Table 4. *Comparative dimensions* (Å *and* °) *of iodonium ylides*

Cpd	C—I—C	I—C(Ph)	I—C(7)	C—C(*)	C(*)—O	I···O	C—I···O
(1)	97·3 (3)	2·104 (9)	2·081 (8)	1·42 (1)	1·20 (1)	2·841 (9)' 2·82 (2)''	176·4 (3)' 169 (1)''
(2)	85·0 (9)/89·8 (7)	2·07 (2)	2·13 (2)	1·41 (4)	1·26 (2)	3·23 (2)'''	160 (1)/164 (1)'''
(3)	98·8	2·090	2·039	1·37	1·233	—	
(4)	98·7	2·089	2·078	1·40	—	2·755	148·9
(5)	98·4	2·106	2·105	1·412/1·356	1·26	*a*	

Notes: (1), (2) this work; (3) Martin-Schultz (1984) (intermolecular distances not reported); (4) Hubbard *et al.* (1980); (5) Druck & Littke (1978). (i) O at $-x-0·5$, $0·5+y$, $1·5-z$; (ii) O at x, $0·5-y$, $0·5+z$; involves C(7); (iii) O at $1-x$, $1-y$, z; involves C(1)/C(111). (*a*) Intramolecular distances to O of C═O group 2·965; 3·138 Å.

* Average distances involving C atoms adjoining C(7)[C(8), C(16) for (1), C(8), C(15) for (2)].

(1). These interactions dominate the packing (Figs. 3, 4).

In both (1) and (2), the I—C (phenyl) bond is about 15° off the perpendicular to the naphthalene plane [dihedral angles: C(ph)—I—C(7) to I—C(7)C_2O_2 of 103 (1)° and 108 (1)/106 (1)° respectively]; this is similar to (3) and (4) but contrasts with the 161° angle in (5) resulting from its close intramolecular contact.

Regioselectivity has been observed in some reactions of (1) with alkenes, leading to the formation of 1,4- rather than 1,2-naphthoquininic furan derivatives (Hatzigrigoriou *et al.*, 1989). This may now be explained from the crystal structure if it is assumed that the conformation of (1), with the phenyl group in an '*anti*' position in relation to O(3), is maintained in solution. The approach of the alkene from the side of O(1) will then be sterically hindered. This is probably the reason why (2) fails to undergo an analogous reaction, since the approach of the alkene from the side of the carbonyl group is hindered, whereas its lactonic carbonyl is unreactive on electronic grounds.

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Structure of N^{α}-Acetyl-*N*-methyl-L-tryptophanamide

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Abstract. $C_{14}H_{17}N_3O_2$ (Ac-L-Trp), $M_r = 259·14$, orthorhombic, $P2_12_12_1$, $a = 8·157$ (1), $b = 11·185$ (1), $c = 15·081$ (2) Å, $V = 1376$ Å^3, $Z = 4$, $D_x = 1·25$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0·8$ cm^{-1}, $F(000) = 552$, $T = 295$ K. Final R value 0·036 for 1407 [$I \geq 1·5\sigma(I)$] observed reflections, GOF = 1·06. The conformational angles are: $\varphi = -81·5$ (2), $\psi = 167·9$ (2), $\chi_1 = -67·1$ (2), $\chi_2 = 94·8$ (3)°. Each molecule is hydrogen bonded to six other molecules. The N-terminal carbonyl group accepts two hydrogen bonds and the H···O···H plane is approximately perpendicular to the peptide group.

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

	x	y	z	B(Å^2)
C(1)	−0·0645 (4)	−0·0205 (2)	0·4366 (2)	5·51 (7)
C(2)	−0·0334 (2)	0·0875 (2)	0·3805 (2)	3·10 (4)
C(3)	−0·0975 (2)	0·2958 (2)	0·3530 (1)	2·89 (4)
C(4)	0·0573 (2)	0·3646 (2)	0·3816 (1)	3·06 (4)
C(5)	0·2432 (3)	0·5261 (2)	0·3355 (2)	4·84 (6)
C(6)	−0·2553 (3)	0·3696 (2)	0·3623 (2)	3·70 (4)
C(7)	−0·3992 (2)	0·3116 (2)	0·3178 (2)	3·33 (4)
C(8)	−0·5132 (3)	0·2396 (2)	0·3556 (2)	4·27 (5)
C(9)	−0·5782 (2)	0·2451 (2)	0·2124 (2)	3·51 (4)
C(10)	−0·4372 (2)	0·3165 (2)	0·2252 (1)	3·07 (4)
C(11)	−0·3697 (3)	0·3748 (2)	0·1519 (2)	4·05 (5)
C(12)	−0·4407 (4)	0·3600 (3)	0·0698 (2)	5·01 (6)
C(13)	−0·5817 (4)	0·2876 (3)	0·0596 (2)	5·07 (6)
C(14)	−0·6501 (3)	0·2299 (2)	0·1294 (2)	4·41 (5)
O(1)	0·0621 (2)	0·0846 (1)	0·3165 (1)	3·53 (3)
O(2)	0·1368 (2)	0·3379 (2)	0·4471 (1)	4·06 (3)
N(1)	−0·1146 (2)	0·1860 (2)	0·4023 (1)	3·11 (3)
N(2)	0·0960 (2)	0·4547 (2)	0·3269 (1)	4·11 (4)
N(3)	−0·6217 (2)	0·1998 (2)	0·2932 (1)	4·41 (4)
H(N1)	−0·189	0·187	0·457	
H(N2)	0·018	0·476	0·275	
H(N3)	−0·731	0·155	0·301	

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $4/3[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab\beta(1,2)\cos\gamma + ac\beta(1,3)\cos\beta + bc\beta(2,3)\cos\alpha]$.

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

C(1)—C(2)	1·495 (4)	C(7)—C(8)	1·356 (3)
C(2)—O(1)	1·240 (3)	C(7)—C(10)	1·431 (3)
C(2)—N(1)	1·327 (3)	C(8)—N(3)	1·366 (3)
C(3)—C(4)	1·539 (3)	C(9)—C(10)	1·412 (3)
C(3)—C(6)	1·536 (3)	C(9)—C(14)	1·393 (4)
C(3)—N(1)	1·443 (3)	C(9)—N(3)	1·367 (3)
C(4)—O(2)	1·218 (3)	C(10)—C(11)	1·395 (4)
C(4)—N(2)	1·340 (3)	C(11)—C(12)	1·376 (4)
C(5)—N(2)	1·446 (3)	C(12)—C(13)	1·414 (5)
C(6)—C(7)	1·501 (3)	C(13)—C(14)	1·354 (4)

C(1)—C(2)—O(1)	121·7 (2)	C(10)—C(9)—C(14)	122·3 (2)
C(1)—C(2)—N(1)	116·5 (2)	C(10)—C(9)—N(3)	107·4 (2)
O(1)—C(2)—N(1)	121·8 (2)	C(14)—C(9)—N(3)	130·3 (2)
C(4)—C(3)—C(6)	113·3 (2)	C(7)—C(10)—C(9)	106·7 (2)
C(4)—C(3)—N(1)	111·2 (2)	C(7)—C(10)—C(11)	134·8 (2)
C(6)—C(3)—N(1)	109·2 (2)	C(9)—C(10)—C(11)	118·5 (2)
C(3)—C(4)—O(2)	122·7 (2)	C(10)—C(11)—C(12)	119·4 (2)
C(3)—C(4)—N(2)	113·4 (2)	C(11)—C(12)—C(13)	120·5 (3)
O(2)—C(4)—N(2)	123·9 (2)	C(12)—C(13)—C(14)	121·5 (3)
C(3)—C(6)—C(7)	112·6 (2)	C(9)—C(14)—C(13)	117·8 (3)
C(6)—C(7)—C(8)	127·1 (2)	C(2)—N(1)—C(3)	122·2 (2)
C(6)—C(7)—C(10)	126·1 (2)	C(4)—N(2)—C(5)	123·8 (2)
C(8)—C(7)—C(10)	106·7 (2)	C(8)—N(3)—C(9)	109·0 (2)
C(7)—C(8)—N(3)	110·2 (2)		
C(2)—N(1)—H(N1)	120 (1)	C(5)—N(2)—H(N2)	117 (1)
C(3)—N(1)—H(N1)	118 (1)	C(8)—N(3)—H(N3)	130 (1)
C(4)—N(2)—H(N2)	119 (1)	C(9)—N(3)—H(N3)	120 (1)

Introduction. We present here the crystal structure of N^{α}-acetyl-*N*-methyl-L-tryptophanamide (Ac-L-Trp) at room temperature as part of work on electron distribution in peptide links. Low-temperature study and electron-distribution results will be published elsewhere (Souhassou, Lecomte, Blessing, Aubry, Rohmer, Wiest, Benard & Marraud, 1990).

Experimental. Colourless crystals of Ac-L-Trp from an ethyl acetate/ethanol solution by slow evaporation. X-ray diffraction data collected on a CAD-4 diffractometer from a crystal of approximate dimensions 0·4 × 0·3 × 0·3 mm. Accurate unit-cell parameters and crystal orientation matrices (together with their e.s.d.'s) from least-squares refinement of the 2θ, ω, χ and φ values of 25 carefully centred reflections with $20 < \theta < 40°$. *hkl* ranges: 0 to 10, 0 to 13, 0 to 18 respectively. ω–2θ scan mode [scan width (°) = 1 + 0·15tanθ, $\sigma_{\text{prescan}} = 1$, $\sigma_{\text{final}} = 0{\cdot}02$, $N_{\text{pipre}} = 6$ and max. time = 90 s] and Cu $K\alpha$ radiation monochromatized by a graphite crystal. During data collection two standard reflections (242 and 211) measured every 120 min to check the stability of the crystal and the electronics. No variation observed. 1576 reflections up to $\theta = 72°$, 1407 with $I \geq 1{\cdot}5\sigma(I)$. Intensities corrected for Lorentz and polarization effects. No absorption correction applied. Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The *E* maps of the set with the best combined figure of merit showed all the heavy atoms. Refinement carried out by full-matrix least squares, allowing the non-H atoms to vibrate anisotropically. Scattering factors taken from *International Tables for X-ray Crystallography* (1974). H atoms localized in the difference Fourier maps and refined in the last cycles. Quantity minimized: $w(|F_o|)^2$ with $w = [\sigma^2(F) + 0{\cdot}00174F^2]^{-1}$. For all calculations *SHELX*76 (Sheldrick, 1976) was used. Final conventional unweighted *R* factor 0·036 and weighted *R* factor 0·040; $S = 1{\cdot}06$. $(\Delta/\sigma)_{\max}$ in final refinement cycle for positional parameters of the non-H atoms 0·40. Max. and min. heights in final

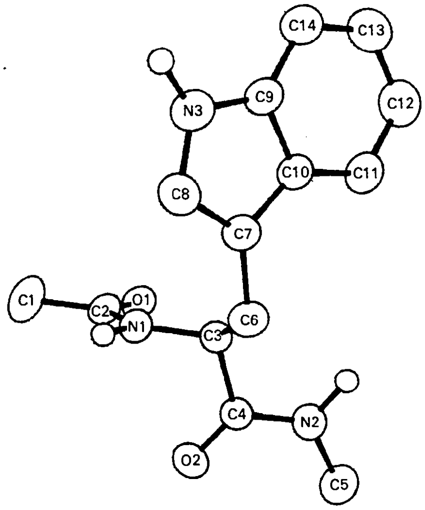

Fig. 1. View of the molecule showing the atomic numbering scheme.

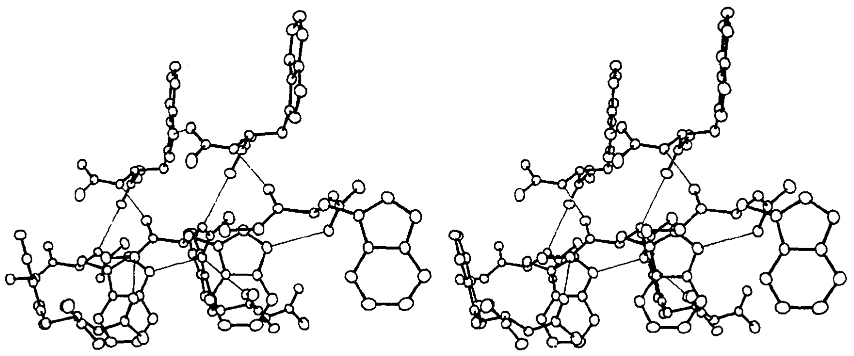

Fig. 2. Packing mode of the molecules.

difference Fourier synthesis ±0·13 e Å^{-3}. According to Taylor & Kennard's (1983) recommendation, the N—H hydrogen atoms were replaced at 1·03 Å from N in the direction obtained by refinement. Table 1 gives the final atomic coordinates and the equivalent isotropic thermal parameters.*

Discussion. The molecular structure with the atomic numbering scheme is shown in Fig. 1. Bond lengths and angles are given in Table 2. Fig. 2 illustrates the packing mode of the molecules.

Bond lengths and bond angles are in good agreement with those of the standard peptide unit (Benedetti, 1982) and both peptide units are nearly *trans* planar. The N-terminal peptide link and the tryptophan cycle are strictly planar. Probably due to the hydrogen bonds, the C-terminal peptide link shows a twist of about 5° between the plane C(3)—C(4)—O(2)—N(2) and the plane C(4)—N(2)—H(N2)—C(5). Table 3 gives the main conformational angles with the nomenclature of the IUPAC–IUB Commission on Biochemical Nomenclature (1970) recommendations in comparison with those of N^{α}-acetyl-N-methyl-DL-tryptophanamide (Harada & Iitaka, 1977).

Table 3. *Conformational angles* (°) *with e.s.d.'s in parentheses*

		Ac-L-Trp	Ac-DL-Trp
ω_1	C(1)—C(2)—N(1)—C(3)	−179·2 (2)	
ω_2	C(3)—C(4)—N(2)—C(5)	−174·7 (2)	
φ	C(2)—N(1)—C(3)—C(4)	−81·5 (2)	−103·7
ψ	N(1)—C(3)—C(4)—N(2)	167·9 (2)	141·4
χ_1	N(1)—C(3)—C(6)—C(7)	−67·1 (2)	−65·3
χ_2^1	C(3)—C(6)—C(7)—C(8)	94·8 (3)	98·7
χ_2^2	C(3)—C(6)—C(7)—C(10)	−81·3 (3)	−78·9

Both derivatives have similar backbone conformations and orientations of the side chain. However, they differ in their packing modes and hydrogen-bond networks. In the case of the chiral derivative each molecule is hydrogen bonded to six molecules in a three-dimensional network (Fig. 2). The N-terminal carbonyl group is engaged in a double hydrogen bond defining an H···O···H plane nearly perpendicular (~70°) to that of the peptide group [N(1)···O(2) $(-\frac{1}{2}+x,\ \frac{1}{2}-y,\ 1-z)$ = 3·057 (2), H(N1)···O(2) = 2·04 (2) Å, N(1)—H(N1)···O(2) = 168 (2)°; N(2)···O(1) $(-x,\ \frac{1}{2}+y,\ \frac{1}{2}-z)$ = 2·907 (3), H(N2)···O(1) = 1·96 (2) Å, N(2)—H(N2)···O(1) = 152 (2)°; N(3)···O(1) $(1-x,\ y,\ z)$ = 2·905 (3), H(N3)···O(1) = 1·88 (2) Å, N(3)—H(N3)···O(1) = 176 (2)°]. The racemic derivative presents an antiparallel association leading to sheets which are bonded by means of an O(1)···N(3) intermolecular hydrogen bond in such a way that the N-terminal carbonyl group also accepts two hydrogen bonds. In this case the angle characterized above is close to 50°.

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* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52386 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.