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CRYSTAL STRUCTURE OF 4,5-BIS-PHENYL-8a-PHENYL-3,4,4a,5,6,8a-HEXAHYDRO-1H,8H-PYRIMIDO[4.5-d]PYRIMIDINE-2,7-DIONE

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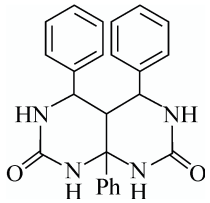
Single crystal X-ray diffraction is used to determine the crystal structure of 4,5-bis-phenyl-8a-phenyl-3,4,4a,5,6,8a-hexahydro-1H,8H-pyrimido[4.5-d]pyrimidine-2,7-dione and identify its conformation characteristics.

Keywords: crystal structure, 4,5-bis-phenyl-8a-phenyl-3,4,4a,5,6,8a-hexahydro-1H,8H-pyrimido[4.5-d]pyrimidine-2,7-dione.

Multicomponent reactions are known to become an ever-developing method to produce new biologically active compounds and pharmaceuticals. With the participation of three or more compounds, they form a new product containing the main structural parts of all precursors.

Taking into consideration the above, we set ourselves a goal to continue the studies on Biginelli reaction modification using aromatic ketones. We chose acetophenone [1] as the aromatic ketone for the investigation. Condensation of benzaldehyde, acetophenone, and carbamide in isopropyl alcohol in the presence of concentrated HCl resulted in the formation of 4,5-bis-phenyl-8a-phenyl-3,4,4a,5,6,8a-hexahydro-1H,8H-pyrimido[4.5-d]pyrimidine-2,7-dione (**I**).



This paper describes the single crystal XRD analysis of 4,5-bis-phenyl-8a-phenyl-3,4,4a,5,6,8a-hexahydro-1H,8H-pyrimido[4.5-d]pyrimidine-2,7-dione (**I**).

Experimental. The synthesis of compound **I** is described in the work [1]. The crystal for XRD was obtained by double crystallization of compound **I** from ethanol. XRD of compound **I** was performed on a Bruker SMART APEX II CCD diffractometer ($T = 296$ K, $\lambda\text{Mo}K_\alpha$ radiation, graphite monochromator, φ- and ω-scanning, $2\theta_{\max} = 56°$).

The crystals of the monohydrate of compound **I** ($C_{24}H_{22}N_4O_2 \cdot H_2O$, $M_r = 416.47$) were white; $T_m = 165\text{-}167°C$, cubic, 0.30×0.30×0.30 mm, monoclinic: $a = 11.2789(12)$ Å, $b = 17.4774(19)$ Å, $c = 10.5751(11)$ Å, $\beta = 94.719(2)°$, $V = 2077.6(4)$ Å^3, $P2_1/c$ space group, $Z = 4$, $d_x = 1.332$ g/cm^3, $\mu = 0.090$ mm^{-1}. We measured the intensities of 23,736 reflections

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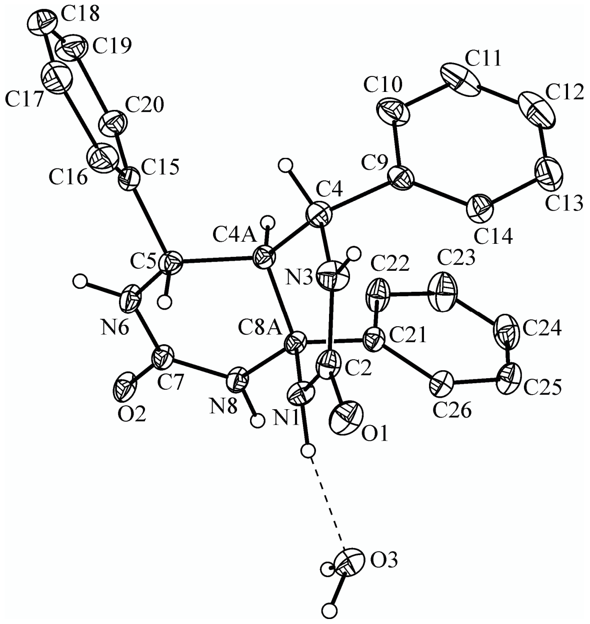

Fig. 1. Molecular structure of the monohydrate of compound **I**. The alternative position of the less occupied disordered molecule is not presented. In the molecule of **I**, the figure shows only hydrogen atoms of the amino groups and those at the asymmetric carbon atoms. The intermolecular hydrogen bond is shown by a dashed line.

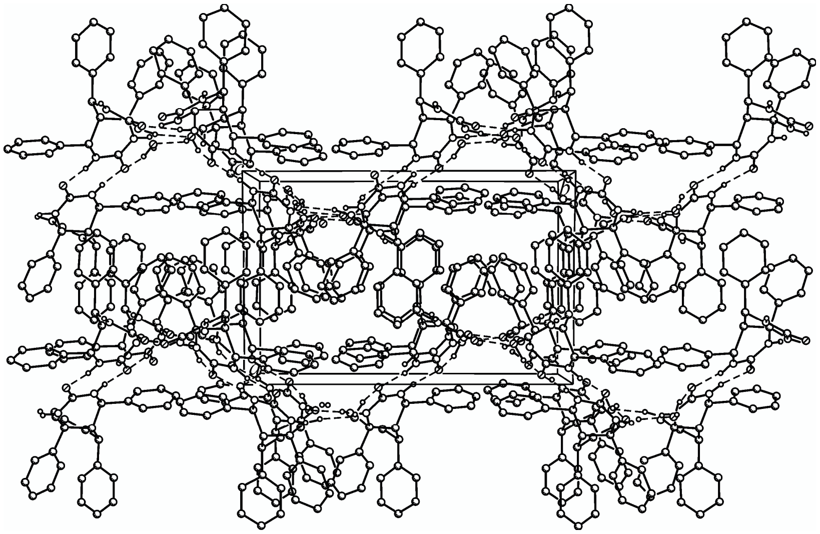

Fig. 2. Hydrogen bonds and packing of the molecules of compound **I** in the crystal (hydrogen bonds are shown by dashed lines).

(5127 independent reflections, $R_{\text{int}} = 0.025$), for which we introduced a semi-empirical correction using the SADABS program [2].

The structure of compound **I** was solved by the direct method and refined by LSM in an anisotropic approximation for non-hydrogen atoms. The crystal of compound **I** contains a solvate water molecule disordered over two positions with the 0.7:0.3 population. Hydrogen atoms of the amino groups and solvate water molecule were objectively identified in difference

TABLE 1. Main Interatomic Distances *d* (Å) and Bond Angles ω (deg) for the Molecule of **I**

Bond	*d*	Bond	*d*	Bond	*d*
O(1)–C(2)	1.2361(14)	N(3)–C(4)	1.4481(16)	N(6)–C(7)	1.3447(15)
O(2)–C(7)	1.2435(14)	C(4)–C(4a)	1.5418(14)	C(7)–N(8)	1.3460(14)
N(1)–C(2)	1.3492(15)	C(4a)–C(5)	1.5422(15)	N(8)–C(8a)	1.4639(13)
N(1)–C(8a)	1.4565(14)	C(4a)–C(8a)	1.5414(15)		
C(2)–N(3)	1.3529(17)	C(5)–N(6)	1.4555(14)		
Angle	ω	Angle	ω	Angle	ω
C(2)–N(1)–C(8a)	124.51(10)	C(4)–C(4a)–C(8a)	111.11(8)	C(7)–N(8)–C(8a)	126.35(9)
N(1)–C(2)–N(3)	117.56(10)	C(5)–C(4a)–C(8a)	108.38(9)	N(1)–C(8a)–C(4a)	107.30(8)
C(2)–N(3)–C(4)	125.98(10)	C(4a)–C(5)–N(6)	108.00(9)	N(1)–C(8a)–N(8)	108.46(9)
N(3)–C(4)–C(4a)	109.28(9)	C(5)–N(6)–C(7)	123.06(9)	C(4a)–C(8a)–N(8)	109.52(8)
C(4)–C(4a)–C(5)	112.11(9)	N(6)–C(7)–N(8)	117.54(10)		

TABLE 2. Hydrogen Bond Parameters (*d*, Å and ω, deg) in the Monohydrate of Compound **I***

Parameter	*d*(D–H)	*d*(H…A)	*d*(D…A)	ω(D–H…A)
N(1)–H(1N)…O(3)	0.91	1.94	2.829(2)	167
N(1)–H(1N)…O(3′)	0.91	1.75	2.657(2)	178
N(6)–H(6N)…O(2)#1	0.92	1.99	2.902(2)	167
N(8)–H(8N)…O(1)#2	0.90	2.11	2.975(2)	160
O(3)–H(3A)…O(2)#3	0.98	1.83	2.753(2)	155
O(3′)–H(3A)…O(2)#3	0.96	1.83	2.756(2)	161
O(3)–H(3B)…O(1)#2	0.95	1.92	2.756(2)	147
O(3′)–H(3B)…O(1)#2	0.96	1.92	2.754(2)	144

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*D is a proton donor, A is a proton acceptor.

Note. Symmetric transformations for equivalent atoms: #1 $-x$, $-y$, $-z+2$; #2 x, $-y+1/2$, $z+1/2$; #3 x, $-y+1/2$, $z-1/2$.

Fourier syntheses and included into the refinement with fixed positions and isotropic thermal parameters ($U_{eq}(H) = 1.5U_{eq}(O)$ and $U_{iso}(H) = 1.2U_{eq}(N)$). The coordinates of other hydrogen atoms were calculated from geometric considerations and refined by the riding model with thermal parameters ($U_{eq}(H) = 1.5U_{eq}(C)$ for the CH_3 groups and $U_{eq}(H) = 1.2U_{eq}(C)$ for all the other groups). The final values of the divergence factors were $R_1 = 0.041$ for 4084 independent reflections with $I > 2\sigma(I)$ and $wR_2 = 0.111$ for all independent reflections. All calculations were made using the SHELXTL program package [3].

The structure of **I** has been deposited with the Cambridge Crystallographic Data Centre (No. 732855).

Results and Discussion. The structure and crystal packing of compound **I** are shown in Figs. 1 and 2; bond lengths and bond angles in the molecule of **I** are given in Table 1.

The bicyclic system of the molecule of **I** is formed by two C(4a)–C(8a) *cis*-conjugated hexahydropyrimidine rings (H(4a)–C(4a)–C(8a)–C(21) torsion angle is 48.2°) (Fig. 1). Both six-membered rings have *sofa* conformation (the deviation of C(4a) atoms from the C(8a)N(1)C(2)N(3)C(4) (plane *A*) and C(5)N(6)C(7)N(8)C(8a) (plane *B*) central planes are –0.611 Å and 0.680 Å respectively). It is noteworthy that the O(1) oxygen atom is considerably out of the plane *A* (the deviation is 0.137 Å), while the O(2) oxygen atom actually lies in the similar plane *B* (the deviation is less than 0.001 Å). This fact appears to be explained by a distortion of the planar trigonal configuration of the N(1) nitrogen atom (the sum of bond angles at the N(1) nitrogen atom is 355.4°) involved in the formation of an intermolecular hydrogen bond with the solvate water molecule (see below). The planar trigonal configurations of other N(3), N(6), and N(8) nitrogen atoms are practically not distorted (the sum of bond angles at N(3), N(6), and N(8) nitrogen atoms are 359.4°, 360.0°, and 360.0° respectively). The phenyl substituents at C(4) and C(5) carbon atoms have a pseudoaxial and pseudoequatorial orientations respectively. The phenyl substituent at the C(8a) carbon atom holds a pseudoaxial position with respect to the N(1)C(2)N(3)C(4)C(4a)C(8a) pyrimidine ring and a pseudoequatorial position with respect to the C(4a)C(5)N(6)C(7)N(8)C(8a) pyrimidine ring.

Compound **I** is a diastereomer with four asymmetric centers at C(4), C(4a), C(5), and C(8a) carbon atoms. The crystal of compound **I** is a racemate with the relative configuration of the chiral atoms: *rac*-4***R****,4a***R****,5***R****,8a***S****.

In the crystal, molecules of **I** are packed into corrugated layers parallel to the 0*YZ* plane by means of hydrogen bonds both directly with each other and through the solvate water molecule (Fig. 2, Table 2). Note that the >N(3)H amino group is not involved in hydrogen bonding.

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