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Reactions of 2-Chloro-4,4-ethylenedioxy-3-phenylsulfonylcyclopent-2-en-1-one with Some Hydride Reducing Agents and Carbon-Centered Nucleophiles

N. A. Ivanova, F. G. Usmanova, and M. S. Miftakhov

Institute of Organic Chemistry, Ufa Research Center, Russian Academy of Sciences, pr. Oktyabrya 71, Ufa, 450054 Bashkortostan, Russia
e-mail: bioreg@anrb.ru

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Abstract—2-Chloro-4,4-ethylenedioxy-3-phenylsulfonylcyclopent-2-en-1-one reacts with $NaBH_4$ and $LiAlH_4$ to give, depending on the conditions, 4,4-ethylenedioxy-2-chloro-3-phenylsulfonylcyclopent-2-en-1-ol and 4,4-ethylenedioxy-3-phenylsulfonylcyclopent-2-en-1-ol, as well as 4,4-ethylenedioxy-3-phenylsulfonylcyclopentan-1-ol. Reactions of the title compound with diethyl malonate under basic conditions occurs at the double-bonded carbon atoms to form a couple of the addition–elimination products at C^2 and C^3.

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We previously described the synthesis and reductive transformations of highly functionalized cyclopentene building block **I** [1] which was designed for the development of new approaches to prostaglandins [2], carbanucleosides [3], and their analogs. In the present work we continued study on the hydride reduction and vinylic nucleophilic substitution reaction of sulfonylcyclopentenone acetal **I** with the goal of estimating its reactivity and obtaining more important precursors of the above compounds.

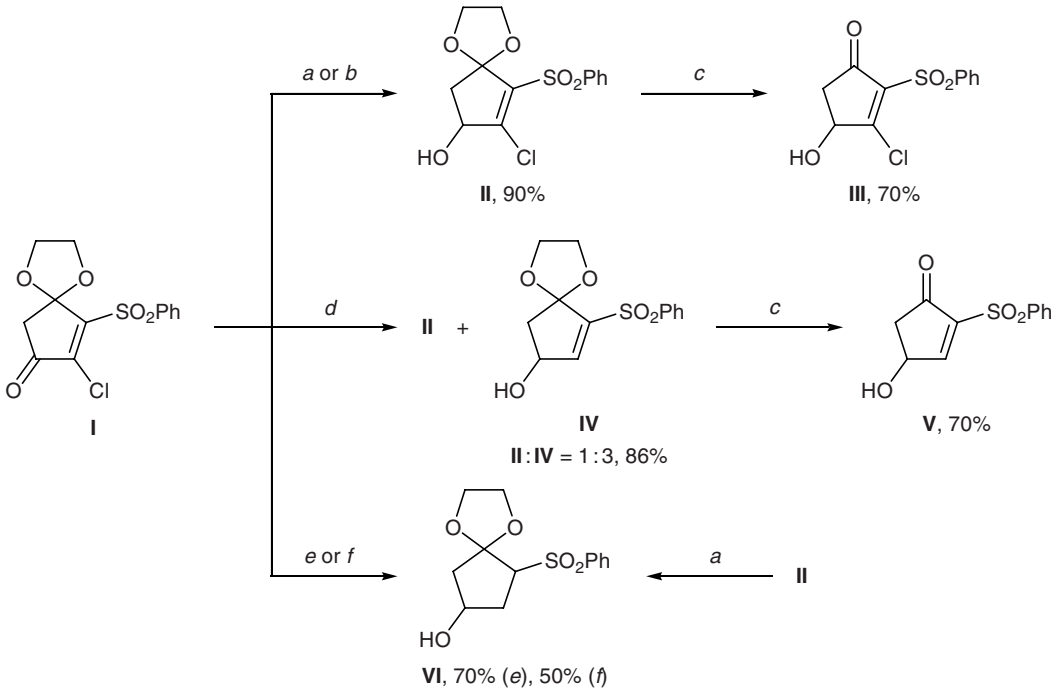
Experiments with accessible reducing agents ($NaBH_4$ and $LiAlH_4$) showed that, depending on the conditions, the reduction may involve the carbonyl group, double C=C bond, and/or vinylic chlorine atom on C^2. Using the system $NaBH_4$–$CeCl_3$ [4] or 1 mol of $NaBH_4$ in EtOH–THF at 0°C, followed by treatment under mild conditions, we succeeded in effecting chemoselective reduction of the ketone group in **I** and obtaining hydroxy acetal **II** (Scheme 1) [1]. The ethylene acetal protecting group in **II** is anomalously sensitive to hydrolysis, so that partial formation of hydroxy ketone **III** was observed even when the reaction mixture was treated with water. Such a behavior of structurally related ketone acetals having no sulfonyl group was discussed by us previously: we presumed intramolecular assistance to hydrolysis by the free hydroxy group in **II** [5, 6].

Increase of the amount of hydride reagent to 1.5 equiv and prolongation of the reaction to 1 h with a view to obtain alcohol **V** led to formation of a mixture of acetals **II** and **IV** at a ratio of 1:3; the product mixture can be readily separated by column chromatography on silica gel. Like hydroxy acetal **II**, compound **IV** is readily converted into the corresponding hydroxy enone **V** on treatment with dilute aqueous solutions of mineral acids.

Further raising the amount of the reducing agent (2 equiv of $NaBH_4$) and reaction time or performing the reduction with $LiAlH_4$ at room temperature resulted in the formation of complete reduction product, alcohol **VI**. Unlike compound **V**, the acetal protecting group in alcohol **VI** is stable, and it remains unchanged upon acid treatment of the reaction mixture. Presumably, conformational or other variations in the molecule eliminate assistance by the hydroxy group. Cyclopentanol **VI** can also be synthesized by hydride reduction of unsaturated alcohol **II**.

We also examined the behavior of unsaturated keto sulfone **I** in reactions with carbon-centered nucleophiles. We found that in the reaction with diethylmalonate in the presence of potassium hydroxide substitution is possible at both vinylic carbon atoms. It should be emphasized that the phenylsulfonyl group was replaced more readily than the chlorine atom, and com-

Scheme 1.



a: $NaBH_4$ (1.0 equiv), EtOH–THF (1 : 1), 0°C, 20 min, $+Me_2CO$; *b*: $NaBH_4$ (4.0 equiv), $CeCl_3 \cdot 7H_2O$ (4 equiv), MeOH, –30°C, 1 h, $+Me_2CO$; *c*: H^+/H_2O; *d*: $NaBH_4$ (1.5 equiv), EtOH–THF (1 : 1), 0°C, 1 h, $+Me_2CO$; *e*: $NaBH_4$ (2.0 equiv), EtOH–THF (1 : 1), 20°C, 4 h, $+H^+/H_2O$; *f*: $LiAlH_4$ (2.0 equiv), Et_2O–THF (1 : 1), 20°C, 4 h, $+H^+/H_2O$.

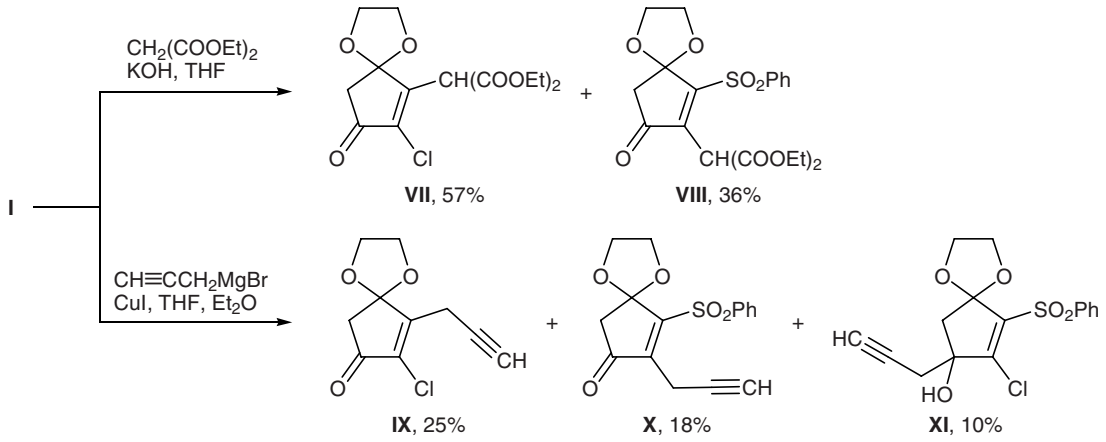
pounds **VII** and **VIII** were formed at a ratio of 3 : 2. Obviously, this is the result of stronger electron-withdrawing effect of the oxo group on the β-carbon atom in the conjugated enone system. The reaction of **I** with copper reagent obtained from prop-2-yn-1-ylmagnesium bromide and CuI was less selective; apart from the corresponding replacement products **IX** and **X**, we isolated 1,2-adduct **XI** (Scheme 2).

Thus the results of our study allowed us to refine some aspects of the reactivity of vinylic sulfone **I** and develop procedures for the preparation of synthetic blocks **II**–**VI** that are interesting as intermediates in the convergent synthesis of prostanoids and other cyclopentanoids.

EXPERIMENTAL

The IR spectra were recorded on UR-20 and Specord M-80 spectrometers from samples prepared as thin films or dispersed in mineral oil. The ^1H and ^{13}C NMR spectra were measured on a Bruker AM-300

Scheme 2.



instrument at 300.13 and 75.47 MHz, respectively, using chloroform-*d* as solvent and reference ($CHCl_3$, δ 7.27 ppm; $CDCl_3$, δ_C 77.00 ppm). The progress of reactions was monitored by TLC on Silufol plates using petroleum ether–ethyl acetate as eluent; spots were visualized by treatment with a *p*-methoxybenzaldehyde-based reagent or an alkaline solition of potassium permanganate [7].

Reaction of compound I with $NaBH_4$. A solution of 0.2 g (0.64 mmol) of keto sulfone **I** in 10 ml of a 1:1 ethanol–tetrahydrofuran mixture was cooled to 0°C, 0.024 g (0.64 mmol) of sodium tetrahydridoborate was added, and the mixture was stirred for 20 min and treated as follows (methods *a* and *b*)

a. The mixture was diluted with 10 ml of acetone, the solvent was distilled under reduced pressure, and the residue was subjected to flash chromatography on silica gel using petroleum ether–ethyl acetate (7:3) as eluent to isolate 0.18 g (90%) of alcohol **II**.

b. The mixture was treated with 5 ml of 5% hydrochloric acid, the solvent was distilled off under reduced pressure, the residue was diluted with 10 ml of a saturated solution of sodium chloride, and the mixture was extracted with chloroform (3×10 ml). The extracts were combined, washed with water, dried over $MgSO_4$, and concentrated under reduced pressure. The residue was subjected to column chromatography on silica gel using petroleum ether–ethyl acetate (7:3) as eluent to isolate 0.14 g (70%) of hydroxy ketone **III**.

2-Chloro-4,4-ethylenedioxy-3-phenylsulfonylcyclopent-2-en-1-ol (II). Colorless oily substance, R_f 0.38 (petroleum ether–ethyl acetate, 7:3; three successive elutions). IR spectrum, ν, cm^{-1}: 1342 (SO_2), 1615 (C–C_{arom}), 3400 (OH). ^1H NMR spectrum, δ, ppm: 2.11 d.d (1H, 5-H_A, J = 4.0, 16.0 Hz), 2.60 d.d (1H, 5-H_B, J = 4.0, 16.0 Hz), 3.70–3.90 m (4H, CH_2O), 4.60 br.s (1H, 4-H), 7.10–7.3 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 45.22 (C^5), 66.13 and 65.76 (CH_2O), 70.96 (C^1), 115.20 (C^4), 146.88 (C^2), 130.14 (C^3), 128.01 (C^o), 129.09 (C^m), 129.95 (C^p), 132.19 (C^i). Found, %: C 49.34; H 4.23; Cl 11.28; S 9.33. $C_{13}H_{13}ClO_5S$. Calculated, %: C 49.29; H 4.14; Cl 11.19; S 9.56.

3-Chloro-4-hydroxy-2-phenylsulfonylcyclopent-2-en-1-one (III). Colorless crystals, mp 111–113°C, R_f 0.34 (petroleum ether–ethyl acetate, 7:3; three successive elutions). IR spectrum, ν, cm^{-1}: 1344 (SO_2), 1584 (C–C_{arom}), 1733 (C=O), 3400 (OH). ^1H NMR spectrum, δ, ppm: 2.23 m (2H, 5-H), 4.39 m (1H, 4-H), 7.50 m (2H, *o*-H), 7.57 m (1H, *p*-H), 7.96 (2H, *m*-H). ^{13}C NMR spectrum, δ_C, ppm: 45.22 (C^5), 78.59 (C^4), 129.10 (C^o), 130.39 (C^m), 133.92 (C^p), 134.82 (C^i), 148.02 (C^2), 176.95 (C^3), 195.92 (C^1). Found, %: C 48.32; H 3.17; Cl 12.85; S 11.58. $C_{11}H_9ClO_4S$. Calculated, %: C 48.45; H 3.33; Cl 13.00; S 11.76.

4,4-Ethylenedioxy-3-phenylsulfonylcyclopent-2-en-1-ol (IV). A solution of 0.2 g (0.64 mmol) of keto sulfone **I** in 10 ml of a 1:1 ethanol–tetrahydrofuran mixture was cooled to 0°C, 0.036 g (0.96 mmol) of sodium tetrahydridoborate was added, and the mixture was stirred for 1 h and was then treated as described above in *a*. By column chromatography on silica gel using petroleum ether–ethyl acetate (7:3) as eluent we isolated 0.04 g (22%) of compound **II** and 0.11 g (64%) of alcohol **IV** as a yellow oily substance, R_f 0.36 (petroleum ether–ethyl acetate, 7:3; three successive elutions). IR spectrum, ν, cm^{-1}: 1340 (SO_2), 1590 (C–C_{arom}), 3390 (OH). ^1H NMR spectrum, δ, ppm: 2.20 d.d (1H, 5-H_A, J = 6.3, 15.4 Hz), 2.72 d.d (1H, 5-H_B, J = 6.3, 15.4 Hz), 3.91–4.32 m (5H, CH_2O, OH), 4.60 m (1H, 1-H), 6.17 m (1H, 3-H) 7.01–7.23 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 46.59 (C^5), 65.78 (CH_2O), 66.32 (CH_2O), 71.15 (C^1), 114.74 (C^4), 125.26 (C^o), 127.37 (C^m), 128.67 (C^p), 135.46 (C^i), 142.01 (C^2), 158.45 (C^3). Found, %: C 55.42; H 5.15; S 11.49. $C_{13}H_{14}O_5S$. Calculated, %: C 55.31; H 5.00; S 11.36.

4-Hydroxy-2-phenylsulfonylcyclopent-2-en-1-one (V). A solution of 0.2 g (0.64 mmol) of keto sulfone **I** in 10 ml of a 1:1 ethanol–tetrahydrofuran mixture was cooled to 0°C, 0.036 g (0.96 mmol) of sodium tetrahydridoborate was added, and the mixture was stirred for 1 h and was then treated as described above in *b*. Column chromatography on silica gel (eluent petroleum ether–ethyl acetate, 7:3) gave 0.13 g (70%) of compound **V** as a colorless oily substance, R_f 0.26 (petroleum ether–ethyl acetate, 1:1). IR spectrum, ν, cm^{-1}: 1344 (SO_2), 1696 (C=C), 1712 (C=O), 3400 (OH). ^1H NMR spectrum, δ, ppm: 3.0 br.s (1H, OH), 3.15 m (2H, 5-H), 4.32 m (1H, 4-H), 6.11 m (1H, 3-N), 7.70 m (2H, *o*-H), 7.77 m (1H, *p*-H), 7.88 m (2H, *m*-H). ^{13}C NMR spectrum, δ_C, ppm: 30.73 (C^5), 65.35 (C^4), 128.91 (C^o), 133.1 (C^m), 134.23 (C^p), 136.32 (C^i), 138.32 (C^2), 163.92 (C^3), 197.25 (C^1). Found, %: C 55.32; H 4.05; S 13.41. $C_{11}H_{10}O_4S$. Calculated, %: C 55.45; H 4.23; S 13.46.

4,4-Ethylenedioxy-3-phenylsulfonylcyclopentan-1-ol (VI). *a*. A solution of 0.50 g (1.56 mmol) of sulfone **I** in 10 ml of anhydrous tetrahydrofuran was added dropwise at 0°C to a suspension of 0.12 g (3.12 mmol) of $LiAlH_4$ in 10 ml of anhydrous diethyl

ether. The mixture was stirred for 4 h at room temperature, 5 ml of 5% hydrochloric acid was added, and the mixture was concentrated under reduced pressure. The residue was diluted with 10 ml of water, and the product was extracted into chloroform (3×10 ml). The extracts were combined, washed with water, and dried over $MgSO_4$, the solvent was distilled off under reduced pressure, and the residue was subjected to column chromatography on silica gel using petroleum ether–ethyl acetate (7:3) as eluent to isolate 0.25 g (50%) of a ~1:1 mixture of diastereoisomeric alcohols **VI** as a colorless oily substance, R_f 0.15 (benzene–ethyl acetate, 7:3; two successive elutions).

b. A solution of 0.2 g (0.64 mmol) of sulfone **I** in 10 ml of a 1:1 ethanol–tetrahydrofuran mixture was cooled to 0°C, 0.048 g (1.28 mmol) of sodium tetrahydridoborate was added, and the mixture was stirred for 4 h and was then treated as described in *b* for compound **II**. The product was isolated by column chromatography on silica gel using petroleum ether–ethyl acetate (7:3) as eluent. Yield 0.13 g (70%). IR spectrum, ν, cm^{-1}: 1306 (SO_2), 1450 (C–C_{arom}), 3508 (OH). ^{1}H NMR spectrum, δ, ppm: 1.92–2.51 m (4H, 2-H, 5-H), 2.88 br.s (1H, OH), 3.53–3.90 m (5H, CH_2O, 3-H), 4.15 q (0.5H, 1-H, J = 6.2 Hz), 4.33 br.s (0.5H, 1-H, J = 6.2 Hz), 7.51–7.88 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 35.54 and 36.38 (C^2), 43.77 and 45.86 (C^5), 64.20 and 65.27 (CH_2O), 64.55 and 65.13 (CH_2O), 67.95 and 68.03 (C^3), 68.36 and 68.82 (C^1), 114.12 and 115.10 (C^4), 128.44 and 128.59 (C^o), 128.80 (C^m), 133.33 and 133.44 (C^p), 139.10 and 139.30 (C^i). Found, %: C 54.80; H 5.54; S 11.35. $C_{13}H_{16}O_5S$. Calculated, %: C 54.91; H 5.67; S 11.28.

Reaction of sulfone I with diethyl malonate. Potassium hydroxide, 0.20 g (3.52 mmol), and diethyl malonate, 0.51 g (3.18 mmol), were added at room temperature to a solution of 0.20 g (0.64 mmol) of sulfone **I** in 8 ml of THF. The mixture was stirred for 20 min, diluted with 25 ml of ethyl acetate and 5 ml of 10% hydrochloric acid, and extracted with ethyl acetate (2×15 ml). The extracts were combined, washed with 10% hydrochloric acid to pH 7 and a saturated solution of sodium chloride, and dried over $MgSO_4$, the solvent was distilled off under reduced pressure, and the residue was subjected to column chromatography on silica gel using petroleum ether–ethyl acetate (8:2) as eluent to isolate 0.08 g (57%) of enone **VII** and 0.05 g (36%) of sulfone **VIII**.

Diethyl 2-(2-chloro-5,5-ethylendioxy-3-oxocyclopent-1-en-1-yl)malonate (VII). Yellow crystals, mp 46–48°C, R_f 0.33 (petroleum ether–ethyl acetate, 7:3; three successive elutions). IR spectrum, ν, cm^{-1}: 1024 (CH_2), 1148 (O–C–O), 1584 (C=C), 1738 (C=O, ester), 1756 (C=O, ketone). ^{1}H NMR spectrum, δ, ppm: 1.24 t (6H, CH_3, J = 7.1 Hz), 2.69 s (2H, 4-H), 4.02 m and 4.10 m (4H, OCH_2CH_2O), 4.22 q (4H, $OC\mathbf{H}_2CH_3$, J = 7.1 Hz), 4.41 s (1H, 1-CH). ^{13}C NMR spectrum, δ_C, ppm: 13.80 (CH_3), 45.40 (C^4), 49.34 (1-CH), 62.33 and 65.94 (CH_2O), 109.44 (C^5), 136.35 (C^2), 154.47 (C^1), 164.95 (OC=O), 194.50 (C^3). Found, %: C 50.67; H 5.01; Cl 10.84. $C_{14}H_{17}ClO_7$. Calculated, %: C 50.54; H 5.15; Cl 10.65.

Diethyl 2-(3,3-ethylenedioxy-5-oxo-2-phenylsulfonylcyclopent-1-en-1-yl)malonate (VIII). Colorless crystals, mp 95–97°C, R_f 0.21 (petroleum ether–ethyl acetate, 7:3; three successive elutions). IR spectrum, ν, cm^{-1}: 1048 (O–C–O), 1240 (SO_2), 1372 (SO_2), 1590 (C–C_{arom}), 1738 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.21 t (3H, CH_3, J = 7.0 Hz), 2.75 s (2H, 4-H), 3.99 m (2H, OCH_2), 4.14 q (4H, OCH_2, J = 6.9 Hz), 4.24 m (2H, OCH_2), 5.38 s (1H, CH) 7.56 q (2H, *m*-H, J = 7.4, 7.8 Hz), 7.67 t (1H, *p*-H, J = 7.2, 7.4 Hz), 7.97 d (2H, *o*-H, J = 7.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 13.91 (CH_3), 48.39 (C^4), 62.40 ($O\mathbf{C}H_2CH_3$), 66.44 (CH_2O), 111.24 (C^2), 128.05 (C^o), 129.27 (C^m), 134.43 (C^p), 140.51 (C^i), 145.42 (C^1), 158.91 (C^2), 164.99 (OC=O), 198.01 (C^5). Found, %: C 54.62; H 5.08; S 7.23. $C_{20}H_{22}O_9S$. Calculated, %: C 54.79; H 5.06; S 7.31.

Reaction of sulfone I with prop-2-yn-1-ylmagnesium bromide. Copper(I) iodide, 0.18 g (0.95 mmol), was added under argon to 0.56 ml (1.43 mmol) of a 2.58 M solution of prop-2-yn-1-ylmagnesium bromide in anhydrous tetrahydrofuran, cooled to 0°C, and the mixture was stirred for 20 min. The mixture was cooled to –50°C, a solution of 0.3 g (0.95 mmol) of sulfone **I** in 4 ml of anhydrous THF was added dropwise over a period of 10 min, and the mixture was stirred for 1 h at 0°C and for 12 h at room temperature. It was then cooled to 0°C, treated with 2 ml of a saturated solution of ammonium chloride, and extracted with ethyl acetate (3×15 ml). The extracts were combined, washed with a saturated solution of sodium chloride, dried over magnesium sulfate, and concentrated under reduced pressure. The residue was subjected to chromatography on silica gel using petroleum ether–ethyl acetate (8:2) as eluent to isolate 0.09 g (30%) of initial sulfone **I**, 0.04 g (25%) of chloro ketone **IX**, 0.04 g (18%) of keto sulfone **X**, and 0.03 g (10%) of alcohol **XI**.

2-Chloro-4,4-ethylenedioxy-3-(prop-2-yn-1-yl)-cyclopent-2-en-1-one (IX). Yellow crystals, mp 90–

93°C, R_f 0.26 (petroleum ether–ethyl acetate, 7:3; three successive elutions). ^{1}H NMR spectrum, δ, ppm: 2.05 t (1H, ≡CH, J = 3.1 Hz), 2.23 d (1H, 5-H$_A$, J = 13.9 Hz), 2.50 d (1H, 5-H$_B$, J = 13.9 Hz), 3.50 d (1H, 1′-H$_A$, J = 12.5 Hz), 3.56 d (1H, 1′-H$_B$, J = 12.5 Hz), 4.05–4.28 m and 4.28–4.48 m (4H, CH_2O). ^{13}C NMR spectrum, δ_C, ppm: 28.09 (C$^{1'}$), 46.98 (C^5), 57.14 and 58.54 (CH_2O), 64.51 (C$^{3'}$), 77.50 (C$^{2'}$), 108.57 (C^4), 135.05 (C^2), 158.79 (C^3), 191.70 (C^1). Found, %: C 56.61; H 4.35; Cl 16.58. $C_{10}H_9ClO_3$. Calculated, %: C 56.49; H 4.27; Cl 16.67.

4,4-Ethylenedioxy-3-phenylsulfonyl-2-(prop-2-yn-1-yl)cyclopent-2-en-1-one (X). Brown oily substance, R_f 0.33 (petroleum ether–ethyl acetate, 7:3; three successive elutions). IR spectrum, ν, cm^{-1}: 1033, 1145, 1342 (SO_2), 1610 (C–C$_{arom}$), 1725 (C=O), 2124 (C≡C), 3305 (≡C–H). ^{1}H NMR spectrum, δ, ppm: 1.98 t (1H, ≡CH, J = 2.7 Hz), 2.28 d (1H, 1′-H$_A$, J = 14.0 Hz), 2.62 d.d (1H, 1′-H$_B$, J = 2.7, 14.0 Hz), 2.47 d (1H, 5-H$_A$, J = 14.0 Hz), 2.72 d.d (1H, 5-H$_B$, J = 2.7, 14.0 Hz), 4.03 m and 4.36 m (4H, CH_2O), 7.55–7.62 m (3H, H$_{arom}$), 8.03 m (2H, H$_{arom}$). ^{13}C NMR spectrum, δ_C, ppm: 29.07 (C$^{1'}$), 50.20 (C^5), 66.22 and 66.37 (CH_2O), 77.32 (≡CH), 79.25 (C$^{2'}$), 114.46 (C^4), 128.13 (C^o), 129.00 (C^m), 133.89 (C^p), 139.20 (C^i), 140.94 (C^2), 151.75 (C^3), 193.75 (C^1). Found, %: C 60.23; H 4.58; S 10.46. $C_{16}H_{14}ClO_4S$. Calculated, %: C 60.37; H 4.43; S 10.37.

2-Chloro-4,4-ethylenedioxy-3-phenylsulfonyl-1-(prop-2-yn-1-yl)cyclopent-2-en-1-ol (XI). Yellow oily substance, R_f 0.13 (petroleum ether–ethyl acetate, 7:3; three successive elutions). ^{1}H NMR spectrum, δ, ppm: 1.93 d (1H, 1′-H$_A$, J = 26.7 Hz), 2.25 d.d (1H, 1′-H$_B$, J = 2.6, 26.7 Hz), 2.00 s (1H, ≡CH), 2.56 d (1H, 5-H$_A$, J = 2.6 Hz), 2.65 d.d (1H, 5-H$_B$, J = 2.6, 25.6 Hz), 2.60 br.s (1H, OH), 4.13–4.36 m (4H, CH_2O), 7.52–7.62 m (3H), 8.00 d (2H). ^{13}C NMR spectrum, δ_C, ppm: 28.80 (C$^{1'}$), 49.91 (C^5), 65.99 and 66.05 (CH_2O), 76.59 (≡CH), 77.84 (C$^{2'}$), 79.14 (C^1), 114.06 (C^4), 127.86 (C^o), 128.76 (C^m), 133.70 (C^p), 138.90 (C^i), 140.78 (C^3), 152.94 (C^2). Found, %: C 54.23; H 4.42; Cl 10.04; S 9.11. $C_{16}H_{15}ClO_5S$. Calculated, %: C 54.16; H 4.26; Cl 9.99; S 9.04.

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