ISSN 1070-3632, Russian Journal of General Chemistry, 2012, Vol. 82, No. 3, pp. 465–467. © Pleiades Publishing, Ltd., 2012.
Original Russian Text © L.D. Popov, S.I. Levchenkov, I.N. Scherbakov, Z.A. Starikova, E.B. Kaimakan, V.V. Lukov, 2012, published in Zhurnal Obshchei Khimii, 2012, Vol. 82, No. 3, pp. 472–474.

Crystal Structure of 2-Acetylbenzimidazole 1'-Phthalazinylhydrazone

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Received January 11, 2011

Abstract—Using X-ray diffraction study of acetylbenzimidazole 2-acetylbenzimidazole 1'-phthalazinylhydrazone we confirmed that this compounds exists as the phthalazonehydrazone tautomer, as was suggested earlier on the basis of IR and NMR spectroscopy and quantum-chemical calculations.

DOI: 10.1134/S1070363212030176

In [1] we have published the results of physicochemical and theoretical studies on the structure and complexing ability of the 2-acetylbenzimidazole 1'-phthalazinylhydrazone (**I**). On the basis of IR and NMR spectroscopy and quantum-chemical calculations in nonempirical approximation within the density functional theory (DFT) framework we suggested that the compound existed predominantly as the phthalazonehydrazone tautomer [2, 3]. In this report, in addition to the previously published data, we present the results of X-ray diffraction study of the 2-acetylbenzimidazole 1'-phthalazinylhydrazone.

A single crystals for the X-ray diffraction analysis was obtained by slow cooling of a solution of **I** in DMSO. Its structure includes water and DMSO molecules. Yellow prisms (M = 398.49) are monoclinic at 120 K, a = 13.1046(15) Å, b = 14.2812(17) Å, c = 10.8040(13) Å, β = 103.202(3)°, V = 1968.5(4) Å^3, space group $P2_1/c$, Z = 4, ρ_{calc} = 1,345 g cm^{-3}. The intensities of 7930 reflections were measured on a Bruker SMART 1000 CCD diffractometer [$\lambda(\mathrm{Mo}K_a)$ = 0.71073 Å, graphite monochromator, ω-scan, $2\theta < 52°$] from a single crystal sample of the size 0.24×0.21×0.14 mm^3. Processing of the source array of measured intensities was performed with the SAINT [4] and SADABS [5] programs. The structure was solved by the direct method and refined in a full-matrix anisotropic approximation for nonhydrogen atoms with respect to F^2_{hkl}. The hydrogen atoms were placed in geometrically calculated positions and refined using a *rider* model [$U_{iso}(H) = nU_{eq}(C)$, where n = 1.5 for carbon atoms of methyl groups, n = 1.2 for the other C atoms]. In the refinement 3844 independent reflections were used (R_{int} = 0.0438). The convergence of the refinement of all independent reflections wR_2 = 0.0661 (R_1 = 0.0379). All calculations were performed using the software package SHELXL-97 [6]. The atomic coordinates and temperature factors are deposited in the Cambridge Structural Database (CCDC 804712).

The crystal structure besides the molecules **I** includes water and DMSO molecules in equimolar ratio. The molecule of compound **I** is close to planar. The hydrazone is in the phthalazone tautomeric form. The conformation of the molecule corresponds to the structure **Ib** given in [1], which, according to calculations, is only by 0.45 kcal mol^{-1} less stable than the most stable conformation corresponding to the 180° rotation about the C^9–N^4 bond [1]. The stabilization of this conformation in the crystal is due to the formation of the strong hydrogen bonds between hydrogen atoms H^5 and H^2 and the oxygen atom of the water molecule O^{1W} (characteristics of the hydrogen bonds are listed in the table). The hydrogen atoms of

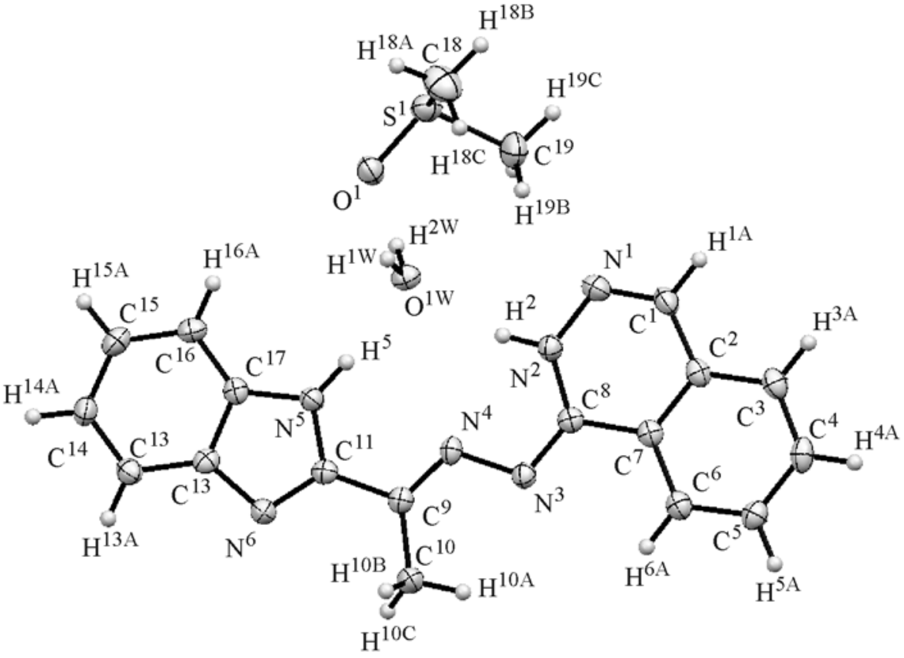

Fig. 1. Structure of 2-acetylbenzimidazole 1'-phthalazinylhydrazone (in thermal ellipsoids of 50% probability)

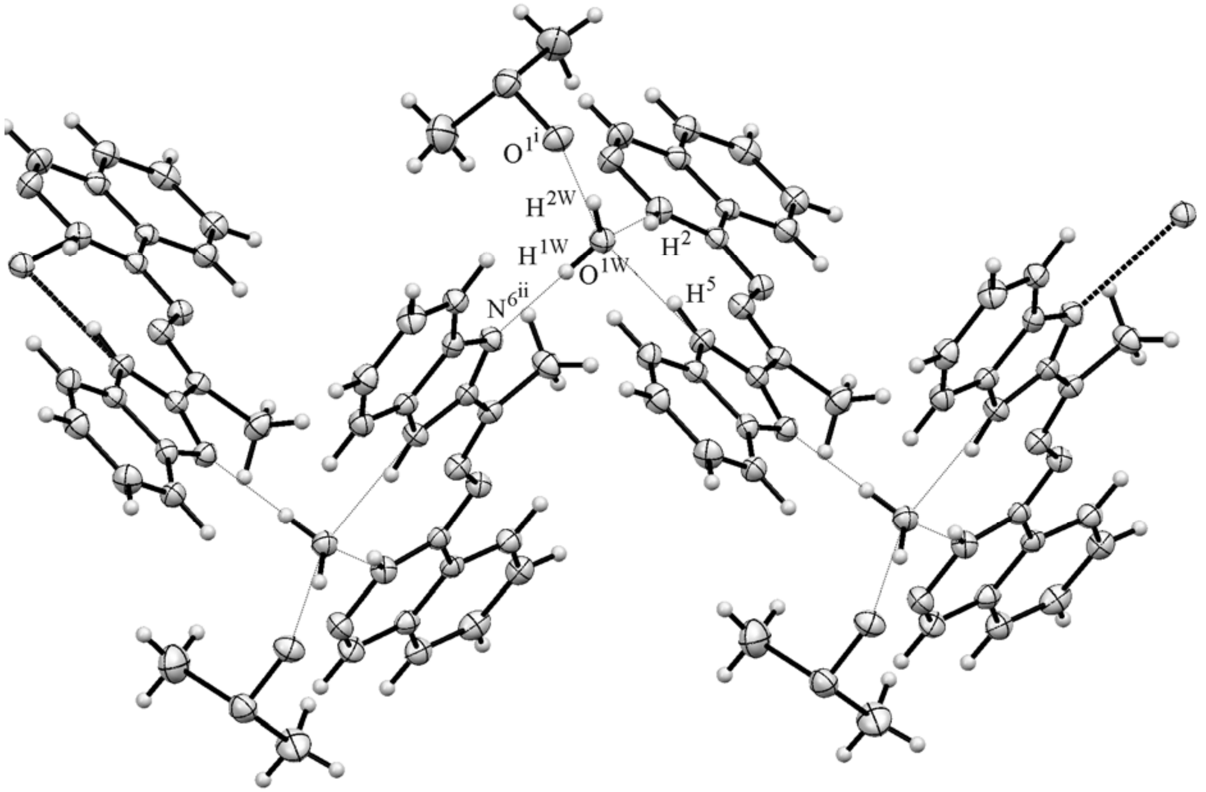

Fig. 2. The packing of molecules in a single crystal of hydrazone **I** (the crystallographic *c* axis is set horizontally)

water molecules H^{1W} and H^{2W}, in turn, form hydrogen bonds with the nitrogen atom N^{6i} of another hydrazone molecule (i: x, $1/2 - y$, $-1/2 + z$) and the oxygen atom of DMSO molecule O^{1ii} (ii: $1 - x$, $-y$, $1 - z$), which leads to the formation of infinite one-dimensional chains of hydrogen bonds involving differently oriented hydrazone **I** along the crystallographic axis (Fig. 2).

Characteristics of hydrogen bonds in crystal **I**[a]

D–H	A	*d*(D–H), Å	*d*(HA), Å	∠DHA, deg	*d*(DA), Å
N^2–H^2	O^{1W}	0.90	2.05	163	2.924(2)
N^5–H^5	O^{1W}	0.92	1.88	169	2.786(2)
O^{1W}–H^{1W}	$N^{6(i)}$	0.89	1.90	170	2.785(2)
O^{1W}–H^{2W}	$O^{1(ii)}$	0.93	1.82	168	2.742(2)

[a] Crystallographic positions: (i) x, $1/2 - y$, $-1/2 + z$; (ii) $1 - x$, $-y$, $1 - z$.

ACKNOWLEDGMENTS

This work was supported by the Ministry of Education and Science under the Federal Target Program “Research and scientific-pedagogical staff of Innovative Russia” for 2009–2011. (State Contract no. 02.740.11.0255).

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