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Reaction of Methyl 1-Bromocycloalkanecarboxylates with Zinc and 2,6-Bis(arylmethylene)cyclohexanones

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Abstract—Methyl 1-bromocyclopentane-, 1-bromocyclohexane-, and 1-bromocycloheptanecarboxylates react with zinc and 2,6-bis(arylmethylene)cyclohexanones to afford 4-aryl-8-(arylmethylene)-5,6,7,8- tetrahydrospiro[chromene-3,1'-cyclopentan]-2(4*H*)-ones, 4-aryl-8-(arylmethylene)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclohexan]-2(4*H*)-ones, and 4-aryl-8-(arylmethylene)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cycloheptan]-2(4*H*)-ones, respectively.

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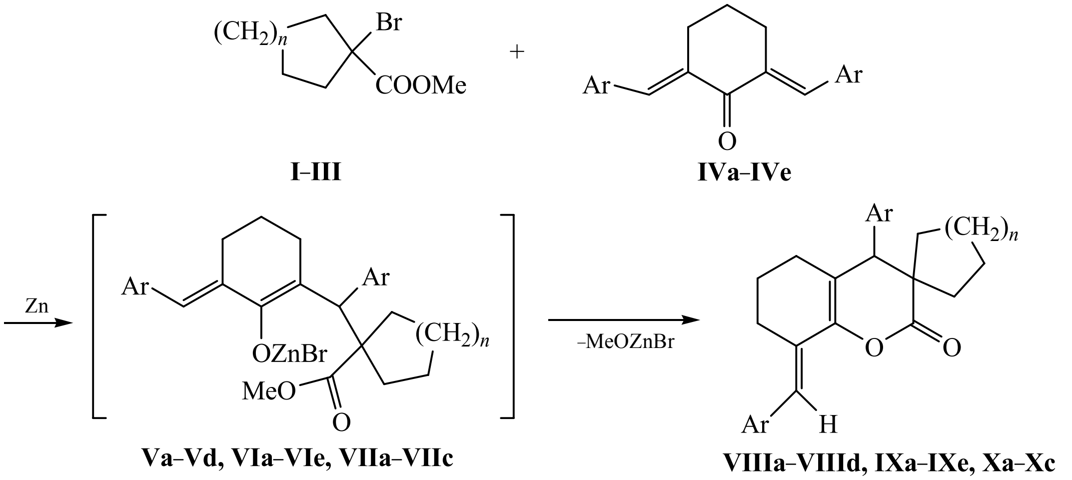
Alicyclic Reformatskii reagents react with α,β-unsaturated ketones to form substituted spirodihydropyranones [1, 2].

We found that the Reformatsky reagents derived from methyl 1-bromocycloalkanecarboxylic acids **I–III** with zinc added to 2,6-bis(arylmethylene)cyclohexanones **IVa–IVe** at 1–4 positions to form the intermediates **Va–Vd, VIa–VId,** and **VIIa–VIIc**. These latter in the reaction course undergo cyclization with the cleavage of methoxyzinc bromide to form 4-aryl-8-(arylmethylene)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclopentan]-2(4*H*)-ones **VIIIa–VIIId**, 4-aryl-8-(arylmethylene)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclohexan]-2(4*H*)-ones **IXa–IXe**, and 4-aryl-8-(arylmethylene)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cycloheptan]-2(4*H*)-ones **Xa–Xc**, respectively.

The composition and structure of compounds obtained were confirmed by the elemental analysis, IR and ^{1}H NMR spectroscopy data. The IR spectra of compounds **VIIIa–VIIId, IXa–IXe,** and **Xa–Xc**

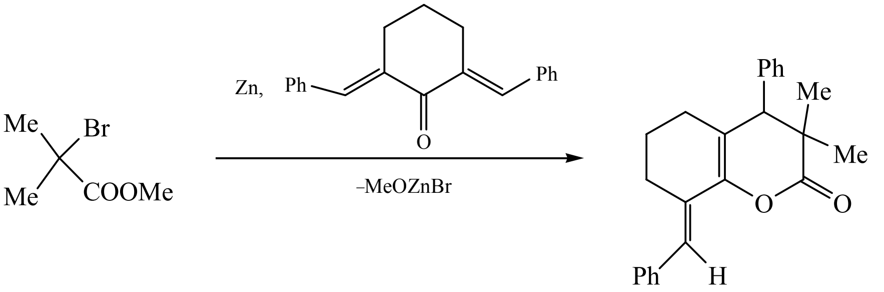


n = 1 (**I, V, VIII**), 2 (**II, VI, IX**), 3 (**III, VII, X**); **IV–X**: Ar = Ph (**a**), 2-FC$_6$H$_4$ (**b**), 3,4-(MeO)$_2$C$_6$H$_3$ (**c**); **IV–VI, VIII, IX**: Ar = 4-ClC$_6$H$_4$ (**d**); **IV, VI, IX**: Ar = 4-MeOC$_6$H$_4$ (**e**).

contain character-istic bands of C=C bonds at 1680–1700 cm^{-1} and of lactone carbonyl groups at 1740–1765 cm^{-1}. The ^{1}H NMR spectra contain one set of signals, among which the most characteristic is a singlet of methine proton in the region of 2.94–3.73 ppm.

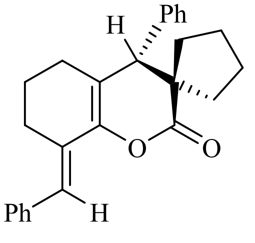
The reaction products have a chiral center, the carbon atom C^{4}, therefore theoretically they can exist as a mixture of *R*- and *S*-enantiomers. The stereoisomerism due to different positions of the substituents at the *exo*-ethylene bond C^{8}=C seems hardly probable, as in the initial 2,6-bis(arylmethylene)cyclohexanone the aryl substituent is *E*-oriented relative to the C$^{2(6)}$=C bond [3], and one of the ArCH=C fragments is not involved in the reaction.

In order to determine the configuration of the reaction products we in more detail investigated the compound **VIIIa** by the methods of ^{1}H and ^{13}C NMR spectroscopy, including 2D experiments ^{1}H–^{13}C HMQS, ^{1}H–^{1}H ROESY and ^{1}H–^{13}C HMBS. The assignment of signals in the ^{1}H and ^{13}C NMR spectra was carried out on the basis of homo- and heteronuclear 2D experiments. Detection of signals of carbon atoms is done after the DEPT experiment. For a more precise assignment of the signals of protons of methylene groups in the ^{1}H NMR spectra we synthesized a model compound 8-benzylidene-3,3-di-methyl-4-phenyl-3,4,5,6,7,8-hexahydro-2*H*-chromene-2-one (**XI**) by the reaction of methyl 2-bromo-2-methylpropanoate with zinc and 2,6-dibenzylidenecyclohexanone.

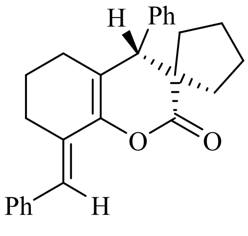


XI

In the gHMBC experiment, in the spectra of compounds **VIIIa** and **XI** a strong interaction was revealed between the proton of the benzylidene group and carbon atom C^{8} and a weaker interaction with the carbon atom C^{7}, suggesting the *E*-configuration of substituents at the *exo*-ethylene bond. However, we failed to determine the steric position of the substituents at the C^{4} atom. Actually, the same enantiomer can contain hydrogen atom and aryl substituent either in the axial, or in equatorial position. In order to identify more stable conformer of compound **VIIIa** we performed *ab initio* calculation of the total energy (E_{tot}, a.u.) of its 4 (*S*)-enantiomers with axial and equatorial phenyl substituent by the DFT B3LYP/6-31G* method.



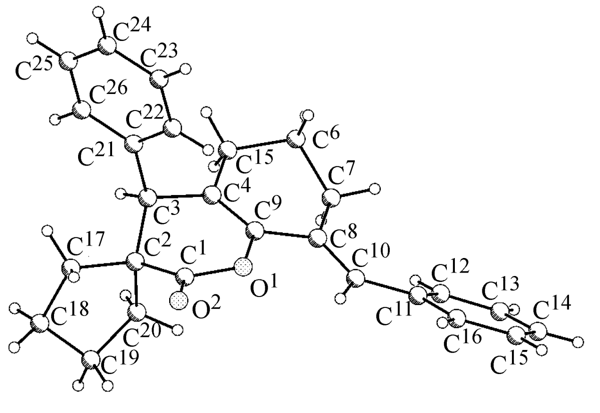
E_{tot} –1156.14466 a.u.



E_{tot} –1156.14269 a.u.

As follows from the calculation, the conformer with equatorial orientation of the hydrogen atom and the axial orientation of the phenyl substituent at the C^{4}–H is more stable than the alternative conformer. But since the calculations describe the structure of molecules in the gas phase, we undertook X-ray diffraction investigation of the crystal of compound **VIIIa**.

According to the results of XRD analysis, the colorless plate crystals of compound **VIIIa** belong to noncentrosymmetric space symmetry group of the monoclinic crystal system. The structure is characterized by pseudoaxially oriented phenyl substituent of the heterocyclic fragment and the *E*-orientation of the phenyl group at the double bond. Atomic positions of the C^{18} and C^{19} atoms in the cyclopentane fragment (see the figure) are disordered over two positions with occupancy 0.5. The bond lengths in the system of conjugated double bonds is leveled as usually in such cases: C^9=C^4 1.323(2) Å, C^9–C^8 1.459(3) Å, C^8=C^{10} 1.326(3) Å. The molecular packing consists of the layers oriented parallel to the crystallographic plane [001]. The packing is characterized by a number of short contacts, the most notable of which is the T-shaped contact between the proton of phenyl substituent and the π-system of C=O group ($H^{25A}\cdots O^2$ [x, $-1+y$, z] 2.616 Å, and $H^{25A}\cdots C^1$ [x, $-1+y$, z] 2.850 Å, as well as contact $H^{20A}\cdots O^2$ [x, $-y$, $-1/2+z$] 2.489 Å, by 0.104 Å, 0.050 Å and 0.231 Å, respectively, less than the sum of the van der Waals radii.



General view of the **VIIIa** molecule and the numbering of atoms adopted in the structural experiment.

EXPERIMENTAL

IR spectra of compounds **VIII–X** as mulls in mineral oil were obtained on a Specord-75IR spectrophotometer. The ^{1}H NMR spectra of solutions of these compounds in $CDCl_3$ were recorded on a Mercury Plus-300 spectrometer (300 MHz), internal reference TMS. The calculation of the molecule **VIIIa** conformers with full optimization of all geometric parameters was executed on a Linux-Cluster running under the control of CentOS 5.5 6×AMD Atlon X2 4800 computer with a Firefly package (PC GAMESS) version 7.1.G [4].

The X-ray study of the compound **VIIIa** was performed along the standard procedure on a Xcalibur 3 diffractometer equipped with a CCD detector [MoK_α, $\lambda = 0.71073$ Å, graphite monochromator, $\omega/2\theta$-scanning with 1° steps, the frame measurement time 20 s, T = 295(2) K]. For the analysis was used a fragment of the lamellar crystal with size 0.42×0.30×0.15 mm. No correction for absorption was introduced because of its smallness. The structure was solved by the direct method with a SHELXS97 program and refined by least-squares method with respect to F^2 with a SHELXL97 program in the anisotropic full-matrix approximation for nonhydrogen atoms [5]. The hydrogen atoms of the CH bonds were set to the geometrically calculated positions and then included in the refinement in isotropic approximation with dependent thermal parameters in the *rider* model. The main parameters of the structural experiment: crystal system monoclinic, space group Pc, a = 9.9930(14) Å, b = 8.9671(10) Å, c = 11.7734 (17) Å, β = 101.633(12)°, V = 1033.3(2) Å^3, the substance of empirical formula $C_{26}H_{26}O_2$ with Z = 2, D_c = 1.191 g cm^{-3}, μ = 0.074 mm^{-1}. In the angular range $3.08 < \theta < 26.37°$ 3340 reflections was collected, of which 2678 were independent (R_{int} = 0.0175), and 1714 with $I > 2\sigma(I)$. The experiment completeness at the angles $\Theta < 26.37°$ was 97.8%. The final refinement parameters: R_1 = 0.0334, wR_2 = 0.0624 for the reflections with $I > 2\sigma(I)$, R_1 = 0.0640, wR_2 = 0.0668 for all reflections, at the quality factor S = 1.001. The peaks of maximum and minimum of the residual electron density are 0.121 and –0.098 e Å^{-3}.

A complete set of crystallographic data of the compound is deposited in the Cambridge Crystallographic Database (CCDC 814140).

4-Aryl-8-(arylmethylene)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclopentan]-2(4*H*)-ones (VIIIa–VIIId). A round bottom flask of 250 ml capacity equipped with reflux condenser was charged with 1.5 g of fine zinc turnings, a catalytic amount of mercurous chloride, 5 mmol of 2,6-bis(arylmethylene)cyclohexanone, 5.2 mmol of methyl 1-bromocyclopentanecarboxylate, 20 ml of benzene, 5 ml of ethyl acetate, and 1 ml of hexamethylphosphortriamide (HMPTA). The reaction mixture was refluxed for 4 h, then cooled, decanted from excess zinc, and decomposed with 5% hydrochloric acid. The organic layer was separated, and from the aqueous layer the reaction products were extracted twice with ethyl acetate. After drying the extract over anhydrous sodium sulfate the solvent was evaporated and residual compound **VIII** was recrystallized from ethyl acetate.

8-(Benzylidene)-4-phenyl-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclopentane]-2(4*H*)-one (VIIIa). Yield 1.6 g (57%), mp 129–130ºC. IR spectrum, ν, cm^{-1}: 1680 (C=C), 1750 (C=O). ^{1}H NMR spectrum, δ, ppm:

1.18–1.38 m, 1.43–1.57 m (2H, C_6H_2), 1.63–2.29 m [10H, C_5H_2, $(CH_2)_4$], 2.51–2.63 2.75–2.85 m (2H, C_7H_2), 3.00 s (1H, C^4H), 7.06 br.s (1H, =CH), 7.10–7.35 m (10H, 2Ph). ^{13}C NMR spectrum, δ, ppm: 22.49 (C^6), 24.87 ($C^{3'}$), 25.30 ($C^{4'}$), 26.80 (C^7), 11.28 (C^5), 32.32 ($C^{2'}$), 39.05 ($C^{5'}$), 52.83 (C^3), 55.45 (C^4), 118.72 (C^{4a}), 122.67 (CH=), 126.51 [C^4 (PhC^4)], 127.36 [C^4 (PhCH=)], 127.98 [C^2, C^6 (PhC^4)], 128.01 [C^3, C^5 (PhC^4)], 128.68 [C^3, C^5 (PhCH=)], 128.88 [C^1 (PhC^4)], 129.25 [C^2, C^6 (PhCH=)], 137.11 [C^1 (PhCH=)], 139.41 (C^8), 143.65 (C^{8a}), 173.36 (C^2=O). Found, %: C 84.47, H 6.90. $C_{26}H_{26}O_2$. Calculated, %: C 84.29, H 7.07.

8-(2-Fluorobenzylidene)-4-(2-fluorophenyl)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclopentane]-2(4*H*)-one (VIIIb). Yield 1.12 g (55%), mp 151–152ºC. IR spectrum, ν, cm^{-1}: 1700 (C=C), 1755 (C=O). ^1H NMR spectrum, δ, ppm: 1.27–1.40 m, 1.47–1.60 m (2H, C_6H_2), 1.68–2.33 m [10H, C_5H_2, $(CH_2)_4$], 2.43–2.56 2.58–2.70 m (2H, C_7H_2), 3.58 s (1H, C^4H), 7.03 s (1H, =CH), 7.05–7.32 m [8H, 2 (2-FC_6H_4)]. Found, %: C 77.09, H 6.14. $C_{26}H_{24}F_2O_2$. Calculated, %: C 76.83, H 5.95.

8-(3,4-Dimethoxybenzylidene)-4-(3,4-dimethoxyphenyl)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclopentane]-2(4*H*)-one (VIIIc). Yield 1.40 g (55%), mp 168–169ºC. IR spectrum, ν, cm^{-1}: 1695 (C=C), 1745 (C=O). ^1H NMR spectrum, δ, ppm: 1.21–1.40 m, 1.46–1.62 m (2H, C_6H_2), 1.65–2.30 m [10H, C_5H_2, $(CH_2)_4$], 2.53–2.66 2.80–2.91 m (2H, C_7H_2), 2.94 s (1H, C^4H), 3.84 s, 3.89 s (12H, 4MeO), 6.61 s, 6.68 d, 6.77 d, 6.86 d, 6.89 s, 6.94 d {6H, 2[3,4-(MeO) $2C_6H_3$], *J* 8.1 Hz}, 6.99 s (1H, =CH). Found, %: C 73.23, H 7.16. $C_{30}H_{34}O_2$. Calculated, %: C 73.45, H 6.99.

8-(4-Chlorobenzylidene)-4-(4-chlorophenyl)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclopentane]-2(4*H*)-one (VIIId). Yield 0.97 g (44%), mp 64–65ºC. IR spectrum, ν, cm^{-1}: 1685 (C=C), 1750 (C=O). ^1H NMR spectrum, δ, ppm: 1.21–1.34 m, 1.44–1.57 m (2H, C_6H_2), 1.63–2.29 m [10H, C_5H_2, $(CH_2)_4$], 2.48–2.62, 2.71–2.82 m (2H, C_7H_2), 2.99 s (1H, C^4H), 6.99 br.s (1H, =CH), 7.05 d, 7.22–7.40 m [8H, 2 (4-ClC_6H_4), *J* 8.4 Hz]. Found, %: C 70.84, H 5.68, Cl 16.33. $C_{26}H_{24}Cl_2O_2$. Calculated, %: C 71.07, H 5.51, Cl 16.14.

4-Aryl-8-(arylmethylene)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclohexane]-2(4*H*)-ones (IXa–IXe) and 4-aryl-8-(arylmethylene)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cycloheptane]-2(4*H*)-ones (Xa–Xc) were obtained similarly to compounds **VIII** from methyl 1-bromocyclohexane and 1-bromocycloheptanecarboxylic acid.

8-(Benzylidene)-4-phenyl-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclohexane]-2(4*H*)-one (IXa). Yield 0.75 g (39%), mp 175–176ºC. IR spectrum, ν, cm^{-1}: 1700 (C=C), 1745 (C=O). ^1H NMR spectrum, δ, ppm: 1.05–2.28 m [14H, C_5H_2, C_6H_2, $(CH_2)_5$], 2.51–2.63, 2.74–2.84 m (2H, C_7H_2), 3.16 s (1H, C^4H), 7.00 s (1H, =CH), 7.10–7.34 m (10H, 2Ph). Found, %: C 84.60, H 7.48. $C_{27}H_{28}O_2$. Calculated, %: C 84.34, H 7.34.

8-(2-Fluorobenzylidene)-4-(2-fluorophenyl)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclohexane]-2(4*H*)-one (IXb). Yield 1.17 g (54%), mp 142–143ºC. IR spectrum, ν, cm^{-1}: 1685 (C=C), 1745 (C=O). ^1H NMR spectrum, δ, ppm: 1.05–2.28 m [14H, C_5H_2, C_6H_2, $(CH_2)_5$], 2.42–2.88 m (2H, C_7H_2), 3.65 s (1H, C^4H), 7.00 s (1H, =CH), 7.02–7.32 m [8H, 2 (2-FC_6H_4)]. Found, %: C 77.39, H 6.08. $C_{27}H_{26}F_2O_2$. Calculated, %: C 77.12, H 6.23.

8-(3,4-Dimethoxybenzylidene)-4-(3,4-dimethoxyphenyl)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclohexane]-2 (4*H*)-one (IXc). Yield 1.67 g (66%), mp 170–171ºC. IR spectrum, ν, cm^{-1}: 1690 (C=C), 1745 (C=O). ^1H NMR spectrum, δ, ppm: 1.06–2.30 m [14H, C_5H_2, C_6H_2, $(CH_2)_5$], 2.54–2.65, 2.80–2.91 m (2H, C_7H_2), 3.11 s (1H, C^4H), 3.84 s, 3.89 s (12H, 4MeO), 6.61 s, 6.67 d, 6.76 d, 6.85 d, 6.89 s, 6.94 d [6H, 2 (3,4-(MeO) $2C_6H_3$), *J* 8.1 Hz], 6.96 s (1H, =CH). Found, %: C 73.98, H 7.01. $C_{31}H_{36}O_6$. Calculated, %: C 73.79, H 7.19.

8-(4-Chlorobenzylidene)-4-(4-chlorophenyl)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclohexane]-2(4*H*)-one (IXd). Yield 0.82 g (36%), mp 85–86ºC. IR spectrum, ν, cm^{-1}: 1700 (C=C), 1765 (C=O). ^1H NMR spectrum, δ, ppm: 1.03–2.28 m [14H, C_5H_2, C_6H_2, $(CH_2)_5$], 2.48–2.60 2.70–2.81 m (2H, C_7H_2), 3.15 s (1H, C^4H), 6.96 br.s (1H, =CH), 7.05 d, 7.23–7.33 m [8H, 2(4–ClC_6H_4), *J* 8.4 Hz]. Found, %: C 71.77, H 5.86, Cl 15.87 $C_{27}H_{26}Cl_2O_2$. Calculated, %: C 71.52, H 5.78, Cl 15.64.

8-(4-Methoxybenzylidene)-4-(4-methoxyphenyl)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cyclohexane]-2(4*H*)-one (IXe). Yield 1.31 g (59%), mp 122–123ºC. IR spectrum, ν, cm^{-1}: 1690 (C=C), 1745 (C=O). ^1H NMR spectrum, δ, ppm: 1.05–2.26 m [14H, C_5H_2, C_6H_2, $(CH_2)_5$], 2.49–2.62, 2.75–2.86 m (2H, C_7H_2), 3.11 s (1H, C^4H), 3.76 s, 3.81 s (6H, 2MeO), 6.80 g, 6.88 g, 7.03 d, 7.28 d [8H, 2 (4-MeOC_6H_4), *J* 8.7 Hz], 6.95 s (1H, =CH). Found, %: C 78.51, H 7.44. $C_{29}H_{32}O_4$. Calculated, %: C 78.35, H 7.26.

8-(Benzylidene)-4-phenyl-5,6,7,8-tetrahydrospiro[chromene-3,1'-cycloheptane]-2(4*H*)-one (Xa). Yield 1.30 g (65%), mp 156–157°C. IR spectrum, ν, cm^{-1}: 1700 (C=C), 1745 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.10–2.27 m [16H, C_5H_2, C_6H_2, $(CH_2)_6$], 2.52–2.64 2.74–2.85 m (2H, C_7H_2), 3.10 s (1H, C^4H), 7.04 s (1H, =CH), 7.09 d, 7.19–7.38 m (10H, 2Ph, *J* 8.1 Hz). Found, %: C 84.17, H 7.69. $C_{28}H_{30}O_2$. Calculated, %: C 84.38, H 7.59.

8-(2-Fluorobenzylidene)-4-(2-fluorophenyl)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cycloheptane]-2(4*H*)-one (Xb). Yield 1.11 g (51%), mp 168–169°C. IR spectrum, ν, cm^{-1}: 1700 (C=C), 1755 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.10–2.30 m [16H, C_5H_2, C_6H_2, $(CH_2)_6$], 2.42–2.68 m (2H, C_7H_2), 3.73 s (1H, C^4H), 7.00 s (1H, =CH), 7.02–7.32 m [8H, 2 (2-FC_6H_4)]. Found, %: C 77.19, H 6.62. $C_{28}H_{28}F_2O_2$. Calculated, %: C 77.40, H 6.50.

8-(3,4-Dimethoxybenzylidene)-4-(3,4-dimethoxyphenyl)-5,6,7,8-tetrahydrospiro[chromene-3,1'-cycloheptane]-2(4*H*)-one (Xc). Yield 1.84 g (71%), mp 170–171°C. IR spectrum, ν, cm^{-1}: 1690 (C=C), 1740 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.12–2.28 m [16H, C_5H_2, C_6H_2, $(CH_2)_6$], 2.53–2.66 2.79–2.90 m (2H, C_7H_2), 3.04 s (1H, C^4H), 3.83 s, 3.84 s, 3.89 s (12H, 4 MeO), 6.58 s, 6.65 d, 6.76 d, 6.85 d, 6.89 s, 6.94 d [6H, 2 (3,4-(MeO) 2C_6H_3), *J* 8.1 Hz], 6.97 s (1H, =CH). Found, %: C 73.88, H 7.50. $C_{32}H_{38}O_6$. Calculated, %: C 74.11, H 7.39.

8-Benzylidene-3,3-dimethyl-4-phenyl-3,4,5,6,7,8-hexahydro-2*H*-chromene-2-one (XI) was obtained similarly to compound **VIII** from methyl 2-bromo-2-methylpropanoic acid and 2,6-bis(benzylidene)cyclohexanone. Yield 1.30 g (48%), mp 158–159°C. IR spectrum, ν, cm^{-1}: 1680 (C=C), 1750 (C=O). ^{1}H NMR spectrum, δ, ppm: 0.98 s (3H, Me), 1.45 s (3H, Me), 1.46–1.60 m, 1.66–1.80 m (2H, C_6H_2), 2.00–2.12 m, 2.14–2.27 m (2H, C_5H_2), 2.53–2.66 m, 2.73–2.84 m (2H, C_7H_2), 2.95 s (1H, C^4H), 7.06 s (1H, =CH), 7.07–7.34 m (10H, 2Ph). ^{13}C NMR spectrum, δ, ppm: 22.41 (Me), 22.51 (C^6), 26.86 (C^7), 26.94 (Me), 28.06 (C^5), 41.47 (C^3), 56.77 (C^4), 117.76 (C^{4a}) , 122.94 (CH=), 126.53 [C^4 ($\underline{Ph}C^4$)], 127.43 [C^4 ($\underline{Ph}$CH=)], 127.98 [C^2, C^6 ($\underline{Ph}C^4$)], 128.12 [C^3, C^5 ($\underline{Ph}C^4$)], 128.18 [C^1 ($\underline{Ph}C^4$)], 128.67 [C^3, C^5 ($\underline{Ph}$CH=)], 129.24 [C^2, C^6 ($\underline{Ph}$CH=)], 137.15 [C^1 ($\underline{Ph}$CH=)], 138.91 (C^8), 143.28 (C^{8a}), 173. 27 (C^2=O). Found, %: C 83.91, H 7.17. $C_{24}H_{24}O_2$. Calculated, %: C 83.69, H 7.02.

REFERENCES

1. Kirillov, N.F. and Gavrilov, A.G., *Zh. Obshch. Khim.*, 2008, vol. 78, no. 7, p. 1189.
2. Kirillov, N.F. and Gavrilov, A.G., *Zh. Org. Khim.*, 2008, vol. 44, no. 7, p. 975.
3. Arbuzov, B.A., Yuldasheva, L.K., and Arshinova, R.P., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1969, no. 5, p. 1011.
4. *http://classic.chem.msu.su/gran/firefly/index.html.*
5. Sheldrick, G.M., *Acta Cryst., A*, 2008, vol. 64, p. 112.