Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

tert-Butyl 2-(1*H*-imidazol-1-yl)acetate

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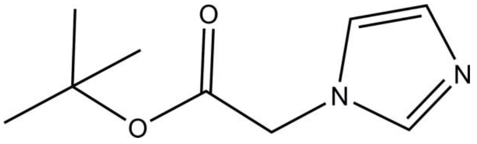
Received 9 January 2012; accepted 10 January 2012

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.097; data-to-parameter ratio = 18.2.

In the title compound, $C_9H_{14}N_2O_2$, the imidazole ring and the acetate O—C═O plane make a dihedral angle of 80.54 (12)°. In the crystal, molecules are connected *via* pairs of C—H⋯O hydrogen bonds, forming centrosymmetric dimers.

Related literature

For related structures, see: Pak *et al.* (2003); Wang *et al.* (2010).



Experimental

Crystal data

$C_9H_{14}N_2O_2$	V = 963.9 (4) Å^3
M_r = 182.22	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 10.558 (2) Å	μ = 0.09 mm^{-1}
b = 9.287 (2) Å	T = 100 K
c = 11.047 (2) Å	0.33 × 0.27 × 0.05 mm
β = 117.157 (4)°	

Data collection

Bruker APEXII CCD diffractometer	6535 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2208 independent reflections
T_{min} = 0.971, T_{max} = 0.996	1688 reflections with $I > 2\sigma(I)$
	R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040	121 parameters
$wR(F^2)$ = 0.097	H-atom parameters constrained
S = 1.05	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
2208 reflections	$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C4—H4B⋯O2^{i}	0.99	2.56	3.3768 (18)	140

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97* and *publCIF* (Westrip, 2010).

The authors thank the University of Malaya for funding this study (FRGS grant No. FP001/2010 A).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5051).

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supplementary materials

Acta Cryst. (2012). E**68**, o445 [**doi:10.1107/S1600536812001067**]

tert-Butyl 2-(1*H*-imidazol-1-yl)acetate

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Comment

The title compound was obtained through the condensation reaction of imidazole with *tert*-butyl chloroacetate. The imidazole ring and the plane passing through C5/O1/O2, make a dihedral angle of 80.54 (12)°. This value is comparable to those calculated for some similar structures (Pak *et al.*, 2003; Wang *et al.*, 2010). In the crystal, each two molecules are connected *via* a pair of C—H···O bonds around a center of inversion.

Experimental

Sodium hydroxide (1.32 g, 0.033 mol) was added to a solution of imidazole (1.5 g, 0.022 mol) in DMF (20 ml), followed by addition of *tert*-butyl chloroacetate (3.15 ml, 0.022 mol). The mixture was refluxed for 1 h. The reaction mass was quenched with cold water (50 ml) and extracted by dichloromethane (3 × 25 ml). The combined organic layers was washed with cold water and brine and dried over anhydrous sodium sulfate. The solvent was evaporated under vacuum and the formed amorphous solid was stirred in *n*-hexane (30 ml) at room temperature. The solid was filtered, washed with hexane (2 × 20 ml), and recrystallized from ethyl acetate to afford off-white crystals of the title compound (melting point = 384–386 K).

Refinement

H atoms were placed at calculated positions and refined in riding mode, with C—H distances of 0.95 (imidazole), 0.98 (methyl) and 0.99 (methylene) Å, and with U_{iso}(H) set to 1.2 (1.5 for methyl) U_{eq}(C).

Figures

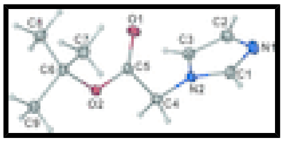

Fig. 1. The molecular structure of the title compound showing displacement ellipsoids at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

tert-Butyl 2-(1*H*-imidazol-1-yl)acetate

Crystal data

$C_9H_{14}N_2O_2$

M_r = 182.22

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

a = 10.558 (2) Å

b = 9.287 (2) Å

$F(000)$ = 392

D_x = 1.256 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 771 reflections

θ = 3.0–29.0°

μ = 0.09 mm^{-1}

c = 11.047 (2) Å	T = 100 K
β = 117.157 (4)°	Plate, colorless
V = 963.9 (4) Å^3	0.33 × 0.27 × 0.05 mm
Z = 4	

Data collection

Bruker APEXII CCD diffractometer	2208 independent reflections
Radiation source: fine-focus sealed tube	1688 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.035
φ and ω scans	θ_{max} = 27.5°, θ_{min} = 2.2°
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -10 \rightarrow 13$
T_{min} = 0.971, T_{max} = 0.996	$k = -12 \rightarrow 12$
6535 measured reflections	$l = -14 \rightarrow 11$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.040	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.097	H-atom parameters constrained
S = 1.05	$w = 1/[\sigma^2(F_o^2) + (0.0352P)^2 + 0.2973P]$ where $P = (F_o^2 + 2F_c^2)/3$
2208 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
121 parameters	$\Delta\rho_{\text{max}}$ = 0.23 e Å^{-3}
0 restraints	$\Delta\rho_{\text{min}}$ = −0.27 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	$U_{\text{iso}}*/U_{\text{eq}}$
O1	0.58413 (11)	0.82030 (11)	0.21337 (11)	0.0244 (3)
O2	0.42983 (10)	0.63596 (10)	0.11631 (10)	0.0177 (2)

N1	1.01459 (13)	0.74927 (14)	0.30780 (13)	0.0250 (3)
N2	0.79333 (12)	0.67798 (12)	0.17766 (12)	0.0164 (3)
C1	0.91215 (15)	0.66189 (16)	0.29694 (15)	0.0210 (3)
H1	0.9207	0.5949	0.3653	0.025*
C2	0.95672 (16)	0.82623 (16)	0.18788 (16)	0.0231 (3)
H2	1.0055	0.8989	0.1650	0.028*
C3	0.82115 (15)	0.78441 (15)	0.10713 (15)	0.0197 (3)
H3	0.7584	0.8211	0.0198	0.024*
C4	0.65738 (15)	0.60761 (15)	0.13621 (15)	0.0188 (3)
H4A	0.6720	0.5166	0.1876	0.023*
H4B	0.6155	0.5835	0.0382	0.023*
C5	0.55466 (15)	0.70313 (15)	0.16122 (14)	0.0169 (3)
C6	0.30955 (14)	0.69930 (15)	0.13399 (14)	0.0178 (3)
C7	0.35240 (16)	0.71586 (17)	0.28396 (15)	0.0228 (3)
H7A	0.3916	0.6247	0.3308	0.034*
H7B	0.2688	0.7420	0.2957	0.034*
H7C	0.4245	0.7916	0.3225	0.034*
C8	0.26438 (16)	0.84140 (16)	0.05766 (16)	0.0230 (3)
H8A	0.3393	0.9133	0.1028	0.035*
H8B	0.1761	0.8747	0.0572	0.035*
H8C	0.2487	0.8278	−0.0362	0.035*
C9	0.19439 (15)	0.58562 (16)	0.06881 (15)	0.0230 (3)
H9A	0.1688	0.5775	−0.0281	0.034*
H9B	0.1102	0.6134	0.0790	0.034*
H9C	0.2299	0.4927	0.1135	0.034*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0231 (6)	0.0202 (5)	0.0320 (6)	−0.0045 (4)	0.0144 (5)	−0.0086 (4)
O2	0.0141 (5)	0.0182 (5)	0.0222 (5)	−0.0015 (4)	0.0096 (4)	−0.0035 (4)
N1	0.0185 (7)	0.0306 (7)	0.0238 (7)	−0.0012 (5)	0.0077 (6)	−0.0052 (6)
N2	0.0143 (6)	0.0174 (6)	0.0177 (6)	−0.0016 (5)	0.0075 (5)	−0.0027 (5)
C1	0.0195 (7)	0.0230 (7)	0.0192 (7)	0.0036 (6)	0.0076 (6)	0.0006 (6)
C2	0.0199 (7)	0.0233 (7)	0.0290 (8)	−0.0039 (6)	0.0138 (7)	−0.0026 (6)
C3	0.0213 (8)	0.0209 (7)	0.0191 (7)	0.0001 (6)	0.0111 (6)	0.0010 (6)
C4	0.0159 (7)	0.0177 (7)	0.0241 (8)	−0.0026 (5)	0.0102 (6)	−0.0037 (6)
C5	0.0163 (7)	0.0188 (7)	0.0148 (7)	−0.0015 (5)	0.0064 (6)	0.0003 (5)
C6	0.0147 (7)	0.0191 (7)	0.0210 (7)	0.0024 (5)	0.0094 (6)	−0.0002 (6)
C7	0.0218 (8)	0.0268 (8)	0.0216 (8)	0.0020 (6)	0.0116 (6)	−0.0015 (6)
C8	0.0216 (8)	0.0222 (7)	0.0259 (8)	0.0028 (6)	0.0114 (7)	0.0028 (6)
C9	0.0165 (8)	0.0241 (7)	0.0288 (8)	−0.0011 (6)	0.0107 (7)	−0.0011 (6)

Geometric parameters (Å, °)

O1—C5	1.2039 (17)	C4—H4B	0.9900
O2—C5	1.3325 (16)	C6—C7	1.513 (2)
O2—C6	1.4910 (16)	C6—C9	1.5207 (19)
N1—C1	1.3131 (19)	C6—C8	1.5209 (19)

N1—C2	1.379 (2)	C7—H7A	0.9800
N2—C1	1.3503 (18)	C7—H7B	0.9800
N2—C3	1.3709 (18)	C7—H7C	0.9800
N2—C4	1.4482 (17)	C8—H8A	0.9800
C1—H1	0.9500	C8—H8B	0.9800
C2—C3	1.353 (2)	C8—H8C	0.9800
C2—H2	0.9500	C9—H9A	0.9800
C3—H3	0.9500	C9—H9B	0.9800
C4—C5	1.5208 (19)	C9—H9C	0.9800
C4—H4A	0.9900		

C5—O2—C6	121.56 (10)	O2—C6—C9	102.05 (11)
C1—N1—C2	104.32 (12)	C7—C6—C9	111.34 (12)
C1—N2—C3	106.85 (12)	O2—C6—C8	109.88 (11)
C1—N2—C4	127.15 (12)	C7—C6—C8	112.35 (12)
C3—N2—C4	125.75 (12)	C9—C6—C8	111.06 (12)
N1—C1—N2	112.36 (13)	C6—C7—H7A	109.5
N1—C1—H1	123.8	C6—C7—H7B	109.5
N2—C1—H1	123.8	H7A—C7—H7B	109.5
C3—C2—N1	110.88 (13)	C6—C7—H7C	109.5
C3—C2—H2	124.6	H7A—C7—H7C	109.5
N1—C2—H2	124.6	H7B—C7—H7C	109.5
C2—C3—N2	105.60 (13)	C6—C8—H8A	109.5
C2—C3—H3	127.2	C6—C8—H8B	109.5
N2—C3—H3	127.2	H8A—C8—H8B	109.5
N2—C4—C5	111.43 (11)	C6—C8—H8C	109.5
N2—C4—H4A	109.3	H8A—C8—H8C	109.5
C5—C4—H4A	109.3	H8B—C8—H8C	109.5
N2—C4—H4B	109.3	C6—C9—H9A	109.5
C5—C4—H4B	109.3	C6—C9—H9B	109.5
H4A—C4—H4B	108.0	H9A—C9—H9B	109.5
O1—C5—O2	126.65 (13)	C6—C9—H9C	109.5
O1—C5—C4	124.35 (13)	H9A—C9—H9C	109.5
O2—C5—C4	108.99 (11)	H9B—C9—H9C	109.5
O2—C6—C7	109.68 (11)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4B···O2$^{\rm i}$	0.99	2.56	3.3768 (18)	140.

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

Fig. 1