

Rafal Kruszynski,^{a*}
Agata Trzesowska,^a
Wojciech Czestkowski,^b
Tadeusz J. Bartczak^a and
Elżbieta Mikiciuk-Olasik^c

^aInstitute of General and Ecological Chemistry, Technical University of Łódź, ul. Żeromskiego 116, 90-924 Łódź, Poland, ^bInstitute of Organic Chemistry, Technical University of Łódź, ul. Żeromskiego 116, 90-924 Łódź, Poland, and ^cDepartment of Chemical Technology of Drugs, Medical University of Łódź, ul. Muszyńskiego 1, 90-145 Łódź, Poland

Correspondence e-mail:
rafal.kruszynski@p.lodz.pl

Key indicators

Single-crystal X-ray study
 $T = 291$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
Disorder in main residue
 R factor = 0.044
 wR factor = 0.155
Data-to-parameter ratio = 16.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

4-(2-Benzylaminoacetyl)-1,2,3,4-tetrahydroquinoxalin-2-one

All interatomic distances of the title compound, $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_2$, are normal. The phenyl ring of the benzylaminomethyl substituent is disordered over two positions. The heteroatom ring of the quinoxalinone system shows a half-chair conformation. The molecules are held together by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds resulting in a chain along the crystallographic c axis. The chains are linked *via* weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds into a three-dimensional hydrogen-bonded network.

Comment

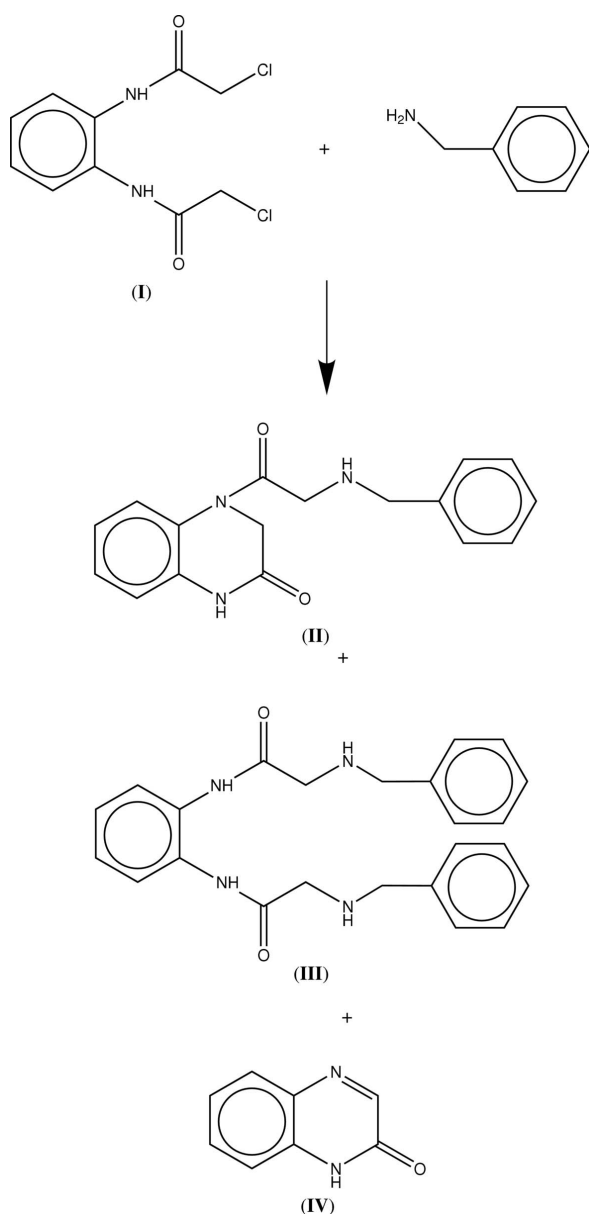
The present work is a continuation of our studies on derivatives of 2,3,4,5,6,7-hexahydro-1*H*-1,4,7-benzotriazine-2,5-dione. Upon introduction of pharmacophoric substituents for a desired activity into those systems, the derivatives exhibit favourable properties such as analeptic activity or anticancer and anti-HIV activities (Bartczak *et al.*, 1995). Fused ten-membered hetero rings with pendant arms possess catalytic properties comparable to the activity of enzymes (Hanson & Jakubke, 1973; Ivanov, 1975). The possibility of linking metal ions to form stable complexes allows the utilization of heterocyclic systems with pendant arms for modelling the cations receptors in proteins (Ovchinnikov, 1974; Müller, 1974). Many similar systems are also useful in environmental protection, in medicinal therapies using complexones, and in the treatment of poisoning with heavy and radioactive metals (Bandot & Jacque, 1977; Num *et al.*, 1983).

Within this area of research, a series of derivatives have been obtained (Mikiciuk-Olasik, 1990; Mikiciuk-Olasik *et al.*, 1993, 1994; Szadowska *et al.*, 1991), but only three structures of these compounds were previously determined: *N,N'*-bis-[2-(4-ethoxyphenyl)amino]-4,5-dimethyl-*o*-phenylenediamine (Kruszynski *et al.*, 2001), 4-[(4-ethoxyphenyl)aminoacetyl]-1,2,3,4-tetrahydroquinoxalin-2-one, hereafter (IIA) (Kruszynski *et al.*, 2002*a*), and 4-(2-benzylamino-1-oxoethyl)-1,2,3,4-tetrahydro-6,7-dimethyl-quinoxalin-2-one, hereafter (IIB) (Kruszynski *et al.*, 2002*b*).

The perspective view of the title compound, (II), together with the atom numbering scheme is shown in Fig. 1. All interatomic distances can be considered as normal. The phenyl group of the benzylaminomethyl substituent is disordered over two positions. The second domain is rotated by 23 (1)° about the vector through C14 and C17.

The molecular geometry of the fused ring system of (II) is comparable to those of (IIA) and (IIB). It is remarkable that the conformation of the side chains in (II) and (IIB) differs significantly (Table 2), although the molecules differ only in two methyl groups attached to the 1,2,3,4-tetrahydroquinoxalin-2-one moiety.

Received 13 August 2006
Accepted 16 August 2006



The heteroatom ring of the quinoxalinone system shows a half-chair conformation with a local pseudo-twofold axis along the mid-points of the N1–C8 and N2–C6 bonds (Duax & Norton, 1975; Duax *et al.*, 1976). The values of the asymmetry parameters are $\Delta C_2(N1-C8) = 2.17(7)$ and $\Delta C_3(N1) = 23.44(8)$. The value of the total puckering amplitude (Cremer & Pople, 1975) is $Q_T = 0.439(9)$ Å, which is close to $Q_T = 0.435(3)$ Å in (IIA) and $Q_T = 0.391(5)$ Å in (IIB). Atom C8 deviates by $0.4143(14)$ Å from the weighted least-squares plane calculated through all quinolinone atoms. Atoms O1 and C11 deviate by $0.085(2)$ and $1.187(2)$ Å, respectively, from the above plane. The weighted least-squares planes of the phenyl disordered domains make dihedral angles of $36.0(6)$ and $30.7(5)^\circ$ with the quinolinone plane.

The molecules of (II) are held together by N–H \cdots O intermolecular hydrogen bonds (Jeffrey & Saenger, 1994),

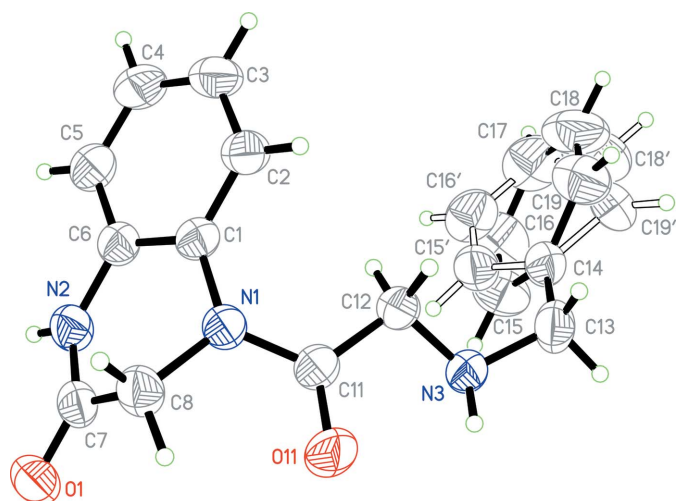


Figure 1
Molecular structure of (II). Displacement ellipsoids are drawn at the 50% probability level. **The minor disordered component of the benzyl ring is indicated by hollow bonds.**

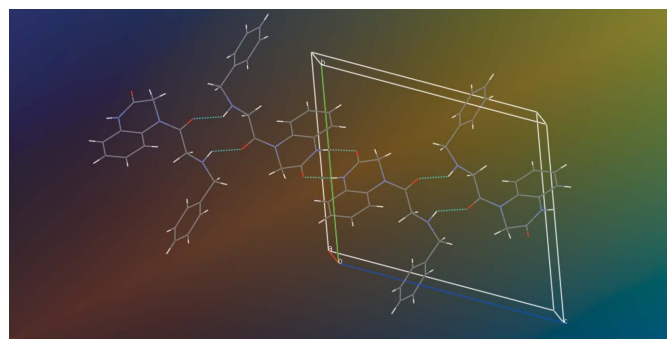


Figure 2
Part of the molecular packing of (II) showing N–H \cdots O intermolecular hydrogen bonds creating a chain structure. The minor disordered component of the benzyl ring has been omitted for clarity. Hydrogen bonds are indicated by dashed lines.

resulting in a chain along the crystallographic *c* axis (Table 1 and Fig. 2). The chains are linked by C–H \cdots O and C–H \cdots N short intermolecular interactions (Table 1), which can be considered as weak hydrogen bonds (Taylor & Kennard, 1982; Desiraju & Steiner, 1999). In this way, a three-dimensional hydrogen-bonded network is created.

Experimental

Compound (II) was prepared according to the method depicted in the scheme. To a stirred solution of chloroacetyl chloride (5.6 g, 0.02 mol) in dry chloroform (100 ml) with anhydrous Na_2CO_3 (10.6 g, 0.1 mol), *o*-phenylenediamine (2.16 g, 0.02 mol) was added in small portions at 277.2 K for 2 h. The following day, methyl alcohol (20 ml) was added. The reaction mixture was stirred and filtered. The precipitate was dried, washed with water and filtered off. The residue was recrystallized from ethyl alcohol and *N,N'*-bis(chloroacetyl)-*o*-phenylenediamine, (I), was obtained (6.3 g, 74% yield). M.p. 472.2 K. Elemental analysis (calculated/found): C 49.84/50.16%, H 4.88/5.259%, N 9.69/9.56%.

Compound (I) (0.01 mol) was dissolved in 500 ml of anhydrous ethanol with fine powdered sodium carbonate (0.07 mol). Benzylamine (0.01 mol) was then added. The reaction mixture was heated at boiling point under reflux for 10 h (Mikiciuk-Olasik, 1990; Mikiciuk-Olasik *et al.*, 1994). The solvent was distilled off and the dry residue was recrystallized from ethanol. Compound II (yield 40%, m.p. 609 K) and a small amount of (III) and (IV) were isolated by fractional crystallization of the solid residue.

Crystal data

C₁₇H₁₇N₃O₂ V = 760.5 (3) Å³
 M_r = 295.34 Z = 2
 Triclinic, P1 D_x = 1.290 Mg m⁻³
 a = 4.7010 (10) Å Cu Kα radiation
 b = 12.415 (2) Å μ = 0.70 mm⁻¹
 c = 14.083 (3) Å T = 291.0 (3) K
 α = 110.77 (3)° Prism, colourless
 β = 93.16 (3)° 0.35 × 0.31 × 0.27 mm
 γ = 96.04 (3)°

Data collection

Kuma KM-4 diffractometer 2835 reflections with I > 2σ(I)
 ω-2θ scans R_{int} = 0.025
 Absorption correction: ψ scan θ_{max} = 80.7°
 (North *et al.*, 1968) 3 standard reflections
 T_{min} = 0.784, T_{max} = 0.820 every 100 reflections
 3332 measured reflections intensity decay: 11.2%
 3191 independent reflections

Refinement

Refinement on F² w = 1/[σ²(F_o²) + (0.077P)²
 R[F² > 2σ(F²)] = 0.044 + 0.1995P]
 wR(F²) = 0.155 where P = (F_o² + 2F_c²)/3
 S = 1.13 (Δ/σ)_{max} < 0.001
 3191 reflections Δρ_{max} = 0.17 e Å⁻³
 199 parameters Δρ_{min} = -0.23 e Å⁻³
 H-atom parameters constrained

Table 1

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
N2—H2N...O1 ⁱ	0.88	2.00	2.879 (2)	175
N3—H3N...O11	0.90	2.25	2.6804 (18)	109
N3—H3N...O11 ⁱⁱ	0.90	2.36	3.056 (2)	134
C2—H2...N3 ⁱⁱⁱ	0.93	2.62	3.535 (2)	169
C8—H8B...O11	0.97	2.23	2.682 (2)	107
C17—H17...O1 ^{iv}	0.93	2.53	3.422 (3)	161

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

Table 2

Comparative torsion angles (°) for compound (II) and (IIB)..

Torsion angle	(II)	(IIB)
C8—N1—C11—C12	174.44 (13)	166.0 (5)
N1—C11—C12—N3	-153.67 (14)	-145.6 (5)
C11—C12—N3—C13	-163.52 (13)	65.3 (6)
C12—N3—C13—C14	-73.1 (2)	176.3 (4)
N3—C13—C14—C15	-45.8 (7)	-12.9 (8)
N3—C13—C14—C15'	-18.3 (6)	-

H atoms bonded to N atoms were located in difference Fourier syntheses. All H atoms were placed in calculated positions and refined as riding on the parent atom, with C—H = 0.93 and 0.97 Å, and N—H = 0.88 and 0.90 Å. U_{iso}(H) was set to 1.2U_{eq}(C,N). The site occupation factors of the disordered atoms of the phenyl ring refined to 0.552 (16)/0.480 (17).

Data collection: *KM-4 Software* (Kuma, 1993); cell refinement: *KM-4 Software*; data reduction: *DATAPROC* (Gałdecki *et al.*, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1990b) *ORTEP-3* (Version 1.062; Farrugia, 1997) and *MERCURY* (Version 1.4; Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

This work was financed by funds allocated by the Ministry of Scientific Research and Information Technology to the Institute of General and Ecological Chemistry, Technical University of Łódź, Poland.

References

Bandot, Ph. & Jacque, M. (1977). *Toxicol. Appl. Pharmacol.* **41**, 13–14.
 Bartzak, T. J., Kajkowski, T., Trzebinski, P., Mikiciuk-Olasik, E. & Kotelko, B. (1995). *Heteroat. Chem.* **6**, 495–498.
 Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
 Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*. Oxford University Press.
 Duax, W. L. & Norton, D. A. (1975). *Atlas of Steroid Structures*, Vol. 1, pp. 16–22. New York: IFI/Plenum.
 Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by N. L. Allinger & E. L. Eliel, pp. 271–383. New York: John Wiley.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Gałdecki, Z., Kowalski, A. & Uszynski, I. (1998). *DATAPROC*. Version 10.0.4. Kuma Diffraction, Wrocław, Poland.
 Hanson, H. & Jakubke, H. D. (1973). *Peptides, 1972 Proceedings of the Twelfth European Peptide Symposium*. New York: Elsevier.
 Ivanov, W. J. (1975). *Ann. N. Y. Acad. Sci.* **246**, 221–222.
 Jeffrey, G. A. & Saenger, W. (1994). *Hydrogen Bonding in Biological Structures*. Heidelberg: Springer-Verlag.
 Kruszynski, R., Bartzak, T. J. & Mikiciuk-Olasik, E. (2001). *Acta Cryst.* **E57**, o953–o955.
 Kruszynski, R., Bartzak, T. J. & Mikiciuk-Olasik, E. (2002a). *Acta Cryst.* **E58**, o98–o101.
 Kruszynski, R., Bartzak, T. J. & Mikiciuk-Olasik, E. (2002b). *Acta Cryst.* **E58**, o461–o464.
 Kuma (1993). *KM-4 Software*. Kuma Diffraction, Wrocław, Poland.
 Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
 Mikiciuk-Olasik, E. (1990). *Pharmazie*, **45**, 436–437.
 Mikiciuk-Olasik, E., Kajkowski, T. & Bartzak, T. J. (1993). *Pharmazie*, **48**, 523–525.
 Mikiciuk-Olasik, E., Trzebinski, P., Nowak, R. & Kotelko, B. (1994). *Acta Pol. Pharm. Drug Res.* **51**, 231–233.
 Müller, W. A. (1974). *Naturwissenschaften*, **61**, 455–456.
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
 Num, A. D., Loberg, M. D. & Conley, R. A. (1983). *J. Nucl. Med.* **24**, 23–24.
 Ovchinnikov, Y. A. (1974). *Membrane Active Complexones*. Amsterdam: Elsevier.
 Sheldrick, G. M. (1990a). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1990b). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
 Szadowska, A., Pakulska, W. & Mikiciuk-Olasik, E. (1991). *Pharmazie*, **46**, 544–545.
 Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.