Table 4. *The deviations* (Å) *of the atoms of the least-squares plane in the thiazolidyne part of the molecule*

Plane: 5·339 x + 12·429 y − 6·972 z = 3·592 (x, y, z are the fractional coordinates along **a**, **b**, **c**).

O(1)	−0·011	S(1)	0·011
C(1)	−0·007	N(1)	0·021
C(2)	0·001	C(4)	−0·038
C(3)	0·014	C(5)	0·010

All e.s.d.'s are 0·002 Å.

that in compound *A* the replacement of the O atom by a dicyanomethylidene group at C5 is possible due to the electron-withdrawing effect of the conjugated system described above.

It can be concluded, as was already suggested by Kucsman, Kapovits, Párkányi, Argay & Kálmán (1984), that the S—O interaction may, by governing the conformation, have a decisive influence on chemical behaviour.

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Structure of (3*S*)-3-*tert*-Butyloxycarbonylamino-2-piperidone

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Abstract. $C_{10}H_{18}N_2O_3$, $M_r = 214·27$, monoclinic, $P2_1$, $a = 15·515$ (2), $b = 6·730$ (1), $c = 12·541$ (2) Å, $\beta = 113·6$ (2)°, $V = 1200·0$ Å^3, $Z = 4$, $D_x = 1·186$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·54$ cm^{-1}, $F(000) = 464$, $T = 295$ K. The final R value for 1611 observed (3139 unique) reflections is 0·055. In both the independent molecules *A* and *B* of the asymmetric unit of the title compound, the conformation of the urethane moiety is *trans*. The lactam group of molecule *A* is non-planar, the C(9)—N(2)—C(10)—C(6) torsion angle being 12·4 (14)°. One main difference between molecules *A* and *B* is in the value of the φ[C(5)—N(1)—C(6)—C(10)] torsion angle [52·3 (11)° for molecule *A* while −86·5 (10)° for molecule *B*] as a consequence of a rotation of the ring relative to the *tert*-butyloxycarbonylamino substituent. A second major difference is the δ-lactam ring conformation which is approximate half-chair for molecule *A* while boat for molecule *B*.

Introduction. Replacement or modification of the peptide backbone function can lead to enzymatically resistant biologically active analogs. Among the factors contributing to altered chemical and biochemical parameters are changes in electronic properties, differences in solubility characteristics, resistance to proteolytic processes, and, perhaps most important, conformational restrictions and changes that can modify receptor recognition (Spatola, 1983).

In particular, we have recently undertaken the synthesis of a series of conformationally constrained analogs of the neuroactive tripeptide H-L-Pro-L-Leu-Gly-NH_2 (Johnson, Yu, Taraporewala, Mishra & Rajakumar, 1985; Yu, Rajakumar, Srivastava, Mishra & Johnson, 1988) in which the γ- and δ-lactam residues developed by Freidinger, Perlow & Veber (1982) have replaced either the leucyl or glycinamide residues. These compounds were synthesized in an attempt to determine whether the *trans* amide bond and the β-bend

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å²) *for the non*-H *atoms of compound* (1) (*with e.s.d.'s in parentheses*)

	x	y	z	U_{eq}*
O(1)A	0·6886 (3)	0·0000 (8)	0·9096 (3)	0·061 (2)
O(2)A	0·8488 (3)	−0·0005 (8)	1·0037 (3)	0·056 (2)
O(3)A	0·8668 (3)	−0·3507 (7)	0·8461 (4)	0·054 (2)
N(1)A	0·7730 (3)	0·0087 (9)	0·8059 (4)	0·046 (2)
N(2)A	1·0030 (3)	−0·2010 (9)	0·8685 (4)	0·050 (2)
C(1)A	0·7046 (7)	−0·2290 (16)	1·0651 (8)	0·085 (4)
C(2)A	0·5618 (5)	−0·0232 (19)	0·9629 (10)	0·086 (5)
C(3)A	0·7091 (8)	0·1355 (19)	1·1007 (10)	0·111 (5)
C(4)A	0·6684 (5)	−0·0292 (13)	1·0131 (6)	0·058 (3)
C(5)A	0·7772 (4)	0·0011 (11)	0·9148 (5)	0·048 (3)
C(6)A	0·8561 (4)	−0·0125 (12)	0·7804 (5)	0·041 (3)
C(7)A	0·9168 (5)	0·1763 (11)	0·8093 (6)	0·051 (3)
C(8)A	1·0070 (5)	0·1314 (12)	0·7909 (6)	0·056 (3)
C(9)A	1·0627 (5)	−0·297 (14)	0·8745 (7)	0·060 (4)
C(10)A	0·9099 (5)	−0·2010 (12)	0·8373 (6)	0·046 (3)
O(1)B	0·1921 (3)	0·0973 (9)	0·4315 (4)	0·054 (2)
O(2)B	0·3447 (3)	0·0764 (10)	0·5594 (4)	0·056 (2)
O(3)B	0·4010 (3)	0·4158 (9)	0·3846 (4)	0·059 (2)
N(1)B	0·3009 (3)	0·0648 (11)	0·3624 (5)	0·052 (3)
N(2)B	0·5189 (4)	0·2612 (11)	0·3588 (5)	0·059 (3)
C(1)B	0·0489 (4)	0·1130 (16)	0·4470 (7)	0·078 (4)
C(2)B	0·1858 (6)	0·2978 (17)	0·5892 (8)	0·089 (4)
C(3)B	0·1820 (6)	−0·0733 (17)	0·5990 (9)	0·098 (5)
C(4)B	0·1545 (4)	0·1073 (12)	0·5202 (5)	0·052 (3)
C(5)B	0·2862 (4)	0·0803 (11)	0·4607 (5)	0·047 (3)
C(6)B	0·3966 (4)	0·0574 (11)	0·3683 (5)	0·047 (3)
C(7)B	0·3975 (5)	−0·0643 (12)	0·2654 (6)	0·069 (3)
C(8)B	0·4937 (5)	−0·0599 (13)	0·2584 (7)	0·070 (4)
C(9)B	0·5632 (5)	0·0764 (14)	0·3462 (6)	0·068 (3)
C(10)B	0·4373 (4)	0·2633 (11)	0·3698 (5)	0·049 (3)

$*U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$

at the -Leu-Gly- sequence that characterize the crystal structure of H-L-Pro-L-Leu-Gly-NH_2 (Reed & Johnson, 1973) are also characteristic of the biologically active conformation of the neuropeptide.

Here we describe the structural characterization by X-ray diffraction of (3*S*)-3-*tert*-butyloxycarbonylamino-2-piperidone (1), a useful intermediate in the synthesis of the δ-lactam analog of H-L-Pro-L-Leu-Gly-NH_2 in which the (3*S*)-3-amino-2-piperidone residue replaces the C-terminal glycinamide moiety. The placement of the δ-lactam residue at the C-terminal position was designed to give rise to an analog of the aforementioned neuropeptide that could not form a β-bend. The aim of the present study was to assess whether the experimentally observed conformation of the δ-lactam derivative (1) is compatible with one of the low-energy conformations calculated for a similar model compound [(3*S*)-3-acetylamino-2-oxo-1-piperidine *N*-methylacetamide] by Freidinger, Veber, Hirschmann & Paege (1980).

Experimental. Colorless prismatic crystals (0·2 × 0·2 × 0·3 mm) of (3*S*)-3-*tert*-butyloxycarbonylamino-2-piperidone were obtained from a mixture of CH_2Cl_2 and petroleum ether (b.p. 303–333 K) solution by slow evaporation. Intensities were collected on a Philips PW 1100 four-circle diffractometer operating in the θ/2θ scan mode (scan width 1·2° and scan speed 0·03° s^{-1}) with graphite-monochromatized Mo *K*α radiation. For measuring lattice parameters, 24 reflections in the range $7{\cdot}5 \le \theta \le 14{\cdot}5°$ were used. No absorption correction was applied. Maximum value of θ reached in intensity measurements 28°; *h, k, l* range −15 to 15, 0 to 7, 0 to 14. During data collection, three standard reflections with 10% intensity variation were measured every 180 min to check stability of the crystal and the electronics. Number of reflections measured 3259; number of unique reflections 3139; value of R_{int} 0·02; number of unobserved [$I < 3\sigma(I)$] reflections 1528.

The structure was solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refined by block-diagonal least squares (based on *F*) with anisotropic thermal parameters for all non-H atoms ($w = 1$). Some of the H atoms were located on the difference Fourier map but not refined and some were calculated. All calculations were performed with atomic scattering values of Sheldrick (1976). Parameters refined 271; $R = 0{\cdot}055$; ratio of maximum least-squares shift to e.s.d. in final refinement cycle 0·8; maximum and minimum height in final difference Fourier synthesis 0·25 and −0·20 e Å^{-3}; $S = 0{\cdot}66$. Table 1 gives the final atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms.*

Discussion. Fig. 1 gives a view of the two independent molecules *A* and *B* in the asymmetric unit of compound (1). Bond lengths, bond angles, and torsion angles are listed in Table 2.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Center as Supplementary Publication No. SUP 51386 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

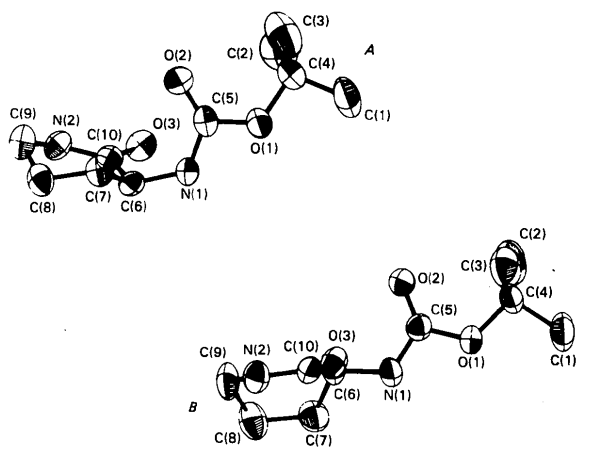

Fig. 1. Thermal-ellipsoid plot (60% level) of molecules *A* and *B* of compound (1) with numbering of the atoms.

Table 2. *Bond lengths* (Å), *bond angles* (°), *and torsion angles* (°) *for compound* (1) (*with e.s.d.'s in parentheses*)

	Molecule *A*	Molecule *B*
O(1)–C(4)	1·465 (10)	1·450 (9)
O(1)–C(5)	1·350 (8)	1·360 (8)
O(2)–C(5)	1·219 (7)	1·207 (7)
O(3)–C(10)	1·238 (10)	1·220 (9)
C(1)–C(4)	1·502 (13)	1·525 (8)
C(2)–C(4)	1·517 (10)	1·514 (13)
C(3)–C(4)	1·510 (14)	1·516 (13)
C(6)–C(7)	1·536 (11)	1·533 (11)
C(6)–C(10)	1·529 (11)	1·520 (10)
C(7)–C(8)	1·537 (12)	1·530 (12)
C(8)–C(9)	1·515 (11)	1·505 (11)
N(2)–C(9)	1·467 (11)	1·460 (12)
N(1)–C(5)	1·342 (9)	1·345 (10)
N(1)–C(6)	1·454 (9)	1·458 (8)
N(2)–C(10)	1·337 (9)	1·327 (10)
C(4)–O(1)–C(5)	122·2 (7)	121·0 (7)
C(5)–N(1)–C(6)	122·3 (7)	120·0 (7)
C(9)–N(2)–C(10)	126·7 (7)	122·0 (8)
C(1)–C(4)–O(1)	109·2 (8)	101·8 (7)
C(2)–C(4)–O(1)	102·3 (8)	110·0 (8)
C(3)–C(4)–O(1)	111·8 (8)	111·6 (8)
C(1)–C(4)–C(2)	111·1 (9)	110·1 (9)
C(1)–C(4)–C(3)	111·5 (8)	111·5 (9)
C(2)–C(4)–C(3)	110·6 (10)	111·4 (7)
O(1)–C(5)–O(2)	125·6 (9)	124·3 (10)
O(1)–C(5) N(1)	108·5 (8)	108·5 (8)
O(2)–C(5)–N(1)	125·9 (10)	127·2 (10)
N(1)–C(6)–C(7)	112·4 (7)	109·1 (8)
N(1)–C(6)–C(10)	110·2 (8)	112·3 (8)
C(7)–C(6)–C(10)	114·1 (8)	110·7 (7)
C(6)–C(7)–C(8)	108·1 (7)	112·1 (7)
C(7)–C(8)–C(9)	109·8 (9)	113·5 (9)
C(8)–C(9)–N(2)	110·9 (8)	111·6 (9)
O(3)–C(10)–N(2)	121·9 (8)	122·8 (8)
O(3)–C(10)–C(6)	120·2 (10)	123·6 (10)
N(2)–C(10)–C(6)	117·7 (9)	113·4 (8)
C(5)–O(1)–C(4)–C(1)	−61·0 (12)	−176·9 (8)
C(5)–O(1)–C(4)–C(2)	−178·8 (9)	66·3 (11)
C(5)–O(1)–C(4)–C(3)	62·9 (12)	−57·8 (12)
C(4)–O(1)–C(5)–O(2)	−8·1 (15)	−1·6 (14)
C(4)–O(1)–C(5)–N(1)	173·3 (8)	177·1 (8)
C(6)–N(1)–C(5)–O(1)	−172·3 (8)	176·1 (8)
C(6)–N(1)–C(5)–O(2)	9·0 (15)	−5·3 (15)
C(5)–N(1)–C(6)–C(7)	−76·1 (11)	150·4 (9)
C(5)–N(1)–C(6)–C(10)	52·3 (11)	−86·5 (10)
C(10)–N(2)–C(9)–C(8)	−26·8 (14)	46·1 (13)
C(9)–N(2)–C(10)–O(3)	−172·6 (9)	177·3 (9)
C(9)–N(2)–C(10)–C(6)	12·4 (14)	0·2 (13)
N(1)–C(6)–C(7)–C(8)	174·4 (8)	173·9 (8)
C(10)–C(6)–C(7)–C(8)	48·1 (10)	49·8 (10)
N(1)–C(6)–C(10)–O(3)	34·1 (12)	11·8 (13)
N(1)–C(6)–C(10)–N(2)	−150·7 (9)	−171·1 (8)
C(7)–C(6)–C(10)–O(3)	161·6 (9)	134·1 (10)
C(7)–C(6)–C(10)–N(2)	−23·3 (12)	−48·9 (11)
C(6)–C(7)–C(8)–C(9)	−62·9 (10)	−5·3 (12)
C(7)–C(8)–C(9)–N(2)	51·1 (11)	−40·4 (12)

The values of bond lengths and bond angles compare well with those found in other *tert*-butyloxycarbonylamino (Benedetti *et al.*, 1980), amide (Chakrabarti & Dunitz, 1982), and δ-lactam (Chakrabarti & Dunitz, 1982; Norskov-Lauritsen, Bürgi, Hofmann & Schmidt, 1985) moieties. In particular: (i) The C—O distances in the two types of carbonyls, particularly those of molecule *A*, correlate with the infrared absorption data: the higher frequency band in the spectrum (1716 cm^{-1}) is attributed to the urethane C(5)=O(2) group, which shows the shorter C—O distance [1·219 (7) Å for molecule *A* and 1·207 (7) Å for molecule *B*], while the lower frequency band (1652 cm^{-1}) is associated with the δ-lactam C(10)=O(3) group, which has the longer distance [1·238 (10) Å for molecule *A* and 1·220 (9) Å for molecule *B*]. (ii) Unfavorable interactions between the bulky *tert*-butyl group and spatially proximate atoms, especially the carbonyl oxygen O(2), result in the alteration of several bond angles relative to values observed in unhindered compounds. (iii) The C(9)—N(2)—C(10) and N(2)—C(10)—C(6) bond angles of the δ-lactam amide group deviate markedly from the 120° value with the former increasing to 126·7 (7)° for molecule *A* and 122·0 (8)° for molecule *B*, and the latter decreasing to 117·7 (9)° for molecule *A* and 113·4 (8)° for molecule *B*.

With regard to torsion angles, the *tert*-butyloxycarbonylamino group is in its usual extended (*trans, trans*, or *b*-type) arrangement (Benedetti *et al.*, 1980). The sequence of torsion angles C(4)—O(1)—C(5)—N(1) and C(6)—N(1)—C(5)—O(1) is 173·3 (8) and −172·3 (8)° for molecule *A* while 177·1 (8) and 176·1 (8)° for molecule *B* (IUPAC–IUB Commission on Biochemical Nomenclature, 1970). A significant conformational difference between molecules *A* and *B* is seen in the value of the φ[C(5)—N(1)—C(6)—C(10)] torsion angle, 52·3 (11)° for molecule *A* but −86·5 (10)° for molecule *B*, as a consequence of a rotation of the ring structure relative to the exocyclic *tert*-butyloxycarbonylamino moiety. The values of the ψ[N(1)—C(6)—C(10)—N(2)] torsion angle, however, are rather close: −150·7 (9)° for molecule *A* and −171·1 (8)° for molecule *B*. In contrast to molecule *B*, the *cis* amide group of molecule *A* is markedly non-planar (Winkler & Dunitz, 1971), the C(9)—N(2)—C(10)—C(6) torsion angle being 0·2 (13)° in the former and 12·4 (14)° in the latter. The dihedral angle between normals to the average planes of the urethane and δ-lactam amides is 73·2 (12)° for molecule *A* and 96·4 (12)° for molecule *B*. An additional interesting difference between molecules *A* and *B* is found in the δ-lactam ring conformation: approximate half-chair for molecule *A* while boat for molecule *B*. The amplitude-phase pair (q_2, φ_2) and the puckering coordinate q_3 have values of 0·331 (8) Å, −86·9 (15)°, −0·381 (8) Å for the δ-lactam ring of molecule *A* and 0·642 (12) Å, −178·3 (7)°, −0·044 (7) Å for the δ-lactam ring of molecule *B*, respectively (Cremer & Pople, 1975). Conformational energy calculations of an (*S*)-3-amino-2-piperidone derivative have been performed by Freidinger *et al.* (1980). One of the two low-energy conformers (strain energy 101·3 J mol^{-1}) found for (3*S*)-3-acetylamino-2-oxo-1-piperidine *N*-methylacetamide is a half-chair with the torsion angle $\psi = -135°$, while one of the two conformers of slightly higher energy (111·8 J mol^{-1}) is a boat with $\psi = -173°$. Thus, the results of the theoretical analysis fit nicely with our experimental findings described here.

There are no intramolecular H bonds in compound (1). Rather, chains of molecule *A* and chains of

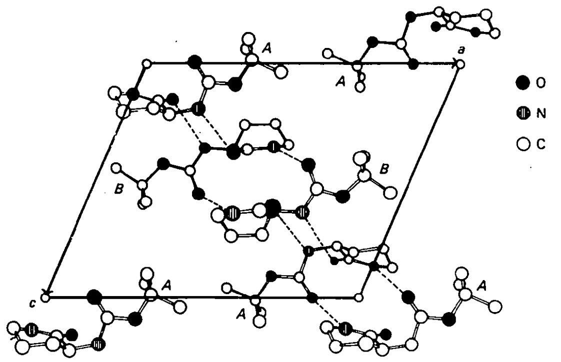

Fig. 2. Packing mode of molecules *A* and *B* of compound (1) viewed down the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines.

molecule *B* are formed *via* (lactam)N—H···O=C-(urethane) intermolecular H bonds along the *y* direction (Fig. 2). The N(2)*A*···O(2)*A* ($2-x, y-\frac{1}{2}, 2-z$) and N(2)*B*···O(2)*B* ($1-x, \frac{1}{2}+y, 1-z$) separations are 2·990 (8) and 2·880 (9) Å, respectively (Ramakrishnan & Prasad, 1971; Taylor, Kennard & Versichel, 1984). These chains are interconnected by the formation of dimeric structures *via* (urethane)N—H···O=C-(lactam) intermolecular H bonds. The N(1)*B*···O(3)*A* ($1-x, \frac{1}{2}+y, 1-z$) and O(3)*B*···N(1)*A* ($1-x, \frac{1}{2}+y, 1-z$) separations are 2·913 (10) and 2·868 (9) Å, respectively.

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Bond Length and Reactivity: 1-Arylethyl Ethers and Esters. 7.* Structure of 1-[3,5-Bis(trifluoromethyl)phenyl]ethyl 4-Nitrobenzoate

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Abstract. $C_{17}H_{11}F_6NO_4$, $M_r = 407{\cdot}3$, monoclinic, $C2/c$, $a = 15{\cdot}002$ (1), $b = 7{\cdot}709$ (1), $c = 30{\cdot}546$ (3) Å, $\beta = 93{\cdot}46$ (1)°, $V = 3526{\cdot}1$ Å^3, $Z = 8$, $D_x = 1{\cdot}53$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 1{\cdot}3$ mm^{-1}, $F(000) = 1648$, $T = 293$ K, $R = 0{\cdot}058$ for 2167 unique observed reflections. The CF_3 groups are disordered, with components of *ca* 0·9, 0·1; this led to slow convergence of refinement. The C—O bond length to

* Part 6: Edwards, Jones & Kirby (1986*a*).

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