to affect the hydrogen-bond network between the host molecules.

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α-*N*-*tert*-Butyloxycarbonyl-L-amino-(*N*-methyl)succinimide (Boc-L-Asu-NMe)

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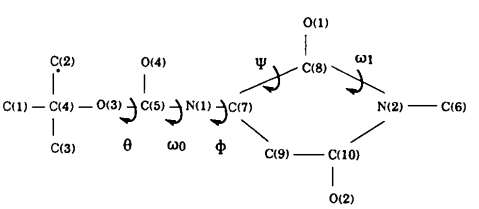
Abstract. $C_{10}H_{16}N_2O_4$, $M_r = 228{\cdot}25$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}768$ (1), $b = 13{\cdot}428$ (1), $c = 16{\cdot}173$ (2) Å, $V = 1252{\cdot}6$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}21$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418, $\mu = 0{\cdot}75$ mm^{-1}, $F(000) = 488$, room temperature, final $R = 0{\cdot}061$ for 1126 observed reflections. The title compound is a model for the aminosuccinyl residue (Asu) in a peptide sequence. In the solid state it adopts a conformation similar to that found in Asu peptides folded in a type II′ β-bend structure. The succinimide ring deviates considerably from planarity and assumes an envelope conformation. In the crystal, chains of N—H···O hydrogen-bonded molecules wind up around the screw axes parallel to **a**.

Introduction. The crystal structure determination of the title compound is part of a research program on the conformational properties of peptides containing the succinimide ring, hereafter referred to as aminosuccinyl peptides or Asu peptides (Capasso, Mattia, Mazzarella & Zagari, 1984*a*,*b*; Capasso, Mazzarella, Sica & Zagari, 1984, 1987; Mazzarella, Schon, Sica & Zagari, 1988). A growing body of experimental data indicates that these peptides strongly prefer a folded conformation of the type II′ β-bend, stabilized by a 4→1 intramolecular hydrogen bond, with the Asu residue in the second position of the bend (Venkatachalam, 1968). As the characterizing fragment of these peptides is the Asu moiety, we have synthesized and studied by X-ray analysis the fully protected Boc-L-Asu-NMe molecule.



Experimental. Synthesis according to a procedure described elsewhere (Capasso, Mazzarella, Sica & Zagari, 1988). Crystal from ethyl acetate, 0·72 × 0·13 × 0·13 mm, Enraf–Nonius CAD-4F diffractometer, Ni-filtered radiation, lattice parameters from 25 reflections ($16 \le \theta \le 25°$); data collection $\omega/2\theta$ scan as suggested by peak-shape analysis, two intensity monitoring reflections (3% variation); 1418 independent reflections with $\theta < 70°$, $0 \le h \le 7$, $0 \le k \le 16$, $0 \le l \le 19$, 1126 with $I > 2{\cdot}5\sigma(I)$, Lp correction, absorption ignored. Structure solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), anisotropic full matrix (on F), H atoms from geometrical considerations, isotropic with the same B_{eq} as the atoms to which they

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are bonded, not refined. Final $R = 0.061$, $wR = 0.081$, $w = 1/\sigma^2(F_o)$, $S = 3.19$; final $(\Delta/\sigma)_{max} = 0.01$, max. and min. heights in $\Delta\rho$ map 0.22 and -0.19 e Å^{-3}; scattering factors from *International Tables for X-ray Crystallography* (1974); Enraf–Nonius (1979) *SDP* software and PDP11/34 and VAX 750 computers of the Centro di Metodologie Chimico-Fisiche dell'Università di Napoli.

Discussion. A view of the molecule is shown in Fig. 1. Fractional coordinates and B_{eq} for non-hydrogen atoms are listed in Table 1.* Geometrical parameters are listed in Table 2. They compare well with those found for all Asu peptides studied (Capasso, Mazzarella *et al.*, 1987; Capasso, Mattia *et al.*, 1984*a*,*b*; Mazzarella *et al.*, 1988). This structure confirms also the tendency of the N(2)–C(8) and N(2)–C(10) bond lengths [1.373 (6) and 1.418 (7) Å respectively] to assume values greater than those usually found in peptides for the C–N bond.

As usual the Boc end group adopts the *trans–trans* conformation about the O(3)–C(5) and C(5)–N(1) bonds [$\theta = -176.2$ (9) and $\omega_0 = 174.1$ (8)°]. The peptide group also assumes a *trans* conformation [$\omega_1 = -173.5$ (9)°].

In the succinimide ring, no atom deviates more than 0.09 Å from the least-squares plane C(7)C(8)C(9)-C(10)N(2). The puckering amplitude (Cremer & Pople, 1975) of the ring is 0.14 Å and the phase is 182°, calculated for the sequence C(7)C(9)C(10)N(2)C(8). The deviation from planarity is in line with a pure envelope conformation with the C(7) atom 0.237 (5) Å out of the mean plane through the remaining four atoms of the ring. The torsion angle ψ, defined by the atoms N(1)–C(7)–C(8)–N(2), is -140.3 (8)° and deviates considerably from the value of -120° corresponding to a fully flat ring.

The title compound, apart from the terminal methyl group, constitutes the *N*-terminal fragment common to several Asu peptides whose conformational preferences have been extensively studied by the authors (Capasso, Mattia *et al.*, 1984*a*,*b*; Capasso, Mazzarella *et al.*, 1984, 1987; Mazzarella, Schon, Sica & Zagari, 1988). The conformation found in the solid state is characterized by a torsion angle φ about the bond N(1)–C(7) of 56.4 (7)°, as found in Asu peptides folded in a type II′ β-bend conformation and presents the urethane plane almost perpendicular (86°) to the succinimide ring (Fig. 2) and the carbonyl O atom O(4) placed only 2.652 (4) Å from the succinimide plane.

The solid-state conformation of Boc-L-Asu-NMe provides a basis to explain the circular dichroism spectra of this compound in solution (Capasso *et al.*, 1988). Indeed the positive Cotton effect at 260 nm, present in the spectra recorded in non-proton-acceptor solvents, can be correlated to the dominance of a conformation similar to that found in the solid state and can be ascribed to the asymmetric perturbation of the π

*Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51282 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

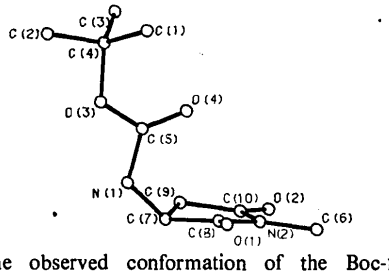

Fig. 1. The observed conformation of the Boc-L-Asu-NMe molecule.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$B_{eq} = \frac{1}{3}\sum_i B_{ii}$.

	x	y	z	B_{eq}
O(1)	0.5355 (8)	0.0933 (3)	0.5457 (2)	5.1 (1)
O(2)	0.0511 (8)	−0.1421 (3)	0.4419 (3)	6.5 (1)
O(3)	0.4089 (7)	0.2804 (2)	0.3329 (2)	4.3 (1)
O(4)	0.4678 (7)	0.1154 (2)	0.3511 (2)	4.7 (1)
N(1)	0.2315 (8)	0.2041 (3)	0.4362 (3)	4.0 (1)
N(2)	0.3206 (8)	−0.0406 (3)	0.5056 (3)	3.8 (1)
C(1)	0.7978 (11)	0.2652 (6)	0.2750 (4)	7.0 (2)
C(2)	0.5238 (19)	0.4021 (5)	0.2434 (5)	9.1 (2)
C(3)	0.4472 (14)	0.2263 (6)	0.1900 (4)	7.4 (2)
C(4)	0.5473 (11)	0.2902 (4)	0.2579 (3)	4.8 (1)
C(5)	0.3817 (9)	0.1940 (3)	0.3698 (3)	3.6 (1)
C(6)	0.4738 (12)	−0.1167 (4)	0.5387 (4)	5.8 (1)
C(7)	0.1577 (9)	0.1163 (3)	0.4816 (3)	3.7 (1)
C(8)	0.3615 (9)	0.0597 (3)	0.5139 (3)	3.6 (1)
C(9)	0.0219 (10)	0.0385 (4)	0.4326 (4)	4.9 (1)
C(10)	0.1196 (10)	−0.0598 (4)	0.4576 (4)	4.4 (1)

Table 2. *Bond lengths* (Å) *and angles* (°) *with their e.s.d.'s in parentheses*

O(1)–C(8)	1.214 (7)	O(2)–C(10)	1.201 (6)
O(3)–C(4)	1.458 (7)	O(3)–C(5)	1.313 (6)
O(4)–C(5)	1.205 (6)	N(1)–C(5)	1.387 (7)
N(1)–C(7)	1.452 (6)	N(2)–C(6)	1.453 (7)
N(2)–C(8)	1.373 (6)	N(2)–C(10)	1.418 (7)
C(1)–C(4)	1.510 (9)	C(2)–C(4)	1.527 (9)
C(3)–C(4)	1.507 (9)	C(7)–C(8)	1.494 (7)
C(7)–C(9)	1.528 (7)	C(9)–C(10)	1.491 (8)

C(4)–O(3)–C(5)	121.6 (7)	C(5)–N(1)–C(7)	119.8 (7)
C(6)–N(2)–C(8)	123.3 (7)	C(6)–N(2)–C(10)	124.8 (8)
C(8)–N(2)–C(10)	111.8 (7)	O(3)–C(4)–C(1)	110.5 (8)
O(3)–C(4)–C(2)	99.7 (9)	O(3)–C(4)–C(3)	110.2 (9)
C(1)–C(4)–C(2)	109.4 (10)	C(1)–C(4)–C(3)	111.9 (9)
C(2)–C(4)–C(3)	114.5 (10)	O(3)–C(5)–O(4)	127.6 (8)
O(3)–C(5)–N(1)	109.9 (7)	O(4)–C(5)–N(1)	122.4 (8)
N(1)–C(7)–C(8)	111.0 (7)	N(1)–C(7)–C(9)	116.2 (7)
C(8)–C(7)–C(9)	103.7 (7)	O(1)–C(8)–N(2)	123.2 (8)
O(1)–C(8)–C(7)	127.5 (8)	N(2)–C(8)–C(7)	109.3 (7)
C(7)–C(9)–C(10)	105.7 (8)	O(2)–C(10)–N(2)	123.5 (9)
O(2)–C(10)–C(9)	129.2 (9)	N(2)–C(10)–C(9)	107.3 (8)

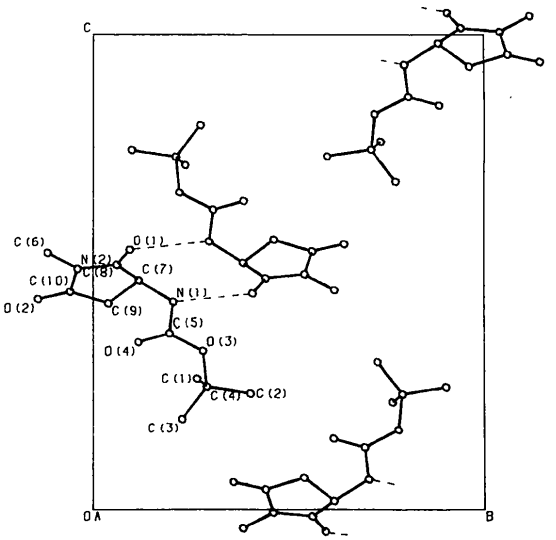

Fig. 2. Packing diagram projected on the *bc* plane showing the intermolecular hydrogen bond (dotted lines).

system of the succinimide chromophore induced by the carbonyl oxygen O(4).

The nitrogen N(1) is the only hydrogen donor atom present in this molecule and it is hydrogen bonded to O(1) of a screw related molecule with an N(1)···O(1′) separation of 2·961 (3) Å as shown in the crystal packing reported in Fig. 2. The presence of only one intermolecular hydrogen bond and the absence of other short intermolecular contacts correlates well with the rather large temperature factors and the relatively low value of the crystal density compared with those of related structures.

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Structure of Cytidinium Dihydrogenphosphate

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Abstract. $C_9H_{14}N_3O_5^+.H_2PO_4^-$, $M_r = 341{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}653$ (1), $b = 7{\cdot}0256$ (9), $c = 19{\cdot}873$ (2) Å, $V = 1347{\cdot}8$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}68$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}04$ cm^{-1}, $F(000) = 712$, $T = 291$ K, $R = 0{\cdot}030$ for 1198 observed reflexions. The ribose conformation is characterized by $P = 163{\cdot}9$ (8)° (^2E) and $\tau_m = 40{\cdot}0$ (6)°. The glycosidic torsion angle χ is in the *anti* region but has a very low value [34·4 (3)°]. The side chain has the preferred *gauche*$^+$ conformation [$\gamma = 46{\cdot}6$ (4)°]. The structure contains infinite ribose···phosphate chains in which the dihydrogenphosphate anions are hydrogen-bonded (*via* their POH donors) to O(3′) and O(5′) of neighboring nucleosides. The ribose···phosphate backbone is further reinforced by a direct O(5′)H···O(2′) bond between adjacent sugar residues. There are also analogous nucleobase···phosphate hydrogen-bonded chains in which the cytosinium cation is a threefold donor [N(4)H$_2$ and the N(3)H protonation site] and the phosphate anion is the acceptor. The hydrogen-bonded columns (along **b**) of anions and cations form pleated sheets which are further connected by an O(3′)H···OP hydrogen bond.

Introduction. The present work continues a series of investigations into the factors influencing dimensions

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