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Oxidation of Substituted 1,5-Hexadien-3-ols with Various Oxidants*

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Abstract—Substituted 1,5-hexadien-3-ols were synthesized by the [2,3]-Wittig rearrangement of unsymmetrical bis-allyl ethers, as well as by reactions of 1-(2-alkenyl)-2-chloromethyloxiranes with Mg/THF. The products were oxidized with pyridinium chlorochromate (PCC), zinc chlorochromate (ZCC), *tert*-butyl hydroperoxide in the presence of OsO_4, and *tert*-butyl hydroperoxide alone. The oxidation of substituted 1,5-hexadien-3-ols with PCC and ZCC gave the corresponding carbonyl compounds. In the reaction with *tert*-butyl hydroperoxide catalyzed by OsO_4 the internal double bond in the substrate was regioselectively converted into epoxy group, whereas allylic oxidation was prevented.

Oxidation reactions are very important for organic chemistry and biological systems. Organic molecules having benzylic or allylic hydrogen atoms are oxidized with atmospheric oxygen or hydroperoxides to give various products. Substituted 1,5-hexadien-3-ols possess allylic hydrogen atoms in different positions, which promote their ready autooxidation. Substituted 1,5-hexadien-3-ols are used in the synthesis of pheromones and antibiotics [1–3]; they also exhibit a very strong antimicrobial activity [4]. Three methods for preparation of substituted 1,5-hexadien-3-ols are known. The first of these is based on the Grignard reaction of unsaturated carbonyl compounds with allyl- or vinylmagnesium halides [5–9]. The second procedure involves four sigmatropic rearrangements initiated by the regioselective [2,3]-Wittig rearrangement of unsymmetrical bisallyl ethers [10–14]. The third method is reduction of 1-(2-alkenyl)-2-chloromethyloxiranes with metallic sodium in methanol or their reaction with magnesium in THF [15–19].

Numerous oxidants are now available for organic compounds, and their list is supplemented almost dayly [20–24]. Among these, the main are pyridinium chlorochromate (PCC), zinc chlorochromate (ZCC), OsO_4, and *t*-BuOOH, which are used in combination with various catalysts. Depending on the oxidant and substrate structure, various oxidation products are formed. Pyridinium chlorochromate and zinc chlorochromate are cheaper and easy to prepare, and procedures utilizing these oxidants are fairly simple. PCC becomes a moderately strong oxidant provided that its hygroscopicity and weakly acidic character are modified by addition of sodium acetate. ZCC is a very potent oxidant with respect to compounds having allylic and benzylic hydrogen atoms. Osmium(VIII) oxide is a very expensive and toxic reagent. A disadvantage of *t*-BuOOH as oxidant is that it promotes radical processes which result in formation of a number of products. The use of *tert*-butyl hydroperoxide in combination with a catalytic amount of metal oxide ensures selective oxidation of hydroxy groups to carbonyl. Oxidation of allyl-like alcohols with *t*-BuOOH in the presence of some catalysts yields the corresponding α,β-epoxy alcohols [25–33]. Martin *et al.* used *tert*-butyl hydroperoxide together with $VO(acac)_2$ to synthesize glycosidase inhibitors. The same oxidizing system was also used for the synthesis of (+)-KDO from furan precursors [34–36]. Ho and Sapp [37] studied by mass spectrometry the mechanism of oxidation of the furan ring with the system *t*-BuOOH–$VO(acac)_2$.

Analysis of published data shows that oxidation reactions of substituted 1,5-hexadienes have not been studied in sufficient detail. The oxidants used in the present work were thoroughly selected, taking into consideration the substrate structure. Initial 1,5-hexadien-3-ols were prepared by two different methods (Scheme 1).

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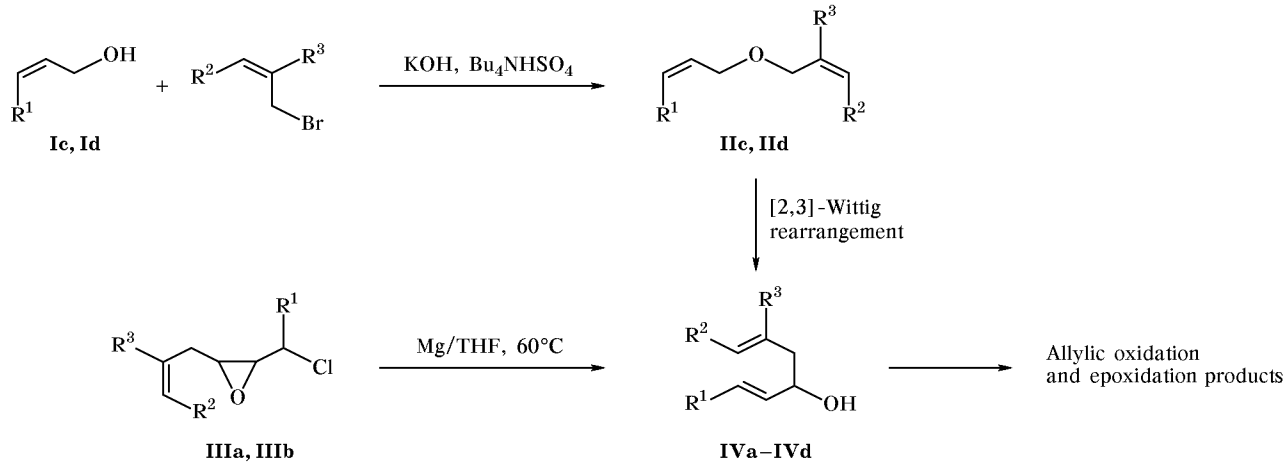
Scheme 1.

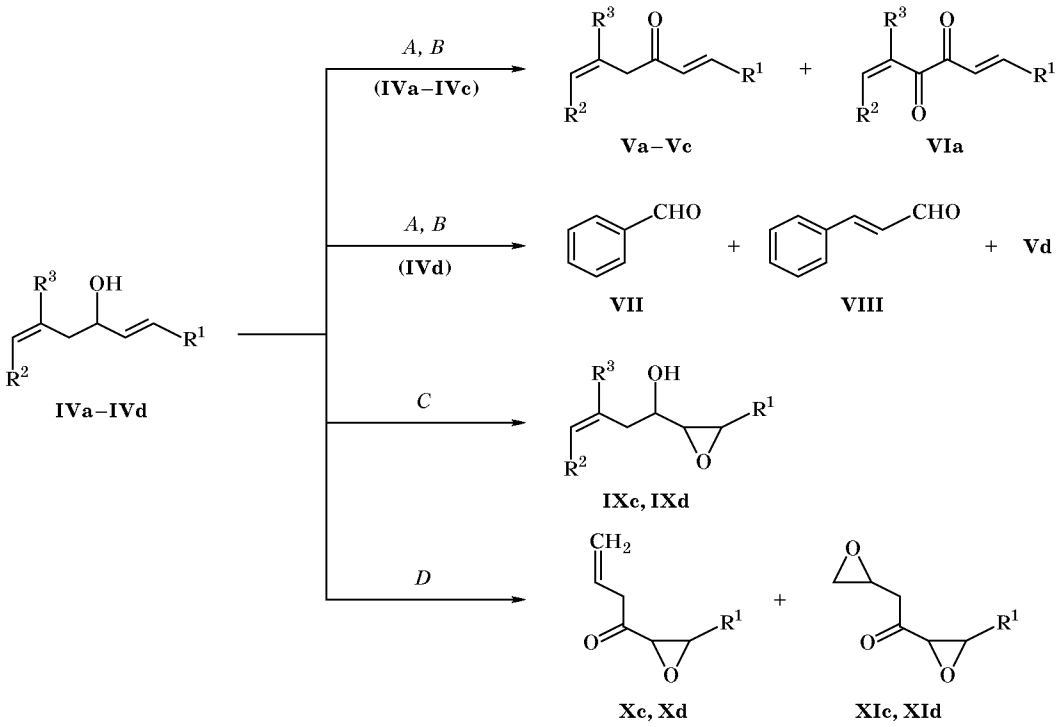


$R^1 = R^2 = H$, $R^3 = CH_3$ (**a**); $R^1 = R^3 = CH_3$, $R^2 = H$ (**b**); $R^1 = CH_3$, $R^2 = R^3 = H$ (**c**); $R^1 = C_6H_5$, $R^2 = R^3 = H$ (**d**).

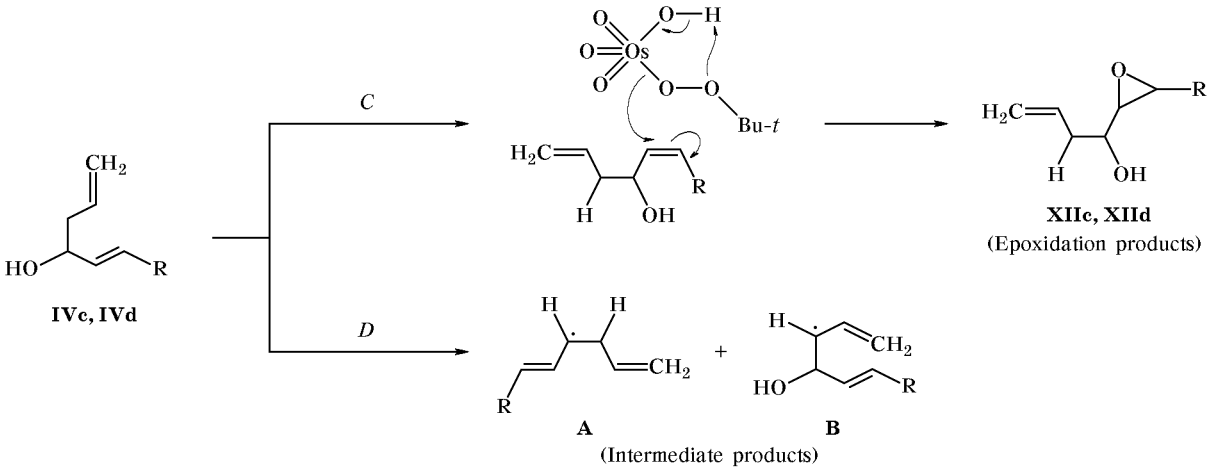
Unsymmetrical bisallyl ethers **IIc** and **IId** were synthesized by reactions of *trans*-2-buten-1-ol (**Ic**) and *trans*-3-phenyl-2-propen-1-ol (**Id**) with allyl bromide in the presence of potassium hydroxide and tetrabutylammonium hydrogen sulfate as phase-transfer catalyst. Substituted 1,5-hexadien-3-ols were obtained in high yield by the [2,3]-Wittig rearrangement of ethers **IIc** and **IId** at –75°C under argon [10–14]. Compounds **IIIa**, **IIIb**, **IVa**, and **IVb** were synthesized by the procedures described in [15–19].

The oxidation of compounds **IVa**–**IVd** with PCC (method *A*) and ZCC (method *B*) gave the corresponding carbonyl compounds **Va** and **Vb**. In the oxidation of **IVa** with PCC product **VIa** was isolated in addition to **Va**. Being a strong oxidant with respect to allylic and benzylic hydrogen atoms, ZCC oxidized alcohol

Scheme 2.



Scheme 3.



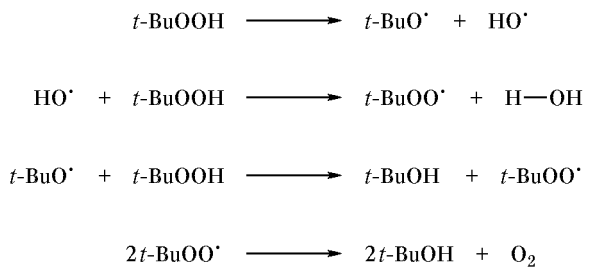
IVa to 2-methyl-1,5-hexadiene-3,4-dione (**VIa**); the latter was formed as a result of oxidation of both allylic hydrogen atom and allylic hydroxy group in **IVa** (Scheme 2). By oxidation of compound **IVd** with PCC and ZCC we obtained benzaldehyde (**VII**), cinnamaldehyde (**VIII**), and 1-phenyl-1,5-hexadien-3-one (**Vd**). The fraction of benzaldehyde was greater when the reaction was performed with ZCC as oxidant. It should be noted that oxidation of **IVd** with both PCC and ZCC gives products with smaller molecular weights owing to decomposition of the initial molecule (Scheme 2).

Presumably, the oxidation of olefins having allylic hydrogen atoms follows two pathways: allylic oxidation and direct attack on the double bond. In the presence of such oxygen carriers as *t*-BuOOH or H_2O_2, the metal can serve as a relay for transfer of oxygen atom from the hydroperoxide to the olefin via an oxometal intermediate. The dismutation of *t*-BuOOH to *t*-BuOH and O_2 in the presence of transition metals has been well documented. *tert*-Butyl hydroperoxide is known to oxidize olefins to epoxy derivatives and allylic oxidation products in the presence of transition metal catalysts.

Catalytic epoxidation and allylic oxidation reactions follow different paths. The first of these involves oxygen transfer, while the second is a radical process (Scheme 3). The epoxidation could occur via activation of the peroxide oxygen atoms.

In the oxidation of substituted 1,5-hexadien-3-ols with *tert*-butyl hydroperoxide catalyzed by OsO_4 (method *C*), only the internal double bond was converted into epoxy group while no allylic oxidation products were formed. α,β-Epoxy alcohols and benzaldehyde were obtained only by oxidation of compound **IVd** according to method *C*. On the other hand, allylic hydrogen atom or allylic hydroxy group could be abstracted by *t*-BuO$^{\cdot}$ or *t*-BuOO$^{\cdot}$ radicals. The oxygen–oxygen bond in alkyl hydroperoxides is very weak, and its dissociation produces radical species which are capable of initiating the other chains (Scheme 4).

Scheme 4.



The oxidation of compounds **IVc** and **IVd** with *t*-BuOOH in the absence of a catalyst (method *D*) gives both epoxidation and allylic oxidation products. We believe that the oxidation with *tert*-butyl hydroperoxide is a completely radical process which involves formation of two different intermediates. Compounds **IVa**–**IVd** have allylic hydrogen atoms, allylic hydroxy groups (**IVa**–**IVc**), and benzylic hydrogen atom (**IVd**) in different positions. *tert*-Butyldioxy radical formed by decomposition of *t*-BuOOH oxidizes the hydroxy group to carbonyl (intermediate **A**); simultaneously, one or both double bonds are converted into epoxy group. The *t*-BuOO$^{\cdot}$ radical behaves very selectively in the formation of intermediate product of allylic oxidation. The methyl group in

IVc acts as electron donor through the hyperconjugation effect. π-Conjugation of the phenyl group with the double bond in compound **IVd** increases the stability of intermediate **A** (Scheme 3), so that none of the oxidation products is formed through intermediate **B**.

When the oxidation was effected with *t*-BuOOH and a catalytic amount of OsO_4, only the internal double bond of the substrate was converted into epoxy group. No allylic oxidation products were obtained following method *C*. Osmium(VIII) oxide prevents both transformation of the terminal double bond into epoxy group and allylic oxidation; OsO_4 also provides regioselective oxidation according to method *C*. In method *D*, *tert*-butyldioxy radical selectively oxidizes the allylic hydroxy group. The reaction with **IVd** is also accompanied by formation of benzaldehyde as a result of benzylic oxidation.

Thus, *tert*-butyl hydroperoxide in the presence of OsO_4 as catalyst selectively oxidizes substituted 1,5-hexadien-3-ols to α,β-epoxy alcohols. The use of *t*-BuOOH alone results in oxidation of the allylic hydroxy group to carbonyl with simultaneous transformation of the double bond into epoxy group.

EXPERIMENTAL

Chromium(VI) oxide, zinc(II) chloride, tetrabutylammonium hydrogen sulfate, butyllithium (as a 1.6 M solution in hexane), *tert*-butyl hydroperoxide (as a 3 M solution in isooctane), pyridine, *trans*-2-buten-1-ol, *trans*-3-phenyl-2-propen-1-ol, and *tert*-butyl alcohol were commercial products. All solvents were used without additional purification.

The progress of reactions was monitored by thin-layer chromatography on silica gel 60 F_{254} applied to aluminum plates; and the products were purified by column chromatography. The IR spectra were recorded on a Wattson FT-IR spectrometer. The ^1H and ^{13}C NMR spectra were measured on Jeol FX-90Q and Bruker AC-200 instruments relative to tetramethylsilane. The GC–MS and elemental analyses were performed at the Center of Science and Technology Research of Turkey (TUBITAK).

Oxidation of substituted 1,5-hexadien-3-ols (*method A; oxidation of compounds **IVa–**IVd** with pyridinium chlorochromate*).** Pyridinium chlorochromate, 1.5 mmol, was dissolved in 2 ml of CH_2Cl_2, 0.03 mmol of sodium acetate was added to the solution, and a solution of 1 mmol of substituted 1,5-hexadien-3-ol in 1.5 ml of CH_2Cl_2 was added through a dropping funnel. After 1–2 h, the reaction completion was checked by TLC. The black mixture was diluted with a fivefold volume of ether. The precipitate was filtered off and washed with two portions of ether. The ether solution was dried over $MgSO_4$ and evaporated under reduced pressure, and the crude product was subjected to column chromatography on silica gel.

*Method B. Oxidation of compounds **IVa**–**IVd** with zinc chlorochromate.* A solution of 8 mmol of compound **IVa**–**IVd** in 70 ml of CH_2Cl_2 was prepared in a 200-ml round-bottom flask equipped with a magnetic stirrer. Zinc chlorochromate, 16 mmol, was added to the solution in four portions over a period of 15 min under vigorous stirring. The mixture was stirred for 2 h, diluted with 120 ml of CH_2Cl_2, and filtered, and the filtrate was carefully evaporated on a rotary evaporator in a vacuum.

*Method C. Oxidation of compounds **IVc** and **IVd** with tert-butyl hydroperoxide in the presence of OsO_4.* A 50-mmol portion of compound **IVc** or **IVd** was dissolved in 125 ml of *t*-BuOH, and 6.5 ml of a 20% aqueous solution of NEt_4OH, 4 ml (103 mmol) of *t*-BuOOH (a 3 M solution of isooctane), and 2.6 ml (0.2 mmol) of a 2.5% solution of OsO_4 in *t*-BuOH were added. The mixture was kept for 12 h at room temperature, 65 ml of a 5% solution of Na_2SO_3 was added, and the mixture was extracted with ether. The extracts were washed with a saturated solution of NaCl, dried over $MgSO_4$, and evaporated.

*Method D. Oxidation of compounds **IVc** and **IVd** with tert-butyl hydroperoxide.* The procedure was the same as in method *C* with the difference that OsO_4 was not added.

(2*E*)-1-Allyloxy-2-butene (IIc). Yield 69%. bp 48–49°C (50 mm). IR spectrum (NaCl), ν, cm^{-1}: 3080, 1650, 1130, 960, 936. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 1.57–1.71 d (3H, J = 7 Hz), 3.88 m (4H), 5.02–5.22 m (2H), 5.22–6.15 m (3H). ^{13}C NMR spectrum, δ_C, ppm: 17.6, 71.0, 116.3, 128.4, 128.7, 135.5. Found, %: C 74.6; H 10.4. $C_7H_{12}O$. Calculated, %: C 75.0; H 10.7.

3-(Allyloxy)-1-phenylpropene (IId). Yield 71%. bp 118–120°C (10 mm). IR spectrum (NaCl), ν, cm^{-1}: 3080, 3040, 1657, 1140, 965, 750–700. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 3.95 m (4H), 4.84–5.35 m (2H), 5.54–6.66 m (3H), 6.94–7.53 m (5H). ^{13}C NMR spectrum, δ_C, ppm: 70.6, 116.2, 124.9, 127.0, 128.5, 130.9, 131.5, 134.3. Found, %: C 83.1; H 8.3. $C_{12}H_{14}O$. Calculated, %: C 82.8; H 8.0.

6-Chloro-4,5-epoxy-2-methyl-1-hexene (IIIa). Yield 76%. bp 70–72°C (20 mm). IR spectrum (NaCl), ν, cm^{-1}: 3080, 1640, 1250, 720. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 1.75 s (3H), 2.2–2.3 m (2H), 2.97–3.00 m (2H), 3.36–3.57 m (2H), 4.82 m (2H). Found, %: C 57.1; H 7.3; Cl 23.8. $C_7H_{11}ClO$. Calculated, %: C 57.3; H 7.5; Cl 24.2.

6-Chloro-2-methyl-4,5-epoxy-1-heptene (IIIb). Yield 79%. bp 92–93°C (4 mm). IR spectrum (NaCl), ν, cm^{-1}: 3081, 1641, 1250, 732. ^1H NMR spectrum ($CDCl_3$, 200 MHz), δ, ppm: 1.55 (3H, *J* = 7 Hz), 1.8 s (3H), 2.22 d.d (1H, *J* = 15, 6 Hz), 2.33 d.d (1H, *J* = 15, 6 Hz), 2.93 d.d (1H, *J* = 7, 2 Hz), 3.85 quint (1H, *J* = 7 Hz), 3.93 t.d (1H, *J* = 6, 2 Hz), 4.82 s (1H), 4.85 s (1H). Found, %: C 59.7; H 7.8; Cl 22.3. $C_8H_{13}ClO$. Calculated, %: C 59.8; H 8.1; Cl 22.1.

5-Methyl-1,5-hexadien-3-ol (IVa). IR spectrum (NaCl), ν, cm^{-1}: 3380, 3050, 1645, 935. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 1.61 s (3H), 2.2 d (4H), 3.04 s (1H), 3.94–4.40 m (1H), 4.57–5.45 m (2H), 5.56–6.12 m (1H). ^{13}C NMR spectrum, δ_C, ppm: 22.7, 46.2, 70.8, 113.7, 114.5, 141.1. Found, %: C 74.6; H 10.9. $C_7H_{12}O$. Calculated, %: C 75.0; H 10.7.

5-Methyl-1,5-heptadien-4-ol (IVb). Yield 74%. bp 55–56°C (10 mm). IR spectrum (NaCl), ν, cm^{-1}: 3370, 3060, 1640, 935. ^1H NMR spectrum ($CDCl_3$, 200 MHz), δ, ppm: 1.67 d (3H, *J* = 7 Hz), 1.78 s (3H), 2.22 d (2H, *J* = 6 Hz), 4.2 t.d (1H, *J* = 7, 5 Hz), 4.78 s (1H), 4.86 s (1H), 5.35–5.76 m (2H). ^{13}C NMR spectrum, δ_C, ppm: 18.1, 22.9, 46.7, 114.0, 127.4, 134.0, 142.8. Found, %: C 75.9; H 11.6. $C_8H_{14}O$. Calculated, %: C 75.0; H 10.7.

(5*E*)-1,5-Heptadien-4-ol (IVc). Yield 62%. bp 72–74°C (35 mm). IR spectrum (NaCl), ν, cm^{-1}: 3600–3200, 3040, 1651, 1260, 1065, 935. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 0.94–1.02 d (3H, *J* = 4 Hz), 2.23 m (2H), 3.94 q (1H, *J* = 6 Hz), 5.14 m (2H), 5.82 m (3H). ^{13}C NMR spectrum, δ_C, ppm: 16.0, 38.6, 76.7, 116.2, 139.3, 140.8. Found, %: C 75.4; H 10.9. $C_7H_{12}O$. Calculated, %: C 75.0; H 10.7.

(1*E*)-1-Phenyl-1,5-hexadien-3-ol (IVd). Yield 72%. bp 130–132°C (2 mm). IR spectrum (NaCl), ν, cm^{-1}: 3600–3200, 3040, 1651, 1260, 740. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 3.78–4.25 m (3H), 4.95–5.46 m (2H), 5.60–6.72 m (3H), 7.1–7.8 m (5H). ^{13}C NMR spectrum, δ_C, ppm: 71.8, 117.0, 127.3, 129.4, 133.0, 135.8, 137.7. Found, %: C 82.1; H 7.9. $C_{12}H_{14}O$. Calculated, %: C 82.8; H 8.1.

5-Methyl-1,5-hexadien-3-one (Va). IR spectrum (NaCl), ν, cm^{-1}: 3080, 1680, 1625, 935. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 2.2 s (3H), 4.47 s (2H), 4.75 s (2H), 6.27–6.31 m (3H). GC–MS data: M^+, *m*/*z* 110; base peak, *m*/*z* 55; retention time 9.378 min. Yield 67.5%. Found, %: C 77.1; H 9.5. $C_8H_{12}O$. Calculated, %: C 77.4; H 9.7.

(5*E*)-2-Methyl-1,5-heptadien-4-one (Vb). IR spectrum (NaCl), ν, cm^{-1}: 3040, 1680, 1625, 990, 935. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 2.2 s (3H), 4.47 s (2H), 4.75 s (2H), 6.0–6.8 m (2H). GC–MS data: M^+, *m*/*z* 124; base peak, *m*/*z* 109; retention time 9.492 min. Yield 58.3%. Found, %: C 77.1; H 9.5. $C_8H_{12}O$. Calculated, %: C 77.4; H 9.7.

(5*E*)-1,5-Heptadien-4-one (Vc). IR spectrum (NaCl), ν, cm^{-1}: 3040, 1680, 1625, 990. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 2.1 m (3H), 4.37 m (2H), 5.0–5.7 m (3H), 6.02–6.76 m (2H). ^{13}C NMR spectrum, δ_C, ppm: 17, 37, 124, 128, 130, 137, 197. Found, %: C 76.8; H 8.9. $C_7H_{10}O$. Calculated, %: C 76.4; H 9.1.

(1*E*)-1-Phenyl-1,5-hexadien-3-one (Vd). IR spectrum (NaCl), ν, cm^{-1}: 3040, 1680, 1625, 1080, 990, 760. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 4.35 m (2H), 5.0–5.7 m (3H), 6.25–7.91 m (2H), 7.2–7.6 m (5H). ^{13}C NMR spectrum, δ_C, ppm: 37, 123, 127, 128, 129, 131, 137, 197. GC–MS data: M^+, *m*/*z* 172; base peak, *m*/*z* 171; retention time 18.376 min. Yield 48.2% (*A*), 36.1% (*B*). Found, %: C 83.2; H 7.3. $C_{12}H_{12}O$. Calculated, %: C 83.7; H 7.0.

2-Methyl-1,5-hexadiene-3,4-dione (VIa). GC–MS data: M^+, *m*/*z* 124; base peak, *m*/*z* 109; retention time 9.492 min. Yield 3%. Found, %: C 67.3; H 6.8. $C_7H_8O_2$. Calculated, %: C 67.7; H 6.5.

Benzaldehyde (VII). GC–MS data: M^+, *m*/*z* 106; base peak, *m*/*z* 77; retention time 7.457 min. Yield 27.5% (*A*), 37.2% (*B*), 21.2% (*C*), 6.3% (*D*).

3-Phenylpropenal (VIII). GC–MS data: M^+, *m*/*z* 132; base peak, *m*/*z* 131; retention time 13.576. Yield 5.53%.

1-(3-Methyloxiranyl)-3-buten-1-ol (IXc). IR spectrum (NaCl), ν, cm^{-1}: 3450–3340, 3060, 1642, 1250, 965. ^1H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 1.32 d (3H, *J* = 7 Hz), 3.41 m (2H), 4.2 m (1H), 4.3–4.9 m (2H), 5.0–5.7 m (3H). GC–MS data: M^+, *m*/*z* 128; base peak, *m*/*z* 55; retention time 10.821 min. Yield 48.8%. Found, %: C 65.7; H 9.6. $C_7H_{12}O_2$. Calculated, %: C 65.6; H 9.4.

1-(3-Phenyloxiranyl)-3-buten-1-ol (IXd). IR spectrum (NaCl), ν, cm^{-1}: 3550–3480, 1641, 1245,

970, 740. ^{1}H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 3.41 m (3H), 4.23 m (1H), 4.4–5.0 m (2H), 5.10–5.67 m (3H), 7.2–7.3 m (5H). GC–MS data: M^+, *m*/*z* 190; base peak, *m*/*z* 91; retention time 16.524 min. Yield 32.2%. Found, %: C 75.1; H 6.7. $C_{12}H_{14}O_2$. Calculated, %: C 75.8; H 6.3.

1-(3-Methyloxiranyl)-3-buten-1-one (Xc). IR spectrum (NaCl), ν, cm^{-1}: 3040, 1715, 1640, 1245, 1080, 970, 760. ^{1}H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 3.95 m (2H), 4.27–5.42 m (2H), 5.10–5.67 m (3H), 7.2–7.3 m (5H). GC–MS data: M^+, *m*/*z* 126; base peak, *m*/*z* 59. Retention time 10.702 min. Yield 10.8%. Found, %: C 66.9; H 7.9. $C_7H_{10}O_2$. Calculated, %: C 66.7; H 7.4.

1-(3-Phenyloxiranyl)-3-buten-1-one (Xd). IR spectrum (NaCl), ν, cm^{-1}: 3040, 1715, 1640, 1245, 1080, 970, 765. ^{1}H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 3.95 m (2H), 4.2–5.3 m (2H), 5.1–5.7 m (3H), 7.2–7.3 m (5H). GC–MS data: M^+, *m*/*z* 188; base peak, *m*/*z* 118; retention time 16.368 min. Yield 16.4%. Found, %: C 76.2; H 6.9. $C_{12}H_{12}O_2$. Calculated, %: C 76.6; H 6.4.

1-(3-Methyloxiranyl)-2-oxiranylethanone (XIc). IR spectrum (NaCl), ν, cm^{-1}: 1720, 1250, 1100. ^{1}H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 1.34 s (3H), 3.42 m (2H), 3.81–4.69 m (3H), 4.57–5.41 m (2H). GC–MS data: M^+, *m*/*z* 142; base peak, *m*/*z* 71; retention time 10.051 min. Yield 33.2%. Found, %: C 59.9; H 7.4. $C_7H_{10}O_3$. Calculated, %: C 59.2; H 7.0.

2-Oxiranyl-1-(3-phenyloxiranyl)ethanone (XId). IR spectrum (NaCl), ν, cm^{-1}: 3040, 1715, 1250, 1100. ^{1}H NMR spectrum ($CDCl_3$, 90 MHz), δ, ppm: 3.42 m (2H), 3.81–4.69 m (3H), 4.63–5.52 m (2H), 7.2–7.3 m (5H). GC–MS data: M^+, *m*/*z* 204; base peak, *m*/*z* 130; retention time 17.257 min. Yield 13.4%. Found, %: C 70.2; H 5.6. $C_{12}H_{12}O_3$. Calculated, %: C 70.6; H 5.9.

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