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(Z)-1-Ethyl-3-(4-chlorobenzylidene)-1,2,3,5-tetrahydroimidazo[2,1-*b*]quinazoline-2,5-dione

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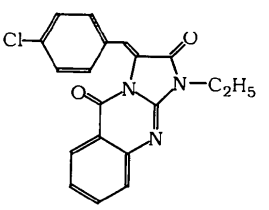
Abstract

The central part of the title molecule, $C_{19}H_{14}ClN_3O_2$, comprises three N atoms bonded to C2(sp^2). The bond lengths of C2 with N1 and N3 (sp^2 N atoms) are comparable [1.371 (5) and 1.392 (6) Å, respectively], while the C2—N4(sp) bond is shorter [1.285 (5) Å]. The main skeleton of the molecule (three condensed π-electron rings inclined slightly towards one another) and the phenyl ring have a *Z* configuration with torsion angle N1—C5—C6—C16 = −8.5 (9)°. Thus, as a result of steric hindrance, the imidazo–quinazolinedione skeleton and the phenyl ring are not coplanar, and form a dihedral angle of 48.1 (1)°.

Comment

In our previous paper, we briefly summarized our structural studies on different hydantoin derivatives with varying biological activities (Karolak-Wojciechowska & Kieć-Kononowicz, 1994). As an extension of our search for new anticonvulsants, we studied arylideneimidazoquinazolinedione derivatives, the molecules being 'enriched' by attaching an additional aromatic ring to the rigid part of the annelated skeleton. The general aim of our project is structure–activity correlations. Since these correlations can be based on structural and electronic parameters derived from a geometrical description of the molecule (Konschin, Tylli, Gynther & Rouvinen, 1989; Diaz-Arauzo, Koehler, Hagen & Cook, 1991), the three-dimensional structures and conformations of all the investigated molecules have to be determined. This prompted us to solve the structure of the title compound, (I), as a crucial material for the molecular modelling of the remaining seven molecules of this class. These data were used for calculation and comparison of the electrostatic potential distribution around the active and inactive molecules (Karolak-Wojciechowska, Kwiatkowski & Kieć-Kononowicz, 1995). The conformation of the molecule together with the atomic numbering is depicted in Fig. 1.



(I)

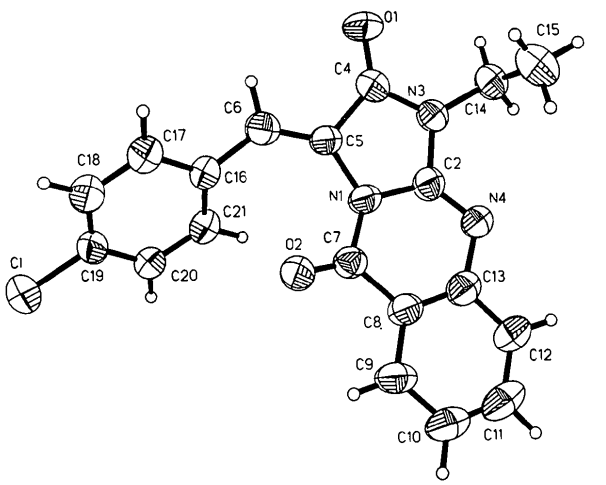


Fig. 1. Molecular structure and atomic numbering showing 50% probability displacement ellipsoids.

Experimental

The preparation of the title compound was performed by Kieć-Kononowicz (1993). The crystals were grown by slow evaporation of a DMF solution.

Crystal data

$C_{19}H_{14}ClN_3O_2$
M_r = 351.8
Monoclinic
$P2_1/c$
a = 11.918 (4) Å
b = 12.296 (3) Å
c = 11.193 (5) Å
β = 90.65 (3)°
V = 1640.1 (5) Å^3
Z = 4
D_x = 1.425 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54184 Å
Cell parameters from 25 reflections
θ = 10–60°
μ = 2.22 mm^{-1}
T = 293 K
Needle
0.4 × 0.2 × 0.2 mm
Light yellow

Data collection

Kuma KM-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3671 measured reflections
2742 independent reflections
1893 observed reflections [$F > 4\sigma(F)$]
R_{int} = 0.038
θ_{max} = 65°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 13$
$l = -14 \rightarrow 14$
2 standard reflections monitored every 50 reflections
intensity decay: 10%

Refinement

Refinement on F
$R = 0.072$
$wR = 0.067$
$S = 4.84$
1893 reflections
227 parameters
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}
Extinction correction:
$F_c^* = F_c(1 - gF_c^2/\sin\omega)$
Extinction coefficient:
$g = 0.0662$
Atomic scattering factors from *SHELXTL/PC* (Sheldrick, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cl	0.5265 (1)	0.6431 (2)	0.0978 (1)	0.092 (1)
O1	1.0741 (3)	0.9032 (3)	−0.3418 (3)	0.071 (1)
O2	0.9412 (3)	0.8881 (3)	0.1057 (3)	0.060 (1)
N1	1.0770 (3)	0.8639 (3)	−0.0335 (3)	0.048 (1)
N3	1.1966 (3)	0.8946 (3)	−0.1826 (3)	0.051 (1)
N4	1.2729 (3)	0.8834 (3)	0.0117 (3)	0.055 (1)
C2	1.1879 (3)	0.8794 (4)	−0.0598 (4)	0.049 (1)
C4	1.0923 (4)	0.8906 (4)	−0.2358 (4)	0.055 (1)
C5	1.0112 (4)	0.8652 (4)	−0.1406 (4)	0.050 (1)
C6	0.9055 (4)	0.8432 (4)	−0.1649 (4)	0.052 (1)
C7	1.0393 (4)	0.8782 (4)	0.0848 (4)	0.047 (1)
C8	1.1323 (4)	0.8783 (4)	0.1702 (4)	0.050 (1)
C9	1.1072 (4)	0.8823 (4)	0.2927 (4)	0.061 (1)
C10	1.1937 (5)	0.8818 (4)	0.3750 (4)	0.070 (2)
C11	1.3026 (5)	0.8763 (5)	0.3378 (5)	0.076 (2)
C12	1.3301 (4)	0.8752 (5)	0.2183 (4)	0.067 (1)
C13	1.2415 (4)	0.8763 (4)	0.1331 (4)	0.054 (1)
C14	1.3006 (4)	0.9200 (5)	−0.2425 (4)	0.065 (2)
C15	1.3261 (5)	1.0405 (6)	−0.2337 (5)	0.091 (2)
C16	0.8146 (3)	0.7961 (4)	−0.0952 (4)	0.048 (1)
C17	0.7059 (4)	0.8300 (4)	−0.1175 (4)	0.058 (1)
C18	0.6172 (4)	0.7848 (5)	−0.0558 (4)	0.063 (1)
C19	0.6375 (4)	0.7008 (5)	0.0228 (4)	0.057 (1)
C20	0.7438 (4)	0.6644 (4)	0.0435 (4)	0.059 (1)
C21	0.8325 (4)	0.7112 (4)	−0.0145 (4)	0.054 (1)

Table 2. *Selected geometric parameters* (Å, °)

O1—C4	1.214 (6)	C4—C5	1.480 (7)
O2—C7	1.201 (6)	C5—C6	1.314 (7)
N1—C2	1.371 (5)	C7—C8	1.455 (7)
N1—C5	1.425 (6)	C8—C9	1.408 (6)
N1—C7	1.414 (6)	C8—C13	1.371 (7)
N3—C2	1.392 (6)	C9—C10	1.375 (7)
N3—C4	1.373 (6)	C10—C11	1.369 (8)
N3—C14	1.450 (6)	C11—C12	1.381 (7)
N4—C2	1.285 (5)	C12—C13	1.415 (7)
N4—C13	1.416 (6)		
C2—N1—C5	109.9 (4)	N1—C2—N3	108.3 (4)
C2—N1—C7	120.1 (4)	N1—C2—N4	128.8 (4)
C5—N1—C7	127.5 (4)	N3—C2—N4	122.9 (4)
C2—N4—C13	112.4 (4)		
N1—C5—C6—C16	−8.5 (9)		

Table 3. *Dihedral angles between mean planes* (°)

Plane *A*: N1, C2, N3, C4, C5. Plane *B*: N1, C2, N4, C13, C8, C7. Plane *C*: C8, C9, C10, C11, C12, C13.

A/B	9.8 (2)
A/C	12.1 (2)
B/C	4.5 (2)

The structure was refined by full-matrix least squares. The positions of all the H atoms were found from a $\Delta\rho$ map and refined using a riding model with fixed isotropic displacement parameters ($1.5U_{eq}$ of the parent atom).

Structure solution: *SHELXTL/PC* (Sheldrick, 1990). Structure refinement: *SHELXTL/PC*. Molecular graphics: *SHELXTL/PC*. Preparation of material for publication: *SHELXTL/PC*.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KA1109). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-(2-Chlorobenzyl)-6-methyl-3(2*H*)-pyridazinone

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Abstract

The crystal and molecular structure of the title compound, $C_{12}H_{11}ClN_2O$, has been determined as part of an investigation into quantitative structure–activity