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Synthesis of Biologically Active Substances Based on Phenoxyethanol Derivatives

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Abstract—By reaction of phenoxyethanol with diverse structure acyl chlorides phenoxyethanol esters were synthesized containing in the molecule fragments of undecene acid and also of some ketocarboxylic acids. The latter acids were obtained by ozonation of alkylcyclenes.

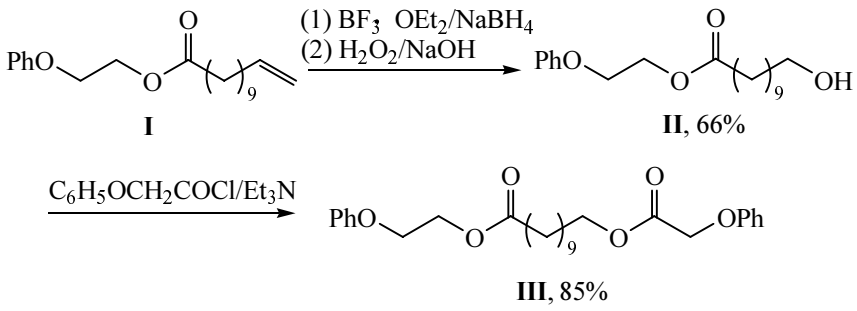
Quite a number of compounds possessing versatile biological activity were found among phenoxyethanol derivatives. The presence in a molecule of two phenoxyethanol fragments separated by a hydrocarbon chain significantly enhances its lipophilicity and facilitates the transport of the active substance to the receptors. It was established that derivatives of undecenoic and decenoic acids most efficiently affected insects metamorphosis [1], and 2,4-decadienoic acid derivatives were the active principle of the pharmacologically active extracts from the *Achillea pharmica* [2] and *Piper nigrum* [3] plants. Some publications describe related structures exhibiting an antitumor activity [4].

The synthesis of these compounds may be based on an ester of the undecenoic acid and phenoxyethanol, 2-phenoxyethyl 11-undecenoate (**I**). The terminal double bond of ester **I** was functionelized via an organoboron compound, then the treatment of alcohol **II** obtained with phenoxyacetyl chloride yielded diester **III**. The transformations were followed by an appearance in the IR spectrum of compound **II** of an absorption band of the alcohol group (3450–3550 cm^{-1}) alongside with disappearance of the absorption bands of the terminal methylene group (905, 1640, 3080 cm^{-1}). The characteristic feature of diester **III** was the singlet at 4.7 ppm in the ^1H NMR spectrum assigned to the methylene protons of the phenoxyacetic acid fragments (Scheme 1).

Ozonation of the terminal double bond in ester **I** afforded aldehyde **IV** that was oxidized with the freshly precipitated silver oxide to obtain acid **V** in a moderate yield. The latter was converted into acyl chloride, and treated with the phenoxyethanol to get diester **VI** (Scheme 2).

The carbonyl group of aldehyde **IV** gives rise in the IR spectrum to a relatively strong absorption band at 1725 cm^{-1}, and in the ^1H to a triplet in the region 9.6 ppm (J 2.1 Hz). The structure of diester **VI** is confirmed by the data of IR and ^1H NMR spectra (absorption bands at 1735, 1515, and 1605 cm^{-1} correspond to vibrations of the ester group and the aromatic ring, the resonances of

Scheme 1.



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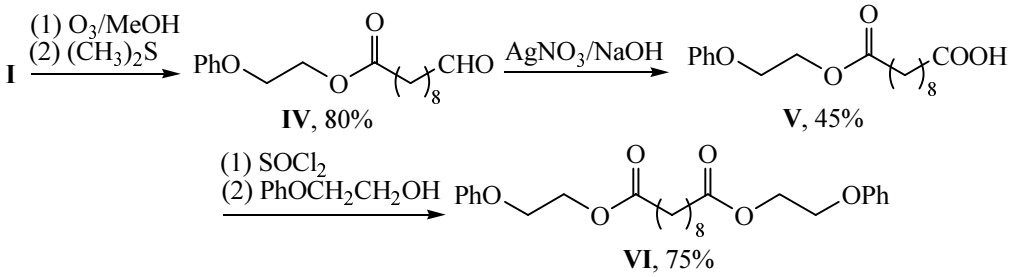
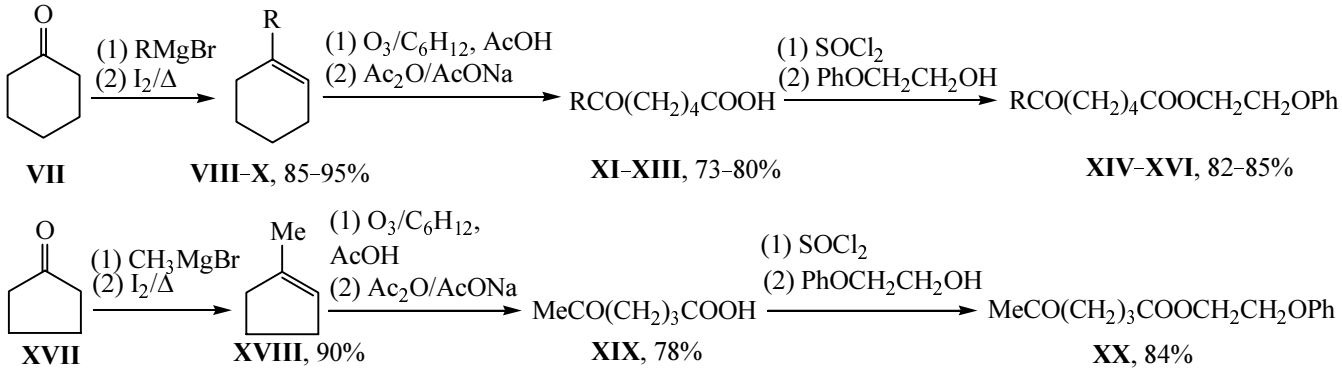
Scheme 2.



Scheme 3.



R = C_6H_{13} (**VIII, XI, XIV**), Pr (**IX, XII, XV**), Me (**X, XIII, XVI**).

the methylene protons of the phenoxyethanol fragment appear at 4.2, 4.4 ppm, of the aromatic ring at 7.3 ppm, of the methylene groups in the α-position with respect to the carbonyl group at 2.3 ppm, and also the signals from the central saturated fragment of the molecule are observed). The ^{13}C NMR spectrum of ester **VI** contains 14 signals from the nonaromatic carbons in accordance with the assumed symmetric structure.

Inasmuch as the replacement of an ester group by a keto function was known to favorably affect the pharmacological activity [4] we carried out the reaction with the phenoxyethanol with 6-oxoheptanoic (**XI**), 6-oxononanoic (**XII**), 6-oxododecanoic (**XIII**), and 5-oxohexanoic (**XIX**) acids prepared in turn by the ozonation of alkylcyclenes **VIII–X**, and **XVIII**. Cycloolefins **VIII** and **IX**, and **XVIII** were obtained by reaction of cyclohexanone or cyclopentanone with alkylmagnesium halides followed by dehydration by heating with I_2 (Scheme 3).

EXPERIMENTAL

IR spectra were measured on spectrophotometers UR-20 and Specord M-80 from thin films or mulls in mineral oil. The ^1H and ^{13}C NMR spectra were registered on spectrometer Bruker AM-300 from solutions of compounds in $CDCl_3$ and $(CD_3)_2CO$ at operating frequencies 300 and 75.46 MHz respectively, internal reference TMS. Chemical shifts are presented in the δ scale. The GLC analysis was carried out on a Chrom-5 chromatograph, column length 1.2 m, stationery phase SE-30 on Chromaton "N-FW-DMCS" (0.16–0.20 mm), carrier gas helium, oven temperature programmed within 50–300°C at a rate 12 deg/min). TLC analysis was performed on Silufol UV 366 plates.

2-Phenoxyethyl 11-hydroxyundecanoate (II). To a solution of 2.5 g (8.2 mmol) of 2-phenoxyethyl 11-undecenoate (**I**) [5] and 1.2 g (7.8 mmol) of boron trifluoride etherate in 40 ml of anhydrous tetrahydrofuran at 10°C was added at stirring avoiding the heating of the mixture 0.32 g (8.0 mmol) of sodium borohydride, and the stirring was continued to the end of gas liberation. Then 40% NaOH solution was added till alkaline reaction of the solution, and 2 ml of 30% hydrogen peroxide was added dropwise. The stirring at 30°C was carried on for 2 h, and the reaction mixture was left standing at room temperature for 12 h. The reaction mixture was washed with a KI solution and then in succession with 5% solution of sodium thiosulfate, 5% solution of HCl, and saturated NaCl solution. On drying the solvent was distilled off. Alcohol **II** was obtained as a residue in an amount of 1.75 g (66%). IR spectrum, cm^{-1}: 920 m, 1080 s, 1250 s,

1505 m, 1600 m, 1740 s, 3400–3600 br.s. The alcohol obtained was used in the synthesis of diester **III** without further purification.

2-Phenoxyethyl 11-(phenoxymethylcarbonyloxy)-undecanoate (III). To a solution of 1.76 g (5.46 mmol) of alcohol **II** and 0.55 g (5.46 mmol) of triethylamine in 50 ml of anhydrous tetrahydrofuran was added at stirring 0.93 g (5.46 mmol) of phenyloxyacetyl chloride in 30 ml of anhydrous tetrahydrofuran (10°C). After stirring for 30 min the reaction mixture was left standing for 12 h. The separated precipitate was filtered off and washed on the filter with tetrahydrofuran.The solution obtained was washed with a saturated NaCl solution, dried with $MgSO_4$, and the solvent was distilled off. The residue (2.6 g) was subjected to chromatography on SiO_2 (eluent hexane–ether, 3 : 2) to isolate 2.1 g (84%) of diester **III** as a light yellow oily substance, n_D^{20} 1.5318. IR spectrum, cm^{-1}: 1205 s, 1500 m, 1605 m, 1725 s, 1745 s. ^1H NMR spectrum, δ, ppm: 1.25–1.40 m (16H, CH_2), 2.35 t (2H, $CH_2C{=}O$, *J* 6.5 Hz), 4.12 t (2H, CH_2O, *J* 7.0 Hz), 4.28 t (2H, CH_2OCO, *J* 5.0 Hz), 4.42 t (2H, CH_2OAr, *J* 5.0 Hz), 4.70 s (2H, $COCH_2O$), 7.28–7.34 m (10H, H_{arom}). Found, %: C 69.89; H 8.04. $C_{27}H_{36}O_6$. Calculated, %: C 71.02; H 7.95.

2-Phenoxyethyl 10-formyldecanoate (IV). Through a solution of 5 g (16.4 mmol) of compound **I** in 70 ml of anhydrous methanol was passed a flow of a mixture O_3/O_2 at –78°C till the solution turned blue (16.8 mmol of O_3 was consumed). The reaction mixture was flushed with argon, and at –78°C was added while stirring 1.69 ml (16.4 mmol) of dimethyl sulfide. The mixture was stirred for 0.5 h, then warmed to the room temperature and left standing for 12 h. The methanol was distilled off, the residue was dissolved in 60 ml of chloroform, the solvent obtained was washed with a saturated NaCl solution, dried with $MgSO_4$, and evaporated to obtain 5.1 g of a substance that was subjected to chromatography a SiO_2 (eluent hexane–ether, 3 : 2) to isolate 3.93 g (80%) of aldehydoester **IV**. IR spectrum, cm^{-1}: 1090 s, 1240 s, 1320 m, 1506 m, 1600 w, 1725 s, 1745 s. ^1H NMR spectrum, δ, ppm: 1.20–1.60 m (12H, CH_2), 2.30 m (4H, $CH_2C{=}O$), 4.12 t (2H, CH_2OAr, *J* 5.0 Hz), 4.42 t (2H, CH_2OCO, *J* 5.0 Hz), 6.95 m and 7.27 m (5H, H_{arom}), 9.6 t (1H, CHO, *J* 2.10 Hz). Found, %: C 70.32; H 8.59. $C_{18}H_{26}O_4$. Calculated, %: C 70.56; H 8.50.

Di[(2-phenoxy)ethyl] decane-1,10-dioate (VI). To a stirred solution of 2 g of $AgNO_3$ in 8.8 ml H_2O was added 1.44 g (4.33 mmol) of aldehydoester **IV**. The mixture obtained was thoroughly stirred, and thereto was added dropwise at 20°C within 20 min 24.4 ml of 1 N water solution of NaOH. The stirring was continued for 12 h. The precipitate was filtered off, washed on the filter with ethyl ether, the solution was dried with $MgSO_4$, and evaporated. We obtained 1.86 g (45%) of acid **V**. IR spectrum, cm^{-1}: 2400–3600 br.s.

Acid **V** without purification was dissolved in 12 ml of anhydrous methylene chloride, to the solution obtained was added at stirring (0°C) 3.7 g (46.8 mmol) of anhydrous pyridine. In 10 min 5.57 g (46.8 mmol) of pure thionyl chloride was added dropwise. The precipitate was filtered off, and to the solution obtained was added dropwise a solution of 1.1 g (8.21 mmol) of phenoxyethanol [5] in 10 ml of anhydrous methylene chloride. After stirring for 30 min the solution was washed with a saturated $NaHCO_3$ solution, dried with $MgSO_4$, and evaporated, The residue (2.05 g) was subjected to column chromatography on SiO_2, eluent hexane–ether, 9 : 1 . We obtained 1.91 g (75%) of compound **VI**. IR spectrum, cm^{-1}: 1080 s, 1240 s, 1515 m, 1605 m, 1735 s. ^1H NMR spectrum, δ, ppm: 1.20–1.45 m (12H, CH_2), 2.31 m (4H, $CH_2C{=}O$), 4.24 t (4H, CH_2OCO, *J* 5.0 Hz), 4.39 t (4H, CH_2OAr, *J* 5.0 Hz), 7.28 m (10H, H_{arom}). ^{13}C NMR spectrum, δ, ppm: 28.87 t, 28.98 t, 29.33 t, 29.44 t, 29.48 t, 29.56 t, 29.74 t, 29.78 t (CH_2), 61.59 t ($\underline{C}H_2OAr$), 69.15 t ($\underline{C}H_2OCO$), 114.64 d ($\underline{C}^4_{arom}$), 121.23 d ($\underline{C}^{2,6}_{arom}$), 129.56 d ($\underline{C}^{3,5}_{arom}$), 158.64 s ($\underline{C}^1_{arom}$), 178.57 s and 178.61 s (C=O). Found, %: C 70.48; H 7.81. $C_{26}H_{34}O_6$. Calculated, %: C 70.56; H 7.76.

1-Hexylcyclohexene (VIII). To the Grignard reagent prepared from 2.4 g (0.1 mol) of Mg and 16.5 g (0.1 mol) of hexyl bromide in 50 ml of anhydrous ethyl ether was added dropwise at 0°C while stirring 8.33 g (0.085 mol) of cyclohexanone, the mixture was heated at reflux for 1 h, and left standing at 20°C for 12 h. The reaction mixture was cooled to 0°C, and 20% water solution of AcOH was added thereto at stirring till the complete dissolution of the precipitate. The ether layer was separated, the reaction products from the water layer were extracted into ether. The combined ether solution was dried with $MgSO_4$, filtered, and evaporated. To the residue 40 ml of toluene and 5.4 g (0.43 mol) of I_2 was added. The mixture obtained was heated at reflux for 3 h, cooled, and washed in succession with a 10% $Na_2S_2O_3$ solution, a saturated solution of $NaHCO_3$, and a saturated solution of NaCl, dried with $MgSO_4$, filtered, and evaporated. The residue was distilled to obtain 12.3 g (87%) of compound **VIII**, bp 103–108°C (15 mm Hg), n_D^{20} 1.4263. ^1H NMR spectrum, δ, ppm: 0.87 t (3H, Me, *J* 6.5 Hz),

1.2–1.4 m (12H, CH_2), 1.52–1.64 m (6H, CH_2C=C), 5.18 t (1H, CH=C, *J* 6.0 Hz). Found, %: C 86.54; H 13.42. $C_{12}H_{22}$. Calculated, %: C 86.76; H 13.26.

1-Propylcyclohexene (IX) was prepared by treating with 8.33 g (0.085 mol) of cyclohexanone the Grignard reagent obtained from 12.3 g (0.10 mol) of propyl bromide and 2.4 g (0.10 mol) of Mg in 50 ml of anhydrous ethyl ether followed by reaction of the alcohol formed with iodine (5.4 g, 0.43 mol). Compound **IX** was isolated similarly to compound **VIII**. Yield 9.4 g (89%), bp 116–119°C (15 mm Hg.), n_D^{20} 1.4298. Found, %: C 87.14; H 12.76. C_9H_{16}. Calculated, %: C 87.02; H 12.90.

1-Methylcyclohexene (X) was prepared by treating with 8.33 g (0.085 mol) of cyclohexanone the Grignard reagent obtained from 14.2 g (0.10 mol) of methyl iodide and 2.4 g (0.10 mol) of Mg in 50 ml of anhydrous ethyl ether followed by reaction of the alcohol formed with iodine (5.4 g, 0.43 mol). Cycloolefin **X** was isolated similarly to compound **VIII**. Yield 7.75 g (95%), bp 106–108°C (15 mm Hg), n_D^{20} 1.4304.

6-Oxododecanoic acid (XI). In a mixture of 40 ml of cyclohexane preliminary passed through a layer of SiO_2 and 2.3 ml of AcOH was dissolved 5 g (0.03 mol) of cycloolefin **VIII**, and through this solution cooled to 0–5°C was passed a flow of ozone–oxygen mixture till 30 mmol of O_3 was consumed. The cyclohexane was decanted from the precipitated ozonide, 5.4 ml of Ac_2O was added thereto and, maintaining the temperature of the mixture below 15°C, 2.3 g of molten sodium acetate was added at stirring by portions as a dispersion in 11.8 ml of AcOH. The reaction mixture was boiled for 30 min at reflux on a water bath, and then left standing for 12 h. Then the acetic acid was distilled off maintaining the bath temperature no higher than 40°C. To the residue 50 ml of benzene–ethyl ether mixture, 1:1, was added. The separated precipitate was filtered off, washed on the filter with 20 ml of benzene–ethyl ether mixture, 1:1, the filtrate was washed with a cold saturated NaCl solution, dried with $MgSO_4$, filtered, and evaporated. We obtained 4.7 g (73%) of ketoacid **XI** as a light yellow oily substance. IR spectrum, cm^{-1}: 1695 s, 1715 s, 2400–3600 br.s.

Methyl 6-oxododecanoate was obtained by treating acid **XI** with diazomethane ether solution. IR spectrum, cm^{-1}: 1715 C, 1735 C. ^1H NMR spectrum, δ, ppm: 0.83 t (3H, Me, *J* 6.5 Hz), 1.15–1.34 m (12H, CH_2), 2.30–2.35 m (6H, CH_2C=O), 3.62 s (3H, OMe). Found, %: C 68.38; H 10.60. $C_{13}H_{24}O_3$. Calculated, %: C 68.49; H 10.51.

6-Oxononanoic acid (XII) was prepared by ozonation of 3.72 g (0.03 mmol) of cycloolefin **IX** in a mixture of 40 ml of cyclohexane and 2.3 ml of AcOH followed by ozonide decomposition by treating in succession with 5.4 ml of Ac_2O and 2.3 g AcONa in 11.8 ml of AcOH. Acid **XII** was isolated in the same way as compound **XI**. Yellow oily substance, yield 4.02 g (78%). IR spectrum, cm^{-1}: 1700 s, 1715 s, 2400–3600 br.s.

Methyl 6-oxononanoate was obtained by treating acid **XII** with diazomethane ether solution. IR spectrum, cm^{-1}: 1715 C, 1735 C. ^1H NMR spectrum, δ, ppm: 0.85 t (3H, Me, *J* 6.5 Hz), 1.20–1.38 m (6H, CH_2), 2.30–2.35 m (6H, CH_2C=O), 3.68 s (3H, OMe). Found, %: C 64.62; H 9.58. $C_{10}H_{18}O_3$. Calculated, %: C 64.49; H 9.74.

6-Oxoheptanoic acid (XIII) was prepared by ozonation of 2.88 g (0.03 mmol) of cycloolefin **X** in a mixture of 40 ml of cyclohexane and 2.3 ml of AcOH followed by ozonide decomposition by treating in succession with 5.4 ml of Ac_2O and 2.3 g AcONa in 11.8 ml of AcOH. Acid **XIII** was isolated in the same way as compound **XI**. Light yellow oily substance, yield 3.46 g (80%). IR spectrum, cm^{-1}: 1695 s, 1715 s, 2400–3600 br.s.

Methyl 6-oxoheptanoate was obtained by treating acid **XIII** with diazomethane ether solution. IR spectrum, cm^{-1}: 1715 C, 1740 C. ^1H NMR spectrum, δ, ppm: 1.25–1.41 m (4H, CH_2), 2.10 s (3H, MeC=O), 2.36 m (4H, CH_2C=O, *J* 7.0 Hz), 3.68 s (3H, OMe). Found, %: C 60.81; H 8.76. $C_8H_{14}O_3$. Calculated, %: C 60.74; H 8.92.

2-Phenoxyethyl 6-oxododecanoate (XIV). To a solution of 5.14 g (0.024 mol) of acid **XI** in 25 ml of anhydrous DMF 2.83 g of $SOCl_2$ was added dropwise. The solution obtained was stirred for 1 h at 30°C, cooled to 20°C, and 60 ml of anhydrous pentane was added. The stirring was continued for 1 h, then the acyl chloride solution in pentane was separated, and 3.3 g (0.024 mol) of phenoxyethanol was added thereto at stirring [5]. The stirring was carried on for 1 h at 20°C, and then the reaction mixture was washed successively with a saturated NaCl solution, 1 N solution of NaOH, with water, then dried with $MgSO_4$. The solvent was distilled off to give as a residue 6.58 g (82%) of ester **XIV** as yellow oily substance that was subjected to chromatography on SiO_2 (eluent hexane–ether, 3:2), n_D^{20} 1.5446. IR spectrum, cm^{-1}: 1080 s, 1205 s, 1505 m, 1600 w, 1715 s, 1745 s. ^1H NMR spectrum, δ, ppm: 0.86 t (3H, Me, *J* 6.5 Hz), 1.20–1.40 m (12H, CH_2), 2.34 m (6H, CH_2C=O), 4.28 t (2H, CH_2OAr, *J* 5.5 Hz), 4.42 t (2H, CH_2O, *J* 5.5 Hz),

7.28 br.s (5H, H_{arom}). Found, %: C 71.63; H 9.18. $C_{20}H_{30}O_4$. Calculated, %: C 71.86; H 9.04.

2-Phenoxyethyl 6-oxononanoate (XV). The corresponding acyl chloride was obtained from 4.13 g (0.024 mol) of acid **XII** by treatment with 2.83 g of $SOCl_2$ as described for compound **XI**. Then to the acyl chloride solution in pentane was added at stirring 3.3 g (0.024 mol) of phenoxyethanol. Similarly to compound **XI** 5.89 g (84%) of ester **XV** was isolated as yellow oily substance and purified by chromatography on SiO_2 (eluent hexane–ether, 3:2), n_D^{20} 1.5398. IR spectrum, cm^{-1}: 1100 s, 1205 s, 1500 m, 1605 w, 1715 s, 1740 s. ^1H NMR spectrum, δ, ppm: 0.89 t (3H, Me, *J* 6.5 Hz), 1.20–1.40 m (6H, CH_2), 2.38 m (6H, CH_2C=O), 4.26 t (2H, CH_2OAr, *J* 5.5 Hz), 4.44 t (2H, CH_2O, *J* 5.5 Hz), 7.26 br.s (5H, H_{arom}). Found, %: C 69.96; H 8.14. $C_{17}H_{24}O_4$. Calculated, %: C 69.86; H 8.21.

2-Phenoxyethyl 6-oxoheptanoate (XVI). The corresponding acyl chloride was obtained from 3.46 g (0.024 mol) of acid **XIII** by treatment with 2.83 g of $SOCl_2$ as described for compound **XI**. Then to the acyl chloride solution in pentane was added at stirring 3.3 g (0.024 mol) of phenoxyethanol. Similarly to compound **XIV** 5.37 g (80%)of ester **XVI** was isolated as yellow oily substance and purified by chromatography on SiO_2 (eluent hexane–ether, 3 : 2), n_D^{20} 1.5401. IR spectrum, cm^{-1}: 1095 s, 1215 s, 1505 m, 1605 w, 1715 s, 1745 s. ^1H NMR spectrum, δ, ppm: 1.24–1.46 m (4H, CH_2), 2.10 s (3H, MeCO), 2.43 m (4H, CH_2C=O), 4.24 t (2H, CH_2OAr, *J* 5.5 Hz), 4.42 t (2H, CH_2O, *J* 5.5 Hz), 7.28 br.s (5H, H_{arom}). Found, %: C 68.23; H 7.49. $C_{15}H_{20}O_4$. Calculated, %: C 68.18; H 7.58.

1-Methylcyclopentene (XVIII) was prepared by treating with 7.14 g (0.085 mol) of cyclopentanone the Grignard reagent obtained from 14.2 g (0.10 mol) of methyl iodide and 2.4 g (0.10 mol) of Mg in 50 ml of anhydrous ethyl ether followed by reacting the arising alcohol with the iodine (5.4 g, 0.43 mol). Cycloolefin **XVIII** was isolated as compound **VIII** above described. Yield 6.55 g (94%). The characteristics of compound **XVIII** were consistent with those published [6].

5-Oxohexanoic acid (XIX) was obtained by ozonation of 2.46 g (0.03 mmol) of cycloolefin **XVII** was prepared by ozonation of 2.88 g (0.03 mmol) of cycloolefin **X** in 40 ml of cyclohexane followed by ozonide decomposition by treating in succession with 5.4 ml of AC_2O and 2.3 g AcONa in 11.8 ml of ACOH. Acid **XIX** was isolated in the same way as compound **XI** as a light yellow oily substance in an yield 3.08 g (79%). IR spectrum, cm^{-1}: 1705 C, 1715 C, 2400–3600 br.s.

Methyl 5-oxohexanoate was obtained by treating acid **XIX** with diazomethane ether solution. IR spectrum, cm^{-1}: 715 s, 1735 s. ^1H NMR spectrum, δ, ppm: 1.34 m (2H, CH_2), 2.08 s (3H, MeC=O), 2.34 m (4H, CH_2C=O), 3.62 s (3H, OMe). Found, %: C 58.43; H 8.39. $C_7H_{12}O_3$. Calculated, %: C 58.32; H 8.39.

2-Phenoxyethyl 5-oxohexanoate (XX). The corresponding acyl chloride was obtained from 4.13 g (0.024 mol) of acid **XIX** by treatment with 2.83 g of $SOCl_2$. The acyl chloride obtained was reacted with 3.3 g (0.024 mol) of phenoxyethanol to isolate 5.04 g (84%) of ester **XX** as a yellow oily substance that was purified by chromatography on SiO_2 (eluent hexane–ether, 3 : 2), n_D^{20} 1.5418. IR spectrum, cm^{-1}: 1090 s, 1200 s, 1500 m, 1605 w, 1715 s, 1745 s. ^1H NMR spectrum, δ, ppm: 1.26–1.40 m (2H, CH_2), 2.10 s (3H, MeCO), 2.34 m (4H, CH_2C=O), 4.24 t (2H, CH_2OAr, *J* 5.5 Hz), 4.68 t (2H, CH_2O, *J* 5.5 Hz), 7.28 br.s (5H, H_{arom}). Found, %: C 67.24; H 7.36. $C_{14}H_{18}O_4$. Calculated, %: C 67.18; H 7.25.

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