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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 σ(C–C) = 0.002 Å
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.

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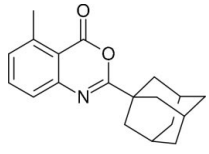
At 150 K, the benzo[*d*][1,3]oxazin-4-one heterocycle in the title compound, $C_{19}H_{21}NO_2$, lies on a crystallographic mirror plane. This group is planar despite the resulting unfavourable steric interaction between the proximal 5-methyl and 4-carbonyl groups.

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 C=O and C=N double bonds are clearly localized, but of the two formally single C—O bonds, O2—C2 is significantly shorter than O2—C1. 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) Å (Table 2). The widened bond angles of 128.39 (12), 121.75 (11) and 123.51 (12)° for O1—C1—C4, C1—C4—C5 and C4—C5—C9, respectively, still result in a short O1···C9 distance of 2.838 (2) Å.



(I)

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.

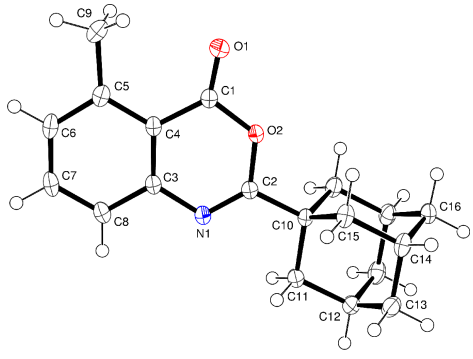


Figure 1
View of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by spheres of arbitrary size. Only one orientation of the disordered methyl group is shown.

Crystal data

$C_{19}H_{21}NO_2$	D_x = 1.352 Mg m^{-3}
M_r = 295.37	Mo $K\alpha$ radiation
Monoclinic, $P2_1/m$	Cell parameters from 8117 reflections
a = 8.3514 (12) Å	θ = 2.5–34.7°
b = 6.7324 (5) Å	μ = 0.09 mm^{-1}
c = 13.3203 (19) Å	T = 150 (2) K
β = 104.275 (11)°	Block, colourless
V = 725.81 (16) Å^3	0.60 × 0.25 × 0.20 mm
Z = 2	

Data collection

Stoe IPDSII area-detector diffractometer	2132 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.061
Absorption correction: none	θ_{max} = 34.7°
10561 measured reflections	$h = -13 \rightarrow 13$
3325 independent reflections	$k = -10 \rightarrow 9$
	$l = -21 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.081P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.048	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.141	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
S = 1.00	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
3325 reflections	Extinction correction: *SHELXL*97
126 parameters	Extinction coefficient: 0.032 (7)
H-atom parameters constrained	

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) $-x, -y, 1-z$.

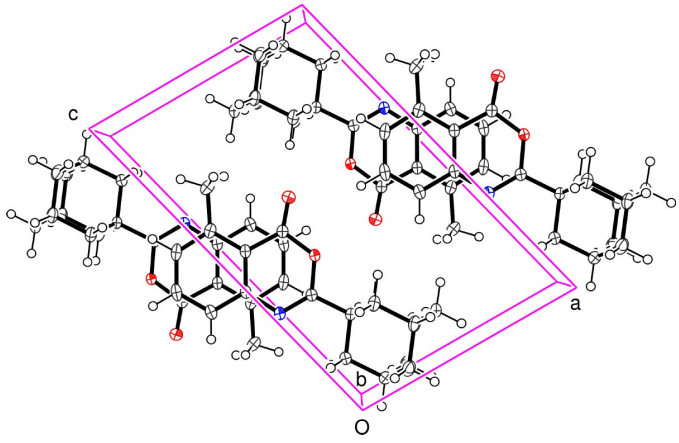


Figure 2
Packing diagram for (I), viewed down the b axis. Only one orientation for the disordered methyl group is shown.

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-RED*32 (Stoe & Cie, 2001); program(s) used to solve structure: *X-STEP*32 (Stoe & Cie, 2001) and *WinGX* (Farrugia, 1999); program(s) used to refine structure: *SHELXL*97 (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.
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Stoe & Cie (2001). *X-AREA*, *X-RED*32 and *X-STEP*32. Stoe & Cie GmbH, Darmstadt, Germany.

supporting information

Acta Cryst. (2004). E**60**, o669–o670 [https://doi.org/10.1107/S1600536804006907]

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$Z = 2$
$F(000) = 316$
$D_x = 1.352$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8117 reflections
$\theta = 2.5$–34.7°
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Block, colourless
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Graphite monochromator
φ and ω scans
10561 measured reflections
3325 independent reflections
2132 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 34.7°$, $\theta_{min} = 2.5°$
$h = -13 \rightarrow 13$
$k = -10 \rightarrow 9$
$l = -21 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.141$
$S = 1.00$
3325 reflections
126 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.032 (7)

Special details

Experimental. The crystal was mounted under the perfluoro-polyether PFO-XR75 (Lancaster Synthesis). A total of 160 frames (2 minute exposure) were collected (phi/omega: 35/90–160, 120/90–180, delta-omega = 1 °.)

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.39794 (12)	0.2500	0.58496 (8)	0.0299 (2)	
O2	0.30446 (10)	0.2500	0.41487 (7)	0.0219 (2)	
N1	0.02377 (13)	0.2500	0.32687 (8)	0.0197 (2)	
C1	0.27710 (15)	0.2500	0.51396 (10)	0.0208 (2)	
C2	0.17667 (14)	0.2500	0.32710 (10)	0.0187 (2)	
C3	−0.01715 (14)	0.2500	0.42282 (10)	0.0189 (2)	
C4	0.10352 (15)	0.2500	0.51764 (10)	0.0188 (2)	
C5	0.05645 (16)	0.2500	0.61268 (10)	0.0219 (2)	
C6	−0.11190 (18)	0.2500	0.60815 (11)	0.0242 (3)	
H6	−0.1462	0.2500	0.6710	0.029*	
C7	−0.23170 (16)	0.2500	0.51463 (12)	0.0254 (3)	
H7	−0.3455	0.2500	0.5144	0.030*	
C8	−0.18492 (15)	0.2500	0.42207 (11)	0.0233 (3)	
H8	−0.2664	0.2500	0.3582	0.028*	
C9	0.1785 (2)	0.2500	0.71724 (11)	0.0317 (3)	
H9A	0.1207	0.2201	0.7711	0.038*	0.50
H9B	0.2307	0.3810	0.7303	0.038*	0.50
H9C	0.2633	0.1490	0.7182	0.038*	0.50
C10	0.23881 (14)	0.2500	0.22986 (9)	0.0179 (2)	
C11	0.09153 (15)	0.2500	0.13392 (10)	0.0248 (3)	
H11A	0.0224	0.1309	0.1349	0.030*	0.50
H11B	0.0224	0.3691	0.1349	0.030*	0.50
C12	0.15514 (16)	0.2500	0.03505 (10)	0.0265 (3)	
H12	0.0589	0.2500	−0.0269	0.032*	
C13	0.25990 (13)	0.43563 (16)	0.03269 (8)	0.0273 (2)	
H13A	0.1923	0.5562	0.0334	0.033*	
H13B	0.2993	0.4370	−0.0316	0.033*	
C14	0.40801 (12)	0.43543 (14)	0.12743 (7)	0.02358 (19)	
H14	0.4765	0.5567	0.1258	0.028*	
C15	0.34586 (12)	0.43626 (14)	0.22713 (7)	0.02296 (19)	
H15A	0.2797	0.5575	0.2293	0.028*	
H15B	0.4412	0.4371	0.2884	0.028*	
C16	0.51278 (16)	0.2500	0.12420 (10)	0.0235 (3)	

H16A	0.5524	0.2500	0.0600	0.028*
H16B	0.6103	0.2500	0.1842	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0196 (4)	0.0484 (7)	0.0209 (5)	0.000	0.0036 (3)	0.000
O2	0.0149 (4)	0.0333 (5)	0.0184 (4)	0.000	0.0059 (3)	0.000
N1	0.0169 (4)	0.0230 (5)	0.0202 (5)	0.000	0.0067 (4)	0.000
C1	0.0186 (5)	0.0250 (6)	0.0198 (5)	0.000	0.0068 (4)	0.000
C2	0.0173 (5)	0.0201 (5)	0.0193 (5)	0.000	0.0058 (4)	0.000
C3	0.0171 (5)	0.0193 (5)	0.0219 (5)	0.000	0.0079 (4)	0.000
C4	0.0180 (5)	0.0191 (5)	0.0212 (5)	0.000	0.0086 (4)	0.000
C5	0.0257 (6)	0.0207 (6)	0.0220 (6)	0.000	0.0111 (5)	0.000
C6	0.0284 (6)	0.0217 (6)	0.0286 (6)	0.000	0.0184 (5)	0.000
C7	0.0207 (5)	0.0241 (6)	0.0362 (7)	0.000	0.0165 (5)	0.000
C8	0.0172 (5)	0.0258 (6)	0.0284 (6)	0.000	0.0085 (5)	0.000
C9	0.0353 (7)	0.0427 (8)	0.0191 (6)	0.000	0.0105 (5)	0.000
C10	0.0167 (5)	0.0210 (5)	0.0174 (5)	0.000	0.0069 (4)	0.000
C11	0.0175 (5)	0.0374 (7)	0.0200 (6)	0.000	0.0058 (4)	0.000
C12	0.0206 (5)	0.0414 (8)	0.0179 (5)	0.000	0.0055 (4)	0.000
C13	0.0314 (5)	0.0293 (5)	0.0245 (4)	0.0079 (4)	0.0133 (4)	0.0080 (4)
C14	0.0278 (4)	0.0209 (4)	0.0259 (4)	−0.0046 (3)	0.0140 (3)	−0.0008 (3)
C15	0.0264 (4)	0.0212 (4)	0.0246 (4)	−0.0038 (3)	0.0126 (3)	−0.0035 (3)
C16	0.0193 (5)	0.0315 (7)	0.0221 (6)	0.000	0.0095 (4)	0.000

Geometric parameters (Å, º)

O1—C1	1.2010 (16)	C10—C11	1.5398 (17)
O2—C2	1.3750 (15)	C10—C15	1.5457 (11)
O2—C1	1.3940 (15)	C10—C15^{i}	1.5457 (11)
N1—C2	1.2762 (15)	C11—C12	1.5368 (18)
N1—C3	1.4019 (16)	C11—H11A	0.9900
C1—C4	1.4625 (17)	C11—H11B	0.9900
C2—C10	1.5094 (16)	C12—C13	1.5303 (13)
C3—C8	1.3988 (17)	C12—C13^{i}	1.5303 (13)
C3—C4	1.4082 (18)	C12—H12	1.0000
C4—C5	1.4151 (17)	C13—C14	1.5339 (14)
C5—C6	1.3923 (19)	C13—H13A	0.9900
C5—C9	1.510 (2)	C13—H13B	0.9900
C6—C7	1.392 (2)	C14—C16	1.5311 (12)
C6—H6	0.9500	C14—C15	1.5405 (12)
C7—C8	1.3821 (19)	C14—H14	1.0000
C7—H7	0.9500	C15—H15A	0.9900
C8—H8	0.9500	C15—H15B	0.9900
C9—H9A	0.9800	C16—C14^{i}	1.5311 (12)
C9—H9B	0.9800	C16—H16A	0.9900
C9—H9C	0.9800	C16—H16B	0.9900

C1···C7^{ii}	3.3984 (4)	C4···C8^{ii}	3.4884 (5)
C3···C5^{ii}	3.4038 (4)	N1···C6^{ii}	3.5083 (5)

C2—O2—C1	122.07 (9)	C15—C10—C15^{i}	108.44 (10)
C2—N1—C3	117.81 (11)	C12—C11—C10	109.70 (10)
O1—C1—O2	116.33 (11)	C12—C11—H11A	109.7
O1—C1—C4	128.39 (12)	C10—C11—H11A	109.7
O2—C1—C4	115.28 (10)	C12—C11—H11B	109.7
N1—C2—O2	124.64 (11)	C10—C11—H11B	109.7
N1—C2—C10	123.61 (11)	H11A—C11—H11B	108.2
O2—C2—C10	111.75 (9)	C13—C12—C13^{i}	109.51 (11)
C8—C3—N1	117.55 (11)	C13—C12—C11	109.74 (7)
C8—C3—C4	120.03 (11)	C13^{i}—C12—C11	109.74 (7)
N1—C3—C4	122.43 (10)	C13—C12—H12	109.3
C3—C4—C5	120.48 (11)	C13^{i}—C12—H12	109.3
C3—C4—C1	117.77 (11)	C11—C12—H12	109.3
C5—C4—C1	121.75 (11)	C12—C13—C14	109.36 (8)
C6—C5—C4	117.48 (12)	C12—C13—H13A	109.8
C6—C5—C9	119.01 (12)	C14—C13—H13A	109.8
C4—C5—C9	123.51 (12)	C12—C13—H13B	109.8
C7—C6—C5	122.28 (12)	C14—C13—H13B	109.8
C7—C6—H6	118.9	H13A—C13—H13B	108.2
C5—C6—H6	118.9	C16—C14—C13	109.15 (9)
C8—C7—C6	119.96 (12)	C16—C14—C15	110.09 (8)
C8—C7—H7	120.0	C13—C14—C15	109.54 (8)
C6—C7—H7	120.0	C16—C14—H14	109.3
C7—C8—C3	119.78 (12)	C13—C14—H14	109.3
C7—C8—H8	120.1	C15—C14—H14	109.3
C3—C8—H8	120.1	C14—C15—C10	109.70 (8)
C5—C9—H9A	109.5	C14—C15—H15A	109.7
C5—C9—H9B	109.5	C10—C15—H15A	109.7
H9A—C9—H9B	109.5	C14—C15—H15B	109.7
C5—C9—H9C	109.5	C10—C15—H15B	109.7
H9A—C9—H9C	109.5	H15A—C15—H15B	108.2
H9B—C9—H9C	109.5	C14^{i}—C16—C14	109.24 (10)
C2—C10—C11	109.81 (9)	C14^{i}—C16—H16A	109.8
C2—C10—C15	110.08 (6)	C14—C16—H16A	109.8
C11—C10—C15	109.19 (7)	C14^{i}—C16—H16B	109.8
C2—C10—C15^{i}	110.08 (6)	C14—C16—H16B	109.8
C11—C10—C15^{i}	109.19 (7)	H16A—C16—H16B	108.3

C2—O2—C1—O1	180.0	N1—C3—C8—C7	180.0
C2—O2—C1—C4	0.0	C4—C3—C8—C7	0.0
C3—N1—C2—O2	0.0	N1—C2—C10—C11	0.0
C3—N1—C2—C10	180.0	O2—C2—C10—C11	180.0
C1—O2—C2—N1	0.0	N1—C2—C10—C15	120.25 (7)
C1—O2—C2—C10	180.0	O2—C2—C10—C15	−59.75 (7)

C2—N1—C3—C8	180.0	N1—C2—C10—C15^{i}	−120.25 (7)
C2—N1—C3—C4	0.0	O2—C2—C10—C15^{i}	59.75 (7)
C8—C3—C4—C5	0.0	C2—C10—C11—C12	180.0
N1—C3—C4—C5	180.0	C15—C10—C11—C12	59.21 (6)
C8—C3—C4—C1	180.0	C15^{i}—C10—C11—C12	−59.21 (6)
N1—C3—C4—C1	0.0	C10—C11—C12—C13	−60.19 (7)
O1—C1—C4—C3	180.0	C10—C11—C12—C13^{i}	60.19 (7)
O2—C1—C4—C3	0.0	C13^{i}—C12—C13—C14	−60.07 (12)
O1—C1—C4—C5	0.0	C11—C12—C13—C14	60.45 (11)
O2—C1—C4—C5	180.0	C12—C13—C14—C16	60.38 (10)
C3—C4—C5—C6	0.0	C12—C13—C14—C15	−60.23 (10)
C1—C4—C5—C6	180.0	C16—C14—C15—C10	−60.25 (10)
C3—C4—C5—C9	180.0	C13—C14—C15—C10	59.79 (10)
C1—C4—C5—C9	0.0	C2—C10—C15—C14	−179.74 (8)
C4—C5—C6—C7	0.0	C11—C10—C15—C14	−59.12 (10)
C9—C5—C6—C7	180.0	C15^{i}—C10—C15—C14	59.77 (12)
C5—C6—C7—C8	0.0	C13—C14—C16—C14^{i}	−60.82 (12)
C6—C7—C8—C3	0.0	C15—C14—C16—C14^{i}	59.45 (13)

Symmetry codes: (i) x, $-y+1/2$, z; (ii) $-x$, $-y$, $-z+1$.