ISSN 1070-4280, Russian Journal of Organic Chemistry, 2012, Vol. 48, No. 2, pp. 296–298. © Pleiades Publishing, Ltd., 2012.
Original Russian Text © V.L. Gein, R.O. Dyrenkov, N.A. Kornienko, M.I. Vakhrin, P.A. Slepukhin, 2012, published in Zhurnal Organicheskoi Khimii, 2012, Vol. 48, No. 2, pp. 303–304.

SHORT COMMUNICATIONS

Formation of *N*,*N*'-Di(5-alkoxycarbonyl-5-methyl-2-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrol-3-yl)-*N*,*N*'-diphenyloxalylamides in the Synthesis of Alkyl 4-Methyl-2,3,6-trioxo-1,5-diphenyl-1,2,3,4,5,6-hexahydropyrrolo[3,4-*b*]pyrrole-4-carboxylates

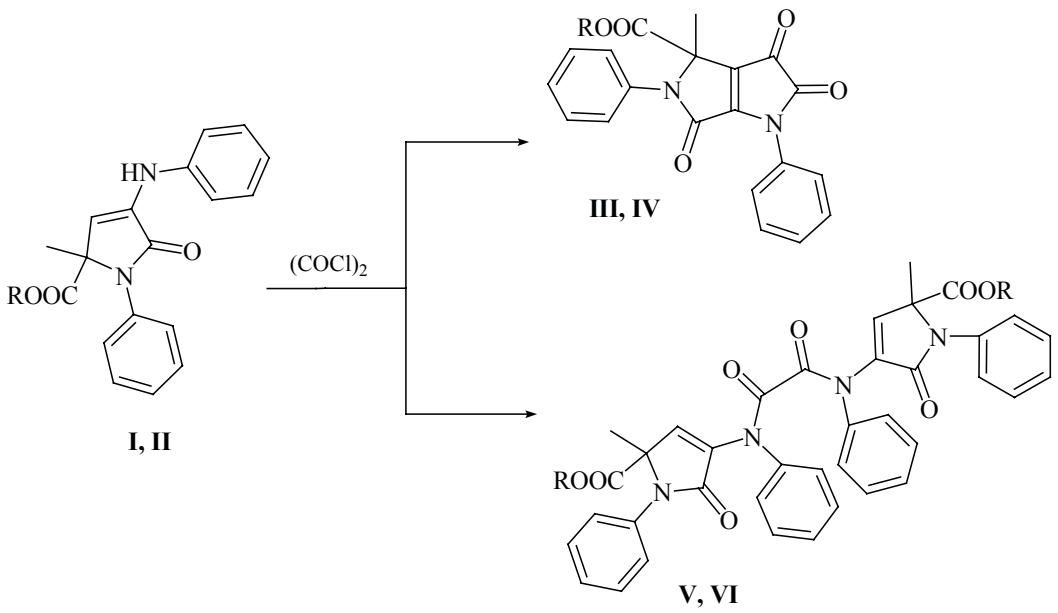
V. L. Gein[a], R. O. Dyrenkov[a], N. A. Kornienko[a], M. I. Vakhrin[a], and P. A. Slepukhin[b]

[a]*Perm State Pharmaceutical Academy of the Federal Agency of Health Protection and Social Development, Perm, 614000 Russia*
e-mail: geinvl48@mail.ru
[b]*Postovsky Institute of Organic Synthesis, Ural Branch, Russian Academy of Sciences, Yekaterinburg, Russia*

Received April 2, 2011

DOI: 10.1134/S1070428012020224

It was established formerly that in the reaction of 5-alkoxycarbonyl-1-aryl-3-arylamino-5-methyl-3-pyrrolin-2-ones with oxalyl chloride alkyl 4-methyl-2,3,6-trioxo-1,5-diphenyl-1,2,3,4,5,6-hexahydropyrrolo[3,4-*b*]pyrrole-4-carboxylates were obtained [1]. In the reaction of pyrrolines **I** and **II** with oxalyl chloride in the ratio 1 : 1 by the procedure [1] at boiling in anhydrous tetrachloromethane over 3–4 h alongside alkyl 4-methyl-2,3,6-trioxo-1,5-diphenyl-1,2,3,4,5,6-hexahydropyrrolo[3,4-*b*]pyrrole-4-carboxylates **III, IV** *N*,*N*'-di(5-alkoxycarbonyl-



5-methyl-2-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrol-3-yl)-*N,N'*-diphenyloxalylamides **V, VI** were isolated for the first time.

The formation of compounds **V, VI** occurred apparently through the acylation of initial enamines **I, II**, in the first stage of the reaction. The formerly unknown compounds **V, VI** synthesized are colorless crystalline substances soluble in DMF, DMSO, and at heating in acetonitrile, ethyl acetate, acetic acid. The IR spectrum of compound **V** contains the absorption bands of the stretching vibrations of the ester carbonyl group (1748 cm^{-1}) and amide carbonyl groups (1672–1712 cm^{-1}).

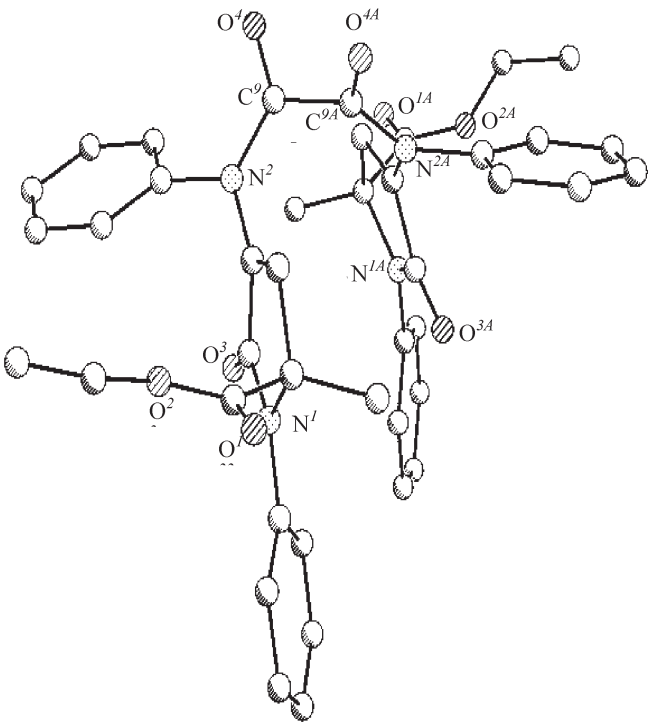
In the ^1H NMR spectra of compounds **V, VI** alongside the signals of the aromatic protons the singlet of three protons of the methyl group attached to the position *5* appears in the region 1.53–1.56 ppm, a singlet of the methoxycarbonyl group in the region 3.65–3.71, triplet and quartet of the ethoxycarbonyl group in the region 1.15 and 4.10 ppm are also present.

In order to establish the spatial arrangement of compounds **V, VI** a single crystal of compound **VI** was obtained by slow crystallization from acetic acid, and it was subjected to XRD analysis (see the figure).

To a solution of 1.0 mmol of compound **I, II** in 30 ml of anhydrous tetrachloromethane was added 1.0 mmol of oxalyl chloride, and the mixture was boiled for 3–4 h. Then the reaction mixture was cooled, the formed precipitate of compound **V, VI** was filtered off, the filtrate was evaporated to isolate compound **III, IV**.

Methyl 4-methyl-2,3,6-trioxo-1,5-diphenyl-1,2,3,4,5,6-hexahydropyrrolo[3,4-*b*]pyrrole-4-carboxylate (III). Yield 2.2 g (51%), mp 191–193°C. IR spectrum, ν, cm^{-1}: 1745 (C=O), 1738 (C^2=O), 1730 (C^3=O), 1705 (C^7=O). ^1H NMR spectrum, δ, ppm: 1.64 s (3H, CH_3), 3.71 s (3H, OCH_3), 7.23–7.36 m (10H, $2C_6H_5$). Found, %: C 70.07; H 4.23; N 7.50. $C_{21}H_{16}N_2O_5$. Calculated, %: C 67.02; H 4.28; N 7.44.

Ethyl 4-methyl-2,3,6-trioxo-1,5-diphenyl-1,2,3,4,5,6-hexahydropyrrolo[3,4-*b*]pyrrole-4-carboxylate (IV). Yield 2.4 g (53%), mp 193–195°C. IR spectrum, ν, cm^{-1}: 1744 (C=O), 1736 (C^2=O), 1728 (C^3=O), 1708 (C^6=O). ^1H NMR spectrum, δ, ppm: 1.15 t (3H, CH_3, *J* 7 Hz), 1.66 s (3H, CH_3), 4.10 q (2H, $\underline{CH_2}CH_3$, *J* 7.5 Hz), 7.09–7.36 m (10H, $2C_6H_5$). Found, %: C 72.59; H 4.75; N 7.15. $C_{22}H_{18}N_2O_5$. Calculated, %: C 69.69; H 4.65; N 7.18.



Structure of the molecule of *N,N'*-di(5-ethoxycarbonyl-5-methyl-2-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrol-3-yl)-*N,N'*-diphenyloxalylamide (**VI**) according to XRD data

***N,N'*-Di(5-methyl-5-methoxycarbonyl-2-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrol-3-yl)-*N,N'*-diphenyloxalylamide (V)**. Yield 1.2 g (31%), mp 275–277°C (acetic acid). IR spectrum, ν, cm^{-1}: 1716 (C=O), 1704 (C=O), 1692 (C^6=O). ^1H NMR spectrum, δ, ppm: 1.53 s (3H, CH_3), 3.65 s (3H, OCH_3), 7.01–7.32 m (10H, $2C_6H_5$). Found, %: C 75.86; H 4.80; N 8.05. $C_{40}H_{34}N_4O_8$. Calculated, %: C 68.76; H 4.90; N 8.02.

***N,N'*-Di(5-ethoxycarbonyl-5-methyl-2-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-yl)-*N,N'*-diphenyloxalylamide (VI).** Yield 1.4 g (34%), mp 247–249°C (acetic acid). IR spectrum, ν, cm^{-1}: 1748 (C=O), 1712 (C=O), 1672 (C^6=O). ^1H NMR spectrum, δ, ppm: 1.15 t (3H, CH_3, *J* 7 Hz), 1.56 s (3H, CH_3), 4.10 q (2H, $\underline{CH_2}CH_3$, *J* 7.5 Hz), 7.19–7.32 m (10H, $2C_6H_5$). Found, %: C 73.52; H 5.25; N 7.60. $C_{42}H_{38}N_4O_8$. Calculated, %: C 69.41; H 5.27; N 7.71.

IR spectra were recorded on a spectrophotometer Specord M-80 from mulls in mineral oil. ^1H NMR spectra were registered on spectrometers Bruker DRX 300 and DRX 500 in DMSO-d_6, internal reference TMS. XRD experiment was carried out on an automatic four-circle

diffractometer Xcalibur S along a common procedure [MoK_α-radiation, 150(2) K, ω/2θ-scanning]. The structure was solved and refined applying SHELXTL software [2]. The results of XRD analysis are deposited in the Cambridge Crystallographic Data Center (CCDC 843691) and are available at the address www.ccdc.cam.ac.uk/data_request/cif.

REFERENCES

1. Gein, V.L., Shumilovskikh, E.V., Voronina, E.V., Gein, L.F., Khokhryakova, N.P., Tendryakova, S.P., Vyaznikova, N.G., and Andreichikov, Yu.S., *Zh. Org. Khim*., 1998, vol. 68, p. 1328.
2. Sheldrick, G.M., *Acta Cryst*., 2008, vol. A64, p. 112.