SYNTHESIS OF STEREOISOMERIC 7,7,10a-TRIMETHYL-trans-PERHYDRONAPHTHO[2,1-c]PYRANS AND 6,6,9a-TRIMETHYL-trans-PERHYDRONAPHTHO[2,1-b]FURANS

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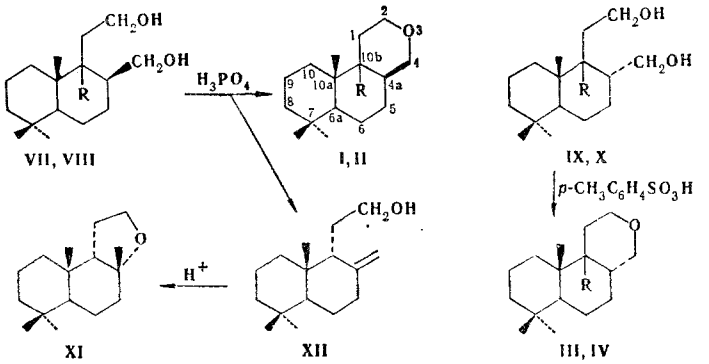
Four diastereomeric [with respect to $C_{(4)}$ and $C_{(10)}$] 7,7,10a-trimethyl-trans-perhydronaphtho[2,1-c]pyrans were synthesized by dehydration of the corresponding 5,5,8a-trimethyl-1-(2-hydroxyethyl)-2-hydroxymethyl-trans-decalins. (1R,2S,8aS)-5,5,8a-Trimethyl-1-(2-hydroxyethyl)-2-hydroxy-trans-perhydronaphthalene, which is dehydrated to give a mixture of epimeric [with respect to $C_{(3a)}$] (9aS,9bR)-6,6,9a-trimethyl-trans-perhydronaphtho[2,1-b]furans, was obtained from (1R,8aS)-5,5,8a-trimethyl-1-(2-hydroxyethyl)-2-methylene-trans-perhydronaphthalene. All of the synthesized oxide compounds have an intense ambergris odor.

In order to study the dependence of the odor on the structure in the decalin series in a continuation of our earlier research [1] we synthesized diastereomeric 7,7,10a-trimethyl-trans-perhydronaphtho[2,1-c]pyrans (I-IV) and epimeric [with respect to $C_{(3a)}$] 6,6,9a-trimethyl-trans-perhydronaphtho[2,1-b]furans (V and VI).

Compounds I-IV were obtained by dehydration of the known diastereomeric 1-(2-hydroxyethyl)-2-hydroxymethyl-trans-decalins VII and VIII [2] and IX and I [3].



I, III, VII, IX R=α-H; II, IV, VIII, X R=β-H

The dehydration of glycols VII and VIII under the influence of reagents that are frequently used for these ends such as p-toluenesulfonic acid in benzene, p-toluenesulfonyl chloride in pyridine, and dimethyl sulfoxide (DMSO) proceeds with difficulty, and the yields of oxides I and II do not exceed 15-28%. Compound I is obtained in good yield (73%) in the dehydration of glycol VII with p-toluenesulfonic acid under more severe conditions (by refluxing in toluene for 80 h). However, orthophosphoric acid was found to be the best dehydrating agent for glycols VII and VIII. In this case oxides I and II are obtained in 89 and 69% yields, respectively.

The known (3aR,9aS,9bS)-3a,6,6,9a-tetramethyl-trans-perhydronaphtho[2,1-b]-furan (XI) [4] was isolated as a side product (6.3%) in the dehydration of diol VIII. It is evidently formed as a result of partial dehydration of glycol VIII to 2-methylene-trans-decalin XII and its subsequent acid-catalyzed cyclization.

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TABLE 1. Characteristics of the Synthesized I-VI

Compound	mp, °C (from methanol)	$[\alpha]_D^{22}$	IR spectrum,* cm^{-1}	Found, %		Empirical formula	Calc., %		Yield, %
				C	H		C	H	
I	60,5—61,5	+17,6°	1104, 1122	81,4	12,1	$C_{16}H_{28}O$	81,3	11,9	89
II	—†	+15,7°	1112, 1145	81,2	12,0	$C_{16}H_{28}O$	81,3	11,9	69
III	51,5—52	+2,5°	1075, 1100, 1127	81,5	11,9	$C_{16}H_{28}O$	81,3	11,9	85
IV	35—36	+21,4°	1108, 1140	81,4	11,7	$C_{16}H_{28}O$	81,3	11,9	96
V	50,5—51,5	+17,1°	1040, 1045, 1053	80,3	11,7	$C_{15}H_{26}O$	81,0	11,8	34
VI	—	−16,7°	1050, 1075, 1093	80,9	11,8	$C_{15}H_{26}O$	81,0	11,8	44

*Frequencies of the vibrations of the tetrahydropyran or tetrahydrofuran ring.
†This compound had n_D^{21} 1.5058.

TABLE 2. Parameters of the PMR Spectra of I-VI

Compound	Solvent	δ, ppm
I	CCl_4	0,92 (6H, s)*; 1,08 (3H, s)*; 3,45 (4H, m, methylene gps. attached to $C_{(2)}$ & $C_{(4)}$)
II	CCl_4	0,85 (6H, s)*; 0,98 (3H, s)*; 2,96 (2H, m); 3,82 (2H, m, CH_2)
III	$CDCl_3$	0,84 (3H, s)*; 0,86 (6H, s)*; 2,90 (1H, t, CH_2, J = 10 Hz); 3,32 (1H, m, CH_2); 3,90 (2H, m, CH_2)
IV	$CDCl_3$	0,85, 0,90, 1,07 (9H, s)*; 3,63 (4H, m, CH_2)
V	CCl_4	0,89 (9H, s, 6- & 9a-CH_3); 3,74 (3H, m, 2- & 3a-H)
VI	CCl_4	0,83 [6H, s, 6-CH_3 (axial) and 9a-CH_3]; 0,88 [3H, s, 6-CH_3 (equatorial)]

*Signals of the 7- and 10a-CH_3 groups.

Let us note that it follows from an examination of molecular models that the B ring in II has a boat conformation.

In contrast to diols VII and VIII, their diastereomers IX and X are dehydrated smoothly by p-toluenesulfonic acid in toluene to give oxides III and IV in 85 and 96% yields, respectively.

The IR spectra of I-IV contain very intense maxima at 1050-1150 cm^{-1} (see Table 1), which are characteristic for the tetrahydropyran ring. Signals of three methyl groups attached to quaternary carbon atoms and multiplet signals of $C_{(2)}$ and $C_{(4)}$ methylene groups (the signal of the former is found at weaker field) are present in their PMR spectra (see Table 2).

A peculiarity of the mass spectra of oxides I-IV is the presence in them of intense peaks of molecular ions (with an intensity of 100% in the spectrum of stereoisomer III) and of an ion with m/z 97 (with an intensity of 100% in the spectra of I, II, and IV). In addition to these peaks, peaks of ions with m/z 221, 180, 151, and 123 (see Table 3), the pathways of the formation of which, which were confirmed by metastable ions in most cases, are presented in the scheme, are found in the mass spectra of oxides I-IV in the high mass number region:

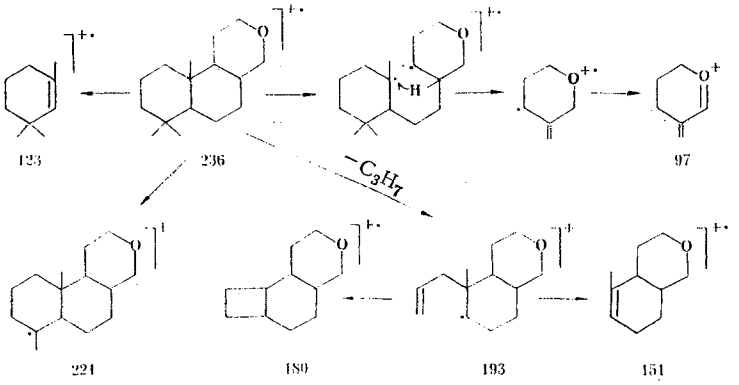


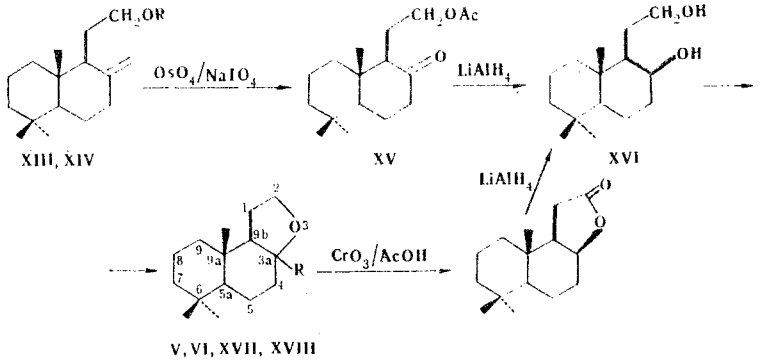
TABLE 3. Mass Spectra of I-VI*

Compound	m/z (relative intensity, %)
I	236 (53), 221 (21), 180 (8), 151 (7), 137 (6), 135 (6), 125 (9), 124 (11), 123 (52), 121 (11), 111 (8), 110 (19), 109 (24), 108 (7), 107 (14), 105 (8), 98 (9), 97 (100), 96 (10), 95 (30), 94 (8), 93 (19), 91 (14), 84 (7), 83 (22), 82 (81), 81 (38), 79 (23), 77 (11), 71 (7), 69 (40), 68 (18), 67 (38), 55 (49), 53 (17), 43 (20), 41 (41), 39 (13)
II	236 (46), 221 (18), 180 (5), 151 (5), 137 (5), 125 (7), 124 (12), 123 (46), 121 (7), 110 (15), 109 (21), 107 (9), 98 (7), 97 (100), 96 (7), 95 (26), 93 (11), 91 (7), 84 (5), 83 (29), 82 (21), 81 (29), 79 (14), 77 (5), 69 (36), 68 (15), 67 (28), 57 (6), 55 (37), 53 (10), 43 (12), 41 (51), 39 (7)
III	236 (100), 221 (58), 207 (5), 180 (5), 177 (5), 152 (11), 151 (25), 135 (6), 138 (6), 137 (6), 125 (11), 124 (16), 123 (44), 122 (9), 121 (10), 111 (7), 110 (18), 109 (21), 108 (6), 107 (13), 105 (7), 98 (9), 97 (79), 96 (12), 95 (29), 94 (7), 93 (16), 91 (10), 83 (19), 82 (18), 81 (30), 80 (5), 79 (77), 77 (9), 69 (24), 68 (17), 67 (28), 57 (5), 56 (5), 55 (32), 53 (13)
IV	236 (89), 221 (29), 180 (7), 177 (8), 151 (15), 137 (6), 135 (7), 125 (11), 124 (17), 123 (54), 122 (9), 121 (13), 111 (8), 110 (20), 109 (26), 108 (10), 107 (21), 105 (11), 98 (9), 97 (100), 96 (14), 95 (39), 94 (9), 93 (23), 91 (13), 84 (7), 83 (25), 82 (27), 81 (45), 80 (6), 79 (25), 77 (9), 71 (11), 70 (10), 69 (46), 68 (20), 67 (42), 57 (9), 56 (10), 55 (54), 53 (17), 44 (5), 43 (24), 42 (6), 41 (73)
V	222 (27), 221 (12), 207 (10), 177 (6), 163 (9), 151 (5), 149 (16), 141 (5), 138 (7), 137 (35), 136 (10), 135 (6), 125 (10), 124 (12), 123 (19), 122 (6), 121 (11), 113 (7), 111 (14), 110 (8), 108 (5), 107 (14), 99 (9), 98 (12), 97 (56), 96 (13), 95 (35), 94 (9), 93 (18), 91 (12), 85 (33), 84 (48), 83 (28), 82 (17), 81 (44), 79 (18), 77 (10), 71 (46), 70 (17), 69 (58), 68 (12), 67 (33), 60 (7), 57 (74), 56 (21), 55 (78), 53 (16), 45 (12), 44 (17), 43 (78), 41 (100)
VI	222 (100), 221 (65), 208 (10), 207 (52), 205 (6), 179 (6), 177 (4), 163 (11), 149 (4), 147 (11), 139 (4), 138 (12), 137 (65), 136 (15), 135 (6), 133 (6), 124 (17), 123 (48), 122 (6), 121 (13), 119 (6), 112 (4), 111 (11), 110 (22), 109 (35), 108 (8), 107 (17), 106 (4), 105 (9), 98 (13), 97 (74), 96 (13), 95 (54), 94 (11), 93 (22), 92 (4), 91 (14), 85 (17), 84 (48), 83 (13), 81 (54), 80 (6), 79 (22), 77 (13), 71 (8), 70 (7), 69 (52), 68 (13), 67 (44), 66 (4), 65 (7), 57 (9), 56 (8), 55 (54), 54 (7), 53 (17), 44 (7), 43 (17), 41 (70)

*The peaks of ions with intensities greater than 3% of the maximum peak are presented.

(1R,8aS)-5,5,8a-Trimethyl-1-(2-hydroxyethyl)-2-methylene-trans-perhydronaphthalene (XIII) [5] served as the starting substance for the synthesis of (3aS,9aS,9bR)- and (3aR,9aS,9bR)-6,6,9a-trimethyl-trans-perhydronaphtho[2,1-b]furans (V and VI). Acetylation of XIII with acetic anhydride in pyridine gives acetate XIV, which is oxidized by osmium tetroxide and sodium periodate to (1R,8aS)-5,5,8a-trimethyl-1-(2-acetoxyethyl)-2-oxo-trans-perhydronaphthalene (XV), the structure of which was proved on the basis of spectral data. Absorption bands that are characteristic for a keto group in a six-membered ring and acetate and gem-dimethyl groups are present in its IR spectrum, while the PMR spectrum contains signals of three methyl groups attached to quaternary carbon atoms and from an acetate group.

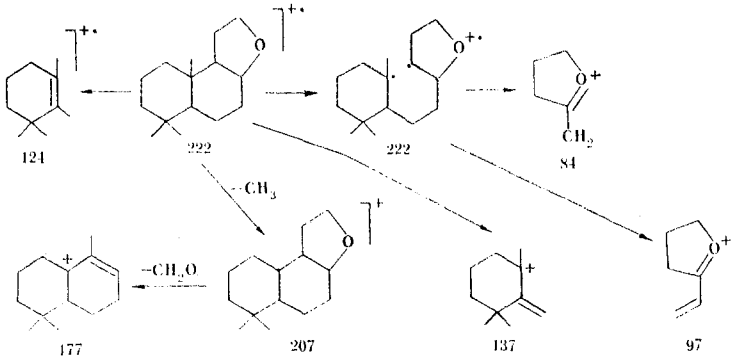
The reduction of keto acetate XV with lithium aluminum hydride proceeds stereoselectively from the less hindered β side of the molecule and leads to the known (1R,2S,8aS)-5,5,8a-trimethyl-1-(2-hydroxyethyl)-2-hydroxy-trans-perhydronaphthalene (XVI) [5].



V R=α-H; VI R=β-H; XIII R=H; XIV R=Ac; XVII R=α-CH_3; XVIII R=β-CH_3

The dehydration of diol XVI with p-toluenesulfonic acid gives a mixture of oxides V and VI, which was separated by column chromatography on silica gel. The structure and stereochemistry of V and VI were proved on the basis of the following data. Three intense bands that are characteristic for the tetrahydrofuran ring are present in their IR spectra at 1000-1100 cm^{-1}. Signals of three methyl groups attached to quaternary carbon atoms appear in the

PMR spectra along with multiplet signals at weaker field (3.3-3.75 ppm) of three protons attached to carbon atoms bonded to an ether oxygen atom. In the PMR spectrum of oxide V the signal of the 9a-CH_3 group appears at weaker (by 0.06 ppm) field as compared with the signal of the same group in its epimer VI, which indicates that it is deshielded by the axially oriented oxygen atom attached to $C_{(3a)}$, i.e., oxide V had a 3aS configuration, while oxide VI has a 3aR configuration. This conclusion was confirmed by chemical means. Diol XVI was obtained by oxidation of V with chromic anhydride under the conditions in [6], in which tetrahydrofuran derivatives are oxidized to the corresponding γ-lactones without isomerization, and subsequent reduction of the oxidation product with lithium aluminum hydride.



The structures of V and VI are confirmed by their mass spectra, which differ from one another with respect to the intensities of the peaks of several ions. Their spectra contain characteristic (for two-ring compounds similar to oxides V and VI [7]) peaks of ions with m/z 177, 137, 123, and 109, as well as peaks of ions with m/z 222 (M^+), 207 ($M-CH_3$), and 97.

Oxides V and VI have an ambergris odor of the same type as their higher homologs XVII and XVIII but with lower intensity. Diastereomeric perhydronaphthopyrans I-IV do not satisfy the requirements of Ohloff's rule [8]; however, they have an intense ambergris odor.

EXPERIMENTAL

See [1] for the individual details of the experimental procedure. The PMR spectra of the compounds were recorded with a Varian XL-100 spectrometer relative to tetramethylsilane. The mass spectra were obtained with an MKh-1303 mass spectrometer with a glass system for direct introduction of the samples directly into the ion source at an ionizing voltage of 70 eV. Analysis by gas-liquid chromatography (GLC) was carried out with a Khrom-4 chromatograph with a flame-ionization detector. Neutral activity 3.5 aluminum oxide, 100/160 μ silica gel, and silica gel impregnated with silver nitrate prepared by the method in [9] were used for column chromatogarphy.

(4aS,10aS,10bS)-7,7,10a-Trimethyl-trans-perhydronaphtho[2,1-c]pyran (I). A mixture of 200 mg (0.79 mmole) of diol VII and 80 mg (0.69 mmole) of 85% phosphoric acid was heated in a sealed test tube at 138-142°C for 4 h, after which it was cooled and treated with water, and the aqueous mixture was extracted with petroleum ether. The extract was washed successively with water, sodium bicarbonate solution, and water, dried, and filtered. The solvent was removed by distillation to give 164 mg of I. The physicochemical characteristics of this and the other compounds that we obtained are presented in Tables 1-3.

(4aS,10aS,10bR)-7,7,10a-Trimethyl-trans-perhydronaphtho[2,1-c]pyran (II). A solution of 640 mg (2.5 mmole) of VIII in 1.2 g (10.4 mmole) of 85% phosphoric acid was heated in a sealed test tube at 138-142°C for 3 h (with monitoring by TLC). It was then worked up as described above to give 523 mg of a liquid substance containing, according to GLC [glass column with a length of 1.2 m and a diameter of 0.35 cm, Celite-545 (45-60 mesh) containing 1.5% OV-17 stationary phase as the solid support, helium as the carrier gas, flow rate 35 ml/min, column temperature 132°C], 86% oxide II, 6.3% tetrahydrofuran derivative XI, and 6.4% of a mixture of four substances with lower polarities. The reaction product was chromatographed with a column filled with 10 g of silica gel impregnated with silver nitrate. Elution with petroleum ether gave three fractions. The first fraction (160 mg) contained oxide II and an impurity with a lower polarity, the second fraction (310 mg) was II, and the third fraction (60 mg) was a mixture of oxide II with a substance with a higher polarity. The second fraction was dissolved in petroleum ether, and the solution was treated with activated charcoal and filtered. The solvent was removed by distillation, and the residue was dried *in vacuo* (2 mm)

for 3 h and then in a vacuum desiccator over P_2O_5 and paraffin to give liquid oxide II (see Tables 1-3). The first fraction was rechromatographed with a column filled with 13 g of the same adsorbent to give 65 mg of oxide II. Repeated chromatography of the third fraction on the same adsorbent gave another 35 mg of oxide II and 7 mg of XI, which was identified by chromatographic and spectral comparison with a genuine sample.

(4aR,10aS,10bS)-7,7,10a-Trimethyl-trans-perhydronaphtho[2,1-c]pyran (III). A solution of 200 mg (0.79 mmole) of glycol IX and 2 mg (0.01 mmole) of p-toluenesulfonic acid in 10 ml of dry toluene was refluxed in a Dean–Stark apparatus for 4 h (with monitoring by TLC), after which the toluene was removed by vacuum distillation, and the residue (198 mg) was dissolved in a small amount of petroleum ether and chromatographed with a column filled with 4 g of silica gel. Gradient elution with petroleum ether–benzene gave 158.8 mg of III (see Tables 1-3).

(4aR,10aS,10bR)-7,7,10a-Trimethyl-trans-perhydronaphtho[2,1-c]pyran (IV). A 1-mg (0.005 mmole) sample of p-toluenesulfonic acid was added to a solution of 51.5 mg (0.2 mmole) of X in 2 ml of dry toluene, after which the toluene was removed by distillation at normal pressure in the course of 10 min. The residue was chromatographed with a column filled with 4 g of aluminum oxide by elution with petroleum ether to give 46.3 mg of oxide IV (see Tables 1-3).

(1R,8aS)-5,5,8a-Trimethyl-1-(2-hydroxyethyl)-2-methylene-trans-perhydronaphthalene Acetate (XIV). A 450-mg (4.41 mmole) sample of freshly distilled acetic anhydride was added to a solution of 400 mg (1.69 mmole) of 1-(2-hydroxyethyl)perhydronaphthalene XIII [5] in 7 ml of dry pyridine, and the mixture was maintained at normal temperature for 3 h. It was then treated with 35 ml of water, and the aqueous mixture was extracted with diethyl ether. The extract was washed successively with 10% sulfuric acid, water, sodium bicarbonate solution, and water, dried, and filtered. The ether was removed by vacuum distillation to give 430 mg (91%) of acetate XIV in the form of a colorless viscous liquid with n_D^{21} 1.4990 and $[\alpha]_D^{21}$ +37° (c 1.9). IR spectrum (thin layer): 890, 1655 (C=C); 1366, 1380 [$C(CH_3)_2$]; 1740 cm^{-1} (OAc). Found: C 78.3; H 10.9%. $C_{18}H_{30}O_2$. Calculated: C 77.6; H 10.9%.

(1R,8aS)-5,5,8a-Trimethyl-1-(2-acetoxyethyl)-2-oxo-trans-perhydronaphthalene (XV). A 1.8-ml sample of water, 1.2 ml of glacial acetic acid, and 12 mg (0.05 mmole) of osmium tetroxide were added to a solution of 360 mg (1.29 mmole) of acetate XIV in 25 ml of dioxane, and the solution was allowed to stand at normal temperature for 30 min until it became turbid. A 607-mg (2.84 mmole) sample of finely ground sodium periodate was added, and the mixture was stirred at the same temperature for 22 h. Water (50 ml) was added to the mixture, and the solution was extracted with ether. The extract was washed successively with water, sodium bicarbonate solution, and water, dried, and filtered. The ether was removed by vacuum distillation, and the residue (350 mg) was chromatographed with a column filled with 8 g of silica gel. Elution under slight pressure [10] with petroleum ether–ethyl acetate (9:1) gave the following three fractions (in the order of increasing polarity). Workup of fraction 1 gave 66 mg of the starting acetate. Workup of fraction 2 gave 225 mg (76% based on the unchanged starting acetate) of XV with $[\alpha]_D^{20}$ −15.3° (c 1.1). IR spectrum (thin layer): 1370, 1380 [$C(CH_3)_2$]; 1715 (C=O); 1740 cm^{-1} (OAc). PMR spectrum (CCl_4): 0.71 (3H, s, axial 5-CH_3), 0.87 (3H, s, equatorial 5-CH_3), 0.97 (3H, s, 8a-CH_3), 1.87 (3H, s, OAc), and 3.71 ppm (2H, m, 2-H). Found: C 72.6; H 10.1%. $C_{17}H_{28}O_3$. Calculated: C 72.8; H 10.1%. Workup of fraction 3 gave 35 mg of a more polar substance, which was not further investigated.

(1R,2S,8aS)-5,5,8a-Trimethyl-1-(2-hydroxyethyl)-2-hydroxy-trans-perhydronaphthalene (XVI). A) A 105-mg (2.76 mmole) sample of lithium aluminum hydride was added to a solution of 220 mg (0.79 mmole) of keto acetate XV in 10 ml of absolute ether, and the mixture was allowed to stand at ordinary temperature for 4 h until the reaction was complete (as monitored by TLC). The excess lithium aluminum hydride was decomposed with ethyl acetate, and the reaction mixture was worked up in the usual manner to give 184 mg (98%) of perhydronaphthol XVI with mp 187-189°C (from petroleum ether) and $[\alpha]_D^{20}$ +41.5° (c 3.2; CH_3OH). Found: C 75.1; H 11.8%. $C_{15}H_{28}O_2$. Calculated: C 75.0; H 11.7%. According to [5], this compound has mp 182-183°C and $[\alpha]_D^{20}$ +39.7° (c 2.5).

B) A solution of 160 mg (1.6 mmole) of chromium trioxide in 1 ml of 98% acetic acid was added to a solution of 10.2 mg (0.05 mmole) of oxide V (see below) in 2 ml of glacial acetic acid, and the mixture was heated at 50-52°C [6] for 3.5 h until the reaction was complete (as monitored by TLC). The reaction mixture was diluted with water and extracted with ether, and the ether extract was washed successively with water, sodium bicarbonate solution, and water, dried, and filtered. The ether was removed by distillation *in vacuo* to give 6.1 mg

of an individual compound (according to TLC). This compound was dissolved in 1 ml of absolute ether, the solution was treated with 10 mg (0.26 mmole) of lithium aluminum hydride, and the mixture was allowed to stand at normal temperature until the reaction was complete (1.5 h). It was then worked up in the usual way to give 4.3 mg (39%) of diol XVI with mp 186-188.5°C (from petroleum ether). No melting-point depression was observed for a mixture of this product with a genuine sample of diol XVI.

Dehydration of Diol XVI. A 180-mg (1.07 mmole) sample of p-toluene-sulfonic acid was added to a solution of 450 mg (1.88 mmole) of XVI in 50 ml of dry benzene, and the solution was refluxed in a Dean–Stark apparatus for 30 min until the reaction was complete (as monitored by TLC). It was then cooled, treated with an equal volume of ether, washed successively with water, sodium bicarbonate solution, and water, dried, and filtered. The ether was removed *in vacuo*, and the residue (409 mg) was chromatographed under slight pressure [10] with a column packed with 90 g of silica gel. Elution with petroleum ether–ethyl acetate (97:3) gave fractions with increasing polarities. The least polar fraction (47.4 mg) was a mixture of hydrocarbons and was not investigated further. Fraction No. 2 [140.7 mg (34%)] was (3aS,9aS,-9bR)-trimethyl-trans-perhydronaphtho[2,1-b]furan (V) (see Tables 1-3), while fraction No. 3 (181.1 mg of a colorless viscous liquid) was its (3aR,9aS,9bR) stereoisomer VI (see Tables 1-3).

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