after the elimination of the solvent was recrystallized from methanol, giving 3.1 g of the N-methylanilide (VIII) with mp 171-173°C and $[\alpha]_D$ −17.2° (c 4.11; $CHCl_3$).

^1H NMR spectrum (CCl_4) (ppm): 0.82 (3 H, s), 0.89 (3 H, s), 0.96 (3 H, s); 3.24 (3 H, s), 7.06-7.63 (5 H, m).

IR (KBr) (cm^{-1}): 3063, 3040, 1766, 1730, 1661, 1599, 1500, 1463, 1425, 1392, 1368, 1250, 1220, 1184, 1148, 1125, 1037, 1003, 779, 706, 667, 561.

The x-ray structural experiment was performed on a Syntex-$P2_1$ diffractometer using Mo radiation with a graphite monochromator. The crystals of the amide (VIII) belong to the rhombic system: $a = 11.504$ (5), b = 12.505 (5), c = 16.753 (7) Å, z = 4, space group $P2_12_12_1$, $d_{calc} = 1.13$ g/cm³, $C_{26}H_{35}O_3$. The intensities of 1953 independent reflections with $2\theta < 50°$ were measured by the standard $2\theta/\theta$ scanning method. The calculations were performed with 1008 reflections having $I > 3\sigma$. The structure was interpretated by the direct method of least squares in the full-matrix anisotropic approximation to R = 0.085 and R_W = 0.096, where $w^{-1} = \sigma_F + (0.01\ F)^2$. No attempts to localize the hydrogen atoms were undertaken in view of the low quality of the single crystal. The values of the coordinates of the atoms of the (VIII) molecule obtained are given in Table 2.

SUMMARY

The structure of the N-methylanilide (VIII) has been studied by the method of x-ray structural analysis and the structure of the "adduct" of caryophyllene with maleic anhydride has been confirmed.

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STEREOSELECTIVITY OF PHOTO-OXIDATION AT THE C_7 DOUBLE BOND OF ISOCEMBROL AND ITS DERIVATIVES

N. I. Yaroshenko and V. A. Raldugin

UDC 547.595.9

The products and stereochemistry of the photo-oxidation at the C_7 double bond of isocembrol and two of its derivatives have been investigated. The stereoselectivity of the transformations performed is discussed.

In connection with the detection in plants of natural terpenoid hydroperoxides [1], the investigations of the photo-oxidation of unsaturated terpenoids modeling the processes of their secondary metabolism in the green parts of plants is acquiring particular importance. In a preceding communication [2] we described the products of the photo-oxidation of isocembrol (I) at the C_{11} double bond — the diols (II-V) and the Δ^{12}-isomer of the diol (V). The photo-oxidation products were analyzed after the conversion of the hydroperoxides into the corresponding alcohols.

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TABLE 1. Chemical Shifts (ppm) and Spin–Spin Coupling Constants (Hz, shown in parentheses) in the PMR Spectra of Compounds (VII), (XX), and (XXI)

Compound	H_2	H_3	H_{5a}	$H_{5б}$	H_6	H_7	H_{11}	Tertiary Me groups	$-CH(\underline{C}H_3)_2$
VII	5,17 dd (16; 9)	5,47 d (16)	2,50 ddd (15; 4; 2)	2,17 dd (15; 10)	5,79 ddd (16; 10; 4)	5,57 dd (16; 2)	5,29 tm (~6)	Me_8 1,25 s Me_4 1,37 s Me_{12} 1,46 s	0,79 d (6) 0,82 d (6)
XX	5,20 dd (15; 10)	5,67 d (15)	2,53 dd (15; 4)	2,13 dd (15; 10)	5 98 ddd (16; 10; 3)	5,57 dd (16; 3)	2,98 dd (11; 2)	1,13 s 1,25 s 1,35 s	0,80 d (6) 0,83 d (6)
XXI	5,25 dd (16; 8; 5)	5,47 d (16)	2,44 dd (14; 6)	2,26 dd (14; 6)	5,70 dt (16; 6)	5 58 d (16)	2,94 dd (10; 3)	1,12 s 1,29 s 1,35 s	0,79 d (6) 0,82 d (6)

Arbitrary symbols. d — Doublet; s — singlet; m — multiplet; dd — doublet of doublets; and so on.

TABLE 2. Chemical Shifts (ppm) and Spin–Spin Coupling Constants (Hz, shown in parentheses) in the PMR Spectra of Compounds (VIII), (IX), and (XIXa)

Compound	H_2	H_3	Me_4	H_7	$>C{=}C\underline{H}_2$	H_{11}	Me_{12}	$-CH(C\underline{H}_3)_2$
VIII	5,27 dd (16; 8,4)	5,50 d (16)	1,33 s	4,14 m	4,94 m 5,12 m	5,00 m	1,50 s	0,77 d (6,7) 0,81 d (6,7)
IX	5,22 dd (15,5; 8,5)	5,59 d (16)	1,32 s	4,06 m	4,90 m 4,89 m	5,00 m	1 50 s	0,77 d (6,7) 0,81 d (6,7)
XIXa* 7-O-(Acetate)	5.33 dd (16; 8)	5,62 d (16)	1,34 s	5,14 tm (~7)	5,10 m 5,07 m	2 54 dd (4,8; 7,5)	1,21 s	0,80 d (6,5) 0,87 d (6,5)

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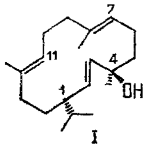
*In addition: Methyl of an acetyl group forms a singlet at 2.03 ppm.



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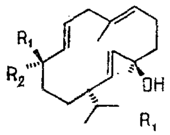
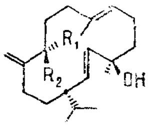
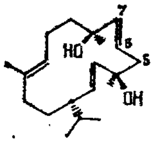


	R_1	R_2
II.	CH_3	OH
III.	OH	CH_3

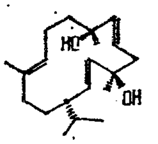


	R_1	R_2
IV.	H	OH
V.	OH	H

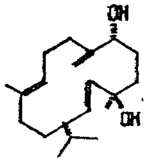
As has been shown [3], reaction at the C_7 double bond of isocembrol leads mainly to the diol (VI), which had previously been isolated by Rowland from tobacco leaves [4]. Since at the present time a fairly large number of cembrane diterpenoids is known in the biosynthesis of which the C_7 double bond is involved [5], we have investigated in more detail the stereochemistry of photo-oxidation at the C_7 double bond of isocembrol and its derivatives. By performing photo-oxidation and reduction of the intermediate hydroperoxides as described previously [2] and chromatographing the products on silica gel we obtained successively unchanged isocembrol (29.3%), then the diols produced by oxidation at the C_{11} double bond [2], and, after these, the diols (VI-IX) with yields of 17.6, 3.2, 5.9, and 1.1% on the isocembrol that had reacted, respectively.



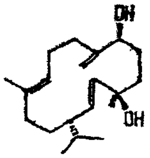
VI



VII



VIII

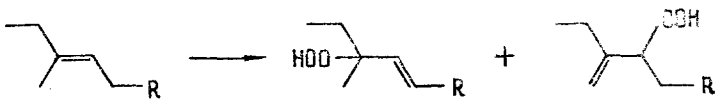


IX

The diol (VI) (mp 150-152°C) was identified by comparing its constants and spectral characteristics with those given in the literature [4], and its epimer (VII) (mp 135-137°C), on the basis of the closeness of its IR spectrum (see the Experimental part) and PMR spectrum (Table 1), with those for the diol (VI). The isomeric structures (II) and (III) were excluded, since they correspond to known compounds [2]. The C_6 double bond in the molecule of the diol (VII) has the trans configuration ($J_{6,7}$ = 15 Hz), and this compound differs from the diol (VI) only by the fact that the asymmetric center present at C_8 has the opposite (R) configuration.

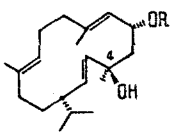
The PMR spectra (Table 2) and the IR spectra of the diols (VIII) and (IX) are very close to one another and are similar to those for the isomeric compounds (IV) and (V) [2]. Their structures, deduced on the basis of the spectra, correspond to the normal products of photo-oxidation at the C_7 double bond on the attack of $^1O_2$ on C_7. The diol (VIII) (oil) gave a crystalline p-nitrobenzoate with mp 127-128°C on the circular dichroism curve of which (for a solution in methanol) a negative Cotton effect was observed at 260 nm, the sign of which corresponds to the R configuration of the C_7 asymmetric center [6]. An additional confirmation of this configuration was obtained by applying Horeau's method [7] to the diol (VIII) — the α-phenylbutyric acid isolated was dextrorotatory. Only one possible structure (7-epi-VIII) remained for the diol (IX) (oil) having, according to its spectra, the same structural fragments as compounds (IV), (V), and (VIII).

Thus, the C_7 double bond of isocembrol, like its C_{11} double bond [2] is attacked by singlet oxygen from both sides, which leads to two pairs of epimeric compounds. However, it must be borne in mind that these two directions of attack lead to different ratios of products: In the one case (S attack) the main product is that with the oxygen atom at C_8 (the 8S-diol (VI)) and in the other (R attack) that with the oxygen at C_7 (the 7R-diol (VIII)). This can be explained by the assumption that in the reacting conformation for which "R attack" is realized the hydrogen atoms at C_6 are present in an unfavorable spatial position with respect to the approaching molecule of singlet oxygen, and a hydrogen atom is split out mainly from the C_8-methyl group. Conversely, in "S attack," for steric reasons the splitting out of a hydrogen atom from the C_6 methylene group with the addition of oxygen at C8 is most favorable. In this connection, it may be mentioned that in the acyclic olefin (X; R = H), which may be regarded as a model compound for the corresponding fragment of the isocembrol molecule, no regiospecific addition of singlet oxygen has been observed — the hydroperoxides (XI) and (XII) were formed in approximately equal amounts (52% and 48%, respectively) [8]:

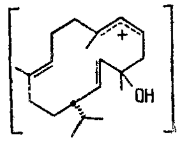


X XI XII

In view of the performance of a biomimetic synthesis of the diol (VI) from isocembrol [3], we cannot fail to mention the potential importance of the intermediate hydroperoxide in the biosynthesis of another cembrane diol — (XIII; R = H), which has also been isolated from tobacco leaves [9]. It is probably capable of undergoing *in vivo* stereoselective isomerization into the hydroperoxide (XIII; R = OH), which is then converted into the corresponding alcohol (XIII; R = H). The possibility of such a stereoselective isomerization has been shown by Lythgoe [10] in *in vitro* experiments for the case of a steroid allyl hydroperoxide. The natural C_4-epimers of the diols (VI) and (XIII) [4, 9] can apparently be obtained similarly from 4-epiisocembrol [11].



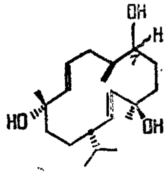
XIII



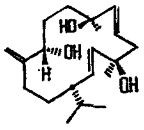
XIV

Enzell [5] has proposed a different scheme for the last stages of the biosynthesis of the diols (VI) and (XIII) and their C_4-epimers which includes in a first stage the epoxidation of cembrene at the C_4 double bond, and the opening of the epoxide ring with the formation of the allyl ion (XIV) the neutralization of which by the OH^- anion should lead to diols (VI) and (XIII) and their epimers. However, this scheme cannot explain the formation of the C_4-epimeric diols (VI) and (XIII), since on epoxidation cembrene gives only one of the two possible C_4-monoepoxides [12].

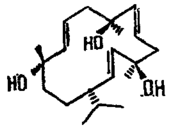
In addition to the diols described above, the photo-oxidation and reduction of isocembrol formed a complex mixture of triols obtained as the result of the further photo-oxidation of the primary hydroperoxides at the remaining trisubstituted double bonds. Under the conditions investigated, these compounds amounted to 34% of the sum of the products. The main ones proved to be the triols (XI), (XVI), and (XVII), which were isolated by chromatography with yields of 5.6, 5.2, and 11.9%, respectively, on the isocembrol that had reacted.



XV



XVI



XVII

The triol (XV) (oil) consisted of a chromatographically inseparable mixture of epimers at C_7 present in a ratio of 3:2 (determined from the ratio of the integral intensities of the noncoincident signals of one of the protons of the exomethylene group in the PMR spectrum present at 4.99 ppm (main epimer) and 5.03 ppm). In favor of the structure (XV) is the formation of the same mixture of epimers in the photo-oxidation of the diol (II), and also the presence in the PMR spectrum of two sets of identical signals, some of which coincide for the two epimers (the signals of the methyl groups, the H_7 and H_{10} atoms, one of the two protons of the exomethylene group, and the methyls of the isopropyl group), while the others are observed separately (Table 3).

The second triol (XVI) was isolated in the form of the 11-O-monoacetate (mp 138-139°C) obtained by the acetylation of the crude fraction with acetic anhydride in pyridine. The structure and stereochemistry of this compound were shown by independent synthesis from the diols (V) and (VI) by their photo-oxidation (followed by acetylation and chromatography of the products).

The structure and stereochemistry of the triol (XVII) (mp 135-137°C) were shown similarly by synthesis from the diols (II) and (VI). In its PMR spectrum, all six of the olefinic protons appeared in the form of a complex multiplet in the 5.30-5.58 ppm region, and

TABLE 3. Chemical Shifts (ppm) and Spin–Spin Coupling Constants (Hz, shown in parentheses) in the PMR Spectra of Compounds (XVa, b)

Compound	H_2	H_3	H_{9a}	H_{9b}
XV* (main epimer)	5,24 dd (15.5; 7.6)	5,35 d (15,5)	2,99 dd (14,5; 6.5)	2 65 dd (14,5; 6.5)
XV* (minor epimer)	5,28 dd (16; 8)	5,38 d (16)	2,93 dd (14.5; 6,5)	2,69 dd (14.5; 6,5)

Compound	H_{10}	H_{11}	$\rangle C{=}C\underline{H}_2$	$-CH(C\underline{H}_3)_2$
XV* (main epimer)	5,46 dt (15; 6,5)	5,63 d (15)	4,84 m 4,99 m	0,77 d (6 0,84 d (6)
XV* (minor epimer)	4,46 dt (15; 6.5)	5,60 d (15)	4,84 m 5,03 m	0,77 d (6) 0,84 d (6))

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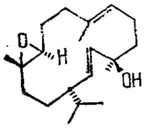
*In addition: tertiary methyl groups giving singlets at 1.21 and 1.25 ppm.

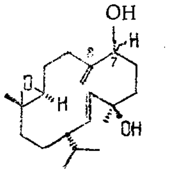
the four allyl protons present at C_6 and C_9 gave a multiplet in the 2.17-2.43 ppm region; the tertiary methyl groups gaves singlets at 1.36, 1.26, and 1.23 ppm and the methyls of the isopropyl groups doublets at 0.80 and 0.85 ppm, J = 6 Hz, each.

When the diol (II) was subjected to photo-oxidation, the triols (XV) and (XVII) were formed in a ratio of 3:4. In this connection it may be mentioned that in comparison with isocembrol the selectivity of oxidation at the C_7 double bond in this case is lower, which is expressed in a decrease in the ratio of the epimers at C_7 (XV) and an increase in the proportion of the latter in the reaction products. The addition of oxygen at C_8 is, as also for isocembrol, a consequence of the predominant "S attack" — the C_8 epimer of the triol (XVII) is formed apparently in small amount, and its presence could be judged only from the PMR spectrum of the mother liquor from the crystallization of a chromatographically pure sample of the triol (XVII) (the multiplet of olefinic protons became somewhat more complex and singlets appeared at 1.26 and 1.34 ppm due to the tertiary methyl groups).

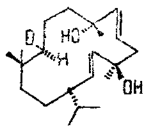
A complete breakdown of selectivity was detected in the photo-oxidation of 11S,12S-epoxyisocembrol (XVIII). The products (XIXa), (XIXb), (XX), and (XXI) were formed in practically equal amounts.



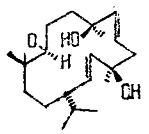
XVIII



XIX a,b



XX



XXI

The epoxydiols (XIXa) and (XIXb) (epimers at C_7) were isolated in the form of a chromatographically inseparable mixture of their 7-O-monoacetates. One of these acetates crystallized from the mixture, forming crystals with mp 133-134°C (for its PMR spectrum, see Table 2). The PMR spectrum of the second epimer, which remained in the mother liquor, differed from the first only by the magnitude of the chemical shift of the H_3 signal (5.61 ppm, doublet, J = 16 Hz). The ratio of these epimers in the mixture was determined from the relative integral intensities of the H_3 signals.

The stereochemistry of the epoxydiol (XXI) (mp 128-129°C) at C_8 was established as S on the basis of its formation from the diol (VI) on epoxidation with peracetic acid. The second ditertiary epoxydiol (XX) had, accordingly, the 8R configuration. Details of the PMR spectra of compound (XX) and (XXI) confirming their structures are given in Table 1.

EXPERIMENTAL

The PMR spectra were recorded on a Bruker WP-200SY instrument (200.13 MHz) for solutions in deuterochloroform (the internal standard was chloroform, the signal of which was taken as 7.4 ppm, δ scale). The other instruments and methods have been described previously [2].

The IR spectra of all the compounds obtained (for solutions in chloroform) contained bands, in identical positions, of a trans-disubstituted double bond (980 cm^{-1}), of a hydroxy group (3610-3620 cm^{-1}), and — for compounds (VIII), (IX), the acetate of the triol (XV), and the acetate of the epoxydiol (XIXa) — the bands of an exomethylene double bond were also observed (905-910, 1650, 3080 cm^{-1}).

Isolation of the Products of the Photo-oxidation of Isocembrol. The photo-oxidation of isocembrol (3.65 g), the working up of the reaction mixture, and chromatography were performed as described previously [2]. In this way we obtained successively 1.07 g of unchanged isocembrol, 1.13 g of products of oxidation at the C_{11} double bond [2], and 0.48 g of the diol (VI) with mp 150-152°C (from acetonitrile), $[\alpha]_D^{20}$ +40.8° (c 6.93) (according to the literature, [4]: mp 150-152°C, $[\alpha]_D^{25}$ +40°); 0.09 g of the diol (VII) with mp 135-137°C (from hexane with diethyl ether), $[\alpha]_D^{20}$ +23.8° (c 1.68), PMR spectrum given in Table 1; 0.16 g of the diol (VIII), oil, PMR spectrum given in Table 2, 7-O-p-nitrobenzoate mp 127-128°C (from hexane with diethyl ether), PMR spectrum: 7.76-8.34 (4 H, multiplet, AA'BB' system of protons of an aromatic ring), 5.44 (1 H, multiplet, H_7), and 5.08 and 4.88 (1 H each; protons of an exomethylene group). We then obtained 0.03 g of the diol (IX), oil, its PMR spectrum being given in Table 2; 0.16 g of a mixture of the epimeric triols (XV), oil, the PMR spectra of the components being given in Table 3; 0.61 g of a mixture of the triols (XVI) and (XVII); and 0.1 g of a mixture of unidentified compounds.

Acetate of the Triol (XVI) and the Triol (XVII). A solution of a mixture of the triols (XVI) and (XVII) obtained as described above (0.61 g) in 10 ml of pyridine was treated with 5 ml of acetic anhydride and the mixture was left at room temperature for 12 h. After the usually working up and chromatography, 0.17 g of the acetate of the triol (XVI) was obtained with mp 138-139°C (from hexane with diethyl ether), $[\alpha]_D^{19}$ +52°C (c 1.73), together with 0.36 g of the triol (XVII) which, after crystallization from the diethyl ether, had mp 135-137°C, $[\alpha]_D^{22}$ +21.8°,(c 6.4).

PMR spectrum of the acetate of the triol (XVI), ppm: 0.81 and 0.84 (3 H each, doublet, J 6 Hz each; methyls of an isopropyl group); 1.22 and 1.35 (3 H, singlets; methyl groups at C_4 and C_8); 2.3 (2 H, multiplet, proton at C_5); 4.86 and 4.88 (1 H each, narrow multiplet, protons of the exomethylene group); 5.22 (1 H, doublet of doublets, H_2, $J_{2,3}$ = 16 Hz, $J_{2,1}$ = 8 Hz); 5.33 (1 H, multiplet (H_{11}); 5.35 (1 H, doublet, H_3, $J_{3,2}$ = 16 Hz); 5.45 (1 H doublet, H_7, $J_{7,6}$ = 15 Hz); 5.48-5.70 (1 H, multiplet of six lines, the B part of a ABLM system formed by the H_7 (A) and H_6 (B) protons and by the two protons at C_5 (L, M).

Photo-oxidation of the Diol (II). A solution of 0.2 g of the diol (II) and 0.02 g of Rose Bengal in 60 ml of methanol was irradiated in a quartz cell with the light of a DRSh-1000 high-pressure mercury lamp at 22°C for 3 h with the passage of air through the solution. After the usual working up [2] and chromatography, 0.04 g of unchanged diol (II), 0.06 g of a mixture of triols (VIII) epimeric at C_7 (the PMR spectrum coincided with that for the product (XV) obtained in the photo-oxidation of isocembrol); and 0.08 g of the triol (XVII) mp 135-137°C, identified by TLC and from its PMR spectrum, were obtained.

Photo-oxidation of the Diols (VI) and (V). This was performed in a similar manner to that of the diol (II). From 0.16 g of compound (VI) were obtained 0.06 g of unchanged diol (VI) and 0.09 g of a fraction which was acetylated with acetic anhydride in pyridine (20°C, 12 h). After the usual working up and chromatography, 0.03 g of the acetate of triol (XVI) (TLC, PMR spectrum) and 0.05 g of the triol (XVII) with mp 135-137°C were isolated.

Similarly, 0.11 g of the diol (V) yielded 0.03 g of the acetate of the triol (XVI) (TLC, PMR spectrum); the other reaction products were not investigated.

Photo-oxidation of 11S,12S-Epoxyisocembrol (XVIII). The photo-oxidation of 1.0 g of compound (XVIII) and the working up of the reaction mixture were carried out under the same conditions as were used for isocembrol [2]. After chromatography, 0.25 g of the initial substance (XVIII); 0.14 g of the epoxydiol (XX) with mp 111-112°C (from hexane with diethyl ether), $[\alpha]_D^{22}$ −17.2° (c 8.13), the PMR spectrum being given in Table 1, and 0.42 g of a mixture of substances giving a single spot in TLC on Silufol were obtained. Its acetylation with acetic anhydride in pyridine (20°C, 12 h) followed by chromatography of the product yielded 0.22 g of a mixture of acetates of the epoxydiols (XIXa and b) and 0.14 g of the epoxydiols (XXI) with mp 128-129°C (from hexane with diethyl ether), $[\alpha]_D^{22}$ −18.7° (c 3.21); its PMR spectrum being given in Table 1.

When a solution of a mixture of the acetates of the epoxydiols (XIXa, b) in petroleum ether containing 10-30% of diethyl ether was kept at 0°C, crystals of one of the epimers (XIXa) with mp 133-134°C deposited from it; its PMR spectrum is given in Table 2; IR spectrum (cm^{-1}): 3620 (OH), 1740 (C=O), 3080, 1650, 910 $(-\underset{/}{C}=CH_2)$, 980 (trans-disubstituted double bond).

The Epoxydiol (XXI) from the Diol (VI). A solution of 0.12 g of the diol (VI) in 20 ml of chloroform cooled to 0°C and stirred was treated with 1.5 g of sodium carbonate, and then 1 ml of a solution of peracetic acid (0.04 g of the peracid) in chloroform was added dropwise. After stirring for 1 h and the usual working up, the product (0.11 g) was chromatographed. This gave 0.08 g of the epoxydiol (XXI) with mp 128-129°C.

SUMMARY

1. The structures of the products of the photo-oxidation of isocembrol and two of its derivatives at the C_7 double bond have been investigated.

2. It has been established that the maximum stereoselectivity of photo-oxidation at the C_7 double bond takes place for isocembrol, and for its derivatives at C_{11} double bond this reaction takes place less selectively.

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