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tert-Butyl imidazole-1-carboxylate

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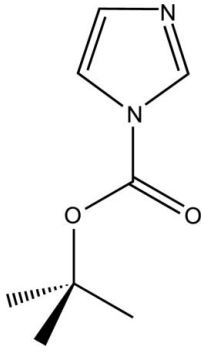
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.104; data-to-parameter ratio = 18.6.

In the title compound, $C_8H_{12}N_2O_2$, molecules are interconnected by weak C—H···O contacts with H···O distances of 2.30 Å, resulting in the formation of chains along [100]. According to graph-set analysis, the unitary descriptor of these chains is $C(5)$. In addition, there are π–π stacking interactions between pyrazole rings (centroid distance = 3.878 Å and ring plane distance = 3.26 Å).

Related literature

The title compound is a well known organic compound and was prepared according to a recently published procedure (Jia *et al.*, 2007). For details of graph-set analysis see: Etter *et al.* (1990); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_{12}N_2O_2$
M_r = 168.19
Monoclinic, $P2_1/c$
a = 5.9952 (2) Å
b = 13.2507 (4) Å
c = 11.5564 (4) Å
β = 94.201 (2)°
V = 915.58 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 200 (2) K
0.50 × 0.38 × 0.38 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
6875 measured reflections
2097 independent reflections
1650 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.07
2097 reflections
113 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2^{i}	0.95	2.30	3.1949 (16)	156

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

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2854).

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supporting information

Acta Cryst. (2009). E**65**, o405 [doi:10.1107/S1600536809003110]

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S1. Comment

The title compound was synthesized in a multistep sythesis in an attempt to create new complexing ligands.

The molecule is an imidazole protected by the *tert*-butyloxycarbonyle (Boc) group in the 1 position (see Fig. 1).

The crystal packing is shown in Fig. 2. In the crystal, weak C—H···O contacts along [100] which can be described according to graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995) with a unitary *C*(5) descriptor (see Fig. 3), lead to chain like structures of dimeric units which are formed by π-type interaction of two imidazole rings (see Fig. 4). The two imidazole rings are separated by about 3.26 Å and shifted, which results in only about half of one ring overlapping with the other ring (see Fig. 5).

Interestingly the imidazoles do not form longer strands of π-type interacting aromatic systems but only dimeric units which might be due to the large space occupied by the Boc protecting group which leads to separate strands of C—H···O bridged dimeric units (see Fig. 2).

S2. Experimental

Boc_2O was reacted solvent free with one equivalent of imidazole. After the CO_2 gas evolution had finished, the byproduct, *t*-butanole, was removed by fine vacuum and big colorless crystals of the title compound were obtained.

S3. Refinement

H atoms were placed in calculated positions (C—H 0.95 Å for aromatic C atoms and C—H 0.98 Å for methyl C atoms) and were included in the refinement in the riding model approximation with U(H) set to $1.2U_{eq}$(C) for aromatic C atoms and $1.5U_{eq}$(C) for methyl C atoms.

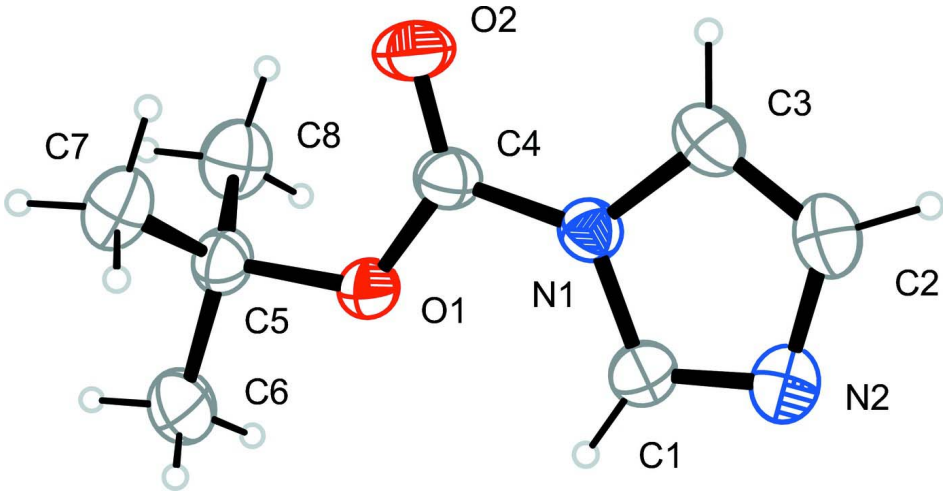

Figure 1

The molecular structure of the title compound with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

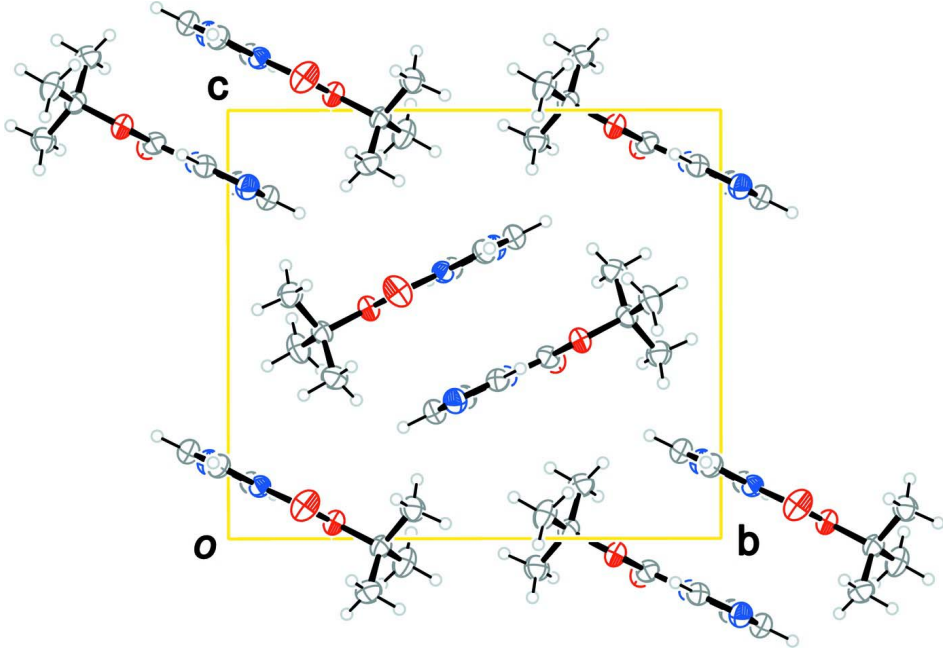

Figure 2

The packing of the title compound, viewed along [$\bar{1}$00].

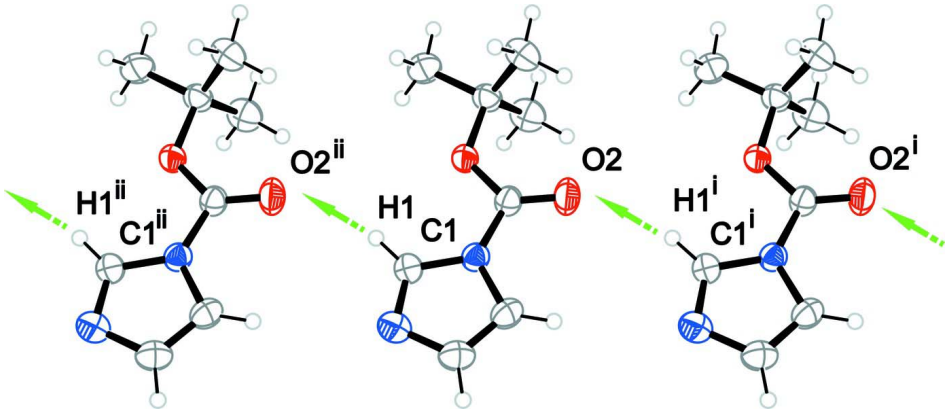

Figure 3

Weak C—H···O interactions lead to chain-like structures in the crystal structure along [100] shown here normal to [001]. Symmetry codes: (i) $x + 1, y, z$; (ii) $x - 1, y, z$.

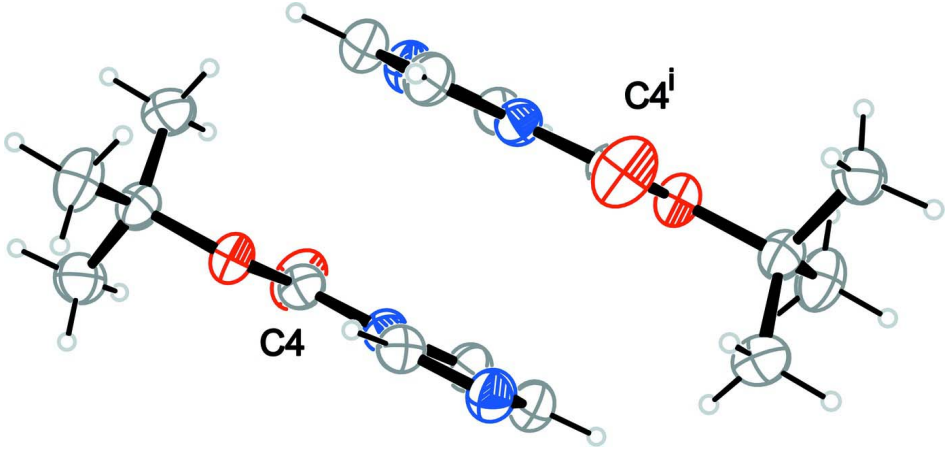

Figure 4

π interaction leads to dimeric units shown here normal to [100]. Symmetry code: (i) $-x, -y, -z$.

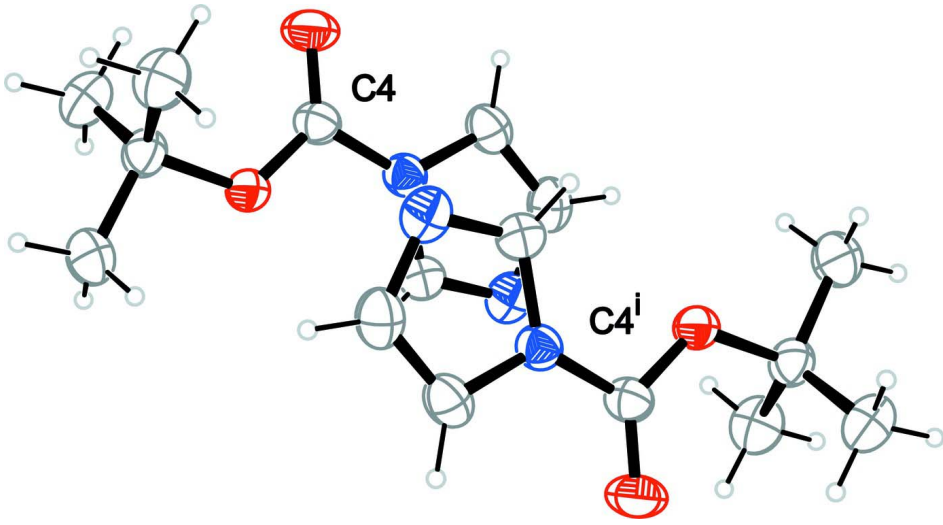

Figure 5

The dimeric units formed by π interaction are shifted, so that only half of the imidazole rings overlap. Two of the imidazole rings are shown here, normal to [001]. Symmetry code: (i) -*x*, -*y*, -*z*.

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Crystal data

$C_8H_{12}N_2O_2$
$M_r = 168.19$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.9952\ (2)$ Å
$b = 13.2507\ (4)$ Å
$c = 11.5564\ (4)$ Å
$\beta = 94.201\ (2)°$
$V = 915.58\ (5)$ Å^3
$Z = 4$
$F(000) = 360$
$D_x = 1.220$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3660 reflections
$\theta = 3.1–27.5°$
$\mu = 0.09$ mm^{-1}
$T = 200$ K
Block, colourless
0.50 × 0.38 × 0.38 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: rotating anode
MONTEL, graded multilayered X-ray optics monochromator
CCD; rotation images; thick slices scans
6875 measured reflections
2097 independent reflections
1650 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.4°$
$h = -7\rightarrow7$
$k = -17\rightarrow17$
$l = -14\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.104$
$S = 1.07$
2097 reflections
113 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
$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 0.2307P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.063 (6)

Special details

Refinement. Hydrogen atoms were placed in calculated positions (C–H 0.95 Å for aromatic C atoms and C–H 0.98 Å for methyl C atoms) and were included in the refinement in the riding model approximation with U(H) set to 1.2 U_{eq}(C) for aromatic C atoms and 1.5 U_{eq}(C) for methyl C atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.15042 (14)	0.21450 (6)	0.03780 (8)	0.0356 (3)
O2	0.50241 (15)	0.15584 (8)	0.07833 (10)	0.0527 (3)
N1	0.19880 (17)	0.06402 (8)	0.12264 (9)	0.0329 (3)
N2	−0.0623 (2)	−0.03954 (9)	0.17875 (11)	0.0442 (3)
C1	−0.0253 (2)	0.04699 (10)	0.13009 (11)	0.0359 (3)
H1	−0.1397	0.0927	0.1031	0.043*
C2	0.1485 (2)	−0.08085 (11)	0.20408 (13)	0.0450 (4)
H2	0.1750	−0.1445	0.2404	0.054*
C3	0.3103 (2)	−0.01943 (10)	0.17071 (12)	0.0410 (3)
H3	0.4671	−0.0307	0.1784	0.049*
C4	0.3034 (2)	0.14897 (10)	0.07701 (11)	0.0350 (3)
C5	0.2187 (2)	0.30947 (10)	−0.01876 (12)	0.0373 (3)
C6	−0.0052 (3)	0.35703 (12)	−0.05516 (15)	0.0526 (4)
H6A	−0.0861	0.3708	0.0138	0.079*
H6B	−0.0929	0.3107	−0.1065	0.079*
H6C	0.0187	0.4204	−0.0963	0.079*
C7	0.3515 (3)	0.37416 (11)	0.06973 (13)	0.0485 (4)
H7A	0.4941	0.3410	0.0926	0.073*
H7B	0.2661	0.3830	0.1382	0.073*
H7C	0.3801	0.4402	0.0357	0.073*
C8	0.3472 (3)	0.28433 (12)	−0.12317 (13)	0.0501 (4)
H8A	0.2566	0.2400	−0.1756	0.075*
H8B	0.4871	0.2501	−0.0974	0.075*
H8C	0.3812	0.3467	−0.1640	0.075*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0293 (5)	0.0336 (5)	0.0438 (5)	−0.0001 (3)	0.0018 (4)	0.0068 (4)
O2	0.0279 (5)	0.0595 (7)	0.0709 (7)	0.0038 (4)	0.0052 (5)	0.0188 (6)
N1	0.0322 (6)	0.0335 (6)	0.0329 (5)	0.0020 (4)	0.0007 (4)	0.0020 (4)
N2	0.0469 (7)	0.0399 (6)	0.0458 (7)	−0.0045 (5)	0.0035 (5)	0.0022 (5)
C1	0.0334 (7)	0.0376 (7)	0.0366 (7)	−0.0013 (5)	0.0017 (5)	−0.0004 (5)
C2	0.0549 (9)	0.0359 (7)	0.0436 (8)	0.0016 (6)	0.0004 (6)	0.0049 (6)
C3	0.0424 (8)	0.0385 (7)	0.0413 (7)	0.0078 (6)	−0.0022 (6)	0.0036 (6)
C4	0.0312 (7)	0.0384 (7)	0.0353 (6)	0.0023 (5)	0.0016 (5)	0.0028 (5)
C5	0.0389 (7)	0.0338 (7)	0.0392 (7)	−0.0034 (5)	0.0036 (5)	0.0049 (5)

C6	0.0507 (9)	0.0407 (8)	0.0654 (10)	0.0045 (7)	−0.0025 (7)	0.0133 (7)
C7	0.0532 (9)	0.0456 (8)	0.0470 (8)	−0.0127 (7)	0.0054 (7)	−0.0033 (7)
C8	0.0600 (10)	0.0514 (9)	0.0397 (8)	−0.0053 (7)	0.0093 (7)	0.0038 (7)

Geometric parameters (Å, º)

O1—C4	1.3187 (15)	C5—C6	1.5144 (19)
O1—C5	1.4892 (15)	C5—C7	1.5147 (19)
O2—C4	1.1958 (15)	C5—C8	1.515 (2)
N1—C1	1.3714 (16)	C6—H6A	0.9800
N1—C3	1.3870 (16)	C6—H6B	0.9800
N1—C4	1.4094 (17)	C6—H6C	0.9800
N2—C1	1.3032 (17)	C7—H7A	0.9800
N2—C2	1.3886 (19)	C7—H7B	0.9800
C1—H1	0.9500	C7—H7C	0.9800
C2—C3	1.344 (2)	C8—H8A	0.9800
C2—H2	0.9500	C8—H8B	0.9800
C3—H3	0.9500	C8—H8C	0.9800
C4—O1—C5	120.04 (10)	C6—C5—C8	111.24 (12)
C1—N1—C3	106.82 (11)	C7—C5—C8	112.92 (12)
C1—N1—C4	128.28 (10)	C5—C6—H6A	109.5
C3—N1—C4	124.90 (11)	C5—C6—H6B	109.5
C1—N2—C2	104.88 (11)	H6A—C6—H6B	109.5
N2—C1—N1	111.73 (11)	C5—C6—H6C	109.5
N2—C1—H1	124.1	H6A—C6—H6C	109.5
N1—C1—H1	124.1	H6B—C6—H6C	109.5
C3—C2—N2	111.44 (12)	C5—C7—H7A	109.5
C3—C2—H2	124.3	C5—C7—H7B	109.5
N2—C2—H2	124.3	H7A—C7—H7B	109.5
C2—C3—N1	105.13 (12)	C5—C7—H7C	109.5
C2—C3—H3	127.4	H7A—C7—H7C	109.5
N1—C3—H3	127.4	H7B—C7—H7C	109.5
O2—C4—O1	128.48 (12)	C5—C8—H8A	109.5
O2—C4—N1	121.79 (11)	C5—C8—H8B	109.5
O1—C4—N1	109.72 (10)	H8A—C8—H8B	109.5
O1—C5—C6	101.94 (10)	C5—C8—H8C	109.5
O1—C5—C7	109.24 (11)	H8A—C8—H8C	109.5
C6—C5—C7	111.30 (12)	H8B—C8—H8C	109.5
O1—C5—C8	109.63 (11)		
C2—N2—C1—N1	−0.07 (15)	C5—O1—C4—N1	−177.82 (10)
C3—N1—C1—N2	0.14 (15)	C1—N1—C4—O2	178.00 (13)
C4—N1—C1—N2	−179.21 (12)	C3—N1—C4—O2	−1.2 (2)
C1—N2—C2—C3	−0.04 (16)	C1—N1—C4—O1	−0.98 (18)
N2—C2—C3—N1	0.12 (16)	C3—N1—C4—O1	179.77 (11)
C1—N1—C3—C2	−0.15 (14)	C4—O1—C5—C6	176.89 (11)
C4—N1—C3—C2	179.23 (12)	C4—O1—C5—C7	−65.27 (15)

C5—O1—C4—O2	3.3 (2)	C4—O1—C5—C8	58.95 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2^{i}	0.95	2.30	3.1949 (16)	156

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