Supporting information:

A Sulfitylation–Oxidation Protocol for the Preparation of Sulfates

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General methods

Standard syringe techniques were applied for the transfer of dry solvents and air- or moisture sensitive reagents. Compounds on TLC plates were visualized with ammonium molybdate solution, potassium permanganate, or UV light. Flash chromatography was carried out using silica gel (0.035–0.070 mm, ca 6 nm pore diameter). Solvents were distilled from appropriate drying agents prior to use. Unless stated otherwise, all chemicals were purchased and used as such.

General chlorosulfite synthesis procedure

Thionyl chloride is put in a three-necked flask (neat) connected to two sequential traps with 10M aqueous NaOH. A little nitrogen pressure is applied so the gas bubbles out of the last trap at moderate speed, to make sure the hydrochloric acid gas byproduct leaves the reaction mixture quickly enough. At 0°C the alcohol is slowly added dropwise over

the course of several hours. After addition the solution is stirred one or two days at room temperature, after which a vacuum distillation is performed.

(*R,S*)-2,3-*O*-isopropylidene-glycerol 1- ethyl sulfate (3b**)**

Sulfite diester **2b** (159 mg, 0.71 mmol), sodium periodate (309 mg, 1.44 mmol) and ruthenium trichloride (cat.) were reacted as described in the general sulfite oxidation procedure,

yielding **3b** as a colorless oil (162 mg, 95 %). IR ν_{max} film: (cm⁻¹) 1376, 1199, 922. ¹H NMR (CDCl₃, 400 MHz, ppm): δ 4.38 (q, 2H, J = 7.1Hz), 4.36 (m, 1H), 4.22 (d, 2H, J = 5.4Hz), 4.11 (dd, 1H, J = 6.5, 8.8Hz), 3.83 (dd, 1H, J = 5.4, 8.8Hz), 1.44 (q, 3H, J = 0.7Hz), 1.44 (t, 3H, J = 7.1Hz), 1.37 (q, 3H, J = 0.7Hz). ¹³C NMR (CDCl₃, 300 MHz, ppm) δ : 110.2, 72.7, 71.9, 70.0, 65.8, 26.4, 25.1, 14.5. HRMS (EI) m/z calcd for C₈H₁₇O₆S (M+H)⁺: 241.0746, found: 241.0748.

(*R,S*)-2,3-*O*-isopropylidene-glycerol 1- isopropyl sulfate (3c**)**

Sulfite diester **2c** (407 mg, 1.71 mmol), sodium periodate (735 mg, 3.44 mmol) and ruthenium trichloride (cat.) were reacted as

described in the general sulfite oxidation procedure, yielding **3c** as a colorless oil (381 mg, 88%). IR ν_{max} film: (cm⁻¹) 1381, 1195, 918. ¹H NMR (CDCl₃, 400 MHz, ppm): δ 4.94 (qq, 1H, J = 6.3, 6.3Hz), 4.38 (dq, 1H, J = 5.4, 6.4Hz), 4.19 (dd, 2H, J = 0.7, 5.4Hz), 4.10 (dd, 1H, J = 6.5, 8.8Hz), 3.84 (dd, 1H, J = 5.4, 8.8Hz), 1.45 (d, 6H, J = 6.3Hz), 1.43 (bq, 3H, J = 0.6Hz), 1.36 (bq, 3H, J = 0.6Hz). ¹³C NMR (CDCl₃, 300 MHz,

ppm) δ : 110.2, 81.1, 72.8, 71.6, 65.9, 26.6, 25.1, 22.4. HRMS (EI) m/z calcd for $C_9H_{19}O_6S$ ($M+H$)⁺: 255,0902, found: 255,0900.

(*R,S*)-2,3-*O*-isopropylidene-glycerol 1-sulfate, sodium salt (4**)**

Sulfate diester **3b** (402 mg, 1.67 mmol) and sodium iodide (252 mg, 1.68 mmol) were reacted as described in the general sulfate deprotection procedure, yielding **4** as a white solid (390 mg, 100%). IR ν_{max} film: (cm⁻¹) 1225, 1074, 996. ¹H NMR (D₂O, 400 MHz, ppm): δ 4.52 (dtd, 1H, J = 3.8, 5.9, 6.6Hz), 4.21 (dd, 1H, J = 6.8, 8.9Hz), 4.16 (dd, 1H, J = 3.8, 10.9Hz), 4.07 (dd, 1H, J = 5.9, 10.9Hz), 3.92 (dd, 1H, J = 5.9, 8.9Hz), 1.49 (s, 3H), 1.42 (s, 3H). ¹³C NMR (H₂O, 300 MHz, ppm): δ 109.9, 73.2, 67.8, 65.0, 25.0, 23.7. HRMS (ESI) m/z calcd for $C_6H_{11}O_6S$ ($M-Na$)⁻: 211.0276, found: 211.0279.

Isopropyl chlorosulfite (5b**)**

Thionyl chloride (120 g, 1.01 mol) and isopropyl alcohol (50.0 g, 0.832 mol) were reacted as described in the general chlorosulfite synthesis procedure. Distillation was performed at 50 °C (40 mbar), yielding **5b** as a colorless liquid (~78%). ¹H NMR (CDCl₃, 400 MHz, ppm): δ 5.50 (qq, 1H, J = 6.3, 6.3Hz), 1.52 (d, 3H, J = 6.3Hz), 1.47 (d, 3H, J = 6.3Hz). ¹³C NMR (CDCl₃, 300 MHz, ppm) δ : 76.8, 68.3, 24.0, 23.8, 23.2. Note: peaks of the rapidly formed degradation product isopropyl chloride are also present in the ¹³C spectrum.

Neopentyl chlorosulfite (5d)

Thionyl chloride (43.5g, 366 mmol) and neopentyl alcohol (26.9g, 305 mmol) were reacted as described in the general chlorosulfite synthesis procedure with exception of a vacuum distillation. Instead excess thionyl chloride was distilled off selectively at 40 °C (50 mbar) yielding **5d** as a colorless liquid (~95%). ^1H NMR (CDCl_3 , 400 MHz, ppm): δ 4.25 (d, 1H, $J = 9.4\text{Hz}$), 3.97 (d, 1H, $J = 9.4\text{Hz}$), 1.02 (s, 9H). ^{13}C NMR (CDCl_3 , 300 MHz, ppm): 76.0, 31.6, 26.4.

1,2:3,4-Di-*O*-isopropylidene- α -D-galactopyranose 6-sulfate, sodium salt (7c)

Sulfate diester **7b** (1255 mg, 3.41 mmol) and sodium iodide (512 mg, 3.42 mmol) were reacted as described in the general sulfate deprotection procedure, yielding **7c** as a white solid (1234 mg, 100%). $[\alpha]_D^{20}$ -40 (c 1.02; MeOH). IR ν_{max} film: (cm^{-1}) 1649, 1389, 1255, 1217, 1074, 999. ^1H NMR (D_2O , 400 MHz, ppm): δ 5.68 (d, 1H, $J = 4.9\text{Hz}$), 4.83 (dd, 1H, $J = 2.3, 7.9\text{Hz}$), 4.58 (dd, 1H, $J = 2.5, 5.0\text{Hz}$), 4.50 (app d, 1H, 7.9Hz), 4.30-4.21 (m, 2H), 4.12 (dd, 1H, $J = 7.7, 10.8\text{Hz}$), 1.60 (s, 3H), 1.50 (s, 3H), 1.42 (s, 6H). ^{13}C NMR (D_2O , 300 MHz, ppm) δ : 109.8, 109.6, 95.3, 69.9, 69.5, 69.4, 66.4, 66.1, 24.4, 24.3, 23.5, 22.7. HRMS (ESI) m/z calcd for $\text{C}_{12}\text{H}_{19}\text{O}_9\text{S}$ (M-Na) $^-$: 339.0750, found: 339.0781.

1-O-Benzyl-2-deoxy-2-acetamido-3,6-di-O-pivaloyl α-D-glucopyranose 4-sulfate, sodium salt (8c)

Sulfate diester **8b** (28.0 mg, 0.048 mmol) and sodium iodide (7.5 mg, 0.050 mmol) were reacted as described in the general sulfate deprotection procedure, yielding **8c** as a white solid (27.3 mg, 99%). $[\alpha]_D^{20} +48$ (c 0.18; MeOH). IR ν_{max} film: (cm⁻¹) 1717, 1644, 1488, 1449, 1280, 1155, 1074, 991, 610. ¹H NMR (CD₃OD, 400 MHz, ppm): δ 7.42-7.28 (m, 5H), 5.30 (dd, 1H, J = 9.1, 11.0Hz), 4.79 (d, 1H, J = 3.6Hz), 3.78 (d, 1H, J = 12.0Hz), 4.54 (d, 1H, J = 11.9Hz), 4.53 (dd, 1H, J = 2.1, 12.0Hz), 4.44 (dd, 1H, J = 2.1, 12.0Hz), 4.32-4.23 (m, 2H), 4.05 (ddd, 1H, J = 2.1, 6.5, 10.1Hz), 3.68 (s, 1H), 1.87 (s, 3H), 1.22 (s, 9H), 1.17 (s, 9H). ¹³C NMR (CD₃OD, 300 MHz, ppm) δ : 129.6, 129.5, 129.2, 97.2, 75.7, 72.2, 70.5, 70.4, 64.8, 53.1, 27.7, 27.7, 22.5. HRMS (ESI) m/z calcd for C₂₅H₃₆NO₁₁S (M-Na)⁻: 558.2009, found: 558.2050.

1-O-Methyl-α-D-glucopyranose 2,3,4,6-tetrasulfate, tetrasodium salt (9c)

Tetrasulfate diester **9b** (66.6 mg, 0.11 mmol) and sodium iodide (88.0 mg, 0.58 mmol) were reacted as described in the general sulfate deprotection procedure, yielding **9c** as a white solid (63.4 mg, 99%). $[\alpha]_D^{20} +133$ (c 0.12; H₂O). IR ν_{max} film: (cm⁻¹) 1644, 1233, 1074, 987. ¹H NMR (D₂O, 400 MHz, ppm): δ 5.21 (d, 1H, J = 3.6 Hz), 4.72-4.61 (m, 2H), 4.46 (ddd, 1H, J = 1.1, 3.7, 9.8Hz), 4.37-4.32 (m, 1H), 4.23-4.14 (m, 2H), 3.52 (d, 3H, 1.1Hz). ¹³C NMR (D₂O, 300 MHz, ppm) δ : 96.3, 75.3, 74.5, 73.8, 68.0, 66.9, 54.9. HRMS (ESI) m/z calcd for C₇H₁₀Na₃O₁₈S₄ (M-Na)⁻: 578.8443, found: 578.8494.

(*R,S*)-Cyclohexane-1,2-diol 1,2-disulfate, disodium salt (10c**)**

Disulfate diester **10b** (883 mg, 2.66 mmol) and sodium iodide (798 mg, 5.33 mmol) were reacted as described in the general sulfate deprotection procedure, yielding **10c** as a white solid (847 mg, 99%). IR ν_{max} film: (cm⁻¹) 1644, 1221, 1069, 1001. ¹H NMR (D₂O, 400 MHz, ppm): δ 4.62-4.57 (m, 2H), 2.07-1.97 (m, 2H), 1.79-1.62 (m, 4H), 1.49-1.39 (m, 2H). ¹³C NMR (D₂O, 300 MHz, ppm) δ : 77.4, 27.2, 20.4. HRMS (ESI) m/z calcd for C₆H₁₀NaO₈S₂ (M-Na)⁻: 296.972, found: 296.974.

1,2:5,6-Di-*O*-isopropylidene- α -D-glucofuranose 3-sulfate, sodium salt (11c**)**

Sulfate diester **11b** (101 mg, 0.27 mmol) and sodium iodide (42 mg, 0.28 mmol) were reacted as described in the general sulfate deprotection procedure, yielding **11c** as a white solid (98 mg, 99%). $[\alpha]_D^{20}$ -10 (c 0.45; MeOH). IR ν_{max} film: (cm⁻¹) 1649, 1376, 1243, 1225, 1014, 858. ¹H NMR (D₂O, 400 MHz, ppm): δ 6.12 (d, 1H, J = 3.7Hz), 5.01 (d, 1H, J = 3.7Hz), 4.87 (d, 1H, J = 2.5Hz), 4.58-4.53 (m, 2H), 4.21-4.15 (m, 1H), 4.11-4.05 (m, 1H), 1.56 (s, 3H), 1.49 (s, 3H), 1.41 (s, 3H), 1.40 (s, 3H). ¹³C NMR (D₂O, 300 MHz, ppm) δ : 112.8, 108.8, 104.3, 82.2, 79.4, 79.1, 72.4, 64.4, 25.2, 24.9, 24.8, 23.4. HRMS (ESI) m/z calcd for C₁₂H₁₉O₉S (M-Na)⁻: 339.0750, found: 339.0782.

3-Benzylglycerol 1,2-disulfate, disodium salt (**12c**)

Disulfate diester **12b** (225 mg, 0.56 mmol) and sodium iodide (220 mg, 1.47 mmol) were reacted as described in the general sulfate deprotection procedure, yielding **12c** as a white solid (217 mg, 100 %). IR ν_{max} film: (cm⁻¹) 1632, 1226, 1052, 1026, 936. ¹H NMR (D₂O, 400 MHz, ppm): δ 7.50-7.39 (m, 5H), 4.75 (dtt, 1H, J = 1.8, 4.8, 4.8Hz), 4.68 (ddd, 2H, J = 1.4, 11.6, 18.8Hz), 4.29 (dddd, 2H, J = 1.9, 4.8, 10.9, 20.2Hz), 3.86 (dddd, 2H, J = 1.6, 5.0, 11.6, 29.1Hz). ¹³C NMR (D₂O, 300 MHz, ppm) δ : 136.6, 128.1, 127.9, 127.7, 75.1, 72.9, 67.6, 66.2. HRMS (ESI) m/z calcd for C₁₀H₁₂O₉S₂ (M-Na)⁻: 362.9820, found: 362.9837.

3-(*tert*-Butyl diphenylsilyl)-glycerol 1,2-disulfate,

disodium salt (**13c**)

Disulfate diester **13b** (796 mg, 1.46 mmol) and sodium iodide (460 mg, 3.07 mmol) were reacted as described in the general sulfate deprotection procedure, yielding **13c** as a white solid (783mg, 100%). IR ν_{max} film: (cm⁻¹) 1640, 1372, 1216, 1203, 914. ¹H NMR (D₂O, 400 MHz, ppm): δ 7.84-7.79 (m, 4H), 7.59-7.48 (m, 6H), 4.60 (tt, 1H, J = 4.9, 4.9Hz), 4.32 (ddd, 2H, J = 4.7, 10.6, 22.7Hz), 3.99 (ddd, 2H, J = 5.0, 11.3, 17.9Hz), 1.09 (s, 9H). ¹³C NMR (D₂O, 300 MHz, ppm) δ : 135.2, 132.6, 129.8, 127.6, 127.4, 76.3, 65.7, 61.5, 25.6, 17.9. HRMS (ESI) m/z calcd for C₁₉H₂₄NaO₉S₂Si (M-Na)⁻: 511.0529, found: 511.0552.

Phenyl sulfate, sodium salt (14c)

Ethyl phenyl sulfate **14b** (59 mg, 0.29 mmol) was dissolved in dry methanol, to which sodium methoxide (33 mg, 0.61 mmol) was added. After stirring for 1 hour the solvent was evaporated. The residue was flash chromatographed (MeCN / H₂O), yielding **14c** as a white solid (56 mg, 100%). IR ν_{max} film: (cm⁻¹) 616, 603. ¹H NMR (D₂O, 300 MHz, ppm): δ 7.55-7.45 (m, 2H), 7.41-7.33 (m, 3H). ¹³C NMR (D₂O, 300 MHz, ppm) δ : 150.6, 129.4, 125.8, 121.0. HRMS (ESI) m/z calcd for C₆H₅O₄S (M-Na)⁻: 172.9909, found: 172.9910.

1,2-*O*-isopropylidene- α -D-glucufuranose 3-[(*R,S*)-ethyl sulfite] (15)

Sulfite **11a** (570 mg, 1.62 mmol) was dissolved in AcOH / H₂O 1 / 1, and heated to 50 °C for 3h. The solvents were evaporated, and the crude product was flash chromatographed (heptane / EtOAc 1 / 1) to yield **15** as a white solid (442 mg, 88 %; 2:3 mixture of diastereomers). IR ν_{max} film: (cm⁻¹) 3469, 1371, 1207, 1082, 1017. ¹H NMR (CDCl₃, 400 MHz, ppm): δ 5.93 (d, 1H, J = 3.7Hz), 5.15+4.93 (app d / td, 1H, J = 2.6Hz / J = 0.5, 2.9 Hz), 4.64+4.58 (d, 1H, J = 3.8Hz), 4.34-4.09 (m, 2H), 3.94-3.68 (m, 3H), 3.33+3.07 (d / br s, 1H, J = 4.7Hz), 1.52+1.51 (s, 3H), 1.41+1.38 (t, 3H, J = 7.1Hz), 1.33+1.32 (app d, 3H, J = 0.5Hz). ¹³C NMR (CDCl₃, 300 MHz, ppm) δ : 112.5+112.4, 105.0+105.0, 83.5, 79.2+79.1, 76.2+74.4, 68.2+67.9, 64.1+64.0, 60.8+59.5, 26.6, 26.2+26.2, 15.3+15.2. HRMS (ESI) m/z calcd for C₁₁H₂₁O₈S (M+H)⁺: 313.0957, found: 313.0950.

6-*O*-acetyl-1,2-*O*-isopropylidene- α -D-glucofuranose 3-[(*R,S*)-ethyl sulfite] (16a**)**

Sulfite **15** (20.8 mg, 0.076 mmol) and acetic anhydride (30.4 mg, 0.30 mmol) were dissolved in dry pyridine. The solvent was evaporated after 1h, and ethyl acetate was added to the reaction mixture. Subsequent washing with water and brine, drying with Na₂SO₄, and filtration yielded the crude product. This was flash chromatographed (heptane / ethyl acetate 3 / 1) to yield **16a** as a white solid (22.0 mg, 93%; 4:5 mixture of diastereomers). IR ν_{max} film: (cm⁻¹) 3490, 1743, 1216, 1086, 1017. ¹H NMR (CDCl₃, 400 MHz, ppm): δ 5.92+5.92 (d, 1H, J = 3.7Hz), 5.12+4.94 (d, 1H, J = 2.5Hz), 4.63+4.59 (d, 1H, J = 3.7Hz), 4.46+4.43 (t, 1H, J = 2.1Hz), 4.32-4.09 (m, 4H), 4.09-3.91 (m, 1H), 3.24+2.98 (d, 1H, J = 4.6Hz), 2.11+2.10 (s, 3H), 1.51+1.50 (s, 3H), 1.40+1.37 (t, 3H, 7.1Hz), 1.33+1.32 (s, 3H). ¹³C NMR (CDCl₃, 300 MHz, ppm) δ : 112.6+112.6, 105.2+105.1, 83.6, 78.9+78.8, 75.9, 74.2, 66.9+66.5, 66.4+66.2, 60.9+59.7, 26.7, 26.4+26.4, 20.9, 15.4+15.3. HRMS (ESI) m/z calcd for C₁₃H₂₃O₉S (M+H)⁺: 355.1063, found: 355.1071.

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 Frequency (MHz): (f1) 400.148
 Original Points Count: (f1) 23971
 Actual Points Count: (f1) 65536
 Acquisition Time (sec): (f1) 3.7440
 Spectral Width (ppm): (f1) 16.001
 Pulse Program: Unknown
 Temperature: 25
 Number of Scans: 8
 Acq. Date: Mar 5 2004

20 Feb 2006
 Document's Title:
 14600
 Frequency (MHz):
 (f1) 75.476
 Original Points Cot
 (f1) 65536
 Actual Points Cour
 (f1) 65536
 Acquisition Time (s)
 (f1) 3.4603
 Spectral Width (pp)
 (f1) 250.933
 Pulse Program:
 ZGDC
 Temperature:
 298
 Number of Scans:
 1024
 Acq. Date:
 Sun Feb 12 12:23:1

Date: 20 Feb 2006
 Document's Title: PROTON.fid
 Frequency (MHz): (f1) 400.148
 Original Points Count: (f1) 23971
 Actual Points Count: (f1) 65536
 Acquisition Time (se (f1) 3.7440
 Spectral Width (ppm, (f1) 16.001
 Pulse Program: Unknown
 Temperature: 29
 Number of Scans: 8
 Acq. Date: Jul 1 2003

Date: 20 Feb 2006
 Document's Title: 31501
 Frequency (MHz): (f1) 75.476
 Original Points Count: (f1) 32768
 Actual Points Count: (f1) 65536
 Acquisition Time (sec): (f1) 1.7302
 Spectral Width (ppm): (f1) 250.933
 Pulse Program: ZGDC
 Temperature: 298
 Number of Scans: 4096
 Acq. Date: Mon Feb 13 07:32:15 AM

Date: 20 Feb 2006
 Document's Title: OK.mrc
 Frequency (MHz): (f1) 400.148
 Original Points Count: (f1) 23971
 Actual Points Count: (f1) 65536
 Acquisition Time (sec): (f1) 3.740
 Spectral Width (ppm): (f1) 16.001
 Pulse Program: Unknown
 Temperature: 29
 Number of Scans: 32
 Acq. Date: Dec 9 2004

Date:
 20 Feb 2006
Document's Title:
 ok.mrc
Frequency (MHz):
 (f1) 75.476
Original Points Count:
 (f1) 32768
Actual Points Count:
 (f1) 65536
Acquisition Time (sec):
 (f1) 1.7302
Spectral Width (ppm):
 (f1) 250.933
Pulse Program:
 ZGDC
Temperature:
 300
Number of Scans:
 1024
Acq. Date:
 Fri Dec 10 06:52:45 AM

Document's Title:
OK.mrc

Frequency (MHz):
(f1) 400.148

Original Points Coun
(f1) 23971

Actual Points Count:
(f1) 65536

Acquisition Time (sec
(f1) 3.7440

Spectral Width (ppm)
(f1) 16.001

Pulse Program:
Unknown

Temperature:
29

Number of Scans:
8

Acq. Date:
Feb 3 2005

20 Feb 2006
 Document's Title:
 24000
 Frequency (MHz):
 (f1) 75.476
 Original Points Count:
 (f1) 32768
 Actual Points Count:
 (f1) 65536
 Acquisition Time (sec):
 (f1) 1.7302
 Spectral Width (ppm):
 (f1) 250.933
 Pulse Program:
 ZGDC
 Temperature:
 298
 Number of Scans:
 1024
 Acq. Date:
 Thu Feb 16 11:35:41 PM

Date: 20 Feb 2006
Document's Title: 31601.mrc
Frequency (MHz): (f1) 75.476
Original Points Count: (f1) 32768
Actual Points Count: (f1) 65536
Acquisition Time (se): (f1) 1.7302
Spectral Width (ppm): (f1) 250.933
Pulse Program: ZGDC
Temperature: 298
Number of Scans: 4096
Acq. Date: Mon Feb 13 12:09:28

Date:

20 Feb 2006

Document's Title:

ok.mrc

Frequency (MHz):

(f1) 400.147

Original Points Count:

(f1) 23971

Actual Points Count:

(f1) 65536

Acquisition Time (sec)

(f1) 3.7440

Spectral Width (ppm):

(f1) 16.001

Pulse Program:

Unknown

Temperature:

25

Number of Scans:

8

Acq. Date:

Jan 6 2004

Date: 20 Feb 2006
 Document's Title: 111000
 Spectrum Title: None
 Frequency (MHz): (f1) 75.476
 Original Points Count: (f1) 32768
 Actual Points Count: (f1) 65536
 Acquisition Time (sec): (f1) 1.7302
 Spectral Width (ppm): (f1) 250.933
 Pulse Program: ZGDC
 Temperature: 298
 Number of Scans: 2048
 Acq. Date: Sun Feb 12 08:00:16 AM

Date: 20 Feb 2006
Document's Title: 12900
Spectrum Title: None
Frequency (MHz): (f1) 75.476
Original Points Count: (f1) 32768
Actual Points Count: (f1) 65536
Acquisition Time (sec): (f1) 1.7302
Spectral Width (ppm): (f1) 250.933
Pulse Program: ZGDC
Temperature: 298
Number of Scans: 2048
Acq. Date: Sun Feb 12 10:52:24 AM