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Synthesis of Higher Spiroacetals by Condensation of Alkyl- and Cycloalkylcyclopentanones and Cyclohexanones with Dihydric Alcohols in the Presence of Heterogenic Catalysts

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Abstract—The condensation of C_3–C_7 alkyl- and cycloalkylcyclopentanones and cyclohexanones with 1,2-ethane-, 1,2-propane-, and 1,3-butanediols in the presence of heterogenic acid catalysts provided new representatives of spiroacetals. The highest yields of reaction products were obtained in the presence of phosphomolybdic heteropolyacid additionally modified with cobalt and bromine, and also in the presence of the chlorinated cationite KU-23 at 110–130°C. The synthesized spiroacetals possess the jasmine and menthol-wooden fragrance of various tinges.

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Spiroacetal containing various functional groups alongside the acetal moieties are included into many pharmaceuticals and, содержащанде наряду с ацетальныmand фрагментамand разлandчные фunqцandональные группы, [1], are applied as synthetic fragrant substances [2], solvents for cellulose ethers [3], biologically active preparations in the agrochemistry [4], promising monomers [5], insecticides of high biological destruction [6]. Scarce published papers describe the preparation of spiroacetals from cyclohexanone [7–9] and its derivatives [10] in the presence of homogenic [7–10] and heterogenic catalysts [11]. Yet the information on the synthesis of higher spiroacetals is virtually absent. We formerly developed the ways and processes of the preparation of C_5–C_7 alkyl- and cycloalkyl-substituted cyclopentanones, cyclohexanones, and a norcamphor [12–14].

The ketones obtained are analogs of the naturally occurring jasmone and possess odors of jasmine and menthol-wooden type with various tinges. Some of them found the application in perfume and cosmetic production [2].

This article describes the preparation of higher spiroacetals by the condensation of C_3–C_7 alkyl-, dialkyl-, and cycloalkyl-substituted cyclanones with 1,2-ethane-, 1,2-propane-, and 1,3-butanediols in the presence of various heterogenic acid catalysts.

The following catalysts were used: phosphomolybdic heteropolyacid modified with cobalt and bromine (PMHA) $P_{0.17}Mo_{2.4}CoBr_{0.27}O_{6.8}$, chlorine-containing cationites KU-2×8 and KU-23, zeocar-2, modified forms of synthetic mordenite (HNaZn-mordenite), and of natural clintoptilolite (Ai-Dag deposit).

The yields of spiroethylene- and propyleneacetals of alkyl-, dialkyl-, and cycloalkylcyclanones and the selectivity of the reactions depended essentially on the catalyst nature, structures, and the molar ratio of initial compounds (Table 1). As seen from Table 1, the most efficient among the applied catalysts were PMHA modified with cobalt and bromine, chlorinated KU-23 (content of Cl 2.2–9.5%), and HNaZn-mordenite that provided the yields of the corresponding spiroacetals on the level 59–80%.

The dependence of the yield of the target product on the ratio of reacting components , temperature, time of the run, and the structure of the initial ketones was monitored by GLC. The reaction proceeds selectively in the medium

of the azeotrope-forming solvent (toluene or xylene) at the molar ratio ketone–diol 1 : 1–3. The increase in this ratio from 1 : 1 to 1 : 3 favored the increase in the yield of the corresponding acetals by 25–35% (Table 2). The yield of acetals also grew at increasing the temperature from 110 to 140°C. The maximum yield of the target product was attained at 130°C and was 52–80% for spiroacetals of alkyl- and dialkylcyclopentanones, 56–79% for cycloalkylcyclopentanones, and 48–83% for cycloalkylcyclohexanones. The raising of the temperature above 140°C decreased the yield of acetals by ~7–10%, apparently due to partial tarring or because of the increased dehydration rate of the used diols.

The presence of alkyl or cycloalkyl substituents in the molecules of cyclopentanone and cyclohexanone essentially affects their reactivity (Table 3). For instance, in going from cyclopentane to the strained bicycloheptane skeleton the yields of the acetals decrease by ~8–25% apparently due to steric factors hampering the nucleophilic attack on the carbonyl group of the ketones.

At all conditions being the same the yields of acetals depend on the structure of initial diols and grow in the sequence 1,2-ethanediol < 1,2-propanediol < 1,3-butane diol. The effect of the methyl group on the condensation process is due to decreasing the role of the steric factors in the conjugation of the hydroxy groups of the diol molecule with the carbonyl group of cyclic ketones [15].

The obtained spiroacetals like the initial ketones possess odors of jasmine and menthol-wooden type but with more delicate various tinge. Some of them found the application as aromatizers of soaps and detergents.

The composition and structure of synthesized acetals **XIa–XIg–XXa–XXd** was confirmed by elemental analysis and the IR and ^{1}H NMR spectra.

In the IR spectra of these compounds the absorption bands in the region 1660 and 1703 cm^{-1} characteristic of the stretching vibrations of the C=O group and the wide band at 3400–3500 cm^{-1} belonging to the associated OH group disappeared. The appearance of absorption bands at 950, 1060, 1150 cm^{-1} characteristic of the stretching vibrations of ether bond may be regarded as the proof of the presence of the spiroacetal structure [16, 17]. The other absorption bands in the spectra correspond to the stretching and bending vibrations of the C–H bonds in the CH, CH_2, and CH_3 groups in the acetal rings containing 4–7 carbon atoms.

EXPERIMENTAL

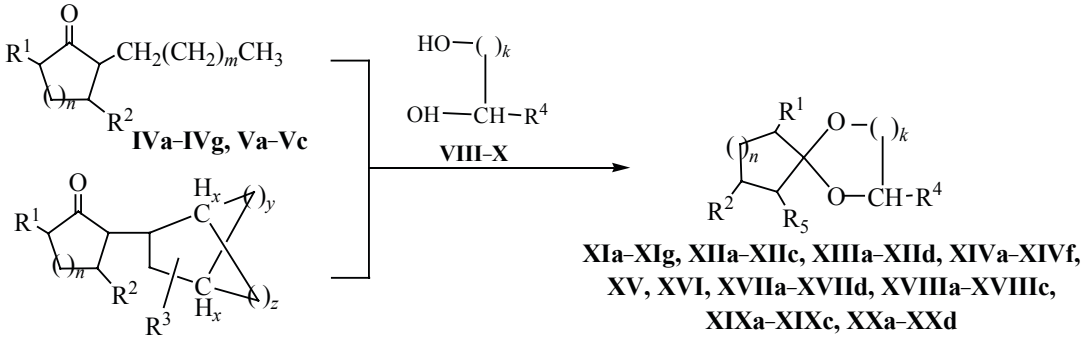
IR spectra were recorded on a spectrophotometer Carl Zeiss UR-20 from thin films. ^{1}H NMR spectra were registered on a spectrometer Bruker (300 MHz) in CCl_4, internal reference HMDS.

The purity and the isomeric composition of compounds were checked by TLC on Silufol UV-254 plates, eluent hexane–ether, 2–4 : 1, development in iodine vapor. GLC was carried out on a chromatograph LKhM -8M-5 equipped with a katharometer, a column 0.3 × 300 cm, stationary phase 5% of XE-60 on Chromaton N-AW DMCS, ramp from 140 to 200°C, carrier gas helium, flow rate 40 ml/min, detector current 75 mA.

Initial ethane-1,2-, propane-1,2-, and butane-1,3-diols

Table 1. Dependence of the yield of spiroacetals on the catalyst nature (ketone–diol 1 : 2, quantity of catalyst 15 wt %)

Compd. no.	Yield, %					
	PMHA	Chlorinated KU-2×8	Chlorinated KU-23	Zeocar-2	HNaZn-mordenite	HNaK-clinoptilolite
XIe	52	46	64	31	55	27
XVa	80	50	60	34	60	26
XVIa	59	47	58	31	56	28
XVIIb	80	38	51	25	50	19
XVIIc	69	42	48	28	51	16
XVIIIb	79	50	48	30	48	27
XVIIIc	70	35	47	22	47	26
XIXb	78	41	52	27	41	19
XIXc	67	32	40	31	47	11
XXb	74	50	49	32	51	25
XXc	65	45	52	25	52	19



IV, $n = 1$: $R^1 = R^2 = H$, $m = 1$ (**a**), 2 (**b**), 3 (**c**), 4 (**d**), 5 (**e**); $m = 5$, $R^1 = H$, $R^2 = Me$ (**f**), $R^1 = Me$, $R^2 = H$ (**g**); **V**, $n = 2$, $R^1 = R^2 = H$, $m = 3$ (**a**), 4 (**b**), 5 (**c**); **VI**, $R^1 = R^2 = R^3 = H$; $n = z = 1$, $x = 2$, $y = 0$, (**a**); $n = 1$, $x = z = 2$, $y = 0$ (**b**); $n = x = y = 1$, $z = 2$ (**c**); $R^1 = R^2 = H$, $R^3 = Me$ (**d**); **VII**, $R^1 = R^2 = R^3 = H$; $n = x = 2$, $y = 0$, $z = 1$ (**a**); $n = x = z = 2$, $y = 0$ (**b**); $n = z = 2$, $x = y = 1$ (**c**); $R^1 = R^2 = H$, $R^3 = Me$ (**d**); $R^1 = Me$, $R^2 = R^3 = H$ (**e**); $R^2 = H$, $R^1 = R^3 = Me$ (**f**); $k = 1$, $R^4 = H$ **VIII**, Me **IX**; $R^4 = Me$, $k = 2$ **X**; **XI**, $n = k = 1$: $R^1 = R^2 = R^4 = H$, $R^5 = Pr$ (**a**), Bu (**b**), C_5H_{11} (**c**), C_6H_{13} (**d**), C_7H_{15} (**e**); $R^5 = C_7H_{15}$, $R^1 = R^4 = H$, $R^2 = Me$ (**f**); $R^1 = Me$, $R^2 = R^4 = H$, $R^5 = C_7H_{15}$ (**g**); **XII**, $n = 2$, $k = 1$, $R^1 = R^2 = R^4 = H$, $R^5 = C_5H_{11}$ (**a**), C_6H_{13} (**b**), C_7H_{15} (**c**); **XIII**, $n = k = 1$, $R^1 = R^2 = R^4 = H$, $R^5 = C_5H_9$ (**a**), C_6H_{11} (**b**), C_7H_{11} (**c**), C_8H_{14} (d); **XIV**, $n = 2$, $k = 1$, $R^1 = R^2 = R^4 = H$, $R^5 = C_5H_9$ (**a**), C_6H_{11} (**b**), C_7H_{11} (**c**), C_8H_{14} (**d**); $R^1 = Me$, $R^2 = R^4 = H$, $R^5 = C_7H_{11}$ (**e**), C_8H_{14} (**f**); $n = 1$, $R^1 = R^2 = H$, $R^4 = CH_3$, $R^5 = C_7H_{15}$, $k = 1$ (**XV**), 2 (**XVI**); **XVII**, $n = k = 1$, $R^1 = R^2 = H$, $R^4 = Me$, $R^5 = C_5H_9$ (**a**), C_6H_{11} (**b**), C_7H_{11} (**c**), C_8H_{13} (**d**); **XVIII**, $k = 1$, $n = 2$, $R^1 = R^2 = H$, $R^4 = Me$, $R^5 = C_5H_9$ (**a**), C_6H_{11} (**b**), C_7H_{11} (**c**); **XIX**, $n = 1$, $k = 2$, $R^1 = R^2 = H$, $R^4 = Me$: $R^5 = C_5H_9$ (**a**), C_6H_{11} (**b**), C_7H_{11} (**c**); **XX**, $n = 2$, $k = 2$, $R^1 = R^2 = H$, $R^4 = Me$: $R^5 = C_5H_9$ (**a**), C_6H_{11} (**b**), C_7H_{11} (**c**), C_8H_{13} (**d**).

Table 2. Dependence of the yield of spiroacetals (%) on the reagents ratio (C_{ketone} 1 mol, 130°C, 6 h)

Ketone no.	Spiroacetal no.	Diol **VIII**, mol			Spiroacetal no.	Diol **XIX**, mol			Spiroacetal no.	Diol **X**, mol		
		1	2	3		1	2	3		1	2	3
IVe	**XIe**	41	52	56	**XVa**	65	60	89	**XVIa**	44	59	61
Vc	**XIIc**	37	66	71		–	–	–		–	–	–
VIc	**XIIIc**	53	72	78	**XVIIc**	54	69	75	**XIXc**	36	67	71
VIIc	**XIVc**	47	69	78	**XVIIIc**	53	70	74	**XXc**	38	65	71

Table 3. Dependence of the yield of spiroacetals on the structure of initial ketone (ketone–**VIII** 1 : 2, 130°C, 6 h)

Ketone no.	Spiroacetal no.	Yield, %		
		PMHA	Chlorinated KU-23	HNaZn-mordenite
IVa	**XIa**	64	63	58
IVb	**XIb**	62	61	55
IVc	**XIc**	66	65	56
IVd	**XId**	57	57	52
IVe	**XIe**	52	64	55
IVf	**XIf**	76	76	69
IVg	**XIg**	53	50	39
Vc	**XIIc**	66	62	44
VIc	**XIIIc**	72	65	52
VIIc	**XIVc**	69	68	43
VIIe	**XIVe**	59	61	40
VIIf	**XIVf**	55	52	35

were of "chemically pure" grade. Ketones were obtained by the procedure we formerly developed [12]. Into a pressure reactor of 2 liter capacity was charged a desired amount of cyclopentanone [methylcyclopentanone (**II**), cyclohexanone (**III**)], C_3–C_7 α-olefins (or cycloolefins), and di-*tert*-butylperoxide in a molar ratio 4 : 1 : 0.25, the reaction proceeded at 140−160°C for 4–6 h. On cooling the reactor the reaction products were subjected first to distillation at the atmospheric pressure, then in a vacuum to isolate the corresponding alkyl-, dialkyl-, or cycloalkylcyclopentanones or cyclohexanones.

The modified PMHA was obtained by boiling a mixture of orthophosphoric acid molybdenum(VI) oxide, and hydrobromic acid followed by mixing the separated precipitate with cobalt(II) carbonate, evaporation, and drying of the mass obtained at 100–120°C [18]. The chlorinated KU-2×8 and KU-23 were prepared as described in [19]. HNaZn-form of mordenite was obtained by impregnation of HNa-mordenite with a solution of $Zn(NO_3)_2$ followed by drying at 100–120°C and calcining at 450–500°C.

Synthesis of spiroacetals. General procedure. The condensation was carried out in a glass reactor of capacity 250 ml equipped with the temperature control (110–140 ± 0.2°C), thermometer, reflux condenser, and Dean-Stark trap. The necessary amount of reagents (0.1 mol of ketone and 0.2 mol of diol) and the solvent was charged into the reactor. The reaction was terminated when the calculated amount of water was separated with the azeotrope. The organic layer was separated from the catalyst, washed till the neutral reaction (against litmus), dried with Na_2SO_4 , and the target product was isolated by distillation.

5-Propyl-1,4-dioxaspiro[4.4]nonane (XIa) was obtained from 12.6 g of compound **IVa** and 12.4 g of compound **VIII**. Yield 10.8 g (64%), bp 109–111°C (18 mm Hg), d_4^{20} 0.9826, n_D^{20} 1.4548. MR_D 46.92, calc. 46.89. IR spectrum, ν, cm^{-1}: 1155–1040, 650. ^{1}H NMR spectrum, δ, ppm: 0.97 t (3H, CH_3), 1.26–1.83 m (10H, $3CH_2$ of cycle and $2CH_2$ Pr), 2.13 m (1H, H^5, CH), 3.84–3.96 d (4H, $H^{2,3}$, $2CH_2O$) [20]. Found, %: C 70.06; H 10.72. $C_{10}H_{18}O_2$. Calculated, %: C 70.89; H 10.59.

5-Butyl-1,4-dioxaspiro[4.4]nonane (XIb) was obtained from 14 g of compound **IVb** and 12.4 g of compound **VIII**. Yield 11.4 g (62%), bp 112–113°C (18 mm Hg), d_4^{20} 0.9615, n_D^{20} 1.4552. IR spectrum, ν, cm^{-1}: 1140–1070, 650. ^{1}H NMR spectrum, δ, ppm: 0.97 t (3H, CH_3), 1.24–1.83 m (12H, $3CH_2$ of cycle and $3CH_2$ Bu), 2.13 m (1H, H^5, CH), 3.84–3.96 d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 71.96; H 11.06. $C_{11}H_{20}O_2$. Calculated, %: C 71.74; H 10.97.

5-Pentyl-1,4-dioxaspiro[4.4]nonane (XIc) was obtained from 15.4 g of compound **IVc** and 12.4 g of compound **VIII**. Yield 13 g (66%), bp 115–117°C (18 mm Hg), d_4^{20} 0.9608, n_D^{20} 1.4556. IR spectrum, ν, cm^{-1}: 1140–1060, 650. ^{1}H NMR spectrum, δ, ppm: 0.97 t (3H, CH_3), 1.25–1.85 m (14H, $3CH_2$ of cycle and $4CH_2$ Alk), 2.13 m (1H, H^5, CH), 3.88–4.02 d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 72.86; H 11.18. $C_{12}H_{22}O_2$. Calculated, %: C 72.73; H 11.11.

5-Hexyl-1,4-dioxaspiro[4.4]nonane (XId) was obtained from 16.8 g of compound **IVd** and 12.4 g of compound **VIII**. Yield 11.9 g (57%), bp 124–125°C (17 mm Hg), d_4^{20} 0.9613, n_D^{20} 1.4558. IR spectrum, ν, cm^{-1}: 1150–1050, 660. ^{1}H NMR spectrum, δ, ppm: 0.97 t (3H, CH_3), 1.24–1.85 m (16H, $3CH_2$ of cycle and $5CH_2$ Alk), 2.12 m (1H, H^5), 3.88–4.02 d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 73.72; H 11.51. $C_{13}H_{24}O_2$. Calculated, %: C 73.58; H 11.32.

5-Heptyl-1,4-dioxaspiro[4.4]nonane (XIe) was obtained from 18.2 g of compound **IVe** and 12.4 g of compound **VIII**. Yield 11.8 g (52%), bp 141–142°C (10 mm Hg), d_4^{20} 0.9524, n_D^{20} 1.4584. IR spectrum, ν, cm^{-1}: 1150–1055, 660. ^{1}H NMR spectrum, δ, ppm: 0.97 t (3H, CH_3), 1.22–1.84 m (18H, $3CH_2$ of cycle and $6CH_2$ Alk), 2.13 m (1H, H^5), 3.85–3.98 d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 74.86; H 11.62. $C_{14}H_{26}O_2$. Calculated, %: C 74.74; H 11.50.

5-Heptyl-6-methyl-1,4-dioxaspiro[4.4]nonane (XIf) was obtained from 19.6 g of compound **IVf** and 12.4 g of compound **VIII**. Yield 18.1 g (76%), bp 126–128°C (1.2 mm Hg), d_4^{20} 0.9524, n_D^{20} 1.4598. IR spectrum, ν, cm^{-1}: 1150–1070, 650. ^{1}H NMR spectrum, δ, ppm: 0.85 t (3H, CH_3 Alk), 1.22 d.d (3H, 6-CH_3), 1.28–1.55 d (16H, $2CH_2$ of cycle and $6CH_2$ Alk), 2.81 d (1H, H^6, CH), 3.02 m (1H, H^5, CH), 3.48–3.60 d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 74.63; H 12.42. $C_{15}H_{28}O_2$. Calculated, %: C 75.0; H 11.67.

5-Methyl-8-heptyl-1,4-dioxaspiro[4.4]nonane (XIg) was obtained from 19.6 g of compound **IVg** and 12.4 g of compound **VIII**. Yield 12 g (53%), bp 125–127°C (1.2 mm Hg), d_4^{20} 0.9518, n_D^{20} 1.4591. IR spectrum, ν, cm^{-1}: 1150–1060, 650. ^{1}H NMR spectrum, δ, ppm: 0.95 t (3H, CH_3Alk), 1.20 d (3H, 5-CH_3), 1.28–1.55 m (16H, $2CH_2$ of cycle and 6 CH_2 Alk), 2.84 d (H^8, CH), 3.00 m

(1H, H^5, CH), 3.48–3.61 d (4H, $H^{2,3}$, CH_2O). Found, %: C 73.96; H 12.65. $C_{15}H_{28}O_2$. Calculated, %: C 75.0; H 11.67.

5-Pentyl-1,4-dioxaspiro[4.5]decane (XIIa) was obtained from 16.8 **r** of compound **Va** and 12.4 g of compound **VIII**. Yield 14.5 g (69%), bp 103–105°C (1.2 mm Hg), d_4^{20} 0.9628, n_D^{20} 1.4582. IR spectrum, ν, cm^{-1}: 1350, 1200–1040, 650. ^1H NMR spectrum, δ, ppm: 0.95 m (3H, CH_3), 1.23–1.77 m (16H, $4CH_2$ of cycle and $4CH_2$ Alk), 2.08 m (1H, H^5, CH), 3.84– 3.96 d.d (4H, $H^{2,3}$, $2CH_2O$). Found %: C 73.86; H 11.76. $C_{13}H_{24}O_2$. Calculated %: C 73.58; H 11.32.

5-Hexyl-1,4-dioxaspiro[4.5]decane (XIIb) was obtained from 18.2 g of compound **Vb** and 12.4 g of compound **VIII**. Yield 15 g (67%), bp 119–121°C (1.2 mm Hg), d_4^{20} 0.9612, n_D^{20} 1.4634. IR spectrum, ν, cm^{-1}: 1340, 1200–1040, 655. ^1H NMR spectrum, δ, ppm: 0.95 m (3H, CH_3), 1.24–1.80 m (18H, $4CH_2$ of cycle and $5CH_2$ Alk), 2.08 m (1H, H^5, CH), 3.84– 3.96 d.d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 74.28; H 11.82. $C_{14}H_{26}O_2$. Calculated, %: C 74.34; H 11.50.

5-Heptyl-1,4-dioxaspiro[4.5]decane (XIIc) was obtained from 19.6 g of compound **Vc** and 12.4 g of compound **VIII**. Yield 15.7 g (66%), bp 132–134°C (1.2 mm Hg), d_4^{20} 0.9582, n_D^{20} 1.4657. IR spectrum, ν, cm^{-1}: 1340, 1175–1040, 650. ^1H NMR spectrum, δ, ppm: 0.97 m (3H, CH_3), 1.24–1.80 m (20H, $4CH_2$ of cycle and $6CH_2$ Alk), 2.06 m (H^5, CH), 3.86–3.98 d.d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 75.64; H 11.88. $C_{15}H_{26}O_2$. Calculated, %: C 75.0; H 11.67.

5-Cyclopentyl-1,4-dioxaspiro[4.4]nonane (XIIIa) was obtained from 15.2 g of compound **VIa** and 12.4 g of compound **VIII**. Yield 15.6 g (79%), bp 144–146°C (3 mm Hg), d_4^{20} 1.0474, n_D^{20} 1.4884. IR spectrum, ν, cm^{-1}: 1200, 1200–1040. ^1H NMR spectrum, δ, ppm: 1.33–1.84 m (14H, $3CH_2$ spiro- and $4CH_2$ Pent), 1.48 d (1H, CH Pent), 2.12 t (1H, H^5, CH), 3.86–3.94 d.d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 43.42; H 10.28. $C_{12}H_{20}O_2$. Calculated, %: C 73.47; H 10.2.

5-Cyclohexyl-1,4-dioxaspiro[4.4]nonane (XIIIb) was obtained from 16.6 g of compound **VIb** and 12.4 g of compound **VIII**. Yield 16.6 g (78%), bp 151–153°C (3 mm Hg), d_4^{20} 1.0453, n_D^{20} 1.4948. IR spectrum, ν, cm^{-1}: 1200–1040. ^1H NMR spectrum, δ, ppm: 0.82–1.62m (16H, $3CH_2$ spiro- and $5CH_2$ Hex), 1.28 t (1H, H^1, CH Hex), 2.12 m (1H, H^5, spiro), 3.86–3.94 d.d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 74.25; H 10.52. $C_{13}H_{22}O_2$. Calculated, %: C 74.29; H 10.48.

5-(Bicyclo[2.2.1]heptyl)-1,4-dioxaspiro[4.4]-nonane (XIIIc) was obtained from 17.8 g of compound **VIc** and 12.4 g of compound **VIII**. Yield 15.9 g (72%), bp 96–98°C (2 mm Hg), d_4^{20} 1.1081, n_D^{20} 1.5108. IR spectrum, ν, cm^{-1}: 1200–1040. ^1H NMR spectrum, δ, ppm: 1.22–1.56 d (8H, $4CH_2$ Hept), 1.36–1.83 m (6H, $3CH_2$ spiro), 1.42 t (1H, H^2, CH Hept), 1.43–1.45 d (2H, $H^{1,4}$, 2CH Hept), 2.14 m (1H, H^5, CH spiro), 3.84–3.96 d.d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 75.64; H 10.03. $C_{14}H_{22}O_2$. Calculated, %: C 75.67; H 9.91.

5-(6-Methylbicyclo[2.2.1]heptyl)-1,4-dioxaspiro[4.4]nonane (XIIId) was obtained from 19.2 g of compound **VId** and 12.4 g of compound **VIII**. Yield 16.2 g (69%), bp 108–110°C (2 mm Hg), d_4^{20} 1.0836, n_D^{20} 1.5078. IR spectrum, ν, cm^{-1}: 1200–1040. ^1H NMR spectrum, δ, ppm: 1.07 d (3H, CH_3), 1.23–1.54 d (6H, $3CH_2$ Hept), 1.36–1.83 m (6H, $3CH_2$ spiro), 1.41 t (1H, H^2, CH), 1.41–1.44 d (2H, $H^{1,4}$, 2CH Hept), 1.62 t (1H, H^6, CH), 2.14 (1H, H^5, CH spiro), 3.84–3.99 d.d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 76.2; H 10.22. $C_{15}H_{24}O_2$. Calculated, %: C 76.27; H 10.17.

5-Cyclopentyl-1,4-dioxaspiro[4.5]decane (XIVa) was obtained from 16.6 g of compound **VIIa** and 12.4 g of compound **VIII**. Yield 17.5 g (83%), bp 157–160°C (3 mm Hg), d_4^{20} 1.0564, n_D^{20} 1.4910. IR spectrum, ν, cm^{-1}: 1445, 1420 [δ(CH_2 of cycle)], 1200–1040 (C–O–C). ^1H NMR spectrum, δ, ppm: 1.26–1.76 m (8H, $4CH_2$ spiro), 1.48 m (1H, H^1, CH Pent), 1.36–1.61 m (8H, $4CH_2$ Pent), 2.14 m (1H, H^5, CH spiro), 3.84–3.96 d.d (4H, $2CH_2O$). Found, %: C 76.42; H 10.52. $C_{13}H_{22}O_2$. Calculated, %: C 76.36; H 10.48.

5-Cyclohexyl-1,4-dioxaspiro[4.5]decane (XIVb) was obtained from 18 g of compound **VIIb** and 12.4 g of compound **VIII**. Yield 16.8 g (75%), bp 149–151°C (1.2 mm Hg), d_4^{20} 1.0574, n_D^{20} 1.5098. IR spectrum, ν, cm^{-1}: 1040–1200. ^1H NMR spectrum, δ, ppm: 0.85–1.64 m (10H, $5CH_2$ Hex), 1.30 m (1H, H^1, CH Hex), 1.32–1.83 m (8H, $4CH_2$ spiro), 2.12 m (1H, H^5, spiro), 3.84–3.96 d.d (4H, $H^{2,3}$, $2CH_2O$). Found, %: C 74.93; H 10.81. $C_{14}H_{24}O_2$. Calculated, %: C 75.0; H 10.71.

5-(Bicyclo[2.2.1]heptyl)-1,4-dioxaspiro[4.5]-decane (XIVc) was obtained from 19.2 g of compound VIIc and 12.4 g of compound **VIII**. Yield 16.2 g (69%), bp 139–141°C (2 mm Hg), d_4^{20} 1.003, n_D^{20} 1.5140. IR spectrum, ν, cm^{-1}: 1200–1055. ^1H NMR spectrum, δ, ppm: 1.24–1.56 d (8H, $4CH_2$ Hept), 1.41 t (1H, H^2, CH Hept), 1.43–1.46 d (2H, $H^{1,4}$, $2CH_2$ Hept), 1.32–1.83 m

(8H, 4CH_2 spiro), 2.14 m (1H, H^5, CH spiro), 3.84–3.96 d.d (4H, H^{2,3}, 2CH_2O). Found, %: C 76.25; H 10.22. $C_{15}H_{24}O_2$. Calculated, %: C 76.27; H 10.17.

5-(6-Methylbicyclo[2.2.1]heptyl)-1,4-dioxaspiro[4.5]decane (XIVd) was obtained from 20.6 g of compound **VIId** and 12.4 g of compound **VIII**. Yield 15.9 g (64%), bp 137–138°C (1 mm Hg), d_4^{20} 1.0735, n_D^{20} 1.5165. IR spectrum, ν, cm^{-1}: 1200–1060. ^{1}H NMR spectrum, δ, ppm: 1.05 d (3H, CH_3), 1.22–1.58 d (6H, 3CH_2 Hept), 1.36–1.83 m (8H, 4CH_2 spiro), 1.40 t (1H, H^2, CH Hept), 1.42–1.44 d (2H, H^{1,4}, CH Hept), 2.12 m (1H, H^5, CH spiro), 3.86–3.94 d.d (4H, H^{2,3}, 2CH_2O). Found, %: C 76.74; H 10.46. $C_{16}H_{26}O_2$. Calculated, %: C 76.8; H 10.4.

5-Methyl-9-(bicyclo[2.2.1]heptyl)-1,4-dioxaspiro[4.5]decane (XIVe) was obtained from 20.6 g of compound VIIe and 12.4 g of compound **VIII**. Yield 14.8 g (59%), bp 130–132°C (1 mm Hg), d_4^{20} 1.0768, n_D^{20} 1.5112. IR spectrum, ν, cm^{-1}: 2870, 1380 (CH_3), 1200–1060 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.22 d (3H, CH_3C^5), 1.23–1.54 m (8H, 4CH_2 Hept), 1.28–1.52 d.d (6H, 3CH_2 spiro), 1.41 t (1H, H^2, Hept), 1.41–1.43 d (2H, H^{1,4}, 2CH Hept), 2.14 m (1H, H^5, CH spiro), 3.84–3.96 d.d (4H, H^{2,3}, 2CH_2O). Found, %: C 76.75; H 10.47. $C_{16}H_{26}O_2$. Calculated, %: C 76.8; H 10.4.

5-Methyl-9-(6-methylbicyclo[2.2.1]heptyl)-1,4-dioxaspiro[4.5]decane (XIVf) was obtained from 22 g of compound **VIIf** and 12.4 g of compound **VIII**. Yield 14.4 g (54%), bp 144–145°C (1 mm Hg), d_4^{20} 1.0525, n_D^{20} 1.5068. IR spectrum, ν, cm^{-1}: 2870, 1380, 1360 (CH_3), 1200–1050 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.07 d (3H, 6-CH_3), 1.22 d (3H, 5-CH_3), 1.23–1.54 m (6H, 3CH_2 Hept), 1.26–1.78 m (6H, 3CH_2 spiro) 1.41 t (1H, H^2, CH Hept), 1.41–1.43 d (2H, H^{1,4}, 2CH Hept), 1.61 t (1H, H^6, CH Hept), 2.12 m (1H, H^5, CH spiro), 3.84–3.96 d.d (4H, H^{2,3}, 2CH_2O). Found, %: C 77.27; H 10.65. $C_{17}H_{28}O_2$. Calculated, %: C 77.27; H 10.6.

5-Heptyl-2-methyl-1,4-dioxaspiro[4.4]nonane (XVa) was obtained from 18.2 g of compound **IVe** and 15.4 g of compound **IX**. Yield 19.3 g (80%), bp 139–141°C (10 mm Hg), d_4^{20} 0.9422, n_D^{20} 1.4502. IR spectrum, ν, cm^{-1}: 1170–1050, 660. ^{1}H NMR spectrum, δ, ppm: 0.95 t (3H, CH_3, Hept), 1.20 d (3H, CH_3 Ht), 3.04 m (1H, H^5, CH), 4.05 m (1H, H^2, CH), 3.73–3.98 m (2H, H^3, CH_2), 1.28–1.55 m (18H, 3CH_2 spiro and 6CH_2 Hept). Found, %: C 74.86; H 11.73. $C_{15}H_{28}O_2$. Calculated, %: C 75.0; H 11.67.

6-Heptyl-2-methyl-1,5-dioxaspiro[4.5]decane (XVIa) was obtained from 18.2 g of compound **IVe** and 18 g of compound **X**. Yield 15.1 g (59%), bp 134–136°C (5 mm Hg), d_4^{20} 0.9575, n_D^{20} 1.4638. IR spectrum, ν, cm^{-1}: 1200–1050, 650. ^{1}H NMR spectrum, δ, ppm: 0.95 t (CH_3, Hept), 1.20 d (3H, 2-CH_3), 1.28–1.55 m (18H, 3CH_2 spiro and 6CH_2 Hept), 1.50–1.72 m (2H, H^3, CH_2), 3.00 m (1H, H^6, CH), 3.74–3.93 m (2H, H^4, CH_2O), 4.05 m (1H, H^2, CH). Found, %: C 75.72; H 11.96. $C_{16}H_{30}O_2$. Calculated, %: C 75.59; H 11.81.

2-Methyl-5-cyclopentyl-1,4-dioxaspiro[4.4]-nonane (XVIIa) was obtained from 15.2 g of compound **VIa** and 15.2 g of compound **IX**. Yield 17.3 g (83%), bp 127–129°C (1.2 mm Hg), d_4^{20} 1.0523, n_D^{20} 1.4971. IR spectrum, ν, cm^{-1}: 1200–1040. ^{1}H NMR spectrum, δ, ppm: 1.22 t (3H, CH_3), 1.28–1.50 d (4H, H^{6,7}, 2CH_2 spiro), 1.33–1.62 m (8H, 4CH_2 Pent), 1.81 d (1H, CH Pent), 2.83 m (2H, H^5, CH), 3.32 t (1H, H^2, OCH), 3.36–3.61 m (2H, H^3, CH_2O), 3.34–3.36 d (2H, H^8, CH_2). Found, %: C 74.32; H 10.64. $C_{13}H_{22}O_2$. Calculated, %: C 74.28; H 10.48.

2-Methyl-5-cyclohexyl-1,4-dioxaspiro[4.4]-nonane (XVIIb) was obtained from 16.6 g of compound **VIb** and 15.2 g of compound **IX**. Yield 18 g (80%), bp 147–149°C (1.2 mm Hg), d_4^{20} 1.0622, n_D^{20} 1.5164. IR spectrum, ν, cm^{-1}: 1200–1040. ^{1}H NMR spectrum, δ, ppm: 0.82–1.62 m (10H, 5CH_2 Hex), 1.22 t (3H, CH_3), 1.28 t (1H, CH Hex), 2.81 m (1H, H^5, CH), 3.32 t (1H, H^2, OCH), 3.38–3.65 m (2H, H^3, CH_2O), 3.34–3.36 (2H, H^8, CH_2). Found, %: C 75.24; H 10.85. $C_{14}H_{24}O_2$. Calculated, %: C 75.0; H 10.71.

2-Methyl-5-(bicyclo[2.2.1]heptyl)-1,4-dioxaspiro[4.4]nonane (XVIIc) was obtained from 18 g of compound **VIc** and 15.2 g of compound **IX**. Yield 16.3 g (68%), bp 143–145°C (1.2 mm Hg), d_4^{20} 1.0478, n_D^{20} 1.5054. IR spectrum, ν, cm^{-1}: 1200–1040. ^{1}H NMR spectrum, δ, ppm: 1.22 t (3H, CH_3) 1.22–1.58 d (8H, 4CH_2 Hept), 1.36–1.83 m (6H, 3CH_2 spiro), 1.42 t (1H, H^2, CH Hept), 1.43–1.45 d (2H, H^{1,4}, 2CH Hept), 2.81 m (1H, H^5, CH), 3.32 t (1H, H^2, OCH), 3.34–3.60 m (2H, H^3, CH_2O). Found, %: C 76.38; H 10.23. $C_{15}H_{24}O_2$. Calculated, %: C 76.27; H 10.17.

2-Methyl-5-(6-methylbicyclo[2.2.1]heptyl)-1,4-dioxaspiro[4.4]nonane (XVIId) was obtained from 19.2 g of compound **VId** and 15.2 g of compound **IX**. Yield 15.8 g (63%), bp 155–157°C (1.2 mm Hg), d_4^{20} 1.0467, n_D^{20} 1.5102. IR spectrum, ν, cm^{-1}: 1200–

1040. ^{1}H NMR spectrum, δ, ppm: 1.07 d (3H, 6-CH_3), 1.22 d (3H, 2-CH_3), 1.23–1.54 m (6H, 3CH_2 Hept), 1.28–1.52 d.d (4H, H^{6,7}, 2CH_2), 2.83 d (1H, H^5, CH), 3.32 d (1H, H^2, OCH), 3.35–3.64 d.d (4H, H^{3,8}, 2CH_2). Found, %: C 76.65; H 10.66. $C_{16}H_{26}O_2$. Calculated, %: C 76.8; H 10.4.

2-Methyl-5-cyclopentyl-1,4-dioxaspiro[4.5]-decane (XVIIIa) was obtained from 16.6 g of compound **VIIa** and 15.2 g of compound **IX**. Yield 18.8 g (84%), bp 134–136°C (1.2 mm Hg), d_4^{20} 1.0562, n_D^{20} 1.5061. IR spectrum, ν, cm^{-1}: 1460, 1440 (CH_2 of cycle), 1380, 1360 (CH_3), 1200–1050 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.22 t (3H, CH_3), 1.26–1.76 m (8H, 4CH_2 spiro), 1.36–1.61 m (8H, 4CH_2 Pent), 1.48 m (1H, H^1, CH Pent), 2.81 m (1H, H^5, CH), 3.32 t (1H, H^2, OCH), 3.38–3.65 m (2H, H^3, CH_2O). Found, %: C 75.34; H 10.96. $C_{14}H_{24}O_2$. Calculated %: C 75.00; H 10.71.

2-Methyl-5-cyclohexyl-1,4-dioxaspiro[4.5]-decane (XVIIIb) was obtained from 18 g of compound **VIIb** and 15.2 g of compound **IX**. Yield 18.7 g (79%), bp 150–152°C (1.2 mm Hg), d_4^{20} 1.0541, n_D^{20} 1.5120. IR spectrum, ν, cm^{-1}: 1460, 1440 (CH_2 of cycle), 1380, 1360 (CH_3), 1200–1050 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 0.85–1.64 m (10H, 5CH_2 Hex), 1.22 t (3H, CH_3), 1.30 m (1H, H^1, CH Hex), 1.32–1.83 m (8H, 4CH_2 spiro), 3.32 t (1H, H^2, OCH), 3.40–3.68 m (2H, H^3, CH_2O). Найдено, %: C 75.82; H 11.12. $C_{15}H_{26}O_2$. Calculated, %: C 75.63; H 10.92.

5-(Bicyclo[2.2.1]heptyl)-2-methyl-1,4-dioxaspiro[4.5]decane (XVIIIc) was obtained from 19.2 g of compound **VIIc** and 15.2 g of compound **IX**. Yield 17.6 g (70%), bp 161–163°C (1.2 mm Hg), d_4^{20} 1.0748, n_D^{20} 1.5123. IR spectrum, ν, cm^{-1}: 1200–1050. ^{1}H NMR spectrum, δ, ppm: 1.22 t (3H, CH_3), 1.24–1.56 d (8H, 4CH_2 Hept), 1.32–1.83 m (8H, 4CH_2 spiro), 1.41 t (1H, H^2, CH Hept), 1.43–1.46 d (2H, H^{1,4}, 2CH Hept), 2.14 m (1H, H^5, CH spiro), 3.32 t (1H, H^2, OCH), 3.40–3.68 m (2H, H^3, CH_2O). Found, %: C 76.86; H 11.06. $C_{16}H_{26}O_2$. Calculated, %: C 76.8; H 10.4.

2-Methyl-6-cyclopentyl-1,5-dioxaspiro[4.5]decane (XIXa) was obtained from 15.2 g of compound **VIa** and 18.0 g of compound **X**. Yield 18.7 g (84%), bp 132–134°C (1.2 mm Hg), d_4^{20} 1.0648, n_D^{20} 1.5092. IR spectrum, ν, cm^{-1}: 1200–1040. ^{1}H NMR spectrum, δ, ppm: 1.22 t (3H, CH_3), 1.26–1.76 m (8H, 4CH_2 spiro), 1.28–1.50 d (4H, H^{7,8}, 2CH_2), 1.48 m (1H, H^1, CH Pent), 1.36–1.61 m (8H, 4CH_2 Pent), 1.50–1.72 m (2H, H^3, CH_2 Ht), 2.81 m (1H, H^6, CH), 3.32 t (1H, H^2, OCH), 3.32–3.36 d (2H, H^9, CH_2), 3.36–3.61 m (2H, H^4, CH_2O). Found, %: C 75.26; H 11.08. $C_{14}H_{24}O_2$. Calculated, %: C 75.00; H 10.71.

2-Methyl-6-cyclohexyl-1,5-dioxaspiro[4.5]decane (XIXb) was obtained from 16.6 g of compound **VIb** and 18.0 g of compound **X**. Yield 18.6 g (78%), bp 147–149°C (1.2 mm Hg), d_4^{20} 1.0622, n_D^{20} 1.5164. IR spectrum, ν, cm^{-1}: 1200–1040. ^{1}H NMR spectrum, δ, ppm: 0.85–1.64 m (10H, 5CH_2 Hex), 1.22 t (3H, CH_3), 1.30 m (1H, H^1, CH Hex), 1.32–1.83 m (8H, 4CH_2 spiro), 1.50–1.72 m (2H, H^3, CH_2 Ht), 3.32 t (1H, H^2, OCH), 3.38–3.65 m (2H, H^4, CH_2O). Found, %: C 75.77; H 11.11. $C_{15}H_{26}O_2$. Calculated, %: C 75.63; H 10.92.

6-(Bicyclo[2.2.1]heptyl)-2-methyl-1,5-dioxaspiro[4.5]decane (XIXc) was obtained from 17.8 g of compound **VIc** and 18.0 g of compound **X**. Yield 16.7 g (67%), bp 159–161°C (1.2 mm Hg), d_4^{20} 1.0724, n_D^{20} 1.5081. IR spectrum, ν, cm^{-1}: 2870, 1380, 1360 (CH_3), 1200–1050 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.20 t (3H, CH_3 Ht), 1.23–1.54 m (6H, 3CH_2 Hept), 1.30–1.53 d (4H, H^{7,8}, 2CH_2 spiro), 1.43–1.45 d (2H, H^{1,4}, 2CH Hept), 2.81 d (1H, H^6, CH), 3.32 t (1H, H^2, OCH), 3.34–3.36 d (2H, H^9, CH_2), 3.36–3.61 m (2H, H^4, CH_2O). Found, %: C 77.43; H 11.13. $C_{17}H_{28}O_2$. Calculated, %: C 77.27; H 10.61.

2-Methyl-6-cyclopentyl-1,5-dioxaspiro[5.5]undecane (XXa) was obtained from 15.6 g of compound **VIIa** and 18.0 g of compound **X**. Yield 20.3 g (85%), bp 152–154°C (1.2 mm Hg), d_4^{20} 1.0566, n_D^{20} 1.5122. IR spectrum, ν, cm^{-1}: 2870, 1380 (CH_3), 1175–1040 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.22 t (3H, CH_3), 1.28 t (1H, H^1, CH Pent), 1.33–1.62 m (8H, 4CH_2 Pent), 2.81 m (1H, H^6, CH), 3.32 t (1H, H^2, OCH), 3.34–3.61 m (4H, H^{4,10}, 2CH_2). Found, %: C 75.86; H 11.18. $C_{15}H_{26}O_2$. Calculated, %: C 75.63; H 10.92.

2-Methyl-6-cyclohexyl-1,5-dioxaspiro[5.5]undecane (XXb) was obtained from 18.0 g of compound **VIIb** and 18.0 g of compound **X**. Yield 18.6 g (74%), bp 167–169°C (1.2 mm Hg), d_4^{20} 1.0652, n_D^{20} 1.5233. IR spectrum, ν, cm^{-1}: 2870, 1380 (CH_3), 1175–1040 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 0.82–1.62 m (10H, 5CH_2 Hex), 1.22 t (3H, CH_3), 1.28 (1H, H^1, CH Hex), 2.81 m (1H, H^6, CH), 3.32 t (1H, H^2, OCH), 3.34–3.36 d (2H, H^{10}, CH_2), 3.38–3.61 m (2H, H^4, 2CH_2O). Found, %: C 76.39; H 11.24. $C_{16}H_{28}O_2$. Calculated, %: C 76.19; H 11.11.

6-(Bicyclo[2.2.1]heptyl)-2-methyl-1,5-dioxaspiro[5.5]undecane (XXc) was obtained from 19.2 g of compound **VIIc** and 18.0 g of compound **X**. Yield

17.1 g (65%), bp 181–183°C (1.2 mm Hg), d_4^{20} 1.0744, n_D^{20} 1.5184. IR spectrum, ν, cm^{-1}: 2870, 1380, (CH_3), 1200–1040 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.22 t (3H, CH_3), 1.24–1.56 d (8H, 4CH_2 Hept), 1.32–1.83 m (8H, 4CH_2 spiro), 1.43–1.46 d (2H, H^{1,4}, 2CH Hept), 2.14 m (2H, H^6, CH spiro), 3.32 t (1H, H^2, OCH), 3.36–3.68 m (2H, H^4, CH_2O). Found, %: C 77.45; H 10.83. $C_{17}H_{28}O_2$. Calculated %: C 77.27; H 10.61.

2-Methyl-6-(6-methylbicyclo[2.2.1]heptyl)-1,5-dioxaspiro[5.5]undecane (XXd) was obtained from 20.6 g of compound **VIId** and 18.0 g of compound **X**. Yield 17.1 g (62%), bp 193–195°C (1.2 mm Hg). IR spectrum, ν, cm^{-1}: 2870, 1380, 1355 (CH_3), 1200–1050 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 1.07 d (3H, CH_3 Hept), 1.22 t (3H, CH_3 Ht), 1.23–1.54 m (6H, 3CH_2 Hept), 1.28–1.53 d (6H, 3CH_2 spiro), 1.43–1.45 d (2H, H^{1,4}, 2CH Hept), 2.81 d (1H, H^6, CH), 3.32 t (1H, H^2, OCH), 3.34–3.36 d (2H, H^{10}, CH_2), 3.36–3.61 m (2H, H^4, CH_2O). Found, %: C 77.85; H 10.92. $C_{18}H_{30}O_2$. Calculated, %: C 77.69; H 10.79.

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