References

BENEDETTI, E., GOODMAN, M., MARSH, R. E., RAPOPORT, H. & MUSICH, J. A. (1975). *Cryst. Struct. Commun.* **4**, 641–645.

BLÁHA, K., BUDĚŠÍNSKÝ, M., KOBLICOVÁ, Z., MALOŇ, P., TICHÝ, M., BAKER, J. R., HOSSAIN, M. B. & VAN DER HELM, D. (1982). *Collect. Czech. Chem. Commun.* **47**, 1000–1019.

BLÁHA, K., FARAG, A. M., VAN DER HELM, D., HOSSAIN, M. B., BUDĚŠÍNSKÝ, M., MALOŇ, P., SMOLÍKOVÁ, J. & TICHÝ, M. (1984). *Collect. Czech. Chem. Commun.* **48**, 712–742.

BLÁHA, K. & MALOŇ, P. (1980). *Acta Univ. Palacki, Olomuc. Fac. Med.* **93**, 81–86.

BLÁHA, K., MALOŇ, P., TICHÝ, M., FRIČ, I., USHA, R., RAMAKUMAR, S. & VENKATESAN, K. (1978). *Collect. Czech. Chem. Commun.* **43**, 3241–3246.

CERRINI, S., FEDELI, W., LUCENTE, G., MAZZA, F., PINNEN, F. & ZANOTTI, G. (1984). *Int. J. Pept. Protein Res.* **23**, 223.

COTRAIT, M. & LEROY, F. (1979). *Cryst. Struct. Commun.* **8**, 819–822.

FRIČ, I. & BLÁHA, K. (1987). *Collect. Czech. Chem. Commun.* Submitted.

GDANIEC, M. (1981). *Pol. J. Chem.* **55**, 1795.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

KARLE, I. L. (1981). *The Peptides*, Vol. 4, ch. 1. New York: Academic Press.

LANGER, V. (1973). *INTER*. UMCH-111. Institute of Macromolecular Chemistry, Praha.

LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A**30**, 580–584.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MAZZA, F., LUCENTE, G., PINNEN, F. & ZANOTTI, G. (1984). *Acta Cryst.* C**40**, 1974–1976.

NARDELLI, M. (1983). *PARST83. A System of Computer Routines for Calculating Molecular Parameters from the Results of Crystal Structure Analysis.* Univ. of Parma, Italy.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SJÖLIN, L. & WLODAWER, A. (1981). *Acta Cryst.* A**37**, 594–604.

SKLENÁŘ, I. & JEČNÝ, J. (1979). *Acta Cryst.* B**35**, 513–515.

SYMERSKÝ, J., BLÁHA, K. & LANGER, V. (1987). *Acta Cryst.* C**43**, 490–493.

TICHÝ, M., FARAG, A. M., MALOŇ, P. & BLÁHA, K. (1984). *Collect. Czech. Chem. Commun.* **49**, 834–839.

WARSHEL, A., LEVITT, M. & LIFSON, S. (1970). *J. Mol. Spectrosc.* **33**, 84–89.

WINKLER, F. K. & DUNITZ, J. D. (1971). *J. Mol. Biol.* **59**, 169–182.

Acta Cryst. (1987). C**43**, 1607–1610

tert-Butyloxycarbonyl-L-aminosuccinyl-glycyl-L-alanine Methyl Ester (Boc-L-Asu-Gly-L-Ala-OMe)

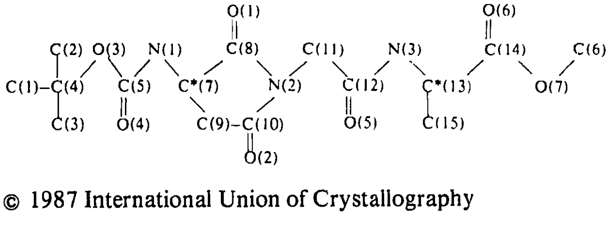
BY SANTE CAPASSO, LELIO MAZZARELLA, FILOMENA SICA AND ADRIANA ZAGARI

Dipartimento di Chimica, Università di Napoli, Via Mezzocannone 4, 80134 *Napoli, Italy*

(*Received* 17 *November* 1986; *accepted* 25 *March* 1987)

Abstract. $C_{15}H_{23}N_3O_7$, $M_r = 357{\cdot}36$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}251\,(5)$, $b = 11{\cdot}179\,(1)$, $c = 35{\cdot}113\,(4)$ Å, $V = 3631\,(3)$ Å^3, $Z = 8$, $D_x = 1{\cdot}307$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}84$ mm^{-1}, $F(000) = 1520$, room temperature, $R = 0{\cdot}045$ for 3330 observed reflections. In agreement with our previous potential-energy calculations [Capasso, Mattia, Mazzarella & Zagari (1984) *Int. J. Pept. Protein Res.* **24**, 85–95], both molecules in the asymmetric unit adopt a type II′ β-turn conformation, with an intramolecular 4→1 hydrogen bond. In the crystal, chains of hydrogen-bonded molecules wind up around the screw axes parallel to **a**.

Introduction. In protein chemistry, the role played by the succinimide ring in the non-enzymatic deamidation of the asparaginyl side chain has been examined by an increasing number of studies (Graf, Bajusz, Patthy, Barat & Cseh, 1971; Aswad, 1984). Moreover, recent studies (Clarke, 1984) have indicated that the succinimide ring is an intermediate in the repair or selective degradation of deamidated proteins. The biological relevance of the succinimide ring has prompted us to study systematically the conformational parameters of model peptides containing the cyclic imide structure, in solution and the solid state (Capasso, Mattia, Mazzarella & Zagari, 1984*a*,*b*; Capasso, Mazzarella, Sica & Zagari, 1984). In this paper we describe the crystal and molecular structure of the fully blocked tripeptide Boc-L-Asu-Gly-L-Ala-OMe.



0108-2701/87/081607-04$01.50

Experimental. Synthesis similar to the procedure previously reported (Capasso, Mazzarella, Sica & Zagari, 1984). Crystals were obtained by evaporation of an ethyl acetate solution. Crystal 0·7 × 0·4 × 0·2 mm. Enraf–Nonius CAD-4 diffractometer. Systematic absences $h00$ for h odd, $0k0$ for k odd, $00l$ for l odd. Cell constants from a least-squares analysis of 25 reflections with $16 \leq \theta \leq 25°$ measured on the diffractometer. Intensity data collected in an ω–2θ scan mode, as suggested by peak-shape analysis. 4218 independent reflections, $2\theta \leq 150°$, Lorentz–polarization correction, no absorption correction. $h = 0$–11, $k = 0$–13, $l = 0$–43. Intensity of two standard reflections recorded periodically to monitor crystal and equipment stability. Structure determined using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). E map revealed a 30-atom fragment; remaining non-H atoms located from a difference Fourier synthesis after a few cycles of least-squares refinement. Anisotropic full-matrix least-squares refinement on F of all 50 non-H atoms led to $R = 0·078$. At this stage, H atoms, fixed by geometrical considerations and a difference Fourier map, were included in the structure factor calculations, but their positional and temperature factors (assumed to be the same as the B_{eq} of the atoms to which they are bonded) were not refined. Further least-squares refinement yielded a residual $R = 0·045$ and $wR = 0·049$, $w = 1/\sigma^2(F_o)$, for 451 refined parameters and 3330 observed data $[I > 2·5\sigma(I)]$. Final value of the standard deviation of an observation of unit weight was 1·99, final $(\Delta/\sigma)_{max} = 0·02$. Final difference Fourier map contained no peak higher than 0·16 e Å^{-3}.

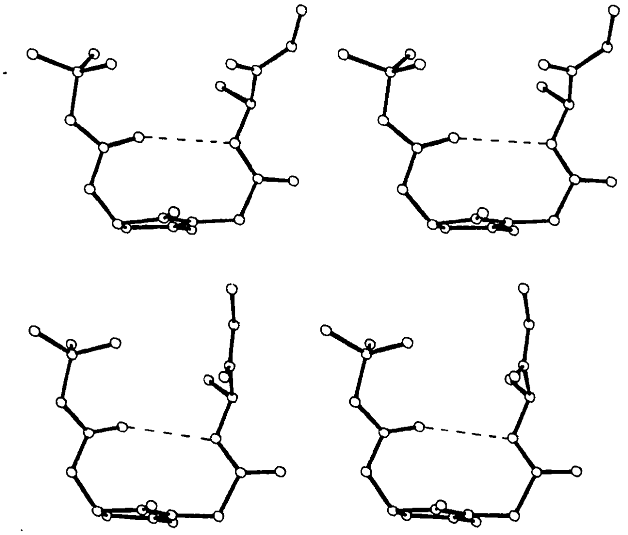


Fig. 1. Stereo diagram illustrating the observed conformations for the primed (bottom) and unprimed (top) molecules of Boc-L-Asu-Gly-L-Ala-OMe. The molecules are seen approximately in the same orientation. The 4→1 intramolecular hydrogen bond is shown by dotted lines.

Table 1. *Fractional coordinates and thermal parameters* (Å^2) *for non*-H *atoms with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*
O(1)	1·3229 (3)	0·2368 (3)	0·9047 (1)	6·11 (7)
O(2)	1·2126 (5)	0·0060 (3)	0·8028 (1)	8·7 (1)
O(3)	0·8836 (3)	0·1788 (3)	0·9430 (1)	6·09 (6)
O(4)	0·9920 (3)	0·2126 (2)	0·8859 (1)	4·90 (5)
O(5)	1·2949 (3)	0·3232 (3)	0·7668 (1)	6·86 (7)
O(6)	0·9853 (3)	0·5189 (2)	0·8458 (1)	4·71 (5)
O(7)	0·9824 (3)	0·5984 (2)	0·7877 (1)	5·38 (6)
N(1)	1·0795 (3)	0·0780 (3)	0·9280 (1)	4·71 (7)
N(2)	1·2840 (3)	0·1367 (3)	0·8488 (1)	4·29 (6)
N(3)	1·1236 (3)	0·3188 (3)	0·8126 (1)	4·35 (6)
C(1)	0·6900 (5)	0·2754 (5)	0·9065 (1)	7·6 (1)
C(2)	0·6961 (5)	0·2673 (4)	0·9775 (1)	7·7 (1)
C(3)	0·8683 (6)	0·3956 (4)	0·9420 (1)	7·3 (1)
C(4)	0·7819 (4)	0·2814 (3)	0·9414 (1)	4·64 (8)
C(5)	0·9855 (4)	0·1624 (3)	0·9163 (1)	4·55 (8)
C(6)	0·9541 (6)	0·7159 (4)	0·8041 (1)	7·2 (1)
C(7)	1·1973 (4)	0·0437 (3)	0·9041 (1)	3·99 (7)
C(8)	1·2756 (4)	0·1530 (3)	0·8879 (1)	3·99 (7)
C(9)	1·1552 (5)	−0·0312 (3)	0·8693 (1)	4·99 (9)
C(10)	1·2180 (5)	0·0340 (3)	0·8358 (1)	4·92 (9)
C(11)	1·3552 (4)	0·2202 (4)	0·8231 (1)	5·9 (1)
C(12)	1·2522 (4)	0·2917 (3)	0·7983 (1)	4·85 (8)
C(13)	1·0216 (4)	0·3913 (3)	0·7918 (1)	4·15 (7)
C(14)	0·9963 (4)	0·5087 (3)	0·8121 (1)	3·63 (6)
C(15)	0·8769 (5)	0·3274 (4)	0·7873 (1)	5·9 (1)
O(1′)	0·6970 (3)	−0·2717 (2)	1·0663 (1)	5·26 (6)
O(2′)	1·0302 (3)	−0·0602 (3)	1·0000 (1)	6·07 (7)
O(3′)	0·8425 (3)	−0·0841 (2)	1·1665 (1)	4·50 (5)
O(4′)	0·9704 (3)	−0·1317 (2)	1·1132 (1)	4·79 (6)
O(5′)	1·1070 (3)	−0·4389 (2)	1·0119 (1)	4·89 (6)
O(6′)	1·0457 (3)	−0·4762 (3)	1·1202 (1)	6·40 (7)
O(7′)	1·2735 (3)	−0·4561 (3)	1·1402 (1)	5·55 (6)
N(1′)	0·7500 (3)	−0·0464 (3)	1·1099 (1)	4·11 (6)
N(2′)	0·8720 (3)	−0·1869 (2)	1·0291 (1)	3·29 (5)
N(3′)	1·0912 (3)	−0·3156 (3)	1·0623 (1)	4·71 (6)
C(1′)	1·0831 (5)	−0·0509 (5)	1·1919 (1)	7·0 (1)
C(2′)	0·8754 (5)	−0·1142 (5)	1·2318 (1)	6·8 (1)
C(3′)	0·9857 (6)	−0·2583 (4)	1·1869 (1)	7·4 (1)
C(4′)	0·9516 (4)	−0·1296 (3)	1·1941 (1)	4·35 (8)
C(5′)	0·8650 (4)	−0·0918 (3)	1·1288 (1)	3·81 (7)
C(6′)	1·2416 (6)	−0·5267 (4)	1·1739 (1)	7·1 (1)
C(7′)	0·7426 (4)	−0·0561 (3)	1·0687 (1)	3·70 (7)
C(8′)	0·7635 (3)	−0·1843 (3)	1·0558 (1)	3·48 (6)
C(9′)	0·8569 (4)	0·0144 (3)	1·0466 (1)	4·33 (8)
C(10′)	0·9319 (4)	−0·0756 (3)	1·0223 (1)	3·85 (7)
C(11′)	0·9204 (4)	−0·2955 (3)	1·0101 (1)	3·97 (7)
C(12′)	1·0474 (4)	−0·3566 (3)	1·0289 (1)	3·59 (7)
C(13′)	1·2152 (4)	−0·3616 (3)	1·0824 (1)	4·37 (8)
C(14′)	1·1647 (4)	−0·4377 (3)	1·1160 (1)	4·28 (8)
C(15′)	1·3146 (4)	−0·2593 (4)	1·0944 (1)	5·09 (9)

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

Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).* All computations performed using the Enraf–Nonius (1979) *SDP* software and PDP11/34 computer of the Centro di Metodologie Chimico-Fisiche dell'Università di Napoli.

Discussion. In Fig. 1 is shown a view of the two molecules in the asymmetric unit, seen approximately in the same orientation. The final positional parameters together with their e.s.d.'s are listed in Table 1. The differences between the bond lengths and angles (Table

---

* 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 43912 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°)

	Un-primed	Primed		Un-primed	Primed
O(1)–C(8)	1·192 (4)	1·213 (4)	N(2)–C(10)	1·377 (5)	1·383 (4)
O(2)–C(10)	1·203 (4)	1·212 (4)	N(2)–C(11)	1·455 (5)	1·456 (4)
O(3)–C(4)	1·485 (5)	1·487 (4)	N(3)–C(12)	1·327 (5)	1·321 (4)
O(3)–C(5)	1·341 (4)	1·344 (4)	N(3)–C(13)	1·443 (5)	1·443 (5)
O(4)–C(5)	1·210 (4)	1·204 (4)	C(1)–C(4)	1·491 (6)	1·503 (6)
O(5)–C(12)	1·227 (4)	1·227 (4)	C(2)–C(4)	1·505 (6)	1·511 (5)
O(6)–C(14)	1·190 (4)	1·191 (5)	C(3)–C(4)	1·506 (6)	1·494 (6)
O(7)–C(6)	1·457 (5)	1·451 (5)	C(7)–C(8)	1·531 (5)	1·515 (5)
O(7)–C(14)	1·325 (4)	1·334 (4)	C(7)–C(9)	1·534 (5)	1·531 (5)
N(1)–C(5)	1·346 (5)	1·352 (4)	C(9)–C(10)	1·499 (5)	1·492 (5)
N(1)–C(7)	1·426 (5)	1·452 (4)	C(11)–C(12)	1·517 (6)	1·510 (5)
N(2)–C(8)	1·388 (4)	1·374 (4)	C(13)–C(14)	1·512 (5)	1·527 (5)
			C(13)–C(15)	1·525 (6)	1·527 (5)
C(4)–O(3)–C(5)	121·7 (5)	121·0 (4)	C(8)–C(7)–C(9)	104·9 (5)	104·3 (5)
C(6)–O(7)–C(14)	116·4 (5)	116·7 (5)	O(1)–C(8)–N(2)	125·1 (6)	124·1 (5)
C(5)–N(1)–C(7)	120·2 (5)	119·8 (5)	O(1)–C(8)–C(7)	128·0 (6)	127·3 (5)
C(8)–N(2)–C(10)	114·2 (5)	113·1 (4)	N(2)–C(8)–C(7)	106·9 (5)	108·5 (4)
C(8)–N(2)–C(11)	123·7 (5)	123·8 (5)	C(7)–C(9)–C(10)	105·1 (5)	105·3 (5)
C(10)–N(2)–C(11)	122·1 (5)	123·2 (5)	O(2)–C(10)–N(2)	123·6 (6)	122·7 (5)
C(12)–N(3)–C(13)	121·5 (5)	123·6 (5)	O(2)–C(10)–C(9)	127·8 (6)	128·5 (6)
O(3)–C(4)–C(1)	110·9 (5)	108·4 (5)	N(2)–C(10)–C(9)	108·6 (5)	108·8 (5)
O(3)–C(4)–C(2)	102·8 (5)	102·4 (5)	N(2)–C(11)–C(12)	114·1 (6)	114·6 (5)
O(3)–C(4)–C(3)	108·5 (5)	111·3 (5)	O(5)–C(12)–N(3)	124·4 (6)	123·7 (5)
C(1)–C(4)–C(2)	112·7 (6)	110·8 (6)	O(5)–C(12)–C(11)	117·8 (6)	118·4 (5)
C(2)–C(4)–C(3)	110·9 (6)	110·9 (6)	N(3)–C(12)–C(11)	117·8 (6)	117·9 (5)
C(1)–C(4)–C(3)	110·7 (6)	112·6 (6)	N(3)–C(13)–C(14)	110·5 (5)	109·5 (5)
O(3)–C(5)–O(4)	126·0 (6)	126·6 (5)	N(3)–C(13)–C(15)	111·4 (5)	110·3 (5)
O(3)–C(5)–N(1)	109·8 (5)	109·7 (5)	C(14)–C(13)–C(15)	108·7 (5)	112·8 (5)
O(4)–C(5)–N(1)	124·2 (6)	123·6 (5)	O(6)–C(14)–O(7)	124·1 (5)	124·3 (6)
N(1)–C(7)–C(8)	115·5 (5)	111·2 (5)	O(6)–C(14)–C(13)	124·4 (5)	125·4 (6)
N(1)–C(7)–C(9)	114·9 (5)	115·8 (5)	O(7)–C(14)–C(13)	111·5 (5)	110·3 (5)

Table 3. *Torsion angles* (°)

		Unprimed unit	Primed unit
φ_1	C(5)–N(1)–C(7)–C(8)	46·7 (5)	52·8 (5)
ψ_1	N(1)–C(7)–C(8)–N(2)	−128·3 (5)	−127·0 (5)
φ_2	C(8)–N(2)–C(11)–C(12)	−108·1 (6)	−93·7 (5)
ψ_2	N(2)–C(11)–C(12)–N(3)	32·0 (6)	7·6 (5)
φ_3	C(12)–N(3)–C(13)–C(14)	−114·7 (6)	−105·7 (5)
ψ_3	N(3)–C(13)–C(14)–O(7)	142·1 (5)	−164·5 (5)

2) of the two molecules are within the e.s.d.'s. On average, their values compare well with those of related compounds (Capasso, Mattia, Mazzarella & Zagari, 1984*a,b*; Capasso, Mazzarella, Sica & Zagari, 1984) and, with the exception of the bond parameters for the succinimide moiety, with those averaged for linear peptides (Benedetti, 1977). In the ring, the lengths of the C(8)—N(2) and C(10)—N(2) distances, as expected, are the same within experimental error in both the unprimed and the primed molecules. It must be noted that, for all the Asu-containing peptides studied by us, these bond lengths are consistently longer (~0·05 Å) than the C—N bond in linear peptides. Their values, together with a smaller but definite shortening of the C—O bonds in the succinimide moiety [on average 1·205 (5) Å for the C(8)—O(1) and C(10)—O(2) distances], indicate a lower π-bond order for the C—N bonds within the ring as compared to a linear peptide.

The relevant dihedral angles of the tripeptide are shown in Table 3. Despite the differences in the packing and hydrogen-bonding network, the two molecules in the asymmetric unit possess a remarkably similar conformation. The largest differences are observed for the values of the ψ_3 dihedral angle, which corresponds to different orientations of the terminal ester moiety in the two molecules. This difference can be ascribed to the intermolecular hydrogen bond which links the O(6) atom of the ester group in the unprimed molecule to the urethane N′H group of the primed unit.

The Boc end group adopts the *trans–trans* conformation about the O(3)—C(5) and C(5)—N(1) bonds [$\theta_1 = 168{\cdot}3$ (6), $\omega_0 = 179{\cdot}3$ (6)° and $\theta'_1 =$ 179·0 (5), $\omega'_0 = -173{\cdot}7$ (6)°], as usually found when N(1) is a secondary N atom. Also, the peptide groups have a *trans* conformation [$\omega_1 = -178{\cdot}1$ (7), $\omega_2 =$ 177·0 (7), $\omega'_1 = -179{\cdot}4$ (6), $\omega'_2 = -176{\cdot}8$ (7)°].

The whole molecule can be divided into four substantially planar moieties: the urethane, the peptide, the ester and the succinimide groups. The first two groups are roughly coplanar so that the urethane carbonyl C=O bond points towards the H—N bond of the peptide group.

In the succinimide rings, no atom deviates more than 0·021 Å from the least-squares plane C(7)C(8)C(9)-C(10)N(2) and no atom deviates more than 0·009 Å from the least-squares plane C(7′)C(8′)C(9′)C(10′)-N(2′). The deformation of the two rings from planarity, though very small, is consistent with a pure envelope conformation, with the C(7) atom 0·055 Å and C(7′) atom 0·024 Å out of the plane of the four remaining atoms. These figures indicate that, in the present

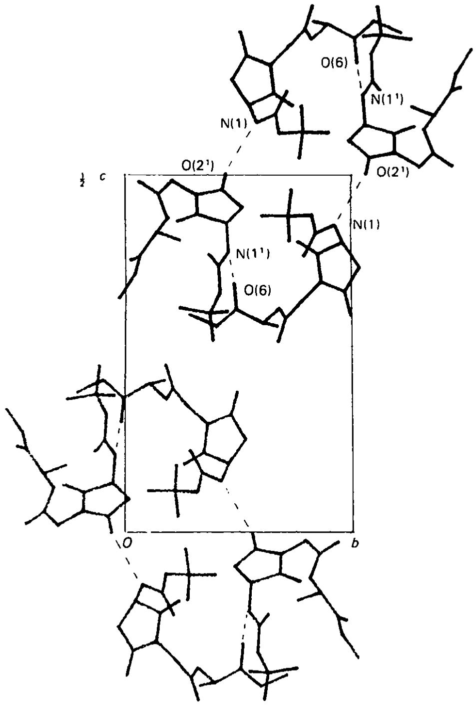


Fig. 2. Packing diagram projected on the *bc* plane showing the intermolecular hydrogen bonds (dotted lines).

structure, the five-membered ring is more planar than that found in the related structures of Asu-containing peptides (Capasso, Mattia, Mazzarella & Zagari 1984*a*,*b*; Capasso, Mazzarella, Sica & Zagari, 1984), where, at least in one case, the maximum deviation from planarity is as high as 0·131 Å. The puckering of the ring, although generally small, significantly affects the dihedral angle ψ_1 about the C^α—C bond, whose value, in all the structures quoted, spans a range of almost 25°.

Inspection of the values of dihedral angles φ_1, ψ_1, φ_2 and ψ_2 in Table 3 clearly indicates that the overall conformation of the two molecules is of the type II′ β-bend, with the L-Asu residue in the second position and the Gly residue in the third position of the turn. This conformation, normally allowed for linear peptides with a D residue in the second position, is stabilized in aminosuccinyl peptides by the five-membered ring, which requires the value of ψ_1 to be close to $-120°$ and was predicted by us, on theoretical grounds, to be very stable for an L-Asu-Gly sequence. In addition, our solution studies on Boc-protected L-Asu-containing peptides show that, in non-H-acceptor solvents, this conformation is indeed the most stable one (manuscript in preparation). The II′ β-bend conformation, also adopted by Boc-L-Asu-L-Ala-Gly-OMe in the solid state (Capasso, Mazzarella, Sica & Zagari, 1984), is stabilized by an intramolecular 4→1 hydrogen bond between the N(3)H group of the L-Ala residue (position 4) and the carbonyl O(4) atom of the Boc group (position 1) [N(3)···O(4) 3·083 (3); N(3′)···O(4′) 2·945 (3) Å].

The folded conformation leaves only one of the two potential donor NH groups per molecule free to form intermolecular hydrogen bonds. The urethane NH group is hydrogen bonded to the carbonyl O(2′) atom, whereas the N′H group is hydrogen bonded to the ester O(6) atom of a screw-related unprimed unit [N(1)···O(2′) 2·998 (3); N(1′)···O(6) 2·918 (3) Å]. Thus, primed and unprimed units form chains of hydrogen-bonded molecules, which wind around the screw axes parallel to **a** (Fig. 2).

References

ASWAD, D. (1984). *J. Biol. Chem.* **259**, 10714–10721.

BENEDETTI, E., (1977). *Peptides: Proceedings of the Fifth American Peptide Symposium*, edited by M. GOODMAN & J. MEINHOFER, pp. 257–273. New York: John Wiley.

CAPASSO, S., MATTIA, C. A., MAZZARELLA, L. & ZAGARI, A. (1984*a*). *Int. J. Pept. Protein Res.* **23**, 248–255.

CAPASSO, S., MATTIA, C. A., MAZZARELLA, L. & ZAGARI, A. (1984*b*). *Int. J. Pept. Protein Res.* **24**, 85–95.

CAPASSO, S., MAZZARELLA, L., SICA, F. & ZAGARI, A. (1984). *Int. J. Pept. Protein Res.* **24**, 588–596.

CLARKE, S. (1984). *Annu. Rev. Biochem.* **54**, 479–506.

Enraf–Nonius (1979). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

GRAF, L., BAJUSZ, S., PATTHY, A., BARAT, E. & CSEH, G. (1971). *Acta Biochim. Biophys. Acad. Sci. Hung.* **6**, 415–418.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht, The Netherlands.)

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Acta Cryst. (1987). C**43**, 1610–1613

Low-Temperature Structure of *N*-Methylphthalazinium Bis(7,7,8,8-tetracyano-*p*-quinodimethanide)

BY GAO YAN AND PHILIP COPPENS

Department of Chemistry, State University of New York at Buffalo, Buffalo, NY 14214, *USA*

(*Received* 11 *June* 1986; *accepted* 6 *May* 1987)

Abstract. $(\mathrm{C_9H_9N_2})^{+}.(\mathrm{C_{12}H_4N_4})_2^{-}$, $M_r = 553·6$, monoclinic, $C2/c$, $a = 28·574\ (12)$, $b = 3·770\ (2)$, $c = 26·183\ (10)$ Å, $\beta = 114·29\ (3)°$, $V = 2570·85$ Å^3, $Z = 4$, $D_m = 1·38$ (measured by flotation in a mixture of chlorobenzene and bromobenzene), $D_x = 1·43$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0·7107$ Å, $\mu = 0·85$ cm^{-1}, $F(000) = 1140$, $T = 130$ K, $R = 0·0343$ for the 1097 reflections used in the rigid-body refinement. The structure consists of separate columns of TCNQ (7,7,8,8-tetracyano-*p*-quinodimethanide) and MPht (*N*-methylphthalazinium), oriented parallel to the *b* axis, each MPht being surrounded by six TCNQ molecules. At 130 K, the average spacings between the TCNQ and MPht molecules in the columns are 3·178 (2) and 3·449 (2) Å, respectively. The average net charge on the TCNQ molecules is estimated to be −0·38 to −0·44 units. The MPht molecules are disordered with two possible orientations of the methyl substituents.

Introduction. Structures of TCNQ salts which are quasi-one-dimensional conductors have been a subject of considerable interest in recent years. The structures

0108-2701/87/081610-04$01.50