Journal of Structural Chemistry, Vol. 42, No. 5, pp. 848-853, 2001

SYNTHESIS AND CRYSTAL AND MOLECULAR STRUCTURE OF 3,5-DIHYDROXY-1-DIPHENYLMETHYLENEAMINO-5-METHOXYCARBONYL-4-*p*-TOLUOYL-2,5-DIHYDRO-2-PYRROLONE

Z. G. Aliev, O. P. Krasnykh, N. A. Konyukhova,
A. N. Maslivets, and L. O. Atovmyan

UDC 547.745

The reaction between methyl 2-diphenylmethylenehydrazino-4-oxo-4-p-tolyl-2-butenic ether and oxalyl chloride gave 1-diphenylmethyleneamino-5-methoxycarbonyl-4-p-toluoyl-2,3-dihydro-2,3-pyrroledione, which adds water to yield substituted 3,5-dihydroxy-2,5-dihydro-2-pyrrolone. The crystal and molecular structure of pyrrolone and the inter- and intramolecular hydrogen bonds in its crystals and solutions were investigated by IR spectroscopy and XRD analysis. The crystals of $C_{27}H_{22}N_2O_6$ *are triclinic with cell dimensions* $a = 9.8540(10)$, $b = 10.0880(10)$, $c = 13.982(2)$ Å, $\alpha = 69.820(10)$, $\beta = 110.57(10)$, $\gamma = 91.350(10)°$, $V = 1213.8(2)$ Å^3, $M = 470.47$, $d_{calc} = 1.287$ g/cm^3, $Z = 2$, *space group* $P\bar{1}$. *The data were collected on a KM-4 (KUMA DIFFRACTION) diffractometer, 3781 reflections with* $I \geq 2\sigma(I)$. *Direct methods, hydrogen atoms localized, least-squares refinement (in an anisotropic approximation for non-hydrogen atoms),* $R = 0.0454$. *The crystal is built from the centrosymmetric dimer associates of molecules bonded by strong (2.64 Å) intermolecular hydrogen bonds (IMHB) of O–H...O type. The dimer associates are linked by weaker (2.82 Å) centrosymmetric IMHB, forming an infinite chain of hydrogen-bonded molecules.*

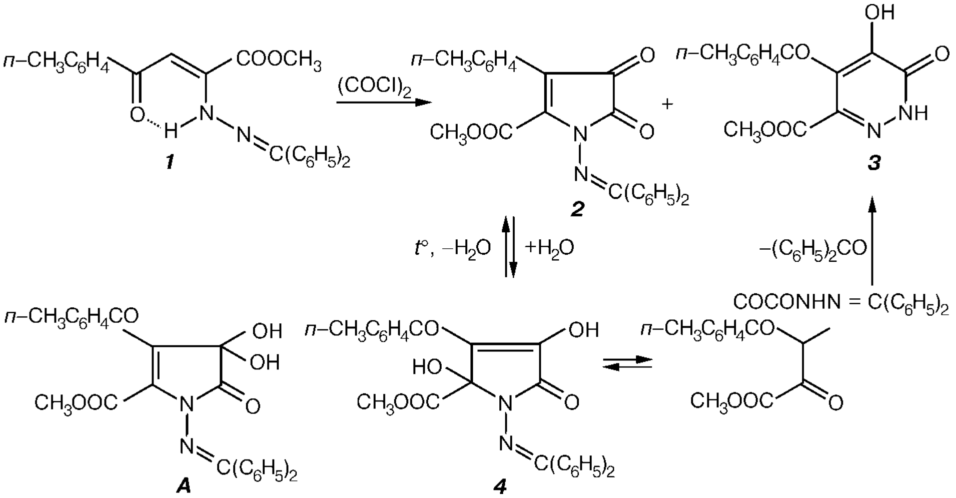
In the structure of substituted 4-acyl-2,3-dihydro-2,3-pyrrolediones, there are several electron-deficient atoms which are potential objects of a nucleophilic attack. These compounds react readily with water, alcohols, and amines, forming adducts as well as de- and recyclization products of various types [1]. For adducts of 4-acyl-2,3-dihydro-2,3-pyrrolediones with water (hydrated forms), gem-diol structure of A type was suggested [2]. Systematic studies of this class of compounds revealed that water is added to pyrrolediones at the carbon atom in the 5 position of heterocycle but not at the carbonyl group, forming substituted 3,5-dihydroxy-4-acyl-2,5-dihydro-2-pyrrolones [3, 4].

A physicochemical study of the hydrated forms of pyrrolediones revealed some problems associated with the fine structure of these molecules. Thus spectral data of pyrrolones indicated that they contain strong intra- and/or intermolecular hydrogen bonds [4]. Based on IR spectral analysis it was suggested that solutions of substituted 1-aryl-4-aroyl-3,5-dihydroxy-2,5-dihydro-2-pyrrolones contain H-chelate type intramolecular hydrogen bonds involving the enol hydroxy group and the ketone aroyl group by analogy with the previously studied 1,5-diaryl-4-aroyl-3-hydroxy-2,5-dihydro-2-pyrrolones [5], and it was not ruled out that intermolecular hydrogen bonds exist in the crystals.

In continuation of our studies on the chemical properties of 4-acyl-2,3-dihydro-2,3-pyrrolediones [6], we synthesized new pyrroledione (**2**) and its hydrate form (**4**) using the known synthetic procedure [1]. The aim of this study is to obtain more reliable data on the type of hydrogen bond in 3,5-dihydroxy-2,5-dihydropyrrolone crystals. The structure of compound **4** was investigated by the XRD method.

Institute of Chemical Physics Problems, Russian Academy of Sciences, Chernogolovka. Perm State University. Translated from *Zhurnal Strukturnoi Khimii*, Vol. 42, No. 5, pp. 1008-1015, September-October, 2001. Original article submitted September 29, 2000.

Methyl 2-diphenylmethylenehydrazino-4-oxo-4-*p*-tolyl-2-butenic ether **1** [7] interacts with oxalyl chloride forming 1-diphenylmethyleneamino-5-methoxycarbonyl-4-*p*-toluoyl-2,3-dihydro-2,3-pyrroledione **2** and 5-hydroxy-3-methoxycarbonyl-4-*p*-toluoyl-1,6-dihydro-6-*p*yridazinone **3** as a minor product.



Compound **2** is a dark red crystalline substance; this is the first representative of the class of 4,5-diacyl-2,3-dihydro-2,3-pyrrolediones containing a diarylmethyleneamine substituent at the nitrogen atom. Its IR spectrum recorded in vaseline oil contains a high-frequency band of the $C^2{=}O$ group at 1750 cm^{-1} typical of this class of compounds. The positions of other absorption bands are also in good agreement with the literature data [3].

Pyridazinone **3** is evidently produced by recyclization involving water, as described previously [8]. This process may be excluded by using freshly distilled oxalyl chloride and absolute chloroform.

As is known, introduction of electron-accepting substituents in the 4 and 5 positions of 2,3-dihydro-2,3-pyrrolediones significantly facilitates the interaction of these compounds with nucleophilic reagents [1, 6]. After short-term storage in contact with air moisture, the red crystals of compound **2** acquired a light-yellow tarnish, and the colored solutions are decolorized as a result of water addition to pyrroledione **2** forming its hydrated form — 2,5-dihydroxy-1-diphenylmethyleneamino-5-methoxycarbonyl-4-*p*-toluoyl-2,5-dihydro-2-pyrrolone **4**. Addition of water is irreversible, as usually takes place in reactions of this type [6].

Pyrrolone **4** is a crystalline substance giving a positive reaction (cherry-red coloring) when tested for the presence of a enol hydroxyl group with an alcohol solution of iron(III) chloride. The positions of the IR absorption bands of crystals **4** in vaseline oil (Table 1) agree with the frequency ranges in the spectra of 2-pyrrolones with close structures [3, 4].

The general view of the molecule is shown in Fig. 1. The bond lengths and angles are presented in Table 1. The chief distinction of this compound from the previously studied pyrroles [9, 10] lies in pyramidality of the nitrogen atom in the cycle.

The sum of bond angles at the N(7) atom is 354.9°, and the height of the pyramid is 0.26 Å. The five-membered ring is nonplanar; the folding angle along C(9)···C(12) is 5.1°, and the N(8) atom lies in the equatorial position. The diphenylmethyleneamine group has an orientation characterized by the torsion angles C(9)N(7)N(8)C(23) –65.2° and N(7)N(8)C(23)C(24) 169.3°. The angle between the planes of the phenyl rings equals 65.1°. The methoxycarbonyl fragment is planar and has a bisecting orientation relative to the heterocycle. The C(15)=O(3) group of the toluoyl fragment is *trans* relative to the C(10)=C(11) bond of the heterocycle but is not coplanar with the latter; the C(10)C(11)C(15)O(3) torsion angle equals –147.0°. The toluoyl fragment is also nonplanar. The O(3)C(15)C(16)C(17) torsion angle is –145.2°. The configuration of the molecule in general as well as the interatomic distances N(8)=C(23) 1.292 Å and C(10)=C(11) 1.344 Å and the distances in the carbonyl groups indicate that the molecule has localized double bonds not markedly involved in conjugations.

There are no intramolecular hydrogen bonds in the molecule. In the solid state, the molecules exist as centrosymmetric dimer associates formed by means of intermolecular hydrogen bonds O(2)–H(2)···O(1). The dimers are stable, as indicated by the parameters of this hydrogen bond: *d*(O···O) 2.62 Å, *d*(O···H) 1.76 Å; the angle at the H(2) atom is 159.6°. It is interesting to note that the structural analog of this compound, 1-benzyl-3-hydroxy-4-ethoxycarbonyl-2,5-dihydro-2-pyrrolone

TABLE 1. Bond Lengths *d* (Å) and Angles ω (deg) in Molecule **4**

Bond	*d*	Bond	*d*
O(1)–C(9)	1.221(2)	O(2)–C(10)	1.323(2)
O(3)–C(15)	1.227(2)	O(4)–C(12)	1.386(1)
O(5)–C(13)	1.197(2)	O(6)–C(13)	1.319(2)
O(6)–C(14)	1.451(2)	N(7)–C(9)	1.359(2)
N(7)–N(8)	1.406(2)	N(7)–C(12)	1.472(2)
N(8)–C(23)	1.292(2)	C(9)–C(10)	1.488(2)
C(10)–C(11)	1.344(2)	C(11)–C(15)	1.468(2)
C(11)–C(12)	1.524(2)	C(12)–C(13)	1.547(2)
C(15)–C(16)	1.487(2)	C(16)–C(21)	1.384(2)
C(16)–C(17)	1.388(2)	C(17)–C(18)	1.381(3)
C(18)–C(19)	1.378(3)	C(19)–C(20)	1.389(4)
C(19)–C(22)	1.518(4)	C(20)–C(21)	1.387(3)
C(23)–C(24)	1.484(2)	C(23)–C(30)	1.486(2)
C(24)–C(25)	1.391(2)	C(24)–C(29)	1.393(2)
C(25)–C(26)	1.394(3)	C(26)–C(27)	1.373(4)
C(27)–C(28)	1.372(3)	C(28)–C(29)	1.380(2)
C(30)–C(31)	1.388(2)	C(30)–C(35)	1.389(2)
C(31)–C(32)	1.387(3)	C(32)–C(33)	1.366(4)
C(33)–C(34)	1.371(4)	C(34)–C(35)	1.400(3)
Angle	ω	Angle	ω
C(13)–O(6)–C(14)	115.9(2)	C(9)–N(7)–N(8)	121.8(1)
C(9)–N(7)–C(12)	111.4(1)	N(8)–N(7)–C(12)	116.7(1)
C(23)–N(8)–N(7)	115.4(1)	O(1)–C(9)–N(7)	127.0(1)
O(1)–C(9)–C(10)	126.7(1)	N(7)–C(9)–C(10)	106.3(1)
O(2)–C(10)–C(11)	129.1(1)	O(2)–C(10)–C(9)	121.2(1)
C(11)–C(10)–C(9)	109.6(1)	C(10)–C(11)–C(15)	128.9(1)
C(10)–C(11)–C(12)	109.0(1)	C(15)–C(11)–C(12)	121.5(1)
O(4)–C(12)–N(7)	109.5(1)	O(4)–C(12)–C(11)	114.1(1)
N(7)–C(12)–C(11)	101.9(1)	O(4)–C(12)–C(13)	109.8(1)
N(7)–C(12)–C(13)	107.9(1)	C(11)–C(12)–C(13)	113.1(1)
O(5)–C(13)–O(6)	126.9(1)	O(5)–C(13)–C(12)	122.0(1)
O(6)–C(13)–C(12)	111.1(1)	O(3)–C(15)–C(11)	118.9(1)
O(3)–C(15)–C(16)	122.1(1)	C(11)–C(15)–C(16)	119.0(1)
C(21)–C(16)–C(17)	118.8(2)	C(21)–C(16)–C(15)	119.8(2)
C(17)–C(16)–C(15)	121.4(1)	C(18)–C(17)–C(16)	120.6(2)
C(19)–C(18)–C(17)	121.0(2)	C(18)–C(19)–C(20)	118.2(2)
C(18)–C(19)–C(22)	120.3(3)	C(20)–C(19)–C(22)	121.5(3)
C(21)–C(20)–C(19)	121.1(2)	C(16)–C(21)–C(20)	120.1(2)
N(8)–C(23)–C(24)	115.6(1)	N(8)–C(23)–C(30)	125.5(1)
C(24)–C(23)–C(30)	118.9(1)	C(25)–C(24)–C(29)	118.5(2)
C(25)–C(24)–C(23)	121.8(2)	C(29)–C(24)–C(23)	119.7(1)
C(24)–C(25)–C(26)	119.9(2)	C(27)–C(26)–C(25)	120.5(2)
C(28)–C(27)–C(26)	119.9(2)	C(27)–C(28)–C(29)	120.1(2)
C(28)–C(29)–C(24)	120.9(2)	C(31)–C(30)–C(35)	119.1(2)
C(31)–C(30)–C(23)	118.9(1)	C(35)–C(30)–C(23)	121.9(2)
C(32)–C(31)–C(30)	121.0(2)	C(33)–C(32)–C(31)	119.8(2)
C(32)–C(33)–C(34)	120.2(2)	C(33)–C(34)–C(35)	120.8(2)
C(30)–C(35)–C(34)	119.1(2)		

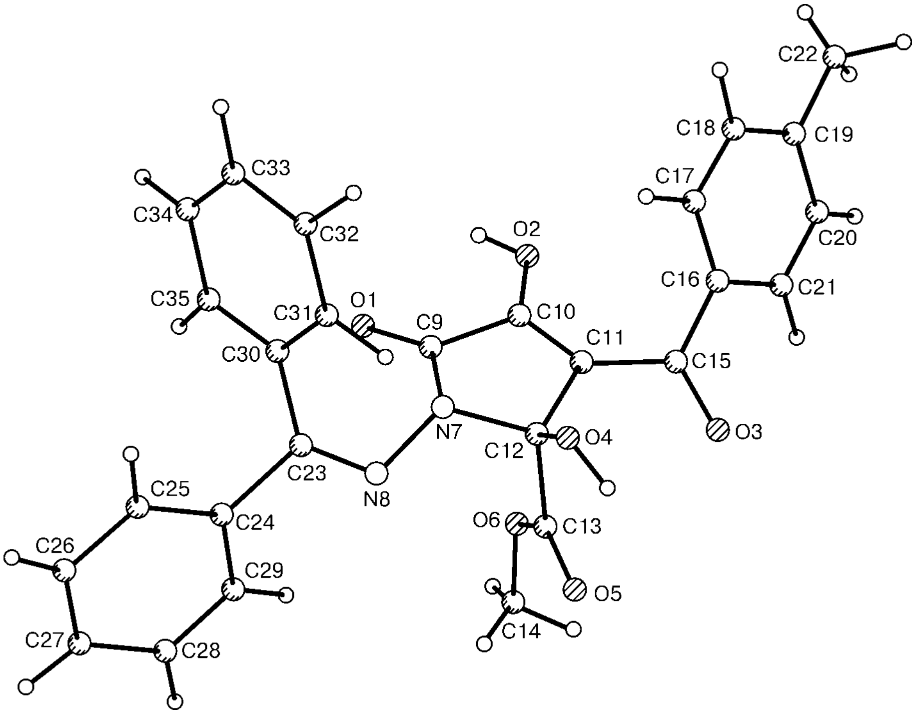


Fig. 1. Structure of molecule **4**.

[10], is also built from dimer associates with an H-bond 2.64 Å in length. The neighboring dimer associates of molecules **4** related by a symmetry center also form H-bonds O(4)–H(4)···O(3). The parameters of this bond (*d*(O···O) 2.82 Å, *d*(O···H) 1.98 Å, angle at H(2) = 149.4°) indicate that it is weaker than the H-bond in the dimer, but yet strong enough. Thus molecules **4** in crystal form centrosymmetric dimer associates connected by hydrogen bonds into an infinite chain in the *c* direction of the crystal (Fig. 2). The O(5) atom of the methoxycarbonyl fragment is not involved in H-bonding, as reflected in the bond length C(13)=O(5) 1.197 Å (this is the shortest bond). All other bonds in the molecule have typical values for the respective atoms and demand no comments.

The IR spectral data of 2-pyrrolone in chloroform (Table 2) indicate that an intramolecular hydrogen bond (IHB) of H-chelate type is formed between C^3–OH and the carbonyl group of the toluoyl substituent in solution. The intensities of the absorption bands at 1665 and 1620 cm^{-1} corresponding to the vibrations of C^2=O and C^4C=O involved in the IMHB decrease relative to the spectra of crystals, whereas the intensity of the absorption band at 1600 cm^{-1} increases considerably

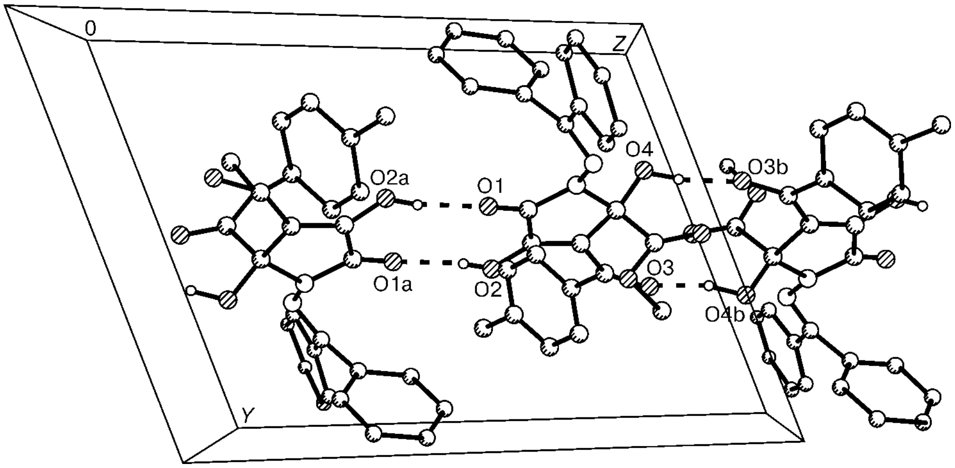


Fig. 2. System of hydrogen bonds in the crystal.

TABLE 2. IR Spectra of Compound **4**

Experimental conditions	OH	$COOCH_3$	$C^2 = O$		ArCO		C = C C = N
			free	in IMHB	in IMHB	in IHB	
Vaseline oil	3395, 3150 (br)	1760	1700	1665	1620		1600
5% chloroform solution	3485, 3430	1725-1735 (br)	1717	1665 (w)	1620 (w)	1600	
1% chloroform solution	3485, 3430	1745 (sh), 1730	1715, 1700 (sh)	1665(v.w.)	1625(w)	1600	

because the C=C, C=N stretching band is superimposed by the intense vibration band of the $C^4C=O$ group involved in the IHB, which is much stronger than the IMHB involving the same group.

A comparison of the spectra of the 5% and 1% solutions shows that in a dilute solution there are less associates due to the formation of IMHB involving $C^2=O$ (the absorption band at 1665 cm^{-1} vanishes), and the absorption bands of the $COOCH_3$ (1745 (sh), 1730 cm^{-1}) and $C^2=O$ groups (1715, 1700(sh) cm^{-1}) are more distinctly resolved into two bands. The latter may be due to the existence of two relatively stable conformers, where the bulky diphenylmethyleneamine substituent is rotated around the N–N bond.

The PMR spectrum of pyrrolone **4** in DMSO-d_6 is typical for this class of compounds: the C^5–OH signal lies in the region of aromatic proton signals, and the C^4–OH signal is very broadened and is not observed at all. This is further support to the fact that the intramolecular hydrogen bonds are strong in this compound.

EXPERIMENTAL

The IR spectra were recorded on a UR-20 instrument. The ^1H NMR spectra were measured on an RYa-2310 (60 MHz, HMDC as internal standard) and Bruker AM-300 spectrometers. The UV spectra were taken on a Specord M40 spectrometer. The purity of the compounds was confirmed by TLC (Silufol UV-254 plates).

1-Diphenylmethyleneamino-5-methoxycarbonyl-4-*p*-toluoyl-2,3-dihydro-2,3-pyrroledione (2), 5-hydroxy-3-methoxycarbonyl-4-*p*-toluoyl-1,6-dihydro-6-pyridazinone (3), and 3,5-dihydroxy-1-diphenylmethyleneamino-5-methoxycarbonyl-4-*p*-toluoyl-2,5-dihydro-2-pyrrolone (4).

Oxalyl chloride (0.94 g, 0.011 mol) was dropped to a solution of methyl 2-diphenylmethylenehydrazino-4-oxo-4-*p*-tolyl-2-butenic ether (4.00 g, 0.010 mol) in absolute benzene (30 ml). The mixture was boiled on a reflux condenser for 2 h 15 min and cooled. Absolute hexane (15 ml) was added, and the precipitate of pyrroledione **2** was filtered off; yield 1.35 g (30%), T_{dec} = 145-146°C (from a 2:1 benzene–hexane mixture). Found, %: C 70.01, H 4.68, N 6.08. Calculated for $C_{27}H_{20}N_2O_5$, %: C 71.67, H 4.46, N 6.19. IR spectrum (vaseline oil): ν/cm^{-1}: 1750 (C^2=O), 1730 ($COOCH_3$, C^3=O), 1630 (C^4C=O). ^1H NMR spectrum ($CDCl_3$, 60 MHz, δ, ppm): 2.29, 3.80 (both s, 3H, CH_3, OCH_3); 7.35 (m, 14H, Ar).

The mother solutions were consolidated, and the mixture was allowed to stay for 24 h for contact with air moisture. The yellow precipitate of the hydrate form **4** was filtered off and recrystallized from a 3:1 chloroform–hexane mixture. Yield 1.86 g (39%), T_m = 164-167°C (with decomp.). Found, %: C 69.08, H 4.56, N 6.04. Calculated for $C_{27}H_{22}N_2O_6$, %: C 68.93, H 4.71, N 5.95. UV spectrum (MeCN), λ_{max}/nm (log ε): 256 (4.26), 405 (3.98). ^1H NMR spectrum (DMSO-d_6, 300 MHz, δ, ppm): 2.42, 3.87 (both s, 3H, CH_3, OCH_3); 7.51 (m, 15H, Ar, C^5–OH).

The filtrate was evaporated, and the residue recrystallized from acetone. Yield of pyridazinone **3** is 0.10 g (3%), T_m = 269-270°C (with decomp.).

X-ray diffraction study of compound 4. The crystals $C_{27}H_{22}N_2O_6$ are triclinic with cell dimensions a = 9.8540(10), b = 10.0880(10), c = 13.982(2) Å, α = 69.820(10), β = 110.57(10), γ = 91.350(10)°, V = 1213.8(2) $Å^3$, M = 470.47, d_{calc} = 1.287 g/cm^3, Z = 2, space group $P\bar{1}$. The data were collected on a KM-4 (KUMA DIFFRACTION) automatic four-circle diffractometer with χ-geometry (θ-2θ scan mode, monochromated CuK_α radiation, $3.6 < 2\theta < 80.3°$). The total number of data collected is 4510. No absorption correction was applied (μ = 0.760 mm^{-1}). The structure was solved by direct methods with subsequent electron density syntheses.

The hydrogen atoms were objectively located on difference electron density maps. The full-matrix least-squares refinement was carried out anisotropically (isotropically for hydrogens). In the final cycles the 3781 data with $I \geq 2\sigma(I)$

TABLE 3. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Thermal Parameters ($U_{eq}\times 10^3$) for **4**

Atom	x	y	z	U_{eq}	Atom	x	y	z	U_{eq}
O(1)	1363(1)	4262(1)	6140(1)	53(1)	C(30)	2412(2)	1285(2)	7640(1)	42(1)
O(2)	–1282(1)	5967(2)	5690(1)	6591)	C(31)	1462(2)	585(2)	8129(2)	58(1)
O(3)	–734(1)	6451(1)	8682(1)	54(1)	C(32)	545(2)	–301(2)	7702(2)	77(1)
O(4)	825(1)	3277(1)	9399(1)	42(1)	C(33)	544(2)	–462(2)	6771(2)	79(1)
O(5)	2785(1)	4992(1)	9986(1)	55(1)	C(34)	1447(3)	248(3)	6259(2)	76(1)
O(6)	2551(1)	6202(1)	8239(1)	51(1)	C(35)	2407(2)	1118(2)	6693(2)	56(1)
N(7)	1634(1)	3687(1)	7950(1)	36(1)	H(2)	–1090(30)	5790(20)	5160(20)	78(7)
N(8)	3120(1)	3158(1)	8429(1)	38(1)	H(4)	1100(30)	3520(20)	10020(20)	80(7)
C(9)	959(2)	4347(2)	6850(1)	40(1)	H(14A)	4540(40)	6380(40)	9130(30)	134(12)
C(10)	–342(2)	5244(2)	6684(1)	42(1)	H(14B)	3790(30)	7610(30)	7950(20)	92(8)
C(11)	–304(2)	5240(2)	7655(1)	38(1)	H(14C)	3500(30)	7570(30)	9030(30)	117(10)
C(12)	1075(1)	4336(1)	8553(1)	35(1)	H(17)	–3140(20)	4700(20)	6792(16)	60(5)
C(13)	2241(2)	5228(2)	9024(1)	40(1)	H(18)	–5630(30)	5360(30)	5660(20)	87(7)
C(14)	3735(2)	7013(3)	8595(2)	72(1)	H(20)	–5340(30)	9180(30)	6010(20)	100(8)
C(15)	–1252(2)	6094(2)	7884(1)	40(1)	H(21)	–2920(30)	8480(30)	7230(20)	81(7)
C(16)	–2796(2)	6530(2)	7126(1)	45(1)	H(22A)	–7370(50)	8890(50)	4590(40)	172(18)
C(17)	–3598(2)	5638(2)	6630(2)	54(1)	H(22B)	–8100(80)	7840(70)	5330(60)	270(30)
C(18)	–5041(2)	6049(3)	5946(2)	70(1)	H(22C)	–7580(60)	7290(60)	4530(50)	220(30)
C(19)	–5709(2)	7368(3)	5711(2)	80(1)	H(25)	4950(30)	–250(20)	8509(19)	73(6)
C(20)	–4904(3)	8261(3)	6206(2)	85(1)	H(26)	7280(40)	–800(40)	8950(30)	125(11)
C(21)	–3465(2)	7844(2)	6915(2)	65(1)	H(27)	9080(30)	870(30)	9220(20)	86(7)
C(22)	–7283(4)	7819(6)	4931(5)	139(2)	H(28)	8190(30)	3140(30)	9160(20)	104(9)
C(23)	3461(2)	2125(1)	8180(1)	37(1)	H(29)	5670(20)	3640(20)	8638(18)	72(6)
C(24)	5047(2)	1738(2)	8513(1)	41(1)	H(31)	1450(20)	710(20)	8847(19)	73(6)
C(25)	5597(2)	420(2)	8612(2)	63(1)	H(32)	–240(30)	–810(30)	8110(30)	116(10)
C(26)	7085(2)	129(3)	8870(2)	78(1)	H(33)	–150(30)	–1150(30)	6510(20)	94(8)
C(27)	8021(2)	1122(3)	9052(2)	71(1)	H(34)	1460(30)	180(30)	5590(20)	94(8)
C(28)	7496(2)	2402(2)	9000(2)	67(1)	H(35)	3110(20)	1600(20)	6398(17)	60(5)
C(29)	6021(2)	2716(2)	8723(2)	53(1)					

yielded $R1 = 0.0454$, $wR2 = 0.1270$; $R1 = 0.0533$, $wR2 = 0.1343$ (all data). The atomic coordinates are given in Table 3. All calculations were carried out on PC/AT using the SHELX-97 program complex [11].

This work was supported by RFFR grant No. 98-03-32888a.

REFERENCES

1. Yu. S. Andreichikov (ed.), *The Chemistry of Five-Membered 2,3-Dioxoheterocycles* [in Russian], Perm State University, Perm (1994).
2. W. Ott, G. Kollenz, and E. Ziegler, *Synthesis*, 546 (1976).
3. T. Sano, Y. Horiguchi, and Y. Tsuda, *Chem. Pharm. Bull.*, **33**, 110 (1985).
4. Yu. S. Andreichikov, A. N. Maslivets, L. I. Smirnova, et al., *Zh. Org. Khim.*, **23**, 1534 (1987).
5. Yu. S. Andreichikov, V. L. Gein, and I. N. Anikina, *ibid.*, **22**, 1749 (1986).
6. A. N. Maslivets, Chemical Sciences Doctoral Dissertation, Saratov State University, Saratov (1996), p. 303.
7. USSR Patent No. 1467956 (1988), *Byull. Izobr.*, 323 (1999).
8. O. P. Krasnykh, A. N. Maslivets, and Yu. S. Andreichikov, *Zh. Org. Khim.*, **30**, 1433 (1994).
9. Z. G. Aliev, S. N. Shurov, E. Yu. Pavlova, et al., *Izv. Ross. Akad. Nauk, Ser. Khim.*, 1552 (1995).
10. B. L. Mylari, T. A. Beyer, and T. W. Siegel, *J. Med. Chem.*, **34**, 1011 (1991).
11. G. M. Sheldrick, *SHELX-97, Programs for Crystal Structure Analysis*, University of Göttingen, Germany.