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N-tert-Butylmorpholine-4-carboxamide

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Key indicators

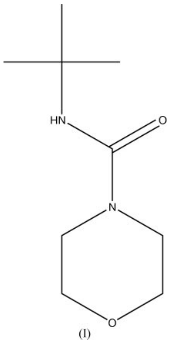
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.065
wR factor = 0.172
Data-to-parameter ratio = 10.6

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

The structure of the title compound, $C_9H_{18}N_2O_2$, has been determined. The morpholine ring exists in an almost ideal chair conformation. The central part of the molecule is almost ideally planar. The molecule is stabilized by weak C—H···N and C—H···O hydrogen bonds. Molecules of the title compound associate *via* N—H···O and C—H···O interactions to form a linear hydrogen-bonded chain along the *z* axis.

Comment

The transparent colourless crystals of the title compound, (I), become brown during the data collection. A reference crystal standing in the diffractometer box, but not in the X-ray beam, did not change colour.



(I)

A perspective view of the structure together with the atom-numbering scheme is shown in Fig. 1. All interatomic distances are normal. The morpholine ring exists in an almost ideal chair conformation, supported by the asymmetry parameters (Fig. 2; Duax & Norton, 1975). The central part of the molecule consisting of atoms N1, C5, N2, C6 and O2 is almost ideally planar. For the least-squares plane calculated through atoms N1, C5, N2 and C6, the maximum deviation of 0.0316 (12) Å occurs for N1 and for the least-squares plane calculated through atoms N1, C5, N2, C6 and O2, the maximum deviation of 0.0413 (15) Å occurs for the same atom. The molecule has additional stabilization provided by intramolecular weak hydrogen bonds, *viz*. C1—H1*A*···N2 and C4—H4*B*···O2. These hydrogen bonds also explain the orientation of the morpholinyl substituent in relation to the urea chain, as shown by the torsion angles (Table 1).

Molecules of the title compound associate *via* N—H···O and C—H···O interactions to form a linear hydrogen-bonded chain along the *z* axis (Fig. 3).

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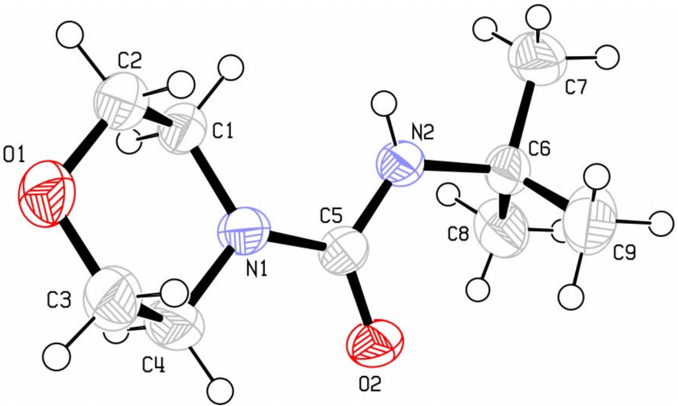

Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

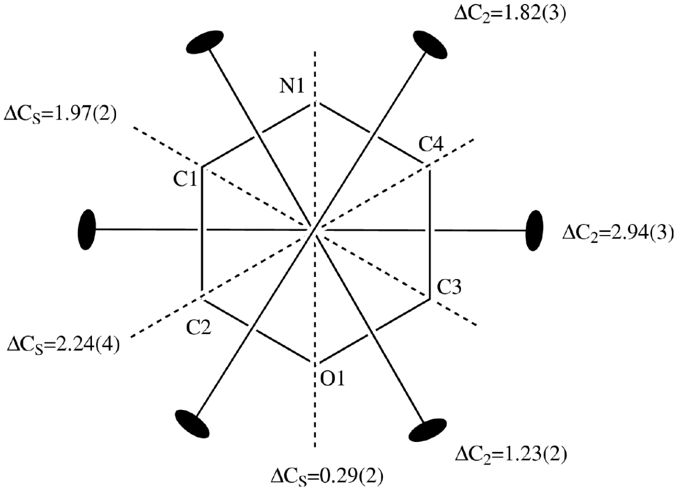

Figure 2
Asymmetry parameters (Duax & Norton, 1975) for (I).

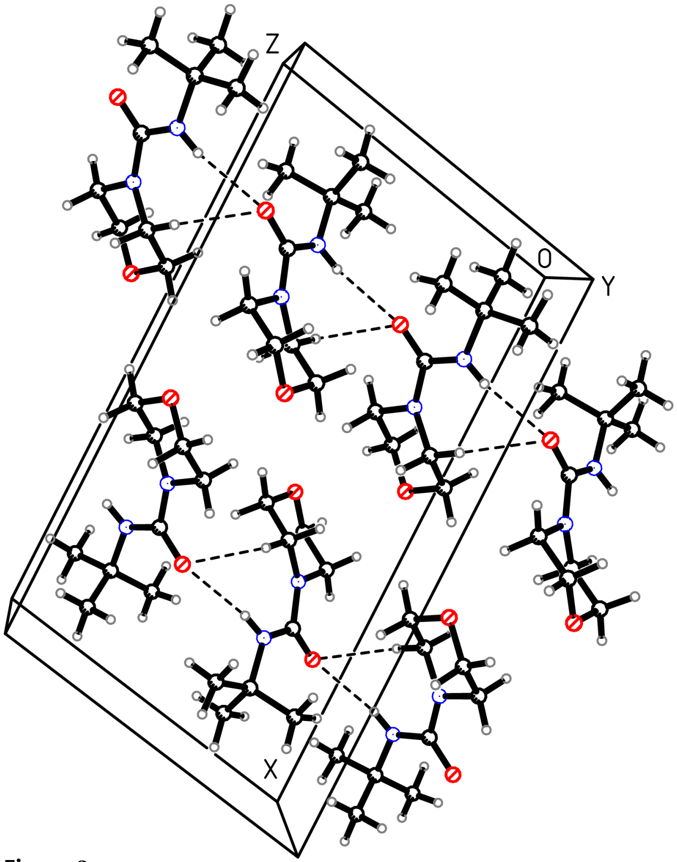

Figure 3
A view along the *z* axis of part of the crystal structure of the title compound showing intermolecular hydrogen bonds creating the chain structure. Hydrogen bonds are indicated by dashed lines.

Experimental

Morpholine (2 mmol, 0.174 g) and *tert*-butyl isocyanate (2.4 mmol, 0.24 g) were dissolved in 20 ml of dry benzene (distilled over CaH_2) and stirred at room temperature for 24 h. Benzene was distilled off under reduced pressure leaving 0.369 g (99.2% theoretical yield) of colourless crystalline solid. Recrystallization from benzene yielded $C(CH_3)_3NHC(O)[N(CH_2CH_2)_2O]$ in the form of colourless well shaped crystals (m.p. 448.7 K) (acc. to the Differential Scanning Calorimetry curve). 1H NMR (in $CDCl_3$, chemical shifts in p.p.m): δ[NH(CO)] 7.26 (*s*, 2H), $\delta(OCH_2CH_2N)$ 3.68–3.71, $\delta[NH(CO)N\text{-}CH_2CH_2O]$ 3.27–3.30 (*t*, 4H), $\delta(C{-}CH_3)$ 1.35 (*s*, 9H). IR (in KBr pellet, wavenumber in cm^{-1}): 2858, 2973 (CH_3); 1622, 1535, 3358 [NHC(O)]; 1359, 1402 [$C(CH_3)_3$]; 1002, 1027, 1109, 1220, 1270 (NCH_2 and OCH_2). UV (in CH_3OH solution): λ_{max} = 226 nm

Crystal data

$C_9H_{18}N_2O_2$
M_r = 186.25
Monoclinic, $P2_1/c$
a = 16.909 (3) Å
b = 6.525 (1) Å
c = 9.622 (2) Å
β = 101.46 (3)°
V = 1040.4 (3) Å^3
Z = 4

D_x = 1.189 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2228 reflections
θ = 5–22°
μ = 0.08 mm^{-1}
T = 293 (2) K
Rectangular prism, colourless
0.30 × 0.11 × 0.11 mm

Data collection

Kuma KM-4–CCD diffractometer
ω scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1999)
T_{min} = 0.975, T_{max} = 0.991
10 532 measured reflections
1838 independent reflections
1711 reflections with $I > 2\sigma(I)$

R_{int} = 0.090
θ_{max} = 25.1°
$h = -20 \rightarrow 20$
$k = -7 \rightarrow 7$
$l = -9 \rightarrow 11$
2 standard reflections every 50 reflections intensity decay: 10.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.172
S = 1.13
1838 reflections
173 parameters
H atoms: see below

$w = 1/[\sigma^2(F_o^2) + (0.1123P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.013 (5)

Table 1
Selected torsion angles (°).

N1—C1—C2—O1	−56.4 (3)	N2—C5—N1—C1	18.4 (3)
C1—C2—O1—C3	58.3 (3)	N2—C5—N1—C4	−178.6 (2)
C2—O1—C3—C4	−58.6 (3)	N1—C5—N2—C6	174.9 (2)
O1—C3—C4—N1	56.8 (3)	O2—C5—N2—C6	−5.1 (3)
C3—C4—N1—C1	−55.6 (3)	C5—N2—C6—C7	−179.8 (2)
C4—N1—C1—C2	55.5 (3)	C5—N2—C6—C8	63.4 (3)
O2—C5—N1—C4	1.4 (3)	C5—N2—C6—C9	−60.5 (3)
O2—C5—N1—C1	−161.6 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2N···O2^{i}	0.84 (3)	2.29 (3)	3.126 (2)	170 (2)
C1—H1*A*···O2^{i}	0.98 (3)	2.43 (3)	3.306 (3)	149 (2)
C2—H2*B*···O1^{ii}	0.98 (3)	2.59 (3)	3.523 (3)	157.4 (18)
C1—H1*A*···N2	0.98 (3)	2.50 (2)	2.859 (3)	101.6 (17)
C4—H4*B*···O2	0.98 (3)	2.33 (2)	2.724 (3)	102.9 (17)
C8—H8*A*···O2	0.88 (3)	2.52 (3)	3.039 (3)	119 (2)
C9—H9*B*···O2	1.01 (3)	2.51 (3)	3.077 (4)	115.1 (17)

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

All H atoms were found on difference Fourier syntheses, calculated after four cycles of anistropic refinement. Their positional parameters were freely refined.

Data collection: Kuma KM-4–CCD software; cell refinement: Kuma KM-4–CCD software; data reduction: Kuma KM-4–CCD software; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: XP in *SHELXTL/PC* (Sheldrick, 1990) and *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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