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8-Chloro-6-phenyl-4*H*-[1,2,4]-triazolo[4,3-*a*][1,5]benzodiazepin-5(6*H*)-one

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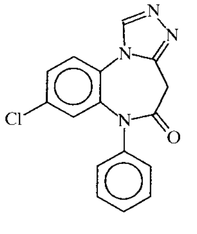
The title compound, $C_{16}H_{11}ClN_4O$, is an anticonvulsant agent. In the crystal, a particularly short C—H···N intermolecular hydrogen bond is noted [H···N 2.22 (2) Å]. The diazepine ring has a boat conformation.

Comment

The title compound, indicated here as RL202, was previously reported to exhibit anticonvulsant activity (Bauer *et al.*, 1974; Fryer *et al.*, 1978). Its crystal structure has now been determined as part of an investigation on the relationships between molecular properties and pharmacological activities of [1,2,4]triazolo[4,3-*a*][1,5]benzodiazepin-5-amines with different substitution patterns (Di Braccio *et al.*, 1990; Roma *et al.*, 1991; Grossi *et al.*, 1993).



RL202

To our knowledge, the present work reports for the first time the crystal structure of a [1,2,4]triazolo[4,3-*a*][1,5]-benzodiazepinone. The structure of RL202 has been solved by direct methods (Gabe *et al.*, 1989). An *ORTEP*II view (Johnson, 1976) is shown in Fig. 1. The diazepine ring has a boat conformation; the asymmetry parameters (*PARST*; Nardelli, 1983, 1995) give evidence of a pseudo mirror plane passing through C4 [$\Delta C_s = 0.0420$ (4)]. The extent of the boat conformation can be described in terms of the (acute) dihedral angles between the central plane, defined by atoms N11, C3a, C5, N6 and the 'bow' and 'stern' planes, defined by atoms C3a, C4, C5 and C6a, C10a, N11, N6, respectively (Hamor & Martin, 1983). The bow and stern angles in RL202 [55.1 (1) and 37.1 (1)°] compare well with analogous values obtained for [1,2,4]triazolo[4,3-*a*][1,4]benzodiazepines, where the ranges for bow angles and stern angles are 53.4–55.5 and 31.2–36.2°, respectively (Hamor & Martin, 1983). For Clobazam, an important 1,5-benzodiazepinedione (Butcher & Hamor, 1985), the corresponding values are 59.4 (5) and 40.7 (5)°, respectively.

The main conformational parameter of RL202 is the torsion angle of the non-condensed phenyl ring. Its rather high value, 57.3 (2)° for the C6a—N6—C1′—C6′ torsion angle, can be referred back to the steric hindrance with neighbouring atoms [C2′···O 2.929 (2), C6′···C7 3.300 (2) Å]. In order to investigate the effects of packing forces on the observed conformation, the geometry of the isolated molecule has been calculated with *ab initio* molecular orbital methods at the HF/6-31G* level (Hariharan & Pople, 1973; full optimization, 356 basis functions). In this case, the internal strain relaxes through an even larger torsion angle of the phenyl ring (68.4°); the two non-bonded distances become 2.97 and 3.53 Å, respectively.

Intermolecular contacts in the crystal state correspond to van der Waals interactions, with the exception of a stronger C—H···N hydrogen bond [C1—H1 1.00 (2), C1···N3 3.191 (2), H1···N3 2.22 (2) Å, C1—H1···N3 163.7(15)°, N3 in $x, \frac{3}{2} - y, -\frac{1}{2} + z$] and of a weaker C—H···O interaction [C6′—H6′ 0.94 (2), C6′···O 3.371 (2), H6′···O 2.45 (2) Å, C6′—H6′···O 166.5(17)°, O in $x, \frac{3}{2} - y, -\frac{1}{2} + z$]. According to a recent review on C—H···N hydrogen bonds (Mascal, 1998), the present H···N distance locates on the short side of the distribution.

Atomic charges have been evaluated for the experimental molecule with re-normalized C—H bond distances; the calculations have been performed at the HF/6-31G* level with the method of the best-fit to the electrostatic potential. The resulting values for exposed heteroatoms in the molecule (O

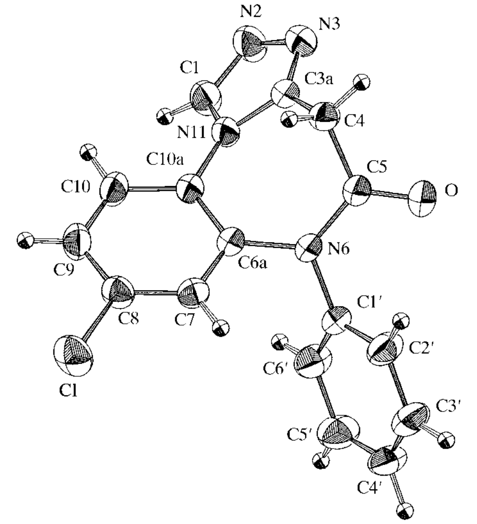


Figure 1
A molecule of RL202 with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are on an arbitrary scale.

−0.59, N3 −0.39, N2 −0.34, Cl −0.13 a.u.), along with the packing geometry, support the observed interactions. Charges calculated for H1 and H6′ are 0.14 and 0.17 a.u., respectively.

Experimental

The compound RL202 was prepared by the method of Grossi *et al.* (1993) and was crystallized from methanol.

Crystal data

$C_{16}H_{11}ClN_4O$
$M_r = 310.74$
Monoclinic, $P2_1/c$
$a = 16.983$ (4) Å
$b = 8.863$ (2) Å
$c = 9.784$ (4) Å
$\beta = 103.67$ (3)°
$V = 1431.0$ (8) Å^3
$Z = 4$
$D_x = 1.442$ Mg m^{-3}
$D_m = 1.441$ Mg m^{-3}
D_m measured by flotation
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 16.5$–19.7°
$\mu = 0.274$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.54 × 0.46 × 0.40 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.823$, $T_{\max} = 0.896$
4161 measured reflections
4161 independent reflections
3462 reflections with $F_o > 4\sigma(F_o)$
$\theta_{\max} = 29.89°$
$h = 0 \rightarrow 23$
$k = 0 \rightarrow 12$
$l = -13 \rightarrow 13$
2 standard reflections
frequency: 90 min
intensity decay: 5%

Refinement

Refinement on F^2
$R(F) = 0.040$
$wR(F^2) = 0.110$
$S = 1.038$
4157 reflections
243 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.3463P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

C1—N2	1.304 (2)	C4—C5	1.512 (2)
C1—N11	1.365 (2)	C5—N6	1.383 (2)
N2—N3	1.395 (2)	N6—C6a	1.430 (2)
N3—C3a	1.302 (2)	C6a—C10a	1.400 (2)
C3a—C4	1.480 (2)	C10a—N11	1.417 (2)
C3a—N11	1.367 (2)		
N2—C1—N11	110.4 (1)	C4—C5—N6	116.0 (1)
C1—N2—N3	107.2 (1)	C5—N6—C6a	124.7 (1)
N2—N3—C3a	107.2 (1)	N6—C6a—C10a	123.7 (1)
N3—C3a—C4	128.8 (1)	C6a—C10a—N11	120.3 (1)
N3—C3a—N11	110.4 (1)	C1—N11—C3a	104.8 (1)
C4—C3a—N11	120.6 (1)	C3a—N11—C10a	124.9 (1)
C3a—C4—C5	108.1 (1)		
N11—C3a—C4—C5	64.4 (2)	C5—N6—C1′—C2′	54.6 (2)
C4—C3a—N11—C10a	9.2 (2)	C6a—N6—C1′—C6′	57.3 (2)
C3a—C4—C5—N6	−68.3 (1)	N6—C6a—C10a—N11	−7.2 (2)
C4—C5—N6—C6a	−1.4 (2)	C6a—C10a—N11—C3a	−43.5 (2)
C5—N6—C6a—C10a	48.9 (2)		

In the final least-squares cycles, zero weight was given to four low-angle reflections, as affected by extinction or experimental error. All the H atoms were localized from difference syntheses and refined isotropically. The range of resulting C—H bond lengths is 0.92 to 1.01 Å with a nearly constant s.u. of 0.02 Å. No correlation matrix elements larger than 0.50 were found.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989) and *CADABS* (local software); program(s) used to solve structure: *NRCVAX*; program(s) used to refine structure: *NRCVAX* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *PARST* (Nardelli, 1983, 1995) and *PARSTCIF* (Nardelli, 1991); molecular orbital methods: *SPARTAN* (Wavefunction Inc., 1998).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1441). Services for accessing these data are described at the back of the journal.

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