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CRYSTAL STRUCTURE OF 2-[BENZYL-(7-OXO-CYCLOHEPTA-1,3,5-TRIENYL)-AMINO]-*N*-CYCLOHEXYLPROPANAMIDE

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The title compound 2-[benzyl-(7-oxo-cyclohepta-1,3,5-trienyl)-amino]-*N*-cyclohexylpropanemide is synthesized and studied by the single crystal X-ray diffraction method. The structure of the product was confirmed by IR, ^{1}H and ^{13}C NMR spectroscopy, and mass spectrometry. The tropone oxygen atom plays an important role in the solid-state molecular structure stabilization by being involved in intramolecular N—H···O and C—H···O contacts.

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Keywords: Single crystal X-ray structure, Smiles rearrangement, Ugi-type coupling, tropolone, cyclohexylisocyanide, benzyl amine, acetaldehyde, Ugi—Smiles-type reaction.

The special features of isocyanide-based multi-component reactions (abbreviated to IMCRs by *Ugi* and *Dömling*) such as the unique synthetic potential, convergent nature, high atom economy, ease of implementation, and the generation of molecular diversity are considered as acceptable factors in the relative efficiency of the reactions [1—6]. In connection with our recent interest in isocyanide chemistry [7—9], we report the synthesis and structure of 2-(*N*,*N*-dialkylamino)-2,4,6-cycloheptatrien-1-one derivative, i.e. 2-[benzyl-(7-oxo-cyclohepta-1,3,5-trienyl)-amino]-*N*-cyclohexylpropanamide, from the Ugi—Smiles-type reaction between acetaldehyde, benzylamine, cyclohexylisocyanide and tropolone. According to our knowledge, solid-state structural data have not as yet been reported for compounds with the 2-(7-oxo-cyclohepta-1,3,5-trienylamino)-acetamide fragment [10].

Experimental. Starting materials and solvents were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. The methods used to follow the reactions were TLC and NMR. TLC and NMR indicated that there was no side products. Melting points were measured on an Electrothermal 9100 apparatus and were uncorrected. IR spectra were measured on a Jasco 6300 FTIR spectrometer. ^{1}H and ^{13}C NMR spectra ($CDCl_3$) were recorded on a BRUKER DRX-250 AVANCE spectrometer at 400.22 MHz and 100.63 MHz respectively. Elemental analyses were performed using a Heraeus CHN—O-Rapid analyzer. Mass spectra were recorded on a FINNIGAN-MATT 8430 mass spectrometer operating at an ionization potential of 70 eV. Preparative thin layer chromatography was performed from Merck silica gel powder.

Preparation of 2-[benzyl-(7-oxo-cyclohepta-1,3,5-trienyl)-amino]-*N*-cyclohexylpropanamide (5). To a magnetically stirred solution of acetaldehyde (**1**) (1 mmol), benzyl amine (**2**) (1 mmol), and tropolone (**4**) (1 mmol) in CH_3OH (7 ml), a solution of cyclohexyl isocyanide (**3**) (1 mmol) in CH_3OH

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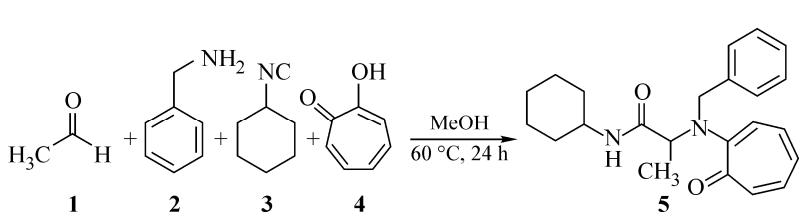
(2 ml) was added dropwise at 60 °C over 5 min. The reaction mixture was refluxed for 24 h. The solvent was removed under reduced pressure and the viscous residue was purified by preparative thin layer chromatography (silica gel; petroleum ether—ethyl acetate (4:1)). The solvent was removed under reduced pressure and product (**5**) was obtained. Yellow crystals, m.p. 133—134 °C, Yield 70 %. IR (KBr): υ = 3291 (NH), 2933 cm^{-1}, 2851 cm^{-1}, 1671 cm^{-1}, 1661 cm^{-1}, 1608 cm^{-1}, 1461 cm^{-1}, 1071 cm^{-1}, 1040 cm^{-1}, 751 cm^{-1}. ^{1}H NMR (400.22 MHz, $CDCl_3$): δ= 1.33 (d, $^3J_{HH}$ = 7.20 Hz, 3H, CH_3), 1.20—2.20 (m, 10H, 5 CH_2 of cyclohexyl), 3.75—3.85 (m, 1H, cyclohexyl, NC*H*), 4.40 (AB-quartet, $^2J_{HH}$ = 16.8 Hz, 2H, N—CH_2Ph), 4.68 (q, $^3J_{HH}$ = 7.0 Hz, 1H, NC*H*, acyclic), 6.58 (d, $^3J_{HH}$ = = 10.0 Hz, 1H, C*H* of tropolone), 6.68 (t, $^3J_{HH}$ = 9.2 Hz, 1H, C*H* of tropolone), 6.86 (t, $^3J_{HH}$ = 10.2 Hz, 1H, C*H* of tropolone), 7.08—7.30 (m, 7H; C*H* of tropolone and Ph), 8.53 (d, $^3J_{HH}$ = 8.00 Hz, 1H, N*H*). ^{13}C NMR (100.63 MHz, $CDCl_3$): δ = 13.23 (*C*H_3), 24.78, 25.65, 33.04, (3 *C*H_2, cyclohexyl), 47.95 (*C*H, cyclohexyl), 49.76 (CH_2 of Ph*C*H_2), 57.72 (*C*H), 123.23, 127.11, 127.25, 127.50, 128.54, 133.53, 135.35, 136.52 (8*C*H, tropolone and Ph), 136.21 (*C*, arom), 157.21 (*C*, tropolone), 170.55 (*C*O, amide) and 184.79 (*C*O, tropolone). Anal. Calcd for $C_{23}H_{28}N_2O_2$ (364.48): C 75.79, H 7.74, N 7.69; Found: C 75.75, H 7.76, N 7.71. MS: *m*/*z* (%) (EI) 364 (M^+, 16), 273 (36), 266 (25), 239 (29), 238 (100), 210 (54), 174 (12), 146 (29), 132 (10), 105 (9), 91 (87), 77(13), 65 (8), 55 (13), 41 (10).

Single crystals of title compound **5** were prepared using the branch tube method [4] in the *n*-hexane/1,4-dioxane (10:1) solvent at 45 °C during a week. The yellow crystals were filtered off, washed with cold *n*-hexane, and dried at room temperature (m. p. 133.5 °C).

X-ray crystallography. The crystallographic measurement was performed on a κ-geometry *Kuma KM4-CCD* automated four-circle diffractometer with graphite monochromatized MoK_α radiation; λ = 0.71073 Å (ω scan). The crystal data were collected at 100(2) K using the *Oxford-Cryosystems* cooler. The data were corrected for Lorentz and polarization effects. Data collection, cell refinement, and data reduction and analysis were carried out with the *Kuma KM4-CCD* software, CRYSALIS CCD and CRYSALIS RED, respectively [11]. The structure was solved by direct methods with the SHELXS-97 program [12], and refined on F^2 by a full-matrix least-squares technique using SHELXL-97 [12] with anisotropic thermal parameters for non-H-atoms. All H atoms were found in difference Fourier maps and were refined isotropically. In the final refinement cycles, all C-bonded H atoms were treated as riding atoms in geometrically optimized positions with C—H = = 0.95—1.00 Å and with U_{iso}(H) = 1.2U_{eq}(C) for CH and CH_2 or 1.5U_{eq}(C) for CH_3. The figures were made using the DIAMOND program [13]. CCDC-848164 contains complete crystallographic data for **5**. These data are freely available upon request *via* http://www.ccdc.cam.ac.uk/data_request/cif.

Crystal data of 5. $C_{23}H_{28}N_2O_2$, M = 364.47, triclinic, space group $P\bar{1}$ (no. 2), a = 7.195(2) Å, b = 11.320(3) Å, c = 13.075(3) Å, α = 66.42(3)°, β = 78.91(3)°, γ = 78.00(3)°, V = 947.6(4) Å^3, Z = 2, T = 100(2) K, μ = 0.08 mm^{-1}, crystal size 0.54×0.54×0.28 mm, yellow block, 13126 reflections measured, 5819 unique (R_{int} = 0.014), 5145 observed ($I > 2\sigma(I)$), θ range 2.92—32.50°, index ranges $-9 \le h \le 10$, $-15 \le k \le 14$, $-17 \le l \le 18$, 0 restraints, 249 parameters, $R1$ = 0.039 (observed refl.), $wR2$ = 0.112 (all data), S = 1.094, $\Delta\rho_{max}$ = 0.47, $\Delta\rho_{min}$ = –0.20 e/Å^3.

Results and discussion. The 1:1 imine intermediate generated by the condensation reaction of benzyl amine **2** with acetaldehyde **1** is trapped by the cyclohexylisocyanide **3** in the presence of tropolone **4**, which leads to the formation of 2-(*N*,*N*-dialkylamino)-2,4,6-cycloheptatrien-1-one derivative (**5**) (Scheme 1). The reaction proceeds smoothly and cleanly under mild and neutral conditions and no



Scheme 1. Four-component synthesis of
2-[benzyl-(7-oxo-cyclohepta-1,3,5-trienyl)-amino]-*N*-cyclohexylpropanamide **5**

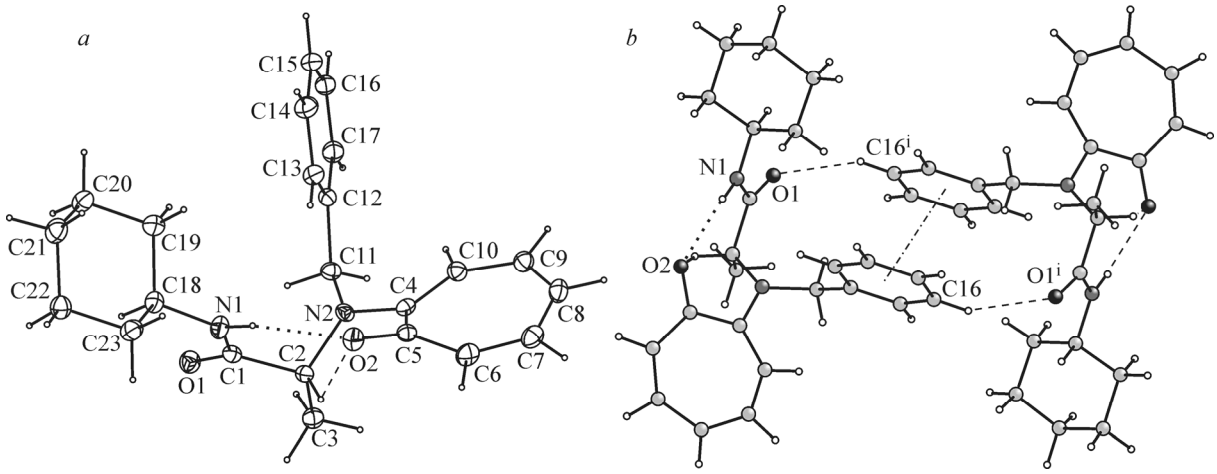


Fig. 1. Molecular structure of **5**, showing the X-ray atom numbering scheme and intramolecular N—H···O and C—H···O contacts (dashed lines). Displacement ellipsoids represent the 50 % probability level. The *R* enantiomer is shown (*a*). Centrosymmetric dimer formed by two enantiomers joined through two C—H···O bonds (dashed lines); accompanying π···π interaction — dash-dot line. Intramolecular contacts — dotted lines (*b*). Symmetry code is given in Table 1. Selected interatomic distances (Å) and bond angles (°): N(2)—C(2) 1.4795(11), N(2)—C(4) 1.3794(11), N(2)—C(11) 1.4583(11), C(4)—C(5) 1.4890(12), C(4)—C(10) 1.3863(12), C(5)—C(6) 1.4480(12), C(6)—C(7) 1.3637(14), C(7)—C(8) 1.4136(14), C(8)—C(9) 1.3681(13), C(9)—C(10) 1.4128(12); C(2)—N(2)—C(4) 120.76(7), C(2)—N(2)—C(11) 114.87(7), C(4)—N(2)—C(11) 120.43(7)

side reactions were observed. The structure of the product was deduced from its IR, ^{1}H NMR, ^{13}C NMR, mass spectrometry, and elemental analysis.

Crystal structure of 5. The centrosymmetric space group shows that the crystal contains racemic compound **5** (Fig. 1). The values of bond lengths and bond angles correspond well with those typical of the respective types of chemical connections [14].

The aminotropone C(4) ~ C(10) ring is not completely planar and shows a pronounced bond lengths alternation. The analysis of the cycloheptatrienyl endocyclic torsion angles reveals the maximum twist on C(4)—C(5) and C(5)—C(6) bonds. The angle of the intersection between the least-squares plane A defined by C(4), C(5), and C(6) and the plane B defined by C(6), C(7), C(8), C(9), C(10), and C(4) (r.m.s. deviation of fitted atoms = 0.051 Å) is 27.6(1)°. This deviation from the ring planarity is also reflected in the mutual orientation of O(2) and N(2) atoms (the N(2)—C(4)—C(5)—O(2) torsion angle is –20.56(10)°). It is worth noting that the O(2) atom acts as an acceptor of two intramolecular hydrogen interactions from N(1) and C(2) atoms, as shown in Fig. 1.

T a b l e 1

Geometry of hydrogen bonds and close contacts for **5** (Å, deg.)

D—H···*A*	*D*—H, Å	H···*A*, Å	*D*···*A*, Å	*D*—H···*A*, deg.
N1—H1···O2	0.87(2)	2.14(2)	2.939(2)	152(1)
C2—H2···O2	1.00	2.27	2.854(2)	116
C6—H6···O1^{i}	0.95	2.60	3.465(2)	152
C16—H16···O1^{ii}	0.95	2.51	3.303(2)	142
C21—H21*A*···O1^{iii}	0.99	2.60	3.501(2)	151

Symmetry codes: (i) –*x*+1, –*y*, –*z*+1; (ii) –*x*+1, –*y*+1, –*z*; (iii) *x*–1, *y*, *z*.

As a result of the formal sp^2-hybridization of the N(2) atom, it has an almost planar environment (the sum of C—N(2)—C angles is 356.1(1)°) and with C(2), C(4) and C(11) atoms it forms the plane C intersecting with the planes A and B at 50.7(1)° and 23.1(1)°, respectively, which is also reflected in the values of C(2)—N(2)—C(4)—C(5) and C(2)—N(2)—C(4)—C(10) torsion angles (–44.03(9) and 146.15(7)°, respectively). A partially-double character of the N(2)—C(4) bond is reflected in its length being 1.3794(11) Å as compared to the N(2)—C(2) and N(2)—C(11) bond lengths of 1.4795(11) Å and 1.4583(11) Å, which are typical values of the single C—N bond. Due to the partial double-bond character of N(2)—C(4), the compound may adopt two different conformations with respect to this bond, i.e. *E* and *Z*. As it is reflected in the C(2)—N(2)—C(4)—C(5) torsion angle (–44.03(9)°), the molecule of compounds **5** is the *Z* conformer with regard to the N(2)—C(4) bond.

The phenyl ring is twisted at about 20° relative to the N(2)—C(11) bond (the N(2)—C(11)—C(12)—C(17) torsion angle is 161.59(7)°).

The amide group (in the *trans* configuration) has the N(1) atom *synclinal* to N(2) and *antiperiplanar* to methyl C(3) and the C(1) atom *synclinal* to benzyl C(11) and *anticlinal* to tropone C(4). It is possible that the mutual *in-plane* orientation of the methyl group and the amide moiety is assisted by the presence of the intramolecular C(2)—H(2)···O(2) interaction (Fig. 1, Table 1).

While the tropone O(2) atom is involved solely in the intramolecular interactions stabilizing the molecular structure of **5**, the amide O(1) atom plays a significant role in the formation of the crystal lattice of **5**, mainly due to the participation in weak intermolecular C—H···O interactions (Table 1). The closest are C(16)—H(16)···O(1)^{ii} contacts joining two enantiomers into centrosymmetric dimers shown in Fig. 1. The mutual orientation of the phenyl rings within the dimer can be accompanied by a weak π···π interaction with an interplanar spacing of 3.558(1) Å and a centroid—centroid separation of 4.039(2) Å. The adjacent dimers are further connected by other weaker C—H···O(1) contacts to form layers parallel to the (011) plane.

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КРАТКИЕ СООБЩЕНИЯ

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CRYSTAL STRUCTURE AND DFT STUDY OF *N*-PHENYL-*N*-(PYRIDIN-4-YL)ACETAMIDE

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The title compound *N*-phenyl-*N*-(pyridin-4-yl)acetamide (**1**) crystallizes in the monoclinic crystal system in the space group $P2_1/n$ with unit cell parameters $a = 9.097(7)$ Å, $b = 11.824(11)$ Å, $c = 10.128(10)$ Å, $\beta = 106.64(2)°$, $V = 1043.8(16)$ Å^3 and $Z = 4$. The structure of the amide unit is almost planar. The dihedral angles of the amide plane with the benzene and pyridine rings are 58.40(5)° and 61.51(5)° respectively, indicating that neither phenyl nor pyridyl group is conjugated with the amide unit.

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K e y w o r d s: crystal structure, DFT calculation, acetamide, pyridine, hydrogen bond.

The conformation of the carbonyl group in *N*,*N*-diarylamides has received interest due to their reactivities [1]. The conformation where the oxygen atom of the carbonyl group has a close contact with an electron-deficient aryl group is considered to be favorable because of the effective conjugation and an intramolecular hydrogen bond with the aryl group. We report the structure and DFT study of *N*-phenyl-*N*-(pyridin-4-yl)acetamide (**1**) where a phenyl group of diphenylacetamide is replaced by the pyridyl group.

Experimental. The preparation of **1** was carried out according to the literature [2, 3]. The single crystals were obtained by recrystallization from dichloromethane. X-ray data from a monoclinic crystal (colorless block, 0.10×0.05×0.05 mm) were collected on a RIGAKU Saturn 724+ CCD device at 93 K. Crystallographic parameters are as follows: space group $P2_1/n$, $a = 9.097(7)$ Å, $b = 11.824(11)$ Å, $c = 10.128(10)$ Å, $\beta = 106.64(2)°$, $V = 1043.8(16)$ Å^3, $Z = 4$, empirical formula $C_{13}H_{12}N_2O$, $M = 212.25$, $d_x = 1.351$ g/cm^{-3}, $F(000) = 448.00$. The intensities of 7652 reflections (multi-layered mirror monochromatic MoK_α radiation, $2\theta_{max} = 52°$, $R_{int} = 0.0418$ for 2027 unique reflections (1652 for $I > 2\sigma(I)$), numerical absorption correction) were measured; $R(I > 2\sigma(I)$, all data) = 0.0425, 0.0567, $wR(I > 2\sigma(I)$, all data) = 0.0926, 0.0996, GOOF on $F^2 = 1.050$, $\Delta\rho_{max} = 0.23$ e/Å^{-3}, $\Delta\rho_{min} = -0.23$ e/Å^{-3}, Data / restrains / parameters = 2027 / 0 / 146.

The structure was solved by a direct method (SHELXD) [4] and was refined by the full-matrix least squares method (SHELXL97) [5]. The positions of the C-bound H atoms were obtained by the calculation and were refined as riding on their parent C atoms. U_{iso}(H) values of the H atoms were set at $1.2U_{eq}$ (parent atom for C). CIF-file containing complete information on studied structure was deposited with CCDC 940755, and is freely available upon request from the following web site: www.ccdc.cam.ac.uk/data_request/cif.

Results and discussion. The crystal structure of **1** is shown in Fig. 1, *a*. The structure of the C1/C7/N1/C12/O1/C13 amide unit is almost planar (r.m.s. deviation = 0.0278 Å). The dihedral angles of the amide plane with the benzene and pyridine rings are 58.40(5)° and 61.51(5)° respectively, indi-

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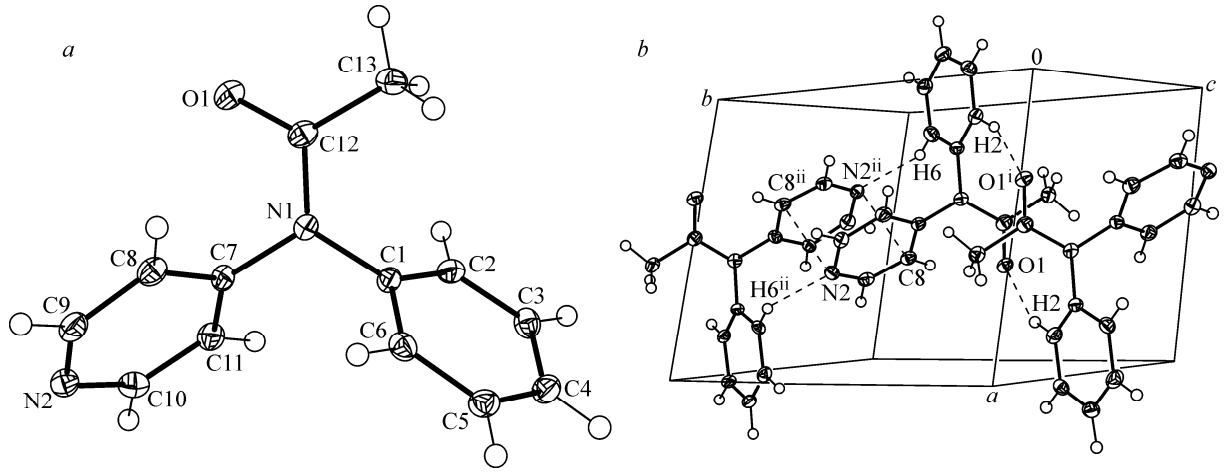

Fig. 1. Crystal structure of **1** (*a*) and the crystal packing structure of the asymmetric unit (b). Intermolecular contacts: C2—H2···O1^{i} = 2.460(2) Å, C6—H6···N2^{ii} = 2.599(2) Å, N2···C8^{ii} = 3.460(3) Å (*symmetry codes*: (i) 1–*x*, –*y*, –*z*, (ii) 1–*x*, 1–*y*, –*z*). Plane-plane and centroid—centroid distances of the pyridine rings: 3.3393(6) Å and 3.945(4) Å

cating that neither phenyl nor pyridyl group is well conjugated with the amide unit. The oxygen atom of the carbonyl group has the same side with the pyridyl ring, but intramolecular hydrogen bonds cannot be recognized. There are five kinds of intermolecular contacts: C2—H2···O1^{i} hydrogen bonds, C6—H6···N2^{ii} hydrogen bonds, and the π···π stacking interaction, as shown in Fig. 1, *b*. The remainning two weak C—H···π interactions are detected as C4—H4···π (N2/C7—C10 pyridyl)^{iii} and C10—H10···π (C1—C6 phenyl)^{iv} (*symmetry codes*: (iii) *x*–1, *y*, *z*, (iv) *x*+1/2, –*y*+1/2, –*z*–1/2), whose geometries are 2.81 Å and 2.77 Å for H4···center and H4···plane, and 2.91 Å and 2.86 Å for H10···center and H4···plane.

DFT calculations converged to two stable conformers (I and II), depending on the direction of the carbonyl group as shown in Fig. 2. Conformer I was estimated to be more stable than conformer II by 7.4 kJ·mol^{-1}, and the energy difference indicated little population of conformer II at room temperature. In conformer I, the pyridyl group has a smaller angle (ca 25.4°) with the amide plane, although the phenyl group has a larger angle (ca 75.6°) with the plane. The smaller dihedral angle of the pyridyl ring is thought to originate in the intramolecular C—H···O hydrogen bond and also in the effective conjugation between the nitrogen lone pair and the ring.

In diarylacetamide derivatives, the dihedral angles of the aryl groups with the amide plane indicate a tendency that the relatively electron-deficient ring has a smaller angle [1, 8, 9]. However, in **1**, the electron-deficient pyridyl ring still has a relatively large dihedral angle which differs from that

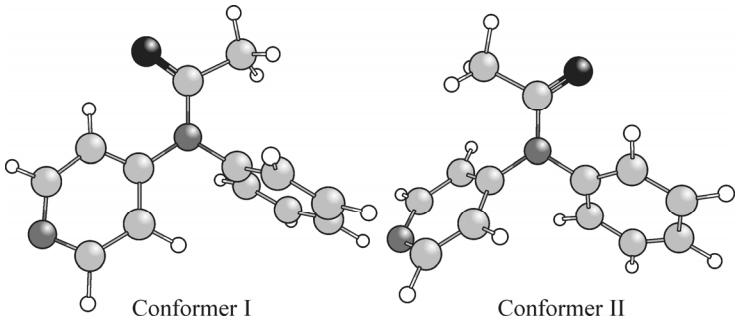

Fig. 2. Two stable conformers of **1** obtained by DFT calculations (GAMESS) at the B3LYP 6-31G(*d*) level. [6, 7]

in the related or optimized structures. This is presumably because the intermolecular hydrogen bonds, the π···π stacking interaction, and the C—H···π interactions stabilize the crystal more effectively.

Conclusions. We have succeeded in the crystal structure determination of **1**. The structure is not very different from that of the related or optimized structures, because the orientation of the C=O group is the same, namely the carbonyl oxygen atom is close to an electron-deficient ring as in literature and calculated structures. Only the interrelation of the dihedral angles of the amide plane with the benzene and pyridine rings is different. The reason for a such thing cannot be explained only by the intermolecular hydrogen bonds and the π···π stacking interaction because there are similar interactions in the referred structures of *N*-(4-nitrophenyl)-*N*-phenylacetamide from [8] and *N*-(4-methoxyphenyl)-*N*-(4-nitrophenyl)acetamide from [9]. This problem is very complicated and the most plausible reason lies in the interplay of intermolecular interactions and the close-packing principle.

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