Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*S*)-*O*-Succinimidyl *N*-[2-(*tert*-butoxycarbonylamino)-propyl]carbamate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.037
wR factor = 0.097
Data-to-parameter ratio = 8.7

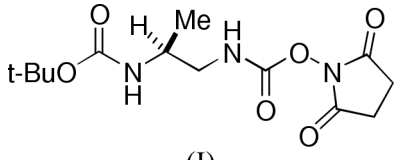
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 10 January 2001
Accepted 5 February 2001
Online 13 February 2001

The molecule of activated carbamate, (*S*)-2,5-dioxopyrrolidin-1-yl *N*-[2-(*tert*-butoxycarbonylamino)propyl]carbamate, ^{t}BuOCONHCH(Me)CH_2NHCOONC$_4$H$_4$O$_2$ or $C_{13}H_{21}N_3O_6$, prepared from *N*-Boc-β^3HAla-OH, assumes a folded conformation with the N—C—C—N torsion angle equal to 55.9 (3)°. Both N—H groups are involved in intermolecular hydrogen bonds, forming infinite chains in the crystal.

Comment

Unnatural biopolymers with a urea backbone, such as *N,N*-linked oligoureas [N(CONHR)-$(CH_2)_m$-]$_n$ (Nowick, 1999), *N,N′*-linked oligoureas [NH-CHR-CH_2-NH-CO-]$_n$ (Burgess *et al.*, 1995, 1997; Kim *et al.*, 1996; Boeijen & Liskamp, 1999; Guichard *et al.*, 1999, 2000; Tamilarasu *et al.*, 1999), ureidopeptoids [N*R*-CH_2-CH_2-NH-CO-]$_n$ (Kruijzer *et al.*, 1997; Wilson & Nowick, 1998) and oligomeric cyclic ureas (Kim *et al.*, 1996) have been described recently as peptide backbone mimetics or as templates for the creation of artificial β-sheets. The urea fragment appears particularly promising for drug discovery because of its expected metabolic stability and interesting hydrogen-bonding properties. We have recently reported a simple and effective synthesis of *O*-succinimidyl 2-(*tert*-butoxycarbonylamino)-2-subsituted-ethylcarbamate derivatives starting from the corresponding N-protected β-amino acids and their use as activated monomers in the synthesis of di- and trisubstituted ureas and *N,N′*-linked oligoureas (Guichard *et al.*, 1999). These derivatives are stable compounds that react readily with amines to form substituted ureas. Furthermore, the mild conditions required for their preparation are compatible with most functionalized side chains of amino acids as well as with standard protecting groups used in solid-phase peptide synthesis (Guichard *et al.*, 2000). Herein, we report the crystal structure of (*S*)-*O*-succinimidyl *N*-[2-(*tert*-butoxycarbonylamino)propyl]carbamate, (I), which was prepared in three steps from Boc-(*S*)-β^3HAla-OH.



(I)

Bond distances and angles of the succinimide ring are in good agreement with those recently published by Tenon *et al.* (2000) and Guichard *et al.* (1999) for *N*-methylsuccinimide and *O*-succinimidyl (2-nitrophenyl)carbamate, respectively. The

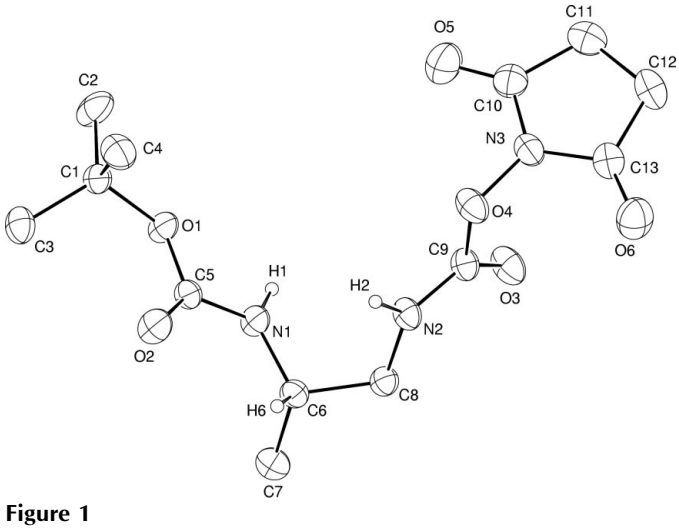


Figure 1
The molecular structure of (I) with the atom-numbering scheme and 25% probability displacement ellipsoids. H atoms are shown only at the N atoms and at the chiral center.

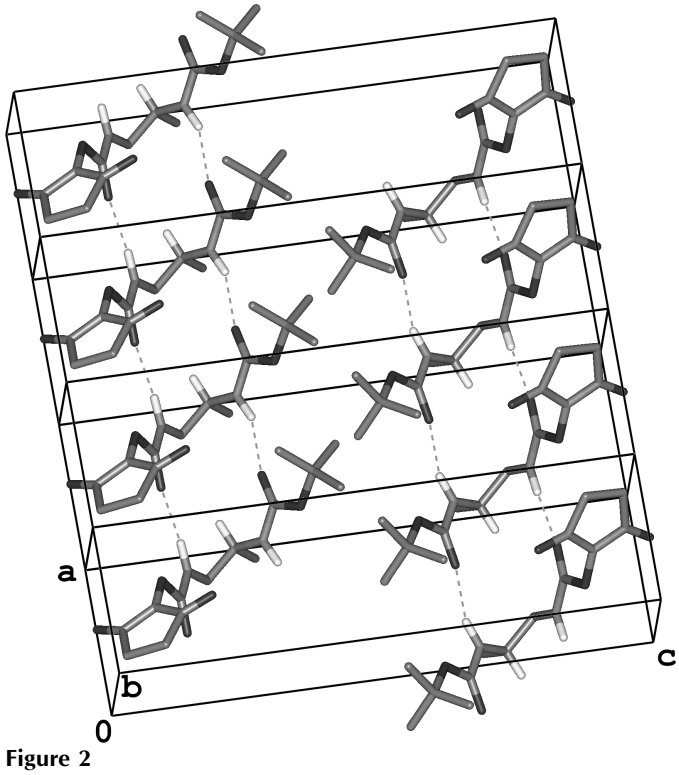


Figure 2
Packing of the molecules showing the infinite chains and the hydrogen-bonding network (in dashed lines).

succinimide ring in (I) is nearly planar, as in the unsubstituted succinimide (Mason, 1961) and *O*-succinimidyl (2-nitrophenyl)carbamate (Guichard *et al.*, 1999) molecules. Indeed, the puckering parameters of the succinimide ring in the title compound are q = 0.010 Å and φ_2 = 314.0° for the sequence N3—C10—C11—C12—C13 (Cremer & Pople, 1975).

The molecule (Fig. 1 and Table 1) assumes a folded shape with the *gauche* conformation about the central C6—C8 bond in the main chain, the N1—C6—C8—N2 torsion angle being equal to 55.9 (3)°. The molecules of the title compound in the crystal are linked into infinite chains *via* C═O···H—N hydrogen bonds (Fig. 2 and Table 2). The chains are stretched along the [100] direction and form a parallel β sheet-like arrangement. All interactions between the chains are purely van der Waals in nature.

Experimental

O-succinimidyl carbamate was prepared by homologation of Boc-*L*-Ala-OH, with subsequent conversion of Boc-(*S*)-β^3HAla-OH [we used the nomenclature proposed by Seebach & Matthews (1997) for β-amino acids] to the corresponding acyl azide and trapping of the intermediate isocyanate, resulting from Curtius rearrangement of the acyl azide, with N-hydroxysuccinimide. Details of the synthetic procedures are available in the CIF file.

Crystal data

$C_{13}H_{21}N_3O_6$	D_x = 1.273 Mg m^{-3}
M_r = 315.33	Mo $K\alpha$ radiation
Monoclinic, $P2$	Cell parameters from 5236 reflections
a = 5.1260 (2) Å	θ = 4.0–26.3°
b = 8.5650 (4) Å	μ = 0.10 mm^{-1}
c = 18.7540 (9) Å	T = 293 (2) K
β = 91.996 (3)°	Prismatic, colorless
V = 822.88 (6) Å^3	0.30 × 0.25 × 0.22 mm
Z = 2	

Data collection

KappaCCD diffractometer	R_{int} = 0.030
φ and ω scans	θ_{max} = 26.3°
5236 measured reflections	$h = 0 \rightarrow 6$
1792 independent reflections	$k = 0 \rightarrow 10$
1436 reflections with $I > 2\sigma(I)$	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.0391P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.037	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.097	$\Delta\rho_{max}$ = 0.11 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
1792 reflections	
205 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (°).

O1—C5—N1—C6	−177.9 (2)	C8—N2—C9—O4	179.76 (19)
C5—N1—C6—C8	−139.7 (2)	N2—C9—O4—N3	−177.1 (2)
N1—C6—C8—N2	55.9 (3)	C9—O4—N3—C10	90.4 (3)
C6—C8—N2—C9	−141.7 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	1.010 (10)	2.037 (18)	2.938 (2)	147 (2)
N2—H2···O3^{ii}	1.016 (10)	2.038 (14)	3.022 (3)	162 (3)

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

The absolute stereochemistry of the title compound is based on the known configuration of Boc-*L*-Ala-OH (purchased from Neosystem, Strasbourg, France) since the homologation using the Arndt–Eistert

reaction is known to proceed without epimerization at the α carbon. The positions of H atoms attached to N atoms were located from a difference map and the N—H bond distance was restrained to 1.03 (1) Å (Taylor & Kennard, 1983). The H atoms connected to carbon were placed in the calculated positions and included in the refinement in the riding model approximation (C—H distances are in the range 0.96–0.98 Å). The isotropic H-atom displacement parameters were restricted to be 30% higher than the equivalent isotropic displacement parameters of the parent atom.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT*; data reduction: *HKL* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *maXus* (Mackay *et al.*, 1999) and *WebLab ViewerPro*3.5 (MSI, 1999).

We would like to thank the Service Commun de Diffraction X sur Monocristaux (Université Henri Poincaré, Nancy I) for providing access to crystallographic experimental facilities.

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