

2-(Adamantan-1-yl)-5-methylbenzo[d]-[1,3]oxazin-4-one

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Key indicators

Single-crystal X-ray study

T = 150 K

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

Disorder in main residue

R factor = 0.047

wR factor = 0.141

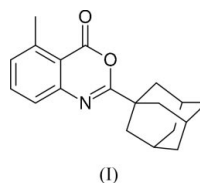
Data-to-parameter ratio = 26.4

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

At 150 K, the benzo[d][1,3]oxazin-4-one heterocycle in the title compound, $\text{C}_{19}\text{H}_{21}\text{NO}_2$, lies on a crystallographic mirror plane. This group is planar despite the resulting unfavourable steric interaction between the proximal 5-methyl and 4-carbonyl groups.

Comment

The title compound, (I), has a planar benzo[d][1,3]oxazin-4-one heterocycle that lies on a crystallographic mirror plane. The molecular structure of (I) is shown in Fig. 1 and selected bond distances and angles are given in Table 1. Within the oxazin-4-one group the $\text{C}=\text{O}$ and $\text{C}=\text{N}$ double bonds are clearly localized, but of the two formally single $\text{C}-\text{O}$ bonds, $\text{O}2-\text{C}2$ is significantly shorter than $\text{O}2-\text{C}1$. The bicyclic heterocycle is planar despite the unfavourable steric interaction between the 5-methyl and 4-carbonyl groups, but the planarity allows π -stacking of these groups in the direction of the *b* axis (Fig. 2), with an inter-layer distance of 3.3662 (4) \AA (Table 2). The widened bond angles of 128.39 (12), 121.75 (11) and 123.51 (12) $^\circ$ for $\text{O}1-\text{C}1-\text{C}4$, $\text{C}1-\text{C}4-\text{C}5$ and $\text{C}4-\text{C}5-\text{C}9$, respectively, still result in a short $\text{O}1 \cdots \text{C}9$ distance of 2.838 (2) \AA .



Experimental

Suitable crystals of the title compound, (I), were prepared by the attempted recrystallization of 2-[(adamantane-1-carbonyl)-amino]-6-methylbenzoic acid from petroleum ether (80/100)-toluene.

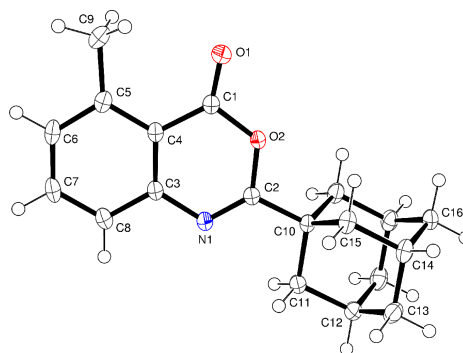


Figure 1

View of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by spheres of arbitrary size. Only one orientation of the disordered methyl group is shown.

Crystal data

$C_{19}H_{21}NO_2$
 $M_r = 295.37$
 Monoclinic, $P2_1/m$
 $a = 8.3514$ (12) Å
 $b = 6.7324$ (5) Å
 $c = 13.3203$ (19) Å
 $\beta = 104.275$ (11)°
 $V = 725.81$ (16) Å³
 $Z = 2$

$D_x = 1.352$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 8117
 reflections
 $\theta = 2.5$ – 34.7 °
 $\mu = 0.09$ mm⁻¹
 $T = 150$ (2) K
 Block, colourless
 $0.60 \times 0.25 \times 0.20$ mm

Data collection

Stoe IPDSII area-detector
 diffractometer
 φ and ω scans
 Absorption correction: none
 10561 measured reflections
 3325 independent reflections

2132 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.061$
 $\theta_{max} = 34.7$ °
 $h = -13 \rightarrow 13$
 $k = -10 \rightarrow 9$
 $l = -21 \rightarrow 17$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.141$
 $S = 1.00$
 3325 reflections
 126 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.081P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.49$ e Å⁻³
 $\Delta\rho_{min} = -0.37$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.032 (7)

Table 1

Selected geometric parameters (Å, °).

O1–C1	1.2010 (16)	N1–C3	1.4019 (16)
O2–C2	1.3750 (15)	C1–C4	1.4625 (17)
O2–C1	1.3940 (15)	C3–C4	1.4082 (18)
N1–C2	1.2762 (15)		
C2–O2–C1	122.07 (9)	N1–C2–C10	123.61 (11)
C2–N1–C3	117.81 (11)	O2–C2–C10	111.75 (9)
O1–C1–O2	116.33 (11)	N1–C3–C4	122.43 (10)
O1–C1–C4	128.39 (12)	C3–C4–C1	117.77 (11)
O2–C1–C4	115.28 (10)	C5–C4–C1	121.75 (11)
N1–C2–O2	124.64 (11)	C4–C5–C9	123.51 (12)

Table 2

Contact distances (Å).

C1...C7 ⁱ	3.3984 (4)	C4...C8 ⁱ	3.4884 (5)
C3...C5 ⁱ	3.4038 (4)	N1...C6 ⁱ	3.5083 (5)

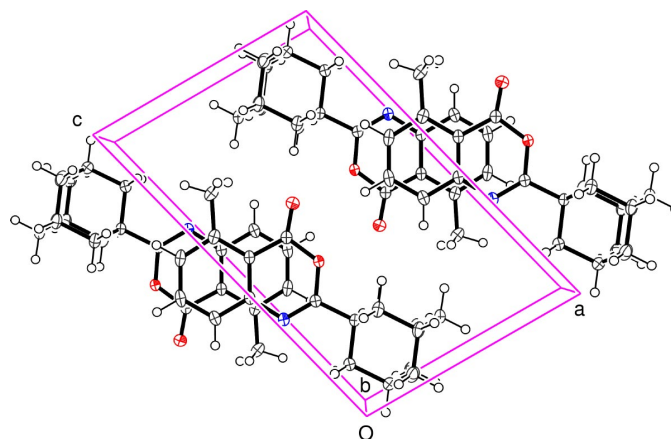
 Symmetry code: (i) $-x, -y, 1 - z$.


Figure 2

Packing diagram for (I), viewed down the b axis. Only one orientation for the disordered methyl group is shown.

All H atoms were initially located in a difference Fourier map. The methyl H atoms were constrained to an ideal geometry with a C–H distance of 0.98 Å, but the group was allowed to rotate freely about its X–C bond. In its final position, the methyl group is not bisected exactly by the mirror plane and hence is disordered 50:50 about the mirror plane. All other H atoms were placed in geometrically idealized positions, with C–H distances of 0.95–1.00 Å. $U_{iso}(H)$ values were set at $1.2U_{eq}(C)$ for all of the H atoms.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2001); program(s) used to solve structure: *X-STEP32* (Stoe & Cie, 2001) and *WinGX* (Farrugia, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX*.

References

- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
 Stoe & Cie (2001). *X-AREA*, *X-RED32* and *X-STEP32*. Stoe & Cie GmbH, Darmstadt, Germany.