Tetrahedron Vol. 48, No. 16, pp. 3335-3344, 1992
Printed in Great Britain

0040-4020/92 $5.00+.00
Pergamon Press Ltd

Cycloaddition of C,N-Diarylnitrones to 2-Butenolide : Synthesis of 2,3,6,6a-Tetrahydrofuro[3,4-d]isoxazol-4(3aH)-one

Avijit Banerji* and Sebanti Basu (nee Sinha)

Centre of Advanced Studies on Natural Products, Department of Chemistry,
Calcutta University, University College of Science,
92, Acharya Prafulla Chandra Road, Calcutta 700 009, India

(*Received in UK* 26 *February* 1992)

Abstract: *The cycloadditions of four C,N-diaryl nitrones (1-4) to the 5-membered conjugated lactone 2-butenolide were investigated. Two stereoisomeric 2-phenyl-3-aryl-2,3,6,6a-tetrahydrofuro[3,4-d]-isoxazol-4(3aH)-one cycloadducts and a ring-opened butanolide derivative were obtained in each case. The structure and stereochemistry of the products were determined by detailed NMR studies. The regiochemical course of the cycloadditions is explained, and the genesis of the ring-opened product rationalised.*

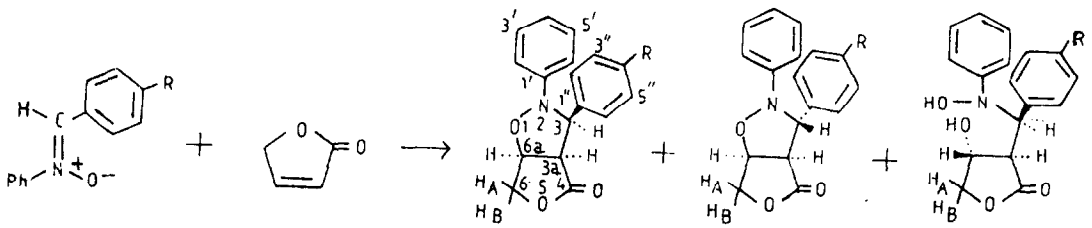
INTRODUCTION

The 1,3-dipolar cycloadditions of nitrones to different unsaturated systems have been the subject of extensive investigations[1,2,3] . A literature search revealed that only one report[4] existed on nitrone cycloadditions to conjugated lactones. This work was performed with δ-lactones derived from sugars. Hence we have undertaken a programme for the detailed study of cycloaddition of conjugated γ-lactones and δ-lactones with the objective of determining the regiochemical and stereochemical course of these reactions. We report here the results of our studies using the γ-lactone 2-butenolide as the substrate. A further objective in this instance was the synthesis of the 2,3,6,6a-tetrahydrofuro[3,4-d]isoxazol-4(3aH)-one ring system, to enrich the family of the comparatively rare bi-heterocycle furo[3,4-d]isoxazoles, the first member of which was synthesised by Quilico et al.[5]

RESULTS AND DISCUSSION

The four nitrones were the C-aryl-N-phenyl derivatives (**1-4**), where the para-substituents on the C-aryl group were varied from the strongly

electron-donating methoxyl to the strongly electron-withdrawing nitro group. The reactions were carried out in refluxing toluene (or in a sealed tube at 110°-115°) for 12 hours. After this period only a small amount of the nitrones survived. The post-reaction mixture yielded the diastereoisomeric 2-phenyl-3-aryl-2,3,6,6a-tetrahydrofuro[3,4-d]isoxazol-4(3aH)-ones, viz. (**6,9,12,14**) and (**7,10,13,15**) as the only cycloadducts. It was observed that the regiochemical course of the reaction could not be changed by varying the substituents on the C-aryl ring. In addition the ring-opened products (**8,11**) and (**16**) were also obtained.



1 : R = NO_2	5	6	7	8
2 : R = Cl		9	10	11
3 : R = OCH_3		12	13	-
4 : R = H		14	15	16

Scheme 1

The structures and relative configurations of the products were established on the basis of spectroscopical data, particularly NMR analysis. The latter included ^{1}H-NMR with decoupling experiments, ^{13}C-NMR and ^{1}H-^{13}C two-dimensional correlations by the XHCORR sequence (using delay parameters differently optimised to enhance one-bond and long-range couplings in separate experiments). The ^{1}H-NMR data are collected in Table 1 while the ^{13}C-NMR assignments are given in Table 2.

The IR spectra of all the bicyclic compounds exhibited γ-lactone bands at 1750-1775 cm^{-1}. ^{1}H-NMR decoupling studies for the eight cycloadducts established the following coupling informations :

$$H_3 - H_{3a} - \underline{H_{6a} - H_{A\text{-}6} - H_{B\text{-}6}}$$

The chemical shifts and the coupling characteristics of these protons decided in favour of the 2,3,6,6a-tetrahydrofuro[3,4-d]isoxazol-4(3aH)-one structure instead of the alternative 2,3,3a,4-tetrahydrofuro[4,3-d]-isoxazol-6(6aH)-one (**17**).

In the latter case, the most upfield proton H-3a would appear as a

Table 1 : 300 MHz ^{1}H-NMR of the Products

	6	7	8*	9	10	11*	12	13	14	15	16*
H-3	5.02(d)	4.94(d)	5.00(d)	4.80(d)	4.79(d)	4.89(d)	4.75(d)	4.73(d)	4.82(d)	4.80(d)	4.93(d)
H-3a	3.84(dd)	3.48(dd)	2.89(t)	3.68(t)	3.44(dd)	2.93(t)	3.66(t)	3.42(dd)	3.69(t)	3.44(dd)	2.96(t)
H-6a	5.23(m)	5.13(m)	4.46(m)	5.12(m)	5.10(m)	4.53(m)	5.15(m)	5.07(m)	5.15(m)	5.06(m)	4.54(m)
H_A-6	4.53	4.60	4.25	4.44	4.56	4.27	4.50	4.52	4.49	4.52	4.26
H_B-6	4.51	4.49	4.21	4.42	4.44	4.22	4.47	4.39	4.45	4.37	4.20
H-2',6'	6.96(d)	6.85(d)	6.58(d)	6.96(m)	6.85(d)	6.64(d)	6.96(m)	6.85(m)	6.96(m)	6.87(m)	6.70(m)
H-4'	7.02(d)	6.94(t)	6.73(t)		6.91(t)	6.75(t)					
H-3',5'	7.21(dd)	7.15(t)	7.05(t)	7.17(t)	7.13(t)	7.07(t)	7.16(t)	7.09(dd)	7.15(t)	7.10(t)	7.06(t)
H-2",6"	7.58(d)	7.63(d)	7.66(d)	7.29(d)	7.27(d)	7.23(d)	7.27(d)	7.29(d)	7.36(d)	7.40(d)	7.43(d)
H-3",5"	8.16(d)	8.17(d)	8.11(d)	7.36(d)	7.36(d)	7.40(d)	6.81(d)	6.79(d)	7.30(m)	7.26(t)	7.26(t)
H-4"	-	-	-	-	-	-	-	-	7.25(t)	7.22(t)	7.18(t)
Others	-	-	5.30-N-OH 3.82-C_{6a}-OH	-	-	5.15-N-OH 4.03-C_{6a}-OH	3.71-OCH_3	3.71-OCH_3	-	-	5.16-N-OH 4.13-C_6-OH
$J_{3,3a}$	9.0	2.9	5.4	8.9	3.0	4.9	9.0	3.0	8.8	2.8	4.0
$J_{3a,6a}$	7.6	6.5	4.8	7.5	6.4	4.7	8.2	6.4	7.5	6.1	4.8
J_{6a,H_A}	2.0	-	-	3.5	-	-	2.8	-	3.1	-	-
J_{6a,H_B}	6.2	4.6	2.8	5.3	4.6	2.7	7.1	4.6	6.3	4.2	3.0
J_{HA,H_B}	10.9	11.2	10.8	10.6	11.2	10.4	10.5	11.2	10.5	11.0	10.2
$J_{2',3'};J_{5',6'}$	8.6	7.7	8.1	ΣJ =	7.8	8.4	ΣJ =	8.8	ΣJ =	ΣJ =	ΣJ =
$J_{3',4'};J_{4',5'}$	7.5	7.4	7.3	15.8	7.4	7.2	15.7	7.3	15.8	14.5	17.0
$J_{2",3"};J_{5",6"}$	8.8	8.7	8.6	8.7	8.5	8.4	8.8	8.8	7.6	7.3	7.2
$J_{3",4"};J_{4",5"}$	-	-	-	-	-	-	-	-	7.1	7.0	7.3

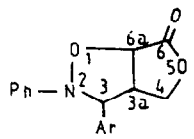
The magnitude of the coupling constants and the chemical shifts were calculated exactly by the aid of the PANIC programme (Parameter Adjustment in NMR by Iteration Calculation) available in Bruker software.

* Numbering of structures for ring-opened products follow those of cycloadducts.

Table 2 : 75.5 MHz ^{13}C-NMR Data of the Products

Str. No.	C-3	C-3a	C-4	C-6	C-6a	C-1'	C-4'	C-2', C-6'	C-3', C-5'	C-1"	C-4"	C-2" C-6"	C-3" C-5"	-OCH_3
6	71.44	54.52	173.50	71.28	77.99	147.86	124.52	117.26	129.21	142.76	148.02	128.82	123.84	-
7	71.28	57.39	175.19	70.88	77.25	147.26	123.84	116.81	128.95	146.06	147.89	128.08	124.27	-
8	54.88	49.87	174.60	74.97	70.39	145.05	120.38	115.86	129.24	148.68	147.52	128.18	123.91	-
9	71.55	54.27	172.94	71.48	77.62	148.14	124.19	117.54	128.83	133.87	134.29	129.15	128.95	-
10	71.27	57.57	175.56	71.16	77.00	147.77	123.46	116.85	128.78	137.62	134.24	128.49	129.25	-
11	54.77	50.16	174.81	74.95	70.79	145.62	120.28	116.15	128.93	138.94	133.56	128.49	129.15	-
12	71.76	54.13	173.27	71.64	77.24	148.22	123.89	117.66	128.79	126.85	159.48	128.69	113.98	55.00
13	71.44	57.38	174.90	71.01	76.79	147.89	122.94	116.60	128.48	130.85	159.31	128.16	114.20	55.10
14	72.18	54.30	172.95	71.45	77.42	148.35	123.88	117.51	128.56	135.18	128.41	127.66	128.76	-
15	71.77	57.43	175.67	70.92	76.90	147.99	122.97	116.51	128.51	139.05	128.06	126.90	128.86	-
16	54.47	50.51	174.94	74.93	71.10	146.02	120.19	116.25	128.84	140.49	127.81	127.05	129.16	-

Assignments were made for compounds (6,8,9,12,13,15) with the help of 1-bond CH - correlations by 2D-XHCORR spectra. Long-range 2D-CH-correlations were carried out for (8,15). Assignments of chemical shifts for C-1" to 6" rest on inter-comparisons and use of additivity parameters[6] (difference between calculated and experimental values were less than 0.5 ppm).



17

multiplet and the most downfield one H-6a as a doublet - this was contrary to observation. Hence the cycloadditions proceeded regioselectively to give only the 2,3,6,6a-tetrahydrofuroisoxazol-4-(3a<u>H</u>)-ones.

The relative configurations of the products were established from the magnitude of J_{3-3a}. The <u>cis</u>-isomers (**6,9,12,14**) showed a larger coupling constant of about 9 Hz between H-3/H-3a while the <u>trans</u>-isomers (**7,10,13, 15**) showed a smaller J_{3-3a} of about 3 Hz. This was in agreement with their stereochemical assignments. J_{3a-6a} was 7.5 to 8.2 Hz indicating the cis orientation of these protons. Interestingly the coupling constants for H_A-6/H-6a and H_B-6/H-6a showed slight but characteristic changes on going from the <u>cis</u>- to the <u>trans</u>-series, which indicated slight conformational changes.

The mass spectral fragmentations of the bicyclic cycloadducts were informative, with some characteristic differences for the two diastereoisomeric series. For example, both p-nitrophenyl derivatives (**6** and **7**) gave a strong M^+ with the base peak at m/z 91 ($C_6H_5N^+$). Common and significant fragments in both cases included those at m/z 226 ($M^+-C_4H_4O_3$), 179 (226-NO-OH), 104 ($226-C_6H_4NO_2$) and 77 ($C_6H_5^+$). Electron-impact induced cycloreversion was more important for (**7**) which showed a peak at m/z 242 (11%), while loss of the elements of water gave a significant peak at m/z 308 only for (**6**).

The molecular formulae of (**8**), $C_{17}H_{16}N_2O_6$, showed the addition of a molecule of water, compared to (**6**) and (**7**). It exhibited IR bands characteristic of hydroxyl groups (3480, 3390 cm^{-1}) and lactone carbonyl (1750 cm^{-1} KBr, 1768 cm^{-1} in $CHCl_3$). ^{1}H-NMR spectrum showed the presence of two exchangeable protons at δ 5.30 (-N-OH) and δ 3.82 (-C-OH). Structure elucidation of this compound as the ring-opened butanolide (**8**) followed from NMR analysis (Tables 1 and 2). The mass spectral fragmentation pattern of (**8**) further corroborated this view, the base peak appearing at m/z 226 [M^+ - { ($C_4H_5O_3$) + OH }] and significant peaks at M-OH (15%), m/z 180 ($226-NO_2$) and 92 (C_6H_5N, 20%). The structures of compounds (**11**) and (**16**) were established similarly.

It was established that the ring-opened product was derived from the <u>cis</u>-isomer. This transformation could be achieved by refluxing this <u>cis</u> isomer (**6**) in moist toluene for 12 hrs., when it was partially converted to (**8**). Under these conditions, the <u>trans</u>-product (**7**) remained unchanged.

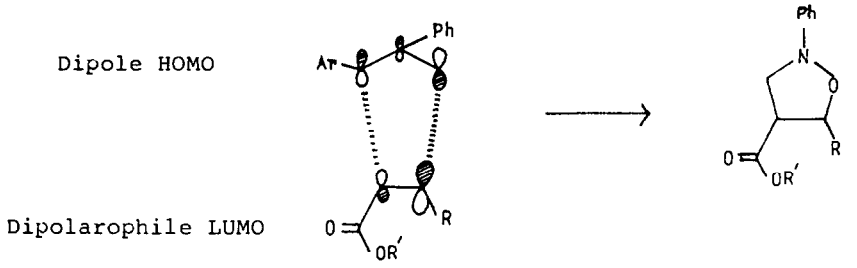
The comparative lability of cis-cycloadducts can be explained on the basis of greater steric interactions present in the molecule, the reaction presumably occurring by a S_N2 displacement at C-6a. The relative configuration at C-3 and C-3a of the ring-opened compounds were the same as the cis-isomers, while the expected inversion at C-6a was confirmed by the reduction in J_{3a-6a} values from ~7.5 Hz in case of the cycloadduct to ~4.8 Hz in case of the ring-opened products. The ring-opened products were found (in small amounts) even when the reactions were carried out with rigorously dried solvent in a sealed tube at 115°, as was evident on TLC of the crude reaction mixture. The ring cleavage under these conditions was initiated presumably by the attack of a second molecule of the nitrone at C-6a to give a compound which further reacted during work-up to give the butanolide derivative.

The experimental observations regarding regioselectivity of the process are in agreement with expectations from theory[7]. The frontier orbital energies and coefficients for some of the diaryl nitrones have been calculated earlier by Joucla et al.[8] The corresponding values of 2-butenolide could be taken to be similar to those of methyl crotonate, which had also been estimated by Joucla[8]. The present authors have utilised Joucla's values to calculate ΔE values for FMO interactions between methyl crotonate and the C-aryl-N-phenyl-nitrones. These values served as a good model for the cycloaddition between 2-butenolide and the corresponding nitrones. The distance of separation of 1.75 Å as assumed between both ends of the addends following Houk[7].

The square of the corrected coefficients $(C\Delta\beta)^2$ for C,N-diphenyl-nitrone are shown on the structure (Scheme 2). The relative magnitudes of the terminal corrected coefficients also hold for all the other nitrones. The difference in the HOMO-LUMO energies clearly indicated that for C,N-diphenylnitrone (**4**), N-phenyl-C-(p-methoxyphenyl) nitrone (**3**) and N-phenyl-C(p-chlorophenyl)nitrone (**2**) the dipole HOMO-dipolarophile LUMO interaction is the predominant one (Sustmann's Type I)[9]. Further, the product of the relevant orbital coefficients was significantly larger for this interaction than for the other FMO interaction. For N-phenyl-C-(p-nitrophenyl) nitrone (**1**) the HOMO-LUMO energy difference interactions are comparable (Sustmann's Type II)[9] : the nitrone HOMO-dipolarophile LUMO interaction would be, however, the determining one since the orbital coefficients of the HOMO dipolarophile at the reacting centres are virtually equal and the product of corrected orbital coefficients $(C\Delta\beta)^2$ in the HOMO-nitrone controlled interaction is much larger.

Thus in all the reactions the dipole HOMO-dipolarophile LUMO interaction would govern the regioselectivity of the process, the

qualitative situation being shown in Scheme 2. Moreover, since C-O bond formation is expected to be more advanced in the transition state than the C-C bond formation, the process would be even more regioselective than indicated by the approximate calculations on the basis of a symmetric T.S. The observed regioselectivity of the process can thus be explained.



Scheme 2

For the dipole HOMO-dipolarophile LUMO interaction the two possible geometries of approach are the diastereoisomeric transition states (exo-) (Fig.1) which give rise to the all - cis products (**6,9,12,14**), and (endo-) which would yield (**7,10,13,15**). The experimental results show that the total yield of all the cis-isomer and the ring-opened product derived from (exo-) approach was always greater in each case compared to the cycloadduct derived from (endo-) approach. The most probable reason is that in the C,N-diaryl nitrones in which the E-form predominates in the (exo-)transition state there would be favourable secondary interactions between the orbitals of the C-aryl ring of the nitrone and the oxygen of the carbonyl group of the unsaturated lactone.

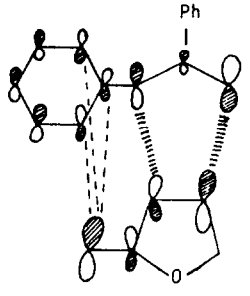


Fig. 1

EXPERIMENTAL

M.ps. were recorded on a Kofler block and are uncorrected. IR spectra were recorded with a Perkin Elmer 782 spectrometer, and mass spectra with a Jeol JMS D-300 mass spectrometer. ^{1}H-N.M.R. and ^{13}C-N.M.R. were recorded for solns. in $CDCl_3$ at 300 MHz and 75.5 MHz respectively on a Bruker AM-300L spectrometer (δ scale, TMS = 0 ppm). XHCORR spectra were recorded using the following pulse sequence suggested by Bax and Morris[10] :

^{1}H = Dec. off - 90° -DO- -DO- D3 - 90° - D4 - CPD Dec.

^{13}C = D1 - 180° - 90° - D4 - FID

with D1 = 2.0-2.5 sec., D3, D4 = 0.0037-0.0038 sec., 0.0018-0.002 sec. for 1-bond CH couplings; D3, D4 = 0.07-0.08 sec., 0.037-0.04 sec. for long-range couplings optimised for $J \approx 7$ Hz.

Analytical samples were routinely dried in vacuo at room temperature. Column and thin-layer chromatography were carried out using silica gel (BDH, 60-120 mesh) and silica gel G (BDH), respectively.

Nitrones 1-4 were prepared from the appropriate aldehydes and phenyl hydroxylamine according to the standard procedure[11,12] . 2-Butenolide was prepared by a literature method[13].

General method of cycloaddition : A soln. of 2-Butenolide (2.2 m mol) in anhydrous toluene (5 ml) was added at a time to a hot soln. of nitrone (2.2 m mol) in anhydrous toluene (15 ml) and refluxed under N_2 for 12 hr. The progress of the reaction was monitored by TLC. The curde post-reaction mixture was evaporated under reduced pressure and the residue was chromatographed to separate the products.

3RS-(3R, 3aR*, 6aS*) and 3RS-(3R*, 3aS*, 6aR*)-2,3,6,6a-Tetrahydro-2-phenyl-3-(p-nitrophenyl)-furo[3,4-d]isoxazol-4(3aH)-one (6,7) and 3RS-(3R*, 3aR*, 6aR*)-3-N-Phenyl-C-(p-nitrophenyl)-N-hydroxyaminomethyl-4-hydroxybutanolide (8).* From 1 and 5 : chromatography yielded 6 (190 mg, 27%) m.p. 177° ($CHCl_3$), Rf = 0.39 (Benzene/AcOEt = 4/1) in the Petrol-Benzene (1/3) eluate; 7 (160 mg, 22%) m.p. 170° (Petrol-AcOEt), Rf = 0.47 (Benzene/AcOEt = 4/1) in the Petrol-Benzene (1/3) eluate and 8 (100 mg, 13%) m.p. 110°-112° ($CHCl_3$), Rf = 0.25 (Benzene/AcOEt = 4/1) in the Benzene-AcOEt (9/1) eluate. 6, IR (KBr) 1770 (s, lactone C=O), 1520, 1350 (s, aromatic nitro group), 840, 735 cm^{-1} (substituted phenyl). MS (m/z) : 326 (M^+, 42%), 308 (M^+-H_2O, 2.5%), 226 ($M^+-C_4H_4O_3$, 5.3%), 204 ($M^+-C_6H_4NO_2$, 3.5%), 179 (226-NO-OH, 5.7%), 128 ($308-C_6H_4NO_2-CO-CHO$, 2.8%), 104 ($226-C_6H_4NO_2$, 4.7%), 91 ($C_6H_5N^+$, 100%), 77

($C_6H_5^+$, 77%). (Found : C, 62.4; H, 4.2; N, 8.3; $C_{17}H_{14}N_2O_5$ requires C, 62.6; H, 4.3; N, 8.6%). **7**, IR (KBr) 1750 (s, lactone C=0), 1515, 1335 (s, aromatic nitro group), 837, 735 cm^{-1} (substituted phenyl). MS (m/z) : 326 (M^+, 12%), 242 ($M^+-C_4H_4O_2$, 11%), 226 ($M^+-C_4H_4O_3$, 4.6%), 195 (242-NO-OH, 2.0%), 92 (C_6H_6N, 8.3%), 91 ($C_6H_5N^+$, 100%), 77 ($C_6H_5^+$, 17%). (Found : C, 62.3; H, 4.2; N, 8.3; $C_{17}H_{14}N_2O_5$ requires C, 62.6; H, 4.3; N, 8.6%). **8**, IR (KBr) 3480, 3390 (m, OH), 1750 (s, lactone C=0), 1515, 1350 (s, aromatic nitro group), 860, 750 cm^{-1} (substituted phenyl); ($CHCl_3$) 3360 (m, -OH), 1768 cm^{-1} (s, lactone C=0). MS (m/z); M^+ peak is absent, 327 (M-OH, 15%), 309 (M^+-H_2O-OH, 1.5%), 226 ($M^+-OH-C_4H_5O_3$, 100%), 180 (226-NO_2, 29%), 192 (C_6H_6N, 20%), 77 ($C_6H_5^+$, 52%). (Found : C, 58.9; H, 4.4; N, 7.8; $C_{17}H_{16}N_2O_6$ requires C, 59.3; H, 4.7; N, 8.1%).

3RS-(3R, 3aR*, 6aS*) and 3RS-(3R*, 3aS*, 6aR*)-2,3,6,6a-Tetrahydro-2-phenyl-3-(p-chlorophenyl)-furo[3,4-d]isoxazol-4(3aH)-one (9,10) and 3RS-(3R*, 3aR*, 6aR*)-3-N-phenyl-C-(p-chlorophenyl)-N-hydroxyaminomethyl-4-hydroxybutanolide (11).* From **2** and **5** : compound **9** (160 mg, 23%) syrup, Rf = 0.44 (Benzene/AcOEt = 4/1) was obtained from the Petrol-Benzene (1/3) eluates. Compound **10** (125 mg, 20%) obtained as a viscous liquid, Rf = 0.62 (Benzene/AcOEt = 4/1) was eluted by Petrol-Benzene (1/1). **11** (100 mg, 18%) m.p. 165°-167° (Benzene), Rf = 0.38 (Benzene/AcOEt = 4/1) was obtained from Benzene-AcOEt (9/1) eluates. **9**, IR (thin liquid film) 1775 (s, lactone C=0), 830, 740 cm^{-1} (substituted phenyl). MS (m/z); 315 (M^+). (Found : C, 64.5; H, 4.2; N, 4.2; $C_{17}H_{14}NO_3Cl$ requires C, 64.7; H, 4.4; N, 4.4%). **10**, IR (thin liquid film) 1770 (s, lactone C=0), 830, 755 cm^{-1} (substituted phenyl). MS (m/z) : 315 (M^+). (Found : C, 64.5; H, 4.3; N, 4.2; $C_{17}H_{14}NO_3Cl$ requires C, 64.7; H, 4.4; N, 4.4%). **11**, IR (KBr) 3390 (m, -OH), 1760 (s, lactone C=0), 835, 750 cm^{-1} (substituted phenyl); ($CHCl_3$) 3360 (w, -OH), 1763 cm^{-1} (m, lactone C=0). (Found : C, 61.6; H, 4.6; N, 4.0; $C_{17}H_{16}NO_4Cl$ requires C, 61.2; H, 4.8; N, 4.2%).

3RS-(3R, 3aR*, 6aS*) and 3RS-(3R*, 3aS*, 6aR*)-2,3,6,6a-Tetrahydro-2-phenyl-3-(p-methoxyphenyl)-furo[3,4-d]isoxazol-4(3aH)-one (12,13).* From **3** and **5**, compound **12** (230 mg, 22%) was obtained as a thick syrup, Rf = 0.5 (Benzene/AcOEt = 4/1) from the Petrol-Benzene (1/3) eluates. **13** (225 mg, 33%) a syrupy mass, Rf = 0.54 (Benzene/AcOEt = 4/1) was isolated after performing preparative TLC (Benzene/AcOEt, 17/3) of the same eluates. **12**, IR (thin liquid film) 1775 (s, lactone C=0), 830, 750 cm^{-1} (substituted phenyl). MS (m/z); 311 (M^+).(Found : C, 69.1; H, 5.3; N, 4.2; $C_{18}H_{17}NO_4$ requires C, 69.4; H, 5.5; N, 4.5%). **13**, IR (thin liquid film) 1770 (s, lactone C=0), 835, 755 cm^{-1} (substituted benzene moiety). MS (m/z); 311 (M^+). (Found : C, 69.2; H,

5.2; N, 4.3; $C_{18}H_{17}NO_4$ requires C, 69.4; H, 5.5; N, 4.5%).

3RS-(3R, 3aR*, 6aS*) and 3RS-(3R*, 3aS*, 6aR*)-2,3,6,6a-Tetrahydro-2,3-diphenylfuro-[3,4-d]isoxazol-4(3aH)-one (14,15) and 3RS-(3R*, 3aR*, 6aR*)-3,N,C-Diphenyl-N-hydroxy* aminomethyl-4-hydroxybutanolide (16). From **4** and **5** : chromatography yielded three products **14** (160 mg, 25%) m.p. 177° (Petrol-AcOEt), Rf = 0.50 (Benzene/AcOEt = 4/1) from Benzene eluates, **15** (133 mg, 21%) m.p. 133° (Petrol-AcOEt), Rf = 0.59 (Benzene/AcOEt = 4/1) from Petrol-Benzene (1/3) eluates and **16** (90 mg, 13%) m.p. 183°-185° ($CHCl_3$), Rf = 0.37 (Benzene/ AcOEt = 4/1) from Benzene-AcOEt (9/1) eluates respectively. **14**, IR (KBr) 1770 (s, lactone C=0) 750, 690 cm^{-1} (substituted phenyl) MS (m/z) : 281 (M^+). (Found : C, 72.4; H, 5.3; N, 4.8; $C_{17}H_{15}NO_3$ requires C, 72.6; H, 5.4; N, 5.0%). **15**, IR (KBr) 1760 (s, lactone C=0), 758, 698 cm^{-1} (substituted phenyl). MS (m/z); 281 (M^+). (Found : C, 72.4; H, 5.3; N, 4.7; $C_{17}H_{15}NO_3$ requires C, 72.6; H, 5.4; N, 5.0%). **16**, IR (KBr) 3490, 3425 (m, -OH), 1737 (s, lactone C=0), 749, 699 cm^{-1} (substituted phenyl); ($CHCl_3$) 3360 (m, -OH), 1765 cm^{-1} (s, lactone C=0). (Found : C, 67.9; H, 5.6; N, 4.4; $C_{17}H_{17}NO_4$ requires C, 68.2; H, 5.7; N, 5.7%).

Acknowledgement : The authors thank R.S.I.C. (C.D.R.I.), Lucknow, India for mass spectral analysis. Thanks are also due to the U.G.C. for funding this research work.

REFERENCES

1. Black, D.St.C.; Crozier, R.F.; Davis, V.C.; Synthesis, **1975**, 4, 205.
2. Banerji, A.; Sahu, A.; J. Scient. and Indus. Research, **1986**, 45, 355.
3. Torssell, K.B.G. in "Nitrile oxides, Nitrones and Nitronates in Organic Synthesis", VCH, New York, Weinheim, **1988**.
4. Panfil, I.; Chmielenski, M.; Tetrahedron, **1985**, 41, 4713.
5. Quilico, A.; Stagno D'Alcontres, G.; Grünanger, P.; Gazz. Chim. Ital., **1950**, 80, 479.
6. Ewing, D.E.; Organic Magnetic Resonance, **1977**, 12(9), 499.
7. Houk, K.N.; Sims, J.; Duke, R.R. Jr.; Strozier, R.W.; George, J.K.; J. Am. Chem. Soc., **1973**, 95, 7287; Houk, K.N.; Sims, J.; Watts, C.R.; Luskus, L.J.; ibid, **1973**, 95, 7301.
8. Joucla, M.; Tonnard, F.; Gree, D.; Hamelin, J.; J. Chem. Res., **1978**, (S), 240; (M), 2901.
9. Sustamann, R.; Tetrahedron lett., **1971**, 2717.
10. Bax, A.; Morris, G.; Jour. Magnetic Ressonance., **1981**, 42, 501.
11. Vogel, A.I.; in "A Text Book of Practical Organic Chemistry", 4th Edn., Longman, 722, **1978**.
12. Wheeler, O.H.; Gore, P.H.: J. Am. Chem. Soc., **1956**, 78, 3363.
13. Price, C.C.; Judge, J.M.; in "Organic Synthesis Collective Volume 5", John Wiley and Sons, 255, **1973**.