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# A simple approach to 5,5′-bis(1,3-dioxolan-4-ones) of tartaric acids

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**Abstract**—An easy access to acetals of aldehydes and tartaric acid is described. A series of bis(1,3-dioxolan-4-ones) was isolated with a high degree of 2,5-*cis*-selectivity. Transformations were performed with both (*S*,*S*)- and (*R*,*R*)-tartaric acid.

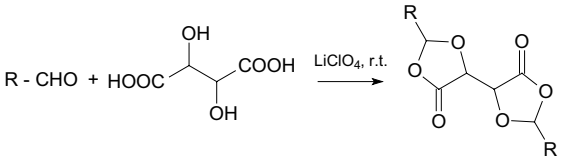

## 1. Introduction

1,3-Dioxolan-4-ones of α-hydroxy acids are important compounds in the pool of chiral building blocks.[1] Since the pioneering work of Seebach et al.,[2] many reported synthetic methods have been based on these chiral building blocks. For example, Kellog et al. described an aldol addition resulting from reacting the acetals of aldehydes and mandelic acid with enol ethers in the presence of boron trifluoride etherate,[3] with chiral ethers of aldol adducts and mandelic acid being isolated. By oxidative cleavage of these ethers, access to chiral aldol products can be achieved. Diastereoselective protonation of dioxolanones of mandelic acid and aldehydes has also been reported.[4] In conjugate addition of the anions of dioxolanones derived from α-hydroxy acids to α,β-unsaturated esters, the corresponding γ-oxo-esters were obtained with up to 86% ee's after flash vacuum pyrolysis.[5] Furthermore, highly diastereoselective Michael additions of lithium enolates of chiral 1,3-dioxolan-4-one of mandelic acid with α,β-unsaturated carbonyl compounds have been described.[6] This method allows, 1,4-dicarbonyl compounds to be obtained with high enantiomeric excess.

This easy access to chiral 1,3-dioxolanones (via simple dehydration methods of aldehydes and α-hydroxy acids) attracts synthetic chemists time and time again. However in all the reactions and examples described in the literature, mostly dioxolanones of mandelic acid, lactic acid, malic acid or phenyl lactic acid have been described. To the best of our knowledge there have been only two examples of 1,3-dioxolan-4-ones derived from tartaric acid reported in the literature. Moreover, these examples only describe the formation of bis(1,3-dioxolanones) of the activated aldehydes chloral[7] and bromal.[8] Also the authors had to use large amounts of concentrated sulfuric acid (up to 10 equiv) in reactions of chloral with tartaric acid.[9]

## 2. Results and discussion

During our investigations of alkylations of aldehydes in the presence of $LiClO_4$ and tartaric acid, we saw that from time to time small amounts of acetals of aldehydes and tartaric acid were produced.[10] In order to analyze and compare these substances we tried to synthesize acetals from aldehydes and tartaric acid [substituted bis(1,3-dioxolan)-4-ones]. We found it was not possible to synthesize these dioxolanones by the classical procedure used for synthesis of dioxolanones of mandelic acid.[4] Upon further investigation, we developed a simple and useful method for the synthesis of dioxolanones of tartaric acid. Herein we present the first synthesis of dioxolanones of unactivated, aliphatic aldehydes and tartaric acid (Scheme 1).




**Scheme 1.**

Acetals of tartaric acid and aldehydes were easily prepared by the reaction of tartaric acid and aldehydes in

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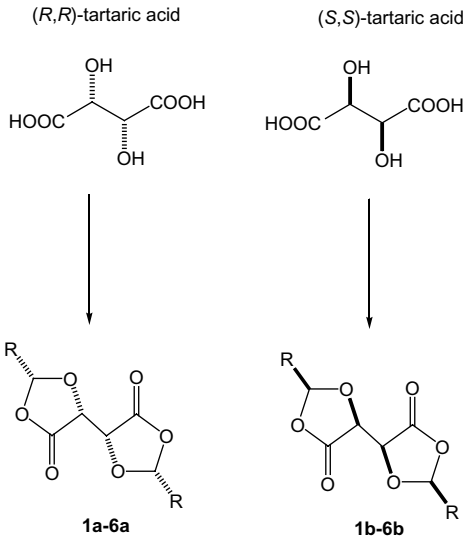
**Table 1**




| Entry R | Compound (yield %)[a] | |
|---|---|---|
| Me | **1a** 43 | **1b** 46 |
| Et | **2a** 36 | **2b** 33 |
| *n*-Pr | **3a** 33 | **3b** 37 |
| *i*-Pr | **4a** 28 | **4b** 23 |
| *t*-Bu | **5a** 45 | **5b** 48 |
| *c*-Hex | **6a** 29 | **6b** 31 |

[a] Yields are not optimized.

the presence of equimolar amounts of dry $LiClO_4$. Several other dry metal salts and the usual Lewis acids were used but without any success. The same highly selective behaviour of $LiClO_4$ was observed during alkylations of aldehydes in the presence of $LiClO_4$ and tartaric acid.[10]

The reactions were performed neat at room temperature for 24 h with only the bis(1,3-dioxolan-4-ones) being obtained in good yields (Table 1). Other acetals, as shown in Figure 1 could not be detected under these conditions.

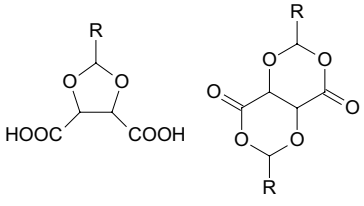



**Figure 1.**

The structures of the 1,3-dioxolan-4-ones were determined unambiguously by X-ray structure analysis of compounds **1b** and **5a** (Fig. 2).[11]

For comparison, both forms of chiral tartaric acid were used in these reactions with the results shown in Table 1.

The dioxolanones were formed with a high degree of 2,2′,5,5′-*cis*-stereoselectivity (**1a**–**6a** in the (*R*,*R*)-series and **1b**–**6b** in the (*S*,*S*)-series, Table 1). The dioxolanones of tartaric acid and acetaldehyde were only isolated in the 2,2′,5,5′-*cis*-configuration (**1a** and **1b**, Table 1). Using higher aldehydes in these reactions, the 2,5-*cis*,2′,5′-*trans*-configurated dioxolanones **7**–**11** were isolated in small amounts as well and described as byproducts; propionaldehyde ⇒ **7**, butyraldehyde ⇒ **8**, isobutyraldehyde ⇒ **9**, pivalaldehyde ⇒ **10** and cyclohexancarboxaldehyde ⇒ **11**.

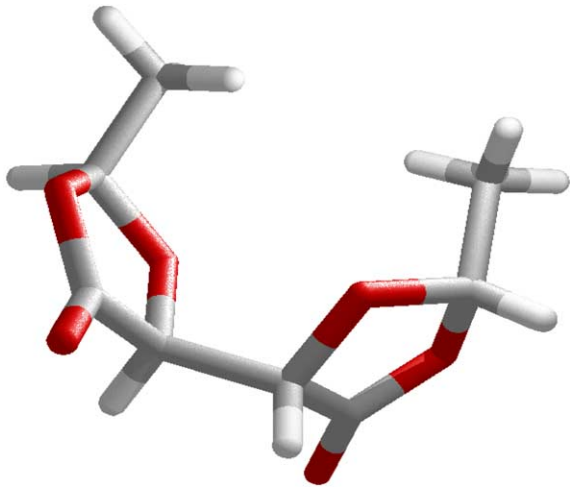


**Figure 2.** X-ray structure of dimethyl-bis(1,3-dioxolan-4-one) **1b**.

The synthesis of dioxolanones of aromatic aldehydes and tartaric acid failed under these reaction conditions. No products were detected on using benzaldehyde. We do not believe that steric reasons are alone responsible for the failure of this reaction, if one considers the results with isobutyraldehyde, pivalaldehyde or cyclohexancarboxaldehyde (Table 1, entries 4–6). Electronic effects may be responsible for this observation. In order to check this assumption we tested several aromatic aldehydes with completely different electrophilic behaviour, *p*-tolualdehyde and *p*-nitrobenzaldehyde, but in both cases no reaction occurred, even with heating. There is no explanation for this phenomenon at this time.

Further investigations of this reaction are currently in progress.

## 3. Experimental

All reactions were performed using oven dried glassware under an atmosphere of dry argon. Aldehydes were distilled before use. Purification of products was accomplished using flash chromatography. $LiClO_4$ was dried at 120 °C in vacuo for 10 h.[12]

$^1H$ NMR and $^{13}C$ NMR spectra were recorded at 300 and 75 MHz in $CDCl_3$, respectively, using a AC-300 spectrometer. Chemical shifts are given in ppm. Thin

layer chromatography was performed out using Merck silica gel 60 $F_{254}$ TLC plates.

The dioxolanones of the (*S*,*S*)-series **1b**–**6b** were described by the rotatory power only, because there are the same physical constants ($^1$H NMR, $^{13}$C NMR and MS) as the acetales in the (*R*,*R*)-series **1a**–**6a**.

### 3.1. General procedure

10 mmol $LiClO_4$, 10 mmol aldehyde and 5 mmol tartaric acid were stirred at room temperature in diethyl ether (ca. 10 mL). The reaction was monitored by thin-layer chromatography. At the end of the reaction (mostly 24 h) the resulting mixture was extracted by diethyl ether and saturated aq $NaHCO_3$-solution. The organic layers were separated, dried ($Na_2SO_4$) and the solvent removed in vacuo. The residue was then purified by column chromatography.

**3.1.1. (2*R*,2′*R*,5*R*,5′*R*)-2,2′-Dimethyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 1a.** Colourless solid; mp: 118–120 °C (petrolether/ethylacetate); $[\alpha]_D = +96$ (*c* 1, ethylacetate); IR (neat) $\nu_{max} = 2999, 2956, 1788, 1400, 1363, 1182, 1140, 1105, 1077, 1041\,cm^{-1}$; $^1$H NMR ($CDCl_3$) $\delta = 5.73$ (2H, q, $J = 4.9$, $H_2$, $H_{2'}$), 4.69 (2H, s, $H_5$, $H_{5'}$), 1.53 (6H, d, $J = 4.9$ Hz, $2\times CH_3$); $^{13}$C NMR ($CDCl_3$) $\delta = 169.1$ ($C_4$, $C_{4'}$), 103.2 ($C_2$, $C_{2'}$), 73.6 ($C_5$, $C_{5'}$), 20.6 ($2\times CH_3$); HRMS: Calcd for $C_8H_{10}O_6$ 202.0477. Found: 202.0459.

**3.1.2. (2*S*,2′*S*,5*S*,5′*S*)-2,2′-Dimethyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 1b.** Colourless solid; mp: 118–120 °C (petrolether/ethylacetate); $[\alpha]_D = -91$ (*c* 1, $CH_2Cl_2$).

**3.1.3. (2*R*,2′*R*,5*R*,5′*R*)-2,2′-Diethyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 2a.** Colourless oil; $[\alpha]_D = +35$ (*c* 0.64, $CH_2Cl_2$); IR ($CH_2Cl_2$) $\nu_{max} = 2978, 2939, 2886, 1804, 1466, 1406, 1364, 1326, 1272, 1264, 1218, 1113, 1014\,cm^{-1}$; $^1$H NMR ($CDCl_3$) $\delta = 5.52$ (2H, t, $J = 4.9$ Hz, $H_2$, $H_{2'}$), 4.69 (2H, s, $H_5$, $H_{5'}$), 1.80 (4H, dq, $J = 4.9$, 7.5 Hz, $2\times CH_2$), 0.95 (6H, t, $J = 7.5$ Hz, $2\times CH_3$); $^{13}$C NMR ($CDCl_3$) $\delta = 169.0$ ($C_4$, $C_{4'}$), 106.2 ($C_2$, $C_{2'}$), 73.2 ($C_5$, $C_{5'}$), 27.2 ($2\times CH_2$), 6.7 ($2\times CH_3$). HRMS: Calcd for $C_{10}H_{14}O_6$ 230.0790. Found: 230.0791.

**3.1.4. (2*S*,2′*S*,5*S*,5′*S*)-2,2′-Diethyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 2b.** Colourless oil; $[\alpha]_D = -32$ (*c* 0.84, $CH_2Cl_2$).

**3.1.5. (2*S*,2′*R*,5*S*,5′*S*)-2,2′-Diethyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 7.** Colourless oil; $[\alpha]_D = -65$ (*c* 1.04, $CH_2Cl_2$); IR (neat) $\nu_{max} = 2978, 2940, 2887, 1794, 1465, 1401, 1365, 1202, 1136, 1113, 1082, 1007\,cm^{-1}$; $^1$H NMR ($CDCl_3$) $\delta = 5.66$ (1H, dt, $J = 1.1$, 4.5 Hz, $H_2$ or $H_{2'}$), 5.50 (1H, dt, $J = 1.1$, 4.9 Hz, $H_2$ or $H_{2'}$), 4.72 (1H, t, $J = 1.1$, 1.5 Hz, $H_5$ or $H_{5'}$), 4.66 (1H, dt, $J = 1.1$, 1.5 Hz, $H_5$ or $H_{5'}$), 1.79 (2H, dq, $J = 4.9$, 7.5 Hz, $CH_2$), 1.61 (2H, dq, $J = 4.5$, 7.5 Hz, $CH_2$), 0.96 (3H, t, $J = 7.5$ Hz, $CH_3$), 0.93 (3H, t, $J = 7.5$ Hz, $CH_3$); $^{13}$C NMR ($CDCl_3$, 75 MHz): $\delta = 169.4$ ($C_4$ or $C_{4'}$), 169.0 ($C_4$ or $C_{4'}$), 108.3 ($C_2$ or $C_{2'}$), 106.0 ($C_2$ or $C_{2'}$), 75.0 ($C_5$ or $C_{5'}$), 72.9 ($C_5$ or $C_{5'}$), 27.9 ($CH_2$), 27.3 ($CH_2$), 6.8 ($CH_3$), 6.4 ($CH_3$). HRMS: Calcd for $C_{10}H_{14}O_6Na$ 253.0681. Found: 253.0680.

**3.1.6. (2*R*,2′*R*,5*R*,5′*R*)-2,2′-Dipropyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 3a.** Colourless oil; $[\alpha]_D = +61$ (*c* 1, $CH_2Cl_2$); IR (neat) $\nu_{max} = 2963, 2877, 1792, 1466, 1404, 1368, 1324, 1254, 1178, 1147, 1102, 1046\,cm^{-1}$; $^1$H NMR ($CDCl_3$) $\delta = 5.56$ (2H, dd, $J = 4.9$, 5.3, $H_2$, $H_{2'}$), 4.67 (2H, s, $H_5$, $H_{5'}$), 1.76 (4H, dddd, $J = 1.9$, 4.9, 5.3, 7.2 Hz, $2\times CH_2$), 1.45 (4H, ddt, $J = 1.9$, 7.2, 7.5 Hz, $2\times CH_2$), 0.91 (6H, t, $J = 7.5$ Hz, $2\times CH_3$); $^{13}$C NMR ($CDCl_3$) $\delta = 169.1$ ($C_4$, $C_{4'}$), 105.6 ($C_2$, $C_{2'}$), 73.3 ($C_5$, $C_{5'}$), 36.2, 16.3 ($4\times CH_2$), 13.7 ($2\times CH_3$); HRMS: Calcd for $C_{12}H_{18}O_6$ 258.1103. Found: 258.1104.

**3.1.7. (2*S*,2′*S*,5*S*,5′*S*)-2,2′-Dipropyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 3b.** Colourless oil; $[\alpha]_D = -55$ (*c* 1, $CH_2Cl_2$).

**3.1.8. (2*S*,2′*R*,5*R*,5′*R*)-2,2′-Dipropyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 8.** Colourless oil; $[\alpha]_D = +33$ (*c* 0.04, $CH_2Cl_2$); IR (neat) $\nu_{max} = 2969, 2925, 2852, 1795, 1737, 1483, 1411, 1260, 1216, 1190, 1093, 1054\,cm^{-1}$; $^1$H NMR ($CDCl_3$) $\delta = 5.68$ (1H, dt, $J = 1.1$, 4.9 Hz, $H_2$ or $H_{2'}$), 5.53 (1H, dt, $J = 1.1$, 4.9 Hz, $H_2$ or $H_{2'}$), 4.70 (1H, dd, $J = 1.1$, 1.5 Hz, $H_5$ or $H_{5'}$), 4.64 (1H, dd, $J = 1.1$, 1.5 Hz, $H_5$ or $H_{5'}$), 1.80–1.69 (4H, m, $2\times CH_2$), 1.51–1.34 (4H, m, $2\times CH_2$), 0.92 (3H, t, $J = 7.5$ Hz, $CH_3$), 0.91 (3H, t, $J = 7.5$ Hz, $CH_3$); $^{13}$C NMR ($CDCl_3$) $\delta = 169.4$ ($C_4$ or $C_{4'}$), 168.9 ($C_4$ or $C_{4'}$), 107.7 ($C_2$ or $C_{2'}$), 105.3 ($C_2$ or $C_{2'}$), 75.0 ($C_5$ or $C_{5'}$), 72.8 ($C_5$ or $C_{5'}$), 36.8, 36.1, 16.3, 15.9 ($4\times CH_2$), 13.7, 13.6 ($2\times CH_3$). HRMS: Calcd for $C_{12}H_{18}O_6Na$ 281.1001. Found: 281.0995.

**3.1.9. (2*R*,2′*R*,5*R*,5′*R*)-2,2′-Diisopropyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 4a.** Colourless oil; $[\alpha]_D = +43$ (*c* 0.6, $CH_2Cl_2$); IR (neat) $\nu_{max} = 3403, 2968, 2929, 1800, 1472, 1399, 1371, 1210, 1127, 1115, 1083, 1053\,cm^{-1}$; $^1$H NMR ($CDCl_3$) $\delta = 5.48$ (2H, d, $J = 5.7$ Hz, $H_2$, $H_{2'}$), 4.90 (2H, s, $H_5$, $H_{5'}$), 2.16 (2H, dsp, $J = 5.7$, 6.8 Hz, $2\times CH$), 1.19 (12H, t, $J = 6.84$ Hz, $4\times CH_3$); $^{13}$C NMR ($CDCl_3$) $\delta = 169.2$ ($C_4$, $C_{4'}$), 108.8 ($C_2$, $C_{2'}$), 73.3 ($C_5$, $C_{5'}$), 32.4 ($2\times CH$), 16.1, 15.9 ($4\times CH_3$); HRMS: Calcd for $C_{12}H_{18}O_6$ 258.1103. Found: 258.1104.

**3.1.10. (2*S*,2′*S*,5*S*,5′*S*)-2,2′-Diisopropyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 4b.** Colourless oil; $[\alpha]_D = -50$ (*c* 0.8, $CH_2Cl_2$).

**3.1.11. (2*S*,2′*R*,5*R*,5′*R*)-2,2′-Diisopropyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 9.** Colourless oil; $[\alpha]_D = +38$ (*c* 0.16,

$CH_2Cl_2$); IR (neat) $\nu_{max} = 2962, 2932, 2876, 1796, 1727, 1464, 1367, 1202, 1119, 973\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 5.59$ (1H, dd, $J = 1.5, 4.5$, $H_2$ or $H_{2'}$), 5.54 (1H, dd, $J = 1.1, 4.9$ Hz, $H_2$ or $H_{2'}$), 4.85 (1H, t, $J = 1.5$ Hz, $H_5$ or $H_{5'}$), 4.77 (1H, dd, $J = 1.1, 1.5$ Hz, $H_5$ or $H_{5'}$), 2.14–2.00 (2H, m, 2×CH), 1.10 (6H, dd, $J = 2.3, 6.8$ Hz, 2×$CH_3$), 1.06 (6H, d, $J = 7.8$ Hz, 2×$CH_3$); $^{13}C$ NMR ($CDCl_3$) $\delta = 169.5$ ($C_4$ or $C_{4'}$), 169.0 ($C_4$ or $C_{4'}$), 110.8 ($C_2$ or $C_{2'}$), 108.5 ($C_2$ or $C_{2'}$), 75.0 ($C_5$ or $C_{5'}$), 73.0 ($C_5$ or $C_{5'}$), 32.9, 32.2 (2×CH), 15.8, 15.8, 15.4, 15.3 (4×$CH_3$). HRMS: Calcd for $C_{12}H_{18}O_6Na$ 281.1001. Found: 281.0996.

**3.1.12. (2*R*,2′*R*,5*R*,5′*R*)-2,2′-Ditertbutyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 5a.** Pale solid; mp: 136–138 °C (hexane/ethylacetate); $[\alpha]_D = +18$ (*c* 1, $Et_2O$); IR (neat) $\nu_{max} = 2961, 2928, 1801, 1728, 1483, 1463, 1409, 1366, 1285, 1203, 1135, 1090, 1073\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 5.17$ (2H, s, $H_2$, $H_{2'}$), 4.66 (2H, s, $H_5$, $H_{5'}$), 0.93 (18H, s, 6×$CH_3$); $^{13}C$ NMR ($CDCl_3$) $\delta = 169.5$ ($C_4$, $C_{4'}$), 109.7 ($C_2$, $C_{2'}$), 72.8 ($C_5$, $C_{5'}$), 34.1 (2×Cq), 23.3 (6×$CH_3$); HRMS: Calcd for $C_{14}H_{22}O_6$ 286.1416. Found: 286.1417.

**3.1.13. (2*S*,2′*S*,5*S*,5′*S*)-2,2′-Ditertbutyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 5b.** Pale solid; $[\alpha]_D = -13$ (*c* 1, $CH_2Cl_2$).

**3.1.14. (2*S*,2′*R*,5*R*,5′*R*)-2,2′-Ditertbutyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 10.** Colourless oil; $[\alpha]_D = +49$ (*c* 0.26, $CH_2Cl_2$); IR (neat) $\nu_{max} = 2960, 2911, 1797, 1482, 1411, 1366, 1325, 1286, 1214, 1197, 1139, 1056, 1038\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 5.38$ (1H, d, $J = 1.5$, $H_2$ or $H_{2'}$), 5.21 (1H, d, $J = 1.5$, $H_2$ or $H_{2'}$), 4.80 (1H, t, $J = 1.5$, $H_4$ or $H_{4'}$), 4.67 (1H, t, $J = 1.5$, $H_4$ or $H_{4'}$), 1.00 (9H, s, 3×$CH_3$), 0.96 (9H, s, 3×$CH_3$); $^{13}C$ NMR ($CDCl_3$) $\delta = 169.7, 169.2$ ($C_4$, $C_{4'}$), 112.7, 109.3 ($C_2$, $C_{2'}$), 75.2, 73.1 ($C_5$, $C_{5'}$), 35.6, 34.3 (2×Cq), 23.4, 23.0 (6×$CH_3$). HRMS: Calcd for $C_{14}H_{22}O_6Na$ 309.1314. Found: 309.1308.

**3.1.15. (2*R*,2′*R*,5*R*,5′*R*)-2,2′-Dicyclohexyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 6a.** Pale solid; mp: 155–158 °C (hexane/ethylacetate); $[\alpha]_D = +43$ (*c* 1, $CH_2Cl_2$); IR (neat) $\nu_{max} = 2923, 2855, 1796, 1452, 1319, 1251, 1219, 1201, 1145, 1068, 1039\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 5.25$ (2H, d, $J = 6.0$ Hz, $H_2$, $H_{2'}$), 4.65 (2H, s, $H_5$, $H_{5'}$), 1.85–1.58 (12H, m), 1.26–0.95 (10H, m); $^{13}C$ NMR ($CDCl_3$) $\delta = 169.2$ ($C_4$, $C_{4'}$), 108.1 ($C_2$, $C_{2'}$), 73.1 ($C_5$, $C_{5'}$), 41.9 (2×CH), 26.5, 26.1, 26.0, 25.2, 25.1 (10×$CH_2$); HRMS: Calcd for $C_{18}H_{26}O_6$ 338.1729. Found: 338.1729.

**3.1.16. (2*S*,2′*S*,5*S*,5′*S*)-2,2′-Dicyclohexyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 6b.** Pale solid; $[\alpha]_D = -46$ (*c* 1, $CH_2Cl_2$).

**3.1.17. (2*S*,2′*R*,5*R*,5′*R*)-2,2′-Dicyclohexyl-5,5′-bis(1,3-dioxolane-4,4′-dione) 11.** Colourless oil; $[\alpha]_D = +24$ (*c* 0.26, $CH_2Cl_2$); IR (neat) $\nu_{max} = 2926, 2854, 1800, 1727, 1451, 1288, 1241, 1206, 1127, 1070, 1018\,cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta = 5.42$ (1H, dd, $J = 1.5, 4.5$ Hz, $H_2$ or $H_{2'}$), 5.24 (1H, dd, $J = 1.5, 5.3$ Hz, $H_2$ or $H_{2'}$), 4.68 (1H, dd, $J = 1.1, 1.5$ Hz, $H_5$ or $H_{5'}$), 4.61 (1H, dd, $J = 1.1, 1.5$ Hz, $H_5$ or $H_{5'}$), 1.81–1.55 (12H, m), 1.28–0.95 (10H, m); $^{13}C$ NMR ($CDCl_3$) $\delta = 169.5$ ($C_4$ or $C_{4'}$), 169.1 ($C_4$ or $C_{4'}$), 110.2 ($C_2$ or $C_{2'}$), 107.9 ($C_2$ or $C_{2'}$), 74.8 ($C_5$ or $C_{5'}$), 72.9 ($C_5$ or $C_{5'}$), 42.3, 41.7 (2×CH), 27.0, 26.5, 26.2, 26.2, 26.0, 25.7, 25.6, 25.3, 25.2, 25.1 (10×$CH_2$). HRMS: Calcd for $C_{18}H_{26}O_6Na$ 361.1627. Found: 361.1626.

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## References and notes

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11. The crystal structures have been deposited at the Cambridge Crystallographic Data Centre and have been allocated the deposition numbers CCDC 219 740 for **1b** and CCDC 219 741 for **5a**.
12. For comments of safety see Armarego, W. L. F.; Perrin, D. D. *Purification of Laboratory Chemicals*; Butterworth-Heinemann: Oxford, 1997.