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Reversible intramolecular Dieckmann-type condensation of 2-(2-hydroxymethyl-5,5,6-trimethoxytetrahydropyran-3-ylcarbonyl)-cyclopentanone: an alternative access to medium-sized lactones

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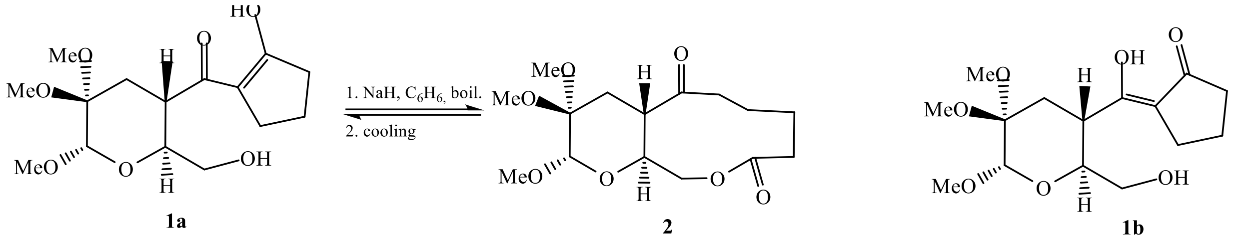
The spectral and analytical data were obtained using the equipment of the *Khimiya* Joint Center at the Institute of Organic Chemistry, Ufa Research Center, Russian Academy of Sciences. ^{1}H and ^{13}C NMR spectra were registered on a spectrometer Bruker AM-300 (300 MHz for ^{1}H and 75.47 MHz for ^{13}C) and on a spectrometer Bruker Avance III (500.13 MHz for ^{1}H and 125.47 MHz for ^{13}C). IR spectra were recorded on spectrophotometers Shimadzu IRPrestige-21 or Bruker Tensor 27 (from films or mulls in mineral oil). Mass spectra were measured on a GC-MS instrument Hewlett Packard, chromatograph HP 6890 with a mass-selective detector HP 5973. Optical rotations were determined on a polarimeter Perkin Elmer-341. Analytical TLC was carried out on Sorbfil plates of the grade PTSKh-AF-A (Sorbpolymer Co., Krasnodar). The melting points were measured on a Boëtius 05 heating block. Electronic structure calculations were carried out using the Gaussian 09, Revision D.01 software package [S1]. Visualization and treatment the results of the quantum chemical calculations were fulfilled in ChemCraft [S2]. The restricted Kohn−Sham method was applied to construct the wave function of reagent and product of Dieckmann reaction (closed shell restricted wave function). The calculations were carried out for standard conditions (298.15 K, 1 atm). Geometry optimization, vibrational frequency calculation, reaction enthalpy, and Gibbs free energy estimation were carried out using hybrid exchange-correlation modification to B97 parametrized by Wilson, Bradley and Tozer (B97-2) [S3]. The triple split valence basis set augmented with *d*- and *p*-type polarization functions and the set of diffuse functions were used [S4]. The structures correspond to minima on the potential energy surface (PES); imaginary vibrational frequencies were not detected in the Hessian calculation. Nonspecific solvation of benzene was accounted for using the polarized continuum model (IEF-PCM) [S5]. Geometrical parameters were determined during the optimization of the PCM-solvated structure.

(2-Hydroxycyclopent-1-en-1-yl)[(2*S*,3*S*,6*S*)-2-hydroxymethyl-5,5,6-trimethoxytetrahydro-2*H*-pyran-3-yl]methanone (1a) [S6], **(1*R*,2*R*,5*S*)-4-[(1*RS*)-2-oxocyclohexyl)-6,8-dioxabicyclo[3.2.1]octan-2-one (6a,b)** [S7], **(1*R*,5*S*)-4-(2-oxocyclohexyl)spiro[7,8-dioxabicyclo[3.2.1]octane-2,2'-[1,3]dioxolane] (7a,b)** [S8], **(4a*S*,6a*S*,10a*R*,*S*,10b*R*)-2,2-ethylenedioxydecahydropyrano[2,3-*c*]chromen-6a(1*H*)-ol (3)** [S9] were prepared as described.

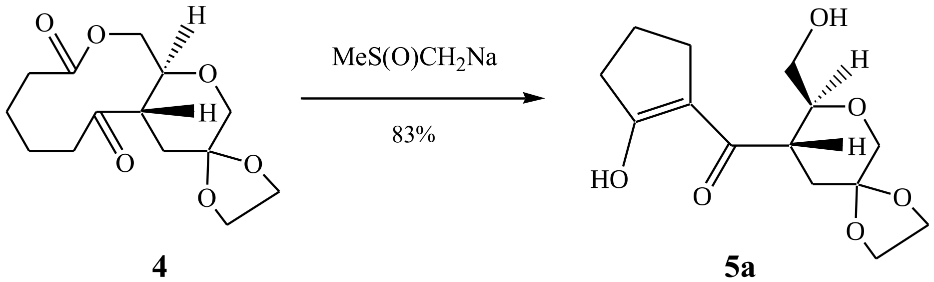
(2*S*,4a*S*,12a*S*)-2,3,3-Trimethoxydecahydropyrano[2,3-*c*]oxecine-5,10-dione (2) and 2-{1-hydroxy-1-[(2*S*,3*S*,6*S*)-2-hydroxymethyl-5,5,6-trimethoxy-tetrahydro-2*H*-pyran-3-yl]methylidene}cyclopentanone (1b).



Method A. Cyclopentenol **1a** (0.04 g, 0.13 mmol) in benzene (1.0 ml) was added to a suspension NaH (0.009 g, 0.39 mmol, obtained by hexane washing of 60% dispersion in mineral oil and removing the residual hexane) in benzene (2.0 ml), and this was boiled under inert atmosphere until the starting material disappeared (TLC monitoring). The reaction mass was then treated with 1% HCl (aq) and extracted with EtOAc (3×5.0 ml). The extract was dried with $MgSO_4$, the solvent was distilled off, the residue was chromatographed on a column of silica gel to afford 2:1 tautomeric mixture **1a,b** (0.017 g, 40%) and lactone **2** (0.017 g, 41%).

Method B. To a solution of compound **1a** (0.05 g, 0.16 mmol) in benzene (3.0 ml) DBU in catalytic quantities (~0.005 ml) was added, and this was boiled under inert atmosphere until the starting material disappeared (TLC monitoring). The solvent was distilled off, the residue was chromatographed on SiO_2 to afford lactone **2** (0.038 g, 75%) and compound **1b** (0.009 g, 18%). Physicochemical characteristics of compound **2** correspond to literature data [S9]. Pale pink oil, R_f 0.14 (petroleum ether-EtOAc, 3:1): ^{1}H NMR ($CDCl_3$), δ, m.d.: 1.58 (m, 4H, $H^{4'A}$, $H^{4'B}$, $H^{3'A}$, $H^{3'B}$), 1.82 (t, 1H, $^2J_{4''A\text{-}4''B}$ 13.1, $^3J_{4''A\text{-}3''}$ 13.1 Hz, $H^{4''A}$), 2.04 (dd, 1H, $H^{4''B}$, $^2J_{4''B\text{-}4''A}$ 13.1, $^3J_{4''B\text{-}3''}$ 3.5 Hz), 2.29 (m, 2H, $H^{5'A}$, $H^{5'B}$), 2.92 (ddd, 1H, $H^{3''}$, $^3J_{3''\text{-}4''A}$ 13.1, $^3J_{3''\text{-}2''}$ 10.6, $^3J_{3''\text{-}4''A}$ 3.8 Hz), 3.24 (s, 3H, OMe), 3.27 (s, 3H, OMe), 3.44 (s, 3H, OMe), 3.52 (dd, 1H, $H^{1'''B}$, $^2J_{1'''B\text{-}1'''A}$ 11.9, $^3J_{1'''B\text{-}2''}$ 4.3 Hz), 3.66 (dd,1H, $H^{1'''A}$, $^2J_{1'''A\text{-}1'''B}$ 11.9, $^3J_{1'''A\text{-}2''}$ 3.0 Hz), 3.99 (ddd,1H, $H^{2''}$, $^3J_{2''\text{-}3''}$ 10.6, $^3J_{2''\text{-}1'''B}$ 4.3, $^3J_{2''\text{-}1'''A}$ 3.0 Hz), 4.57 (s, 1H, $H^{6''}$). ^{13}C NMR ($CDCl_3$), δ: 22.71 ($C^{4'}$), 24.31 ($C^{3'}$), 28.54 ($C^{4''}$), 34.03 ($C^{5'}$), 46.31 ($C^{3''}$), 47.87 (OMe), 48.28 (OMe), 55.03 (OMe), 63.21 ($C^{1'''}$), 69.55 ($C^{2''}$), 96.55 ($C^{5''}$), 97.46 ($C^{6''}$), 110.22 ($C^{1'}$), 173.39 (C^{1}), 210.40 (C=O). Mass spectrum, *m/z*: 317 $[MH]^+$. Found, %: C 56.98, H 7.59. $C_{15}H_{24}O_7$. Calculation, %: C 56.95, H 7.65.

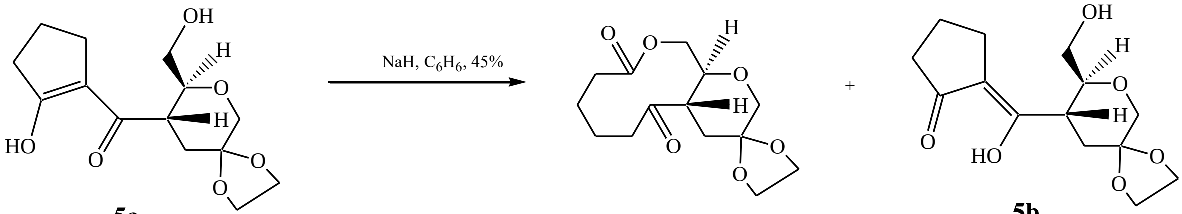
(2-Hydroxycyclopent-1-en-1-yl)[(8*S*,9*S*)-8-hydroxymethyl-1,4,7-trioxaspiro[4.5]dec-9-yl]methanone 5a.



4 **5a**

To a DMSO (0.3 ml) solution of NaH (0.006 g, 0.26 mmol, prepared by hexane washing of 60% dispersion in mineral oil and removing the hexane residue) under argon, a solution of lactone **4** (0.035 g, 0.13 mmol) in DMSO (0.3 ml) was added for 30 minutes, and this was stirred at room temperature until the starting material disappeared (TLC control). The reaction mass was then treated with water (0.5 ml), the products were extracted with EtOAc (3×3.0 ml), the extract was dried with $MgSO_4$, the solvent was distilled off, and the residue was chromatographed on SiO_2 to afford product **5a** (0.029 g, 83%). Colourless oil, $[\alpha]_D^{20}$ +74° (*c* 0.5, $CHCl_3$), R_f 0.3 (EtOAc). ^{1}H NMR ($CDCl_3$), δ, m.d.: 1.85 (m, 1H, $H^{4''A}$), 1.92 (m, 2H, $H^{4'A}$, $H^{4'B}$), 2.12 (m, 1H, $H^{4''B}$), 2.42 м 2H, $H^{3'A}$, $H^{3'B}$), 2.60 (m, 2H, $H^{5'A}$, $H^{5'B}$), 2.76 (ddd, 1H, $H^{3''}$, $^3J_{3''\text{-}2''}$ 10.5, $^3J_{3''\text{-}4''A}$ 10.5, $^3J_{3''\text{-}4''B}$ 5.2 Hz), 3.42 (d, 1H, $H^{6''B}$, $^2J_{6''B\text{-}6''A}$ 11.7 Hz), 3.55 (dd, 1H, $H^{1''B}$, $^2J_{1'''B\text{-}1'''A}$ 11.7 Hz), 3.72 (m, 3H, $H^{6''A}$, $H^{1'''A}$, $H^{2''}$), 3.92-4.05 (m, 4H, $-OCH_2-CH_2O-$). ^{13}C NMR ($CDCl_3$), δ: 20.37 ($C^{4'}$), 25.26 ($C^{5'}$), 35.29 ($C^{4''}$), 37.38 ($C^{3''}$), 40.52 ($C^{3'}$), 63.28 ($C^{1'''}$), 64.72 ($-OCH_2-$), 65.00 ($-OCH_2-$), 70.97 ($C^{6''}$), 78.26 ($C^{2''}$), 103.95 ($C^{5''}$), 110.40 ($C^{1'}$), 174.01 ($C^{2'}$), 207.96 (C=O). Mass spectrum, *m/z*: 284.13 $[MH]^+$. Found, %: C 59.09, H 7.11. $C_{14}H_{20}O_6$. Calculation, %: C 59.14, H 7.09.

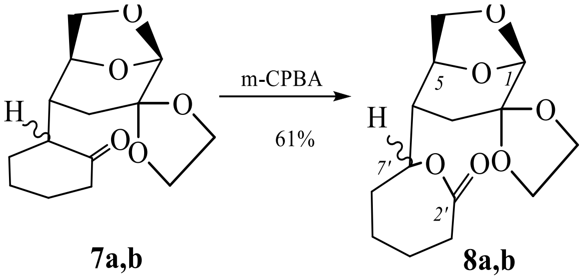
(4a*S*,12a*S*)-Octahydrospiro[1,3-dioxolane-2,3-pyrano[2,3-*c*]oxeceine]-5,10-dione (4) and 2-[hydroxy(8-hydroxymethyl-1,4,7-trioxaspiro[4.5]decan-9-yl)methylidene]-cyclopentanone (5b).



5a **4** **5b**

The title products were obtained from cyclopentenol **5a** (0.05 g, 0.018 mmol) analogously to Method A for compounds **2** and **1b**. Yields: **5a,b** (2:1, 0.01 g, 25%), and **4** (0.02 g, 45%). Physicochemical characteristics of compound **4** correspond to the literature [S9].

(1*R*,5*S*)-4-(7-Oxooxepan-2-yl)spiro[7,8-dioxabicyclo[3.2.1]octane-2,2'-[1,3]dioxolane] (8a,b)

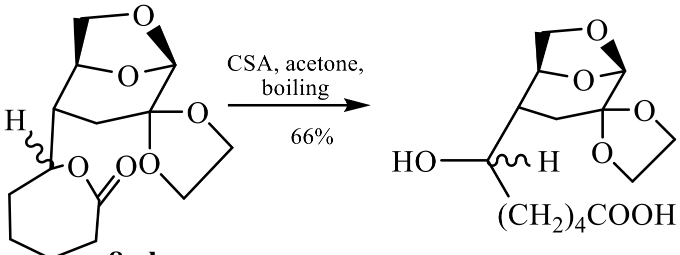


To a stirred solution of ketone **7a,b** (0.21 g, 0.78 mmol) in chloroform (7.0 ml), mCPBA (0.67 g, 3.9 mmol) was added in small portions. After 24 hours (TLC control), the mixture was treated with saturated $NaHCO_3$ solution (3×10 ml), the products were extracted with chloroform (3×20 ml), the combined organic layers were dried over $CaCl_2$, the solvent was evaporated, and the residue was chromatographed on SiO_2 to afford 0.14 g (88%) of lactone **8a,b**. Colourless crystals, m.p. 88°C. R_f 0.23 (petroleum ether-EtOAc, 1:1). $[\alpha]_D^{20}$ –62.5° (*c* 1.0, $CHCl_3$).

β-Isomer: ^{1}H NMR ($CDCl_3$), δ, m.d.: 1.54 (m, 1H, H$^{6'B}$), 1.66 (m, 2H, H$^{5'B}$, H$^{4'B}$), 1.83 (dd, 1H, H^{4}, $^3J_{4\text{-}3}$ 6.4, $^3J_{4\text{-}7'}$ 9.8 Hz), 1.94 (dd,1H, H^{3B}, $^2J_{3B\text{-}3A}$ 15.0, $^3J_{3B\text{-}4}$ 9.8 Hz), 1.96 (m, 2H, H$^{5'A}$, H$^{4'A}$), 2.18 (d, 1H, H^{3B}, $^2J_{3A\text{-}3B}$ 15.0 Hz), 2.20 (m, 1H, H$^{6'A}$), 2.65 (m, 1H, H$^{3'B}$), 2.76 (m,1H, H$^{3'A}$), 3.90-3.97 (m, 4H, CH_2O), 3.91 (d, 1H, H^{6B}, $^2J_{6B\text{-}6A}$ 7.6 Hz), 3.95 (dd, 1H, H^{6A}, $^2J_{6A\text{-}6B}$ 7.6, $^3J_{6A\text{-}5}$ 4.9 Hz), 4.51 (d, 1H, H^{5}, $^3J_{5\text{-}6A}$ 4.9 Hz), 4.76 (dd, 1H, H$^{7'}$, $^3J_{7'\text{-}4}$ 9.8, $^3J_{7'\text{-}6'B}$ 9.8 Hz), 5.0 (s, 1H, H^{1}). ^{13}C NMR ($CDCl_3$), δ: 22.50 (C$^{5'}$), 26.76 (C$^{3'}$), 27.24 (C$^{4'}$), 31.39 (C$^{6'}$), 34.09 (C^{3}), 42.71 (C^{4}), 64.88 (CH_2O), 65.33 (CH_2O), 67.93 (C^{6}), 73.13 (C^{5}), 78.27 (C$^{7'}$), 101.63 (C^{1}), 104.57 (C^{2}), 175.38 (C=O).

α-Isomer: ^{1}H NMR ($CDCl_3$), δ, m.d.: 1.49 (m, 1H, H$^{6'B}$), 1.60 (d, 1H, H^{3B}, $^2J_{3B\text{-}3A}$ 15.0 Hz), 1.64 (m, 2H, H$^{5'B}$, H$^{4'B}$), 1.83 (ddd,1H, H^{4}, $^3J_{4\text{-}7}$ 9.8, $^3J_{4\text{-}3A}$ 7.2, $^3J_{4\text{-}5}$ 1.9 Hz), 1.97 (m, 2H, H$^{5'A}$, H$^{4'A}$), 2.11 (m,1H, H$^{6'A}$), 2.16 (dd, 1H, H^{3A}, $^2J_{3A\text{-}3B}$ 15.0, $^3J_{3A\text{-}4}$ 7.2 Hz), 2.66 (m,1H, H$^{3'B}$), 2.77 (m,1H, H$^{3'A}$), 3.88 (m, 2H, H^{6A}, H^{6B}), 3.92-4.0 (m,4H, CH_2O), 4.77 (dd, 1H, H$^{7'}$, $^3J_{7'\text{-}4}$ 9.8, $^3J_{7'\text{-}6'B}$ 9.8 Hz), 4.91 (ddd, 1H, H^{5}, $^3J_{5\text{-}6A}$ 4.6, $^3J_{5\text{-}4}$ 1.9, $^3J_{5\text{-}6B}$ 1.9 Hz), 5.0 (s,1H, H^{1}). ^{13}C NMR ($CDCl_3$), δ: 22.86 (C$^{5'}$), 27.65 (C$^{4'}$), 28.25 (C$^{3'}$), 31.88 (C$^{6'}$), 34.42 (C^{3}), 42.54 (C^{4}), 64.91 (CH_2O), 65.48 (CH_2O), 67.67 (C^{6}), 72.27 (C^{5}), 78.35 (C$^{7'}$), 100.75 (C^{1}), 104.39 (C^{2}), 175.67 (C=O). Mass spectrum, *m/z*: 284.30 [*M*H]$^+$. Found, %: C 59.10, H 7.05. $C_{14}H_{20}O_6$. Calculation, %: C 59.14, H 7.09.

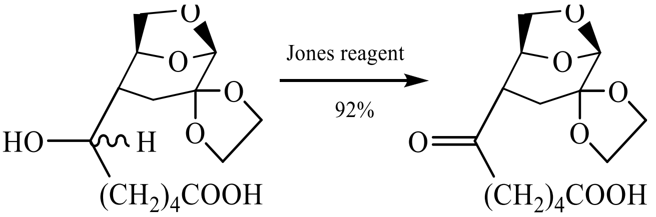
6-Hydroxy-6-{(1*S*,2*R*,5*R*)-spiro[7,8-dioxabicyclo[3.2.1]octane-2,2'-[1,3]dioxolan]-2-yl}hexanoic acid (9a,b).



8a,b **9a,b**

To a solution of compound **8a,b** (0.12 g, 0.40 mmol) in acetone (4.0 ml), 10-camphorsulfonic acid (0.03 g, 0.01 mmol) was added, and this boiled for 10 h (TLC control). The solvent was distilled off, the residue was chromatographed on SiO_2. Yield 0.08 g (66%). Colorless crystals, mp. 86°C. R_f 0.38 (EtOAc). ^{1}H NMR (CD_3OD), δ, m.d.: 1.38-1.71 (m, 4H, H$^{4'A}$, H$^{4'B}$, H$^{5'A}$, H$^{5'B}$) [1.37-1.72 (m, 4H, H$^{4'A}$, H$^{4'B}$, H$^{5'A}$, H$^{5'B}$)][1], 1.79 (m, 1H, H^4) [1.80 (m, 1H, H^4)], 1.86 (dd, 1H, H^{3B}, $^2J_{3B\text{-}3A}$ 14.5, $^3J_{3B\text{-}4}$ 6.8 Hz) [2.01 (dd, 1H, H^{3B}, $^2J_{3B\text{-}3A}$ 14.5, $^3J_{3B\text{-}4}$ 6.8 Hz)], 2.22 (d,1H, H^{3A}, $^2J_{3A\text{-}3B}$ 14.5 Hz) [2.21 (d,1H, H^{3A}, $^2J_{3A\text{-}3B}$ 14.5 Hz)], 2.31 (m, 4H, H$^{2'A}$, H$^{2'B}$, H$^{3'A}$, H$^{3'B}$) [2.32 (m, 4H, H$^{2'A}$, H$^{2'B}$, H$^{3'A}$, H$^{3'B}$)], 3.79 (dd,1H, H^{6A}, $^2J_{6A\text{-}6B}$ 7.5, $^3J_{6A\text{-}5}$ 5.0 Hz) [3.78 (dd, 1H, H^{6A}, $^2J_{6A\text{-}6B}$ 7.5, $^3J_{6A\text{-}5}$ 5.0 Hz)], 3.93 (d,1H, H^{6B}, $^2J_{6B\text{-}6A}$ 7.5 Hz) [3.97 (d, 1H, H^{6B}, $^2J_{6B\text{-}6A}$ 7.5 Hz)], 3.85-4.06 (m, 4H, CH_2O) [3.84-4.02 (m, 4H, CH_2O)], 4.52 (d, 1H, H^5, $^3J_{5\text{-}6A}$ 5.0 Hz) [4.55 (d, 1H, H^5, $^3J_{5\text{-}6A}$ 5.0 Hz)], 4.85 (m,1H, H$^{6'}$) [4.85 (m, 1H, H$^{6'}$)], 4.97 (s, 1H, H^1) [4.99 (s, 1H, H^1)]. ^{13}C NMR (CD_3OD), δ: 26.43 [26.06] (C$^{5'}$), 26.51 [26.24] (C$^{4'}$), 28.42 [29.42] (C^3), 35.32 [35.20] (C$^{3'}$), 35.75 [35.54] (C$^{2'}$), 45.98 [45.97] (C^4), 65.96 [66.14] (CH_2O), 66.70 [66.72] (CH_2O), 69.40 [69.02] (C^6), 71.02 [70.33] (C$^{6'}$), 75.30 [74.62] (C^5), 102.74 [102.46] (C^1), 106.32 [106.33] (C^2), 177.98 [177.97] (C=O). Mass spectrum, *m/z*: 302.31 [*M*H]$^+$. Found, %: C 55.59, H 7.30. $C_{14}H_{22}O_7$. Calculation, %: C 55.62, H 7.33.

6-Oxo-{(1*R*,5*S*)-spiro[7,8-dioxabicyclo[3.2.1]octane-2,2'-[1.3]dioxolan]-4-yl}hexanoic acid (10).



9a,b **10**

To a solution of hydroxy acid **9a,b** (0.08 g, 0.03 mmol) in acetone (5.0 ml), Jones reagent (0.3 ml) was added dropwise, and this was stirred for 15 minutes (TLC control). Isopropanol (1.0 ml) was then added, the solution was filtered and evaporated, the residue was chromatographed on SiO_2. Yield of oxo acid **10** was 0.07 g (92%), colourless crystals, m.p.113°C. R_f 0.24 (petroleum ether - EtOAc, 1:1). ^{1}H NMR ($CDCl_3$), δ, m.d.: 1.65 (m, 4H, H$^{4'A}$, H$^{4'B}$, H$^{3'A}$, H$^{3'B}$), 2.21 (dd, 1H, H^{3B}, $^2J_{3B\text{-}3A}$ 14.6, $^3J_{3B\text{-}4}$ 7.9 Hz), 2.34 (m, 2H, H^{3A}, H^4), 2.70 (m, 4H, H$^{2'A}$, H$^{2'B}$, H$^{5'A}$, H$^{5'B}$), 3.87-4.05 (m, 6H, H^{6A}, H^{6B}, CH_2O), 4.98 (c, 1H, H^1), 5.02 (m, 1H, H^5). ^{13}C NMR ($CDCl_3$), δ: 22.61 (C$^{3'}$), 23.90 (C$^{4'}$), 27.68 (C^3), 33.64 (C$^{2'}$), 39.48 (C$^{5'}$), 50.02 (C^4), 65.06 (CH_2O), 65.18 (CH_2O), 67.46

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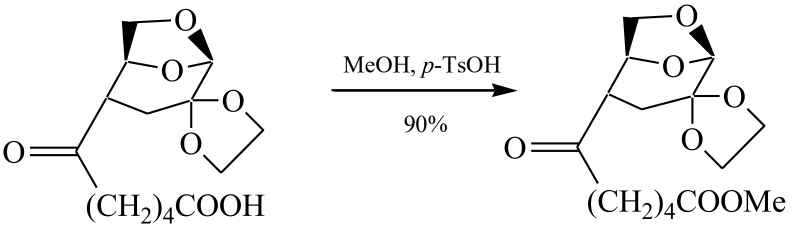
[1] In square brackets, the signals of minor diastereomer are given.

(C^{6}), 72.21 (C^{5}), 101.02 (C^{1}), 103.97 (C^{2}), 179.14 (C=O), 207.81 (C=O). Mass spectrum, *m/z*: 300.30 [*M*H]$^{+}$. Found, %: C 55.95, H 6.68. $C_{14}H_{20}O_{7}$. Calculation, %: C 55.99, H 6.71.

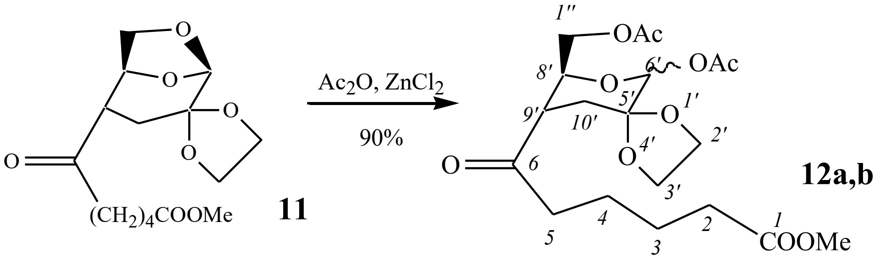
Methyl 6-oxo-6-{(1*R*,5*S*)-spiro[7,8-dioxabicyclo[3.2.1]octane-2,2'-[1,3]dioxolan]-4-yl}hexanoate (11).



10 **11**

Catalytic amounts of TsOH were added to a solution of carboxylic acid **10** (0.02 g, 0.07 mmol) in MeOH (2.0 ml), and this was stirred for 6 hours (TLC control). The solvent was evaporated, and the residue was chromatographed with in SiO_2. Yield 0.023 g (90%). Colourless oil. $[\alpha]_D^{20}$ –142° (*c* 0.7, $CHCl_3$). ^{1}H NMR ($CDCl_3$), δ, m.d.: 1.64 (m, 4H, H$^{4'A}$, H$^{4'B}$, H$^{3'A}$, H$^{3'B}$), 2.21 (dd, 1H, H^{3B}, $^{2}J_{3B\text{-}3A}$ 14.7, $^{3}J_{3B\text{-}4}$ 8.0 Hz), 2.31 (m, 3H, H^{4}, H$^{5'A}$, H$^{5'B}$), 2.42 (d, 1H, H^{3A}, $^{2}J_{3A\text{-}3B}$ 14.7 Hz), 2.68 (m, 2H, H$^{2'A}$, H$^{2'B}$), 3.68 (s, 3H, OCH_3), 3.81-4.02 (m, 6H, H^{6A}, H^{6B}, CH_2O), 4.99 (s, 1H, H^{1}), 5.05 (m, 1H, H^{5}). ^{13}C NMR ($CDCl_3$), δ: 22.63 (C$^{3'}$), 24.08 (C$^{4'}$), 27.54 (C^{3}), 33.59 (C$^{2'}$), 39.40 (C$^{5'}$), 49.32 (C^{4}), 51.29 (OCH_3), 64.93 (CH_2O), 65.05 (CH_2O), 67.33 (C^{6}), 72.09 (C^{5}), 100.91 (C^{1}), 103.85 (C^{2}), 173.69 (C=O), 207.57 (C=O).

Methyl 6-[(6*RS*,8*S*,9*S*)-6-acetoxy-8-acetoxymethyl-1,4,7-trioxaspiro[4.5]dec-9-yl]-6-oxohexanoate 12a,b.



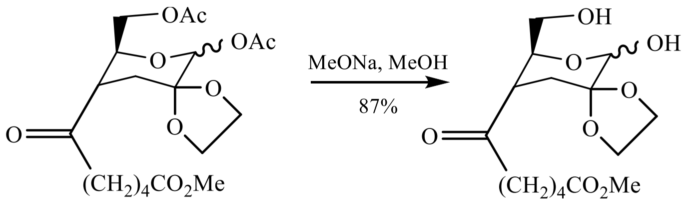
11

12a,b

To a solution of compound **11** (1.5 g, 4.70 mmol) in Ac_2O (20.0 ml), $ZnCl_2$ (0.65 g, 4.70 mmol) was added with stirring and cooling to 0°C, and stirring was continued until the disappearance of the starting material (TLC control). The mixture was poured into saturated ice-cold aqueous $NaHCO_3$ solution (30.0 ml), the products were extracted with EtOAc (3×25.0 ml), dried with $MgSO_4$. The solvent was distilled off, and the residue was chromatographed on SiO_2. Yield 1.79 g (90%), 3:1 mixture of α:β epimers, colourless oil. R_f 0.3 (EtOAc). ^{1}H NMR ($CDCl_3$), δ, m.d.: 1.55 (m, 4H, H^{4A}, H^{4B}, H^{3A}, H^{3B}) [1.56 (m, 4H, H^{4A}, H^{4B}, H^{3A}, H^{3B})], 1.84 (t, 1H, H$^{10'B}$, $^{2}J_{10'B\text{-}10'A}$ 13.0, $^{3}J_{10'B\text{-}9'}$ 13.0 Hz) [2.61 (dd, 1H, H$^{10'B}$, $^{2}J_{10'B\text{-}10'A}$ 16.7, $^{3}J_{10'B\text{-}9'}$ 7.8 Hz)], 1.99 (dd, 1H, H$^{10'A}$, $^{2}J_{10'A\text{-}10'B}$ 13.0, $^{3}J_{10'A\text{-}9'}$ 4.2 Hz) [2.77 (dt, 1H, H$^{10'A}$, $^{2}J_{10'A\text{-}10'B}$ 16.7, $^{3}J_{10'A\text{-}9'}$ 1.8, $^{3}J_{10'A\text{-}8'}$ 1.8, Hz)], 2.00 (s, 3H, CH_3) [2.01 (s, 3H, CH_3)], 2.10 (s, 3H, CH_3) [2.09 (s, 3H, CH_3)], 2.28 (m, 2H, H^{2A}, H^{2B}) [2.29 (m, 2H, H^{2A}, H^{2B})], 2.40 (m, 1H, H^{5B}) [2.42 (m, 1H, H^{5B})], 2.50 (m, 1H, H^{5A}) [2.53 (m, 1H, H^{5A})], 2.97 (ddd, 1H, H$^{9'}$, $^{3}J_{9'\text{-}10'B}$ 13.0, $^{3}J_{9'\text{-}8'}$ 10.1, $^{3}J_{9'\text{-}10'A}$ 4.2 Hz) [3.07 (td, 1H, H$^{9'}$, $^{3}J_{9'\text{-}10'B}$ 7.8, $^{3}J_{9'\text{-}8'}$ 1.8, $^{3}J_{9'\text{-}10'A}$ 1.8 Hz)], 3.61 (s, 3H, OCH_3) [3.62 (s, 3H, OCH_3)], 3.90-4.15 (m, 4H, CH_2O) [3.91-4.16 (m, 4H, CH_2O)], 3.99 (dd, 1H, H$^{1''B}$, $^{2}J_{1''B\text{-}1''A}$ 7.4, $^{3}J_{1''B\text{-}8'}$ 5.5 Hz) [4.28 (dd,1H,

$H^{1''B}$, $^2J_{1''B\text{-}1''A}$ 12.1, $^3J_{1''B\text{-}8'}$ 3.5 Hz)], 4.15 (d, 1H, $H^{1''A}$, $^2J_{1''A\text{-}1''B}$ 7.4 Hz) [4.54 (dd, 1H, $H^{1''A}$, $^2J_{1''A\text{-}1''B}$ 12.1, $^3J_{1''A\text{-}8'}$ 7.2 Hz)], 5.00 (m, 1H, $H^{8'}$) [4.11 (m, 1H, $H^{8'}$)], 5.59 (s,1H, $H^{6'}$) [5.83 (s, 1H, $H^{6'}$)]. ^{13}C NMR ($CDCl_3$), δ: 20.75 [20.87] (CH_3), 20.10 [21.06] (CH_3), 22.50 [22.94] (C^3), 24.19 [24.27] (C^4), 33.66 [33.76] (C^2), 36.19 [31.86] ($C^{10'}$), 42.09 [39.78] (C^5), 48.05 [52.83] ($C^{9'}$), 51.54 [51.50] (OCH_3), 64.04 [65.01] ($C^{1''}$), 65.94 [65.50] (CH_2O), 66.57 [65.64] (CH_2O), 74.40 [73.58] ($C^{8'}$), 93.42 [90.70] ($C^{6'}$), 103.20 [102.82] ($C^{5'}$), 168.84 [168.97] (C=O), 170.60 [170.93] (C=O), 173.71 [173.76] (C=O), 209.05 [207.55] (C=O).

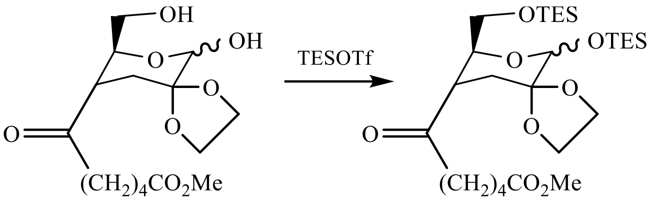
Methyl 6-[(6*RS*,8*S*,9*S*)-6-hydroxy-8-hydroxymethyl-1,4,7-trioxaspiro[4.5]-dec-9-yl]-6-oxohexanoate 13a,b.



12a,b **13a,b**

Acetates **12a,b** (0.20 g, 0.48 mmol) were dissolved in MeOH (2.0 ml), and MeONa (0.04 g, 0.96 mmol) was added. After completion the reaction (TLC), AcOH was added to pH = 7, MeOH was evaporated, the residue was extracted with EtOAc (3x5.0 ml), dried with $MgSO_4$, the solvent was distilled off, and the residue was chromatographed. Yield 0.14 g (87%), 1:0.8 mixture of α:β epimers, colourless oil, R_f 0.15 (EtOAc). ^{1}H NMR (CD_3OD), δ, m.d.: 1.54 (m, 4H, H^{4A}, H^{4B}, H^{3A}, H^{3B}) [1.55 (m, 4H, H^{4A}, H^{4B}, H^{3A}, H^{3B})], 1.72 (dd, 1H, $H^{10'A}$, $^2J_{10'A\text{-}10'B}$ 13.0, $^3J_{10'A\text{-}9'}$ 2.4 Hz) [1.93 (dd, 1H, $H^{10'A}$, $^2J_{10'A\text{-}10'B}$ 13.2, $^3J_{10'A\text{-}9'}$ 2.0 Hz)], 2.14 (t, 1H, $H^{10'B}$, $^2J_{10'B\text{-}10'A}$ 13.0, $^3J_{10'B\text{-}9'}$ 13.0 Hz) [1.81 (t, 1H, $H^{10'B}$, $^2J_{10'B\text{-}10'A}$ 13.2, $^3J_{10'B\text{-}9'}$ 13.0 Hz)], 2.28 (m, 2H, H^{2A}, H^{2B}) [2.29 (m, 2H, H^{2A}, H^{2B})], 2.48 (m, 2H, H^{5A}, H^{5B}) [2.49 (m, 2H, H^{5A}, H^{5B})], 3.01 (ddd, 1H, $H^{9'}$, $^3J_{9'\text{-}10'B}$ 13.0, $^3J_{9'\text{-}8'}$ 10.4, $^3J_{9'\text{-}10'A}$ 2.4 Hz) [3.02 (ddd, 1H, $H^{9'}$, $^3J_{9'\text{-}10'B}$ 13.0, $^3J_{9'\text{-}8'}$ 10.4, $^3J_{9'\text{-}10'A}$ 2.4 Hz)], 3.50 (dd, 1H, $H^{1''B}$, $^2J_{1''B\text{-}1''A}$ 10.5, $^3J_{1''B\text{-}8'}$ 6.2 Hz) [3.49 (dd, 1H, $H^{1''B}$, $^2J_{1''B\text{-}1''A}$ 10.5, $^3J_{1''B\text{-}8'}$ 6.2 Hz)], 3.58 (dd, 1H, $H^{1''A}$, $^2J_{1''A\text{-}1''B}$ 10.5, $^3J_{1''A\text{-}8'}$ 4.9 Hz) [3.59 (dd, 1H, $H^{1''A}$, $^2J_{1''A\text{-}1''B}$ 10.5, $^3J_{1''A\text{-}8'}$ 4.9 Hz)], 3.63 (s, 3H, OCH_3) [3.62 (s, 3H, OCH_3)], 3.96-4.18 (m, 4H, CH_2O) [3.97-4.17 (m, 4H, CH_2O)], 4.25 (m, 1H, $H^{8'}$) [3.79 (m, 1H, $H^{8'}$)], 4.80 (s, 1H, $H^{6'}$) [4.69 (s, 1H, $H^{6'}$)]. ^{13}C NMR ($CDCl_3$), δ: 22.61 [22.94] (C^3), 24.20 [24.21] (C^4), 30.73 [35.61] ($C^{10'}$), 33.73 [33.74] (C^2), 42.05 [42.68] (C^5), 47.49 [47.96] ($C^{9'}$), 51.55 [51.56] (OCH_3), 63.24 [62.82] ($C^{1''}$), 65.31 [66.26] (CH_2O), 65.75 [68.12] (CH_2O), 69.40 [77.07] ($C^{8'}$), 96.17 [92.18] ($C^{6'}$), 104.63 [104.09] ($C^{5'}$), 173.88 [173.89] (C=O), 210.42 [210.47] (C=O). Mass spectrum, *m/z*: 333 [*M*H]$^+$. Found, %: C 54.19, H 7.23. $C_{15}H_{24}O_8$. Calculation, %: C 54.21, H 7.28.

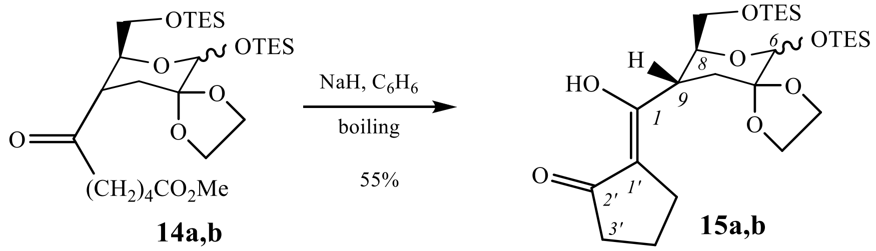
Methyl 6-oxo-6-[(6*RS*,8*S*,9*S*)-6-triethylsilyloxy-8-triethylsilyloxymethyl-1,4,7-trioxaspiro[4.5]dec-9-yl]hexanoate 14a,b



13a,b **14a,b**

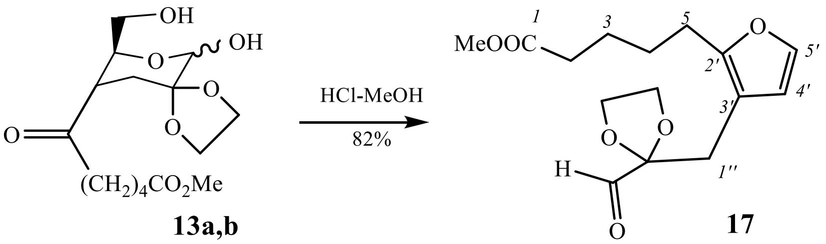
To a solution of diols **13a,b** (0.4 g, 1.16 mmol) in CH_2Cl_2 (13 ml), Et_3SiOTf (1.05 g, 4.0 mmol) and Et_3N (1.2 ml, 8.4 mmol) were added at 0°C, and this was stirred up to the disappearance of the starting material (TLC control). The reaction mass was then treated with a saturated aqueous NH_4Cl solution, extracted with CH_2Cl_2 (3×15 ml). The extract was dried with $MgSO_4$, the solvent was distilled off, and the residue was chromatographed on SiO_2. Yield 0.35 g (54%), silyl ethers **14a,b** in the ratio 1:0.4. Colourless oil. R_f 0.3 (petroleum ether-EtOAc, 2:1). ^1H NMR ($CDCl_3$), δ, m.d.: 0.50 (quartet, 2H, OTES) [0.51 (quartet, 2H, OTES)], 0.91 (t, 3H, OTES) [0.92 (t, 3H, OTES)], 1.56 (m, 4H, H^{4A}, H^{4B}, H^{3A}, H^{3B}) [1.57 (m, 4H, H^{4A}, H^{4B}, H^{3A}, H^{3B})], 1.81 (dd, 1H, $H^{10'A}$, $^{2}J_{10'A-10'B}$ 13.0, $^{3}J_{10'A-9'}$ 5.0 Hz) [1.55 (dd, 1H, $H^{10'A}$, $^{2}J_{10'A-10'B}$ 13.0, $^{3}J_{10'A-9'}$ 5.0 Hz)], 1.86 (t, 1H, $H^{10'B}$, $^{2}J_{10'B-10'A}$ 13.0, $^{3}J_{10'B-9'}$ 13.0 Hz) [2.18 (t, 1H, $H^{10'B}$, $^{2}J_{10'B-10'A}$ 13.0, $^{3}J_{10'B-9'}$ 13.0 Hz)], 2.30 (m, 2H, H^{2A}, H^{2B}) [2.29 (m, 2H, H^{2A}, H^{2B})], 2.49 (m, 2H, H^{5A}, H^{5B}) [2.50 (m, 2H, H^{5A}, H^{5B})], 2.93 (ddd, 1H, $H^{9'}$, $^{3}J_{9'-10'B}$ 13.0, $^{3}J_{9'-8'}$ 10.0, $^{3}J_{9'-10'A}$ 5.0 Hz) [2.94 (ddd, 1H, $H^{9'}$, $^{3}J_{9'-10'B}$ 13.0, $^{3}J_{9'-8'}$ 10.0, $^{3}J_{9'-10'A}$ 5.0 Hz)], 3.57 (dd, 1H, $H^{1''B}$, $^{2}J_{1''B-1''A}$ 10.5, $^{3}J_{1''B-8'}$ 5.0 Hz) [3.51 (dd, 1H, $H^{1''B}$, $^{2}J_{1''B-1''A}$ 10.5, $^{3}J_{1''B-8'}$ 5.0 Hz)], 3.68 (dd, 1H, $H^{1''A}$, $^{2}J_{1''A-1''B}$ 10.5, $^{3}J_{1''A-8'}$ 5.0 Hz) [3.67 (dd, 1H, $H^{1''A}$, $^{2}J_{1''A-1''B}$ 10.5, $^{3}J_{1''A-8'}$ 5.0 Hz)], 3.64 (s, 3H, OCH_3) [3.63 (s, 3H, OCH_3)], 3.77 (td, 1H, $H^{8'}$, $^{3}J_{8'-9'}$ 10.0, $^{3}J_{8'-1''A}$ 5.0, $^{3}J_{8'-1''B}$ 5.0 Hz) [4.15 (td, 1H, $H^{8'}$, $^{3}J_{8'-9'}$ 10.0, $^{3}J_{8'-1''A}$ 5.0, $^{3}J_{8'-1''B}$ 5.0 Hz], 3.94-4.20 (m, 4H, CH_2O) [3.90-4.20 (m, 4H, CH_2O)], 4.64 (s, 1H, $H^{6'}$) [4.66 (s, 1H, $H^{6'}$)]. ^{13}C NMR ($CDCl_3$), δ: 6.36 [6.48] (OTES), 6.71 [6.62] (OTES), 22.58 [22.64] (C^3), 24.39 [24.44] (C^4), 33.82 [33.84] (C^2), 36.50 [30.69] ($C^{10'}$), 43.05 [42.08] (C^5), 49.96 [49.74] ($C^{9'}$), 51.40 [51.41] (OCH_3), 64.29 [64.49] ($C^{1''}$), 65.74 [65.01] (CH_2O), 67.01 [65.09] (CH_2O), 77.36 [69.29] ($C^{8'}$), 97.60 [92.29] ($C^{6'}$), 104.40 [105.23] ($C^{5'}$), 173.73 [173.78] (C=O), 209.70 [209.61] (C=O).

(6*RS*,8*S*,9*S*)-9-[1-Hydroxy-1-(2-oxocyclopentylidene)methyl]-6-triethylsilyloxy-8-triethylsilyloxymethyl-1,4,7-trioxaspiro[4.5]decane 15a,b



To a suspension of NaH (0.03 g, 1.10 mmol) in dry THF (2.0 ml), a THF (1.0 ml) solution of keto esters **14a,b** (0.20 g (0.36 mmol) was added, and this was boiled under inert atmosphere until the starting material disappeared (TLC control). The reaction mass was then treated with 1% HCl (aq, 2.0 ml), and the product was extracted with EtOAc (3×3.0 ml). The extract was dried with $MgSO_4$, the solvent was distilled off, and the residue was chromatographed on SiO_2. Yield 0.10 g (55%). Colorless oil, R_f 0.3 (petroleum ether-EtOAc, 2:1). ^{1}H NMR ($CDCl_3$), δ, m.d.: 0.52 (quartet, 2H, OTES) [0.53 (quartet, 2H, OTES)], 0.91 (t, 3H, OTES) [0.92 (t, 3H, OTES)], 1.78 (dd, 1H, H^{10A}, $^2J_{10A\text{-}10B}$ 13.1, $^3J_{10A\text{-}9}$ 4.0 Hz) [1.89 (dd, 1H, H^{10A}, $^2J_{10A\text{-}10B}$ 13.1, $^3J_{10A\text{-}9}$ 4.0 Hz)], 1.90 (m, 2H, $H^{4'A}$, $H^{4'B}$) [1.91 (m, 2H, $H^{4'A}$, $H^{4'B}$)], 2.12 (t, 1H, H^{10B}, $^2J_{10B\text{-}10A}$ 13.1, $^3J_{10B\text{-}9}$ 13.1 Hz), 2.40 (m, 2H, $H^{3'A}$, $H^{3'B}$) [2.31 (m, 2H, $H^{3'A}$, $H^{3'B}$)], 2.57 (m, 2H, $H^{5'A}$, $H^{5'B}$) [2.59 (m, 2H, $H^{5'A}$, $H^{5'B}$)], 2.69 (ddd, 1H, H^9, $^3J_{9\text{-}10A}$ 13.1, $^3J_{9\text{-}8}$10.1, $^3J_{9\text{-}10A}$ 4.0 Hz) [2.74 (ddd, 1H, H^9, $^3J_{9\text{-}10A}$ 13.1, $^3J_{9\text{-}8}$ 10.1, $^3J_{9\text{-}10A}$ 4.0 Hz)], 3.61 (dd, 1H, $H^{1'''B}$, $^2J_{1'''B\text{-}1'''A}$ 10.7, $^3J_{1'''B\text{-}8}$ 3.7 Hz) [3.63 (dd, 1H, $H^{1'''B}$, $^2J_{1'''B\text{-}1'''A}$ 10.7, $^3J_{1'''B\text{-}8}$ 3.7 Hz)], 3.67 (dd, 1H, $H^{1'''A}$, $^2J_{1'''A\text{-}1'''B}$ 10.7, $^3J_{1'''A\text{-}8}$ 6.0 Hz) [3.70 (dd, 1H, $H^{1'''A}$, $^2J_{1'''A\text{-}1'''B}$ 10.7, $^3J_{1'''A\text{-}8}$ 6.0 Hz)], 3.85 (ddd, 1H, H^8, $^3J_{8\text{-}9}$ 10.1, $^3J_{8\text{-}1'''B}$ 6.0, $^3J_{8\text{-}1'''A}$ 4.0 Hz) [3.68 (ddd, 1H, H^8, $^3J_{8\text{-}9}$ 10.1, $^3J_{8\text{-}1'''B}$ 6.0, $^3J_{8\text{-}1'''A}$ 4.0 Hz)], 3.97-4.24 (m, 4H, CH_2O) [3.95-4.23 (m, 4H, CH_2O)], 4.70 (s,1H, H^6) [4.68 (s, 1H, H^6)]. ^{13}C NMR ($CDCl_3$), δ: 6.39 [6.59] (OTES), 6.80 [6.71] (OTES), 20.35 [20.60] ($C^{4'}$), 25.44 [24.38] ($C^{5'}$), 36.23 [35.69] (C^{10}), 37.17 [38.96] ($C^{3'}$), 42.01 [38.89] (C^9), 63.95 [63.72] ($C^{1'''}$), 65.78 [65.07] (CH_2O), 67.11 [66.95] (CH_2O), 77.72 [78.27] (C^8), 97.95 [97.71] (C^6), 104.45 [105.23] (C^5), 110.06 [109.42] ($C^{1'}$), 176.17 [176.74] (C^1), 206.42 [206.57] (C=O). Mass spectrum, *m/z*: 529 $[MH]^+$. Found, %: C 59.09, H 9.20. $C_{26}H_{48}O_7Si_2$. Calculation, %: C 59.05, H 9.15.

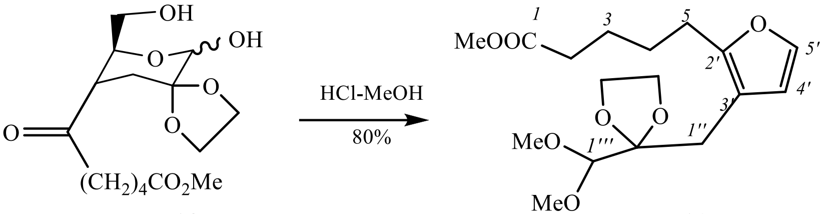
Methyl 5-{3-[(2-formyl-1,3-dioxolan-2-yl)methyl]furan-2-yl}pentanoate 17.



Method A. Diols **13a,b** (0.10 g, 0.30 mmol) were dissolved in MeOH (3.0 ml), trimethyl formate (0.03 ml, 0.30 mmol) and TsOH (0.05 g, 0.03 mmol) were added. The solution was stirred for 5 hours at room temperature (TLC control). The reaction mass was then neutralized with saturated $NaHCO_3$ (aq, pH ~ 6), the products were extracted with EtOAc (3×2.0 ml). The extract was dried with $MgSO_4$, the solvent was distilled off, and the residue was chromatographed on SiO_2. Yield 0.08 g (90%).

Method B. Diols **13a,b** (0.15 g, 0.45 mmol) were treated with 8% methanolic solution of HCl (1.2 ml), and this was stirred for 12 hours at room temperature (TLC control). The reaction mass was neutralized with saturated $NaHCO_3$ (aq., pH ~ 6), the products were extracted with EtOAc (3×2.0 ml). The extract was dried with $MgSO_4$, the solvent was distilled off, and the residue was chromatographed on SiO_2. Yield 0.11 g (82%). Colourless oil, R_f 0.4 (EtOAc). ^{1}H NMR ($CDCl_3$), δ, m.d.: 1.62 (m, 4H, H^{3A}, H^{3B}, H^{4A}, H^{4B}), 2.27 (m, 2H, H^{2A}, H^{2B}), 2.57 (m, 2H, H^{5A}, H^{5B}), 2.80 (s, 2H, $H^{1''A}$, $H^{1''B}$), 3.63 (s, 3H, OMe), 3.70 - 4.01 (m, 4H, CH_2O), 6.28 (d, 1H, $H^{4'}$, $^3J_{4'\text{-}5'}$ 1.2 Hz), 7.20 (d, 1H, $H^{5'}$, $^3J_{5'\text{-}4'}$ 1.2 Hz), 9.48 c (1H, O=C-H). ^{13}C NMR ($CDCl_3$), δ: 24.39 (C^5), 25.45 (C^3), 27.80 (C^4), 29.45 ($C^{1''}$), 33.78 (C^2), 51.49 (OMe), 66.21 (CH_2O), 66.92 (CH_2O), 92.11 ($C^{2''}$), 98.94 ($C^{3'}$), 113.23 ($C^{4'}$), 139.80 ($C^{5'}$), 153.06 ($C^{2'}$), 174.05 (C=O), 196.25 (O=C-H). Mass spectrum, *m/z*: 297 $[MH]^+$. Found, %: C 60.77, H 6.77. $C_{15}H_{20}O_6$. Calculation, %: C 60.80, H 6.80.

Methyl 5-{3-[(2-dimethoxymethyl-1,3-dioxolan-2-yl)methyl]furan-2-yl}pentanoate 18.



13a,b **18**

Diols **13a,b** (0.20 g, 0.58 mmol) were treated with 17% methanolic solution of HCl (1.5 ml), and this was stirred for 12 hours at room temperature (TLC control). The reaction mass was then neutralized with saturated $NaHCO_3$ (aq., pH ~ 6), the reaction products were extracted with EtOAc (3×2.0 ml). The extract was dried with $MgSO_4$, the solvent was distilled off, and the residue was chromatographed on SiO_2. Yeild 0.16 g (80%). Colourless oil, R_f 0.5 (EtOAc). ^1H NMR ($CDCl_3$), δ, m.d.: 1.60 (m, 4H, H^{3A}, H^{3B}, H^{4A}, H^{4B}), 2.30 (m, 2H, H^{2A}, H^{2B}), 2.58 (m, 2H, H^{5A}, H^{5B}), 2.70 (c, 2H, $H^{1''A}$, $H^{1''B}$), 3.48 (c, 6H, OMe), 3.63 (c, 3H, OMe), 3.68 (m, 2H, CH_2O), 3.92 (m, 2H, CH_2O), 4.07 (c, 1H, $H^{1'''}$), 6.29 (d, 1H, $H^{4'}$, $^3J_{4'\text{-}5'}$ 1.8 Hz), 7.19 (d, 1H, $H^{5'}$, $^3J_{5'\text{-}4'}$ 1.8 Hz). ^{13}C NMR ($CDCl_3$), δ: 24.51 (C^5), 25.47 (C^3), 27.86 (C^4), 29.09 ($C^{1''}$), 33.85 (C^2), 51.44 (OMe), 57.07 (OMe), 57.08 (OMe), 66.10 (CH_2O), 66.11 (CH_2O), 106.70 ($C^{1'''}$), 109.92 ($C^{2''}$), 112.73 ($C^{3'}$), 113.36 ($C^{4'}$), 139.62 ($C^{5'}$), 153.03 ($C^{2'}$), 174.07 (C=O). Mass spectrum, *m/z*: 343 $[MH]^+$. Found, %: C 59.59, H 7.60. $C_{17}H_{26}O_7$. Calculation, %: C 59.64, H 7. 65.

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