{-1}), and NH groups (3056–3101 and 3155–3207 cm^{-1}). In the ^1H NMR spectra of **Ia**–**Ic** we observed a multiplet from aromatic protons at δ 6.84–7.63 ppm, signals from olefinic protons at δ 6.90–6.98 and 6.90–7.03 ppm, and signals from NH protons at δ 12.1–12.5 (N^4H) and 13.5–13.7 ppm (N^1H).

3-Benzoylmethylene-5-(4-chlorobenzylidene)-piperazine-2,6-dione (Ia). To a solution of 0.01 mol of glycine amide hydrochloride and 0.01 mol of sodium carbonate in 5 ml of water we added a solution of 0.01 mol of *p*-chlorobenzaldehyde in 5 ml of ethanol. The mixture was heated to 60–70°C, 0.01 mol of methyl benzoylpyruvate was added, and the mixture was heated for 5 min under reflux and was left to stand for 24 h at room temperature. The precipitate was filtered off. Yield 25%, mp 166–168°C (from ethanol). IR spectrum (mineral oil), ν, cm^{-1}: 1664 (C=O, ketone), 1752 (C=O, imide), 3086 (NH), 3190 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 6.90 s (1H, CH=), 7.00 s (1H, COCH=), 7.51–8.07 m (9H, H_{arom}), 12.23 s (1H, NH), 13.35 s (1H, NH).

3-Benzoylmethylene-5-(3,4-dimethoxybenzylidene)piperazine-2,6-dione (Ib) was synthesized in a similar way. Yield 23%, mp 263–265°C. IR spectrum (mineral oil), ν, cm^{-1}: 1625 (C=O, ketone), 1697 (C=O, imide), 3056 (NH), 3155 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 3.88 s (CH_3O), 3.99 s (CH_3O), 6.90 s (1H, CH=), 7.05 s (1H, COCH=), 7.33–8.00 m (8H, H_{arom}), 12.12 s (1H, NH), 13.45 s (1H, NH).

3-Benzoylmethylene-5-(4-bromobenzylidene)-piperazine-2,6-dione (Ic) was synthesized in a similar

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way. Yield 20%, mp 266–268°C. IR spectrum (mineral oil), ν, cm^{-1}: 1620 (C=O, ketone), 1704 (C=O, imide), 3101 (NH), 3207 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 6.90 s (1H, CH=), 6.98 s (1H, COCH=), 7.50–8.00 m (9H, H_{arom}), 12.20 s (1H, NH), 13.33 s (1H, NH).

The IR spectra were taken on Specord and UR-20 instruments, and the ^1H NMR spectra were recorded on a Bruker WM-250 spectrometer (250 MHz).

ACKNOWLEDGMENTS

This study was financially supported by the Russian Foundation for Basic Research (project no. 02-03-96415).