Chemistry of 5(2*H*)-Isoxazolones: Novel Conversion of Positional Isomers [1]

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Reactions of 4-arylidene-3-methyl-5(4*H*)-isoxazolones with nucleophiles lead to 4-substituted-benzyl-3-methyl-5(2*H*)-isoxazolones. Formation of 5(4*H*)- and 5(2*H*)-isoxazolone derivatives have been observed when 4-substituted-arylidene-3-methyl-5(4*H*)-isoxazolones are reacted with methyl magnesium iodide. Oxidation of 4-substituted-benzyl-3-methyl-5(2*H*)-isoxazolones with manganic acetate and pyridinium chlorochromate, gives 4,4'-bis[4-substituted-benzyl-3-methyl-5(4*H*)-isoxazolone]. Oxidation of 4-substituted-benzyl-3-methyl-5(2*H*)-isoxazolones with metachloroperbenzoic acid gives 4-hydroxy-4-substituted-benzyl-3-methyl-5(4*H*)-isoxazolones and reactions of the same substrates with *N*-bromosuccinimide furnish 4-bromo-4-substituted-benzyl-3-methyl-5(4*H*)isoxazolones.

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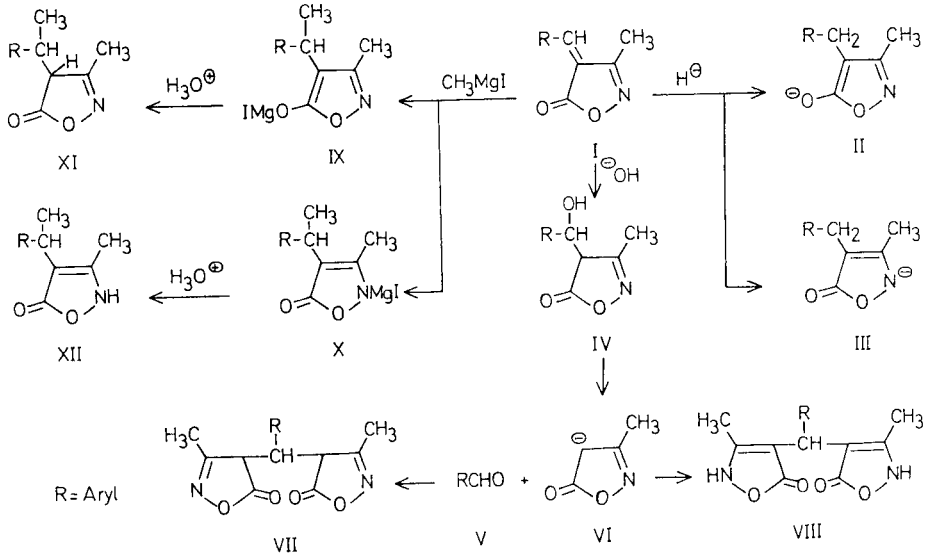
The chemistry of 5-isoxazolones, extensively studied by earlier workers [2-5], does not permit understanding of the structural assignments of the reaction products because the existance of various tautomers in this class of compounds has been reported by Katritzky only in 1961 [6]. After the sixties, reactions of 5(4*H*)-isoxazolones have been studied [7-16], but the chemistry of 5(2*H*)-isoxazolones has remained unexplored. This is presumably because of the nonavailability of a facile synthesis for obtaining substituted 5(2*H*)-isoxazolones. In principle, appropriately substituted 5(4*H*)-isoxazolones can become substrates for conversion to 5(2*H*)-isoxazolones but such an attempt has not been made by earlier workers. The chemistry of 5(2*H*)-isoxazolones is expected to be interesting since these are activated cyclic enamines. The present communication, therefore, reports the preparation and chemistry of 5(2*H*)-isoxazolones.

On the basis of reaction mechanism (Scheme 1), 4-substituted-benzylidine-3-methyl-5(4*H*)-isoxazolones **I** can serve as starting materials for the preparation of 4-substituted-benzyl-3-methyl-5(2*H*)-isoxazolones **III** since a hydride attack may lead to two possible tautomers **II** and **III** and of these **III** is likely to stablize. In order to ascertain the feasibility of this approach, the required 4-substituted-benzylidene-3-methyl-5(4*H*)-isoxazolones **1-7** (Scheme 2) have been prepared by reacting appropriate aldoximes with ethyl acetoacetate in the presence of 3*N* hydrochloric acid. This method essentially represents the synthetic strategy reported earlier [2] and of the seven compounds prepared, compounds **1-4** have been reported by earlier workers [8]. As would be expected, sodium borohydride reductions of **1-7** have indeed furnished the corresponding 5(2*H*)-isoxazolones **8-14** in excellent yields. This observation is significant because a recent report [16] indicates that 4-alkylidene-5(4*H*)-isoxazolones undergo reduction with sodium borohydride to yield 5(4*H*)-isoxazolones and

Scheme 1



Scheme 2

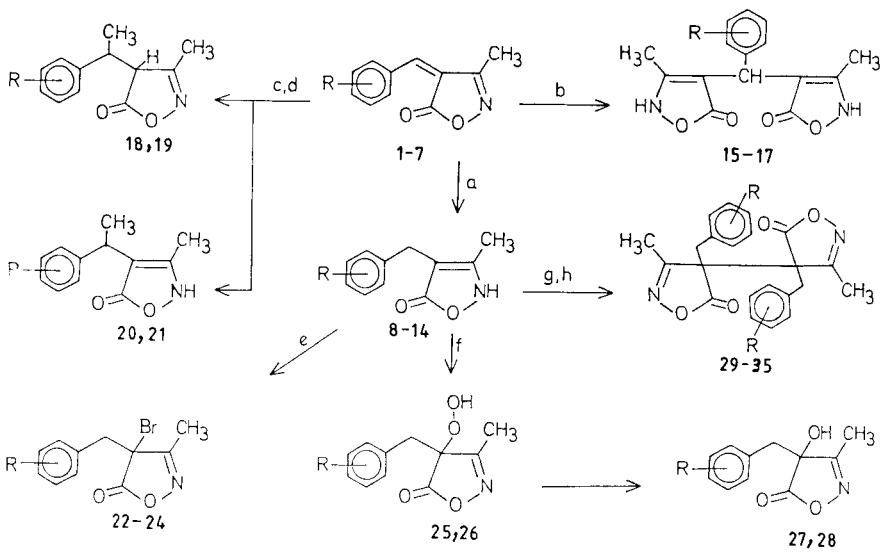


Table 1

Physical and Analytical Data

Compound No.	R	Yield (%)	mp °C (°C)	Formula	Analysis Calcd/Found C	H	N
5	3,4-$(OCH_3)_2$	67	162	$C_{13}H_{13}NO_4$	63.2 63.1	5.3 5.2	5.7 5.7
6	2,5-$(OCH_3)_2$	60	143	$C_{13}H_{13}NO_4$	63.2 63.5	5.3 5.3	5.7 5.9
7	3,4-$(Cl)_2$	75	167	$C_{11}H_7Cl_2NO_2$	51.6 51.9	2.7 3.0	5.5 5.5
8	H	86	109	$C_{11}H_{11}NO_2$	69.7 69.4	5.8 6.0	7.4 7.2
9	4-CH_3	90	116	$C_{12}H_{13}NO_2$	70.9 70.5	6.4 6.5	6.9 6.5
10	3,4-(OCH_2O)	89	108	$C_{12}H_{11}NO_4$	61.8 61.7	4.8 4.8	6.0 5.6
11	4-OCH_3	86	122	$C_{12}H_{13}NO_3$	65.6 65.8	5.9 5.8	6.3 6.4
12	3,4-$(OCH_3)_2$	90	107	$C_{13}H_{15}NO_4$	62.6 62.4	6.0 6.1	5.6 5.2
13	2,5-$(OCH_3)_2$	82	86	$C_{13}H_{15}NO_4$	62.6 62.4	6.0 6.1	5.6 5.3
14	3,4-$(Cl)_2$	86	109	$C_{11}H_9Cl_2NO_2$	51.2 51.6	3.5 3.7	5.4 5.4
15	H	94	150	$C_{15}H_{14}N_2O_4$	62.9 62.8	4.8 4.9	9.7 9.3
16	4-CH_3	92	98	$C_{16}H_{16}N_2O_4$	64.0 64.3	5.3 5.4	9.3 9.6
17	3,4-(OCH_2O)	92	128	$C_{16}H_{14}N_2O_6$	58.2 58.6	4.2 4.2	8.5 8.8
18,20	H	57	107	$C_{13}H_{15}NO_2$	70.9 71.0	6.4 6.4	6.9 6.8

Table 1 (continued)

Compound No.	R	Yield (%)	mp °C (°C)	Formula	Analysis Calcd/Found C	H	N
19,21	4-CH_3	53	Oil	$C_{13}H_{15}NO_2$	71.9 72.0	6.9 6.9	6.4 6.4
22	H	83	74	$C_{11}H_{10}BrNO_2$	49.2 49.6	3.7 3.7	5.2 5.2
23	4-CH_3	81	Oil	$C_{12}H_{12}BrNO_2$	51.1 51.0	4.2 4.3	4.9 5.0
24	3,4-(OCH_2O)	84	98	$C_{12}H_{10}BrNO_4$	46.2 46.2	3.2 3.4	4.5 4.6
27	H	39	Oil	$C_{11}H_{11}NO_3$	64.4 64.3	5.4 5.6	6.8 6.8
28	4-CH_3	38	Oil	$C_{12}H_{13}NO_3$	65.7 65.9	5.9 6.0	6.6 6.7
29	H	76	122	$C_{22}H_{20}N_2O_4$	70.2 70.4	5.3 5.4	7.4 7.2
30	4-CH_3	70	132	$C_{24}H_{24}N_2O_4$	71.3 71.2	5.9 6.2	6.9 6.8
31	3,4-(OCH_2O)	66	151	$C_{22}H_{20}N_2O_8$	62.0 61.8	4.3 4.4	6.0 6.0
32	4-OCH_3	74	127	$C_{24}H_{24}N_2O_6$	66.1 66.4	5.5 5.8	6.3 6.2
33	3,4-(OCH_3)$_2$	68	134	$C_{26}H_{28}N_2O_8$	62.9 63.2	5.6 5.6	5.6 5.8
34	2,5-(OCH_3)$_2$	66	145	$C_{26}H_{28}N_2O_8$	62.9 63.1	5.6 5.5	5.6 5.7
35	3,4-(Cl)$_2$	60	142	$C_{22}H_{16}Cl_2N_2O_4$	51.4 51.7	3.1 3.5	5.4 5.4

Table 2

Spectral Data

Compound No.	MS	UV λ max	IR (C=O) cm^{-1}	^1H NMR δ ppm
5	247		1725	(deuteriochloroform): 2.18 (s, 3-CH_3, 3H), 3.90 (s, Ar 3,4-OCH_3, 6H), 6.76-7.66 (m, Ar, 3H), 8.58, 8.60 (2s, 4-=CH, 1H)
6	247		1716	(deuteriochloroform and hexadeuteriodimethyl sulfoxide): 2.19 (s, 3-CH_3, 3H), 3.18 (s, Ar 2,5-OCH_3, 6H), 6.65-7.39 (m, Ar, 4H), 8.70, 8.80 (2s, 4-=CH, 1H)
7	256		1715	(deuteriochloroform and hexadeuteriodimethyl sulfoxide): 2.25 (s, 3-CH_3, 3H), 7.4-8.10 (s, Ar, 3H), 8.08, 8.10 (2s, 4-=CH, 1H)
8	189	260	1675	(deuteriochloroform): 1.90 (s, 3-CH_3, 3H), 3.40 (s, 4-CH_2, 2H), 6.98-7.23 (br m, Ar, 5H)
9	203	258	1680	(deuteriochloroform): 1.98 (s, 3-CH_3, 3H), 2.22 (s, Ar 4-CH_3, 3H), 3.40 (s, 4-CH_2, 2H), 6.91-7.12 (br m, Ar, 4H)
10	233		1680	(deuteriochloroform): 1.96 (s, 3-CH_3, 3H), 3.31 (s, 4-CH_2, 2H), 5.8 (s, Ar 3,4-OCH_2O, 2H), 6.49-6.67 (br m, Ar, 3H)
11	219		1670	(deuteriochloroform): 1.88 (s, 3-CH_3, 3H), 3.30 (s, 4-CH_2, 2H), 3.62 (s, Ar 4-OCH_3, 3H), 6.90-7.20 (br m, Ar, 4H)
12	249		1680	(deuteriochloroform): 2.00 (s, 3-CH_3, 3H), 3.38 (s, 4-CH_2, 2H), 3.62, 3.70 (2s, Ar 2,5-OCH_3, 6H), 6.58-6.78 (br m, Ar, 3H)
13	249		1660	(deuteriochloroform): 2.00 (s, 3-CH_3, 3H), 3.38 (s, 4-CH_2, 2H), 3.62, 3.70 (2s, Ar 2,5-OCH_3, 6H), 6.58-6.78 (br m, Ar, 3H)

Table 2 (continued)

Compound No.	MS	UV λ max	IR (C=O) cm^{-1}	^1H NMR δ ppm
14	258		1660	(deuteriochloroform): 2.00 (s, 3-CH_3, 3H), 3.34 (s, 4-CH_2, 2H), 6.94-7.19 (br m, Ar, 3H)
15		260	1660, 1690	(deuteriochloroform and hexadeuteriodimethyl sulfoxide); 1.88 (s, 2 x 3-CH_3, 6H), 4.78 (s, 1-CH, 1H), 7.17 (s, Ar, 5H)
16			1660, 1690	(deuteriochloroform and hexadeuteriodimethyl sulfoxide): 1.90 (s, 2 x 3-CH_3, 6H), 2.50 (s, Ar 4-CH_3, 3H), 4.75 (s, 1-CH, 1H), 7.02 (s, Ar, 4H)
17			1660,1675	(deuteriochloroform and hexadeuteriodimethyl sulfoxide): 1.91 (s, 2 x 3-CH_3, 6H), 4.68 (s, 1-CH, 1H), 6.52 (s, Ar, 3H)
18, 20 [a]	203	254	1640-1670 (broad)	(deuteriochloroform): 1.40, 1.49 (d, ethyl-CH_3, 3H, J = 8 Hz), 1.51, 1.60 (d, ethyl-CH_3, 3H, J = 8 Hz), 1.79, 1.89 (d, 2 x 3-CH_3, 6H), 3.29-3.60 (br m, 4-H, 1H), 3.72-3.8 (d, 2 x 2-CH, 2H, J = 9 Hz), 7.2 (s, Ar, 10H)
19, 21 [a]	217	257	1660-1690 (broad)	(deuteriochloroform): 1.19, 1.28 (d, ethyl-CH_3, 3H, J = 8 Hz), 1.39, 1.48 (d, ethyl-CH_3, 3H, J = 8 Hz), 2.15 (s, 2 x Ar 4-CH_3, 6H), 2.60, 2.69 (d, 2 x 2-CH, 2H, J = 9 Hz), 7.15 (m, Ar, 8H)
22 [b]		232	1785	(deuteriochloroform): 2.30 (s, 3-CH_3, 3H), 3.19, 3.33 (d, 4-CH_2, 1H, J = 14 Hz), 3.47, 3.61 (d, 4-CH_2, 1H, J = 14 Hz), 6.92-7.28 (m, Ar, 5H)
23			1785 (Neat)	(carbon tetrachloride): 2.1 (s, 3-CH_3, 3H), 2.20 (s, Ar 4-CH_3, 3H), 3.14, 3.28 (d, 4-CH_2, 1H, J = 14 Hz), 3.42, 3.56 (d, 4-CH_2, 1H, J = 14 Hz), 6.80-7.08 (m, Ar, 5H)
24			1780	(carbon tetrachloride): 2.14 (s, 3-CH_3, 3H), 3.08, 3.22 (d, 4-CH_2, 1H, J = 14 Hz), 3.36, 3.52 (d, 4-CH_2, 1H, J = 14 Hz), 6.48-6.7 (m, Ar, 4H)
27	205	284	1785 (Neat)	(deuteriochloroform): 2.01 (s, 3-CH_3, 3H), 3.09 (s, 4-CH_2, 2H), 3.58 (s, 4-OH, 1H), 6.9-7.36 (m, Ar, 5H)
28	219		1785 (Neat)	(deuteriochloroform): 2.04 (s, 3-CH_3, 3H), 2.25 (s, Ar 4-CH_3, 3H), 3.09 (s, 4-CH_2, 2H), 3.57 (s, 4-OH, 1H), 6.96-7.2 (m, Ar, 4H)
29	376		1740, 1775	(deuteriochloroform): 1.96, 2.04 (2s, 2 x 3-CH_3, 6H), 3.52, 3.59 (2s, 2 x 4-CH_2, 4H), 7.08-7.38 (m, Ar, 10H)
30	404		1740, 1780	(deuteriochloroform): 1.96, 2.00 (2s, 2 x 3-CH_3, 6H), 2.23 (s, 2 x Ar 4-CH_3, 6H), 3.45, 3.52 (2s, 2 x 4-CH_2, 4H), 6.82-7.12 (m, Ar, 8H)
31	464		1720, 1775	(deuteriochloroform): 1.85, 2.06 (2s, 2 x 3-CH_3, 6H), 3.41, 3.49 (2s, 2 x 4-CH_2, 4H), 5.81, 5.85 (2s, 2 x Ar 3,4-OCH_2O, 4H), 6.47-6.73 (m, Ar, 8H)
32	436		1720, 1775	(deuteriochloroform): 1.99, 2.04 (2s, 2 x 3-CH_3, 6H), 3.49, 3.70 (2s, 2 x 4-CH_2, 4H), 3.70 (s, 2 x Ar 4-OCH_3, 6H), 6.67, 7.14 (m, Ar, 8H)
33	496		1740, 1790	(deuteriochloroform); 1.96, 2.01 (2s, 2 x 3-CH_3, 6H), 3.42, 3.50 (2s, 2 x 4-CH_2, 4H), 3.68 (s, 4 x Ar 3,4-OCH_3, 12H), 6.59-7.10 (m, Ar, 6H)
34	496		1740, 1790	(deuteriochloroform): 1.88, 2.04 (2s, 2 x 3-CH_3, 6H), 3.46 (s, 2 x 4-CH_2, 4H), 3.65, 3.68 (2s, 4 x Ar 2,5-OCH_3, 12H), 6.57-6.82 (m, Ar, 6H)
35	514		1740, 1770	(deuteriochloroform): 1.92, 2.04 (2s, 2 x 3-CH_3, 6H), 3.46 (s, 2 x 4-CH_2, 4H), 6.77-7.39 (m, Ar, 6H)

[a] Spectroscopy data of the mixture have been given. [b] Also see reference number [14].

not 5(2*H*)-isoxazolones. The spectroscopic data of **8-14** supported their assigned structures. Unlike hydride attack, the attack of hydroxyl ions on the arylidene carbon in 5(4*H*)-isoxazolone **I** may lead to **IV**. This intermediate **IV** can split into aldehyde **V** and 5(4*H*)-isoxazolone **VI** and their recombination [2] can furnish **VII**. In the present study it has been observed that even a mild alkali like sodium bicarbonate is able to split the intermediate (**IV**) and the recombination of the two components **V** and **VI** yields **VIII**. Formation of **VIII**, compounds **15-17**, (Scheme 2) is also supported by its spectral data.

Similar to the site of hydride attack on **I**, reaction of methyl magnesium iodide with **I** may lead to **IX** and/or **X** since both the tautomers **IX** and **X** can stabilize in Grignard reaction. In the present study the reactions of 4-substituted-benzylidene-3-methyl-5(4*H*)-isoxazolones **1-2** with methyl magnesium iodide have furnished mixtures of 5(4*H*)- and 5(2*H*)-isoxazolone derivatives **18-21**. The formation of the two positional isomers is evident from their uv and ^1H nmr spectra. No attempt has been made to separate these isomers.

The chemistry of 5(2*H*)-isoxazolones is attractive since

carbon 4 in these compounds represents the β-carbon atom of cyclic enamines and is electron rich. Free radical reactions and oxidation reactions involving this centre are expected to yield interesting compounds. Electron push from the enamine nitrogen enables 5(2*H*-)isoxazolone to revert to 5(4*H*)-isoxazolone provided C-4 is appropriately functionalized. This envisaged pathway has been studied by reacting 4-substituted-benzyl-3-methyl-5(2*H*)-isoxazolones **8-10** with *N*-bromosuccinimide. The resultant 4-bromo-4-substituted-benzyl-3-methyl-5(4*H*)-isoxazolones **22-24** have been obtained in good yields. The ir and ^{1}H nmr spectra of **22-24** supported the assigned structures. In the light of this observation, the reactions of **8** and **9** with *meta*-chloroperbenzoic acid have been studied. In principle, either the hydroperoxides **25** and **26** or their corresponding decomposition products **27** and **28**, would be formed in these reactions. The structures of the isolated products have indicated them to be hydroxy compounds **27-28**. Oxidations of **8-14** with pyridinium chlorochromate (PCC) have furnished 4,4′-bis[substituted-benzyl-3-methyl-5(4*H*)-isoxazolones] **29-35**. Oxidations of **8-14** with manganic acetate have also furnished similar dimerised products **29-35**.

It can be concluded from the present study that 4-substituted-benzylidene-3-methyl-5(4*H*)-isoxazolones can be easily converted to 5(2*H*)-isoxazolone derivatives during their reactions with sodium borohydride and aqueous sodium bicarbonate. Unlike these reactions, Grignard reactions of 5(4*H*)-isoxazolones have furnished mixture of 5(4*H*)- and 5(2*H*)-isoxazolone derivatives. Oxidations of 5(2*H*)-isoxazolones have been found to yield dimers of 5(4*H*)-isoxazolones.

EXPERIMENTAL

Melting points have been determined on a hot stage apparatus and are uncorrected. The ir spectra were recorded on Beckman Acculab-1 grating instruments. Unless stated otherwise, all spectra have been recorded in potassium bromide. Methanolic solutions have been used to record uv spectra on Perkin Elmer Lambda-15. The ^{1}H nmr spectra were recorded on Perkin-Elmer R-32 using TMS as internal reference. The ms spectra have been obtained from Jeol-D-300 instrument. The physico-chemical and spectral data of **5-24** and **27-35** are reported in Tables 1 and 2.

4-Substituted-benzylidene-3-methyl-5(2*H*)-isoxazolones **5-7**.

To a solution of the required aldoxime (1 g, 0.008 mole) in methanol-water (1:1), was added ethyl acetoacetate (2.1 ml, 0.016 mole) and 3 *N* hydrochloric acid (3 ml). This reaction mixture was stirred at 30° for 24 hours. The separated solid was filtered, washed with water and crystallized from chloroform-hexane mixture.

4-Substituted-benzyl-3-methyl-5(2*H*)-isoxazolones **8-14**.

To the required solution of **1-7** (1 g, 0.005 mole) in methanol (10 ml) was added sodium borohydride (0.5 g, 0.01 mole) at 0-5° under stirring. The reaction mixture was stirred for 3.5 hours at room temperature (30°) and was then made acidic with 30% hydrochloric acid to *p*H 2. This was then extracted with ethyl acetate, removal of the solvent and crystallization of the residue with chloroform-hexane mixture furnished the desired compounds.

1,1-Bis[3-methyl-5-oxo-(2*H*)-isoxazol-4-yl]-1-substituted-phenylmethanes **15-17**.

A suspension of compounds **1-3** (1 g, 0.005 mole) in aqueous sodium bicarbonate (5%, 14 ml) was heated at 70° for one hour. During this period an oil separated out. On cooling this reaction mixture was extracted with ether and the aqueous layer was made acidic with hydrochloric acid (20%). The precipitated material was further triturated with this acid and the separated solid was crystallized from aqueous methanol.

4-(1-Substituted-phenyl)ethyl-3-methyl-5(2*H*)- and 5(4*H*)-isoxazolones **18-21**.

To a solution of magnesium (0.5 g), methyl iodide (4.0 ml) in dry ether (50 ml) was added **1** or **2** (1 g, 0.005 mole) in dry ether (30 ml). The reaction mixture was refluxed for 2 hours. This was followed by the removal of the solvent and the residue was decomposed with cold dilute aqueous hydrochloric acid (1:15). It was then extracted with ether, the organic layer was separated and washed with water. Careful removal of solvent gave an oil which on trituration with ethanol furnished a solid. This was then recyrstallized with aqueous ethanol.

4-Bromo-4-substituted-benzyl-3-methyl-5(4*H*)-isoxazolones **22-24**.

To the desired solution of **8-10** (1 g, 0.005 mole) in dry chloroform was added *N*-bromosuccinimide (0.980 g, 0.005 mole) and the reaction mixture was stirred at room temperature (25°-30°) for 1.5 hours. It was then washed with warm water and extracted with chloroform. The solvent was removed and the residue was crystallized from benzene-hexane mixture.

4-Hydroxy-4-substituted-benzyl-3-methyl-5(4*H*)-isoxazolones **27-28**.

To the required solution of **8-9** (1 g, 0.005 mole) in dry dichloromethane (5 ml) was added *meta*-chloroperbenzoic acid (1.8 g, 0.01 mole) at room temperature (30°) and the reaction was allowed to proceed for 25 hours. It was then filtered and the filtrate was passed through a column of silica gel. Removal of the solvent furnished the desired products as oils.

4,4′-Bis[4-substituted-benzyl-3-methyl-5(4*H*)-isoxazolones] **29-35**.

i. To the desired solution of **8-14** (1 g, 0.005 mole) in acetic acid (8 ml) was added manganic acetate dihydrate (2.68 g, 0.01 mole) at 40° and was stirred at 70°. On cooling ice water was added and the separated solid was filtered. It was crystallized from benzene-hexane mixture.

ii. To the required solution of **8-14** (1 g, 0.005 mole) in dry dichloromethane (6 ml) was added pyridinium chlorochromate (2 g, 0.01 mole) at room temperature (30°). The reaction was allowed to proceed for 25 minutes and was then filtered. The filtrate was passed through a column of florisil and the column was finally washed with ether. The solvents from the combined organic layer were removed and the residue was crystallized from benzene-hexane mixture.

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