

2-Hydroxy-4,4-dimethyl-N-(4-methylphenyl)-6-oxocyclohexanylacetamide

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

Disorder in main residue

R factor = 0.045

wR factor = 0.127

Data-to-parameter ratio = 20.5

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

In the title compound, $\text{C}_{17}\text{H}_{21}\text{NO}_3$, the cyclohexene ring adopts an envelope conformation. The phenyl ring forms a dihedral angle of $35.77(4)^\circ$ with the amide plane. In the crystal, the symmetry-related molecules are linked through $\text{N}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{O}$ and weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, to form molecular networks parallel to (110) .

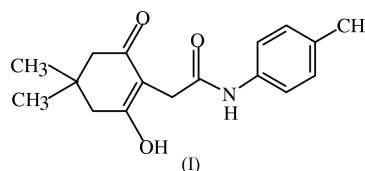
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Comment

An X-ray study of the title compound, (I), is of interest because some cyclohexane derivatives are used as reagents for the separation and identification of aldehydes (Horning & Horning, 1946). These derivatives are important chiral building blocks in the synthesis of natural products (Enders *et al.*, 2000). Also, these derivatives induce hydroxy chlorophyll in tobacco cell suspension cultures (Wang *et al.*, 2000).



The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. The C—C bond lengths in the phenyl ring are comparable with the overall average value [1.384 (13) Å] given by Allen *et al.* (1987). The bond lengths and angles observed in the cyclohexene ring are comparable with those of related structures (Semmingsen, 1974; Katrusiak, 1990, 1991, 1993; Bolte *et al.*, 1997). Atom N1 is sp^2 hybridized; the N1—C1 [1.420 (2) Å] and N1—C8 [1.341 (2) Å] bonds have lengths intermediate between single and double bonds.

The cyclohexene ring adopts an envelope conformation, as indicated by the lowest asymmetry parameter $\Delta_s(\text{C}13)$ of 0.045 (1); atom C13 deviates by 0.620 (1) Å from the weighted least-squares plane through atoms C10, C11, C12, C14 and C15 (Cremer & Pople, 1975; Nardelli, 1983). The phenyl and cyclohexane (excluding atom C13) rings form dihedral angles of $35.77(4)$ and $85.64(4)^\circ$, respectively, with the amide plane consisting of atoms N1, O1, C8 and C9. The torsion angles C1—N1—C8—O1 [$2.1(2)^\circ$] and N1—C8—C9—C10 [$143.1(1)^\circ$] indicate +synperiplanar and +antiperiplanar conformations for the phenyl and cyclohexene rings with respect to the central amide group. These conformations are influenced by the $\text{C}-\text{H}\cdots\text{O}$ intramolecular interactions involving atoms O1 and O3 (Table 2). In the crystal, the symmetry-related molecules are linked by $\text{N}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ intermolecular hydrogen bonds (Table 2 and Fig. 2), to form molecular networks parallel to the ab plane.

Experimental

Stirring dimedone with bromoacetic acid in the presence of K_2CO_3 in DMF for 24 h afforded 2-hydroxy-4,4-dimethyl-6-oxocyclohexanylacetic acid. This was stirred with $SOCl_2$ in the presence of a few drops of pyridine in CH_2Cl_2 . The resulting acid chloride was stirred with *p*-toluidine, in the presence of pyridine, to afford the title compound.

Crystal data

$C_{17}H_{21}NO_3$
 $M_r = 287.35$
 Monoclinic, $P2_1/n$
 $a = 10.1239$ (1) Å
 $b = 5.8366$ (1) Å
 $c = 26.9176$ (1) Å
 $\beta = 97.403$ (1)°
 $V = 1577.28$ (3) Å³
 $Z = 4$

$D_x = 1.210$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 5446 reflections
 $\theta = 2.0$ – 28.2 °
 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.48 \times 0.34 \times 0.26$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 10 433 measured reflections
 3912 independent reflections
 2972 reflections with $I > 2\sigma(I)$

$R_{int} = 0.029$
 $\theta_{max} = 28.3$ °
 $h = -13 \rightarrow 13$
 $k = -6 \rightarrow 7$
 $l = -35 \rightarrow 30$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.127$
 $S = 1.03$
 3912 reflections
 191 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.2846P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.26$ e Å⁻³
 $\Delta\rho_{min} = -0.29$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1—C8	1.229 (2)	C10—C15	1.354 (2)
O2—C11	1.234 (2)	C10—C11	1.440 (2)
O3—C15	1.336 (2)	C11—C12	1.503 (2)
N1—C8	1.341 (2)	C13—C14	1.523 (2)
N1—C1	1.420 (2)	C13—C17	1.528 (2)
C8—C9	1.511 (2)	C13—C16	1.531 (2)
C9—C10	1.506 (2)	C14—C15	1.496 (2)
C8—N1—C1—C6	36.0 (2)	N1—C8—C9—C10	143.1 (1)
C1—N1—C8—O1	2.1 (2)	C8—C9—C10—C11	-70.39 (15)
O1—C8—C9—C10	-38.60 (18)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C6—H6 \cdots O1	0.93	2.45	2.8824 (18)	109
C9—H9A \cdots O3	0.97	2.44	2.8235 (15)	103
O3—H3O \cdots O1 ⁱ	0.82	1.82	2.6423 (12)	174
C14—H14A \cdots O1 ⁱ	0.97	2.53	3.1301 (17)	120
N1—H1N \cdots O2 ⁱⁱ	0.86	2.00	2.8591 (15)	174

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

After their location in a difference map, all the H atoms were positioned geometrically and allowed to ride on their attached atoms using *SHELXL97* (Sheldrick, 1997) defaults for bond lengths and displacement parameters. One of the methyl groups (C7) was found to be disordered; it was treated as an idealized disordered methyl group, with two positions rotated from each other by 60°, and the

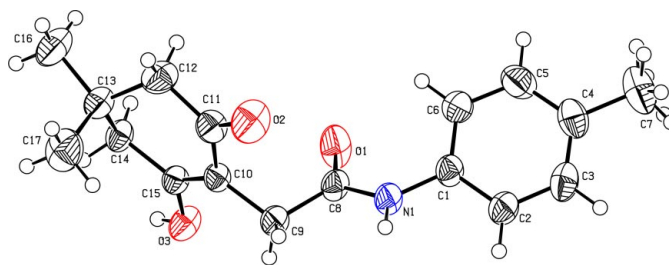


Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

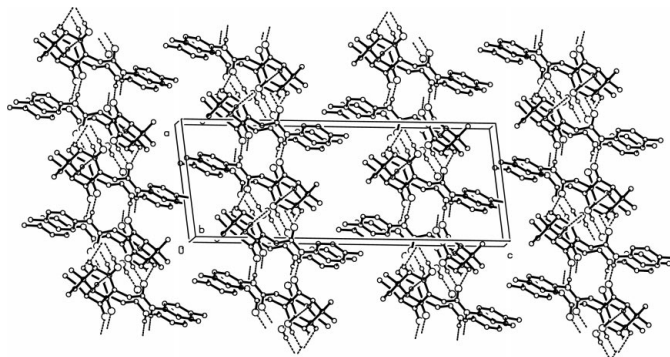


Figure 2

Packing of the molecules, viewed down the *b* axis.

occupation factors fixed at 0.5. Rotating group refinement was used for the OH group.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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