

STRUCTURE OF ORGANIC
COMPOUNDS

X-Ray Mapping in Heterocyclic Design:
18. X-Ray Diffraction Study of a Series of Derivatives
of 3-Cyanopyridine-2-one with Annelated Heptane
and Octane Cycles

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Received January 13, 2016

Abstract—Seven new, previously unknown, bicyclic and tricyclic heterocycles based on derivatives of 3-cyanopyrid-2-ones are obtained: 2-oxo-2,5,6,7,8,9-hexahydro-1*H*-cyclohepta[*b*]pyridine-3-carbonitrile, C₁₁H₁₂N₂O (**1a**); 2-[2-(4-chlorophenyl)-2-oxoethoxy]-6,7,8,9-tetrahydro-5*H*-cyclohepta[*b*]pyridine-3-carbonitrile, C₁₉H₁₇ClN₂O₂ (**2a**); (3-amino-6,7,8,9-tetrahydro-5*H*-cyclohepta[*b*]furo[3,2-*e*]pyridin-2-yl)(4-chlorophenyl)methanone, C₁₉H₁₇ClN₂O₂ (**3**); 2-oxo-1,2,5,6,7,8,9,10-octahydrocycloocta[*b*]pyridine-3-carboxamide, C₁₂H₁₆N₂O₂ (**4**); 2-[2-(4-chlorophenyl)-2-oxoethoxy]-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine-3-carboxamide, C₂₀H₂₁ClN₂O₃ (**5a**); 1-[2-(4-chlorophenyl)-2-oxoethyl]-2-oxo-1,2,5,6,7,8,9,10-octahydrocycloocta[*b*]pyridine-3-carboxamide, C₂₀H₂₁ClN₂O₃ (**5b**); and 2-[2-(4-chlorophenyl)-2-oxoethoxy]-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine-3-carbonitrile, C₂₀H₁₉ClN₂O₂, (**6**). All compounds are characterized by ¹H NMR spectroscopy, and their crystal structures are determined by X-ray diffraction.

DOI: 10.1134/S1063774517020249

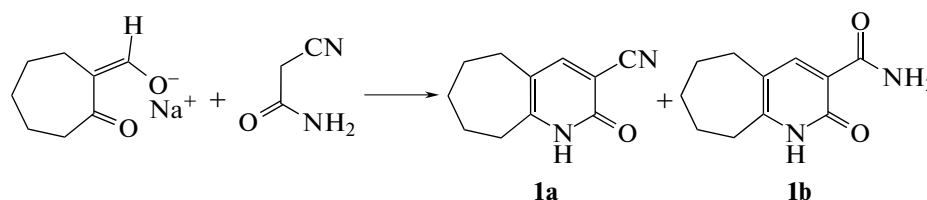
INTRODUCTION

Earlier [1–3] synthetic pathways to obtain pyridine-2-ones, their 3-cyano derivatives containing cyclopentane and cyclohexane cycles annelated at the C5–C6 bond of pyridone, and products of their *N*-/*O*-phenacylation and subsequent heterocyclizations were considered and the structures of these compounds were determined. In this study, the range

of objects is extended to annelated cycles of larger size, namely, cycloheptane and cyclohexane.

EXPERIMENTAL

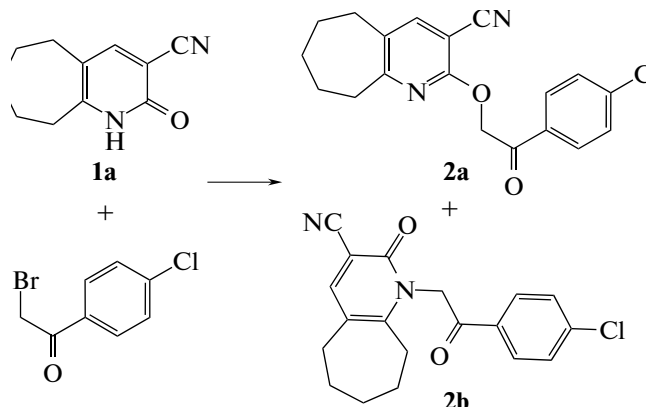
Transformations of the cycloheptane derivative. Synthesis of 2-oxo-2,5,6,7,8,9-hexahydro-1*H*-cyclohepta[*b*]pyridine-3-carbonitrile (**1a**) was performed by the procedure described in [4]:



A mixture of compounds **1a** and **1b** was obtained. Individual compound **1a** (48%) was isolated chro-

matographically (SiO₂; chloroform as an eluent); the **1a** : **1b** ratio was 4:1.

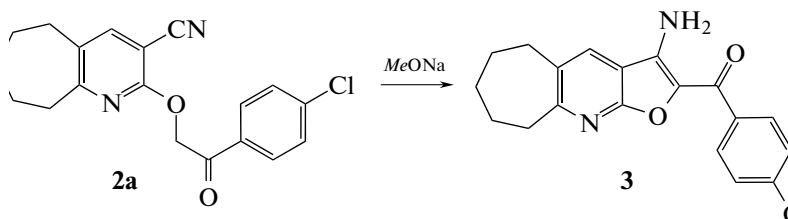
Phenacylation of **1a** was performed by the procedure given in [5]:



The *O*-isomer 2-[2-(4-chlorophenyl)-2-oxoethoxy]-6,7,8,9-tetrahydro-5*H*-cyclohepta[*b*]pyridine-3-carbonitrile (**2a**) and the *N*-isomer 1-[2-(4-chlorophenyl)-2-oxoethyl]-2-oxo-2,5,6,7,8,9-hexahydro-1*H*-cyclohepta[*b*]pyridine-3-carbonitrile (**2b**) were obtained in 76% and 21% yields, respectively.

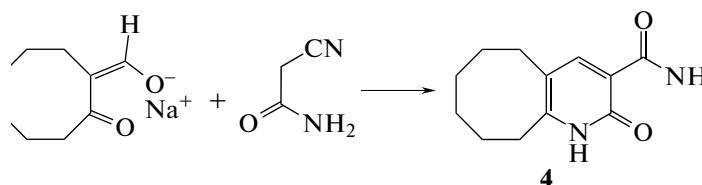
The isomers were separated using column chromatography (SiO₂; chloroform as an eluent).

(3-Amino-6,7,8,9-tetrahydro-5*H*-cyclohepta[*b*]furo[3,2-*e*]pyridin-2-yl)(4-chlorophenyl)methanone (**3**) was synthesized in 72% yield by the procedure given in [5].

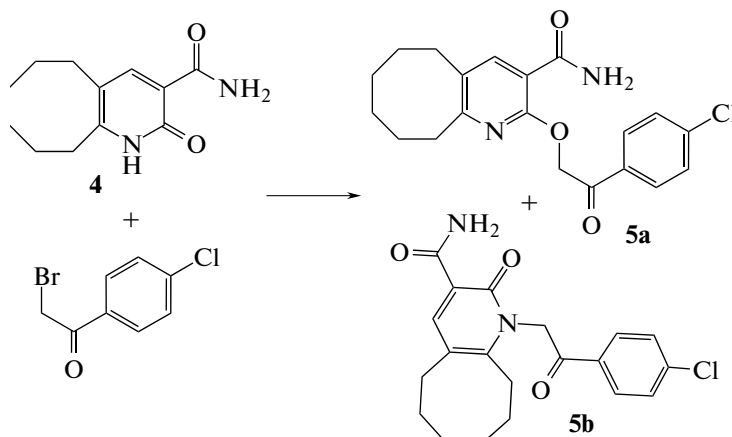


Transformations of the cyclooctane derivative. 2-Oxo-1,2,5,6,7,8,9,10-octahydrocycloocta[*b*]pyri-

dine-3-carboxamide (**4**) was synthesized in 53% yield by the procedure described in [4].



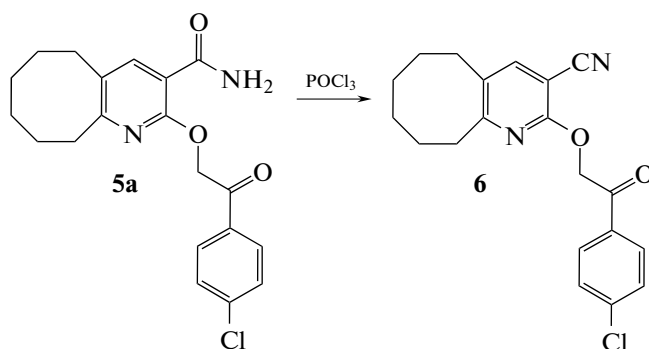
Phenacylation of **4** was performed according to the procedure [5].



The *O*-isomer 2-[2-(4-chlorophenyl)-2-oxoethoxy]-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine-3-carboxamide (**5a**) and the *N*-isomer 1-[2-(4-chlorophenyl)-2-oxoethyl]-2-oxo-1,2,5,6,7,8,9,10-octahydrocycloocta[*b*]pyridine-3-carboxamide (**5b**) were obtained in 87% and 7% yields, respectively. The isomers were separated using column chromatogra-

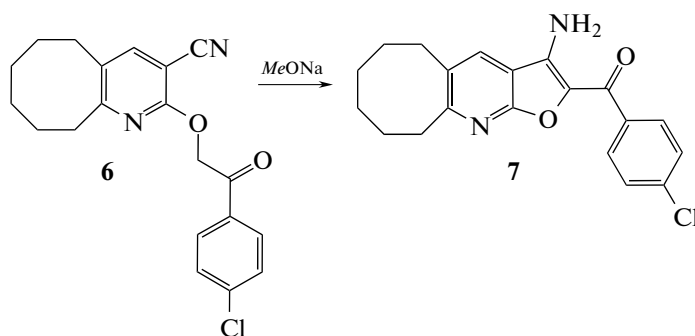
phy (SiO₂; 1 : 1 chloroform–hexane mixture as an eluent).

Synthesis of 2-[2-(4-chlorophenyl)-2-oxoethoxy]-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine-3-carbonitrile (**6**) (with the cyano group instead of the amide group) was performed by an original procedure on heating of **5a** with POCl₃.



Triethylamine (1 ml) and POCl₃ (1 ml) were added to a solution of 0.37 g **5a** in 150 ml of absolute dichloromethane. The mixture was refluxed for 15 h on an apparatus equipped with calcium chloride drying tube. The reaction mass was cooled and washed with an aqueous solution of soda. The organic layer

was separated, dried with anhydrous MgSO₄, and evaporated. (3-Amino-5,6,7,8,9,10-hexahydrocycloocta [b]furo[3,2-*e*]pyridin-2-yl)(4-chlorophenyl)methanone (**7**) was obtained in 65% yield by the reaction of pyridine **6** with sodium methylate according to the procedure in [5].



Physicochemical characteristics of the compounds prepared are given in Table 1.

X-ray diffraction studies. The crystal structures of compounds **1a**, **2a**, **3**, **4**, **5a**, **5b**, and **6** were determined. Attempts to grow X-ray-quality single crystals of **7** failed.

Crystal data, details of data collection, and parameters of structure refinement are summarized in Table 2. The crystal structures were determined by the direct method followed by the refinement of the positional and anisotropic displacement parameters of all non-hydrogen atoms. The positions of hydrogen atoms, with the exception of those involved in hydrogen bonds, were determined based on geometrical

considerations and rigidly bound to the positions of corresponding carbon atoms in the refinement. The thermal parameters of these H atoms were calculated from those of parent atoms as $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The H atoms involved in hydrogen bonds were localized on difference syntheses of electron density and refined independently in the isotropic approximation.

The crystallographic data on the structures studied have been deposited at the Cambridge Crystallographic Data Center, CCDC nos 1445799 (**1a**), 1445802 (**2a**), 1445803 (**3**), 1445840 (**4**), 1445841 (**5a**), 1445804 (**5b**), and 1445843 (**6**).

Table 1. Physicochemical characteristics of the compounds obtained

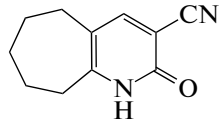
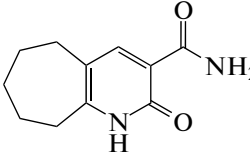
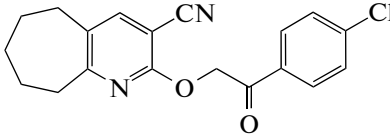
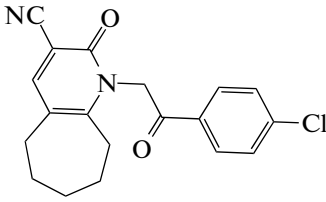
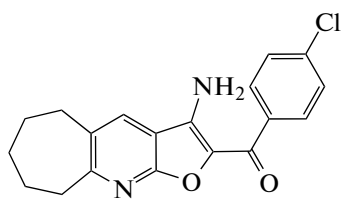
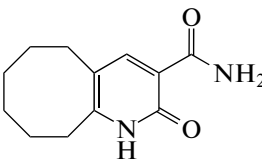
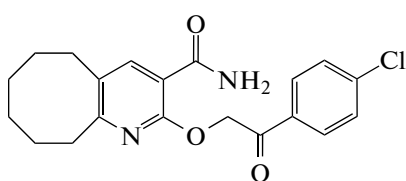
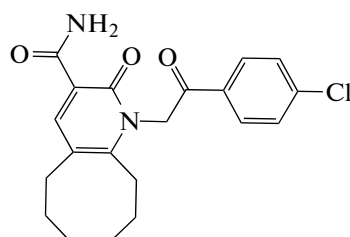
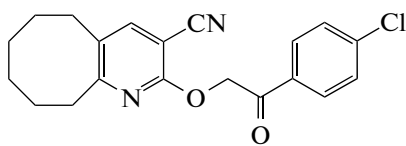
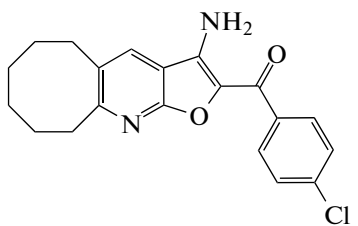
	Compound	T_m , °C	^1H NMR (DMSO- d_6 ; δ ppm)
1a		255	12.42 (s, 1H, 1-NH); 7.94 (s, 1H, 4-CH); 2.74 (m, 2H, 9-CH ₂); 2.55 (m, 2H, 5-CH ₂); 1.74, 1.57, and 1.49 (m, 2H+2H+2H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂)*
1b		342	12.25 (s, 1H, 1-NH); 9.08 (s, 1H, NH in CONH ₂); 7.35 (s, 1H, NH in CONH ₂); 8.09 (s, 1H, 4-CH); 2.76 (m, 2H, 9-CH ₂); 2.61 (m, 2H, 5-CH ₂); 1.76, 1.57, and 1.51 (m, 2H+2H+2H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂)*
2a		151	8.01 and 7.64 (m, 4H, Ar); 8.01 (s, 1H, 4-CH); 5.81 (s, 2H, O-CH ₂); 2.72 (m, 4H, 5-CH ₂ + 9-CH ₂); 1.74 and 1.53 (m, 2H, 4H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂)*
2b		264	8.10 and 7.69 (m, 4H, Ar); 8.06 (s, 1H, 4-CH); 5.85 (s, 2H, 1-CH ₂); 2.82 (m, 2H, 9-CH ₂); 2.69 (m, 2H, 5-CH ₂); 1.74 and 1.54 (m, 2H+4H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂)*
3		270	8.17 (s, 1H, 4-CH); 8.11 and 7.63 (m, 4H, Ar); 7.54 (s, 2H, -NH ₂); 3.03 (m, 2H, 9-CH ₂); 2.86 (m, 2H, 5-CH ₂); 1.84 and 1.64 (m, 2H, 4H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂)*
4		302	12.23 (s, 1H, 1-NH); 9.10 (s, 1H, NH in CONH ₂); 7.39 (s, 1H, NH in CONH ₂); 8.09 (s, 1H, 4-CH); 2.69 (m, 2H, 10-CH ₂); 2.56 (m, 2H, 5-CH ₂); 1.65, 1.56, and 1.35 (m, 2H, 2H, 4H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂ , 9-CH ₂)*
5a		209	8.02 and 7.63 (m, 4H, Ar); 8.00 (s, 1H, 4-CH); 7.87 (s, 1H, NH in CONH ₂); 7.71 (s, 1H, NH in CONH ₂); 5.77 (s, 2H, O-CH ₂); 2.69 (q, 4H, 5-CH ₂ + 10-CH ₂); 1.59, 1.53, and 1.26 (m, 2H, 2H, 4H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂ , 9-CH ₂)*
5b		276	8.83 (s, 1H, NH in CONH ₂); 7.43 (s, 1H, NH in CONH ₂); 8.21 (s, 1H, 4-CH); 8.12 and 7.68 (m, 4H, Ar); 5.68 (s, 2H, 1-CH ₂); 2.87 (m, 2H, 10-CH ₂); 2.70 (m, 2H, 5-CH ₂); 1.61, 1.46, and 1.32 (m, 4H, 2H, 2H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂ , 9-CH ₂)*

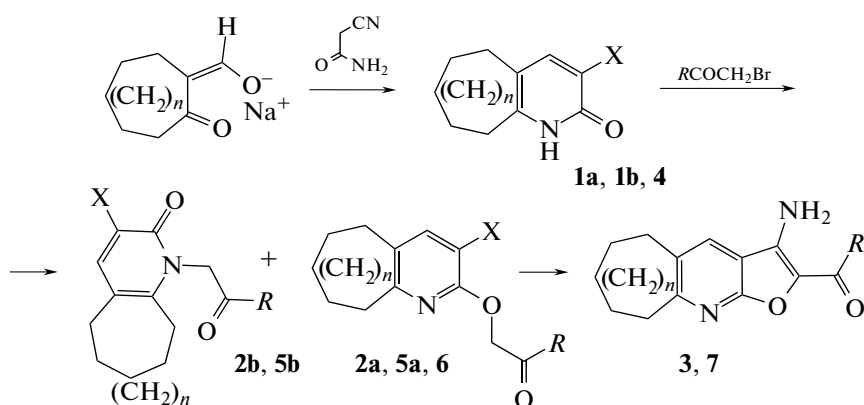
Table 1. (Contd.)

Compound	$T_m, ^\circ\text{C}$	$^1\text{H NMR}$ (DMSO- d_6 ; δ ppm)
	145	8.01 and 7.64 (m, 4H, Ar); 7.99 (s, 1H, 4-CH); 5.79 (s, 2H, O-CH ₂); 2.69 and 2.65 (m, 4H, 5-CH ₂ , 10-CH ₂)*; 1.59, 1.49, and 1.25 (m, 2H, 2H, 4H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂ , 9-CH ₂)*
	266	8.19 (s, 1H, 4-CH); 8.13 and 7.63 (m, 4H, Ar); 7.55 (s, 2H, -NH ₂); 2.98 (m, 2H, 10-CH ₂); 2.87 (m, 2H, 5-CH ₂); 1.71 and 1.33 (m, 8H, 6-CH ₂ , 7-CH ₂ , 8-CH ₂ , 9-CH ₂)*

* Inverse assignment of the signals of CH₂ groups is possible.

RESULTS AND DISCUSSION

The transformations described above are shown on the general scheme:



In the course of cyclization of pyridones, the nitrile group is partially (seven-membered cycle, **1a** + **1b**) or completely (eight-membered cycle, **4**) saponified to the amide group. At the second stage, on phenacylation of pyridones **1a** and **4**, the yields are close to quantitative, namely, 97% for the seven-membered cycle (**2a** + **2b**) and 94% for the eight-membered cycle (**5a** + **5b**). In both cases, the *O*-isomer (**2a**, **5a**) is the dominant product owing to the steric effect of the ring.

Compound **2a** with the seven-membered cycle contains the CH acidic nucleophilic group at the oxygen atom and the electrophilic center of the cyano group. As a result, under the effect of a base compound **2a** easily undergoes cyclization to form tricyclic furopyridone **3**. An analogous compound **5a** contains a less electrophilic amide group; nonetheless, it was

converted to nitrile (compound **6**). Under the effect of a base, this compound was also converted to homologous furopyridine **7**. The possibility of cyclization of *N*-isomers **2b** and **5b** to indolizine was not studied in this work [4]. Consider specific structural features of the compounds prepared.

Crystal structures. Molecular structures of the compounds studied with the atomic numbering are shown in Figs. 1–7. Selected bond lengths and angles are given in Tables 3–9.

Compound 1a. Unlike the “rigid” pyridine heterocycle, the conformationally flexible seven-membered fragment has a chair conformation: the planar C6/C5/C11/C10 (1) fragment forms an angle of 53.80(7)° with the C6/C7/C9/C10 (2) plane, and the dihedral angle between plane 2 and C7/C8/C9 (3) is

Table 2. Crystal data, details of data collection, and parameters of structure refinement for the compounds studied

Empirical formula, compound	$C_{11}H_{12}N_2O$, 1a	$C_{19}H_{17}ClN_2O_2$, 2a	$C_{19}H_{17}ClN_2O_2$, 3	$C_{12}H_{16}N_2O_2$, 4	$C_{20}H_{21}ClN_2O_3$, 5a	$C_{20}H_{21}ClN_2O_3$, 5b	$C_{20}H_{19}ClN_2O_2$, 6
Crystal system, space group, Z	Monoclinic, $P2_1/n$, 4	Triclinic, $P\bar{1}$, 2	Triclinic, $P\bar{1}$, 2	Triclinic, $P\bar{1}$, 4	Triclinic, $P\bar{1}$, 2	Triclinic, $P\bar{1}$, 2	Monoclinic, $P2_1/c$, 4
a , b , c , Å	9.9847(12), 5.6732(17), 16.853(3)	12.7258(11), 5.1648(12), 26.607(3)	7.0314(7), 10.747(2), 12.3055(12)	9.321(2), 10.031(2), 12.614(4)	7.741(4), 8.566(3), 14.763(3)	7.4562(17), 10.8253(12), 12.9343(13)	11.961(8), 10.830(7), 14.499(8)
α , β , γ , deg	90.00, 93.220(10), 90.00	90.00, 103.0700(10), 90.00	115.067(12), 102.383(9), 93.350(13)	98.82(2), 105.53(2), 95.709(18)	79.00(2), 80.53(2), 72.03(2)	67.358(9), 76.077(9), 71.933(9)	90.00, 105.92(3), 90.00
V , Å ³	953.1(4)	1703.5(5)	810.9(2)	1110.6(5)	908.2(6)	907.2(2)	1806.1(19)
D_x , g/cm ³	1.312	1.329	1.396	1.317	1.363	1.365	1.305
Radiation; λ , Å			CuK α : 1.5418				MoK α : 0.7107
μ , cm ⁻¹	0.691	2.093	2.198	0.736	2.050	2.052	0.227
T , K	295(2)	295(2)	295(2)	295(2)	295(2)	295(2)	295(2)
Crystal size, mm	$0.30 \times 0.30 \times 0.30$	$0.20 \times 0.20 \times 0.20$	$0.30 \times 0.30 \times 0.30$	$0.20 \times 0.20 \times 0.20$	$0.20 \times 0.20 \times 0.20$	$0.20 \times 0.20 \times 0.20$	$0.30 \times 0.30 \times 0.30$
Diffractometer			Enraf–Nonius CAD-4				
Scan mode			$\omega/2\theta$				
Absorption correction;	None	DIFABS,	DIFABS,	DIFABS,	DIFABS,	DIFABS,	DIFABS,
T_{\min} , T_{\max}		0.2150, 0.6810	0.209, 0.676	0.1488, 0.9290	0.072, 0.518	0.187, 0.657	0.128, 0.598
θ_{\max} , deg	74.84	74.93	74.88	54.96	69.93	69.92	25.97
Limiting indices	$-12 \leq h \leq 12$, $0 \leq k \leq 7$, $0 \leq l \leq 21$	$-15 \leq h \leq 15$, $0 \leq k \leq 6$, $0 \leq l \leq 33$	$-8 \leq h \leq 8$, $-13 \leq k \leq 12$, $0 \leq l \leq 15$	$-9 \leq h \leq 9$, $-10 \leq k \leq 10$, $0 \leq l \leq 13$	$-8 \leq h \leq 9$, $-10 \leq k \leq 10$, $0 \leq l \leq 17$	$-8 \leq h \leq 9$, $-12 \leq k \leq 13$, $0 \leq l \leq 15$	$-14 \leq h \leq 14$, $0 \leq k \leq 13$, $0 \leq l \leq 17744$
Number of reflections: measured/unique (N_1), $R_{\text{int}}/\text{with } I > 2\sigma(I)$ (N_2)	2004/1939, 0.0457/1728	3474/3406, 0.0476/2073	3386/3227, 0.0524/2901	2767/2767, 0.0000/761	3542/3399, 0.0790/1436	3499/3332, 0.1288/1253	3692/3535, 0.0482/2051
Refinement method		Full-matrix least-squares on F^2					
Number of parameters	132	218	225	289	243	235	227
Extinction correction, coefficient	SHELXL, 0.0051(10)	SHELXL, 0.00028(8)	—	—	—	—	SHELXL, 0.0015(4)
R_1/wR_2 for N_1	0.0500/0.1240	0.0814/0.0614	0.0525/0.1322	0.2226/0.0919	0.1437/0.1064	0.1638/0.1036	0.0827/0.0816
R_1/wR_2 for N_2	0.0453/0.1195	0.0384/0.0507	0.0466/0.1248	0.0452/0.0518	0.0499/0.0767	0.0480/0.0651	0.0354/0.0660
S	1.043	0.946	1.037	0.677	0.864	0.737	0.958
$\Delta\rho_{\min}/\Delta\rho_{\max}$, e/Å ³	-0.197/0.243	-0.160/0.108	-0.305/0.244	-0.139/0.162	-0.140/0.157	-0.161/0.151	-0.120/0.094
Programs			CAD-4 [6], WinGX [7], SHELX97 [8], ORTEP-3 [7]				

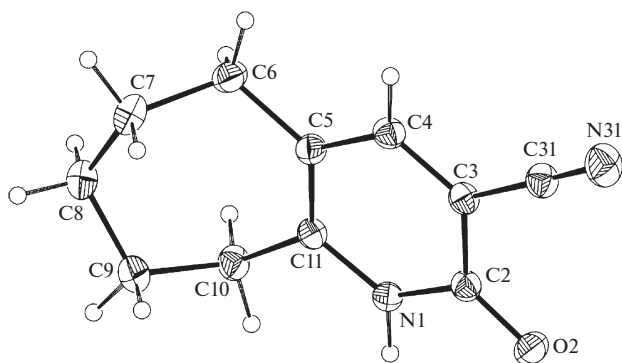


Fig. 1. Molecular structure of **1a** with atomic numbering. Hereinafter, atomic displacements are shown at the 30% probability level and the hydrogen atoms are shown with circles of an arbitrary radius.

$55.19(12)^\circ$. Planar fragments 1 and 3 are nearly parallel: the angle between them is only $1.42(11)^\circ$. The six-membered heterocycle retains the pseudodiene $O2=C2-C3=C4-C5=C11$ structure (Fig. 1, Table 3).

In crystal molecules form centrosymmetric dimers owing to the classical $N1-H1\cdots O2^i(-x+1, -y, -z+1)$ hydrogen bond ($N1-H1 = 0.931(19)$, $N1\cdots O2^i = 2.795(2)$, and $H1\cdots O2^i = 1.865(19)$ Å; angle $N1-H1\cdots O2^i = 177(2)^\circ$). In addition, there are nonclassical hydrogen bonds in the structure with C atoms of the six-membered heterocycle (C4) and annelated seven-membered cycle (C6–C10) acting as hydrogen donors. The N31, O2, and N1 atoms act as acceptors. The donor–acceptor contacts involving the N31 nitrogen atom of the nitrile group (4 contacts) fall in the range $3.418(2)$ – $3.643(2)$ Å, analogous contacts involving the O2 atom (3 contacts) are $3.560(2)$ – $3.695(2)$ Å, and the only contact involving N1 is $3.446(2)$ Å.

Compound 2a. This compound is the product of *O*-phenacylation of compound **1a**. As in molecule **1a**, the seven-membered fragment has the chair conformation: the planar C6/C5/C11/C10 (1) fragment forms a dihedral angle of $53.87(9)^\circ$ with the C6/C7/C9/C10 (2) plane, and the dihedral angle between planes 2 and C7/C8/C9 (3) is $55.51(13)^\circ$. Planar fragments 1 and 3 are almost parallel: the angle between them is only $1.66(12)^\circ$. In consequence of the formation reaction, the six-membered heterocycle adopts the aromatic character and the bonds in the C2–C3–C4–C5–C11 chain become equal (Fig. 2, Table 4).

In the crystal structure of this compound, there are no classical hydrogen bonds, but nonclassical hydrogen bonds are found. The N1, N31, O12, O14, and C118 atoms act as acceptors. The contacts can be divided into two ranges, shorter and longer than 3 Å. Contacts $C13\cdots N1 = 2.689(2)$ and $C20\cdots O14 = 2.819(2)$ Å belong to the former range. The remaining $C\cdots N$ and $C\cdots O$ contacts fall in the range $3.200(2)$ –

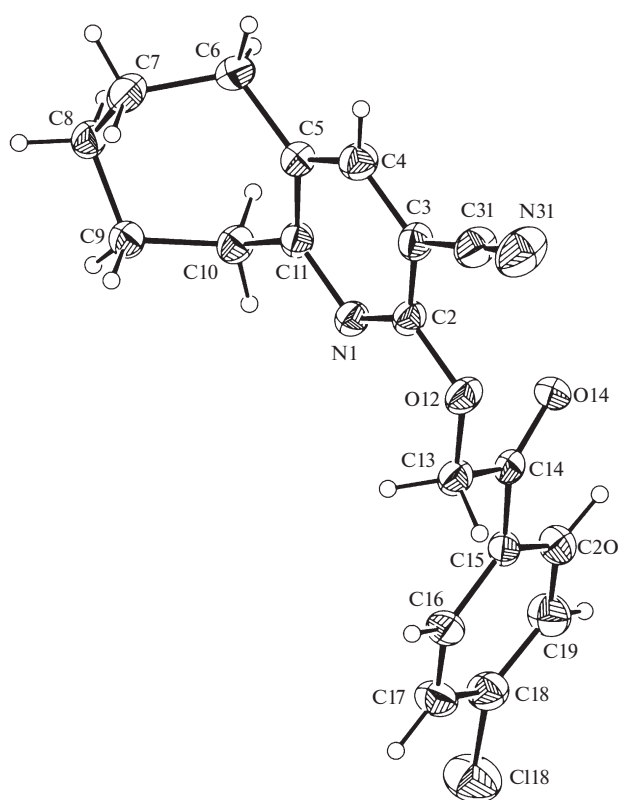


Fig. 2. Molecular structure of **2a** with atomic numbering.

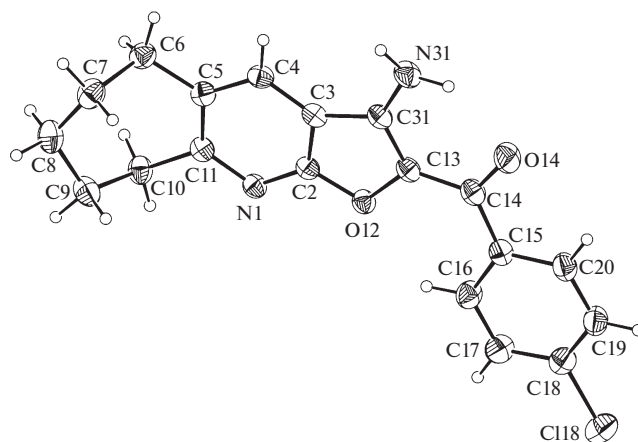


Fig. 3. Molecular structure of **3** with atomic numbering.

$3.590(2)$ Å, and contact $C7\cdots C118 = 3.702(2)$ Å. Most likely, these contacts should be considered van der Waals interactions.

Compound 3. This compound is the product of cyclization of **2a**. The tendency to the retention of the chair conformation of the seven-membered cycle is also observed in this molecule: the planar C6/C5/C11/C10 (1) fragment forms a dihedral angle of $56.27(1)^\circ$ with the C6/C7/C9/C10 (2) plane, and

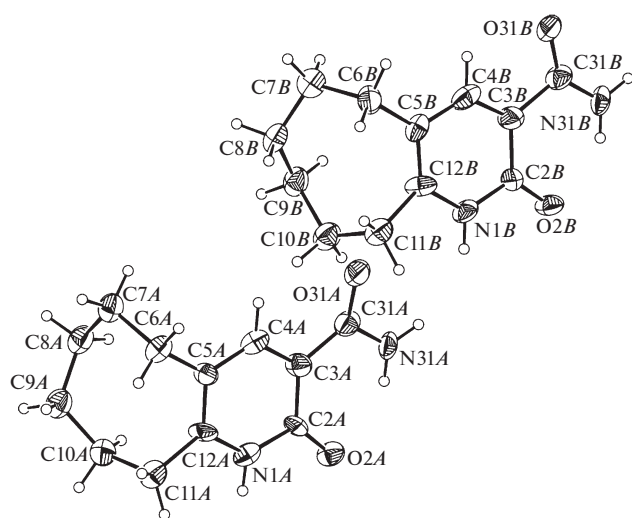


Fig. 4. Molecular structure of **4** with atomic numbering.

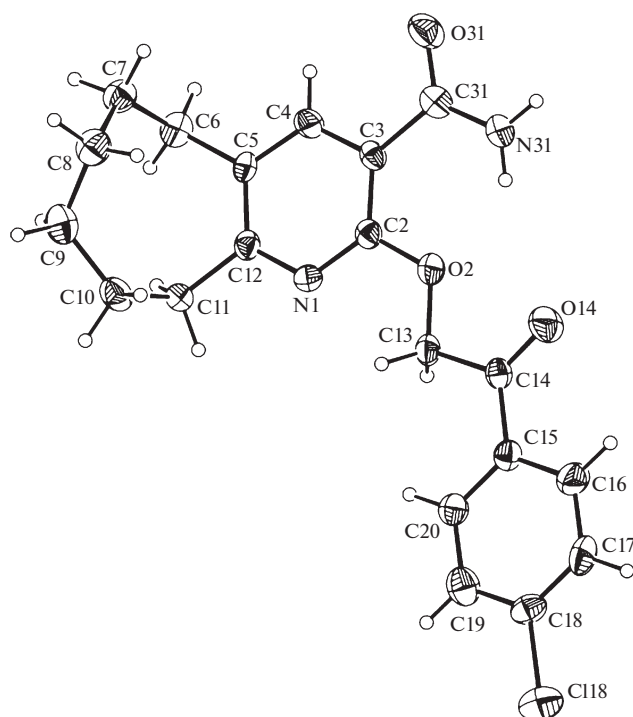


Fig. 5. Molecular structure of **5a** with atomic numbering.

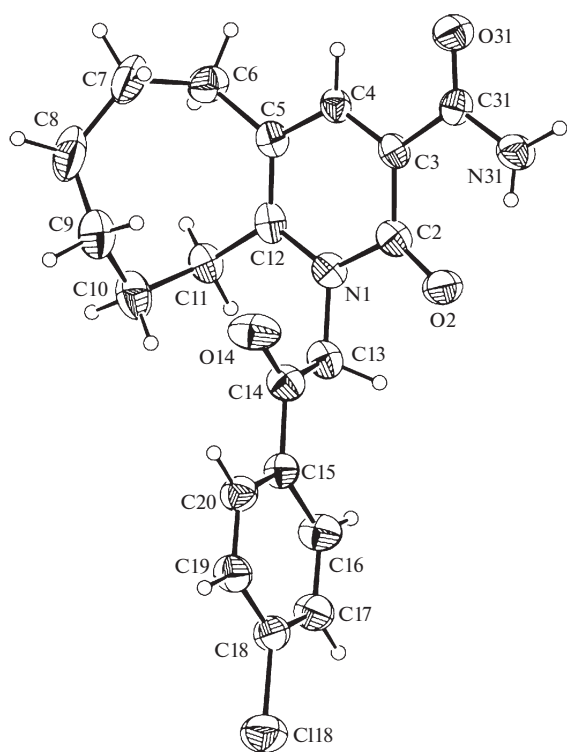


Fig. 6. Molecular structure of **5b** with atomic numbering.

the dihedral angle between planes 2 and C7/C8/C9 (3) is $54.91(1)^\circ$; the angle between fragments 1 and 3 is $1.77(1)^\circ$. As in molecule **2a**, the six-membered heterocycle retains the aromatic character; bond lengths in the C2–C3–C4–C5–C11 chain are equal within the error (Fig. 3, Table 5).

Two classical hydrogen bonds are found in crystal **3**. The intramolecular hydrogen bond is formed by the

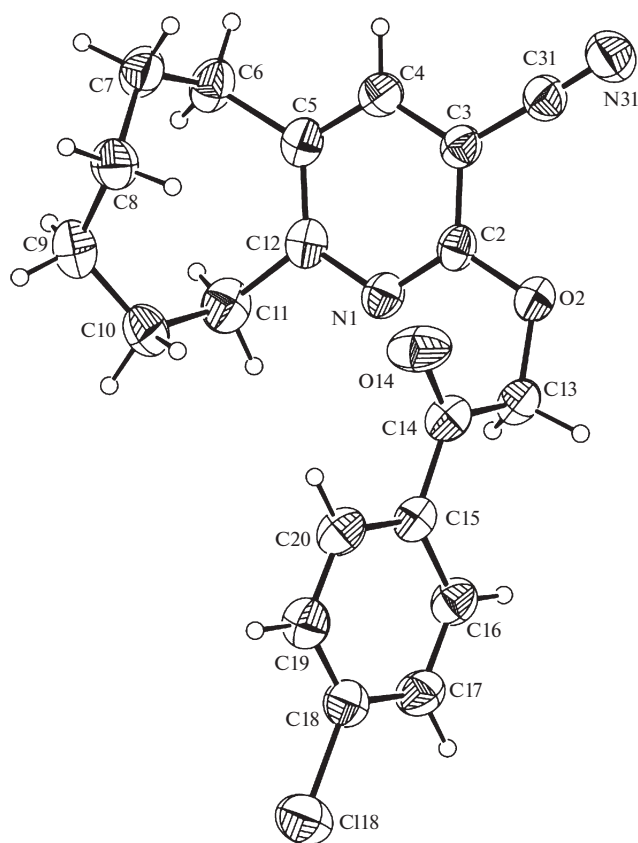


Fig. 7. Molecular structure of **6** with atomic numbering.

Table 3. Selected bond lengths d (Å) and angles ω (deg) in structure **1a**

Bond	d	Bond	d
N1–C11	1.3625(17)	N1–C2	1.3799(16)
N1–H1	0.93(2)	C2–O2	1.2472(16)
C2–C3	1.4326(18)	C3–C4	1.3775(19)
C3–C31	1.4386(18)	C31–N31	1.1377(19)
C4–C5	1.4030(18)	C5–C11	1.3777(18)
C5–C6	1.5112(18)	C6–C7	1.531(2)
C7–C8	1.517(2)	C8–C9	1.517(2)
C9–C10	1.536(2)	C10–C11	1.4998(17)
Angle	ω	Angle	ω
C11–N1–C2	126.08(11)	C11–N1–H1	121.0(11)
C2–N1–H1	112.9(11)	O2–C2–N1	120.48(12)
O2–C2–C3	125.80(12)	N1–C2–C3	113.72(11)
C4–C3–C2	120.99(12)	C4–C3–C31	120.88(12)
C2–C3–C31	118.14(12)	N31–C31–C3	179.11(17)
C3–C4–C5	122.14(12)	C11–C5–C4	117.22(12)
C11–C5–C6	121.57(12)	C4–C5–C6	121.21(12)
C5–C6–C7	115.03(12)	C8–C7–C6	114.35(12)
C9–C8–C7	115.90(13)	C8–C9–C10	115.35(13)
C11–C10–C9	113.89(12)	N1–C11–C5	119.84(12)
N1–C11–C10	116.19(11)	C5–C11–C10	23.97(12)

Таблица 4. Selected bond lengths d (Å) and angles ω (deg) in structure **2a**

Bond	d	Bond	d
N1–C2	1.319(2)	N1–C11	1.359(2)
C2–O12	1.3567(19)	C2–C3	1.394(2)
C3–C4	1.399(2)	C3–C31	1.426(2)
C31–N31	1.140(2)	C4–C5	1.382(2)
9C5–C11	1.393(2)	C5–C6	1.512(2)
C6–C7	1.532(2)	C7–C8	1.507(2)
C8–C9	1.517(2)	C9–C10	1.533(2)
C10–C11	1.504(2)	O12–C13	1.4237(18)
C13–C14	1.515(2)	C14–O14	1.212(2)
C14–C15	1.489(2)	C18–C118	1.7373(18)
Angle	ω	Angle	ω
C2–N1–C11	118.14(15)	N1–C2–O12	120.43(16)
N1–C2–C3	123.86(17)	O12–C2–C3	115.66(16)
C2–C3–C4	116.86(17)	C2–C3–C31	121.15(18)
C4–C3–C31	121.95(17)	N31–C31–C3	178.4(3)
C5–C4–C3	120.98(17)	C4–C5–C11	117.12(17)
C4–C5–C6	120.53(17)	C11–C5–C6	122.35(17)
C5–C6–C7	114.76(17)	C8–C7–C6	115.02(16)
C7–C8–C9	115.39(16)	C8–C9–C10	114.61(16)
C11–C10–C9	114.84(15)	N1–C11–C10	115.31(15)
N1–C11–C5	123.03(17)	C5–C11–C10	121.66(17)
C2–O12–C13	117.70(14)	O12–C13–C14	112.95(15)
O14–C14–C15	122.22(18)	O14–C14–C13	120.78(17)
C15–C14–C13	116.95(16)	C16–C15–C14	122.40(16)
C20–C15–C14	119.17(17)		

N3–H31A amino group (as a donor) and the O14 oxygen atom of the phenacyl fragment (as an acceptor); it has the following parameters: H31A...O14 = 2.086 and N31...O14 = 2.739 Å, angle N31–H31A...O14 = 125.84°. The other hydrogen bond is intermolecular; it is also formed by the N31–H31B amino group (as a donor) and the N1ⁱ ($x-1, y, z$) atom the neighboring molecule (as an acceptor) and has the following parameters: H31B...N1ⁱ = 2.435 and N31...N1ⁱ = 3.291 Å, angle N31–H31B...N1ⁱ = 156.36°. In addition to this hydrogen bond, there is a nonclassical hydrogen bond between molecules: H17...O14ⁱⁱ ($x+1, y, z$) = 2.412 and C17...O14ⁱⁱ = 3.146 Å, angle C17–H17...O14ⁱⁱ = 135.74°. Two nonclassical intramolecular hydrogen bonds (C16–H16...O12 and C20–

Table 5. Selected bond lengths d (Å) and angles ω (deg) in structure **3**

Bond	d	Bond	d
N1–C2	1.3254(18)	N1–C11	1.3483(19)
C2–O12	1.3601(17)	C2–C3	1.3902(19)
C3–C4	1.388(2)	C3–C31	1.433(2)
C31–N31	1.3520(19)	C31–C13	1.386(2)
N31–H31A	0.93(3)	N31–H31B	0.91(2)
C4–C5	1.380(2)	C5–C11	1.421(2)
C5–C6	1.508(2)	C6–C7	1.536(2)
C7–C8	1.510(3)	C8–C9	1.523(3)
C9–C10	1.533(2)	C10–C11	1.504(2)
O12–C13	1.4096(16)	O14–C14	1.2470(19)
C13–C14	1.437(2)	C14–C15	1.488(2)
C18–C118	1.7410(18)		
Angle	ω	Angle	ω
C2–N1–C11	114.93(12)	N1–C2–O12	121.45(12)
N1–C2–C3	126.77(14)	O12–C2–C3	111.78(12)
C4–C3–C2	117.50(13)	C4–C3–C31	136.23(13)
C2–C3–C31	106.26(13)	N31–C31–C13	127.73(15)
N31–C31–C3	126.34(15)	C13–C31–C3	105.93(13)
C31–N31–H31A	115.2(16)	C31–N31–H31B	117.8(14)
H31A–N31–H31B	119(2)	C5–C4–C3	118.52(13)
C4–C5–C11	118.81(14)	C4–C5–C6	119.93(14)
C11–C5–C6	121.21(14)	C5–C6–C7	113.11(14)
C8–C7–C6	113.47(15)	C7–C8–C9	115.61(16)
C8–C9–C10	115.26(16)	C11–C10–C9	114.89(14)
N1–C11–C5	123.40(14)	N1–C11–C10	116.19(13)
C5–C11–C10	120.40(14)	C2–O12–C13	105.36(11)
C31–C13–O12	110.66(13)	C31–C13–C14	125.46(14)
O12–C13–C14	123.75(14)	O14–C14–C13	116.28(15)
O14–C14–C15	118.88(15)	C13–C14–C15	124.84(14)
C20–C15–C14	116.95(15)	C16–C15–C14	125.26(15)

Таблица 6. Selected bond lengths d (Å) and angles ω (deg) in structure 4

Bond	d	Bond	d	Angle	ω	Angle	ω
N1A–C12A	1.383(7)	N1B–C12B	1.371(7)	C12A–N1A–C2A	127.9(6)	C12B–N1B–C2B	123.5(6)
N1A–C2A	1.362(8)	N1B–C2B	1.381(8)	O2A–C2A–N1A	118.1(7)	O2B–C2B–N1B	119.5(7)
C2A–O2A	1.203(7)	C2B–O2B	1.209(7)	O2A–C2A–C3A	129.8(7)	O2B–C2B–C3B	126.8(8)
C2A–C3A	1.441(8)	C2B–C3B	1.422(8)	N1A–C2A–C3A	112.0(7)	N1B–C2B–C3B	113.7(7)
C3A–C4A	1.381(8)	C3B–C4B	1.342(9)	C4A–C3A–C2A	120.4(7)	C4B–C3B–C2B	118.7(8)
C3A–C31A	1.473(9)	C3B–C31B	1.509(9)	C4A–C3A–C31A	115.2(7)	C4B–C3B–C31B	121.1(7)
C31A–O31A	1.224(7)	C31B–O31B	1.195(7)	C2A–C3A–C31A	124.3(7)	C2B–C3B–C31B	120.1(7)
C31A–N31A	1.344(8)	C31B–N31B	1.333(7)	O31A–C31A–N31A	114.8(8)	O31B–C31B–N31B	133.8(8)
C4A–C5A	1.386(8)	C4B–C5B	1.421(9)	O31A–C31A–C3A	130.8(8)	O31B–C31B–C3B	108.3(7)
C5A–C12A	1.368(8)	C5B–C12B	1.358(8)	N31A–C31A–C3A	114.4(7)	N31B–C31B–C3B	117.9(7)
C5A–C6A	1.480(8)	C5B–C6B	1.480(8)	C3A–C4A–C5A	124.3(7)	C3B–C4B–C5B	129.6(7)
C6A–C7A	1.514(7)	C6B–C7B	1.525(7)	C12A–C5A–C4A	115.9(7)	C12B–C5B–C4B	108.3(6)
C7A–C8A	1.523(7)	C7B–C8B	1.533(8)	C12A–C5A–C6A	119.9(7)	C12B–C5B–C6B	126.8(7)
C8A–C9A	1.522(8)	C8B–C9B	1.511(7)	C4A–C5A–C6A	124.2(7)	C4B–C5B–C6B	124.8(7)
C9A–C10A	1.524(7)	C9B–C10B	1.502(7)	C5A–C6A–C7A	123.0(6)	C5B–C6B–C7B	119.2(6)
C10A–C11A	1.509(8)	C10B–C11B	1.524(7)	C6A–C7A–C8A	106.6(5)	C8B–C7B–C6B	112.1(6)
C11A–C12A	1.494(8)	C11B–C12B	1.481(8)	C9A–C8A–C7A	120.9(6)	C9B–C8B–C7B	123.1(6)
				C8A–C9A–C10A	116.5(6)	C10B–C9B–C8B	116.7(6)
				C11A–C10A–C9A	126.1(6)	C11B–C10B–C9B	120.7(6)
				C12A–C11A–C10A	104.9(6)	C12B–C11B–C10B	109.1(6)
				N1A–C12A–C5A	119.4(7)	N1B–C12B–C5B	126.1(7)
				N1A–C12A–C11A	114.2(6)	N1B–C12B–C11B	110.7(6)
				C5A–C12A–C11A	126.1(7)	C5B–C12B–C11B	123.0(7)

Таблица 7. Selected bond lengths d (Å) and angles ω (deg) in structure 5a

Bond	d	Bond	d	Angle	ω	Angle	ω
C18–C18	1.721(4)	N1–C2	1.333(4)	C2–N1–C12	118.2(3)	N1–C2–O2	117.3(3)
N1–C12	1.348(4)	C2–O2	1.372(4)	N1–C2–C3	125.0(4)	O2–C2–C3	117.7(4)
C2–C3	1.383(5)	O2–C13	1.417(4)	C2–O2–C13	117.7(3)	C2–C3–C4	115.2(4)
C3–C4	1.399(5)	C3–C31	1.506(5)	C2–C3–C31	127.1(4)	C4–C3–C31	117.6(4)
C31–O31	1.246(4)	C31–N31	1.321(4)	O31–C31–N31	122.0(4)	O31–C31–C3	119.1(4)
C4–C5	1.385(5)	C5–C12	1.393(5)	N31–C31–C3	119.0(4)	C5–C4–C3	121.9(4)
C5–C6	1.514(4)	C6–C7	1.521(5)	C4–C5–C12	117.4(4)	C4–C5–C6	120.1(4)
C7–C8	1.502(5)	C8–C9	1.495(5)	C12–C5–C6	122.5(4)	C5–C6–C7	111.8(3)
C9–C10	1.541(5)	C10–C11	1.514(5)	C8–C7–C6	117.7(4)	C9–C8–C7	117.1(4)
C11–C12	1.495(4)	C13–C14	1.507(5)	C8–C9–C10	118.0(4)	C11–C10–C9	116.2(3)
C14–O14	1.233(4)	C14–C15	1.470(5)	C12–C11–C10	116.8(3)	N1–C12–C5	122.2(3)
				N1–C12–C11	114.7(3)	C5–C12–C11	122.8(4)
				O2–C13–C14	107.7(3)	O14–C14–C15	122.0(4)
				O14–C14–C13	120.0(4)	C15–C14–C13	117.9(3)
				C20–C15–C14	122.0(4)	C16–C15–C14	119.1(4)

Таблица 8. Selected bond lengths d (Å) and angles ω (deg) in structure 5b

Bond	d	Bond	d	Angle	ω	Angle	ω
N1–C12	1.383(4)	N1–C2	1.423(4)	C12–N1–C2	124.0(3)	C12–N1–C13	124.0(3)
N1–C13	1.466(4)	C2–O2	1.236(4)	C2–N1–C13	111.1(3)	O2–C2–N1	117.9(3)
C2–C3	1.438(4)	C3–C4	1.351(4)	O2–C2–C3	127.3(4)	N1–C2–C3	114.8(3)
C3–C31	1.492(4)	C31–O31	1.224(4)	C4–C3–C2	120.1(3)	C4–C3–C31	117.8(3)
C31–N31	1.335(4)	N31–H31A	0.93(4)	C2–C3–C31	122.1(3)	O31–C31–N31	122.2(4)
N31–H31B	0.79(4)	C4–C5	1.409(4)	O31–C31–C3	120.7(3)	N31–C31–C3	117.0(3)
C5–C12	1.377(4)	C5–C6	1.485(4)	C31–N31–H31A	122(2)	C31–N31–H31B	117(3)
C6–C7	1.535(5)	C7–C8	1.562(5)	H31A–N31–H31B	122(4)	C3–C4–C5	123.8(3)
C8–C9	1.517(5)	C9–C10	1.510(5)	C12–C5–C4	117.8(3)	C12–C5–C6	122.9(3)
C10–C11	1.503(5)	C11–C12	1.498(4)	C4–C5–C6	119.2(3)	C5–C6–C7	115.2(3)
C13–C14	1.507(5)	C14–O14	1.210(4)	C6–C7–C8	115.9(3)	C9–C8–C7	117.3(4)
C14–C15	1.491(5)	C18–C118	1.728(4)	C10–C9–C8	115.9(4)	C11–C10–C9	114.4(4)
				C12–C11–C10	114.8(3)	C5–C12–N1	119.5(3)
				C5–C12–C11	121.1(3)	N1–C12–C11	119.3(3)
				N1–C13–C14	110.0(3)	O14–C14–C15	119.4(4)
				O14–C14–C13	121.2(3)	C15–C14–C13	119.3(3)
				C16–C15–C14	122.4(4)	C20–C15–C14	119.7(3)

Таблица 9. Selected bond lengths d (Å) and angles ω (deg) in structure 6

Bond	d	Bond	d	Angle	ω	Angle	ω
N1–C2	1.320(2)	N1–C12	1.353(2)	C2–N1–C12	118.29(13)	N1–C2–O2	118.85(14)
C2–O2	1.3598(19)	C2–C3	1.387(2)	N1–C2–C3	123.73(15)	O2–C2–C3	117.40(15)
O2–C13	1.4348(18)	C3–C4	1.388(2)	C2–O2–C13	115.96(13)	C2–C3–C4	117.08(16)
C3–C31	1.432(2)	C31–N31	1.144(2)	C2–C3–C31	121.57(15)	C4–C3–C31	121.34(14)
C4–C5	1.379(2)	C5–C12	1.389(2)	N31–C31–C3	177.85(18)	C5–C4–C3	120.78(15)
C5–C6	1.515(2)	C6–C7	1.520(2)	C4–C5–C12	117.53(15)	C4–C5–C6	120.28(15)
C7–C8	1.524(2)	C8–C9	1.526(2)	C12–C5–C6	122.05(16)	C5–C6–C7	111.77(15)
C9–C10	1.516(3)	C10–C11	1.532(3)	C6–C7–C8	115.68(15)	C7–C8–C9	116.67(18)
C11–C12	1.504(2)	C13–C14	1.512(2)	C10–C9–C8	116.37(16)	C9–C10–C11	116.28(17)
O14–C14	1.2128(18)	C18–C118	1.7377(19)	C12–C11–C10	113.35(16)	N1–C12–C5	122.54(16)
				N1–C12–C11	113.55(15)	C5–C12–C11	123.86(16)
				O2–C13–C14	110.55(13)	O14–C14–C15	121.46(16)
				O14–C14–C13	119.16(15)	C15–C14–C13	119.37(14)
				C16–C15–C14	123.06(16)	C20–C15–C14	118.35(15)

H20 \cdots O14) are 2.261 and 2.393 Å long. Two more intermolecular contacts—C4 \cdots N31ⁱⁱⁱ ($-x-1, -y, -z+1$) equal to 3.634 Å and C8 \cdots O14^{iv} ($x+1, y, z+1$) equal to 3.684 Å—should most likely be considered van der Waals interactions.

Compound 4. Compound 4 is tricyclic. The structure contains two crystallographically independent

molecules, *A* and *B*. Unlike the rigid pyridone heterocycle, conformationally flexible eight-membered fragment has the chair conformation: in molecules *A* and *B*, the planar C5/C6/C11/C12 (1) fragment forms with the C6/C7/C10/C11 (2) plane a dihedral angle of 67.50(2)° and 67.66(2)°, respectively. The C8*A* and C8*B* atoms deviate from plane 2 by 0.21 and -0.49 Å, respectively. The C9*A* and C9*B* atoms deviate from

plane 2 by -0.57 and 0.35 Å, respectively. In principle, these deviations account for the distinction in the conformations of eight-membered cycles. The six-membered heterocycles retain the pseudodiene $C2=C3=C4=C5=C12$ structure (Fig. 4, Table 6).

In crystal **4** H atoms of the amide groups form classical intramolecular hydrogen bonds with the ketone O atom. These bonds close stable six-membered $=C2-C3-C31-N31-H31\cdots O2=$ cycles with the following parameters: $H31\cdots O2A = 2.067$ and 1.967 Å, $N31\cdots O2 = 2.756$ and 2.644 Å, and angle $N31-H31\cdots O2 = 136.66^\circ$ and 134.86° for molecules *A* and *B*, respectively. In addition to intramolecular hydrogen bonds, molecules *A* and *B* form two eight-membered centrosymmetric dimers each. One of them involves the pyridone N1 atom (as a donor) and the O2 atom of the keto group (as an acceptor): $-N1A-H1A\cdots O2A^i = C2A^i-N1A^i-H1A^i\cdots O2A = C2A-$ and $-N1B-H1B\cdots O2B^{ii} = C2B^{ii}-N1B^{ii}-H1B^{ii}\cdots O2B=C2B-$. Symmetry operations: (i) $-x, -y + 1, -z + 2$; and (ii) $-x, -y + 2, -z + 1$. Parameters of these hydrogen bonds are as follows: $H1A\cdots O2A^i = 1.925$ and $N1A\cdots O2A^i = 2.739$ Å, angle $N1A-H1A\cdots O2A^i = 157.40^\circ$; $H1B\cdots O2B^{ii} = 2.143$ and $N1B\cdots O2B^{ii} = 2.993$ Å, angle $N1B-H1B\cdots O2B^{ii} = 169.87^\circ$. The second pair is $-N31A-H31A\cdots O31A^{iii} = C31A^{iii}-N31A^{iii}-H31A^{iii}\cdots O31A = C31A-$ and $-N31B-H31B\cdots O31B^{iv} = C31B^{iv}-N31B^{iv}-H31B^{iv}\cdots O31B = C31B-$. Symmetry operations: (iii) $-x, -y + 1, -z + 1$; and (iv) $-x, -y + 2, -z$. Parameters of these hydrogen bonds are as follows: $H31A\cdots O31A^{iii} = 2.145$ and $N31A\cdots O31A^{iii} = 2.994$ Å, angle $N31A-H31A\cdots O31A^{iii} = 169.27^\circ$; $H31B\cdots O31B^{iv} = 2.135$ and $N31B\cdots O31B^{iv} = 2.928$ Å, angle $N31B-H31B\cdots O31B^{iv} = 153.04^\circ$.

In addition to the classical bonds, nonclassical hydrogen bonds involving only C atoms of annelated eight-membered cycles are formed in the crystal structure. The N31, O2, and N1 atoms act as acceptors. Thus, two contacts involving the N31 atom of the nitrile group are 3.455 and 3.590 Å, four contacts involving the O2 atom are 3.336 – 3.745 Å, and six contacts involving the O31 atom are 3.048 – 3.696 Å.

Compound 5a. This compound is the product of *O*-phenacylation of **4**. As in **4**, the eight-membered fragment has the chair conformation: the planar C6/C5/C11/C12 (1) fragment forms a dihedral angle of $67.22(9)^\circ$ with the C6/C7/C10/C11 (2) plane, and the C8 and C9 atoms deviate from plane 2 by -0.31 and 0.56 Å, respectively. In consequence of the formation reaction, the six-membered heterocycle adopts the aromatic character and the bonds in the $C2-C3-C4-C5-C12$ chain become equal (Fig. 5, Table 7).

In structure **5a**, there are two classical intramolecular hydrogen bonds involving the amide group, which close the six-membered $-N31-H31B\cdots O2-C2-C3-C31-$ cycle and the nine-membered $-N31-H31B\cdots O14=C14-C13-O2-C2-C3-C31-$ cycle

with the following parameters: $H31B\cdots O2 = 2.024$ and $N31\cdots O2 = 2.686$ Å, angle $N31-H31B\cdots O2 = 133.05^\circ$ for the former; $H31B\cdots O14 = 2.832$ and $N31\cdots O14 = 3.649$ Å, angle $N31-H31B\cdots O14 = 159.38^\circ$ for the latter. Intermolecular hydrogen bonds formed by the amide groups of two molecules close centrosymmetric eight-membered cycle $-N31-H31A\cdots O31^i=C31^i-N31^i-H31A^i\cdots O31=C31-$: $H31A\cdots O31^i = 2.058$ Å, angle $N31-H31A\cdots O31^i = 171.04^\circ$, and (i) denotes $-x, -y + 2, -z + 1$. The following intermolecular contacts are found in the crystal structure: $C6-H6B\cdots O2^{ii}$, $C7-H7B\cdots O31^{iii}$, and $C9-H9A\cdots O14^{iv}$ with $C6\cdots O2^{ii} = 3.551$, $C7\cdots O31^{iii} = 3.501$, and $C9\cdots O14^{iv} = 3.616$ Å; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x, -y + 1, -z + 1$; (iv) $x, y - 1, z$. These contacts should most likely be considered van der Waals interactions.

Compound 5b. Compound **5b** is the product of *N*-phenacylation of **4**. As in **4**, the eight-membered fragment has the chair conformation: the planar C6/C5/C11/C12 (1) fragment forms a dihedral angle of $67.47(19)^\circ$ with the C6/C7/C10/C11 (2) plane; the C8 and C9 atoms deviate from plane 2 by $0.533(5)$ and $-0.366(5)$ Å. The six-membered heterocycle retains the pseudodiene $C2=C3=C4=C5=C12$ structure (Fig. 6, Table 8). The C31/O31/N31 amide group is almost coplanar with the six-membered heterocycle: the rotation angle is only $2.9(3)^\circ$. The C15–C20 benzene ring is almost perpendicular ($88.5(1)^\circ$) to the heterocycle.

Two types of classical hydrogen bonds are found in the structure. One of them is the intramolecular $N31-H31B\cdots O2$ bond having parameters $H31B\cdots O2 = 2.05(4)$ Å, $N31\cdots O2 = 2.704(5)$ Å, and angle $N31-H31B\cdots O2 = 141(4)^\circ$; the other is the intermolecular $N31-H31A\cdots O31^i$ bond having parameters $H31A\cdots O31^i = 1.98(4)$ Å, $N31\cdots O31^i = 2.904(4)$ Å, and angle $N31-H31A\cdots O31^i = 168(4)^\circ$. The (i) symmetry operation is $-x, -y + 2, -z + 1$. Nonclassical hydrogen bonds are also found in the crystal structure of this compound. The O2, O14, O31, and C118 atoms act as acceptors on these bonds. Their contacts can be divided into two ranges, namely, shorter and longer than 3 Å. The intramolecular $C13\cdots O2 = 2.552(4)$ Å contact falls in the former range. One contact involving O2 ($3.417(5)$ Å), three contacts involving O14 ($3.261(5)$ – $3.554(6)$ Å), three contacts involving O31 ($3.474(4)$ – $3.797(5)$ Å), and two contacts involving C118 ($3.845(5)$ and $3.571(5)$ Å) fall in the latter range. These contacts should most likely be considered van der Waals interactions.

Compound 6. Compound **6** has a special place in the series of compounds with annelated eight-membered cycle, since it is yet the only member of the series with the nonhydrolyzed nitrile group in the 3-position. The conformationally flexible eight-membered cycle adopts the chair conformation (Fig. 7). Planes C5/C6/C11/C12 (1) and C6/C7/C10/C11 (2)

form an angle of $69.34(8)^\circ$, and the C8 and C9 atoms deviate from plane 2 by 0.345 and -0.572 \AA .

There are no classical hydrogen bonds in the crystal structure. Intermolecular contacts $C6 \cdots O14^i = 3.173(3)$, $C7 \cdots O14^i = 3.255(3)$, $C16 \cdots N31^{ii} = 3.755(3)$, $C13 \cdots O2^{ii} = 3.525(3)$, $C19 \cdots O2^{iii} = 3.771(3)$, and $C20 \cdots N31^{iii} = 3.395(3) \text{ \AA}$ (symmetry operations: (i) $-x + 1, y - 1/2, -z + 3/2$; (ii) $-x, -y, -z + 1$; and (iii) $x, -y + 1/2, z + 1/2$) should most likely be considered van der Waals interactions. Selected interatomic distances and bond angles are given in Table 9.

In summary, seven new, previously unknown, heterocyclic compounds were obtained in this work using familiar and modified (based on specific features of starting compounds) synthetic procedures. Their physicochemical characteristics, namely, ^1H NMR spectra and melting points of individual compounds, were obtained. Single crystals suitable for X-ray diffraction study were grown and the crystal structures were determined.

ACKNOWLEDGMENTS

This study was supported by the Russian Foundation for Basic Research and the State Committee of Science of the Ministry of Education and Science of Republic Armenia within Joint Scientific Programs 15-53-05064 and 15RF-027.

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Translated by I. Polyakova

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