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REACTION OF β-ARYLACRYLOXIRANES WITH PHENYL AZIDE. SYNTHESIS AND CHEMICAL TRANSFORMATIONS OF β-PHENYLAMINO-β-ARYLACRYLYLOXIRANES

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UDC 547.824'724'555.1'391.1'422.22-31.07:541.124

The reaction of β-arylacrylyloxiranes with phenyl azide by refluxing in dioxane or toluene leads to β-phenylamino-β-aryl-acrylyloxiranes. Epoxypropionyl- and β-hydroxypropionyltriazoles are also isolated when the reaction is carried out in the dark without heating. It is shown that β-phenylamino-β-arylacrylyloxiranes undergo cyclization to 3(2H)-furanones in an acidic medium, whereas they are converted to 2,3-dihydro-4-pyridones under basic-catalysis conditions.

Epoxypropionylpyrazolines, which were found to be convenient starting substances for the synthesis of compounds of the bipyrazole and pyrrolidino[1,2-b]pyrazole series, were previously obtained in a study of the cycloaddition of diazo compounds to β-arylacrylyloxiranes [1, 2]. In a continuation of our study of the cycloaddition of 1,3-dipoles to β-arylacrylyloxiranes and in order to synthesize functionally substituted ketones of the heterocyclic series we have investigated the reactions of β-arylacrylyloxiranes with phenyl azide and some synthetic possibilities of the resulting β-phenylamino-β-arylacrylyloxiranes.

We have established that the reaction of β-arylacrylyloxiranes Ia-f with phenyl azide by refluxing in an aprotic solvent (dioxane, toluene) for 15-20 h leads to epoxy enamino ketones II and V-IX (in 50-75% yields), the structures of which were confirmed by the results of elementary analysis and IR and PMR spectral data (Table 1), as well as by alternative synthesis of II from β-phenylpropionyloxirane Ig. The existence of enamino ketones II and V-IX in the cis-s-cis-chelate-bonded form is in agreement with the set of spectral data, viz., the chemical shift of the NH proton (12.05-12.20 ppm), as well as the characteristic frequencies of vibrations of NH, C=O, and C=C bonds at 3170, 1610, 1605, 1590, and 1560 cm^{-1} [3].

For the isolation and identification of the intermediates formed during the described reaction, epoxy enone Ia was maintained with phenyl azide in dioxane at room temperature in

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0009-3122/88/2408-0839$12.50

TABLE 1. Characteristics of II-XXII

Compound	mp, °C	R_f*	IR spectrum, ν, cm^{-1}	PMR spectrum, ppm (J, Hz)**
II	145...146	0,67	1560, 1590, 1605, 3170	1,40 (s, 3H); 2,72, 2,80 (two d, 2H, J=5,0); 5,40 (s, 1H); 6,90 (m, 5H); 7,20 (s, 5H); 12,05 (s, 1H)
III	131...132	0,55	1550, 1595, 1690	1,60 (s, 3H); 2,80, 3,30 (two d, 2H, J=5,0); 7,24 (s, 5H); 7,30 (s, 5H)
IV	116...117	0,39	1550, 1595, 1680, 3520	1,27 (d, 3H, J=6,5); 2,35 (t, 1H, J=6,0); 3,88 (dd, 2H, J=6,0, J=6,0); 4,02 (d, m 1H, J=6,5, J=6,0); 7,17 (m, 8H); 7,29 (m, 2H)
V	132...133	0,69	1560, 1590, 1605, 3170	1,46 (s, 3H); 2,86, 2,90 (two d, 2H, J=5,0); 5,50 (s, 1H); 7,00 (m, 5H); 7,36 (s, 4H); 12,18 (s, 1H)
VI	104...106	0,68	1560, 1590, 1605, 3170	1,45 (s, 3H); 2,84, 2,88 (two d, 2H, J=5,0); 5,50 (s, 1H); 7,73 (m, 5H); 7,59 (s, 4H); 12,20 (s, 1H)
VII	101...103	0,68	1565, 1590, 1605, 3170	1,24 (s, 3H); 1,30 (s, 3H); 3,18 (s, 1H); 5,45 (s, 1H); 6,80 (m, 5H); 7,20 (s, 5H); 12,20 (s, 1H)
VIII	114...116	0,67	1560, 1590, 1605, 3150	1,12 (d, 3H, J=6,5); 1,33 (s, 3H); 2,94 (q, 1H, J=6,5); 5,40 (s, 1H); 6,78 (m, 5H); 7,20 (s, 5H); 12,20 (s, 1H)
IX	126...127	0,67	1560, 1580, 1600, 3160	3,40, 3,98 (two d, 2H, J=2,0); 5,50 (s, 1H); 6,88 (m, 5H); 7,22 (s, 10H); 12,26 (s, 1H)
X	Oil	0,40	1560, 1590, 1605, 3170, 3330, 3430	1,20 (s, 3H); 2,28 (s, 3H); 2,40, 2,85 (two d, 2H, J=12,0); 2,75 (s, 2H); 5,53 (s, 1H); 6,70 (m, 5H); 7,10 (s, 5H); 12,05 (s, 1H)
XI	Oil	0,20	1560, 1590, 1605, 3170, 3450	1,16 (s, 3H); 2,16 (s, 6H); 2,24, 2,80 (two d, 2H, J=12,0); 3,27 (s, 1H); 5,80 (s, 1H); 6,70 (m, 5H); 7,27 (s, 5H); 12,15 (s, 1H)
XII	Oil	0,20	1560, 1590, 1605, 3150, 3450	0,86 (t, 6H, J=7,0); 1,18 (s, 3H); 2,30, 2,90 (two d, 2H, J=12,0); 2,44 (q, 4H, J=7,0); 4,15 (s, 1H); 5,90 (s, 1H); 6,75 (m, 5H); 7,20 (s, 5H); 12,10 (s, 1H)
XIII	159...160	0,43	1600, 1685, 3450	1,02 (s, 3H); 3,38 (d, 2H, J=6,0); 4,80 (t, 1H, J=6,0); 5,95 (s, 1H); 6,70 (m, 5H); 7,50 (m, 5H)
XIV	103...104	0,14	1600, 1695, 3370	1,34 (s, 3H); 2,20 (s, 1H); 2,30 (s, 3H); 2,78 (s, 2H); 5,82 (s, 1H); 7,30 (m, 3H); 7,66 (m, 2H)
XV	Oil	0,55	1600, 1595	1,28 (s, 3H); 2,18 (s, 6H); 2,38, 2,50 (two d, 2H, J=13,0); 5,85 (s, 1H); 7,30 (m, 3H); 7,60 (m, 2H)
XVI	Oil	0,55	1600, 1695	0,82 (t, 6H, J=7,0); 1,26 (s, 3H); 2,48 (q, 4H, J=7,0); 3,66 (s, 2H); 6,00 (s, 1H); 7,40 (m, 3H); 7,78 (m 2H)
XVII	69...70	0,52	1600, 1700	1,44 (s, 3H); 3,65 (s, 2H); 6,10 (s, 1H); 7,42 (m, 3H); 7,80 (m, 2H)
XVIII	115...116	0,55	1600, 1700	1,40 (s, 3H); 3,76 (s, 3H); 6,10 (s, 1H); 7,45 (m, 3H); 7,80 (m, 2H)
XIX	154...155	0,28	1600, 1630, 3470	1,30 (s, 3H); 3,90, 4,15 (two d, 2H, J=11,0); 4,26 (s, 1H); 5,20 (s, 1H); 7,10 (m, 5H); 7,55 (m, 5H)
XX	142...143	0,28	1600, 1630, 3470	1,28 (s, 3H); 3,82, 4,10 (two d, 2H, J=11,0); 4,16 (s, 1H); 5,15 (s, 1H); 7,00 (m, 4H); 7,20 (s, 5H)
XXI	174...175	0,29	1605, 1630, 3470	1,20 (s, 3H); 1,25 (d, 3H, J=7,0); 3,93 (q, 1H, J=7,0); 4,23 (s, 1H); 5,06 (s, 1H); 6,87 (s, 5H); 7,08 (s, 5H)
XXII	225...227	0,65	1560, 1630	1,93 (d, 3H, J=0,8); 6,10 (s, 1H); 7,15 (s, 5H); 7,20 (s, 5H); 7,62 (q, H, J=0,8)

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*Solvents: ether—hexane (2:1) for V-IX, XIII, and XIX-XXI,
**Solvents for the PMR spectra: d_6-acetone for II, III, V-IX, XI, and XIV-XVI.

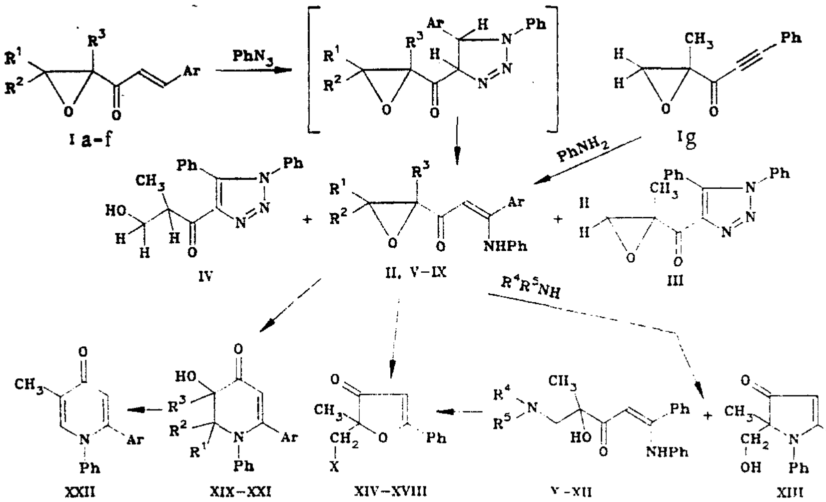
Found, %			Empirical formula	Calc., %			Yield, %
C	H	N		C	H	N	
77,3	6,0	5,0	$C_{18}H_{17}NO_2$	77,4	6,1	5,0	75
70,8	5,7	13,7	$C_{18}H_{15}N_3O_2$	70,8	5,0	13,8	25
70,0	5,7	13,5	$C_{18}H_{17}N_3O_2$	70,3	5,6	13,7	28
68,8	5,0	4,4	$C_{18}H_{16}ClNO_2$	69,9	5,2	4,5	66
60,4	4,6	4,0	$C_{18}H_{16}BrNO_2$	60,3	4,5	3,9	59
77,7	6,5	4,6	$C_{19}H_{19}NO_2$	77,8	6,5	4,8	56
77,9	6,5	4,6	$C_{19}H_{19}NO_2$	77,8	6,5	4,8	61
81,0	5,7	4,0	$C_{23}H_{19}NO_2$	80,9	5,6	4,1	50
73,6	7,1	8,8	$C_{19}H_{22}N_2O_2$	73,5	7,2	9,0	90
73,9	7,7	8,6	$C_{20}H_{24}N_2O_2$	74,0	7,5	8,6	90
74,9	8,0	7,9	$C_{22}H_{28}N_2O_2$	75,0	8,0	8,0	80
77,4	6,0	4,9	$C_{18}H_{17}NO_2$	77,4	6,1	5,0	10
71,0	6,6	6,9	$C_{12}H_{13}NO_2$	70,9	6,5	6,9	71
71,7	6,8	6,3	$C_{13}H_{15}NO_2$	71,9	7,0	6,5	66
73,5	7,7	5,7	$C_{15}H_{19}NO_2$	73,4	7,8	5,7	70
53,9	4,1	—	$C_{12}H_{11}BrO_2$	54,0	4,2	—	90
64,5	5,0	—	$C_{12}H_{11}ClO_2$	64,7	5,0	—	92
77,3	6,0	4,9	$C_{18}H_{17}NO_2$	77,4	6,1	5,0	95
68,8	5,1	4,6	$C_{18}H_{16}ClNO_2$	68,9	5,2	4,5	92
77,8	6,6	4,7	$C_{19}H_{19}NO_2$	77,8	6,5	4,8	97
82,6	5,6	5,3	$C_{18}H_{15}NO$	82,7	5,8	5,3	60

and chloroform—methanol (4:3) for XXII.
XII, XIII, and XVII-XXII, $CDCl_3$ for IV, and CCl_4 for X,

the dark for 30 months (until the starting compound vanished). As a result of chromatographic separation of the reaction mixture, in addition to oxiranyl enamino ketone II (in 35% yield), we isolated epoxypropionyltriazole III and β-hydroxypropionyltriazole IV. The formation of enamino ketone II and triazoles III and IV evidently proceeds through an intermediate triazoline, which, however, could not be isolated from the reaction mixture. The transformation of the latter is evidently determined by the character and ratio of the resulting products. The principal pathway (it becomes the only pathway at 100-110°C) includes cleavage of the triazoline ring with the formation of α-diazo β-amino ketones, which, by elimination of nitrogen, are converted to epoxypropionyl enamino ketones II and V-IX [4]. At room temperature the competitive oxidation fo the triazoline by air oxygen, which leads to stable epoxypropionyltriazole III, begins to play a significant role. At the same time, aromatization of the triazoline ring is possible through an intramolecular redox process with the formation of β-hydroxypropionyltriazole IV. Thus the reaction of phenyl azide with β-arylacrylyloxiranes at 100-110°C can serve as a preparative method for the synthesis of β-phenylamino-β-arylacrylyloxiranes; this supplements the known methods for obtaining compounds of this series [5, 6].

Despite the extensive use of amino vinyl ketones in organic synthesis [3], the chemistry of epoxypropionyl enamino ketones has not been adequately studied [7,8]. In the present research we investigated some possibilities for the use of compounds of this series for synthetic purposes. It was shown that the corresponding amino ketones X-XII are formed in the reaction of amino ketone II with methyl- and dimethyl(ethyl)amine in isopropyl alcohol. The use of aqueous solutions of methyl- or dimethylamine leads to an increase in the reaction rate; however, the process is accompanied by the formation of up to 10% side product — 3(2H)-pyrrolone XIII. 3(2H)-Furanone hydrochlorides, which were identified in the form of the corresponding bases XIV-XVI, are formed in an attempt to obtain the hydrochlorides of aminohydroxy ketones X-XII. It should be noted that the cyclization of N-methylamino hydroxy ketone X, as in the case of dialkylamino ketones XI and XII, also proceeds with the participation of the hydroxy group rather than the methylamino group.

It was found that the formation of 3(2H)-furanones in an acidic medium has general character in the hydroxy (epoxy) enamino ketone series. Thus the reaction of enamino ketone II with hydrobromic or hydrochloric acid in acetic acid does not stop at the step involving the formation of hydrohalides, and the subsequent heterocyclization, which is accompanied by the elimination of aniline hydrobromide (hydrochloride), leads to 2-bromo(chloro)-3(2H)-furanones XVII and XVIII. Furanone XVIII is also isolated in an attempt to acetylate II with acetyl chloride in chloroform.



Ia—c II, V, VI, XIX, XX $R^1=R^2=H$, $R^3=CH_3$; Ia, II, XIX $Ar=C_6H_5$; Ib V, XX $Ar=4\text{-}ClC_6H_4$; Ic, VI $Ar=BrC_6H_4$; Id, VII $R^1=R^2=CH_3$, $R^3=H$, $Ar=C_6H_5$; Ie, VIII, XXI $R^1=R^3=CH_3$, $R^2=H$, $Ar=C_6H_5$; If, IX $R^1=Ar=C_6H_5$, $R^2=R^3=H$; X $R^4=CH_3$, $R^5=H$; XI $R^4=R^5=CH_3$; XII $R^4=R^5=C_2H_5$; XIV $X=CH_3NH$; XV $X=(CH_3)_2N$; XVI $X=(C_2H_5)_2N$; XVII $X=Br$; XVIII $X=Cl$

It has been previously shown that β-arylamino vinyl epoxy ketones do not undergo cyclization to dihydropyridones upon prolonged refluxing in isopropyl alcohol [7, 8]. However, it was found that the reaction proceeds readily under basic-catalysis conditions. Thus the corresponding 2-hydroxy-2,3-dihydro-4-pyridones XIX-XXI are formed when tetrabutylammonium hydroxide in dioxane or triethylamine in isopropyl alcohol is added to II, V, and VIII in the course of 0.5-2.0 h. Dehydration with the formation of 4-pyridone XXII occurs when XIX is heated in acetic anhydride.

Thus the synthesized epoxopropionyl enamino ketones are convenient starting substances for the synthesis of functionally substituted 3(2H)-furanones and 2,3-dihydro-4-pyridones.

EXPERIMENTAL

The IR spectra of solutions of the substances in CCl_4 and $CHCl_3$ ($c = 10^{-1}$ μ, $\ell = 0.01$ cm) were recorded with a Specord 75-IR spectrophotometer. The PMR spectra of solutions of the compounds in CCl_4 and d_6-acetone were obtained with a Tesla BS-467-A spectrometer (60 MHz) with hexamethyldisiloxane as the internal standard. The course of the reactions was monitored by means of TLC on Silufol plates in ether—hexane (2:1) and chloroform—methanol (4:3). The characteristics of the synthesized compounds are presented in Table 1.

5-Aryl-2-methyl-5-phenylamino-1,2-epoxy-4-penten-3-ones II, V, and VI, 2(3)-Methyl-6-phenyl-6-phenylamino-2,3-epoxy-5-hexen-4-ones VII and VIII, and 1,5-Diphenyl-5-phenylamino-1,2-epoxy-4-penten-3-one (IX). A 13-g (110 mmole) sample of phenyl azide was added in portions to a solution of 100 mmole of epoxy enone Ia-f in 50-100 ml of dioxane or toluene, and the reaction mixture was refluxed for 15-20 h. The solvent was evaporated with a film evaporator, the solid residue was dissolved in ether, and the solution was filtered through a layer of silica gel (elution with ether). The solvent was removed by distillation, and II and V-IX were crystallized from acetone—hexane (1:2) or ether—hexane (1:1).

2-Methyl-5-phenyl-5-phenylamino-1,2-epoxy-4-penten-3-one (II), 1,5-Diphenyl-4-(2-methyl-2,3-epoxypropionyl)-1,2,3-triazole (III), and 1,5-Diphenyl-4-(3-hydroxy-2-methylpropionyl)-1,2,3-triazole (IV). A mixture of 4.7 g (25 mmole) of Ia and 3.2 g (27 mmole) of phenyl azide in 10 ml of dioxane was maintained for 30 months in the dark, after which the solvent was evaporated in vacuo, and the residue was chromatographed with a column packed with silica gel [elution with ether—hexane (1:1)]. Triazoles III and IV were crystallized from ether—hexane (2:1).

1-Alkyl(dialkyl)amino-2-hydroxy-2-methyl-5-phenyl-5-phenylamino-4-penten-3-ones X-XII and 2-Hydroxymethyl-2-methyl-1,5-diphenyl-3(2H)-pyrrolone (XIII). A) A 20-mmole sample of methyl-, dimethyl-, or diethylamine in 5-10 ml of alcohol was added to a solution of 2.7 g (10 mmole) of II in 20 ml of isopropyl alcohol, and the mixture was stirred at 40-60°C for 12 h until epoxy ketone II had dissolved completely. The solvent was evaporated with a rotary evaporator to isolate the individual amino hydroxy ketones X-XII in the form of yellow oils.

B) The reaction was carried out as described in experiment A using a 30% aqueous solution of methyl- or dimethylamine. After 2-3 h, the reaction mixture was filtered through a layer of silica gel. Successive elution with ether and methanol gave pyrrolone XIII and amino hydroxy ketones X and XI, respectively. Compound XIII was crystallized from ether—hexane (1:2).

2-Alkyl(dialkyl)amino-2-methyl-5-phenyl-3(2H)-furanones XIV-XVI. An ether solution of hydrogen chloride or a twofold excess of hydrochloric acid was added to a solution of 10 mmole of amino hydroxyketone X-XII in ether or acetic acid, and the mixture was maintained for 1 h at room temperature. After removal of the ether, the residue was diluted with water, and the aqueous mixture was made alkaline with sodium carbonate solution and extracted with ether. The ether solution was dried over sodium sulfate. After removal of the ether, XIV was crystallized from ether—hexane (1:2), while XV and XVI were separated from the aniline by filtration through a layer of silica gel.

2-Bromo(chloro)methyl-2-methyl-5-phenyl-3(2H)-furanones XVII and XVIII. A) A 2.5-ml sample of 47% hydrobromic acid or 36% hydrochloric acid was added to a solution of 2.7 g (10 mmole) of II in 10 ml of acetic acid. After 1 h, the reaction mixture was poured into ice water, and the aqueous mixture was extracted with ether (three 50-ml portions). The ether solution was washed successively with sodium carbonate solution and dried over sodium sulfate. Compounds XVII and XVIII crystallized after partial removal of the ether (up to 30 ml) and the addition of 30 ml of hexane.

B) A 5-ml sample of acetyl chloride was added to a solution of 1.4 g (5 mmole) of enamino ketone II in 20 ml of chloroform. After 12 h, the solvent was removed, and furanone XVIII was isolated as described in experiment A.

3-Hydroxy-6-aryl-3-methyl-1-phenyl-2,3-dihydro-4-pyridones XIX and XX and 3-Hydroxy-2,3-dimethyl-1,6-diphenyl-2,3-dihydro-4-pyridone (XXI). A 0.2-0.3-g sample of tetrabutylammonium hydroxide (or 1 ml of triethylamine) was added to a solution of 10 mmole of enamino ketone II, V, or VIII in 10 ml of dioxane (or isopropyl alcohol), and the solvent was then evaporated after 0.5 h (2 h). The residue was filtered through a layer of silica gel (elution with ether), and dihydropyridones XIX-XXI were crystallized from ether.

3-Methyl-1,6-diphenyl-4-pyridone (XXII). A solution of 5 mmole of dihydropyridone XIX in 5 ml of acetic anhydride was refluxed for 2 h, after which the mixture was diluted with water, and the aqueous mixture was made alkaline with sodium carbonate solution and extracted with ether. The ether solution was washed with sodium carbonate solution and water and dried. Compound XXII crystallized after partial removal of the ether.

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