Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

tert-Butyl 6-acetamido-3,4-dihydro-2*H*-1,4-benzoxazine-4-carboxylate

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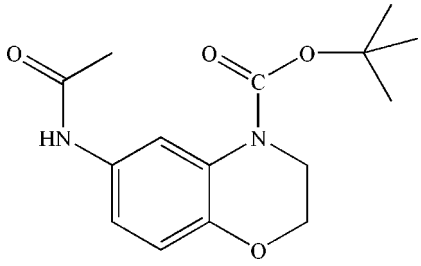
Received 26 August 2010; accepted 15 September 2010

Key indicators: single-crystal X-ray study; T = 103 K; mean σ(C–C) = 0.002 Å; R factor = 0.048; wR factor = 0.114; data-to-parameter ratio = 17.0.

The title molecule, $C_{15}H_{20}N_2O_4$, contains a benzene ring fused to an oxazine ring and one *tert*-butoxycarbonyl group bound to the N atom. An intramolecular C—H···O interaction occurs. In the crystal, molecules are linked through intermolecular N—H···O and C—H···O hydrogen bonds.

Related literature

For the pharmacological properties of phenylmorpholine derivatives, see: Bourlot *et al.* (1998); Albanese *et al.* (2003); La *et al.* (2008). For structures, see: Chen *et al.* (2003); Olmstead *et al.* (2003); Vergeer *et al.* (1999).



Experimental

Crystal data

$C_{15}H_{20}N_2O_4$
M_r = 292.33
Orthorhombic, *Pbca*
a = 9.675 (2) Å
b = 13.137 (3) Å
c = 23.128 (5) Å
V = 2939.7 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 103 K
0.43 × 0.40 × 0.18 mm

Data collection

Rigaku SPIDER diffractometer
20907 measured reflections
3367 independent reflections
3005 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.114
S = 1.00
3367 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*N*···O4^{i}	0.879 (19)	2.051 (19)	2.9251 (17)	173.1 (16)
C5—H5···O4	0.95	2.27	2.8771 (19)	121
C11—H11*B*···O3^{ii}	0.98	2.49	3.456 (2)	168

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This work was supported financially by Jiangsu Province 's Key Medical Talents Program (No. RC2007097).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2200).

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supporting information

Acta Cryst. (2010). E**66**, o2600 [doi:10.1107/S1600536810036937]

tert-Butyl 6-acetamido-3,4-dihydro-2*H*-1,4-benzoxazine-4-carboxylate

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S1. Comment

The stucture of the title compound, (I), is an important phenylmorpholine product. Phenylmorpholine compounds are used as α2 C adrenergic receptor agonists. Numerous phenylmorpholine derivatives possess various pharmacological properties (Bourlot, *et al.*, 1998; Albanese, *et al.*, 2003; La *et al.*, 2008).

We report here the crystal structure of the title compound. (Fig. 1). The title molecule of (I) contains a benzene ring fused to an oxazine ring and one *tert*-butoxycarbonyl bound to the N atom. The N1—C8 bond distance is 1.4208 (18) Å and agrees with literature values (Vergeer, *et al.*, 1999; Chen, *et al.*, 2003; Olmstead, *et al.*, 2003). The conformation of the six-membered heterocyclic ring shows that C1 and C2 are out of the plane of the remaining four atoms by 0.5950 (16) and -0.0818 (16) Å, respectively.

The molecules are linked through hydrogen-bonding interactions of types N—H···O and C—H···O (Table 1).

S2. Experimental

The title compound was crystallized from a mixed solvent composed of dichloromethane and hexane (1:1); colorlessblock-shaped crystals were obtained after several days.

S3. Refinement

Positional parameters of all the H atomsbonded to C atoms were calculated geometrically and were allowed to ride on the C atoms to which they were bonded, with C—H distances of 0.95Å (CH), 0.98Å (CH_3) or 0.99Å (CH_2), and with U_{iso}(H) =1.2$U_{eq}$of the parent atoms. The H-atoms bonded to N atoms was taken from a difference map and was allowed to refine freely.

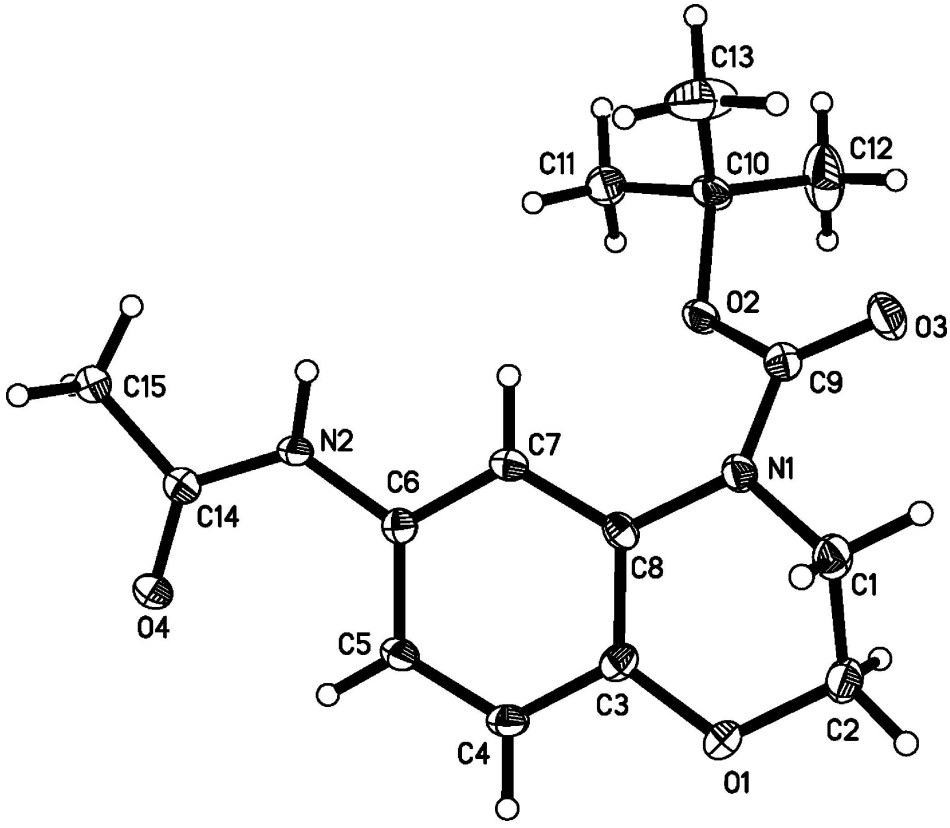

Figure 1

A view of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 50% probability level.

tert-Butyl 6-acetamido-3,4-dihydro-2*H*-1,4-benzoxazine-4-carboxylate

Crystal data

$C_{15}H_{20}N_2O_4$
M_r = 292.33
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 9.675 (2) Å
b = 13.137 (3) Å
c = 23.128 (5) Å
V = 2939.7 (12) Å^3
Z = 8

$F(000)$ = 1248
D_x = 1.321 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8192 reflections
θ = 3.1–27.5°
μ = 0.10 mm^{-1}
T = 103 K
Chunk, colorless
0.43 × 0.40 × 0.18 mm

Data collection

Rigaku SPIDER diffractometer
Radiation source: Rotating Anode
Graphite monochromator
ω scans
20907 measured reflections
3367 independent reflections

3005 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −12→12
k = −16→17
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.114$
$S = 1.00$
3367 reflections
198 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 1.960P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Special details

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.16030 (11)	0.52261 (8)	0.61658 (5)	0.0221 (3)
O2	0.53747 (10)	0.73649 (7)	0.57927 (4)	0.0173 (2)
O3	0.62739 (12)	0.58152 (8)	0.55786 (5)	0.0239 (3)
O4	0.08299 (11)	0.93078 (8)	0.77211 (5)	0.0222 (3)
N1	0.43426 (13)	0.59636 (9)	0.61318 (5)	0.0164 (3)
N2	0.28810 (12)	0.88225 (9)	0.73211 (5)	0.0146 (3)
C1	0.40682 (16)	0.48702 (11)	0.61007 (7)	0.0201 (3)
H1A	0.4110	0.4569	0.6493	0.024*
H1B	0.4775	0.4534	0.5857	0.024*
C2	0.26528 (17)	0.47066 (11)	0.58444 (7)	0.0212 (3)
H2A	0.2647	0.4954	0.5440	0.025*
H2B	0.2445	0.3969	0.5839	0.025*
C3	0.19649 (15)	0.61199 (11)	0.64273 (6)	0.0162 (3)
C4	0.09200 (15)	0.66258 (11)	0.67188 (6)	0.0178 (3)
H4	0.0014	0.6348	0.6718	0.021*
C5	0.11645 (15)	0.75263 (11)	0.70112 (6)	0.0166 (3)
H5	0.0431	0.7872	0.7200	0.020*
C6	0.25037 (15)	0.79196 (10)	0.70245 (6)	0.0140 (3)
C7	0.35627 (14)	0.74149 (10)	0.67329 (6)	0.0145 (3)
H7	0.4475	0.7681	0.6745	0.017*
C8	0.33000 (15)	0.65256 (11)	0.64237 (6)	0.0145 (3)
C9	0.54187 (15)	0.63514 (11)	0.58119 (6)	0.0159 (3)
C10	0.65864 (16)	0.79588 (12)	0.56032 (7)	0.0200 (3)
C11	0.60965 (17)	0.90434 (11)	0.56794 (7)	0.0237 (3)

H11A	0.5321	0.9174	0.5417	0.028*
H11B	0.6855	0.9512	0.5591	0.028*
H11C	0.5796	0.9146	0.6080	0.028*
C12	0.6938 (3)	0.77433 (14)	0.49806 (9)	0.0433 (5)
H12A	0.7274	0.7043	0.4944	0.052*
H12B	0.7658	0.8216	0.4851	0.052*
H12C	0.6111	0.7831	0.4741	0.052*
C13	0.7775 (2)	0.77283 (15)	0.60145 (11)	0.0425 (5)
H13A	0.7467	0.7825	0.6414	0.051*
H13B	0.8548	0.8189	0.5933	0.051*
H13C	0.8077	0.7022	0.5961	0.051*
C14	0.20747 (14)	0.94428 (11)	0.76449 (6)	0.0155 (3)
C15	0.28116 (15)	1.03281 (11)	0.79194 (6)	0.0188 (3)
H15A	0.2406	1.0966	0.7780	0.023*
H15B	0.3793	1.0306	0.7816	0.023*
H15C	0.2715	1.0289	0.8341	0.023*
H2N	0.375 (2)	0.9008 (13)	0.7287 (8)	0.022 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0204 (6)	0.0195 (5)	0.0263 (6)	−0.0049 (4)	0.0015 (4)	−0.0083 (4)
O2	0.0147 (5)	0.0148 (5)	0.0224 (5)	−0.0012 (4)	0.0056 (4)	0.0001 (4)
O3	0.0232 (6)	0.0193 (5)	0.0292 (6)	0.0037 (5)	0.0103 (5)	−0.0013 (5)
O4	0.0124 (5)	0.0268 (6)	0.0275 (6)	−0.0001 (4)	0.0030 (4)	−0.0073 (5)
N1	0.0167 (6)	0.0136 (6)	0.0189 (6)	0.0006 (5)	0.0039 (5)	−0.0008 (5)
N2	0.0107 (6)	0.0153 (6)	0.0179 (6)	−0.0017 (5)	0.0012 (4)	−0.0010 (5)
C1	0.0240 (8)	0.0134 (7)	0.0230 (8)	0.0007 (6)	0.0048 (6)	0.0001 (6)
C2	0.0268 (8)	0.0163 (7)	0.0205 (7)	−0.0020 (6)	0.0038 (6)	−0.0035 (6)
C3	0.0188 (7)	0.0153 (7)	0.0145 (6)	−0.0021 (6)	−0.0023 (5)	0.0000 (5)
C4	0.0128 (7)	0.0205 (7)	0.0202 (7)	−0.0035 (6)	−0.0006 (5)	0.0001 (6)
C5	0.0120 (6)	0.0187 (7)	0.0191 (7)	−0.0006 (5)	0.0022 (5)	−0.0001 (5)
C6	0.0151 (7)	0.0150 (6)	0.0119 (6)	−0.0003 (5)	−0.0001 (5)	0.0016 (5)
C7	0.0125 (7)	0.0164 (7)	0.0146 (6)	−0.0016 (5)	0.0004 (5)	0.0020 (5)
C8	0.0144 (7)	0.0159 (7)	0.0130 (6)	0.0019 (5)	0.0016 (5)	0.0020 (5)
C9	0.0156 (7)	0.0167 (7)	0.0153 (6)	0.0014 (6)	−0.0011 (5)	0.0005 (5)
C10	0.0166 (7)	0.0195 (7)	0.0237 (8)	−0.0036 (6)	0.0076 (6)	0.0011 (6)
C11	0.0250 (8)	0.0182 (7)	0.0280 (8)	−0.0039 (6)	0.0068 (6)	−0.0011 (6)
C12	0.0707 (15)	0.0260 (9)	0.0330 (10)	−0.0041 (10)	0.0303 (10)	−0.0012 (8)
C13	0.0241 (10)	0.0336 (10)	0.0697 (15)	−0.0061 (8)	−0.0121 (9)	0.0070 (10)
C14	0.0146 (7)	0.0170 (7)	0.0148 (7)	0.0012 (5)	−0.0007 (5)	0.0020 (5)
C15	0.0169 (7)	0.0198 (7)	0.0197 (7)	−0.0007 (6)	0.0016 (5)	−0.0034 (6)

Geometric parameters (Å, º)

O1—C3	1.3664 (17)	C5—C6	1.395 (2)
O1—C2	1.4317 (18)	C5—H5	0.9500
O2—C9	1.3328 (18)	C6—C7	1.394 (2)

O2—C10	1.4749 (17)	C7—C8	1.393 (2)
O3—C9	1.2132 (18)	C7—H7	0.9500
O4—C14	1.2301 (18)	C10—C12	1.506 (2)
N1—C9	1.3752 (19)	C10—C11	1.512 (2)
N1—C8	1.4208 (18)	C10—C13	1.523 (2)
N1—C1	1.4624 (19)	C11—H11A	0.9800
N2—C14	1.3540 (18)	C11—H11B	0.9800
N2—C6	1.4180 (18)	C11—H11C	0.9800
N2—H2N	0.88 (2)	C12—H12A	0.9800
C1—C2	1.508 (2)	C12—H12B	0.9800
C1—H1A	0.9900	C12—H12C	0.9800
C1—H1B	0.9900	C13—H13A	0.9800
C2—H2A	0.9900	C13—H13B	0.9800
C2—H2B	0.9900	C13—H13C	0.9800
C3—C4	1.385 (2)	C14—C15	1.505 (2)
C3—C8	1.398 (2)	C15—H15A	0.9800
C4—C5	1.383 (2)	C15—H15B	0.9800
C4—H4	0.9500	C15—H15C	0.9800

C3—O1—C2	117.24 (12)	C3—C8—N1	117.45 (13)
C9—O2—C10	120.86 (11)	O3—C9—O2	125.95 (14)
C9—N1—C8	126.92 (12)	O3—C9—N1	122.72 (13)
C9—N1—C1	118.35 (12)	O2—C9—N1	111.33 (12)
C8—N1—C1	113.88 (12)	O2—C10—C12	111.35 (14)
C14—N2—C6	128.51 (12)	O2—C10—C11	102.40 (12)
C14—N2—H2N	115.7 (12)	C12—C10—C11	111.06 (14)
C6—N2—H2N	115.8 (12)	O2—C10—C13	108.04 (13)
N1—C1—C2	108.92 (12)	C12—C10—C13	112.90 (17)
N1—C1—H1A	109.9	C11—C10—C13	110.57 (14)
C2—C1—H1A	109.9	C10—C11—H11A	109.5
N1—C1—H1B	109.9	C10—C11—H11B	109.5
C2—C1—H1B	109.9	H11A—C11—H11B	109.5
H1A—C1—H1B	108.3	C10—C11—H11C	109.5
O1—C2—C1	111.86 (12)	H11A—C11—H11C	109.5
O1—C2—H2A	109.2	H11B—C11—H11C	109.5
C1—C2—H2A	109.2	C10—C12—H12A	109.5
O1—C2—H2B	109.2	C10—C12—H12B	109.5
C1—C2—H2B	109.2	H12A—C12—H12B	109.5
H2A—C2—H2B	107.9	C10—C12—H12C	109.5
O1—C3—C4	116.16 (13)	H12A—C12—H12C	109.5
O1—C3—C8	124.19 (13)	H12B—C12—H12C	109.5
C4—C3—C8	119.64 (13)	C10—C13—H13A	109.5
C5—C4—C3	121.58 (13)	C10—C13—H13B	109.5
C5—C4—H4	119.2	H13A—C13—H13B	109.5
C3—C4—H4	119.2	C10—C13—H13C	109.5
C4—C5—C6	119.10 (13)	H13A—C13—H13C	109.5
C4—C5—H5	120.4	H13B—C13—H13C	109.5
C6—C5—H5	120.4	O4—C14—N2	123.82 (13)

C7—C6—C5	119.71 (13)	O4—C14—C15	121.00 (13)
C7—C6—N2	116.27 (13)	N2—C14—C15	115.18 (12)
C5—C6—N2	124.02 (13)	C14—C15—H15A	109.5
C8—C7—C6	120.86 (13)	C14—C15—H15B	109.5
C8—C7—H7	119.6	H15A—C15—H15B	109.5
C6—C7—H7	119.6	C14—C15—H15C	109.5
C7—C8—C3	119.04 (13)	H15A—C15—H15C	109.5
C7—C8—N1	123.38 (13)	H15B—C15—H15C	109.5
C9—N1—C1—C2	−116.78 (14)	C4—C3—C8—C7	2.4 (2)
C8—N1—C1—C2	53.44 (16)	O1—C3—C8—N1	−0.5 (2)
C3—O1—C2—C1	32.01 (17)	C4—C3—C8—N1	178.35 (12)
N1—C1—C2—O1	−56.42 (16)	C9—N1—C8—C7	−41.1 (2)
C2—O1—C3—C4	177.85 (13)	C1—N1—C8—C7	149.70 (13)
C2—O1—C3—C8	−3.3 (2)	C9—N1—C8—C3	143.12 (14)
O1—C3—C4—C5	178.58 (13)	C1—N1—C8—C3	−26.10 (18)
C8—C3—C4—C5	−0.3 (2)	C10—O2—C9—O3	−15.9 (2)
C3—C4—C5—C6	−1.6 (2)	C10—O2—C9—N1	164.66 (12)
C4—C5—C6—C7	1.5 (2)	C8—N1—C9—O3	179.49 (14)
C4—C5—C6—N2	−178.52 (13)	C1—N1—C9—O3	−11.7 (2)
C14—N2—C6—C7	−178.52 (13)	C8—N1—C9—O2	−1.1 (2)
C14—N2—C6—C5	1.5 (2)	C1—N1—C9—O2	167.70 (12)
C5—C6—C7—C8	0.5 (2)	C9—O2—C10—C12	64.04 (18)
N2—C6—C7—C8	−179.43 (12)	C9—O2—C10—C11	−177.21 (13)
C6—C7—C8—C3	−2.5 (2)	C9—O2—C10—C13	−60.47 (18)
C6—C7—C8—N1	−178.20 (12)	C6—N2—C14—O4	−1.4 (2)
O1—C3—C8—C7	−176.45 (13)	C6—N2—C14—C15	178.02 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···O4^{i}	0.879 (19)	2.051 (19)	2.9251 (17)	173.1 (16)
C1—H1*B*···O3	0.99	2.31	2.748 (2)	105
C5—H5···O4	0.95	2.27	2.8771 (19)	121
C7—H7···O2	0.95	2.40	2.7940 (18)	104
C11—H11*B*···O3^{ii}	0.98	2.49	3.456 (2)	168
C12—H12*A*···O3	0.98	2.39	2.957 (2)	117
C13—H13*C*···O3	0.98	2.52	3.073 (2)	116

Symmetry codes: (i) $x+1/2, y, -z+3/2$; (ii) $-x+3/2, y+1/2, z$.