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Peroxy-Containing Acetylenic Alcohols and Ethers Derived from 4-Methoxybenzophenone and 1- and 2-Acetonaphthones

E. A. Dikusar, E. V. Vashkevich, N. G. Kozlov, V. I. Potkin,
K. L. Moiseichuk, O. I. Isakovich, and A. P. Yuvchenko

Institute of Physical Organic Chemistry, National Academy of Sciences of Belarus, Minsk, Belarus
Institute of Chemistry of New Materials, National Academy of Sciences of Belarus, Minsk, Belarus

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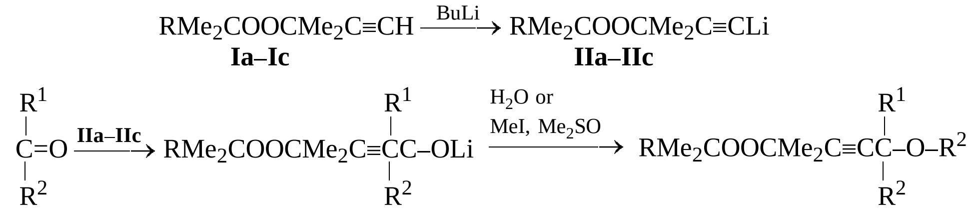
Abstract—A procedure was suggested for the synthesis of 2,5-dimethyl-2-*tert*-alkylperoxy-5-lithiooxy-5-methyl(phenyl)-5-naphthyl(aryl)alk-3-ynes by the reactions of the corresponding monosubstituted peroxyalkynes with butyllithium, followed by the reactions of the resulting lithium peroxy acetylenides with 4-methoxybenzophenone and 1- and 2-acetonaphthones. Lithium peroxy alcoholates are hydrolyzed with water to form peroxy-containing alcohols and react with methyl iodide in the presence of dimethyl sulfoxide to form the corresponding 2,5-dimethyl-2-*tert*-alkylperoxy-5-methoxy-5-methyl(phenyl)-5-naphthyl(aryl)alk-3-ynes. The thermal stability of the peroxides prepared was evaluated by thermal analysis.

It is known that photosensitized decomposition of some alkyl and aryl alkyl peroxides in polyethylene occurs only in the presence of naphthalene [1]; therefore, it is important to prepare peroxides containing a naphthalene fragment. We have shown previously [2, 3] that accessible lithium peroxy acetylenides **IIa**–**IIc** are convenient selective agents for preparing functional derivatives of peroxides. Here we report on the synthesis of 2,5-dimethyl-2-*tert*-alkylperoxy-5-lithiooxy-5-methyl(phenyl)-5-naphthyl(aryl)alk-3-ynes **IV** by the reactions of lithium peroxy acetylenides **IIa**–**IIc**, prepared by treatment of 3-methyl-3-*tert*-alkylperoxy-1-butynes **Ia**–**Ic** [4] with butyllithium, with 4-methoxybenzophenone **IIIa**, 1-acetonaphthone **IIIb**, and 2-acetonaphthone **IIIc**. Intermediate lithium peroxy alcoholates **IV**, which are subjected to further transformations without isolation, are reactive compounds. Their hydrolysis yielded peroxy alcohols **Va**–**VIIa, Vb**–**VIIb**, and **Vc**–**VIIc** in 64–78% yields.



$$RMe_2COOCMe_2C{\equiv}CH \xrightarrow{BuLi} RMe_2COOCMe_2C{\equiv}CLi$$

Ia–**Ic** **IIa**–**IIc**

$$\underset{R^2}{\overset{R^1}{C}}{=}O \xrightarrow{\textbf{IIa–IIc}} RMe_2COOCMe_2C{\equiv}C\underset{R^2}{\overset{R^1}{C}}–OLi \xrightarrow{H_2O\ \text{or}\ MeI,\ Me_2SO} RMe_2COOCMe_2C{\equiv}C\underset{R^2}{\overset{R^1}{C}}–O–R^2$$

IIIa–**IIIc** **IV** **Va**–**Xa, Vb**–**Xb, Xc**–**Xc**

I, II, V–**X**, R = Me (**a**), Et (**b**), Pr (**c**); **III**, R^1 = Ph, R^2 = 4-MeO–C_6H_4 (**a**), R^1 = Me, R^2 = 1-$C_{10}H_7$ (**b**), 2-$C_{10}H_7$ (**c**); R^1 = Ph, R^2 = 4-MeO–C_6H_4, R^3 = H (**V**), Me (**VIII**); R^1 = Me, R^2 = 1-$C_{10}H_7$, R^3 = H (**VI**), Me (**IX**); R^1 = Me, R^2 = 2-$C_{10}H_7$, R^3 = H (**VII**), Me (**X**).

Lithium peroxy alcoholates **IV** react with methyl iodide in the presence of dimethyl sulfoxide to form the corresponding 2,5-dimethyl-2-*tert*-alkylperoxy-5-methoxy-5-methyl(phenyl)-5-naphthyl(aryl)alk-3-ynes **VIIIa**–**Xa**, **VIIIb**–**Xb**, and **VIIIc**–**Xc** in 61–70% yield. In the absence of dimethyl sulfoxide, there is no reaction.

Peroxy alcohols **Va**–**VIIa**, **Vb**–**VIIb**, and **Vc**–**VIIc** were purified by molecular distillation [5], and peroxy-containing methyl ethers **VIIIa**–**Xa**, **VIIIb**–**Xb**, and **VIIIc**–**Xc**, by column chromatography on Al_2O_3.

Compounds **Va**–**Xa**, **Vb**–**Xb**, and **Vc**–**Xc** are colorless or weakly colored liquids readily soluble in com-

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Table 1. Yields, physicochemical constants, and elemental analyses of **Va**–**Xa, Vb**–**Xb**, and **Vc**–**Xc**

Comp. no.	Yield, %	d_4^{20}	n_D^{20}	Found, %		Formula	Calculated, %		*M*	
				C	H		C	H	found	calculated
Va	78	1.0791	1.5390	75.12	7.81	$C_{23}H_{28}O_4$	74.97	7.66	354.8	368.5
Vb	77	1.0778	1.5360	75.58	8.03	$C_{24}H_{30}O_4$	75.36	7.91	369.4	382.5
Vc	76	1.0686	1.5345	76.04	8.29	$C_{25}H_{32}O_4$	75.73	8.13	380.6	396.5
VIa[a]	74	–	–	77.54	7.99	$C_{21}H_{26}O_3$	77.27	8.03	319.4	326.4
VIb	64	1.0516	1.5505	78.05	8.19	$C_{22}H_{28}O_3$	77.61	8.29	323.0	340.5
VIc	69	1.0533	1.5465	78.20	8.63	$C_{23}H_{30}O_3$	77.93	8.53	336.0	354.5
VIIa	65	1.2299	1.5460	77.53	8.21	$C_{21}H_{26}O_3$	77.27	8.03	309.7	326.4
VIIb	71	1.1692	1.5390	77.84	8.51	$C_{22}H_{28}O_3$	77.61	8.29	321.4	340.5
VIIc	68	1.0079	1.5410	78.05	8.66	$C_{23}H_{30}O_3$	77.93	8.53	339.8	354.5
VIIIa	69	1.1486	1.5305	75.51	7.98	$C_{24}H_{30}O_4$	75.36	7.91	370.6	382.5
VIIIb	66	1.0189	1.5230	76.08	8.31	$C_{25}H_{32}O_4$	75.73	8.13	382.7	396.5
VIIIc	64	0.9349	1.5260	76.18	8.45	$C_{26}H_{34}O_4$	76.06	8.35	389.1	410.6
IXa	68	1.0510	1.5305	77.84	8.45	$C_{22}H_{28}O_3$	77.61	8.29	326.3	340.5
IXb	65	1.0624	1.5370	78.16	8.79	$C_{23}H_{30}O_3$	77.93	8.53	343.0	354.5
IXc	70	0.9486	1.5200	78.51	8.99	$C_{24}H_{32}O_3$	78.22	8.75	359.3	368.5
Xa	61	0.9587	1.5330	77.88	8.43	$C_{22}H_{28}O_3$	77.61	8.29	324.0	340.5
Xb	68	0.9793	1.5245	78.14	8.81	$C_{23}H_{30}O_3$	77.93	8.53	341.6	354.5
Xc	69	0.9512	1.5225	78.43	9.03	$C_{24}H_{32}O_3$	78.22	8.75	350.4	368.5

[a] mp 73–74°C.

Table 2. ^{1}H NMR data for **Va**–**Xa, Vb**–**Xb**, and **Vc**–**Xc**

Comp. no.	^{1}H NMR spectrum, δ, ppm
Va	1.22 s (9H, Me_3COO), 1.52 s (6H, Me_2C), 2.87 s (1H, OH), 3.76 s (3H, MeO), 6.65–7.65 m (9H, Ph and C_6H_4)
Vb	0.87 t (3H, *Me*CH_2), 1.18 s (6H, Me_2COO), 1.52 s (6H, Me_2C), 1.54 q (2H, CH_2), 2.82 s (1H, OH), 3.75 s (3H, MeO), 6.75–7.68 m (9H, Ph and C_6H_4)
Vc	0.88 t [3H, *Me*$(CH_2)_2$], 1.18 s (6H, Me_2COO), 1.40–1.57 m [4H, $(CH_2)_2$], 1.53 s (6H, Me_2C), 2.84 s (1H, OH), 3.74 s (3H, MeO), 6.75–7.67 m (9H, Ph and C_6H_4)
VIa	1.21 s (9H, Me_3COO), 1.45 s (6H, Me_2C), 2.10 s (3H, Me), 2.82 s (1H, OH), 7.20–8.08 m, 8.72–8.88 m (7H, $C_{10}H_7$)
VIb	0.86 t (3H, *Me*CH_2), 1.16 s (6H, Me_2COO), 1.46 s (6H, Me_2C), 1.50 q (2H, CH_2), 2.16 s (3H, *Me*COH), 2.77 s (1H, OH), 7.20–8.16 m, 8.72–8.86 m (7H, $C_{10}H_7$)
VIc	0.85 t [3H, *Me*$(CH_2)_2$], 1.15 s (6H, Me_2COO), 1.48 s (6H, Me_2C), 1.07–1.60 m [4H, $(CH_2)_2$], 2.16 s (3H, *Me*COH), 7.21–8.17 m, 8.71–8.88 m (7H, $C_{10}H_7$)
VIIa	1.22 s (9H, Me_3COO), 1.54 s (6H, Me_2C), 1.82 s (3H, Me), 2.62 s (1H, OH), 7.40–8.18 m (7H, $C_{10}H_7$)
VIIb	0.89 t (3H, *Me*CH_2), 1.21 s (6H, Me_2COO), 1.52 s (6H, Me_2C), 1.55 q (2H, CH_2), 1.83 s (3H, *Me*COH), 2.78 s (1H, OH), 7.38–8.15 m (7H, $C_{10}H_7$)
VIIc	0.88 t [3H, *Me*$(CH_2)_2$], 1.22 s (6H, Me_2COO), 1.43–1.57 m [4H, $(CH_2)_2$], 1.52 s (6H, Me_2C), 1.83 s (3H, *Me*COH), 2.62 s (1H, OH), 7.40–8.16 m (7H, $C_{10}H_7$)
VIIIa	1.23 s (9H, Me_3COO), 1.56 s (6H, Me_2C), 3.33 s (3H, MeOCC≡C), 3.75 s (3H, *Me*OC_6H_4), 6.75–7.63 m (9H, Ph and C_6H_4)
VIIIb	0.88 t (3H, *Me*CH_2), 1.18 s (6H, Me_2COO), 1.56 s (6H, Me_2C), 1.57 q (2H, CH_2), 3.31 s (3H, MeOCC≡C), 3.74 s (3H, *Me*OC_6H_4), 6.74–7.62 m (9H, Ph and C_6H_4)
VIIIc	0.89 t [3H, *Me*$(CH_2)_2$], 1.18 s (6H, Me_2COO), 1.43–1.55 m [4H, $(CH_2)_2$], 1.52 s (6H, Me_2C), 3.31 s (3H, MeOCC≡C), 3.75 s (3H, *Me*OC_6H_4), 6.75–7.67 m (9H, Ph and C_6H_4)

Table 2. (Contd.)

Comp. no.	^{1}H NMR spectrum, δ, ppm
IXa	1.28 s (9H, Me_3COO), 1.59 s (6H, Me_2C), 1.99 s (3H, MeCC≡C), 3.24 s (3H, MeO), 7.32–8.13 m, 8.72–8.91 m (7H, $C_{10}H_7$)
IXb	0.91 t (3H, *Me*CH_2), 1.24 s (6H, Me_2COO), 1.56 q (2H, CH_2), 1.58 s (6H, Me_2C), 2.00 (3H, MeCC≡C), 3.24 s (3H, MeO), 7.30–8.15 m, 8.75–8.91 m (7H, $C_{10}H_7$)
IXc	0.89 t [3H, *Me*$(CH_2)_2$], 1.24 s (6H, Me_2COO), 1.40–1.60 m [4H, $(CH_2)_2$], 1.58 s (6H, Me_2C), 1.99 s (3H, MeOCC≡C), 3.21 s (3H, MeO), 7.40–8.15 m, 8.75–8.90 m (7H, $C_{10}H_7$)
Xa	1.30 s (9H, Me_3COO), 1.61 s (6H, Me_2C), 1.79 s (3H, MeCC≡C), 3.22 s (3H, MeO), 7.40–8.20 m (7H, $C_{10}H_7$)
Xb	0.91 t (3H, *Me*CH_2), 1.25 s (6H, Me_2COO), 1.55 q (2H, CH_2), 1.60 s (6H, Me_2C), 1.78 s (3H, MeCC≡C), 3.23 s (3H, MeO), 7.40–8.15 m (7H, $C_{10}H_7$)
Xc	0.89 t [3H, *Me*$(CH_2)_2$], 1.26 s (6H, Me_2COO), 1.40–1.55 m [4H, $(CH_2)_2$], 1.60 s (6H, Me_2C), 1.78 s (3H, MeCC≡C), 3.23 s (3H, MeO), 7.40–8.18 m (7H, $C_{10}H_7$)

Table 3. UV data for **IIIa–IIIc, Va–Xa, Vb–Xb**, and **Vc–Xc**

Comp. no.	λ_{max}, nm ($\varepsilon \times 10^{-3}$, l mol^{-1} cm^{-1})
IIIa	204 (14), 221 (8), 254 (6), 290 (12)
IIIb	210 (62), 239 (29), 295 (12)
IIIc	208 (13), 240 (31), 247 (34), 283 (6)
Va	204 (13), 233 (8)
Vb	204 (13), 232 (8)
Vc	205 (14), 233 (9)
VIa	222 (80)
VIb	223 (81)
VIc	223 (82)
VIIa	225 (80)
VIIb	225 (81)
VIIc	223 (81)
VIIIa	208 (18), 234 (13)
VIIIb	204 (18), 232 (14)
VIIIc	206 (18), 232 (12)
IXa	224 (81)
IXb	224 (81)
IXc	225 (81)
Xa	225 (80)
Xb	224 (80)
Xc	224 (81)

mon organic solvents and insoluble in water. These compounds are stable in storage at 0–5°C. The physicochemical constants and yields of **Va**–**Xa**, **Vb**–**Xb**, and **Vc**–**Xc** are given in Table 1; the ^{1}H NMR data, in Table 2; and the UV data, in Table 3.

The IR spectra of **Va**–**Xa**, **Vb**–**Xb**, and **Vc**–**Xc** are consistent with their structure [6]. The spectra contain no absorption bands of the C=O and C≡C–H groups of the starting ketones **IIIa**–**IIIc** and 3-methyl-3-*tert*-alkylperoxy-1-butynes **Ia**–**Ic**. The spectra of peroxy alcohols **Va**–**VIIa**, **Vb**–**VIIb**, and **Vc**–**VIIc** contain a band of associated OH groups at 3440 ± 20 cm^{-1}. Ethers **VIIIa**–**Xa**, **VIIIb**–**Xb**, and **VIIIc**–**Xc** exhibit an absorption band of the C–O–C stretching vibrations at 1090 ± 10 cm^{-1}. The peroxides derived from 4-methoxybenzophenone (**Va**–**Vc**, **VIIIa**–**VIIIc**) exhibit the following absorption bands confirming the presence of aromatic groups (cm^{-1}): 3085 ± 5, 3060 ± 5, 3030 ± 5 (CH_{Ar}); 1605 ± 5, 1580 ± 5, 1500 ± 5 (skeleton vibrations of Ar); 835 ± 5, 800 ± 5, 765 ± 10, 745 ± 5, 725 ± 5, 700 ± 5 (CH_{Ar}). The spectra of the peroxides derived from 1-acetonaphthone (**VIa**–**VIc**, **IXa**–**IXc**) contain the following absorption bands (cm^{-1}): 3095 ± 5, 3050 ± 5 (CH_{Ar}); 1600 ± 5, 1575 ± 5, 1505 ± 5 (skeleton vibrations of Ar); 805 ± 5, 785 ± 5 (CH_{Ar}). The peroxides derived from 2-acetonaphthone (**VIIa**–**VIIc**, **Xa**–**Xc**) exhibit the following absorption bands (cm^{-1}): 3060 ± 5 (CH_{Ar}); 1600 ± 5, 1505 ± 5 (skeleton vibrations of Ar); 820 ± 5, 750 ± 5 (CH_{Ar}).

The thermal stability of **Va**–**Xa**, **Vb**–**Xb**, and **Vc**–**Xc** was evaluated by thermal analysis [7]. The results (data for the first decompostion step) are listed in Table 4. It is seen that the thermal stability of peroxy alcohols **Va**–**VIIa**, **Vb**–**VIIb**, and **Vc**–**VIIc** and of peroxy ethers **VIIIa**–**Xa**, **VIIIb**–**Xb**, and **VIIIc**–**Xc** is relatively high. Peroxy alcohols **Va**–**VIIa**, **Vb**–**VIIb**, and **Vc**–**VIIc** start to decompose at a noticeable rate with a pronounced exothermic effect only at 124–142°C. Peroxy ethers **VIIIa**–**Xa**, **VIIIb**–**Xb**, and **VIIIc**–**Xc** in which the hydroxy group is replaced by the methoxy group have somewhat higher thermal stability. The increase in the thermal stability upon methylation is especially pronounced in ethers **IXa**–**IXc** and **Xa**–**Xc** formed, respectively, from 1- and 2-acetonaphthone derivatives **VIa**–**VIc** and **VIIa**–**VIIc**. For example, ethers **IXa** and **Xa** containing *tert*-butylperoxy groups start to decompose at 148 and 142°C, respectively, which is 6–8°C higher than the decomposition point of the respective peroxy alcohols **VIa** and **VIIa**.

EXPERIMENTAL

The IR spectra were recorded on a Specord IR-75 spectrophotometer (thin films for **Va**–**Xa**, **Vb**–**Xb**, and **Vc**–**Xc** except **VIa**; KBr pellet for **VIa**). The ^{1}H NMR spectra were taken on a Tesla BS-567A spectrometer in $CDCl_3$, internal reference TMS. The UV spectra were measured on a Specord UV-Vis spectrophotometer for methanolic solutions of concentration 1×10^{-3} M for **IIIa**, **Va**–**Vc**, and **VIIIa**–**VIIIc** or 1×10^{-4} M for **IIIb**, **IIIc**, **VIa**–**VIc**, **VIIa**–**VIIc**, **IXa**–**IXc**, and **Xa**–**Xc**. The molecular weights were determined cryoscopically in benzene.

The thermal behavior of the compounds was studied on a Paulik–Paulik–Erdey derivatograph under argon at a linear heating rate of 7 deg min^{-1} (sample weight 100 mg, DTA 1/10, DTG 1/10). The purity of the compounds was checked by thin-layer chromatography on 20-cm-high Silufol plates, eluent hexane–ether (3 : 1), developer *N,N*-dimethyl-*p*-phenylenediamine dihydrochloride. Column chromatography was performed on Al_2O_3, L 40/250 μm, Brockmann grade II, neutral. Determination of the content of available oxygen in **Va**–**Xa**, **Vb**–**Xb**, and **Vc**–**Xc** by iodometric titration in concentrated HCl [8] gave overestimated results. The starting peroxyalkynes **Ia**–**Ic** [9] and butyllithium [10] were prepared by published procedures.

2,5-Dimethyl-2-*tert*-alkylperoxy-5-hydroxy-5-methyl(phenyl)-5-naphthyl(aryl)alk-3-ynes Va–VIIa, Vb–VIIb, and Vc–VIIc *(general procedure).* A hexane solution of 0.011 mol of butyllithium was added in an argon flow over a period of 0.5 h to a vigorously stirred solution of 0.013 mol of peroxyalkyne **Ia**–**Ic** in 20 ml of tetrahydrofuran, cooled to –40 to –20°C. The mixture was stirred for an additional 1 h, after which 0.01 mol of ketone **IIIa**–**IIIc** was added, and the mixture was warmed to 20–23°C over a period of 1–2 h, stirred for an additional 3–4 h, and allowed to stand for 18 h. To the resulting lithium peroxy alcoholates **IV**, we added 100 ml of water; peroxy alcohols **Va**–**VIIa**, **Vb**–**VIIb**, and **Vc**–**VIIc** were extracted with hexane, the extract was dried over $CaCl_2$, and the solvent was removed. Compounds **Va**–**Xa**, **Vb**–**Xb**, and **Vc**–**Xc** (except **VIa**) were purified by molecular distillation (110°C, 5×10^{-2} mm), and **VIa**, by recrystallization from hexane.

Table 4. Characteristics of the first step of thermolysis of peroxides **Va–Xa, Vb–Xb**, and **Vc–Xc** (thermal analysis data)

Comp. no.	T_{dec}, °C			Weight loss, %
	onset	end	maximum	
Va	142	215	182	23.0
Vb	138	205	177	23.5
Vc	135	205	178	22.0
VIa	140	198	180	31.0
VIb	135	195	175	25.0
VIc	138	195	176	23.0
VIIa	136	185	164	23.5
VIIb	130	205	175	24.5
VIIc	122	185	160	21.0
VIIIa	145	213	187	23.0
VIIIb	138	198	171	25.0
VIIIc	140	205	178	24.0
IXa	148	212	176	29.5
IXb	142	218	188	29.0
IXc	141	208	182	32.0
Xa	142	195	172	23.0
Xb	136	215	190	28.0
Xc	132	198	172	27.0

2,5-Dimethyl-2-*tert*-alkylperoxy-5-methoxy-5-methyl(phenyl)-5-naphthyl(aryl)alk-3-ynes VIIIa–Xa, VIIIb–Xb, and VIIIc–Xc *(general procedure).* To a solution coontaining 0.01 mol of lithium peroxy alcoholates **IV** (intermediates in synthesis of peroxy alcohols **Va**–**VIIa**, **Vb**–**VIIb**, and **Vc**–**VIIc**), we added 0.012 mol of methyl iodide and 3 ml of dimethyl sulfoxide. The reaction was complete in 18 h at 20–23°C. Then 100 ml of hexane was added, the organic layer was washed with water and 30% NaOH and dried over $CaCl_2$, and the solvent was removed. Compounds **VIIIa**–**Xa**, **VIIIb**–**Xb**, and **VIIIc**–**Xc** were purified by column chromatography on Al_2O_3 (eluent hexane).

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