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Key indicators

Single-crystal X-ray study
T = 291 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.058
wR factor = 0.132
Data-to-parameter ratio = 16.9

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

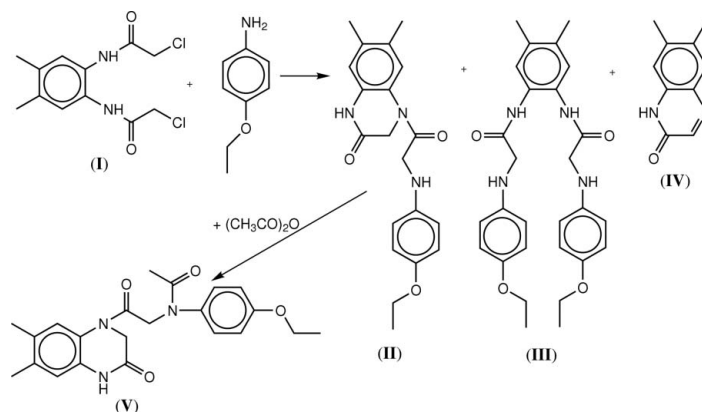
4-[(4-Ethoxyphenyl)aminoacetyl]-6,7-dimethyl-1,2,3,4-tetrahydroquinoxalin-2-one

All the interatomic distances in the title compound, $\text{C}_{22}\text{H}_{25}\text{N}_3\text{O}_4$, are normal. The heteroatom ring of the quinoxalinone system shows a half-chair conformation, slightly distorted towards a sofa. In the crystal structure, the molecules dimerize *via* a pair of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The dimers are linked by $\text{C}-\text{H}\cdots\text{O}$ and possible $\text{C}-\text{H}-\pi$ interactions into a three-dimensional network.

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Comment

The present work is a continuation of our studies of derivatives of 2,3,4,5,6,7-hexahydro-1*H*-1,4,7-benzotriazine-2,5-dione (see, for example, Mikiciuk-Olasik *et al.*, 1994). Such compounds possess important pharmacological properties (see, for example, Bartczak *et al.*, 1995) and also have applications in environmental protection (Num *et al.*, 1983).



The crystal structures of four derivatives of this family have been described previously, namely *N,N'*-bis(2-(4-ethoxyphenyl)amino)-4,5-dimethyl-*o*-phenylenediamine, (III) (Kruszynski *et al.*, 2001), 4-[(4-ethoxyphenyl)aminoacetyl]-1,2,3,4-tetrahydroquinoxalin-2-one, (VA) (Kruszynski *et al.*, 2002*a*), 4-(2-benzylamino-1-oxoethyl)-1,2,3,4-tetrahydro-6,7-dimethylquinoxalin-2-one (Kruszynski *et al.*, 2002*b*) and 4-(2-benzylamino-1-oxoethyl)-1,2,3,4-tetrahydroquinoxalin-2-one (Kruszynski *et al.*, 2006). We now describe the synthesis and crystal structure of the title compound, (V) (Fig. 1), in which all the interatomic distances can be considered normal.

The molecular geometry of the fused ring system of (V) is comparable with those of the above-mentioned compounds. The puckered heteroatom ring (C1/C6–C8/N1/N2) in (V) shows a half-chair conformation, slightly distorted towards a sofa, with a local pseudo-twofold axis passing through the midpoints of the N1–C8 and N2–C6 bonds. The values of the asymmetry parameters (Duax & Norton, 1975) are $\Delta\text{C}_2(\text{N1}-$

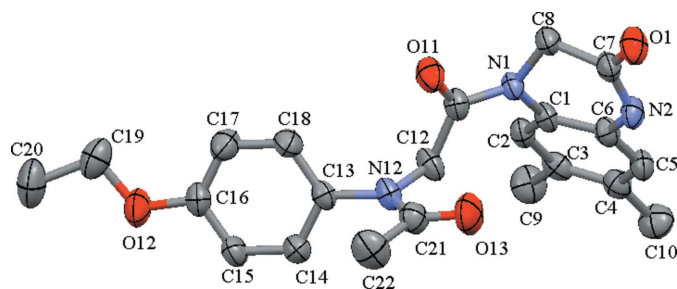


Figure 1
The molecular structure of (V). Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

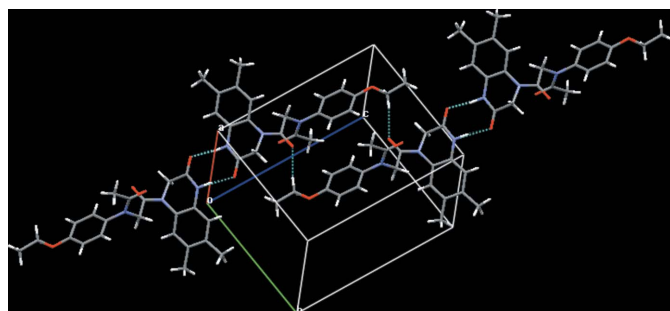


Figure 2
Part of the molecular packing of (V), showing intermolecular N—H...O and C—H...O hydrogen bonds (dashed lines) creating a chain structure.

C8) = 10.00 (4)° and $\Delta C_s(N1) = 17.51$ (7)°. The total puckering amplitude $Q_T = 0.421$ (7) Å (Cremer & Pople, 1975) lies within the range of 0.391–0.439 Å found for the above-mentioned similar compounds. Atomic deviations from the mean plane defined by atoms C1–C6/N1/N2 are: C7 0.441 (5), C8 0.767 (5), O1 0.586 (6), C9 0.034 (10), C10 – 0.188 (10) and C11 – 0.892 (4) Å. The aromatic ring of the ethoxyphenyl unit makes a dihedral angle of 43.19 (9)° with the quinolinone plane (N1/N2/C1–C8).

The conformations of the side chains in (V) and (VA) show some significant differences, although the molecules differ only in two methyl groups attached to the 1,2,3,4-tetrahydroquinoxalin-2-one unit. For example, the C12–N12–C13–C14 torsion angle in (V) is 104.2 (3)°, and the equivalent angle in (VA) is 75.5 (4)°. The C11–C12–N12–C13 angle in (V) is 109.7 (3)°, while that in (VA) is 93.1 (3)°.

The molecules of (V) create dimers *via* inversion symmetry-generated pairs of N—H...O hydrogen bonds (Table 1). These dimers are linked by C—H...O interactions into chains propagating along the [111] axis (Fig. 2). A very acute intramolecular C—H...O bond and two weak intermolecular C—H... π interactions (Table 1) may also affect the crystal packing of (V).

Experimental

Compound (V) was prepared according to the method depicted in the scheme. Compound (I) (Kruszynski *et al.*, 2006) (0.02 mol) was dissolved in anhydrous ethanol (500 ml) with fine-powdered sodium carbonate (0.07 mol). 1-Amino-4-ethoxybenzene (0.02 mol) was then

added and the reaction mixture was heated at boiling point under reflux for 10 h. The solvent was then distilled off and the dry residue was recrystallized from ethanol. Compound (II), and small amounts of compounds (III) and (IV), were isolated by fractional crystallization of the solid residue. Compound (II) (0.01 mol) was dissolved in ethanoic anhydride (40 ml) and the mixture was heated at boiling point for 1 h. The solvent was then distilled off and the dry residue was recrystallized from ethanol to yield crystals of (V). Elemental analysis, calculated for $C_{22}H_{25}N_3O_4$ (found): C 66.82 (66.65), H 6.34 (6.32), N 10.63 (10.69), O (by difference) 16.21 (15.99)%. Yield 85%, m.p. 496–497 K. 1H NMR (in DMSO/TMS, δ , p.p.m.): 1.55 [*t*, 3H, $CH_3(Et)$, $J = 7$ Hz], 1.95 (*s*, 3H, CH_3CO), 2.4 (*s*, 6H, 2 CH_3 -ar), 4.25 [*q*, 2H, $CH_2(Et)$, $J = 7$ Hz], 4.5 (*s*, 2H, ar-NH CH_2CON -ar), 4.75 (*s*, 2H, ar-NH CH_2CON -ar), 7.25 [*m*, 6H, H(ar)], 11.00 (*s*, 1H, NHCO).

Crystal data

$C_{22}H_{25}N_3O_4$	$V = 1031.1$ (4) Å ³
$M_r = 395.45$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.274$ Mg m ⁻³
$a = 6.634$ (1) Å	Mo $K\alpha$ radiation
$b = 10.547$ (2) Å	$\mu = 0.09$ mm ⁻¹
$c = 15.276$ (3) Å	$T = 291.0$ (3) K
$\alpha = 94.21$ (3)°	Needle, colourless
$\beta = 90.10$ (3)°	$0.35 \times 0.11 \times 0.07$ mm
$\gamma = 104.64$ (3)°	

Data collection

Kuma KM-4 diffractometer	1787 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{int} = 0.054$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$\theta_{max} = 27.6^\circ$
$T_{min} = 0.971$, $T_{max} = 0.996$	3 standard reflections
4993 measured reflections	every 100 reflections
4518 independent reflections	intensity decay: 4.2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.058$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.132$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.87$	$\Delta\rho_{max} = 0.18$ e Å ⁻³
4518 reflections	$\Delta\rho_{min} = -0.20$ e Å ⁻³
267 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	(Sheldrick, 1997)
	Extinction coefficient: 0.018 (2)

Table 1

Hydrogen-bond geometry (Å, °).

$Cg1$ and $Cg2$ are the centroids of the C1- and C13-containing rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2–H2N...O1 ⁱ	0.99	1.88	2.852 (3)	166
C8–H8A...O11	0.97	2.30	2.736 (3)	107
C19–H19B...O11 ⁱⁱ	0.97	2.46	3.380 (4)	158
C15–H15...Cg1 ⁱⁱⁱ	0.93	2.83	3.652 (5)	148
C22–H22C...Cg2 ^{iv}	0.96	2.72	3.474 (5)	136

Symmetry codes: (i) $-x + 3, -y + 2, -z + 2$; (ii) $-x + 2, -y + 1, -z + 1$; (iii) $-x + 2, -y + 2, -z + 1$; (iv) $x + 1, y, z$.

The N-bound H atom was located in a difference map and refined as riding in its as-found relative position. The C-bound H atoms were positioned geometrically (C–H = 0.93–0.99 Å) and refined as riding. The methyl groups were allowed to rotate about their local threefold axis. $U_{iso}(H)$ was set equal to $1.2U_{eq}(\text{carrier})$ or $1.5U_{eq}(\text{methyl carrier})$.

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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1990) and *MERCURY* (Version 1.4; Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997) and *PLATON* (Spek, 2003).

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