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Tertiary Acetylenic Alcohols and Diols on the Basis of Phenylacetylene and 2-Methyl-3-butyn-2-ol*

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Abstract—A procedure has been proposed for the synthesis of tertiary acetylenic alcohols and diols by treatment of phenylacetylene or 2-methyl-3-butyn-2-ol with butyllithium and subsequent reaction of lithium phenylacetylide or lithium 4-lithio-2-methyl-3-butyn-2-olate thus obtained with alicyclic, aromatic, and terpene ketones.

We previously [1, 2] described the synthesis of acetylenic alcohols by addition of 1-octynyl- and 1-octadecynyllithium to various ketones. Acetylenic alcohols and diols attract interest as potential biologically active substances [3]; acetylenic diols are also promising synthons for studying their transformations under conditions of the Ritter reaction [4–6].

The goal of the present work was to develop a procedure for preparation of tertiary alcohols and diols containing an alicyclic or aromatic moiety and an aliphatic chain having a triple C≡C bond in the β-position with respect to the hydroxy group. We have found that treatment of phenylacetylene (**Ia**) or 2-methyl-3-butyn-2-ol (**IIa**) with 1 or 2 equiv of butyllithium, respectively, and subsequent reaction of lithium phenylacetylide (**Ib**) or lithium 4-lithio-2-methyl-3-butyn-2-olate (**IIb**) with alicycic (**IIIa**–**IIId**) and aromatic ketones (**VIa**–**VIe**), (*R*)-(–)- and (*S*)-(+)-carvone (**VIIIa**, **VIIIb**), isocamphanone (**X**), (±)-camphor (**XII**), (1*S*)-(+)-fenchone (**XIV**), and 2-adamantanone (**XVI**) lead to formation of the corresponding tertiary acetylenic alcohols **IVa**–**IVd**, **VIIa**–**VIIe**, **IXa**, **IXb**, **XVa**, and **XVIIa** and diols **Va**–**Vd**, **XI**, **XIII**, **XVb**, and **XVIIb** (Scheme 1). The products were isolated in 63–88% yield. Their yields, physical constants, and analytical data are given in Table 1, ^{1}H NMR spectra, in Table 2, and IR and UV spectra, in Table 3.

The IR spectra of the products contain absorption bands at 3225–3600 (O−H) and 995–1085 cm^{-1} (C−OH). The triple carbon–carbon bond gives rise to a weak band in the region 2220–2245 cm^{-1}.

In the ^{1}H NMR spectra of the prepared tertiary alcohols and diols we observed signals typical of the corresponding initial compounds and also broadened singlets from the hydroxy proton at δ 1.85–3.08 ppm. In the spectra of carvone, isocamphanone, camphor, and fenchone derivatives **IX**, **XI**, **XIII**, and **XV** the OH signal is overlapped by signals from alicyclic protons. Signals from the methyl protons in the *exo* and *endo* positions of compounds **XI**, **XIII**, and **XV** were assigned on the basis of published data [4] for initial ketones **X**, **XII**, and **XIV**, taking into account that reactions with organolithium reagents occur only at the carbonyl group, so that the configuration of the other molecular fragments remains unchanged. The upfield signal was assigned to the *endo*-methyl group, and the downfield signal, to the *exo*-oriented methyl group.

According to the data of spectral methods and TLC analysis, tertiary acetylenic alcohols and diols based on terpene ketones **VIIIa**, **VIIIb**, **X**, **XII**, and **XIV** are formed as a single isomer. Taking into account the results of our previous detailed spectral studies of related compounds [7, 8], these products were assigned structures with *endo*- (**XI**) and *exo*-orientation of the hydroxy group (**IXa**, **IXb**, **XIII**, **XV**) (see Scheme 1).

Tertiary acetylenic alcohols derived from natural optically active ketones, carvone (**VIIIa**/**VIIIb**) and

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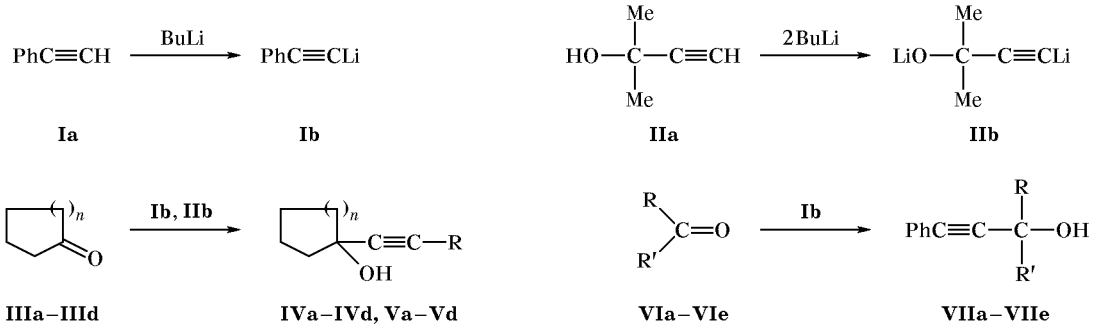
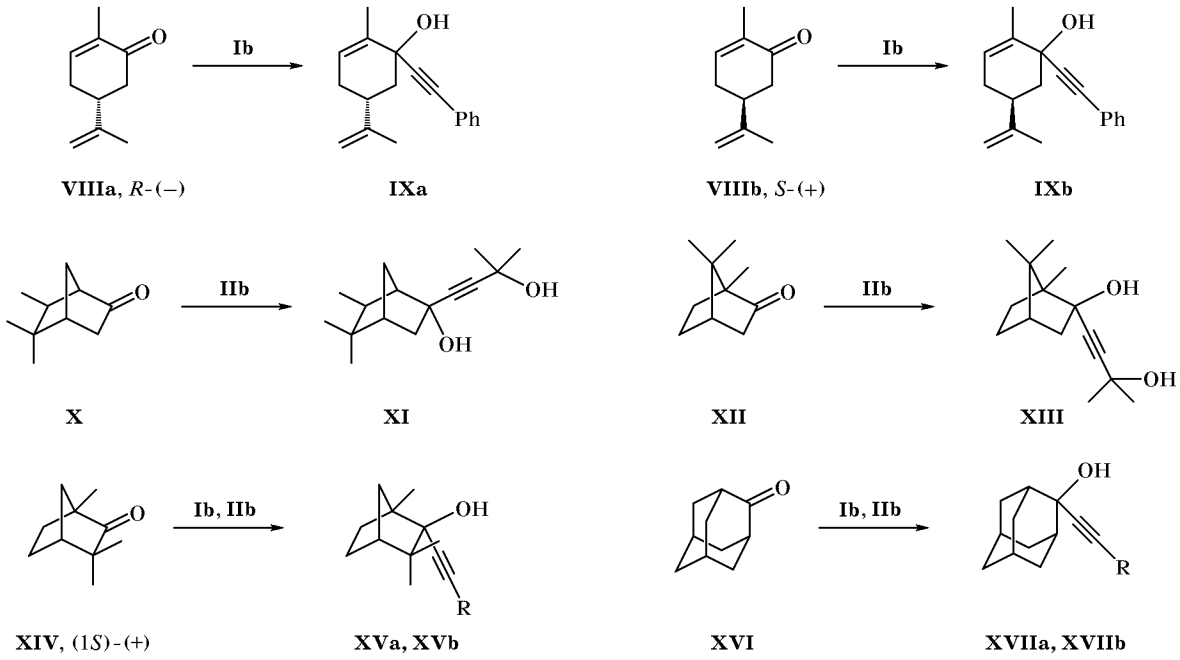
Scheme 1.



III–**V**, *n* = 1 (**a**), 2 (**b**), 3 (**c**), 8 (**d**); **IV**, R = Ph; **V**, R = $HOCMe_2$; **VI**, **VII**, R = Me, R′ = Ph (**a**), 1-naphthyl (**b**), 2-naphthyl (**c**); R = Ph, R′ = 4-$MeOC_6H_4$ (**d**); R = R′ = 4-$Me_2NC_6H_4$ (**e**).



XV, **XVII**, R = Ph (**a**), $HOCMe_2$ (**b**).

fenchone (**XIV**), are also optically active compounds, $[\alpha]_D^{20}$, deg: –335 (**IXa**), +347 (**IXb**), –24 (**XVa**), –8 (**XVb**).

EXPERIMENTAL

The IR spectra were recorded on a Specord 75IR spectrometer from samples prepared as thin films (compounds **IVa**, **Vc**, **VIIa**, **VIIc**, **IXa**, **IXb**, **XI**, **XIII**, **XVa**, and **XVIIa**) or KBr pellets (**IVb**–**IVd**, **Va**, **Vb**, **Vd**, **VIIb**, **VIId**, **VIIe**, **XVb**, and **XVIIb**). The ^1H NMR spectra were obtained on a Tesla BS-567A instrument in $CDCl_3$ using tetramethylsilane as internal reference. The UV spectra were measured on a Specord UV-Vis spectrophotometer from 1×10^{-3} M solutions of **Va**–**Vd**, **XI**, **XIII**, **XVb**, and **XVIIb**, 1×10^{-4} M solutions of **IVa**–**IVd**, **VIIa**, **VIId**, **IXa**, **IXb**, **XVa**, and **XVIIa**, and 1×10^{-5} M solutions of **VIIb**, **VIIc**, and **VIIe** in methanol. The optical rotations of compounds **IXa**, **IXb**, **XVa**, and **XVb** were measured on a SM-2 polarimeter from 3.5% solutions in methanol. The molecular weights were determined by cryoscopy in benzene. Neutral aluminum oxide, L 40/250 μm, Brockmann activity grade II, was used for column chromatography. Butyllithium was prepared by the procedure described in [9].

Table 1. Yields, physical constants, and elemental analyses of compounds **IVa**–**IVd**, **Va**–**Vd**, **VIIa**–**VIIe**, **IXa**, **IXb**, **XI**, **XIII**, **XVa**, **XVb**, **XVIIa**, and **XVIIb**

Comp. no.	Yield, %	bp, °C (*p*, mm), or mp, °C	d_{20}^{20}	n_D^{20}	Found, %		Formula	Calculated, %		*M*	
					C	H		C	H	found	calcd.
IVa	75	100–101 (5×10^{-2})	1.0681	1.5790	84.07	7.84	$C_{13}H_{14}O$	83.83	7.58	176	186.3
IVb	80	54–55	–	–	84.18	8.30	$C_{14}H_{16}O$	83.96	8.05	198	200.3
IVc	80	41–42	–	–	84.25	8.64	$C_{15}H_{18}O$	84.07	8.47	209	214.3
IVd	73	88–89	–	–	84.91	10.32	$C_{20}H_{28}O$	84.45	9.92	274	284.4
Va	53	57–58	–	–	71.68	9.84	$C_{10}H_{16}O_2$	71.39	9.59	163	168.2
Vb	69	102–103	–	–	72.81	10.11	$C_{11}H_{18}O_2$	72.49	9.95	177	182.3
Vc	85	122–123 (5×10^{-2})	–	–	73.58	10.33	$C_{12}H_{20}O_2$	73.43	10.27	190	196.3
Vd	82	110–111	–	–	77.03	11.51	$C_{17}H_{30}O_2$	76.64	11.35	252	266.4
VIIa	78	130–131 (5×10^{-2})	1.1130	–	73.58	10.33	$C_{12}H_{20}O_2$	73.43	10.27	190	196.3
VIIb	74	69–70	–	–	88.31	6.28	$C_{20}H_{16}O$	88.20	5.92	265	272.3
VIIc	71	–	–	–	88.39	6.11	$C_{20}H_{16}O$	88.20	5.92	262	272.3
VIId	71	90–91	–	–	84.18	5.84	$C_{22}H_{18}O$	84.05	5.77	304	314.4
VIIe[a]	65	166–167	–	–	81.17	7.27	$C_{25}H_{26}N_2O_2$	81.05	7.07	360	370.5
IXa	77	133–134 (5×10^{-2})	1.1525	1.5740	85.91	8.13	$C_{18}H_{20}O$	85.67	7.99	244	252.4
IXb	74	123–124 (5×10^{-2})	1.1318	1.5755	85.94	8.08	$C_{18}H_{20}O$	85.67	7.99	243	252.4
XI	86	125–126 (5×10^{-2})	–	–	76.61	10.44	$C_{15}H_{24}O_2$	76.23	10.23	226	236.4
XIII	81	107–108 (5×10^{-2})	0.9930	1.5025	76.38	10.25	$C_{15}H_{24}O_2$	76.23	10.23	226	236.4
XVa	74	111–112 (5×10^{-2})	1.9749	1.5620	85.35	8.91	$C_{18}H_{22}O$	84.99	8.82	244	254.4
XVb	71	88–89	–	–	76.44	10.31	$C_{15}H_{24}O_2$	76.23	10.23	233	236.4
XVIIa	88	144–145 (5×10^{-2})	–	–	85.91	8.16	$C_{18}H_{20}O$	85.67	7.99	246	252.4
XVIIb	83	148–149	–	–	76.84	10.01	$C_{15}H_{22}O_2$	76.88	9.46	229	234.3

[a] Found N, %: 7.54. Calculated N, %: 7.56.

Table 2. ^{1}H NMR spectra of compounds **IVa**–**IVd**, **Va**–**Vd**, **VIIa**–**VIIe**, **IXa**, **IXb**, **XI**, **XIII**, **XVa**, **XVb**, **XVIIa**, and **XVIIb**

Comp. no.	Chemical shifts δ, ppm
IVa	1.55–3.10 m [8H, $(CH_2)_4$], 2.34 br.s (1H, OH), 7.15–7.60 m (5H, Ph)
IVb	1.00–2.20 m [10H, $(CH_2)_5$], 2.40 br.s (1H, OH), 7.15–7.47 m (5H, Ph)
IVc	1.10–2.55 m [12H, $(CH_2)_6$], 2.30 br.s (1H, OH),7.12–7.50 m (5H, Ph)
IVd	1.10–1.95 m [22H, $(CH_2)_{11}$], 2.00 br.s (1H, OH), 7.15–7.52 m (5H, Ph)
Va	1.53 s (6H, Me_2C), 1.51–2.20 m [8H, $(CH_2)_4$], 2.59 br.s (2H, 2OH)
Vb	0.90–2.50 m [10H, $(CH_2)_5$], 1.54 s (6H, Me_2C), 3.08 br.s (2H, 2OH)

Table 2. (Contd.)

Comp. no.	Chemical shifts δ, ppm
Vc	1.40–2.20 m [12H, $(CH_2)_6$], 1.52 s (6H, Me_2C), 3.00 br.s (2H, 2OH)
Vd	1.20–1.95 m [22H, $(CH_2)_{11}$], 1.51 s (6H, Me_2C), 2.17 s and 2.44 br.s (2H, 2OH)
VIIa	1.86 s (3H, Me), 2.59 br.s (1H, OH), 7.15–7.78 m (10H, 2Ph)
VIIb	2.13 s (3H, Me), 2.75 br.s (1H, OH), 7.15–8.95 m (12H, $C_{10}H_7$, Ph)
VIIc	1.94 s (3H, Me), 2.69 br.s (1H, OH), 7.15–8.22 m (12H, $C_{10}H_7$, Ph)
VIId	2.94 br.s (1H, OH), 3.75 s (3H, MeO), 6.70–7.75 m (14H, C_6H_4, 2Ph)
VIIe	2.78 br.s (1H, OH), 2.92 s (12H, $2Me_2N$), 6.57–6.80 m, 7.17–7.65 m (13H, $2C_6H_4$, Ph)
IXa	1.16–1.85 m (6H, OH, CH, $2CH_2$), 1.78 s and 1.91 s (6H, 2Me), 4.70–4.85 m (2H, $=CH_2$), 5.40–5.75 m (1H, =CH), 7.15–7.45 s (5H, Ph)
IXb	1.60–1.85 m (6H, OH, CH, $2CH_2$), 1.78 s and 1.92 s (6H, 2Me), 4.62–4.80 m (2H, $=CH_2$), 5.40–5.80 m (1H, =CH), 7.12–7.45 m (5H, Ph)
XI	0.85 d (3H, 6-CH_3, J = 7.0 Hz), 0.90 s (3H, *endo*-5-CH_3), 1.02 s (3H, *exo*-5-CH_3), 1.50 s (6H, $Me_2CC{\equiv}C$), 1.55–2.35 m (9H, 2OH, 3CH, $2CH_2$)
XIII	0.86 s (3H, 1-CH_3), 0.94 s (3H, *endo*-7-CH_3), 1.06 s (3H, *exo*-7-CH_3), 1.10–2.40 m (9H, 2OH, CH, $3CH_2$), 1.52 s (6H, Me_2C)
XVa	1.01 s (3H, 1-CH_3), 1.10–2.20 m [8H, OH, CH, CH_2, $(CH_2)_2$], 1.22 s (3H, *endo*-3-CH_3), 1.27 s (3H, *exo*-3-CH_3), 7.15–7.45 m (5H, Ph)
XVb	0.96 s (3H, 1-CH_3), 1.12 s (3H, *endo*-3-CH_3), 1.17 s (3H, *exo*-3-CH_3), 1.20–2.00 m [6H, CH_2, $(CH_2)_2$], 1.52 s (6H, Me_2C), 1.73 br.s and 2.17 br.s (2H, 2OH)
XVIIa	1.35–2.15 m (14H, Ad), 2.08 br.s (1H, OH), 7.15–7.45 m (5H, Ph)
XVIIb	1.34–2.15 m [14H, Ad], 1.51 s (6H, Me_2C), 2.03 br.s (2H, 2OH)

Table 3. IR and UV spectra of compounds **IVa**–**IVd**, **Va**–**Vd**, **VIIa**–**VIIe**, **IXa**, **IXb**, **XI**, **XIII**, **XVa**, **XVb**, **XVIIa**, and **XVIIb**

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)
IVa	3360 (OH); 3080, 3055, 3030 (CH_{arom}); 2960, 2870 (CH_{aliph}); 2220 (C≡C); 1595, 1570, 1480 (Ar); 1440 (CH_2); 995 (C–OH); 755, 690 (CH_{arom})	204 (21 000), 241 (20 000), 252 (18 000)
IVb	3225 (OH); 3080, 3055, 3020 (CH_{arom}); 2930, 2855 (CH_{aliph}); 2240 (C≡C); 1595, 1565, 1480 (Ar); 1445 (CH_2); 1070 (C–OH); 755, 690 (CH_{arom})	205 (20 000), 241 (20 000), 252 (18 000)
IVc	(CH_{aliph});3370 (OH); 3080, 3055, 3030, 3020 (CH_{arom}); 2925, 2855 (CH_{aliph}); 2225 (C≡C), 1595, 1570, 1480 (Ar); 1450, 1440 (CH_2); 1020 (C–OH); 750, 690 (CH_{arom})	251 (20 000)
IVd	3325 (OH); 3100, 3075 3060, 3045, 3030, 3020 (CH_{arom}); 2940, 2910, 2860, 2845 (CH_{aliph}); 2225 (C≡C); 1600, 1575, 1490 (Ar); 1470, 1445 (CH_2); 1060 (C–OH); 755, 695 (CH_{arom})	206 (24 000), 241 (22 000), 251 (20 000)
Va	3275 (OH); 2980, 2940, 2870 (CH_{aliph}); 2240 (C≡C); 1460 (CH_2); 1170, 995 (C−OH)	203 (150)
Vb	3350 (OH); 2980, 2855 (CH_{aliph}); 2230 (C≡C); 1450 (CH_2); 1180, 1070 (C–OH)	203 (150)
Vc	3340 (OH); 2975, 2930, 2855 (CH_{aliph}); 2230 (C≡C); 1455 (CH_2); 1170, 1025 (C−OH)	203 (200)
Vd	3320 (OH); 2935, 2855, 2845 (CH_{aliph}); 2230 (C≡C); 1470 (CH_2); 1160, 1005 (C−OH)	202 (200)

Table 3. (Contd.)

Comp. no.	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (ε)
VIIa	3540, 3370 (OH); 3080, 3055, 3030 (CH_{arom}); 2985, 2930, 2860 (CH_{aliph}); 2230 (C≡C), 1595, 1570, 1485, 1445 (Ar); 1085 (C–OH); 755, 700, 685 (CH_{arom})	252 (17000)
VIIb	3600, 3390 (OH); 3080, 3055, 3025 (CH_{arom}); 2985, 2930, 2870 (CH_{aliph}); 2230 (C≡C); 1595, 1520, 1505, 1480, 1440 (Ar); 1025 (C–OH); 800, 775, 755, 690 (CH_{arom})	241 (30000), 252 (28000)
VIIc	3590, 3370 (OH); 3080, 3055, 3020 (CH_{arom}); 2980, 2930, 2855 (CH_{aliph}); 2230 (C≡C); 1595, 1500, 1485, 1440 (Ar); 1075 (C–OH); 810, 755, 690 (CH_{arom})	241 (31000), 252 (28000)
VIId	3500 (OH); 3080, 3065, 3040, 3010 (CH_{arom}); 2960, 2940, 2840 (CH_{aliph}); 2230 (C≡C); 1605, 1585, 1505, 1490 (Ar); 1025 (C–OH); 835, 755, 745, 695 (CH_{arom})	242 (25000), 252 (20000)
VIIe	3425 (OH), 3100, 3080, 3030 (CH_{arom}); 2980, 2960, 2880, 2840 (CH_{aliph}); 2230 (C≡C), 1605, 1590, 1520, 1505 (Ar); 1050 (C–OH); 840, 820, 805, 790, 755, 690 (CH_{arom})	205 (66000), 252 (34000)
IXa	3355 (OH); 3080, 3020 (=C–H); 3080, 3055, 3030 (CH_{arom}); 2965, 2945, 2915, 2880, 2850, 2835 (CH_{aliph}); 2220 (C≡C); 1645 (C=C); 1595, 1570, 1480 (Ar); 1440 (CH_2); 1025 (C–OH); 755, 690 (CH_{arom})	254 (13000)
IXb	3350 (OH); 3080, 3020 (=C–H); 3060, 3030 (CH_{arom}); 2965, 2945, 2915, 2880, 2855, 2835 (CH_{aliph}); 2220 (C≡C); 1640 (C=C); 1595, 1560, 1485 (Ar); 1440 (CH_2); 1030 (C–OH); 755, 690 (CH_{arom})	205 (20000), 242 (15000), 253 (14000)
XI	3390 (OH); 2980, 2960, 2945, 2875 (CH_{aliph}); 2240 (C≡C); 1475, 1450 (CH_2); 1155, 1045 (C−OH)	204 (200)
XIII	3400 (OH); 3025, 2955, 2940, 2875 (CH_{aliph}); 2235 (C≡C); 1470 (CH_2); 1150, 1055 (C−OH)	203 (400), 237 (450)
XVa	3475 (OH); 3080, 3055, 3030, 3020 (CH_{arom}); 2995, 2960, 2925, 2870 (CH_{arom}); 2220 (C≡C); 1595, 1565, 1490 (Ar); 1055 (C–OH); 755, 690 (CH_{arom})	205 (26000), 243 (24000), 252 (20000)
XVb	3350 (OH); 2975, 2955, 2930, 2875 (CH_{aliph}); 2240 (C≡C); 1470, 1450 (CH_2); 1160, 1045 (C−OH)	204 (200)
XVIIa	3450 (OH); 3080, 3055, 3025 (CH_{arom}); 2990, 2930, 2900, 2850 (CH_{aliph}); 2225 (C≡C); 1595, 1570, 1485 (Ar); 1450 (CH_2); 1075 (C−OH); 755, 690 (CH_{arom})	206 (23000), 241 (21000), 252 (18000)
XVIIb	3240 (OH); 2975, 2900, 2850 (CH_{aliph}); 2245 (C≡C); 1445 (CH_2); 1150, 1080 (C−OH)	204 (150)

1-Hydroxy-1-(2-phenylethynyl)- and 1-hydroxy-1-(3-hydroxy-3-methyl-1-butynyl)cycloalkanes IVa–IVd and Va–Vd, 2,4-diphenyl-3-butyn-2-ol (VIIa), 2-(1-naphthyl)-4-phenyl-3-butyn-2-ol (VIIb), 2-(2-naphthyl)-4-phenyl-3-butyn-2-ol (VIIc), 1-(4-methoxyphenyl)-3-phenyl-2-propyn-1-ol (VIId), 1,1-bis(4-dimethylaminophenyl)-3-phenyl-2-propyn-1-ol (VIIe), (5*R*)-(–)-5-isopropenyl-2-methyl-1-(2-phenylethynyl)-2-cyclohexenol (IXa), (5*S*)-(+)-5-isopropenyl-2-methyl-1-(2-phenylethynyl)-2-cyclohexenol (IXb), *exo*-2-(3-hydroxy-3-methyl-1-butynyl)-5,5,6-trimethylbicyclo[2.2.1]heptan-*endo*-2-ol (XI), (±)-*endo*-2-(3-hydroxy-3-methyl-1-butynyl)-1,7,7-trimethylbicyclo[2.2.1]heptan-*exo*-ol (XIII), (1*S*)-(–)-*endo*-2-(2-phenylethynyl)-1,3,3-trimethylbicyclo[2.2.1]heptan-*exo*-2-ol (XVa), (1*S*)-(–)-*endo*-2-(3-hydroxy-3-methyl-1-butynyl)-1,3,3-trimethylbicyclo[2.2.1]heptan-*exo*-2-ol (XVb), 2-hydroxy-2-(2-phenylethynyl)adamantane (XVIIa), and 2-hydroxy-2-(3-hydroxy-3-methyl-1-butynyl)-adamantane (XVIIb) (*general procedure***).** A solution of 0.03 mol or 0.07 mol of butyllithium in hexane was added under argon over a period of 0.5 h to a solution of 0.035 mol of phenylacetylene (**Ia**) in 50 ml of anhydrous tetrahydrofuran or of 0.035 mol of anhydrous 2-methyl-3-butyn-2-ol (**IIa**) in 100 ml of tetrahydrofuran, respectively, which was vigorously stirred at –40 to –20°C. The mixture was stirred for 1 h, 0.025 mol of ketone **IIIa**–**IIId**, **VIa**–**VIe**, **VIIIa**, **VIIIb**, **X**, **XII**, **XIV**, or **XVI** was added, and the mixture was allowed to warm up to 20–23°C over a period of 1–2 h, stirred for 3–4 h at that temperature,

and was left to stand for 18 h. The resulting lithium or dilithium alkoxide was hydrolyzed with 300 ml of water, the product was extracted into diethyl ether, the extract was washed with water, dried over $CaCl_2$, and evaporated, and the residue was kept in a vacuum. Compounds **IVa**, **Vc**, **VIIa**, **IXa**, **IXb**, **XI**, **XIII**, **XVa**, and **XVIIa** were purified by vacuum distillation, compounds **IVb**–**IVd**, **Va**, **Vb**, **Vd**, **VIId**, **VIIe**, **XVb**, and **XVIIb** were recrystallized from hexane, and compounds **VIIb** and **VIIc** were subjected to molecular distillation [10] with subsequent recrystallization from hexane (**VIIb**). Compounds **Vc**, **VIIc**, **XI**, and **XVIIa** are colorless viscous glassy liquids.

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