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Reactions of 2-Butylsulfanyl-2-alkenals with Alcohols and Water

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Abstract—2-Butylsulfanyl-2-alkenals react with alcohols at room temperature in the presence of acid catalysts to give 45–90% of the corresponding acetals. Acetals derived from 2-butylsulfanylpropenal readily undergo hydrolysis at the vinylsulfanyl group (20°C, catalysis by HCl or TsOH) with formation of 2-oxopropionaldehyde *O,O*- or *O,S*-acetals in 70–90% yield. Unlike 2-butylsulfanyl-2-propenal *O,O*-dialkyl acetals, the initial aldehydes and 2,4-dinitrophenylhydrazones derived therefrom are stable to hydrolysis under analogous conditions: the vinyl sulfide moiety remains unchanged even under considerably more severe conditions (100°C, 3 h; HCl, H_2SO_4, CF_3SO_2OH, or $TiCl_4$).

2-Alkenals are known to take up alcohols at the carbonyl group in the presence of acid catalysts [1]. These reactions often occur at room temperature [2, 3]. When a small amount of a solvent immiscible with water is used, the liberated water separates from the mixture, and no azeotropic removal of water is necessary [3]. However, it was proposed to synthesize acetals from high-boiling alcohols with removal of water by azeotropic distillation [4]. In this case, the reaction can be accompanied by formation of an appreciable amount of the corresponding 1,1,3-trialkoxypropane [4, 5].

The goal of the present work was to examine the regioselectivity in reactions of 2-butylsulfanyl-2-alkenals with alcohols and water. These substrates may be regarded as a combination of acrylic and vinyl sulfide moieties. The presence of the latter leads us to expect that addition of alcohols will follow the Markownikoff rule [6]. This pattern is typical of reactions of 2-alkoxypropenals with equimolar amounts of alcohols, where regioselective attack on the C=C bond occurs [7]. Provided that polarization of the C=C bond is determined mainly by electron-acceptor effect of the carbonyl group, nucleophilic attack on the latter might be expected.

Experiments showed that, unlike 2-alkoxypropenals, 2-butylsulfanyl-2-alkenals **Ia** and **Ib** take up alcohols in the presence of a catalytic amount of a Brønsted or Lewis acid (TsOH, HBr, $BF_3 \cdot OEt_2$, $HgCl_2$) at room temperature exclusively at the carbonyl group to give 52–100% of the corresponding 2-alkylsulfanyl-2-alkenal dialkyl acetals **IIIa**–**IIId** (Scheme 1). Compounds **III** undergo appreciable decomposition during vacuum distillation (at 1 mm); however, they can be isolated by molecular distillation.

Acetals **IV**, **V**, and dibutyl disulfide are often formed as by-products (5–6, 7–14, and 5–8 wt %, respectively, according to the GC–MS data), regardless of the mode of water removal from the reaction mixture (azeotropic distillation or trapping with 3- or 4-Å molecular sieves). Compounds **IV** and **V** are likely to result from hydrolysis of the vinylsulfanyl group in **III**. Facile hydrolysis of the vinylsulfanyl fragment in **III** was demonstrated by carrying out the reaction of 2-butylsulfanylpropenal dibutyl acetal (**IIIc**) with water at 20°C in the presence of *p*-toluenesulfonic acid (Scheme 2). After 9 days, the yield of **Vc** reached 89% (^{1}H NMR data).

Likewise, acetal **Vc** is readily formed in the reaction of 2-butylsulfanylpropenal (**Ib**) with butanol, catalyzed by concentrated hydrochloric acid, unless liberated water is trapped. After 24 h, the weight fraction of **Vc** in the reaction mixture reaches 70% (GC–MS). In this experiment, one fraction isolated by distillation contained about 50% of acetal **IVc**. Usually, the concentration of **IVc** in the reaction mixture is low, so that it cannot be determined by ^{1}H NMR spectroscopy or isolated by distillation (it can be detected only by GC–MS). Accumulation of a considerable amount of 2-oxopropanal dialkyl acetal in the reaction mixture led us to presume that this compound is a precursor of mixed *O,S*-acetal **V**; as a rule, the

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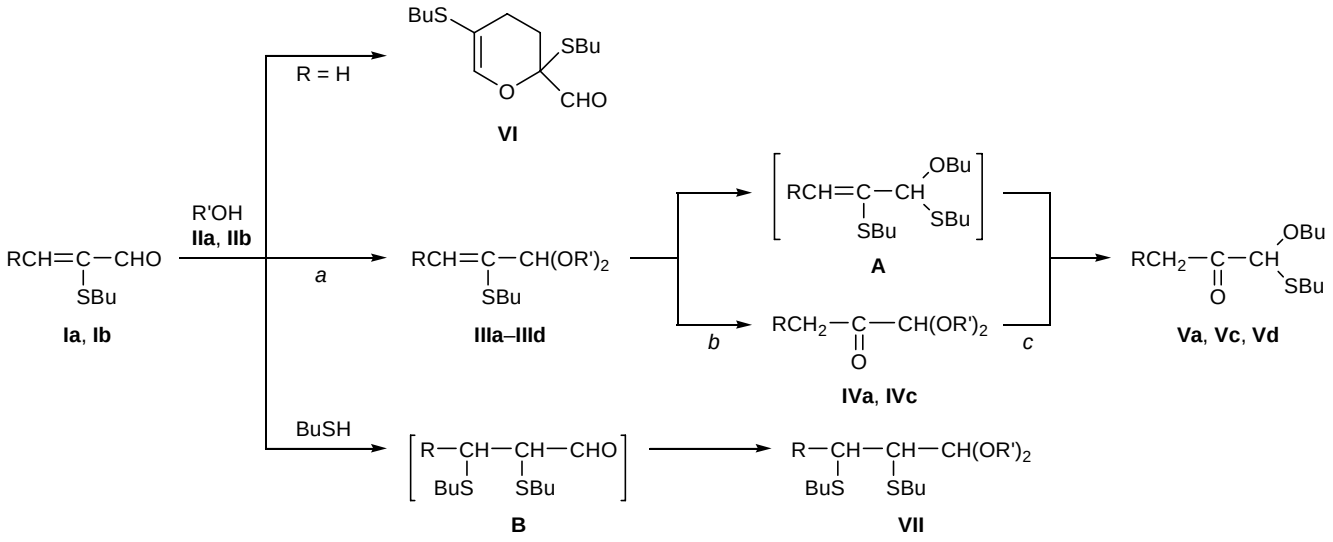
Scheme 1.

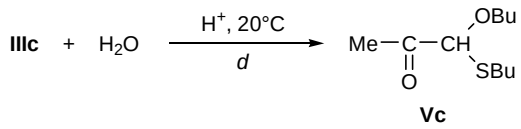


I, R = Me (**a**), H (**b**); **II**, R' = Bu (**a**), Et (**b**); **III**, R = Me, R' = Bu (**a**), Et (**b**); R = H, R' = Bu (**c**), Et (**d**); **IV**, R = Me, R' = Bu (**a**), R = H, R' = Bu (**c**); **V**, R = Me, R' = OBu (**a**); R = H, R' = Bu (**c**), Et (**d**); **VII**, R = Me, R' = Et.

concentration of **IV** is twice as low as that of **V**. Alternatively, compound **V** may be formed by hydrolysis of intermediate acetal **A** (Scheme 1). Presumably, the latter was detected in up to 4% yield by GC–MS (*m*/*z* 304) in the reaction of aldehyde **Ia** with butanol.

The reaction sequence *a*–*b*–*c* operating in the absence of water acceptors and reaction *d* (Scheme 2) may be regarded as a new preparative route to 2-oxopropanal *O*,*S*-acetals. Alkylsulfanyl group may be introduced into molecules of *O*,*O*-acetals **IV** via nucleophilic replacement of the RO group by butanethiol which could be formed by partial decomposition of the initial aldehyde or acetal **III**. An analogous process was observed previously in the reaction of 2-oxopropanal diethyl acetal with EtSH [8]. However, such a reaction seems to be surprising, taking into account 20–40-fold excess of alcohol used in our experiments. These data convincingly demonstrate predominating activity of the RS nucleophile and a fairly high stability of monothioacetals **V** to disproportionation.

Scheme 2.



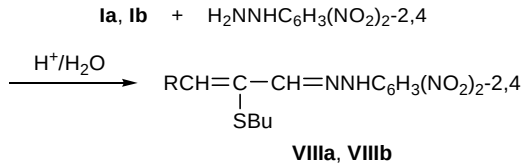
Apart from the above products resulting from hydrolytic cleavage of acetals **IV** and **V** of both crotonic and acrylic series, reactions of 2-butylsulfanylpropenal (**Ib**) with alcohols give rise to a large amount of the corresponding dimer, 2,5-bis(butylsulfanyl)-2,3-dihydro-4*H*-pyran-2-carbaldehyde (**VI**) (28–60%, according to the ^{1}H NMR data). As shown in [9], aldehyde **Ib** is capable of undergoing cyclodimerization at a high rate. In order to minimize this process, the reactions were performed using a 10–20-fold excess of alcohols, and the initial aldehyde was diluted with ether. Under these conditions, the yield of acetal **IIIc** reached 80%, while the yield of **IIId** was as low as 30–45%. Among other reasons, the low yield of **IIId** is explained by the fact that initial aldehyde **Ib** (which is prepared by retro-Diels–Alder reaction from dimer **VI** [10]) usually contains 20–30% of the dimer. As with crotonaldehyde derivative **Ia**, the reaction of aldehyde **Ib** with ethanol gives up to 8% of monothioacetal **Vd** as by-product. Another side reaction is nucleophilic 1,4-addition of thiol formed by decomposition of butyl vinyl sulfide in acid medium, as well as by hydrolysis of dimer **VI**. Intermediate formation of 2,3-bis(butyl-sulfanyl)propanal **B** was detected by ^{1}H NMR and GC–MS; the spectral data were compared with those reported in [11]. Diethyl acetal **VII** was isolated by distillation (yield up to 6%).

Thus 2-butylsulfanyl-2-enals take up alcohols only at the carbonyl group. No Markownikoff addition of alcohols or thiols at the C=C bond, which is typical of 2-alkoxypropenals [7, 8] or vinyl sulfides [6], was observed.

Unlike acetals **III**, hydrolysis of the vinyl sulfide moiety in initial 2-butylsulfanyl-2-alkenals **I** is con-

siderably more difficult. We previously showed that aldehyde **Ib** reacts with 2,4-dinitrophenylhydrazine in aqueous ethanol in the presence of a large amount of concentrated sulfuric acid to afford 2-butylsulfanyl-2-propenal 2,4-dinitrophenyl-hydrazone as the only product (yield 35%) [12]. Likewise, the reaction of 2-butylsulfanyl-2-butenal (**Ia**) with 2,4-dinitrophenylhydrazine in the presence of concentrated hydrochloric acid (100°C, 3 h) gave only the corresponding hydrazone **VIIIa** (yield 50%, Scheme 3) [13].

Scheme 3.



R = Me (**a**), H (**b**).

Hydrolysis of substituted vinyl sulfides usually requires severe conditions, e.g., catalysis by $HgCl_2$ (2 equiv, 50–80°C, 20–40 h) [14], 70% perchloric acid (6 equiv)–benzenethiol (2 equiv) [15], or $TiCl_4$ (2 equiv) [16]. Our attempts to effect hydrolysis of 2-butylsulfanylpropenal (**Ib**) in the presence of $TiCl_4$ under analogous conditions were unsuccessful: only tarry material was obtained. The hydrolysis of aldehyde **Ib** in the presence of a catalytic amount of CF_3SO_2OH was also accompanied by tarring. No expected 2-oxopropanal was detected by spectral methods (^{1}H NMR, IR). We can conclude that, if a molecule possesses an acrylic bond system (or its imino analog), the vinylsulfanyl moiety therein (aldehydes **I**) or in the corresponding 2,4-dinitrophenylhydrazones (compounds **VIII**) is stable to hydrolysis.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer (400 MHz for ^{1}H and 100.61 MHz of ^{13}C), respectively, using $CDCl_3$ as solvent and HMDS as internal reference. Gas chromatographic–mass spectrometric analysis was performed on a Hewlett–Packard HP 5971A mass-selective detector (electron impact, 70 eV) coupled with an HP 5890 chromatograph (Ultra-2 column, 5% of phenylmethylsilicone, injector temperature 250°C, oven temperature programming from 70 to 280°C at 20 deg/min).

2-Butylsulfanyl-2-butenal (**Ia**) was synthesized by the procedure described in [13]. ^{13}C NMR spectrum, δ_C, ppm: 13.74 (CH_3, Bu), 16.85 (**C**H_3CH=), 21.73 (CH_2, Bu), 31.86 (SCH_2), 32.20 (CH_2, Bu), 139.50 (C=), 155.44 (CH=), 191.10 (CHO); the signals were assigned using standard JMOD technique. ^{13}C NMR spectrum of 2-butylsulfanylpropenal (**Ib**), δ_C, ppm: 13.57 (CH_3), 22.1 (CH_2), 29.65 (CH_2), 29.82 (CH_2), 126.44 (H_2C=), 147.89 (=**C**SBu), 190.60 (CHO); the signals were assigned from the proton-coupled spectrum.

1,1-Dibutoxy-2-butylsulfanyl-2-butene (IIIa). *a*. A mixture of 2.76 g (0.017 mol) of 2-butenal (**Ia**), 23.7 g (0.37 mol) of butyl alcohol, 51.4 g of diethyl ether, 0.3 g (0.0017 mol, 10 mol %) of *p*-toluenesulfonic acid, and 5.8 g of 4-Å molecular sieves was kept for 13 days at room temperature. An additional portion of 4-Å molecular sieves, 8.83 g, was then added, the mixture was kept for 3 days and neutralized with dry potassium carbonate, and diethyl ether and excess butyl alcohol were removed under reduced pressure. According to the NMR data, the residue contained 94.9% of acetal **IIIa**. By triple vacuum distillation we isolated 1.3 g (26%) of dibutyl acetal **IIIa**, bp 125–127°C (6 mm). The still residue contained a lot of tars. ^{1}H NMR spectrum, δ, ppm: 0.89 t (6H, C**H**$_3$CH$_2$, ^3J = 7.1 Hz), 0.91 t (3H, C**H**$_3$CH$_2$), 1.38 m (6H, C**H**$_2$CH$_3$), 1.54 m (6H, C**H**$_2$CH$_2$CH$_3$), 1.87 d (3H, C**H**$_3$CH, ^3J = 6.8 Hz), 2.68 t (2H, SCH_2), 3.38 d.t and 3.57 d.t (4H, OCH_2, ^3J = 6.5 Hz), 4.78 s (1H, OCHO), 6.18 q (1H, CH_3C**H**, ^3J = 6.8 Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 288 (9) $[M]^+$, 216 (40) $[M - C_3H_7CHO]^+$, 215 (19) $[M - C_3H_7CH_2O]^+$, 159 (50) $[C_4H_9OCHOC_4H_9]^+$, 103 (75) $[C_4H_9OCHOC_4H_9 - C_4H_8]^+$, 57 (100) $[C_4H_9]^+$. Found, %: C 66.18; H 11.10; S 11.20. $C_{16}H_{32}O_2S$. Calculated, %: C 66.67; H 11.10; S 11.12.

b. The reaction was carried out as described above in *a* with the difference that 5 mol % of HBr was used as catalyst. After 3 days, the mixture contained the following products (wt %, GC–MS): initial aldehyde **Ia** (18), acetal **IIIa** (38), acetal **Va** (12), dibutyl disulfide (6). Compounds **Ia** and **IIIa** were identified by comparing their GC–MS parameters with those of authentic samples. Mass spectrum of **Va**, *m*/*z* (I_{rel}, %): 175 (30) $[BuOCHSBu]^+$, 119 (62) $[BuOCHSBu - C_4H_8]^+$, 57 (80) $[CH_3CH_2CO]^+$, 29 (100) $[CH_3CH_2]^+$. Mass spectrum of Bu_2S_2, *m*/*z* (I_{rel}, %): 178 (30) $[M]^+$, 122 (25), 87 (10), 79 (8), 57 (98) $[Bu]^+$, 45 (20), 41 (80), 29 (100) $[Et]^+$.

2-Butylsulfanyl-1,1-diethoxy-2-butene (IIIb). A mixture of 0.8 g (0.005 mol) of aldehyde **Ia**, 11.58 g (0.25 mol) of ethyl alcohol, 0.04 g of HBr in 1.25 ml of $CHCl_3$ (10 mol %), and 1.1 g of 3-Å molecular

sieves was kept for 6 days at room temperature. The mixture was neutralized with potassium carbonate, and the solvent was evaporated. According to the NMR data, the residue was 100% of acetal **IIIb**. Vacuum distillation afforded 0.38 g (32%) of **IIIb** with bp 125–127°C (3 mm). ^{1}H NMR spectrum, δ, ppm: 0.88 t (3H, CH_3 in Bu), 1.21 t (6H, CH_3 in Et, ^3J = 7.0 Hz), 1.38 m (2H, C**H**$_2$CH$_3$ in Bu), 1.52 m (2H, SCH$_2$C**H**$_2$), 1.88 d (3H, C**H**$_3$CH, ^3J = 6.8 Hz), 2.70 t (2H, SCH_2, ^3J = 7.4 Hz), 3.46 d.q and 3.60 d.q (4H, OCH_2, ^3J = 7.0 Hz), 4.82 s (1H, OCHO), 6.24 q (1H, CH, ^3J = 6.8 Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 232 (5) $[M]^+$, 188 (9) $[M - CH_3CHO]^+$, 187 (9) $[M - C_2H_5O]^+$, 159 (2), 143 (1) $[M - SC_4H_9]$, 131 (4), 103 (100) $[C_2H_5OCHOC_2H_5]^+$, 75 (58) $[C_2H_5OCHOC_2H_5 - C_2H_4]^+$, 47 (42) $[C_2H_5OCHOC_2H_5 - 2C_2H_4]^+$. Found, %: S 13.92. $C_{12}H_{24}O_2S$. Calculated, %: S 13.80. The ^{1}H NMR spectrum of **IIIb** coincided with that reported in [13] for a sample prepared by reaction of **Ia** with $Si(OEt)_4$.

3,3-Dibutoxy-2-butylsulfanyl-1-propene (IIIc). A mixture of 5.76 g (0.04 mol) of 2-butylsulfanyl-2-propenal (**Ib**), 37 g (0.5 mol) of butanol, 77.9 g (1.05 mol) of diethyl ether, 0.24 g (0.0014 mol, 3.5 mol %) of *p*-toluenesulfonic acid, and 6.84 g of 4-Å molecular sieves was kept for 10 days at room temperature. The mixture was neutralized with dry potassium carbonate, and the solvent was distilled off under reduced pressure. According to the NMR data, the residue contained 73% of acetal **IIIc**, 20% of dimer **VI**, and 7% of acetal **Vc**. The mass spectrum of the latter was identical to that of an authentic sample (see below). By double vacuum distillation we isolated 3.8 g (32.5%) of acetal **IIIc**, bp 117–120°C (2 mm). ^{1}H NMR spectrum, δ, ppm: 0.87 t (6H, CH_3 in OBu), 0.88 t (3H, CH_3 in SBu), 1.38 m (6H, C**H**$_2$CH$_3$), 1.56 m (6H, C**H**$_2$CH$_2$CH$_3$), 2.67 t (2H, SCH_2), 3.40 d.t and 3.52 d.t (4H, OCH_2), 4.84 s (1H, =CH_2), 4.89 s (1H, =CH_2), 5.43 s (1H, OCHO). Mass spectrum, *m*/*z* (I_{rel}, %): 274 (2) $[M]^+$, 217 (1) $[M - C_4H_9]^+$, 202 (30) $[M - C_3H_7CHO]^+$, 201 (8) $[M - C_4H_9O]^+$, 159 (22) $[C_4H_9OCHOC_4H_9]^+$, 146 (7), 145 (11), 103 $[C_4H_9OCHOC_4H_9 - C_4H_9]^+$, 89 (24) $[C_4H_9S]^+$, 73 (7), 57 (100) $[C_4H_9]^+$. Found, %: C 65.60; H 11.09; S 11.42. $C_{15}H_2O_2S$. Calculated, %: C 65.14; H 11.02; S 11.68.

Reaction of 2-butylsulfanyl-2-propenal with ethyl alcohol. A mixture of 5.22 g (0.036 mol) of aldehyde **Ib**, 32.26 g (0.7 mol) of ethyl alcohol, 0.41 g (6.4 mol %) of *p*-toluenesulfonic acid, 121.47 g of diethyl ether, and 7.43 g of 3-Å molecular sieves was kept for two weeks at room temperature and was then neutralized with dry potassium carbonate. According to the ^{1}H NMR spectrum, the yield of acetal **IIId** was 30%, of aldehyde **VII**, 12%, and of acetal **Vd**, 13%. The ^{1}H NMR spectrum and GC–MS parameters of **Vd** were consistent with those reported in [8]. By vacuum distillation we isolated 1 g (12.65%) of 2-butylsulfanyl-3,3-diethoxy-1-propene (**IIId**), bp 98–100°C (5 mm). ^{1}H NMR spectrum, δ, ppm: 0.9 t (3H, CH_3 in Bu), 1.2 t (6H, CH_3 in Et), 1.38 m (2H, CH_2 in Bu), 1.60 m [2H, CH_2 in Bu), 2.65 m (2H, SCH_2), 3.47 d.q and 3.59 d.q (4H, OCH_2), 4.90 s (1H, =CH_2), 4.87 s (1H, =CH_2), 5.44 s (1H, OCHO). Mass spectrum, *m*/*z* (I_{rel}, %): 218 (2) $[M]^+$, 189 (1) $[M - C_2H_5]^+$, 174 (40) $[M - CH_3CHO]^+$, 173 (8) $[M - CH_3CH_2O]^+$, 145 (3), 117 (16), 103 (100) $[C_2H_5OCHOC_2H_5]^+$, 89 (24) $[C_4H_9S]^+$, 75 (83) $[C_2H_5OCHOC_2H_5 - C_2H_4]^+$, 47 (73) $[C_2H_5OCHOC_2H_5 - 2C_2H_4]^+$. Found, %: C 60.12; H 10.27; S 14.68. $C_{11}H_{22}O_2S$. Calculated, %: C 60.55; H 10.08; S 14.69. In addition, vacuum distillation gave (third fraction) 1.25 g (11.3%) of 2,3-bis(butylsulfanyl)-1,1-diethoxypropane (**VII**), bp 155°C (5 mm). ^{1}H NMR spectrum, δ, ppm: 0.85 t (6H, CH_3 in Bu), 1.2 t (6H, CH_3 in Et), 1.35 m (4H, CH_2 in Bu), 1.50 m (4H, CH_2 in Bu), 2.4 m (4H, SCH_2), 2.67 m (1H, CH), 2.80 d.d and 2.87 d.d (2H, C**H**$_2$CH, ^3J = 5.3, ^2J = 12.4 Hz), 3.53 d.q and 3.67 d.q (4H, OCH_2, ^3J = 7.0 Hz), 4.56 d (1H, OCHO, ^3J = 5.0 Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 308 (1) $[M]^+$, 263 (3) $[M - OC_2H_5]^+$, 219 (1) $[M - SC_4H_9]^+$, 205 (1) $[M - CH_2SC_4H_9]^+$, 173 (2), 154 (2), 103 (100) $[C_2H_5OCHOC_2H_5]^+$, 75 (33) $[C_2H_5OCHOC_2H_5 - C_2H_4]^+$, 47 (4) $[C_2H_5OCHOC_2H_5 - 2C_2H_4]^+$. Found, %: C 58.54; H 10.40; S 20.88. $C_{15}H_{32}O_2S_2$. Calculated, %: C 58.39; H 10.38; S 20.80.

1-Butoxy-1-butylsulfanyl-2-propanone (Vc). A mixture of 7.33 g (0.05 mol) of 2-butylsulfanyl-2-propenal (**Ib**), 22.24 g (0.3 mol) of butyl alcohol, and 0.5 ml of concentrated hydrochloric acid was kept for 24 h at room temperature. The mixture was neutralized with K_2CO_3 and evaporated, and the residue was subjected to double vacuum distillation to isolate 3.05 g (28%) of compound **Vc** with bp 175–180°C (1 mm), n_D^{20} = 1.4690. ^{1}H NMR spectrum, δ, ppm: 4.81 s (1H, SCHO), 3.8 m (2H, CH_2S), 3.4 m (2H, CH_2O), 2.24 s (3H, CH_3CO), 1.45 m (4H, CH_2), 1.49 m (4H, CH_2), 0.91 m (6H, CH_3). Mass spectrum, *m*/*z* (I_{rel}, %): 175 (33) $[C_4H_9SCHOC_4H_9]^+$, 119 (100) $[C_4H_9SCHOC_4H_9 - C_4H_8]^+$, 63 (23) $[C_4H_9SCHOH - C_4H_8]^+$, 57 (79) $[C_4H_9]^+$, 43 (26) $[CH_3CO]^+$. Found, %: C 60.60; H 10.20; S 14.75. $C_{11}H_{22}O_2S$. Calculated, %: C 60.55; H 10.09; S 14.68. The preceding fraction contained acetals **Vc** and **IVc** at a ratio of 1:1. The ^{1}H NMR

spectrum of **IVc** was in agreement with the data given in [17]. Mass spectrum of **IVc**, *m*/*z* (I_{rel}, %): 159 (6) $[BuOCHOBu]^+$, 129 (2), 103 (14) $[M - C_4H_8]^+$, 57 (100) $[Bu]^+$, 47 (4), 43 (22) $[CH_3CO]^+$, 41 (40).

Reaction of 3,3-dibutoxy-2-butylsulfanyl-1-propene (IIIc) with water. A mixture of 2.97 g (0.01 mol) of acetal **IIIc**, 0.2 g (0.1 mol) of water, and 0.19 g (0.001 mol, 10 mol %) of *p*-toluenesulfonic acid was kept for 9 days at room temperature. According to the NMR data, the mixture contained 89% of acetal **Vc**; the GC–MS data of **Vc** coincided with those given above.

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