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Methyl 3-carboxy-7-(*tert*-butoxycarbonyl)-4,7-diazaheptanoate

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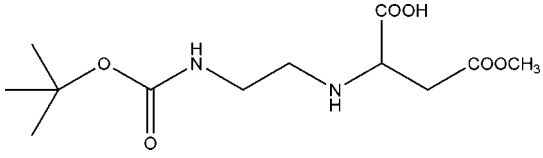
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.057; wR factor = 0.129; data-to-parameter ratio = 8.5.

In the molecule of the title compound, $C_{12}H_{22}N_2O_6$, the dihedral angle between the planar carbamoyloxy and ester C—COO units is 22.23 (3)°. In the crystal structure, intermolecular O—H···N and N—H···O hydrogen bonds link the molecules into chains.

Related literature

For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{12}H_{22}N_2O_6$
M_r = 290.32
Orthorhombic, $Pca2_1$
a = 10.824 (2) Å
b = 15.204 (3) Å
c = 8.9300 (18) Å
V = 1469.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.40 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.954, T_{max} = 0.990
1921 measured reflections
1540 independent reflections
984 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.129
S = 0.90
1540 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···N2^{i}	0.82	2.21	3.001 (6)	161
N1—H1···O3^{ii}	0.86	2.01	2.849 (8)	166
N2—H2···O3^{ii}	0.86	2.48	3.128 (8)	132
N2—H2···O4^{iii}	0.86	2.50	3.060 (6)	123

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2260).

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supplementary materials

Acta Cryst. (2007). E**63**, o3079 [**doi:10.1107/S1600536807025871**]

Methyl 3-carboxy-7-(*tert*-butoxycarbonyl)-4,7-diazaheptanoate

J.-Y. Xu, Z.-Y. Wu, W.-Y. Wu, S.-L. Dong and J.-T. Wang

Comment

In the process of synthesis, we obtained the title compound, (I), and we herein report its crystal structure.

In the molecule of (I), (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The dihedral angle between the planar (O1/O2/N1/C5) and (O5/O6/C10/C11) units is 22.23 (3)°.

In the crystal structure, the intermolecular O—H···N and N—H···O hydrogen bonds (Table 1) link the molecules into chains, in which they may be effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, maleic anhydride (10 g, 100 mmol) and methanol (50 ml) were added into a four-necked round-bottom flask fitted with a mechanical stirrer, dropping funnel and thermometer. After the mixture was refluxed in a water bath for 30 min, excess methanol was distilled off. Triethylamine (20 ml) was added to the reside, cooled in ice, and stirred very slowly. Then, *N*-(*tert*-butoxycarbonyl)-1,2-ethane diamine (16 g, 100 mmol) was added, and the mixture was stirred in a water bath for 2 h. The mixture was filtrated and washed twice with hot acetone (100 ml). Crystals of (I) suitable for X-ray analsyis were obtained by slow evaporation of methanol (10 ml) (yield; 23.5 g, 72%, m.p. 454 K).

Refinement

H atoms were positioned geometrically, with O—H = 0.82 Å (for OH), N—H = 0.86 Å (for NH) and C—H = 0.98, 0.97 and 0.96 Å for methine, methylene and methyl H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*N*,*O*), where x = 1.5 for NH and methyl H atoms, and x = 1.2 for all other H atoms.

Figures

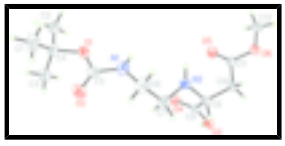

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

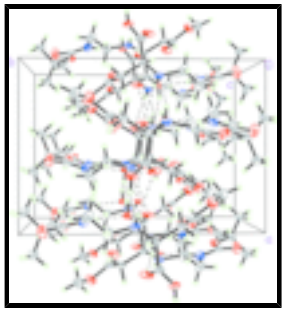

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

Methyl 3-carboxy-7-(*tert*-butoxycarbonyl)-4,7-diazaheptanoate

Crystal data

$C_{12}H_{22}N_2O_6$	D_x = 1.312 Mg m^{-3}
M_r = 290.32	Melting point: 454 K
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2c -2ac	Cell parameters from 25 reflections
a = 10.824 (2) Å	θ = 9–12º
b = 15.204 (3) Å	μ = 0.11 mm^{-1}
c = 8.9300 (18) Å	T = 298 (2) K
V = 1469.6 (5) Å^3	Block, colourless
Z = 4	0.40 × 0.30 × 0.10 mm
F_{000} = 624	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.042
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 1.3º
T = 298(2) K	h = 0→13
ω/2θ scans	k = 0→18
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→10
T_{min} = 0.954, T_{max} = 0.990	3 standard reflections
1921 measured reflections	every 200 reflections
1540 independent reflections	intensity decay: none
984 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 3.5659P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.057	$(\Delta/\sigma)_{max} < 0.001$

$wR(F^2) = 0.129$

$S = 0.90$

1540 reflections

182 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$\Delta\rho_{max} = 0.23$ e Å^{-3}

$\Delta\rho_{min} = -0.13$ e Å^{-3}

Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.9191 (4)	0.8093 (3)	0.6834 (6)	0.0502 (12)
O2	0.9209 (5)	0.8738 (3)	0.4545 (6)	0.0675 (15)
O3	1.1376 (4)	0.4304 (3)	0.1506 (6)	0.0587 (15)
H3	1.2097	0.4253	0.1777	0.088*
O4	1.1218 (3)	0.5130 (3)	0.3553 (5)	0.0538 (12)
O5	0.8910 (4)	0.3791 (3)	0.4610 (6)	0.0600 (13)
O6	0.7747 (4)	0.2899 (3)	0.3195 (6)	0.0562 (13)
N1	0.8734 (5)	0.7303 (4)	0.4871 (7)	0.0542 (15)
H1	0.8748	0.6869	0.5487	0.065*
N2	0.8842 (4)	0.5550 (3)	0.3022 (6)	0.0385 (13)
H2	0.8391	0.5417	0.3782	0.046*
C1	0.8483 (4)	0.9585 (4)	0.7424 (5)	0.072 (3)
H1B	0.8520	0.9787	0.6407	0.108*
H1C	0.8605	1.0072	0.8091	0.108*
H1A	0.7690	0.9325	0.7610	0.108*
C2	0.9464 (4)	0.8585 (4)	0.9272 (5)	0.070 (3)
H2B	1.0113	0.8162	0.9403	0.105*
H2C	0.8682	0.8314	0.9484	0.105*
H2A	0.9591	0.9070	0.9944	0.105*
C3	1.0736 (4)	0.9238 (4)	0.7211 (4)	0.070 (2)
H3A	1.0700	0.9451	0.6200	0.105*
H3B	1.1318	0.8763	0.7272	0.105*
H3C	1.0991	0.9706	0.7864	0.105*

C4	0.9472 (4)	0.8915 (4)	0.7681 (5)	0.055 (2)
C5	0.9062 (4)	0.8111 (4)	0.5360 (5)	0.0466 (17)
C6	0.8359 (4)	0.7149 (4)	0.3337 (4)	0.065 (2)
H6A	0.8419	0.7698	0.2786	0.078*
H6B	0.7499	0.6969	0.3328	0.078*
C7	0.9117 (4)	0.6461 (4)	0.2543 (4)	0.0552 (19)
H7A	0.8972	0.6509	0.1474	0.066*
H7B	0.9986	0.6579	0.2720	0.066*
C8	0.9439 (4)	0.4897 (4)	0.2033 (4)	0.0359 (14)
H8	0.9422	0.5149	0.1023	0.043*
C9	1.0804 (4)	0.4764 (4)	0.2425 (5)	0.0365 (14)
C10	0.8706 (4)	0.4040 (4)	0.1954 (4)	0.0362 (14)
H10A	0.7907	0.4162	0.1507	0.043*
H10B	0.9209	0.3647	0.1354	0.043*
C11	0.8504 (4)	0.3591 (4)	0.3417 (4)	0.0420 (15)
C12	0.7384 (4)	0.2387 (4)	0.4468 (5)	0.070 (2)
H12A	0.8051	0.2366	0.5175	0.105*
H12B	0.7182	0.1801	0.4152	0.105*
H12C	0.6674	0.2652	0.4931	0.105*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.046 (3)	0.045 (2)	0.059 (3)	−0.009 (2)	−0.003 (3)	−0.002 (2)
O2	0.086 (4)	0.050 (3)	0.067 (4)	−0.005 (3)	0.000 (3)	0.013 (3)
O3	0.025 (2)	0.052 (3)	0.099 (4)	0.002 (2)	0.016 (3)	−0.012 (3)
O4	0.0221 (18)	0.092 (4)	0.047 (3)	−0.010 (2)	−0.008 (2)	0.009 (3)
O5	0.049 (3)	0.076 (3)	0.055 (3)	−0.013 (2)	−0.007 (3)	−0.005 (3)
O6	0.049 (3)	0.058 (3)	0.062 (3)	−0.015 (2)	0.010 (3)	−0.010 (3)
N1	0.061 (4)	0.048 (3)	0.053 (4)	0.008 (3)	−0.009 (3)	−0.006 (3)
N2	0.020 (2)	0.050 (3)	0.045 (3)	0.001 (2)	−0.001 (2)	0.002 (3)
C1	0.051 (5)	0.067 (5)	0.102 (9)	0.024 (4)	−0.010 (6)	−0.022 (6)
C2	0.049 (4)	0.087 (7)	0.074 (6)	−0.011 (4)	0.004 (4)	−0.028 (5)
C3	0.045 (4)	0.061 (4)	0.104 (7)	−0.006 (3)	−0.008 (5)	−0.008 (5)
C4	0.034 (3)	0.047 (4)	0.082 (6)	0.002 (3)	−0.007 (4)	−0.018 (4)
C5	0.027 (3)	0.061 (5)	0.052 (4)	0.005 (3)	0.003 (3)	0.008 (4)
C6	0.071 (5)	0.067 (5)	0.059 (5)	0.025 (4)	−0.011 (5)	−0.002 (5)
C7	0.076 (5)	0.049 (4)	0.041 (4)	0.011 (4)	−0.002 (4)	0.006 (3)
C8	0.018 (2)	0.045 (3)	0.044 (4)	0.001 (2)	0.001 (3)	0.000 (3)
C9	0.018 (2)	0.047 (3)	0.044 (4)	0.005 (3)	−0.001 (3)	0.002 (3)
C10	0.021 (3)	0.052 (3)	0.036 (3)	−0.001 (3)	−0.001 (3)	−0.008 (3)
C11	0.020 (3)	0.052 (4)	0.054 (4)	0.003 (3)	0.002 (3)	−0.016 (4)
C12	0.058 (4)	0.070 (4)	0.083 (6)	−0.004 (4)	0.029 (5)	−0.002 (5)

Geometric parameters (Å, °)

O1—C5	1.324 (8)	C2—H2C	0.9600
O1—C4	1.492 (8)	C2—H2A	0.9600
O2—C5	1.211 (8)	C3—C4	1.513 (9)

O3—C9	1.244 (7)	C3—H3A	0.9600
O3—H3	0.8200	C3—H3B	0.9600
O4—C9	1.235 (7)	C3—H3C	0.9600
O5—C11	1.192 (8)	C6—C7	1.508 (9)
O6—C11	1.348 (7)	C6—H6A	0.9700
O6—C12	1.433 (8)	C6—H6B	0.9700
N1—C5	1.351 (8)	C7—H7A	0.9700
N1—C6	1.448 (10)	C7—H7B	0.9700
N1—H1	0.8600	C8—C9	1.531 (7)
N2—C8	1.477 (7)	C8—C10	1.528 (7)
N2—C7	1.479 (8)	C8—H8	0.9800
N2—H2	0.8600	C10—C11	1.491 (9)
C1—C4	1.495 (9)	C10—H10A	0.9700
C1—H1B	0.9600	C10—H10B	0.9701
C1—H1C	0.9600	C12—H12A	0.9600
C1—H1A	0.9600	C12—H12B	0.9600
C2—C4	1.507 (11)	C12—H12C	0.9600
C2—H2B	0.9600		
C5—O1—C4	120.5 (6)	N1—C6—C7	113.8 (6)
C9—O3—H3	109.5	N1—C6—H6A	108.8
C11—O6—C12	118.3 (6)	C7—C6—H6A	108.8
C5—N1—C6	121.8 (6)	N1—C6—H6B	108.8
C5—N1—H1	119.1	C7—C6—H6B	108.8
C6—N1—H1	119.1	H6A—C6—H6B	107.7
C8—N2—C7	111.6 (5)	N2—C7—C6	113.8 (6)
C8—N2—H2	124.2	N2—C7—H7A	108.8
C7—N2—H2	124.2	C6—C7—H7A	108.8
C4—C1—H1B	109.5	N2—C7—H7B	108.8
C4—C1—H1C	109.5	C6—C7—H7B	108.8
H1B—C1—H1C	109.5	H7A—C7—H7B	107.7
C4—C1—H1A	109.5	N2—C8—C9	112.0 (5)
H1B—C1—H1A	109.5	N2—C8—C10	111.9 (4)
H1C—C1—H1A	109.5	C9—C8—C10	113.5 (5)
C4—C2—H2B	109.5	N2—C8—H8	106.3
C4—C2—H2C	109.5	C9—C8—H8	106.3
H2B—C2—H2C	109.5	C10—C8—H8	106.3
C4—C2—H2A	109.5	O4—C9—O3	127.6 (5)
H2B—C2—H2A	109.5	O4—C9—C8	118.5 (5)
H2C—C2—H2A	109.5	O3—C9—C8	113.9 (6)
C4—C3—H3A	109.5	C11—C10—C8	115.2 (5)
C4—C3—H3B	109.5	C11—C10—H10A	108.5
H3A—C3—H3B	109.5	C8—C10—H10A	108.6
C4—C3—H3C	109.5	C11—C10—H10B	106.5
H3A—C3—H3C	109.5	C8—C10—H10B	105.1
H3B—C3—H3C	109.5	H10A—C10—H10B	113.0
O1—C4—C1	110.3 (6)	O5—C11—O6	123.7 (7)
O1—C4—C2	101.5 (6)	O5—C11—C10	127.8 (5)
C1—C4—C2	111.5 (7)	O6—C11—C10	108.5 (6)
O1—C4—C3	108.4 (6)	O6—C12—H12A	109.5

C1—C4—C3	112.5 (6)	O6—C12—H12B	109.5
C2—C4—C3	112.0 (6)	H12A—C12—H12B	109.5
O2—C5—O1	126.9 (7)	O6—C12—H12C	109.5
O2—C5—N1	123.8 (7)	H12A—C12—H12C	109.5
O1—C5—N1	109.3 (6)	H12B—C12—H12C	109.5
C5—O1—C4—C1	58.8 (9)	C7—N2—C8—C10	−150.7 (5)
C5—O1—C4—C2	177.1 (6)	N2—C8—C9—O4	6.5 (8)
C5—O1—C4—C3	−64.9 (7)	C10—C8—C9—O4	−121.5 (6)
C4—O1—C5—O2	4.1 (10)	N2—C8—C9—O3	−171.6 (5)
C4—O1—C5—N1	−176.0 (5)	C10—C8—C9—O3	60.5 (7)
C6—N1—C5—O2	−8.8 (10)	N2—C8—C10—C11	−58.4 (6)
C6—N1—C5—O1	171.2 (6)	C9—C8—C10—C11	69.6 (6)
C5—N1—C6—C7	123.3 (7)	C12—O6—C11—O5	1.6 (8)
C8—N2—C7—C6	169.8 (5)	C12—O6—C11—C10	−177.6 (5)
N1—C6—C7—N2	74.1 (8)	C8—C10—C11—O5	−5.2 (8)
C7—N2—C8—C9	80.5 (6)	C8—C10—C11—O6	173.9 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···N2^{i}	0.82	2.21	3.001 (6)	161
N1—H1···O3^{ii}	0.86	2.01	2.849 (8)	166
N2—H2···O3^{ii}	0.86	2.48	3.128 (8)	132
N2—H2···O4^{iii}	0.86	2.50	3.060 (6)	123

Symmetry codes: (i) *x*+1/2, −*y*+1, *z*; (ii) −*x*+2, −*y*+1, *z*+1/2; (iii) *x*−1/2, −*y*+1, *z*.

Fig. 1

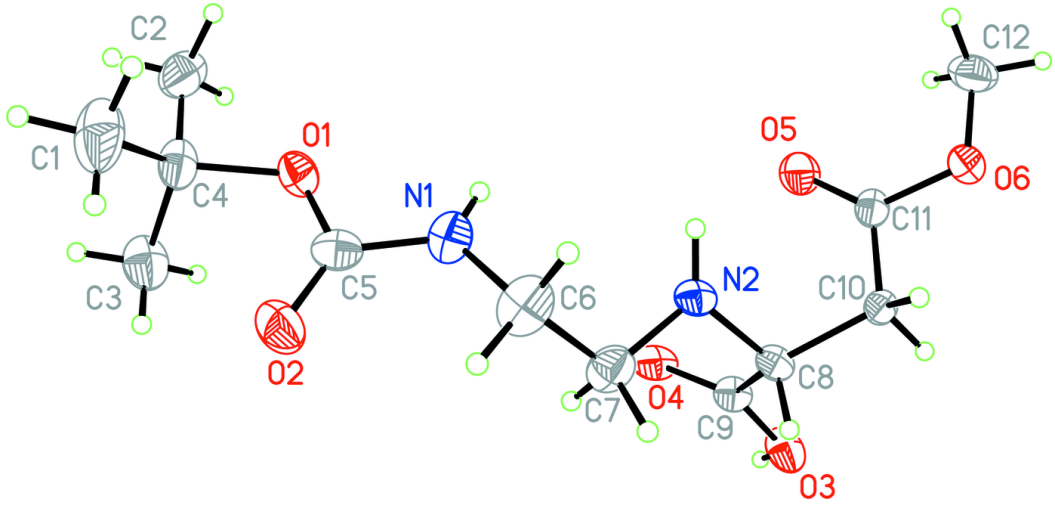

Fig. 2