PREPARATION OF N-METHACRYLOXYETHYL DERIVATIVES OF SPIROPYRANS OF THE INDOLINE SERIES*

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Reaction of 2,3,3-trimethylindolenine with ethylene iodohydrin gave 1-hydroxyethyl-2,3,3trimethylindoleninium iodide, which gives 3a,4,4-trimethyloxazolidino[3,2-a]indoline on treatment with sodium carbonate solution. Condensation of the latter with 5-nitro- and 3methoxy-5-nitrosalicylaldehydes gives, respectively, N-hydroxyethyl derivatives of 6'nitro- and 6'-nitro-8'-methoxyspiro(indoline-2,2'-benzopyrans) (V, VI), while condensation with 3-nitrosalicylaldehyde gives 4,4-dimethyl-3a-(2-hydroxy-3-nitrostyryl)oxazolidino[3,2-a]indoline (VII). Acylation of nitrospiran V and oxazolidinoindoline VII with methacrylyl chloride in pyridine proceeds at the free hydroxyl groups, while a mixture of derivatives of $1-(\beta$ -methacryloxyethyl)spiropyran and 3a-(2-methacryloxy-3-nitrostyryl)oxazolidinoindoline is formed under the same conditions from nitromethoxyspiropyran VI. Acylation of V in acetone in the presence of sodium bicarbonate gives 4,4-dimethyl-3a-(2-methacryloxy-5-nitrostyryl)oxazolindo-[3,2-a]indoline. The mechanism of the isomeric transformations of the various forms that contain spiropyran and oxazolidino-indoline rings was studied by means of electronic spectra. The photochromic properties of the spiropyrans were investigated.

Spiropyrans that contain unsaturated groupings are of interest as monomers for the preparation of photochromic polymers, but compounds of this type have received very little study [2-5]. An attempt to obtain 1-methacryloxyethyl-3,3-dimethyl-6'-nitrospiro(indoline-2,2'-[2H-1]-benzopyran) is described in a patent [5], but neither the methods used to purify the product nor its characteristics are given. The aim of the present investigation was the synthesis of 1-methacryloxyethyl derivatives of spiropyrans of the in-doline series.

Heating of 2,3,3-trimethylindolenine (I) with ethylene iodohydrin gave a quantitative yield of a quaternary salt, to which we assigned the 1-hydroxyethyl-2,3,3-trimethylindoleninium iodide structure (II). However, on treatment of the latter with sodium carbonate solution, we isolated 3a,4,4-trimethyloxazolidino-[3,2-a]indoline (III) in 93.5% yield instead of the expected methylene base.



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Signals of methyl groups at 1.05, 1.20, and 1.33 ppm and a multiplet at 3.35 ppm (the center of the multiplet). which corresponds to the methylene groups of the oxazolidine ring, are observed in the PMR spectrum of III (Fig. 1). The multiplet structure of the signal of the methylene chain of the condensed oxazolidine ring is in agreement with the data in [6] on the PMR spectra of 2oxazoline. The addition of trifluoroacetic acid does not change the form of the spectrum; this indicates the absence of an OH group. The absorption bands at 3200-3600 cm⁻¹ that are characteristic for the stretching vibrations of the OH group are absent in the IR spectrum of III.



The fact mentioned above compelled us to turn again to the problem of the structure of the salt in order

to choose between structures II and IV. For this purpose we studied the IR spectra of the salt and of 2,3,3trimethylindoleninium chloride, which we obtained for comparison. It was found that the absorption bands at 2300-2600 cm⁻¹ that are characteristic for the NH⁺ stretching vibrations [7] are absent in the spectrum of the salt, while they are quite intense in the spectrum of trimethylindoleninium chloride. In addition, an intense band at 3300 cm⁻¹, which can be assigned to the stretching vibrations of OH groups ties up in hydrogen bonds, is observed in the spectrum of the salt. When the hydrogen of the OH group is replaced by deuterium, the absorption band at 3300 cm⁻¹ decreases, and a band at 2450 cm⁻¹, which is related to OD stretching vibrations, appears. Consequently, the quaternary salt that we obtained has structure II.

When III reacts with 5-nitro- and 3-methoxy-5-nitrosalicylaldehydes, the oxazolidine ring opens and hydroxyethyl derivatives of spiropyrans (V, VI) are formed in good yields. On the other hand, reaction of III with 3-nitrosalicylaldehyde proceeds without cleavage of the ring and leads to styrene derivatives (VII) in the trans form. In the latter case, subsequent transformation of VII to a spiropyran does not occur, apparently because of the formation of an intramolecular hydrogen bond between the OH and NO2 groups.



Signals at δ 1.20 and 1.30 ppm, which are related to the two methyl groups of the $C(CH_3)_2$ grouping. are observed in the PMR spectra of spiropyrans V and VI (the spectrum of VI also contains a signal at 3.82 ppm, which corresponds to the OCH₃ group). The signals of the two methylene groups have the characteristic triplet shape with δ 3.42 and 3.74 ppm and a spin-spin coupling constant (J) of 6 Hz. The signals of the protons in the 3' and 4' positions at about 6 ppm (the precise assignment of the lines of the AB spectrum is difficult because of the superimposition of signals from the aromatic protons) have a spinspin coupling constant of 10.5 Hz; this attests to their cis orientation relative to the double bond [8]. The intervals of change in the shift of the singlet from 1.78 to 2.56 ppm at temperatures from +60 to -60° (for V) and from 1.84 to 2.00 ppm at +60 to -40° (for VI) confirm the presence of a weakly associated OH group.

The spectrum of VII, like the spectrum of III, contains signals at 1.20 and 1.48 ppm, which are characteristic for methyl groups, and a signal at 3.7 ppm, which corresponds to the methylene groups in the oxazolidine ring. The signal of the OH group at 11.11 ppm is practically independent of the solution temperature (up to 60°) and concentration. This indicates the presence of a structure with an intramolecular hydrogen bond. Similar conclusions are drawn in [9] on the basis of the PMR spectral data for o-nitro-



Fig. 2. Absorption spectra in pyridine $(l \ 1 \ cm)$: 1) V, c 5.06 $\cdot 10^{-5}$ M (freshly prepared solution); 2) V, c 5.0 $\cdot 10^{-5}$ M (solution stored for 24 h); 3) VII, c 5.06 $\cdot 10^{-5}$ M; 4) VI, c 5.01 $\cdot 10^{-5}$ M (freshly prepared solution); 5) VI, c 5.01 $\cdot 10^{-5}$ M (after irradiation for 30 sec with 303-313 nm light); 6) VI, c 5.02 $\cdot 10^{-5}$ M (solution stored for 24 h).

phenols. The planar structure of the H-chelate ring is apparently a large steric hindrance to the existence of the cis form, and VII is therefore found in the trans form. The signals of the H_{α} and H_{β} protons of the trans form are situated at 6.42 and about 7.25 ppm with J = 16 Hz.

The stretching vibrations of the hydroxyl group in the IR spectrum of spiropyran V, suspended in mineral oil, appear at 3400 cm⁻¹ as a broad absorption band, which is characteristic for OH groups tied up in intermolecular hydrogen bonds. When this substance is dissolved in benzene, the hydrogen bonds are ruptured, and the band of a free hydroxyl group appears at 3600 cm^{-1} in place of the band at 3400 cm^{-1} . The absorption band of stretching vibrations of the OH group is found at 3220 cm^{-1} in the IR spectrum of VII (in mineral oil). Its position and intensity do not change in the spectrum of a benzene solution of the compound. The OH group in this case apparently has different character (phenolic hydroxyl group) than in spiropyran V (alcoholic hydroxyl group).

The merocyanine forms of spiropyrans V and VI are intensely colored crystalline substances that are light-insensitive [sic]. In both polar and nonpolar solvents they form colored solutions that gradually become decolorized under the influence of visible light or in the dark. An exception to this

is a solution of VI in alcohol, in which the equilibrium is shifted markedly to favor the colored form. The rate of dark decolorization of colored solutions of VI in other solvents (acetone, dioxane, and chloroform) is less than the rate of decolorization of corresponding solutions of V. The colorless solutions become colored on irradiation with UV light (a new absorption band in the visible region with λ_{max} 545 nm appears in the spectrum of an alcohol solution of V).

In contrast to V and VI, VII is not photochromic in solution in hexane, benzene, and ether (solutions in these solvents are yellow). We were able to observe the photochromism of VII only in alcohol solution at -20 to -30° . The mechanism of this process seems to be as follows. When the compound is dissolved in alcohol, the oxazolidine ring is cleaved, and the colored merocyanine form is generated (a solution of VII in alcohol is violet). At low temperatures this form is converted, under the influence of visible light, to the corresponding photochromic spiropyran, which can be converted to the colored form under the influence of UV light or by heating.



A direct confirmation of this assumption is offered by the following experiment. If an alcohol solution of VII is evaporated and the residual lilac-colored substance is dissolved in ether, one obtains a blue solution that retains its photochromic properties for three to four decolorization-coloring cycles. The solution becomes yellow after loss of its photochromic properties. In the case described, the merocyanine form is gradually isomerized to nonphotochromic compound VII. The fact that yellow oxazolidine form VII is produced during synthesis in alcohol solutions is apparently associated with the fact that this form in solution is in equilibrium with the merocyanine form but precipitates because of its lower solubility in alcohol.

To synthesize the methacryloxyethyl derivatives we treated V-VII with methacrylyl chloride in pyridine. The acylation of V was also carried out in acetone in the presence of sodium bicarbonate. In the acylation of V and VII in pyridine we obtained, as expected, the corresponding derivatives of spiropyran (VIII) and (hydroxystyryl)oxazolidino-indoline (IX).



 $IX R^{1} = NO_{2}; R^{2} = H; X R^{1} = H; R^{2} = NO_{2}$

Treatment of spiropyran VI under similar conditions gave (according to PMR and IR spectroscopy) a mixture of approximately equal amounts of spiropyran and (hydroxystyryl)oxazolidinoindoline derivatives; we were unable to separate this mixture. Acylation of V in acetone gives only X in low yield (17%), and primarily unchanged V is recovered.

To clarify the above-noted peculiarities in the acylation, we studied the electronic spectra of V-VII in pyridine, acetone, and acetone saturated with hydrogen chloride. As seen from Fig. 2, an absorption band at about 475 nm is present in the absorption spectrum of a pyridine solution of VII, which contains an oxazolidine ring; this band is absent in the spectrum of a hexane solution. The appearance of an absorption band at 475 nm can be explained by the formation of a complex of the pyridine—phenol type. The spectrum of VI contains two bands at 370 and 475 nm. The first band is related to the form with a spiropyran structure, while the presence of the second band attests to the formation in solution of V. When solutions of V and VI are stored in the dark, an increase in the absorption band at 455-475 nm is observed in 24 h or when they are irradiated with UV light. This indicates the buildup in solution of the form with the oxazolidine ring. The development of a complex of the pyridine—phenol type is undoubtedly the principal reason for the formation of compounds that contain an oxazolidine ring. Thus the interconversions of the various forms in pyridine solutions of V and VI can be reflected by means of the following scheme:

$$\mathbf{A} \xrightarrow{hv_1, \ k_1 \mathrm{T}}_{k_2 \mathrm{T}, \ hv_2} \quad \mathbf{B} \xrightarrow{\text{pyridine}} \mathbf{C},$$

where A is the starting form with a spiropyran structure, B is the colored merocyanine form, and C is the form with an oxazolidine ring. The concentrations of forms A and C are determined by the ratio of the rate constants of the elementary reactions represented by the scheme. As has already been noted above, the introduction of an OCH_3 group into the 8' position leads to a decrease in the rate of dark decolorization of the solutions. This may apparently explain the fact that the rate of buildup of the form with the oxazolidine ring in the case of VI is substantially greater than rate of buildup of the analogous form in the case of V. In the acylation of V, form C is practically absent, and the only reaction product is spiropyran VIII. However, in the acylation of V, a reaction product containing (according to the IR spectra) about 90% of VIII and about 10% of X was obtained 24 h after dissolving the compound in pyridine.

The absorption spectra of solutions of V and VII in acetone saturated with hydrogen chloride (Fig. 3) have an absorption band at 420 nm, which corresponds to salt XI. This fact is evidence that both the spiropyran and oxazolidine rings are opened in acid media. Thus it can be assumed that acylation of V in acetone proceeds via the following scheme:



It is not at all clear to us why acylation will lead only to phenol derivatives under these conditions. However, it is important to note that the result is associated with the presence of free hydrogen chloride in the reaction mixture. Sodium bicarbonate is apparently a less effective reagent that ties up the hydrogen chloride. This conclusion is confirmed by the fact that a product containing about 66% VIII and about 34% X (according to the IR spectra) is formed in the acylation of V in the presence of one mole of triethylamine, when the reaction proceeds under homogeneous conditions. It should be pointed out that the corre-



Fig. 3. Absorption spectra in acetone $(l \ 1 \ cm)$: 1) V, c $6.9 \cdot 10^{-5}$ M; 2) VII, c $6.9 \cdot 10^{-5}$ M; 3) VII, c $2.76 \cdot 10^{-5}$ M (solution saturated with HCl); 4) V, c $2.3 \cdot 10^{-5}$ M (solution saturated with HCl).



Fig. 4. IR spectra: 1) V (in mineral oil); 2) VIII (in CCl_4); 3) VII (in mineral oil); 4) IX (in CCl_4); 5) product of acylation of VI.

sponding o-acetoxyarylvinylpyrylium salts [10] are formed in the acylation of spiropyrans with acetic anhydride in the presence of perchloric acid.

The structure of acyl derivatives VIII-X was confirmed by the spectral data. The PMR spectrum of VIII (Fig. 1), like the spectra of V and VI, contains signals at δ 1.18 and 1.30 ppm, which are characteristic for gem-methyl groups, and triplets at 3.51 and 4.34 ppm, which correspond to methylene groups. The presence of a methacrylyl group is confirmed by the presence of methyl proton signals (1.92 ppm) and protons of a terminal =CH₂ group (5.57 and 6.10 ppm). The cis configuration of the 3'-H and 4'-H protons relative to the double bond is confirmed by the J constant (10.5 Hz).

As in the spectra of III and VII, two signals from the protons of gem-methyl groups at 1.16 and 1.42 ppm and signals at 3.61 ppm, which are characteristic for the oxazolidine ring, are observed in the PMR spectrum of X (Fig. 1). The trans configuration of the 3'-H and 4'-H protons relative to the double bond is confirmed by the J constant (16 Hz). The signals at 2.07 ppm are related to the methyl protons, while the signals at 5.85 and 6.43 ppm are related to the protons attached to the double bond of the methacrylyl group.

The IR spectra of acyl derivatives VIII and IX are presented in Fig. 4. The band at 1730 cm^{-1} should be assigned to the stretching vibrations of the carbonyl group in the first case, while the band at 1760 cm⁻¹ should be assigned to the carbonyl stretching vibrations in the second case. Thus there is a possibility for the analysis of the relative percentage of these substances by IR spectroscopy if a mixture of them is formed in the reaction. The spectrum of this sort of product of the reaction of spiropyran VI with methacrylyl chloride is presented in Fig. 4. It is also interesting to note that the absorption bands in the region characteristic for the vibrations of the benzene ring (1500-1600 cm⁻¹) undergo a marked change in intensity on acylation of VII; the absorption band at 1600 cm^{-1} , which is usually the most intense band in this region when conjugation is present [11], decreases considerably. This is not observed in the acylation of V – the ratio of the intensities of the absorption bands at $1500-1600 \text{ cm}^{-1}$ remains unchanged.

Acylspiropyran VIII is a white substance that is nonphotochromic in the crystalline state. Solutions of VIII in benzene.

ethyl acetate, ether, and other solvents have clearly expressed photochromic and dark properties. When solutions of VIII in toluene are irradiated with UV light, they become blue (an absorption band with λ_{\max} 600 nm appears). The color disappears rapidly after the action of the light is ceased because of dark cyclization.

The copolymerization of VIII with methyl methacrylate and the properties of the photochromic polymer will be described in the next paper of this series.

EXPERIMENTAL

The PMR spectra were measured with an RC-60 spectrometer. Carbor tetrachloride (III) and deuterochloroform (V-VIII, X) were used as the solvents. Solutions of V and VI were allowed to stand in light at room temperature for about 1 h to decolorize them prior to recording of the spectra. The chemical shifts were measured on the δ scale relative to tetramethylsilane as the internal standard; the accuracy in the measurement of the shifts was ± 0.05 ppm, while the accuracy in the measurement of the spin-spin coupling constants was ± 0.2 Hz. The IR spectra were recorded with a UR-10 spectrometer. The UV spectra were recorded with a Unicam SP-700 spectrophotometer.

<u>3a,4,4-Trimethyloxazolidino[3,2-a]indoline (III)</u>. A mixture of 1 g (6.3 mmole) of 2,3,3-trimethylindolenine (I) and 1.4 g (8.1 mmole) of ethylene iodohydrin was heated at 95-100° for 3 h. The resulting solid was washed several times with benzene and dried in a vacuum desiccator to give 2.08 g (100%) of 1-(β -hydroxyethyl)-2,3,3-trimethylindoleninium iodide (II) as white crystals with mp 162-163° (from alcohol). Found,%: C 47.5; H 5.4; I 38.6; N 4.7. C₁₃H₁₈INO. Calculated,%: C 47.1; H 5.4; I 38.3; N 4.2.

A 2-g (6.3 mmole) sample of II was dissolved in 60 ml of water, and the solution was filtered and treated with 0.6 g (5.6 mmole) of anhydrous sodium carbonate in 15 ml of water. The resulting emulsion was extracted with ether, and the extract was dried with anhydrous magnesium sulfate. The ether was vacuum evaporated, and the residue was chromatographed with a column filled with basic activity II/III aluminum oxide and elution with benzene-hexane (1:1) to give 1.14 g (93.5%) of III with bp 137-138° (20 mm) and mp 44° (from light petroleum ether). Found, %: C 76.9; H 8.4; N 7.3. $C_{13}H_{17}NO$. Calculated, %: C 76.8; H 8.4; N 6.9.

 $\frac{1-(\beta - \text{Hydroxyethyl})-3,3-\text{dimethyl}-6'-\text{nitrospiro}(\text{indoline}-2,2'-[2H-1]\text{benzopyran}) (V). A mixture of 2.03 g (10 mmole) of III and 1.67 g (10 mmole) of 5-nitrosalicylaldehyde was refluxed for 1.5 h in 30 ml of alcohol, after which it was allowed to stand overnight. The precipitate was separated and washed with alcohol to give 2.97 g (84.5%) of V as dark cherry-red crystals with mp 163-165° [from benzene-hexane (1:2)] and R_f* 0.36 (orange spot). UV spectrum (alcohol), <math>\lambda_{\max}$, nm (ϵ): 268 (15,900), 342 (8600). Found,%: C 68.4; H 5.7; N 7.8. C₂₀H₂₀N₂O₄. Calculated,%: C 68.2; H 5.7; N 8.0.

 $\frac{1-(\beta - \text{Hydroxyethy})-3,3-\text{dimethyl}-6'-\text{nitro}-8'-\text{methoxyspiro}(\text{indoline}-2,2'-[2H-1]\text{benzopyran}) (VI).}{\text{This compound was similarly obtained from 2.1 g (10.4 mmole) of III and 2.05 g (10.5 mmole) of 3-meth-oxy-5-nitrosalicylaldehyde in 50 ml of alcohol. The yield of dark-green crystals with a metallic luster and mp 205-206° (from alcohol) was 3.4 g (86%). UV spectrum (methanol), <math>\lambda_{\text{max}}$, (D): 285 (0.388), 373 (0.541), 550 (0.49), c 3.82 $\cdot 10^{-5}$ M, l 1 cm. Found,%: C 66.2; H 6.1; N 7.6. $C_{21}H_{22}N_2O_5$. Calculated,%: C 66.0; H 5.8; N 7.3.

<u>4,4-Dimethyl-3a-(2-hydroxy-3-nitrostyryl)oxazolidino[3,2-a]indoline (VII)</u>. This compound was similarly obtained from 2.18 g (10.7 mmole) of III and 1.77 g (10.7 mmole) of 3-nitrosalicylaldehyde in 32 ml of alcohol. The yield of yellow crystals with mp 169-169.5° [from benzene-hexane (1:6)] was 3.38 g (89.5%). UV spectrum, λ_{max} , nm (ϵ): 240 (28,200), 370 (3900) (hexane); 240, 350, 435, 550 (alcohol). Found,%: C 68.4; H 5.6; N 7.6. C₂₀H₂₀N₂O₄. Calculated,%: C 68.2; H 5.7; N 7.6.

<u>1-(β -Methacryloxyethyl)-3,3-dimethyl-6'-nitrospiro(indoline-2,2'-[2H-1]benzopyran) (VIII).</u> A 7-g (67 mmole) sample of methacrylyl chloride was added dropwise with vigorous stirring in the course of an hour to a cooled (to 3-4°) freshly prepared solution of 1.89 g (5.4 mmole) of V in 30 ml of dry pyridine, during which the intense rose color gradually disappeared and a precipitate appeared. Stirring was continued for another 3 h at 15-18°, after which the mixture was cooled with ice and treated carefully with 20 ml of cold water. The mixture was poured into 0.5 kg of ground ice, and the precipitate was removed by filtration, dried in a vacuum desiccator, and dissolved in benzene. The benzene solution was chromatographed with a column filled with hydrated silicic acid in benzene. The yield of white crystals of VIII with mp 111-112° [from benzene - hexane (1:10)] was 1.35 g (60%); the substance became blue at 105°, and the melt was blue; R_f 0.95 (orange spot). UV spectrum (ethyl acetate), λ_{max} , nm (ϵ): 269 (16,000), 335 (9500). Found, %: C 68.5; H 5.6; N 6.6. $C_{24}H_{24}N_2O_5$. Calculated, %: C 68.6; H 5.7; N 6.7.

<u>4,4-Dimethyl-3a-(2-methacryloxy-3-nitrostyryl)oxazolidino[3,2-a]indoline (IX)</u>. This compound was obtained as in the case of VIII from 0.95 g (2.7 mmole) of VII and 3.5 g (33 mmole) of methacrylyl chloride in 15 ml of pyridine. After chromatography (which gave a light-brown oily substance), the reaction product was triturated with 10 ml of alcohol, and the solid was separated and washed with 3-4 ml of cold alcohol to give 0.49 g (43.5%) of beige crystals of IX with mp 115-116° (from alcohol). Found, 7: C 68.7; H 5.6; N 6.3. $C_{24}H_{24}N_2O_5$. Calculated, 7: C 68.6; H 5.7; N 6.7.

<u>4,4-Dimethyl-3a-(2-methacryloxy-5-nitrostyryl)oxazolidino-[3,2-a]indoline (X)</u>. A 1.06-g (3 mmole) sample of V was dissolved in 30 ml of absolute acetone and 0.28 g (3.3 mmole) of sodium bicarbonate was added; 0.35 g (3.3 mmole) of methacrylyl chloride in 3.5 ml of absolute acetone was then added dropwise with stirring at 10° in the course of 25 min. The mixture was then refluxed for 15 min, after which 100 ml of water, 50 ml of acetone, and sodium bicarbonate (to pH 7) were added, and the mixture was extracted

^{*} The R_f values were obtained with plates of the Silufol UV₂₅₄ type with benzene-ether (7:3). The spots were colored by irradiation with UV light.

with ether. The ether was evaporated, and the residue was chromatographed with a column filled with hydrated silicic acid in benzene. The solvent was vacuum evaporated, and the dark-yellow oily residue was triturated with a small amount of alcohol. The solid was separated and washed with cold alcohol to give 0.21 g (17%) of yellow crystals of X with mp 138-138.5° (from alcohol) and R_f 0.74 (yellow spot). UV spectrum (alcohol), λ_{max} 235 nm (ϵ 21,300). Found,%: C 68.6; H 5.8; N 6.6. $C_{24}H_{24}N_2O_5$. Calculated,%: C 68.6; H 5.7; N 6.7.

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