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1,3-Dipolar cycloadditions: Part II†—Cycloaddition of 1-pyrroline 1-oxide to 1-cinnamoyl piperidine and 1-(4′-chlorocinnamoyl) piperidine

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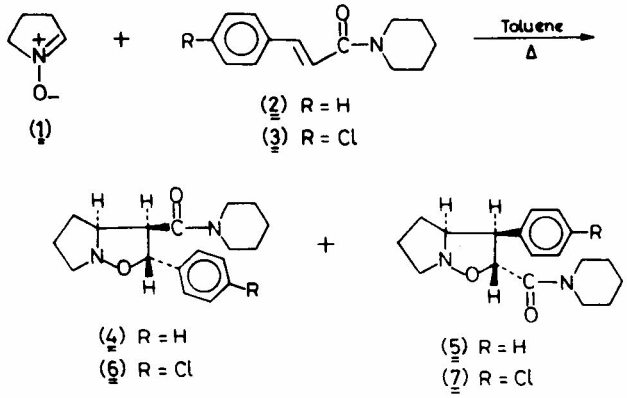
Cycloaddition of 1-pyrroline 1-oxide **1** to 1-cinnamoylpiperidine **2** and 1-(4′-chlorocinnamoyl) piperidine **3** gives two regioisomeric cycloadducts in each case. The structure and stereochemistry of the cycloadducts have been confirmed from spectroscopic and X-ray crystallographic analysis.

Cycloaddition reactions of nitrones to unsaturated substrates constitute the best procedure for the construction of the isoxazolidine ring system[1-3] with regio- and stereo-chemical controls. The isoxazolidine ring containing two heteroatoms can be considered to be the masked form of several functional group combinations. Thus, nitrone cycloadducts are attractive intermediates for the synthesis of several classes of biologically active compounds and Natural Products[1-4].

We have earlier reported out investigations on the cycloaddition of C,N-diarylnitrones to the conjugated γ-lactone 2-butenolide[5]. We report here the course of cycloaddition of the five-membered cyclic nitrone 1-pyrroline-1-oxide **1**[6] to 1-cinnamoyl piperidine **2** and 1-(4′-chlorocinnamoyl)piperidine **3**.

The cycloadditions were performed by refluxing equimolar amounts of the dipoles and dipolarophiles in anhydrous toluene for 12 hr under nitrogen atmosphere. After this period only small amounts of the dipolarophiles survived, and apparently a part of the nitrone had decomposed. The post-reaction mixture on chromatography yielded two regioisomeric cycloadducts 2-aryl-3-piperidinyloxo-2,3, 3a,4,5,6-hexahydropyrrolo [1,2-b] isoxazole **4** **6** and 3-aryl-2-piperidinyloxo-2,3,3a,4,5,6-hexahydropyrrolo [1,2-b] isoxazole **5**/**7** (Scheme I).

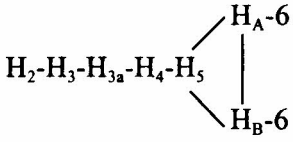
The structure and relative configuration of the



Scheme I

products were established on the basis of spectroscopic data, particularly detailed NMR analysis including 2D-COSY.

Compounds **4**, **5**, **6** and **7** exhibited a strong IR band for the amide carbonyl at 1635, 1645, 1650 and 1660 cm^{-1}, respectively. The 300 MHz ^1H NMR assignments of **4**, **5**, **6** and **7** are given in Table I. Decoupling and COSY -experiments established the following coupling information **a** for the non-aromatic protons.



The H-2 proton appeared as a doublet in both **4** and **5**. In compound **5** this proton resonated at δ 4.62 ppm, i.e. about 1 ppm upfield when compared to H-2 proton of **4** (δ 5.52 ppm). The H-3 proton

†For Part I of the series, see ref.5.

Table I—300 MHz ^{1}H NMR assignments of compounds **4**, **5**, **6**, and **7**

Proton	Chemical shifts in δ ppm,, multiplicity (*J* in Hz),			
	4	**5**	**6**	**7**
H-2	5.52,d(9.3)	4.62,d(7.8)	5.49,d(9.3)	4.55,d(7.9)
H-3	3.59,dist.t(8.8)	3.99,dd(7.8,6.0)	3.51,dist.t(8.8)	4.00,dd(7.9,5.9)
H-3a	4.03,dist.q	3.81,q(6.0)	4.01,dist.q	3.79,q(5.9)
H-4		1.92,m		1.93,m.
H_A-5	1.72-1.89,m	2.12,m	1.70-1.87,m	2.09,m
H_B-5		1.75,m		1.75,m
H_α-6	3.29, m	3.44,m	3.27,m	3.38,m
H_β-6	2.02, m	3.05,dt(13.0,7.8)	2.03,m	3.02,dt(13.0,7.8)
H_A-2′	3.72, m	3.61,m	3.72,m	3.62,m
H_B-2′	3.29, m	3.44,m	3.27,m	3.38,m
H-3′,4′,5′	1.45-1.60, m	1.40-1.60,m	1.44-1.60,m	1.40-1.60,m
H_A-6′	3.43, m	3.50,m	3.41,m	3.54,m
H_B-6′	3.29, m	3.38,m	3.27,m	3.38,m
H-2″,6″	7.48, d(7.0)	7.41,d(7.2)	7.42,d(8.4)	7.35
H-4″		7.33,dist.t	-	— *AB* System (8.6)
H-3″, 5″	7.29, m	7.25,d(7.5)	7.27,d(8.7)	7.30

Assignments are in agrrement with COSY-90° experiments of compounds **4** and **5**. Additional long-range couplings evident from COSY-LR-90° were H-2 and H-3 with the corresponding *ortho* protons in **4** and **5** respectively. W-coupling of H-3a with Hα -6 in both **4** and **5**.

appeared at δ 3.99 ppm as a double doublet for compound **5**, a downfield shift of ~0.5 ppm compared to **4** in which the same proton appeared at δ 3.59 ppm as a distorted triplet. In simple systems of the type >CH-Ph the methine proton appears at ~δ 2.8-2.9 ppm while for compounds of the type >CHCONR$_2$ the methine proton resonates at ~2.2 ppm[7,8]. This trend in chemical shift differences of the methines attached to an aryl and carboxamido group is maintained in **4** and **5**. The chemical shifts of H-3a proton for both compounds were comparable. From the above observantions, it seemed apparent that in **4** the phenyl group is attached to C-2 whereas in compound **5** the carboxamido group is linked to C-2, clearly showing that the cycloadducts bear a regioisomeric relationship with one another. The NMR characteristics of **6** and **7** were very similar to those of the cycloadducts **4** and **5** respectively (Table I).

The proposed regiochemistry of **4** and **5** were confirmed by LR-COSY analysis. The C-2 proton showed a long-range correlation with the aromatic *ortho*-protons at δ 7.48 ppm in **4**, whereas a similar long-range correlation of C-3 proton with the aromatic *ortho*-protons was observed for **5** (Figures 1 and 2, Table I).

This regiochemical relationship was in agreement with mass spectral analyses. The EI-MS of compound **4** showed that the molecular ion (M^+) peak at m/z 300 (7.3%) whereas the CI-MS of compound **5** showed the (M+1) peak at m/z 301 (2.7%). The MS fragmentation patterns of **4** and **5** were broadly similar in some respects which reflected the general similarity in their structures. In both compounds peaks at m/z 215 and 86 were obtained with comparable relative intensities, and arose due to initial electron-impact induced cycloreversion. The fragmentation pattern of compound **4** (Scheme II) showed two fragments at m/z 194 and 106, whereas similar fragmentation of **5** gave two peaks at m/z 159 and 142 respectively. This is reflective of the regioisomeric nature of the two cycloadducts.

The relative configuration of **4** was established by X-ray crystallographic analysis. The established it to be the 3-3a *cis*-isomer (Figure 3), the *trans*-relationship between C_2 - and C_3 - substituents being maintained from the substrate. The A-B ring juncture is *cis* - with the lone pair of the tertiary

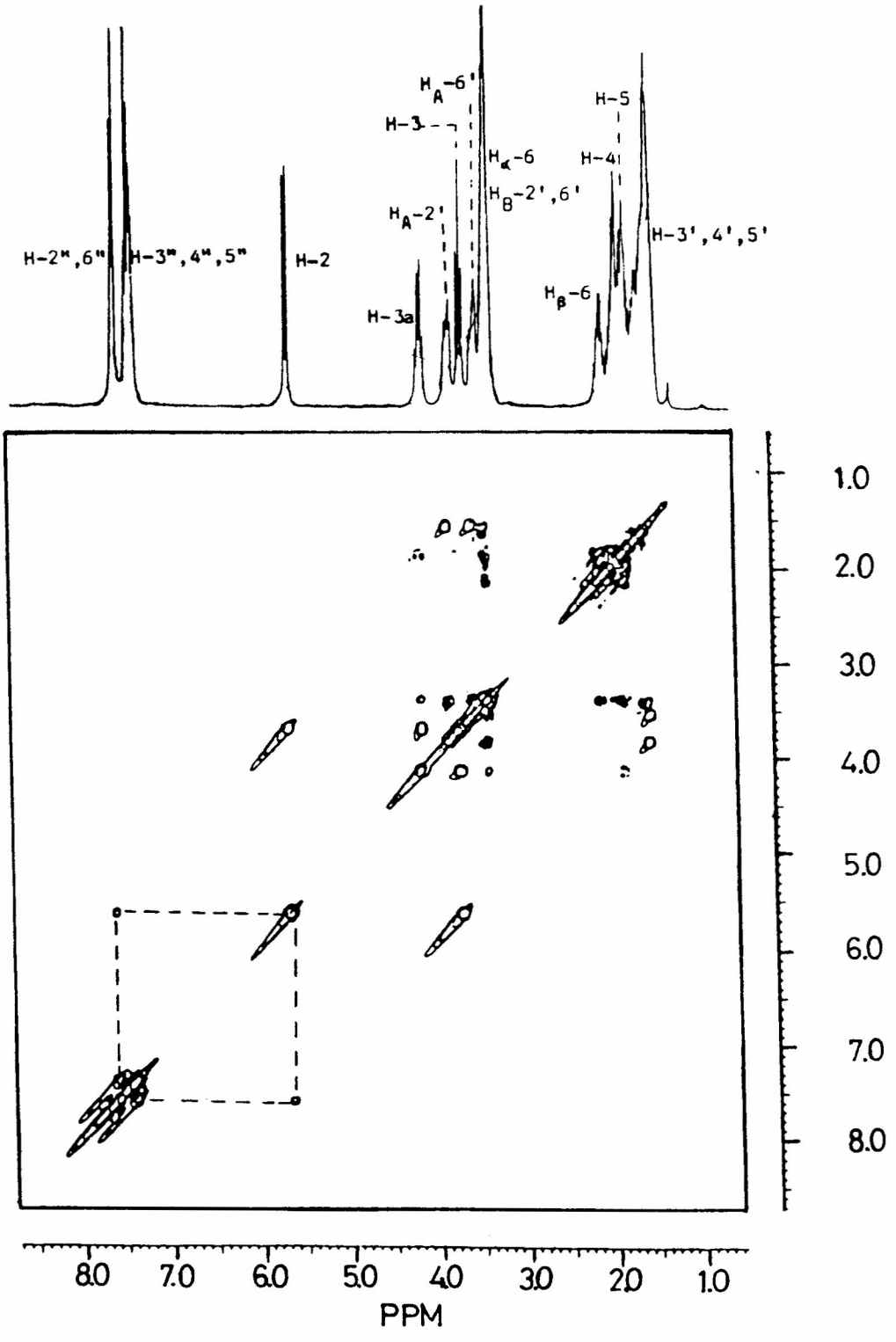


Figure 1—300 MHz ^{1}H-^{1}H-COSY-LR-90° spectrum of **4** in $CDCl_3$

nitrogen pointing in the same direction as the $C_{(3a)}$-H bond with the N-atom clearly pyramidal. The projection diagram is given in Figure 3 while the relevant data are given in Tables III-IV.

The $J_{3\text{-}3a}$ values for the pair of regioisomers were similar (Table I) corresponding to the dihedral angles of 15-20°, indicating that they had the same relative stereochemistry at 3-3a position.

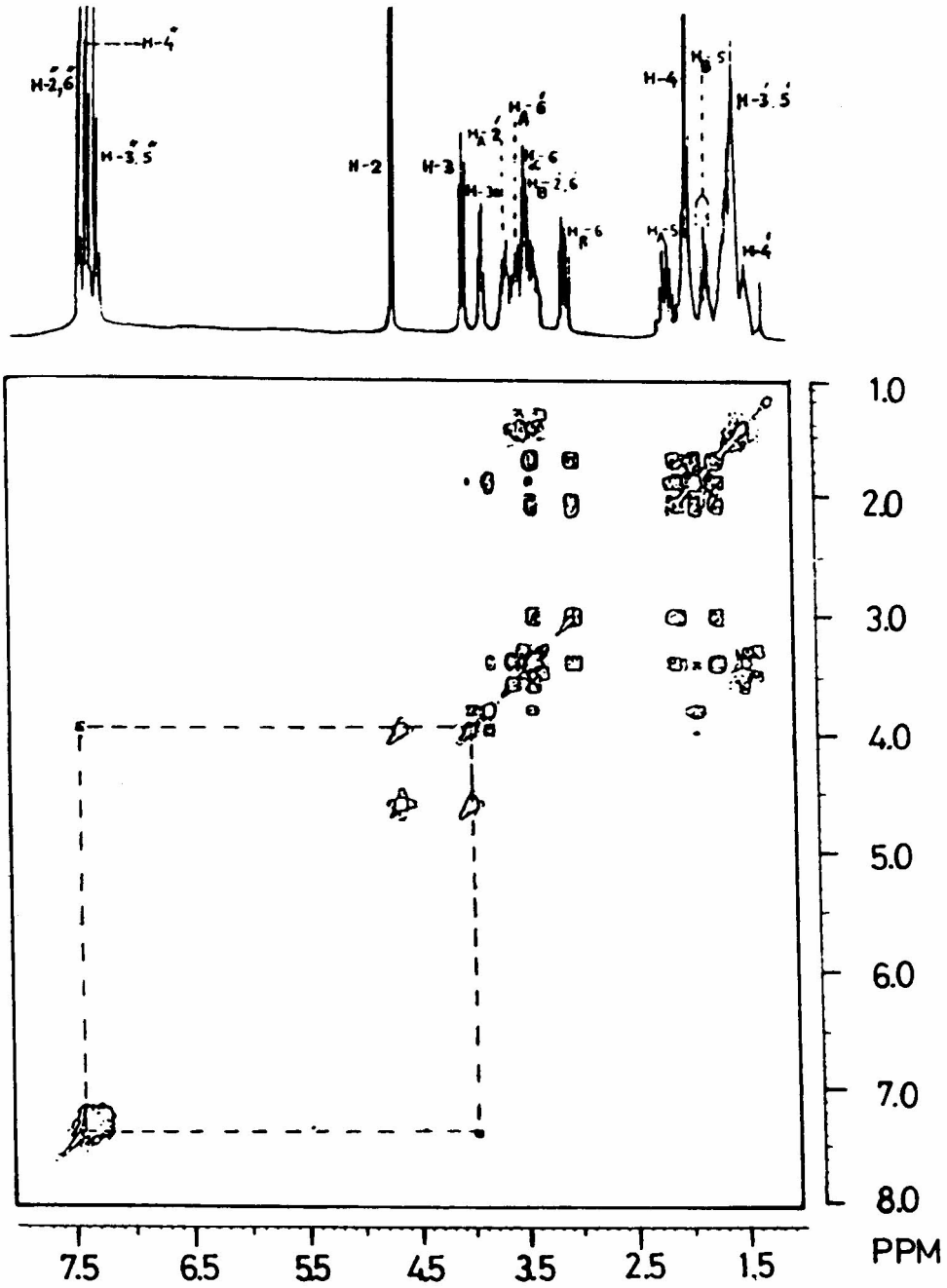
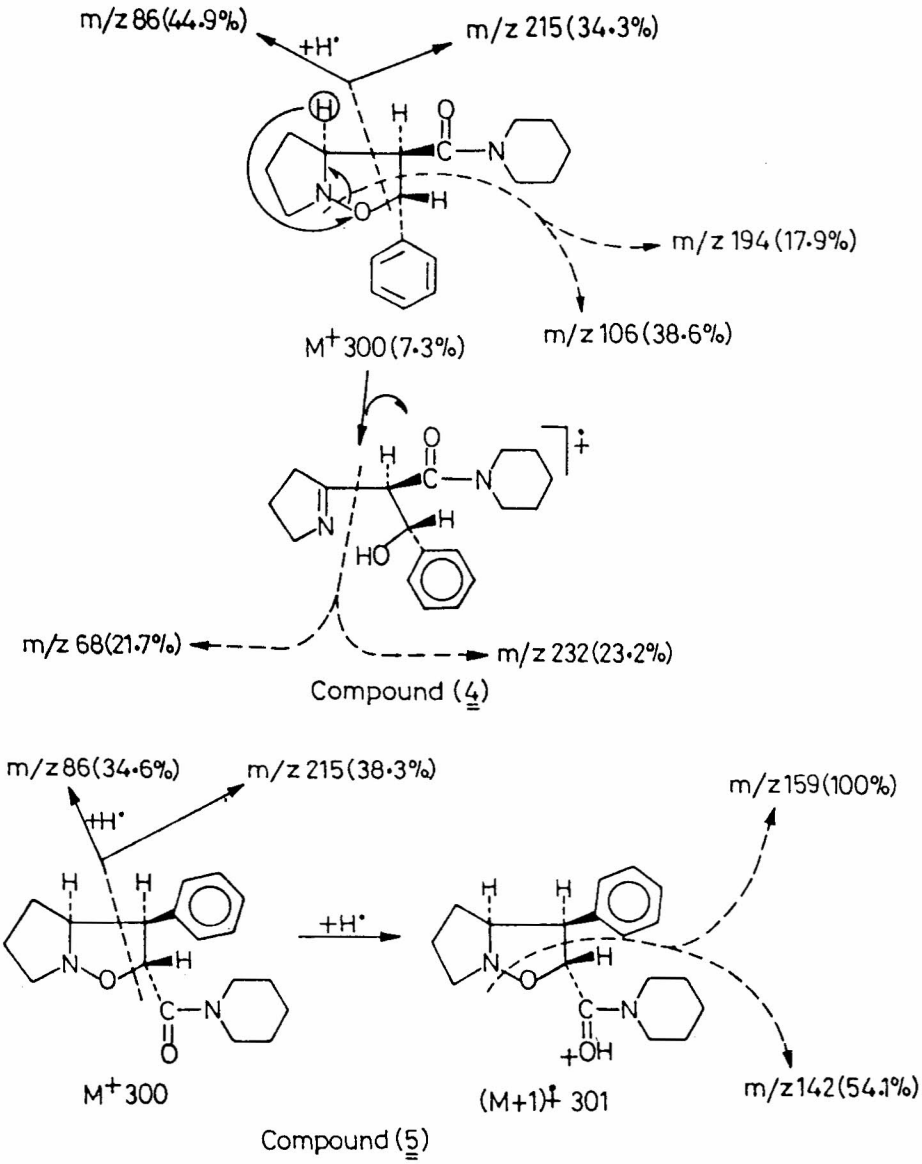


Figure 2—300 MHz H-H-COSY-LR-90° spectrum of **5** in $CDCl_3$

The 75.5 MHz ^{13}C NMR spectra of the two cycloadducts **4** and **5** were broadly similar with slight variation in the chemical shift values reflective of their regioisomeric relationship (assignments in Table II). The C-2 carbon appearing at δ 80.02 in compound **4**, was shifted 3.55 ppm downfield to δ 83.57 ppm in compound **5**. The regio- and stereo-chemical assignments for **6** and **7** followed from a close similarity of their spectroscopic data, particularly NMR, with those of **4** and **5** respectively (Tables I and II).

Nitrone cycloadditions are usually classified as Sustmann[9] type-II processes where both HOMO-LUMO interactions can contribute effectively to the stabilisation of the transition state[10]. The lack of pronounced ragio-selectivity could be due to the high reactivity of the cyclic nitrone and the



Scheme II

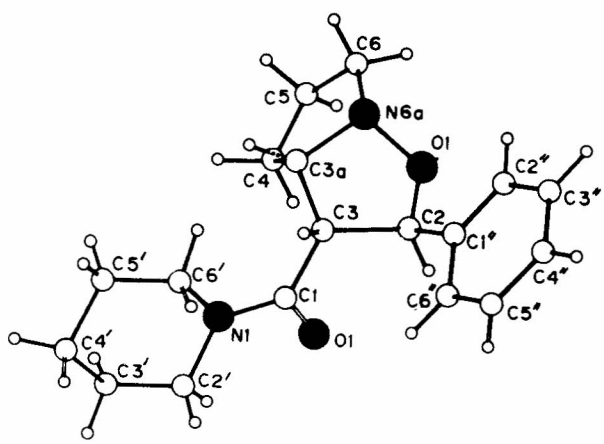


Figure 3 —X-ray crystallographic structure of **4**.

possibility of some equilibration occurring under the reaction conditions.

Experimentally it was observed that for both the regioisomers H-3, and H-3a had a *cis*- relationship. This is in line with the preference of *endo*- mode of attack (**A**) where facourable secondary orbital interactions are large enough to overcome the steric encumbrances present in the endo-transition state[4] (Figure 4).

Experimental Section

General. Melting points were recorded on a Köfler block apparatus and are uncorrected. IR spectra were recorded on a Perkin-Elmer 782

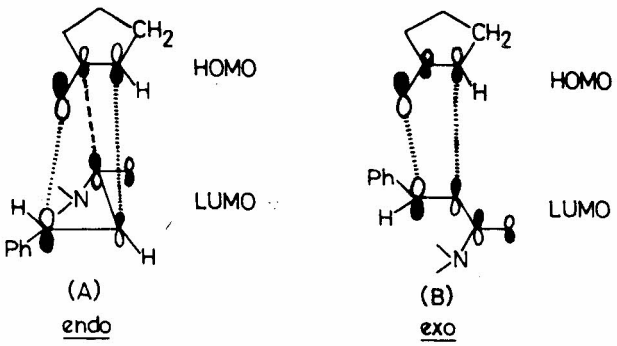


Figure 4

Table II—75.5 MHz ^{13}C NMR assignment of compounds **4**, **5**, **6** and **7**

Carbon	^{13}CNMR chemical shift (in δ,ppm),			
	4	**5**	**6**	**7**
2	80.2	83.57	79.46	83.85
3	58.17	58.04	58.56	57.83
3a	66.96	74.62	67.06	74.51
4	27.63	30.35	27.09	30.26
5	23.59‡	23.27‡	24.42‡	23.58‡
6	57.11	56.92	57.33	56.79
2′	42.93	43.49	43.10	43.47
3′	25.51	25.55	25.64	25.52
4′	23.92‡	24.53‡	24.50‡	24.48‡
5′	26.54	26.55	26.70	26.38
6′	46.92	46.74	46.40	46.89
2″,6″	126.65	128.18	128.13	129.35
4″	127.62	126.98	132.04	132.34
3″,5"	128.21	128.76	128.56	128.70
1"	139.39	140.59	138.21	138.54
>C=O	167.04	165 90	167.47	167.02

‡Assignments are interchangeable.

Table III—Positional parameters (×10^4) and mean recalculated isotropic factors (×10^3) for non-hydrogen atoms (*)

Atom	*X*	*Y*	*Z*	<*U*>
01	9679 (6)	2797 (6)	1205 (7)	74 (9)
C2	8357 (7)	3313 (9)	500 (10)	54 (11)
C3	8254 (7)	3302 (8)	−1239 (10)	54 (11)
C3a	9641 (8)	2762 (10)	−1275 (11)	71 (13)
C4	9870 (9)	1026(10)	−2188(13)	89(16)
C5	11266(10)	716(12)	− 952(16)	113(17)
C6	11697 (8)	2244(10)	707(13)	85(16)
N6a	10528 (6)	3290 (7)	611 (9)	69(10)
C1	7100 (8)	2279(10)	−2821(11)	66(12)
N1	6614 (7)	2508 (8)	4272 (9)	87(11)
01	6601 (5)	1290 (6)	−2748 (7)	81 (9)
C1"	8098 (7)	4860 (9)	1720(10)	55(11)
C2"	6846 (8)	5517 (10)	1219(12)	73(14)
C3"	6516 (8)	6910(11)	2341(11)	86(16)
C4"	7462 (10)	7684(10)	4017(11)	82(15)
C5"	8718 (9)	7041(11)	4495(12)	78(15)
C6"	9038 (7)	5649 (9)	3365(11)	61(12)
C2′	7082(12)	3702(14)	−4486(11)	133(15)
C3′	7087(13)	3293(16)	−6080(11)	163(18)
C4′	5851(11)	2471(14)	−7636(11)	124(16)
C5′	5451(11)	1198(14)	−7366(10)	128(17)
C6′	5384(11)	1700(14)	−5727(10)	127(17)

*Given in Å^2 and calculated as $<U>= \sum_i \sum_j U_{ij}.a^*_i\, a^*_j a_i.a_j$

spectrometer and mass spectra on a Jeol JMS D-300 mass spectrometer. 300 MHz ^{1}H NMR spectra and 75.5 MHz ^{13}C NMR spectra as as well as 2D COSY and LR-COSY spectra were recorded on a Bruker AM-300L superconducting magnet NMR spectrometer using a 5 mm ^{1}H-^{13}C-dual probe operating with the Bruker DISR 871 software (chemical shifts are in δ, ppm relative to TMS as internal reference for solutions in $CDCl_3$). Elemental analyses were carried out by the Microanalytical Laboratory, Department of Chemistry, Calcutta University.

Neutral alumina (Acme's) was used for column chromatography. Analytical TLC was performed using Merck silica gel G support. Organic extracts were dried over anhydrous Na_2SO_4. Analytical samples were routinely dried over P_2O_5 *in vacuo*.

Nitrone **1** was prepared by SeO_2 catalysed H_2O_2 oxidation of pyrrolidine according to the procedure of Murahashi and Shiota[6]. 1-Cinnamoylpiperidine **2** and 1-(4′-chlorocinnamoyl)piperidine **3** were prepared by a standard procedure[9,10].

General method of cycloaddition. To a hot solution of the nitrone **1** (1.9 mmoles) in anhydrous toluene (10 mL), a solution of piperidine (1.9 mmoles) in anhydrous toluene (25 mL) was added over a short period of time and the reaction mixture the refluxed under nitrogen atmosphere for 12 hr. The progress of the reaction was monitored by TLC. The crude post-reaction mixture was chromatographed over neutral alumina to separate the products.

2R*S*(2*R, 3*R**, 3a*S**)-2,3,3a,4,5,6-hexahydro-2-phenyl-3-piperidinocarbonylpyrrolo[1, 2-*b*] isoxazole 4**. Yield 210 mg (30%), m.p. 152° (petrol - benzene 1:1), obtained from 1% ethyl

Table IV—Anisotropic thermal parameters ($\times 10^4$) for non-hydrogen atoms

Atom	*U*11	*U*22	*U*33	*U*23	*U*13	*U*12
01	701(38)	622(37)	613(42)	309(33)	297(32)	74(29)
C2	485(45)	469(50)	427(52)	169(43)	212(40)	– 7(36)
C3	551(46)	436(47)	331(49)	76(39)	78(39)	– 197(37)
C3a	582(51)	694(61)	543(61)	290(50)	208(46)	– 126(43)
C4	824(67)	555(58)	796(76)	– 13(55)	464(61)	– 66(49)
C5	800(71)	748(74)	1200(87)	233(73)	378(72)	78(56)
C6	514(51)	620(61)	964(85)	250(61)	276(55)	136(45)
N6a	578(41)	598(44)	561(49)	239(38)	161(37)	– 136(35)
C1	543(49)	646(57)	465(58)	228(47)	80(42)	– 202(42)
N1	900(52)	841(53)	385(47)	253(41)	– 101(40)	– 636(43)
01	762(38)	767(41)	479(40)	260(32)	42(31)	– 421(33)
C1"	427(43)	560(53)	438(53)	255(45)	154(40)	– 62(39)
C2"	623(58)	655(62)	501(61)	98(51)	133(48)	– 216(47)
C3"	529(54)	761(70)	830(82)	237(63)	248(58)	2(49)
C4"	819(66)	538(56)	687(74)	72(53)	409(60)	– 105(51)
C5"	711(61)	664(64)	592(66)	208(55)	260(52)	– 147(49)
C6"	529(47)	579(56)	452(56)	188(47)	177(45)	– 118(42)
C2'	1351(94)	1332(97)	578(77)	441(71)	– 147(67)	– 966(80)
C3'	1329(95)	1919(93)	827(98)	776(97)	– 174(80)	–1048(98)
C4'	947(75)	1563(90)	618(75)	617(74)	– 55(60)	– 536(73)
C5'	1044(81)	1280(91)	814(92)	413(81)	22(70)	– 661(73)
C6'	1165(85)	1360(99)	642(79)	467(75)	– 14(67)	– 802(76)

acetate in benzene eluates (Found: C, 71.8; H,8.1; N, 9.2. $C_{18}H_{24}N_2O_2$ requires C, 71.9 ; H, 8.0 ; N, 9.3%); IR (KBr): 2920-2840, 1635, 745, 700 cm^{-1}; 70 eV EIMS: m/z 300 (M^+, 7.3%), 232 (M^+–C_4H_6N, 23.2%), 216 (M^++1–$C_{14}H_{17}NO$, 58.1%), 215 (M^+–$C_{14}H_{17}NO$, 34.3%), 194 (M^+–C_7H_6O, 17.9%), 188 (M^+–$C_5H_{10}NCO$, 55.8%), 138 (215–C_6H_5, 87.3%), 131 (215–$C_5H_{10}NO$, 70.7%), 112 ($C_5H_{10}N$–CO^+, 33.9%), 106 ($C_7H_6O^+$, 38.6%), 103 (215–$C_6H_{10}NO$, 30.8%), 86 ($C_4H_7NOH^+$, 26.3%), 84 ($C_5H_{10}N^+$, 100%), 77 ($C_6H_5^+$, 80.1%), 69 ($C_4H_6N^+$, 63.1%), 68 ($C_4H_6N^+$, 21.7%).

2R*S*(2R*, 3R*, 3a*S), 2,3,3a,4,5,6-hexahydro-3- phenyl-2-piperidinocarbonylpyrrolo [1,2-*b*] isoxazole, 5**. Yield 160 mg (22%), m.p. 145° (petrol-benzene 1:1), obtained from 2% ethyl acetate in benzene eluates (Found : C,71.7; H,8.1; N,9.3. $C_{18}H_{24}N_2O_2$ required C,71.9 ; H,8.0 ; N,9.3%); IR (KBr) : 2940-2850, 1645, 745, 710 cm^{-1}; 70 eV EIMS : m/z 301 (M^++1, 2.7%), 216 (M^++1–$C_{14}H_{17}NO$, 47.5%), 215 (M^+–$C_{14}H_{17}NO$, 38.3%), 188 (M^+–$C_6H_{10}NO$, 51.2%), 159 (M^+–$C_7H_{11}NO_2$, 100%), 142 ($C_7H_{12}NO_2$, 15.4%), 138 (215–C_6H_5, 33.4%), 131 (215–$C_5H_{10}N$, 90.2%), 112 ($C_6H_{10}NO^+$, 34.2%), 103 (131-CO, 63.7%), 91 (159–C_4H_7N, 76.4%), 86 (C_4H_7NO, 34.6%), 84 ($C_5H_{10}N^+$, 68.7%) 77 ($C_6H_5^+$, 39.1%), 69 ($C_4H_7N^+$, 87.0%); 70 eV CIMS : m/z 301 (M^++1, 100%), 216 (M^++1–$C_{14}H_{17}NO$, 40.8%), 188 (M^+–$C_6H_{10}NO$, 43.2%), 159 (M^+–$C_7H_{11}NO_2$, 61.5%), 142 ($C_7H_{12}NO_2$, 15.4 %), 91 (159–C_4H_7N, 10.2%), 69 ($C_4H_7H^+$, 40.2%); 25 eV EIMS: m/z 301 (M^++1, 6.3%), 216 (M^++1–$C_{14}H_{17}NO$, 8.0%), 215 (M^+–$C_{14}H_{17}NO$, 10.2%), 188 (M^+–$C_6H_{10}NO$, 60.6%), 159 (M^+–$C_7H_{11}NO_2$, 100%), 142 ($C_7H_{12}NO_2$, 10.3%).

2R*S*(2*R, 3*R**, 3a*S**)-2,3,3a,4,5,6-hexahydro-2-(4"-chlorophenyl)-3- piperidinocarbonylpyrrolo[1,2-*b*]isoxazole, 6.** Yield 200 mg (28%), gummy mass obtained from 2% ethyl acetate in benzene eluates (Found: C,64.5 ; H,6.8 ; N,8.3. $C_{18}H_{23}N_2O_2Cl$ requires C,64.5, ; H,6.8 ; N, 8.3%). IR (KBr) : 1645, 1100, 1150, 820 cm^{-1}.

2R*S*(2*R, 3*R**, 3a*S**) 2,3,3a,4,5,6-hexahydro-3-(4"-chlorophenyl)-2- piperidinocarborylpyrrolo [1,2-*b*] isoxazole 7**. Yield 115 mg (16%), Gummy mass obtained from 2% ethyl acetate in benzene eluates; (Found: C,64.5 ; H,6.8 : N, 8.3.

Table V—Distances (Å) for non-hydrogen atoms with *e.s.d.*'s given in parentheses

O1 - N6a	1.493(10)	C1'' - C2''	1.283(14)
O1 - C2	1.417(11)	C1'' - C6''	1.379(12)
N6a - C3a	1.493(11)	C2'' - C3''	1.388(15)
N6a - C6	1.480(13)	C3'' - C4''	1.395(15)
C3a - C3	1.521(13)	C4'' - C5''	1.378(16)
C3a - C4	1.542(14)	C5'' - C6''	1.385(14)
C3 -C1	1.527(12)	C4 - C5	1.554(17)
C3 - C2	1.570(11)	C5 - C6	1.549(16)
C1 - N1	1.335(11)	C2' - C3'	1.354(17)
C1 - O1	1.231(12)	C3' - C4'	1.509(18)
N1 - C2'	1.472(16)	C4' - C5'	1.537(20)
N1 - C6'	1.471(14)	C5' - C6'	1.408(17)

$C_{18}H_{23}N_2O_2Cl$ requires C, 64.5 ; H,6.8 ; H,8.3%}; IR (KBr): 1655, 1100, 1120, 860 cm^{-1}.

X-ray diffraction studies were carried out using Philips PW 110D automatic four circle diffractometer operating with Cu-K_α radiation (λ)= 1.5418Å monochromated by graphite.

Crystallographic data of 4' : Mol. formula $C_{18}H_{24}N_2O_2$, M_W = 300, triclinic, space group P_1 parameters : $Z = 2$, $a = 15.336(4)$, $b = 9.492(4)$, $c = 9.181(3)$, $\alpha = 115.8°(1)$, $\beta = 100.9°(1)$, $\gamma = 81.6°(1)$. The structure was solved by means of direct methods[13] and refined with isotropic, then anisotropic thermal factors, for the non-hydrogen atoms by full matrix least squares procedures. All hydrogen atoms were calculated at their theoretical places. Isotropic thermal factor was reined for the hydrogens. The final agreement factor $R = \Sigma|F_o|-|F_c|/\Sigma|F_o|$ converged to 0.079 and 0.074 for the weighted R_w factor = $\Sigma\ W_i|F_o|-|F_c|/\Sigma\ W_i|F_o|$. Positional parameters are given in Table III and the distance (Å) for non-hydrogen atoms and bond angles with estimated deviations, are given in the Tables V and VI.

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Table VI—Bond angles (deg.) for non-hydrogen with *e.s.d.*'s given in parentheses

N6a - 01 - C2	106.3 (6)	C3 - C2 - C1''	112.4 (7)
01 - N6a - C3a	99.3 (6)	C2 -C1'' - C2''	119.0 (8)
01 - N6a - C6	105.1 (7)	C2 - C1'' - C6''	122.8 (8)
C3a - N6a - C6	106.2 (7)	C'' - C1'' - C6''	118.2 (8)
N6a - C3a - C3	103.0 (7)	C1'' -C2''-C3''	121.6 (9)
N6a - C3a - C4	107.8 (8)	C2'' - C3'' - C4''	119.8(10)
C3 - C3a - C4	118.6 (8)	C3'' - C4'' - C5''	118.2 (10)
C3a - C3 - C1	113.8 (7)	C4'' - C5'' - C6''	121.5 (10)
C3a - C3 - C2	103.1 (7)	C1 - C6'' - C5''	120.6 (9)
C1 - C3 - C2	112.1 (7)	C3a - C4 - C5	104.8 (9)
C3 - C1 - N1	118.8 (8)	C4 - C5 - C6	105.9 (9)
C3 - C1 - 01	120.8 (8)	N6a - C6 - C5	108.2 (8)
N1 - C1 - 01	120.4 (8)	N1 - C2' - C3'	115.5(11)
C1 - N1 - C2''	125.4 (9)	C2' - C3' - C4'	116.9(12)
C1 - N1 - C6'	120.1 (9)	C3' - C4'- C5'	108.9(11)
C2 - N1 - C6'	113.9 (9)	C3' - C5' - C6'	113.7(11)
01 - C2 - C3	104.6 (7)	N1 - C6' - C5'	112.9(11)
01 - C2 - C1''	112.1 (7)		

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