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Camphor and Isocamphanone in the Synthesis of Disubstituted Acetylene Alcohols, Ethers, and Esters

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Abstract—By treating 1-octyne and phenylacetylene with butyllithium the corresponding lithium acetylides were obtained that, with camphor and isocamphanone, provided along streospecific process 2-*exo*-(1-octynyl or 2-phenyl-1-ethynyl)-2-*endo*-lithiumoxy-5,5,6-trimethylbicyclo[2.2.1]heptane and 2-*endo*-(1-octynyl or 2-phenyl-1-ethynyl)-2-*exo*-lithiumoxy-1,7,7-trimethylbicyclo[2.2.1]heptane. The hydrolysis of these lithium alcoholates occurred selectively and resulted in individual tertiary terpene alcohols containing *exo*-acetylene substituent in the case of camphor, *endo*-acetylene fragment in the case of isocamphanone. The alcohols reacted with methyl, ethyl, or butyl iodides in the presence of hexamethylphosphoramide to afford ethers, and with benzoyl chloride to furnish disubstituted esters of benzoic acid.

The interest to chemical transformations of terpene compounds is due to the availability and chemical lability of this compounds class. As a result, it is possible to prepare therefrom the products of versatile structures. Acetylene-containing terpene derivatives do not exist in the nature and are not described in the literature [1, 2]. However quite a number of medicines is known to contain acetylene fragments, in particular, substituted acetylene alcohols possess a wide range of biological activity [3]. Therefore the building into the structure of the natural biologically active compounds a pharmacophoric acetylene moiety providing new potentially bioactive substances is an urgent problem.

The target of this study was a synthesis of a multipurpose synthon that would permit preparation of previously unknown individual isomeric disubstituted acetylene alcohols, ethers, and esters containing in the structure a bicyclic terpene moiety from the known drug, camphor [4], or its closest analog, isocamphanone.

We showed previously [5, 6] that as such synthons could be used highly reactive lithium alcoholates of various structures. They were brought into reaction *in statu* nascendi, without isolation, and they were able to undergo further transformation conserving the initial configuration [7, 8]. In this study we carried out for the first time a stereospecific synthesis of 2-*exo*-(1-octynyl or 2-phenyl-1-ethynyl)-2-*endo*-lithiumoxy-5,5,6-trimethylbicyclo[2.2.1]heptane (**IVa, b**) and 2-*endo*-(1-octynyl or 2-phenyl-1-ethynyl)-2-*exo*-lithiumoxy-1,7,7-trimethylbicyclo[2.2.1]heptane (**VIIIa, b**) from lithium acetylides (**IIa, b**) prepared by treating with butyllithium 1-octyne (**Ia**) or phenylacetylene (**Ib**) reacted with camphor (**IIIb**) or isocamphanone (**IIIa**).

The alcoholates thus prepared **IVa, b** and **VIIIa, b** were hydrolysed with water without separation from the reaction mixture. The individual isomeric tertiary terpene alcohols were isolated in 68—84% yield. The acetylene fragment was in endo-position in alcohols **Va, b** obtained from the isocamphanone, and in *exo*-position in alcohols **IXa, b** from camphor.

The structure of compounds obtained was confirmed by IR and NMR spectra. In their IR spectra are lacking the absorption bands of C=O group and C_{sp}–H bond, and appear characteristic bands of the stretching vibrations of the C≡C bond at 2220 cm^{-1} and of an associated hydroxy group at 3455 cm^{-1}. In the ^1H NMR spectra of alcohols **Va, b** and **IXa, b** are present proton signals both from the bicyclic moiety and from the substituents in the side chain (Table 1), and the pattern of the spectra is characteristic of individual isomers evidencing the selectivity of the reaction. However the ^1H NMR spectra are not sufficient for unambiguous location of the tertiary hydroxy group.

The determination of the spatial arrangement of the acetylene alcohols **V, IX** was carried out with the use of ^{13}C NMR spectra by comparison of the experimental and calculated chemical shifts. The calculations were based on the chemical shifts of model

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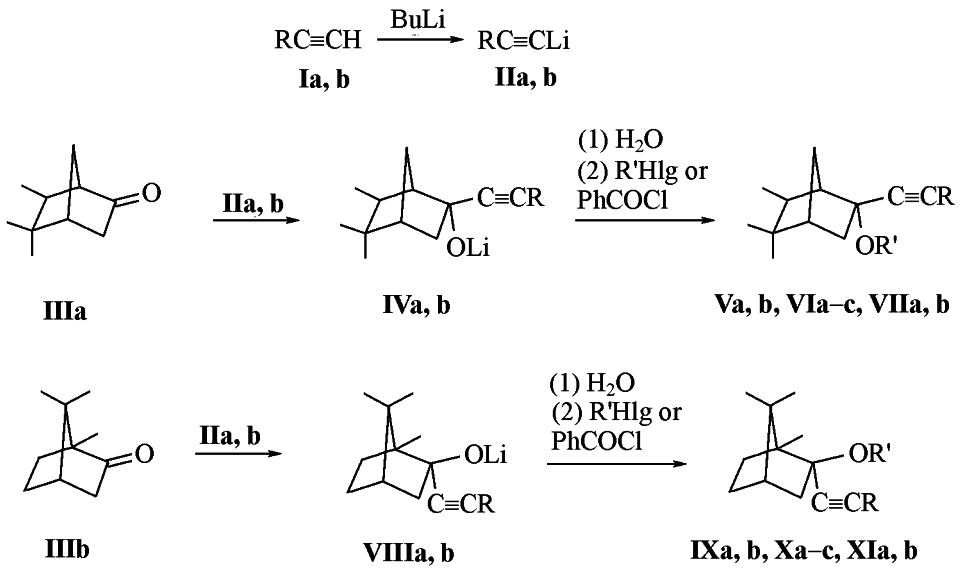

I, II, IV, V, VII–IX, XI, R = $(CH_2)_5CH_3$ (**a**), Ph (**b**); **V, IX**, R' = H; **VI** < **X**, R = $(CH_2)_5CH_3$ (**a**–**c**), Ph (**d**–**f**); R' = CH_3 (**a, d**), $(CH_2)_3CH_3$ (**c, f**); **VII, XI**, R' = COPh.

compounds, derivatives of [2.2.1]heptane [9–11]. The chemical shift of C^2 atom in the synthesized tertiary alcohols is 80 ppm. The comparison of the spectra of alcohols **V, IX** with those of borneol, isoborneol, 2-*exo*- and 2-*endo*-methylborneols [12], 2-methyl- and 2-phenylisoborneols [13] indicated that hydroxy group at the C^2 atom had endo-orientation in the case of alcohols **V** obtained from isocamphanone, and *exo*-orientation for alcohols **IX** prepared from camphor. Therefore the acetylene fragment was located *exo* in alcohols **V** and *endo* in alcohols **IX**. The configuration of the substituents at the C^2 atom is deduced from the chemical shifts of C^3, C^6, and C^{8-10} atoms that are sensitive to the change from *endo* to *exo* orientation of the substituents due to the alteration of the 1,4-through-space interaction [7, 8].

Lithium alcoholates **IVa, b** and **VIIIa, b** readily react with methyl, ethyl, and butyl iodides in the presence of hexamethylphosphoramide (HMPA) to afford the corresponding ethers **VIa–f, Xa–f**) in 65–85% yield. Without HMPA the reaction does not proceed. In the IR spectra of the ethers appears the absorption band of stretching vibrations of the C–O–C bonds at 1090 cm^{-1}. The ^1H NMR spectra of the ethers unlike those of the initial compounds contain the signal from OAlk groups at 3.5 ppm. Since it had been shown before [7, 8] that configuration of the substituents remained the same in the course of this reaction, the *endo*-configuration was assigned to ethers **VI**, and to ethers **X** *exo*-configuration.

Lithium alcoholates **IV, III** with benzoyl chloride afforded esters **VIIa, b, XIa, b** in 63–78% yield. In the IR spectra of the esters the stretching vibrations band of carbonyl group appears at 1730 cm^{-1}. To the phenyl groups in the IR spectra of these compounds correspond three bands of stretching vibrations from C–H in the benzene ring at 3080, 3060, and 3030 cm^{-1}, and bands of bending vibrations of the benzene ring at 1600, 1580, 1500, 755, and 700 cm^{-1}. In the ^1H NMR spectra of compounds **VII, XI** the phenyl signals are present downfield at 7–8 ppm.

Compounds obtained are either colorless fluids **Va, VIa–f", VIIa, IXa, Xa–f, XIa** or crystalline compounds **Vb, VIIb, XIb** well soluble in the common organic solvents and insoluble in water. Their ^1H NMR spectra are presented in Table 1, physical characteristics and yields in Table 2.

UV spectra of compounds obtained contain the following absorption bands λ_{max}, nm (ε): 204±2 (300±100) – (**Va, IXa, Xc**); 204±3 (25000±2000), 241±1 (23000±3000), 252±1 (12000±2000) – (**Vb, VId–f, VIIb, IXb, Xd–f**); 204±1 (200±100), 235±5 (200±150) – (**VIa–c, Xa, b**); 203 (12000), 231 (24000) – (**XIa**); 204 (30000), 237 (29000), 242 (28000), 253 (22000) – (**XIb**).

EXPERIMENTAL

IR spectra were measured on IR Fourier spectrometer Protege-460 of Nicolet Co. ^1H NMR spectra

Table 1. ^{1}H NMR spectra of compounds **Va, b, VIa–f, VIIa, b, IXa, b, Xa–f, XIa, b**

Compd. no.	δ, ppm
Va	0.80–1.05 m (12H, Me_2C^5, MeC^6, $\underline{Me}CH_2$), 1.05–2.10 m [16H, OH, 3CH, CH_2, $(CH_2)_4$], 2.18 (2H, $CH_2C{\equiv}C$)
Vb	0.89 d (3H, MeC^6, $^3JH^1$–H 7.0 Hz), 0.92 s (3H, MeC^5-*endo*), 1.09 s (3H, MeC^5-*exo*), 1.55–2.10 m [7H, OH, 2CH, $2CH_2$], 2.16 br.s (1H, HC^1), 7.20–7.45 m (5H, Ph)
VIa	0.77–1.03 m (12H, Me_2C^5, MeC^6, $\underline{Me}CH_2$), 1.03–2.02 m [15H, 3CH, $2CH_2$, $(CH_2)_4$], 2.21 t (2H, $CH_2C{\equiv}C$, 3.26 s (3H, MeO)
VIb	0.77–1.03 m (12H, Me_2C^5, MeC^6, $\underline{Me}CH_2$), 1.20 t (3H, $MeCH_2O$), 1.20–2.03 m [15H, 3CH, $2CH_2$, $(CH_2)_4$], 2.21 t (2H, $CH_2C{\equiv}C$), 3.17–3.80 m (2H, CH_2O)
VIc	0.77–1.03 m (15H, Me_2C^5, MeC^6, $2\underline{Me}CH_2$), 1.15–2.03 m [19H, 3CH, $2CH_2$, $(CH_2)_2$, $(CH_2)_4$], 2.22 t (2H, $CH_2C{\equiv}C$), 3.15–3.70 m (2H, CH_2O)
VId	0.88 d (3H, MeC^6, $^3J_{H^1\text{–}H}$ 7.0 Hz), 0.91 s (3H, MeC^5-*endo*), 1.03 s (3H, MeC^5-*exo*), 1.60–2.03 m [6H, 2CH, $2CH_2$], 2.16 br.s (1H, HC^1), 3.36 s (3H, MeO), 7.22–7.50 m (5H, Ph)
VIe	0.88 d (3H, MeC^6, $^3J_{H^5}1_{\text{–}H}$ 7.0 Hz), 0.91 s (3H, MeC^5-*endo*), 1.04 s (3H, MeC^5-*exo*), 1.22 t (3H, $\underline{Me}CH_2$), 1.54–2.05 m [6H, 2CH, $2CH_2$], 2.17 br.s (1H, HC^1), 3.28–3.92 m (2H, CH_2O), 7.22–7.48 m (5H, Ph)
VIf	0.88 d (3H, MeC^6, $^3J_{H^1\text{–}H}$ 7.0 Hz), 0.91 s (3H, MeC^5-*endo*), 0.93 t (3H, $MeCH_2$), 1.04 s (3H, MeC^5-*exo*), 1.25–2.05 m [10H, 2CH, $2CH_2$, $(CH_2)_2$], 2.16 br.s (1H, HC^1), 3.25–3.81 m (2H, CH_2O), 7.20–7.48 m (5H, Ph)
VIIa	0.75–1.03 d (12H, Me_2C^5, MeC^6, $\underline{Me}CH_2$), 1.07–2.05 m [15H, 3CH, $2CH_2$, $(CH_2)_4$], 2.22 t (2H, $CH_2C{\equiv}C$), 7.30–8.15 m (5H, Ph)
VIIb	0.89 d (3H, MeC^6, $^3J_{H^1\text{–}H}$ 7.0 Hz), 0.91 s (3H, MeC^5-*endo*), 1.03 s (3H, MeC^5-*exo*), 1.60–2.40 m [6H, 2CH and $2CH_2$], 2.75 br.s (1H, HC^1), 7.15–8.12 m (10H, 2Ph)
IXa	0.83 s, 0.88 t, 0.92 s (9H. Me_2C^7, $\underline{Me}CH_2$), 1.05 s (3H, MeC^1), 1.10–2.10 m [16H, OH, CH, $3CH_2$, $(CH_2)_4$], 2.21 t (2H, $CH_2C{\equiv}C$)
IXb	0.86 s, 0.98 s, 1.08 s (9H, Me_2C^7, MeC^1), 1.06–2.40 m (8H, OH, CH, $3CH_2$), 7.20–7.48 m (5H, Ph)
Xa	0.84 s, 0.88 t and 0.90 s (9H, Me_2C^7, $\underline{Me}CH_2$), 0.98 s (3H, MeC^1), 1.02–2.07 m [15H, CH, $3CH_2$, $(CH_2)_4$], 2.23 t (2H, $CH_2C{\equiv}C$), 3.24 s (3H, MeO)
Xb	0.84 s, 0.89 t, 0.91 s [9H, Me_2C^7, $\underline{Me}(CH_2)_5$], 1.00 s (3H, MeC^1), 1.11 t (3H, $MeCH_2$), 1.15–2.10 m [15H, CH, $3CH_2$, $(CH_2)_4$], 2.22 t (2H, $CH_2C{\equiv}C$), 3.32–3.87 m (2H, CH_2O)
Xc	0.83 s, 0.89 s, 0.90 t, 1.02 s (15H, Me_2C^7, $2\underline{Me}CH_2$, MeC^1), 1.03–2.15 m [19H, CH, $3CH_2$, $(CH_2)_2$, $(CH_2)_4$], 2.21 t (2H, $CH_2C{\equiv}C$),
Xd	0.89 s, 0.98 s and 1.03 s (9H, Me_2C^7, MeC^1), 1.10–2.45 m (7H, CH, $3CH_2$), 3.34 s (3H, MeO), 7.20–7.52 m (5H, Ph)
Xe	0.91 c, 1.00 c, 1.08 c (9H, Me_2C^7, MeC^1), 1.10–2.50 m (7H, CH and $3CH_2$), 1.19 t (3H, $\underline{Me}CH_2$), 3.35–3.85 m (2H, CH_2O), 7.17–7.50 m (5H, Ph)
Xf	0.88–1.10 m (12H, Me_2C^7, $\underline{Me}CH_2$, MeC^1), 1.10–2.50 m [11H, CH, $3CH_2$, $(CH_2)_2$], 3.30–3.80 m (2H, CH_2O), 7.12–7.50 m (5H, Ph)
XIa	0.88 t (3H, $\underline{Me}CH_2$), 0.89 s, 0.96 s, 1.10 s (9H, Me_2C^7, MeC^1), 1.10–2.10 m [15H, CH, $3CH_2$, $(CH_2)_4$], 2.20 t (2H, $CH_2C{\equiv}C$), 7.26–8.15 m (5H, Ph)
XIb	1.00 s, 1.11 s, 1.26 s (9H, Me_2C^7, MeC^1), 1.28–2.80 m (7H, CH and $3CH_2$), 7.19–8.12 m (10H, 2Ph)

Table 2. Characteristics of compounds **Va, b, VIa–f, VIIa, b, IXa, b, Xa–f, XIa, b**

Compd. no.	Yield, %	bp, °C (*p*, mm Hg), mp, °C	d_{20}^{20}	n_D^{20}	Found, %		Formula	Calculated, %		*M*	
					C	H		C	H	found	calculated
Va	81	104–105 ($5–10^{-2}$)	0.9187	1.4925	82.61	11.58	$C_{18}H_{30}O$	82.38	11.52	255.8	262.4
Vb	84	72–73	–	–	85.16	8.77	$C_{18}H_{22}O$	84.99	8.72	244.4	254.4
VIa	79	90–91 ($5–10^{-2}$)	1.0171	1.4760	82.64	11.70	$C_{19}H_{32}O$	82.55	11.67	271.4	276.5
VIb	72	100–101 ($5–10^{-2}$)	0.9776	1.4735	82.70	11.80	$C_{20}H_{34}O$	82.73	11.87	281.7	290.5
VIc	69	127–128 ($5–10^{-2}$)	0.8823	1.4745	83.12	12.07	$C_{22}H_{38}O$	82.95	12.02	311.6	318.5
VId	74	103–104 ($5–10^{-2}$)	1.0356	1.5465	85.12	9.10	$C_{19}H_{24}O$	85.03	9.01	260.3	268.4
VIe	71	111–112 ($5–10^{-2}$)	1.0145	1.5375	85.11	9.40	$C_{20}H_{26}O$	85.06	9.28	271.6	282.4
VIf	65	128–129 ($5–10^{-2}$)	1.1380	1.5310	85.21	9.83	$C_{22}H_{30}O$	85.11	9.74	304.3	310.5
VIIa	78	175–176 ($5–10^{-2}$)	1.1116	1.5210	82.11	9.35	$C_{25}H_{34}O_2$	81.92	9.35	358.3	366.5
VIIb	64	95–96	–	–	83.91	7.43	$C_{25}H_{26}O_2$	83.76	7.31	347.1	358.5
IXa	68	103–104 ($5–10^{-2}$)	1.0053	1.4840	82.50	11.63	$C_{18}H_{30}O$	82.38	11.52	254.6	262.4
IXb	80	125–126 ($5–10^{-2}$)	1.0668	1.5600	85.11	8.81	$C_{18}H_{22}O$	84.99	8.72	245.3	254.4
Xa	67	95–96 ($5–10^{-2}$)	1.0457	1.4795	82.59	11.71	$C_{19}H_{32}O$	82.55	11.67	269.3	276.5
Xb	71	98–98 ($5–10^{-2}$)	1.0075	1.4755	82.77	11.84	$C_{20}H_{34}O$	82.73	11.87	282.0	290.5
Xc	74	108–109 ($5–10^{-2}$)	0.9345	1.4730	83.04	12.06	$C_{22}H_{38}O$	82.95	12.02	310.1	318.5
Xd	81	102–103 ($5–10^{-2}$)	1.0382	1.5505	85.13	9.04	$C_{19}H_{24}O$	85.03	9.01	257.8	268.4
Xe	75	105–106 ($5–10^{-2}$)	0.9974	1.5410	85.18	9.41	$C_{20}H_{26}O$	85.06	9.28	270.4	282.4
Xf	82	115–116 ($5–10^{-2}$)	1.0681	1.5305	85.20	9.84	$C_{22}H_{30}O$	85.11	9.74	303.1	310.5
XIa	63	186–187 ($5–10^{-2}$)	1.1165	1.5165	82.04	9.43	$C_{25}H_{34}O_2$	81.92	9.35	359.3	366.5
XIb	78	131–132	–	–	83.84	7.33	$C_{25}H_{26}O_2$	83.76	7.31	350.4	358.5

were registered on spectrometers Tesla BS-567A (100 MHz) and Bruker AC-300 (300 MHz) from 2–5% solutions in $(CD_3)_2SO$, internal reference TMS. The ^{13}C NMR spectra were recorded on spectrometer Tesla BS-587 A at operating frequency 20 MHz. Mass spectra were taken on Chrommass GC/MS Hewlett Packard 5890/5972 instrument, column HP-5MS (70 eV), eluent dichloromethane. UV spectra were registered on spectrophotometer Specord UV Vis from solutions in methanol of concentration 1.10^{-2} M for compounds **Va, VI a–c, IXa, Xa–c** or 1.10^{-4} M for compounds **Vb, VId–f, VIIa, b, IXb, Xd–f, XIa, b**. Molecular weight of compounds was measured cryoscopically in benzene. For column chromatography was used neutral Al_2O_3 of **II** grade Brockmann activity.

Butyllithium was prepared along procedure [14].

2-*exo*-[1-Octynyl (**IVa**) or 2-phenyl-1-ethynyl (**IVb**)]-2-*endo*-lithiumoxy-5,5,6-or (±)-2-*endo*-[1-octynyl (**VIIIa**) or 2-phenyl-1-ethynyl (**VIIIb**)]-2-*exo*-lithiumoxy-1,7,7-trimethylbicyclo[2.2.1]heptanes. To a cooled to–40÷–20°C solution of 0.013 mol of alkyne **Ia, b** in 20 ml of anhydrous tetrahydrofuran was added at vigorous stirring within 0.5 h 0.011 mol of butyllithium in hexane. The mixture was stirred at this temperature for 1 h, and then 0.01 mol of ketone **IIIa, b** was added, the temperature was raised to 20°C within 1 h, and the stirring was continued for 3 h more. Then the reaction mixture was left overnight. Compounds **IVa, b** and **VIIIa, b** were used as obtained without isolation.

2-*exo*-[1-Octynyl (Va) or 2-phenyl-1-ethynyl (Vb)]-2-*endo*-hydroxy-5,5,6-or (±)-2-endo-[1-octynyl (IXa) or 2-phenyl-1-ethynyl (IXb)]-2-*exo*-hydroxy-1,7,7-trimethylbicyclo[2.2.1]heptanes. To a solution containing 0.01 mol of lithium alcoholates **IVa, b, VIIIa, b** was added 100 ml of water, the alcohols **Va, b, IXa, b** were extracted into hexane, the extract was dried with $CaCl_2$, the solvent was removed, and the reaction products were purified by vacuum distillation (**Va, IXa, b**) or by recrystallization from hexane (**Vb**).

2-*exo*-[1-Octynyl (VIa–c) or 2-phenyl-1-ethynyl (VId–f)]2-endo-[methoxy (VIa, d), ethoxy (VIb, e) or butoxy (VIc, f)-5,5,6- or (±)-2-endo-[1-octynyl (Xa–c) or 2-phenyl-1-ethynyl (Xd–f)]2-*exo*-[methoxy (Xa, d), ethoxy (Xb, e) or butoxy (Xc, f]-1,7,7-trimethylbicyclo[2.2.1]heptanes. To a solution containing 0.01 mol of lithium alcoholates **IVa, b, VIIIa, b** was added 0.01 mol of methyl or ethyl iodide or butyl bromide and 3 ml of HMPA. With methyl and ethyl iodides the reaction was completed in 18 h at 20°C. With butyl bromide the reaction mixture was refluxed for 50 h under argon atmosphere. To the reaction mixture was added 100 ml of hexane, the organic solution was washed with water and 30% solution of NaOH, dried on $CaCl_2$, the solvent was removed, and compounds **VIa–e, Xa–e** were distilled in a vacuum.

2-***exo*-[1-Octynyl (VIIa) or 2-phenyl-1-ethynyl (VIIb)]2-endo-benzoyloxy-5,5,6- or (±)-2-*endo*-[1-octynyl (XIa) or 2-phenyl-1-ethynyl (XIb)]-2-*exo*-benzoyloxy-1,7,7-trimethylbicyclo[2.2.1]heptanes.** To a solution containing 0.01 mol of lithium alcoholates **IVa, b, VIIIa, b** was added in one portion 0.012 mol of benzoyl chloride, the mixture was stirred for 3 h and left overnight. Then 100 ml of water was added, the reaction products were extracted into hexane, the organic layer was washed with water, with saturated water solution of $NaHCO_3$, and dried on $CaCl_2$. The solvent was removed. Benzoates **VIIa, XIa** were purified by vacuum distillation, compounds **VIIb, XIb** were recrystallized from hexane.

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