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Methyl 4-(Benzylamino)-6-methyl-2-oxo-3-cyclohexene-1-carboxylate, $C_{16}H_{19}NO_3$

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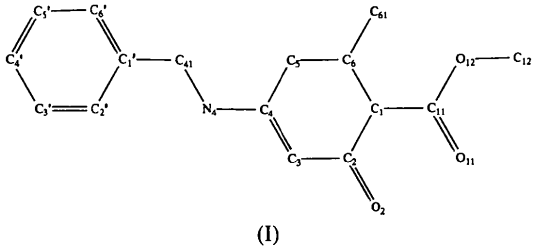
Abstract

The cyclohexene ring adopts an almost ideal sofa conformation. The bond lengths of the C=C—C=O fragment indicate strong conjugation through these bonds. Other planar fragments of the molecule, *i.e.* the phenyl ring and the ester group, make angles of 60.82 (4) and 75.20 (6)°, respectively, with the plane of the cyclohexene ring. The methyl substituent occupies an equatorial position. In the crystal structure, the intermolecular N—H···O hydrogen bonds form infinite chains of molecules along the [010] direction.

Comment

The title compound (I) has been found to exhibit anticonvulsant activity with a remarkable lack of neurotoxicity. The protective index, PI (defined as the ratio of the median toxic dose, TD_{50}, to the median effective dose, ED_{50}), was found to be 7.7 in mice and 18.7 in rats (Edafiogho *et al.*, 1992). These values compare favourably with the respective values for currently marketed anticonvulsants.



(I)

The cyclohexene ring exists in a sofa conformation with atoms C1–C5 almost perfectly coplanar and the largest deviation from the least-squares plane being 0.006 (1) Å; atom C6 deviates 0.618 (2) Å from this plane. A small value for the asymmetry parameter ΔC_s^3 (Duax & Norton, 1975) of 0.75 indicates only minor distortion from an ideal sofa conformation. Also, the values of the intra-annular torsion angles are close to the values calculated by Bucourt & Hainaut (1965) for this conformation. The bond lengths C2—O2 and C3—C4 are significantly lengthened while the bond C2—C3 is shortened in comparison with typical values for an O=C—C=C group (Allen *et al.*, 1987; Abell, Allen, Bugg, Doyle & Raithby, 1988), and the fragment O2—C2—C3—C4—N4 is nearly planar. These features indicate significant conjugation in this fragment. The phenyl ring is planar with a maximum deviation of 0.006 (1) Å. The angle between the plane of the ester group O11=C11—O12—C12 and the plane of phenyl ring is 45.68 (7)°. The methyl group (C61) is in an equatorial position, as indicated by the values of the torsion angles C4—C5—C6—C61 and C2—C1—C6—C61 of −173.3 (1) and 172.9 (1)°, respectively. In the crystal structure, the molecules are connected by N4—H4···O2(x, $-1+y$, z) hydrogen bonds [N···O 2.930 (1), H···O 2.13 (1) Å, N—H···O 162 (1)°] to form infinite chains.

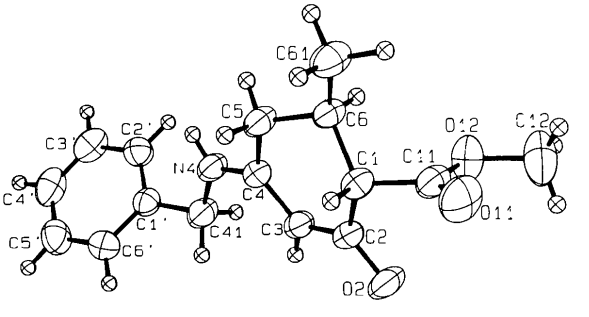


Fig. 1. Thermal ellipsoid representation of the molecule (Johnson, 1976) with the atomic numbering scheme. Anisotropic ellipsoids are drawn at the 50% probability level; H atoms are represented as spheres of arbitrary radii.

Experimental

Crystal data

$C_{16}H_{19}NO_3$
M_r = 273.32
Monoclinic
$P2_1/n$
a = 8.9562 (4) Å
b = 7.1033 (3) Å
c = 22.7440 (10) Å
β = 90.017 (4)°
V = 1446.94 (11) Å^3
Z = 4
D_x = 1.255 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 30.30–53.55°
μ = 0.701 mm^{-1}
T = 293 (2) K
Plate
0.35 × 0.35 × 0.10 mm
Colourless

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Data collection

CAD-4F diffractometer	R_{int} = 0.0241
$\omega/2\theta$ scans	θ_{max} = 74.87°
Absorption correction: none	$h = 0 \rightarrow 11$
3160 measured reflections	$k = 0 \rightarrow 8$
2967 independent reflections	$l = -28 \rightarrow 28$
2683 observed reflections $[I < 2\sigma(I)]$	3 standard reflections frequency: 2000 s intensity variation: 4.3%

Refinement

Refinement on F^2	$\Delta\rho_{max}$ = 0.207 e Å^{-3}
$R[F > 4\sigma(F)]$ = 0.0410	$\Delta\rho_{min}$ = −0.121 e Å^{-3}
$wR(F^2)$ = 0.1115	Extinction correction: *SHELXL92* (Sheldrick, 1993)
S = 1.085	Extinction coefficient: 0.004 (0)
2967 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
258 parameters	
All H-atom parameters refined	
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 0.2196P]$ where $P = (F_o^2 + 2F_c^2)/3$	

Data collection: CAD-4F. Cell refinement: CAD-4F. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL92* (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL92*.

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

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C1	0.90179 (14)	0.0325 (2)	0.58423 (5)	0.0425 (3)
C11	0.89133 (15)	0.1740 (2)	0.63345 (6)	0.0473 (3)
O11	0.98764 (13)	0.2810 (2)	0.64762 (5)	0.0701 (3)
C12	0.7313 (3)	0.3008 (4)	0.70549 (11)	0.0879 (6)
O12	0.75905 (12)	0.1641 (2)	0.66032 (5)	0.0648 (3)
C2	0.79022 (15)	0.0854 (2)	0.53615 (5)	0.0440 (3)
O2	0.76018 (14)	0.25507 (12)	0.52939 (5)	0.0639 (3)
C3	0.73021 (15)	−0.0582 (2)	0.50051 (5)	0.0438 (3)
C4	0.77063 (13)	−0.2447 (2)	0.50684 (5)	0.0400 (3)
N4	0.72052 (13)	−0.3800 (2)	0.47135 (5)	0.0461 (3)
C41	0.6254 (2)	−0.3418 (2)	0.42086 (6)	0.0546 (4)
C1′	0.61022 (13)	−0.5087 (2)	0.38074 (5)	0.0431 (3)
C2′	0.5491 (2)	−0.6772 (2)	0.39992 (6)	0.0520 (3)
C3′	0.5324 (2)	−0.8263 (2)	0.36136 (8)	0.0612 (4)
C4′	0.5753 (2)	−0.8084 (2)	0.30354 (7)	0.0660 (4)
C5′	0.6355 (2)	−0.6416 (3)	0.28411 (7)	0.0663 (4)
C6′	0.6540 (2)	−0.4937 (2)	0.32255 (6)	0.0545 (3)
C5	0.8804 (2)	−0.3034 (2)	0.55307 (6)	0.0461 (3)
C6	0.88543 (14)	−0.1711 (2)	0.60564 (5)	0.0421 (3)
C61	1.0114 (2)	−0.2240 (2)	0.64782 (7)	0.0587 (4)

Table 2. *Geometric parameters* (Å, °)

C1—C11	1.508 (2)	N4—C41	1.455 (2)
C1—C2	1.528 (2)	C41—C1′	1.502 (2)
C1—C6	1.533 (2)	C1′—C6′	1.385 (2)
C11—O11	1.194 (2)	C1′—C2′	1.387 (2)
C11—O12	1.335 (2)	C2′—C3′	1.383 (2)
C12—O12	1.436 (2)	C3′—C4′	1.376 (3)
C2—O2	1.2441 (15)	C4′—C5′	1.375 (3)
C2—C3	1.410 (2)	C5′—C6′	1.377 (2)
C3—C4	1.381 (2)	C5—C6	1.522 (2)
C4—N4	1.333 (2)	C6—C61	1.528 (2)
C4—C5	1.498 (2)		
C11—C1—C2	109.04 (10)	C4—N4—C41	122.73 (11)
C11—C1—C6	112.79 (10)	N4—C41—C1′	112.64 (11)
C2—C1—C6	113.40 (10)	C6′—C1′—C2′	118.64 (12)
O11—C11—O12	123.48 (13)	C6′—C1′—C41	119.62 (13)
O11—C11—C1	125.44 (13)	C2′—C1′—C41	121.71 (12)
O12—C11—C1	111.08 (11)	C3′—C2′—C1′	120.26 (14)
C11—O12—C12	116.5 (2)	C4′—C3′—C2′	120.3 (2)
O2—C2—C3	123.20 (12)	C5′—C4′—C3′	119.75 (14)
O2—C2—C1	117.93 (11)	C4′—C5′—C6′	120.06 (15)
C3—C2—C1	118.84 (10)	C5′—C6′—C1′	120.92 (15)
C4—C3—C2	122.32 (11)	C4—C5—C6	113.50 (10)
N4—C4—C3	122.70 (11)	C5—C6—C61	111.28 (11)
N4—C4—C5	116.44 (10)	C5—C6—C1	109.63 (10)
C3—C4—C5	120.80 (11)	C61—C6—C1	111.17 (11)
C6—C1—C2—C3	−25.1 (2)	C3—C4—C5—C6	26.1 (2)
C1—C2—C3—C4	−1.1 (2)	C4—C5—C6—C1	−49.88 (15)
C2—C3—C4—C5	0.7 (2)	C2—C1—C6—C5	49.39 (14)

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71403 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1068]

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