Supporting Information

Aluminium chloride hexahydrate ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$): An efficient, facile, mild, catalytic and highly chemoselective deprotection of *tert*-butyldimethylsilyl (TBS) ethers

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1. GENERAL EXPERIMENTAL INFORMATION

The starting materials were purchased from Aldrich Chemical Co. and were used without further purification. Flash column chromatography was performed with silica gel 60 (230–400 mesh). Silica plates of 0.20 mm thickness were used for thin layer chromatography which was visualized by UV light at 254 nm or by staining using base solution of $\text{CoCl}_2/\text{H}_2\text{SO}_4$ ac. followed by heating. Melting points were determined with a Fischer-Johns Scientific melting point apparatus and they are uncorrected. ^1H and ^{13}C NMR spectra were recorded using a Bruker Avance 300 MHz, and a Varian 500 MHz. The chemical shifts (δ) are given in ppm relative to TMS as internal standard (0.00). The microwave-assisted reactions were performed using a focused microwave unit Anton-Paar Synthos 300, constant factor of the microwave 1.214. The temperature was monitored with an infrared temperature sensor. In all experiments the microwave temperature was held constant. Reactions were carried out in 5 mL glass vessels with a magnetic stir bar, which were sealed with a cap septum. The specific reaction time corresponds to the total irradiation time. After completion of the reaction, the mixture was cooled to 25 °C via air-jet cooling. For analytical purposes the mass spectra were recorded on a Shimadzu GCMS-QP2010 Plus.

2. CHARACTERIZATION DATA OF ALL COMPOUNDS

(3aR,4S,5R,6aS)-5-hydroxy-4-(hydroxymethyl)hexahydro-2H-cyclopenta[*b*]furan-2-one (Ref. 1).

Beige solid, m.p. 115–117 °C.

¹H NMR (500 MHz, CDCl₃) δ = 2.0 (m, 4H), 2.42 (td, *J* = 5.4, 15 Hz, 1H), 2.53 (dd, *J* = 2.4 Hz, 1H), 2.63 (m, 1H), 2.81 (dd, *J* = 9.6 Hz, 1H), 3.65 (m, 2H), 4.18 (q, *J* = 6.3 Hz, 1H), 4.94 (td, *J* = 6.9, 2.3 Hz, 1H) ppm.

¹³C NMR (125 MHz, CDCl₃) δ = 35.2 (CH₂), 39.4 (CH), 40.7 (CH₂), 55.1 (CH), 63.7 (CH₂), 75.6 (CH), 83.4 (CH), 177.02 (C=O) ppm.

MS [EI⁺] *m/z* (%): 173 (M⁺+1), 154 (10), 126 (35), 95 (18), 82 (42), 67 (25), 54 (100), 41 (30).

(3aR,4S,5R,6aS)-5-((*tert*-butyldimethylsilyloxy)-4-((*tert*-butyl dimethylsilyloxy)methyl)hexahydro-2H-cyclopenta[*b*]furan -2-one (Ref. 2)

White solid, m.p. 60–62 °C.

¹H NMR: (300 MHz, CDCl₃) δ = 0.04 (d, *J* = 2.4 Hz, 12H), 0.87 (d, *J* = 7.8 Hz, 18H), 2.01–1.93 (m, 2H), 2.23 (ddd, *J* = 14.8, 6.9, 5.8 Hz, 1H), 2.53 (dd, *J* = 18.0, 3.0 Hz, 1H), 2.66 (m, 1H), 2.77 (dd, *J* = 18.0, 10.6 Hz, 1H), 3.51 (ddd, *J* = 31.4, 10.3, 5.4 Hz, 2H), 4.12 (dd, *J* = 10.4, 4.8 Hz, 1H), 4.92 (td, *J* = 7.2, 2.3 Hz, 1H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = -5.5 (4xCH₃), 18.2 (2xC), 25.8 (6xCH₃), 35.5 (CH₂), 39.1 (CH), 41.0 (CH₂), 56.9 (CH₂), 62.5 (CH), 74.5 (CH), 84.1 (CH), 177.2 (C=O).

MS [EI⁺] *m/z* (%): 401 (M⁺+1), 343 (25), 301 (6), 269 (13), 193 (15), 147 (50), 89 (45), 73 (100), 59 (45), 43 (31).

3-phenoxypropane-1,2-diol (Ref. 3)

White solid, m.p. 50–53 °C

¹H NMR δ (500 MHz, CDCl₃) = 3.2 (s, 2H), 3.6 (m, 2H), 3.7 (m, H), 4.0 (m, 2H), 6.8 (m, 2H), 6.9 (m, H), 7.2 (m, 2H) ppm.

¹³C NMR δ (125 MHz, CDCl₃) = 63.7 (CH₂), 68.9 (CH₂), 70.5 (CH), 114.5 (2xCH), 121.2 (CH), 129.5 (2xCH), 158.4 (C).

MS [EI⁺] *m/z* (%): 168 (M⁺), 150 (50), 136 (22), 119 (30), 94 (100), 77 (28), 66 (35).

2,2,3,3,8,8,9,9-octamethyl-5-(phenoxy)methyl-4,7-dioxo-3,8-disiladecane

Colorless liquid

¹H NMR δ (300 MHz, CDCl₃) = 0.09 (m, 12H), 0.9 (s, 18H), 3.7 (d, 2H), 3.9 (m, H), 4.1 (m, 2H), 6.9 (m, 3H), 7.3 (t, 2H) ppm.

¹³C NMR δ (75 MHz, CDCl₃) = -0.45 (4xCH₃), 18.3 (2xC), 25.9 (6xCH₃), 65.0 (CH₂), 69.9 (CH₂), 72.1 (CH), 114.4 (2xCH), 120.5 (CH), 129.3 (2xCH), 159.0 (C).

MS [EI⁺] *m/z* (%): 397 (M⁺), 303 (70), 277 (10), 249 (12), 207 (11), 171 (52), 133 (75), 89 (12), 73 (16).

2-(1H-indol-3-yl)ethanol (Ref. 4)

Beige solid, m.p. 56–59 °C

¹H NMR δ (500 MHz, CDCl₃) = 1.9 (s, H), 2.9 (t, 2H), 3.8 (t, 2H), 6.9 (s, H), 7.11 (t, H), 7.18 (t, H), 7.29 (d, H), 7.6 (d, H), 8.16 (s, H) ppm.

¹³C NMR δ (125 MHz, CDCl₃) = 28.7 (CH₂), 62.5 (CH₂), 111.3 (CH), 112.1 (C), 118.8 (CH), 119.4 (CH), 122.1 (CH), 122.6 (CH), 127.4 (C), 136.4 (C).

MS [EI⁺] m/z (%): 161 (M⁺), 159 (12), 133 (25), 117 (12), 102 (6), 85 (100), 77 (7).

3-(2-(tert-butyldimethylsilyloxy)ethyl)-1H-indole (Ref. 5)

Amber oil

¹H NMR δ (500 MHz, CDCl₃) = 0.0 (s, 6H), 0.8 (s, 9H), 2.9 (t, 2H), 3.8 (t, 2H), 6.9 (d, H), 7.0 (t, H), 7.1 (t, H), 7.2 (d, H), 7.5 (d, H), 7.85 (s, H) ppm.

¹³C NMR δ (125 MHz, CDCl₃) = 0.0 (2xCH₃), 23.6 (C), 31.2 (3xCH₃), 34.2 (CH₂), 69.1 (CH₂), 116.3 (CH), 118.3 (C), 124.1 (CH), 124.4 (CH), 127.1 (CH), 127.3 (CH), 132.9 (C), 141.3 (C).

MS [EI⁺] m/z (%): 275 (M⁺), 260 (42), 218 (61), 200 (12), 144 (100), 130 (8).

Menthol (Ref. 6)

White solid, m.p. 41–44 °C

¹H NMR: (500 MHz, CDCl₃) δ = 0.82 (d, 3H), 0.92 (d, 6H), 1.05 (m, 1H), 1.63 (m, 1H), 3.41 (m, 1H) ppm.

¹³C NMR: (125 MHz, CDCl₃) δ = 16.02 (CH₃), 20.98 (CH₃), 22.17 (CH₃), 23.09 (CH₂), 25.73 (CH), 31.62 (CH), 34.5 (CH₂), 45.02 (CH₂), 50.07 (CH), 71.4 (CH).

MS [EI⁺] m/z (%): 156 (M⁺), 138 (100), 119 (20), 106 (55), 95 (30), 86 (10), 57 (40)

triisopropyl(2-isopropyl-5-methylcyclohexyloxy)silane (Ref. 7)

Colorless liquid

¹H NMR: (300 MHz, CDCl₃) δ = 0.74 (d, 3H), 0.89 (d, 6H), 1.06 (m, 21H), 1.35 (m, 1H), 1.61 (m, 3H), 1.95 (m, 1H), 3.5 (m, 1H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 12.29 (3xCH), 17.7 (6xCH₃), 18.33 (2xCH₃), 21.45 (CH₃), 22.7 (CH₂), 24.95 (CH), 31.73 (CH), 34.59 (CH₂), 45.76 (CH₂), 50.75 (CH), 72.78 (CH)

***tert*-butyl(2-isopropyl-5-methylcyclohexyloxy)diphenylsilane** (Ref. 8)

White solid, m.p. 61–63 °C.

¹H NMR: (500 MHz, CDCl₃) δ = 0.5 (d, 3H), 0.7 (d, 3H), 0.8 (d, 3H), 1.1 (s, 9H), 1.26 (t, 1H), 1.53 (d, 2H), 1.7 (d, 1H), 2.3 (t, 1H), 3.4 (m, 1H), 7.3 (m, 6H), 7.6 (m, 4H) ppm.

¹³C NMR: (125 MHz, CDCl₃) δ = 15.7 (CH₃), 19.7 (CH₃), 21.5 (CH₃), 22.4 (CH₂), 25.2 (CH), 27.3 (3xCH₃), 31.7 (CH), 34.6 (CH₂), 45.4 (CH₂), 50.6 (CH), 73.9 (CH), 127 (6xCH), 129 (4xCH), 134.4 (2xC).

MS [EI⁺] m/z (%): 337 (30), 217 (20), 199 (80), 139 (100), 137 (28), 95 (21), 81 (57).

cyclopentane-1,1-diyl dimethanol (Ref. 9)

White solid, m.p. 91–93 °C

¹H NMR: (300 MHz, CDCl₃) δ = 1.4 (m, 4H), 1.5 (m, 4H), 3.23 (s, 2H), 3.57 (s, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 25.14 (2xCH₂), 31.79 (2xCH₂), 48.32 (C), 70.14 (2xCH₂).

MS [EI⁺] m/z (%): 131 (M⁺+1), 112 (3), 99 (15), 95 (8), 81 (100), 67 (75), 55 (32), 41 (55).

(cyclopentane-1,1-diylbis(methylene))

bis(oxy)bis(triisopropylsilane)

Colorless liquid

¹H NMR: (300 MHz, CDCl₃) δ = 1.05 (m, 42H), 1.4 (m, 4H), 1.5 (m, 4H), 3.5 (s, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 12 (6xCH), 18.11 (12xCH₃), 25.78 (2xCH₂), 31.37 (2xCH₂), 50.31 (C), 67.19 (2xCH₂).

(cyclopentane-1,1-diylbis(methylene))bis(oxy)bis(*tert*-butyldiphenylsilane)

Yellow oil

¹H NMR: (500 MHz, CDCl₃) δ = 1.04 (s, 18H), 1.44 (dd, *J* = 6.4, 4.0 Hz, 8H), 3.5 (s, 4H), 7.32 (m, 8H), 7.34 (m, 4H), 7.6 (m, 8H) ppm.

¹³C NMR: (125 MHz, CDCl₃) δ = 19.4 (2xC), 25.3 (2xCH₂), 26.9 (6xCH₃), 31.4 (2xCH₂), 50.2 (C), 67 (2xCH₂), 127 (20xCH), 134 (4xC).

MS [EI⁺] m/z (%): 607 (M⁺), 335 (10), 299 (20), 273 (95), 213 (50), 153 (100), 105 (35), 79 (11).

Acetaminophen (Ref. 10)

White solid, m.p. 169–171 °C

¹H NMR: (300 MHz, CDCl₃+DMSO) δ = 2.0 (s, 3H), 6.6 (m, 2H), 7.2 (m, 2H), 9.1 (s, H) ppm.

¹³C NMR: (75 MHz, CDCl₃+DMSO) δ = 23 (CH₃), 114 (2xCH), 120 (2xCH), 129.9 (C), 152.7 (C), 167.7 (C=O).

 MS [EI⁺] m/z (%): 151 (M⁺), 121 (4), 109 (100), 80 (10), 65 (2), 53 (4), 42 (5), 26 (5).

N-(4-(tert-butyldimethylsilyloxy)phenyl)acetamide (Ref. 11)

White solid, m.p. 95–97 °C

¹H NMR: (300 MHz, CDCl₃) δ = 0.17 (s, 6H), 0.96 (s, 9H), 2.1 (s, 3H), 6.7 (d, *J* = 9.0 Hz, 2H), 7.3 (d, *J* = 9.0 Hz, 2H), 7.6 (s, H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = -4.5 (2xCH₃), 18.1 (C), 24.2 (CH₃), 25.6 (3xCH₃), 120.2 (2xCH), 121.6 (2xCH), 131.5 (C), 152.3 (C), 168.4 (C=O).

 MS [EI⁺] m/z (%): 265 (M⁺), 250 (5), 208 (100), 192 (5), 180 (8), 166 (30), 149 (8), 134 (5), 116 (4), 106 (15), 73 (37), 43 (35).

N-(4-(triisopropylsilyloxy)phenyl)acetamide

White solid, m.p. 64–66 °C

¹H NMR: (300 MHz, CDCl₃) δ = 1.1 (m, 18H), 1.2 (m, 3H), 2.0 (s, 3H), 6.8 (d, *J* = 9.0 Hz, 2H), 7.3 (d, *J* = 9.0 Hz, 2H), 7.9 (s, H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 12.5 (3xCH), 17.8 (6xCH₃), 24.1 (CH₃), 119.8 (2xCH), 121.5 (2xCH), 131.4 (C), 152.6 (C), 168.5 (C=O).

 MS [EI⁺] m/z (%): 307 (M⁺), 264 (82), 236 (60), 222 (18), 208 (80), 194 (45), 178 (12), 134 (12), 93 (12), 75 (22), 43 (100).

N-(4-(tert-butyldiphenylsilyloxy)phenyl)acetamide

Slightly yellow oil

¹H NMR: (500 MHz, CDCl₃) δ = 1.0 (s, 9H), 2.0 (d, 3H), 6.6 (m, 2H), 7.1 (m, 2H), 7.3 (m, 7H), 7.7 (m, 3H), 7.9 (s, 1H) ppm.

¹³C NMR: (125 MHz, CDCl₃) δ = 19.4 (C), 24.2 (CH₃), 26.6 (3xCH₃), 115.8 (CH), 119.8 (CH), 121.5 (CH), 122.6 (CH), 127.4 (CH), 129.5 (4xCH), 129.7 (3xCH), 131.2 (C), 132.8 (CH), 134.9 (2xCH), 152.3 (C), 154.1 (C), 162.7 (C), 168 (C=O).

 MS [EI⁺] m/z (%): 389 (M⁺), 350 (100), 290 (40), 272 (38), 231 (25), 149 (20), 77 (8).

Hydroquinone (Ref. 12)

White solid, m.p. 171 °C

¹H NMR: (300 MHz, CDCl₃) δ = 6.6 (s, 4H), 7.4 (s, 2H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 115.8 (4xCH), 149.5 (2xC).

 MS [EI⁺] m/z (%): 110 (M⁺), 81 (33), 63 (8), 53 (32), 39 (21)

1,4-bis(*tert*-butyltrimethylsilyloxy)benzene (Ref. 13)

White solid, m.p. 42–44 °C

¹H NMR: (300 MHz, CDCl₃) δ = 0.16 (s, 12H), 0.97 (s, 8H), 6.6 (s, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = -4.4 (4xCH₃), 18.1 (2xC), 25 (6xCH₃), 120.5 (4xCH), 149.7 (2xC).

MS [EI⁺] m/z (%): 138 (M⁺), 323 (5), 281 (100), 239 (10), 225 (15), 112 (12), 73 (75), 57 (12), 41 (12).

1,4-bis(triisopropylsilyloxy)benzene

White solid, m.p. 22 °C

¹H NMR: (300 MHz, CDCl₃) δ = 1.08 (m, 32H), 1.2 (m, 6H), 6.7 (s, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 12.5 (6xCH), 17.8 (12xCH₃), 120.3 (4xCH), 150 (2xC).

MS [EI⁺] m/z (%): 422 (M⁺), 379 (97), 351 (40), 337 (15), 323 (40), 309 (25), 195 (10), 112 (70), 105 (50), 87 (28), 73 (39), 59 (55), 43 (22).

1,4-bis(*tert*-butylphenylsilyloxy)benzene

White solid, m.p. 121–123 °C

¹H NMR: (300 MHz, CDCl₃) δ = 1.0 (s, 18H), 6.4 (s, 4H), 7.3 (m, 12H), 7.6 (m, 8H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 19.4 (2xC), 26.5 (6xCH₃), 120.0 (4xCH), 127.6 (CH), 129.6 (CH), 135.5 (CH), 149.5 (2xC).

MS [EI⁺] m/z (%): 586 (M⁺), 391 (10), 318 (3), 215 (100), 199 (83), 139 (57), 79 (75), 63 (28).

tert-butyl(3-methoxy-4-

(triisopropylsilyloxy)benzyloxy)dimethylsilane

Yellow oil

¹H NMR: (300 MHz, CDCl₃) δ = 0.03 (s, 3H), 0.089 (s, 3H), 0.94 (s, 9H), 1.1 (d, *J* = 7.2 Hz, 18H), 1.25 (m, 3H), 3.8 (s, 3H), 4.67 (s, 2H), 6.73 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 6.87 (d, *J* = 1.5 Hz, 1H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = -5.1 (CH₃), -2.94 (CH₃), 12.88 (3xCH), 17.91 (6xCH₃), 18.4 (C), 25.66 (3xCH₃), 55.38 (CH₃), 64.99 (CH₂), 110.47 (CH), 118.28 (CH), 119.99 (CH), 134.6 (C), 144.39 (C), 150.73 (C) ppm.

(3-methoxy-4-(triisopropylsilyloxy)phenyl)methanol

Yellow oil

¹H NMR: (300 MHz, CDCl₃) δ = 1.1 (d, *J* = 7.2 Hz, 18H), 1.32–1.18 (m, 3H), 3.8 (s, 3H), 4.58 (s, 2H), 6.75 (dd, *J* = 8.0, 1.8 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.87 (d, *J* = 1.8 Hz, 1H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 12.87 (3xCH), 17.89 (6xCH₃), 55.43 (CH₃), 65.39 (CH₂), 111.25 (CH), 119.41 (CH), 120.22 (CH), 134.08 (C), 145.11 (C), 150.95 (C) ppm.

***tert*-butyl(4-((*tert*-butyldimethylsilyloxy)methyl)-2-methoxyphenoxy)diphenylsilane**

Yellow oil

¹H NMR: (300 MHz, CDCl₃) δ = -0.316 (s, 3H), -0.14 (s, 3H), 0.7 (m, 9H), 0.9 (m, 9H), 4.3 (m, 3H), 5.2 (s, 2H), 6.5–7.0 (m, 3H), 7.2–7.35 (m, 6H), 7.58–7.65 (m, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = -4.97 (CH₃), -4.6 (CH₃), 10.17 (C), 19.4 (C), 26.5 (3xCH₃), 31.6 (3xCH₃), 55.1 (CH₃), 64.8 (CH₂), 115.5 (2xCH), 119.5 (2xCH), 121.6 (CH), 126.9 (2xCH), 127.7 (2xCH), 129.8 (2xCH), 132.9 (CH), 134.8 (CH), 135.5 (2XC), 136.9 (C), 138.3 (C), 154.9 (C) ppm.

(4-(*tert*-butyldiphenylsilyloxy)-3-methoxyphenyl)methanol

Yellow oil

¹H NMR: (300 MHz, CDCl₃) δ = 1.1 (m, 9H), 2.97 (s, OH), 3.55 (s, 3H), 4.49 (s, 2H), 6.5–6.9 (m, 3H), 7.2–7.5 (m, 6H), 7.7 (d, J = 7.8 Hz, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 19.0 (C), 26.6 (3xCH₃), 55.4 (CH₃), 65.3 (CH₂), 111.5 (CH), 119.3 (CH), 120.0 (CH), 127.3 (CH), 127.5 (CH), 127.7 (CH), 128.1 (CH), 129.3 (CH), 129.6 (CH), 129.7 (CH), 133.6 (CH), 134.1 (CH), 134.4 (CH), 138.8 (2xC), 135.3 (C), 144.6 (C), 150.6 (C) ppm.

***tert*-butyldimethyl(3-(triisopropylsilyloxy)benzyloxy)silane**

Yellow oil

¹H NMR: (300 MHz, CDCl₃) δ = 0.199 (s, 6H), 1.05 (s, 9H), 1.2 (d, J = 7.0, 18H), 1.45–1.29 (m, 3H), 4.79 (s, 2H), 6.85 (dd, J = 7.8, 2.1 Hz, 1H), 7.04–6.91 (m, 2H), 7.22–7.34 (m, 1H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = -5.28 (2xCH₃), 12.69 (3xCH), 17.92 (6xCH₃), 18.0 (C), 25.91 (3xCH₃), 64.71 (CH₂), 117.47 (CH), 118.39 (CH), 118.49 (CH), 129.0 (CH), 143.03 (C), 156.11 (C) ppm.

(3-(triisopropylsilyloxy)phenyl)methanol

Yellow oil

¹H NMR: (300 MHz, CDCl₃) δ = 1.11 (d, J = 7.2 Hz, 18H), 1.33–1.18 (m, 3H), 1.77 (s, OH), 4.61 (s, 2H), 6.8 (d, J = 8.0 Hz, 1H), 6.9 (d, J = 8.0 Hz, 2H), 7.19 (t, J = 8.0 Hz, 1H), ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 12.68 (3xCH), 17.92 (6xCH₃), 65.19 (CH₂), 118.39 (CH), 119.01 (CH), 119.42 (CH), 129.45 (CH), 142.49 (C), 156.31 (C) ppm.

***tert*-butyl(3-((*tert*-butyldimethylsilyloxy)methyl)phenoxy)diphenylsilane**

Yellow oil

¹H NMR: (300 MHz, CDCl₃) δ = 0.0 (s, 6H), 0.87 (s, 9H), 1.1 (s, 9H), 4.57 (s, 2H), 6.6 (dd, *J*=8.0, 2.0 Hz, 1H), 6.8 (d, *J*= 2.0 Hz, 1H), 6.83 (s, 1H), 7.0 (t, *J*= 8.0 Hz, 1H), 7.3 (m, 6H), 7.7 (d, *J*= 8.0 Hz, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = -5.3 (2xCH₃), 18.36 (C), 19.49 (C), 25.94 (3xCH₃), 26.57 (3xCH₃), 64.7 (CH₂), 117.47 (CH), 118.23 (CH), 118.72 (CH), 127.74 (CH), 128.92 (2xCH), 129.82 (4xCH), 133.07 (2xC), 135.54 (4xCH), 142.9 (C), 155.62 (C) ppm.

(3-(*tert*-butyldiphenylsilyloxy)phenyl)methanol

Yellow oil

¹H NMR: (300 MHz, CDCl₃) δ = 1.1 (s, 9H), 1.58 (s, OH), 4.48 (s, 2H), 6.62 (dd, *J*=8.0, 2.0 Hz, 1H), 6.82 (t, *J*=7.5 Hz, 2H), 7.0 (t, *J*= 7.8 Hz, 1H), 7.3 (m, 6H), 7.7 (d, *J*=7.5 Hz, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 19.48 (C), 26.55 (3xCH₃), 65.08 (CH₂), 118.32 (CH), 118.9 (CH), 119.56 (CH), 127.78 (CH), 129.3 (2xCH), 129.9 (4xCH), 132.9 (2xC), 135.5 (4xCH), 132.9 (2xC), 135.5 (4xCH), 142.3 (C), 155.81 (C) ppm.

(3a*R*,4*S*,5*R*,6a*S*)-4-((*tert*-butyldimethylsilyloxy)methyl)-5-(*tert*-butyldiphenylsilyloxy)hexahydro-2*H*-cyclopenta[*b*]furan-2-one

Colorless oil

¹H NMR: (300 MHz, CDCl₃) δ = 0.01 (d, *J*= 3.0 Hz, 6H), 0.87 (s, 9H), 1.12 (s, 9H), 2.0 (m, 3H), 2.7 (m, 2H), 2.89 (dd, *J*= 18.6, 11.0 Hz, 1H), 3.35 (dd, *J*= 10.0 6.0 Hz, 1H), 3.47 (dd, *J*=, 10.0 6.0 Hz, 1H), 4.1 (q, *J*= 5.0 Hz, 1H), 7.46 (m, 7H), 7.7 (d, *J*= 7.5 Hz, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = -5.59 (CH₃), -5.56 (CH₃), 18.1 (C), 19.03 (C), 25.84 (3xCH₃), 26.87 (3xCH₃), 35.93 (CH₂), 39.9 (CH₂), 41.14 (CH), 57.15 (CH), 63.32 (CH₂), 76.32 (CH), 84.65 (CH), 127.7 (2xCH), 129.7 (4xCH), 133.6 (2xC), 135.8 (4xCH), 177.41 (C=O) ppm.

(3a*R*,4*S*,5*R*,6a*S*)-5-(*tert*-butyldiphenylsilyloxy)-4-(hydroxymethyl)hexahydro-2*H*-cyclopenta[*b*]furan-2-one

White solid, m.p. 141–144 °C

¹H NMR: (300 MHz, CDCl₃) δ = 1.06 (s, 9H), 1.61 (s, OH), 2.05 (q, *J*= 5.0 Hz, 3H), 2.54 (d, *J*= 2.4 Hz, 1H), 2.61 (d, *J*= 2.4 Hz, 1H), 2.67 (m, 1H), 2.8 (dd, *J*= 17.4, 10.0 Hz, 1H), 3.38 (t, *J*= 6.3 Hz, 2H), 4.1 (q, *J*= 5.1 Hz, 1H), 7.4 (m, 7H), 7.66 (m, 5H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 19.02 (C), 26.86 (3xCH₃), 35.73 (CH₂), 39.03 (CH₂), 40.83 (CH), 56.8 (CH), 62.12 (CH₂), 75.61 (CH), 84.21 (CH), 127.7 (2xCH), 129.9 (4xCH), 133.4 (2xCH), 135.9 (4xCH), 177.49 (C=O) ppm.

***tert*-butyl((1*S*,2*R*)-2-(2-(*tert*-butyldimethylsilyloxy)ethyl)cyclopent-3-enyloxy)diphenylsilane**

Thick yellow oil

¹H NMR: (300 MHz, CDCl₃) δ = 0.02 (s, 6H), 0.85 (s, 9H), 1.01 (s, 9H), 1.56 (m, 1H), 2.1 (m, 3H), 2.53 (m, 1H), 3.63 (m, 2H), 4.4 (q, *J* = 7.0 Hz, 1H), 5.48 (m, 1H), 5.64 (m, 1H), 7.33 (m, 7H), 7.62 (m, 5H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 5.1 (2xCH₃), 18.37 (C), 19.36 (C), 26.04 (3xCH₃), 27.04 (3xCH₃), 31.92 (CH₂), 39.94 (CH₂), 45.3 (CH), 62.15 (CH₂), 75.26 (CH), 127.6 (CH), 129.5 (2xCH), 133.77 (4xCH), 134.11 (2xC), 134.75 (4xCH), 135.84 (CH) ppm.

2-((1*R*,5*S*)-5-(*tert*-butyldiphenylsilyloxy)cyclopent-2-enyl)ethanol

Colorless oil

¹H NMR: (300 MHz, CDCl₃) δ = 1.1 (s, 9H), 1.76 (m, 1H), 2.0 (m, 2H), 2.14 (dd, *J* = 17.0, 7.5 Hz, 1H), 2.3 (m, 1H), 2.62 (m, 1H), 3.7 (m, 2H), 4.56 (q, *J* = 7.2 Hz, 1H), 5.57 (m, 1H), 5.62 (m, 1H), 7.4 (m, 6H), 7.68 (m, 4H) ppm.

¹³C NMR: (75 MHz, CDCl₃) δ = 19.26 (C), 27.0 (3xCH₃), 31.66 (CH₂), 39.83 (CH₂), 45.59 (CH), 61.36 (CH₂), 75.21 (CH), 127.68 (CH), 128.43 (2xCH), 129.73 (4xCH), 133.03 (2xC), 133.74 (2xCH), 134.2 (2xCH), 135.82 (CH) ppm.

1-(4-(*tert*-butyldimethylsilyloxy)phenyl)propan-1-one

White solid, m.p. 20–22 °C

¹H NMR δ (300 MHz, CDCl₃) = 0.2 (s, 6H), 0.9 (s, 9H), 1.2 (t, 3H), 2.9 (q, 2H), 6.8 (m, 2H), 7.8 (m, 2H) ppm.

¹³C NMR δ (75 MHz, CDCl₃) = -4.4 (2xCH₃), 8.4 (CH₃), 18.2 (C), 25.5 (3xCH₃), 31.3 (CH₂), 119.8 (2xCH), 130.1 (2xCH), 130.5 (C), 160 (C), 199.5 (C=O) ppm.

MS [EI⁺] m/z (%): 264 (M⁺), 235 (40), 207 (100), 179 (17), 151 (15), 135 (10), 121 (5), 105 (2), 89 (5), 73 (7), 57 (11).

1-(4-hydroxyphenyl)propan-1-one

White solid, m.p. 147–149 °C

¹H RMN δ (300 MHz, CDCl₃+DMSO) = 1 (t, 3H), 2.8 (q, 2H), 6.7 (m, 2H), 7.7 (m, 2H) ppm.

¹³C RMN δ (75 MHz, CDCl₃+DMSO) = 7.5 (CH₃), 30 (CH₂), 114.3 (2xCH), 127.5 (C), 129.24 (2xCH), 161 (C), 198 (C=O) ppm.

MS [EI⁺] m/z (%): 150 (M⁺), 120 (2), 93 (20), 65 (10), 63 (3), 38 (2), 26 (2).

4-(*tert*-butyldimethylsilyloxy)phenyl benzoate

White solid, m.p. 79–81 °C

$^1\text{H NMR}$ δ (300 MHz, CDCl_3) = 0.2 (s, 6H), 1.0 (s, 9H), 6.8 (d, 2H), 7.0 (d, 2H), 7.5 (m, 2H), 7.6 (m, H), 8.1 (d, 2H) ppm.

$^{13}\text{C NMR}$ δ (75 MHz, CDCl_3) = -4.4 (2xCH₃), 18.1 (C), 25.6 (3xCH₃), 120.5 (2xCH), 122.3 (2xCH), 128.5 (2xCH), 129.7 (C), 130.1 (2xCH), 133.4 (CH), 144.9 (C), 153.3 (C), 165.4 (C=O) ppm.

MS [EI^+] m/z (%): 328 (M^+), 271 (65), 166 (5), 138 (5), 123 (6), 105 (100), 77 (70), 51 (12).

4-hydroxyphenyl benzoate

White solid, m.p. 163–165°C

$^1\text{H NMR}$ δ (300 MHz, CDCl_3) = 6.8 (m, 2H), 7.0 (m, 2H), 7.5 (m, 2H), 7.6 (m, H), 8.1 (m, 2H) ppm.

$^{13}\text{C NMR}$ δ (75 MHz, CDCl_3) = 115.5 (2xCH), 121.7 (2xCH), 128 (CH), 129.2 (C), 129.5 (2xCH), 133.0 (C), 142.8 (2xCH), 154.6 (C), 165.1 (C=O) ppm.

MS [EI^+] m/z (%): 214 (M^+), 105 (100), 77 (25), 51 (5), 28 (7).

4-(*tert*-butyldiphenylsilyloxy)-3-methoxybenzaldehyde

Yellow oil

$^1\text{H NMR}$ δ (300 MHz, CDCl_3) = 1.0 (s, 9H), 3.6 (s, 3H), 7.3 (m, 9H), 7.7 (m, 5H), 9.7 (s, H) ppm.

$^{13}\text{C NMR}$ δ (75 MHz, CDCl_3) = 19.0 (C), 26.5 (3xCH₃), 109–135 (13xCH), 151.1 (C), 151.3 (C), 191 (C=O).

MS [EI^+] m/z (%): 391 ($\text{M}^+ + 1$), 333 (70), 318 (70), 261 (10), 217 (55), 199 (100), 152 (30), 139 (60), 79 (70), 63 (11) ppm.

methyl 3-(*tert*-butyldiphenylsilyloxy)benzoate

Yellow oil

$^1\text{H NMR}$ δ (300 MHz, CDCl_3) = 1.1 (m, 9H), 3.83 (s, 3H), 6.83 (d, J = 8.5 Hz, 1H), 7.0 (q, J = 7.5 Hz, 1H), 7.4 (m, 6H), 7.54 (m, 1H), 7.7 (m, 4H) ppm.

$^{13}\text{C NMR}$ δ (75 MHz, CDCl_3) = 19.6 (C), 26.7 (3xCH₃), 52.26 (CH₃), 121.11 (CH), 122.51 (CH), 124.4 (CH), 127.9 (CH), 128.04 (CH), 129.8 (CH), 130.2 (2xCH), 132.68 (2xCH), 135.01 (2xCH), 135.47 (2xC), 135.7 (C), 155.79 (C), 167.11 (C) ppm.

3. REFERENCES

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4. Copies of ^1H -NMR and ^{13}C -NMR spectra of compounds

Compound 1

Compound 1a

CCIQ3 UAEM-UNAH NZ
Dr. Carlos Gonzalez/David
Clave: ALQ-13
CDCl₃
1H
Varian-NMRSystem 500MHz
no. reg. 716
3-06-2011
File: exp
Pulse Sequence: s2pul

CCIQ3 UAEM-UNAH NZ
Dr. Carlos Gonzalez/David
Clave: ALQ-13
CDCl₃
13C
Varian-NMRSystem 500MHz
no. reg. 716
3-06-2011
File: exp
Pulse Sequence: s2pul

Compound 2a

Universidad Autonoma del Estado de Mexico
Facultad de Quimica
Centro de Investigación en Química Sustentable
DCG-AP
1H RMN
CDCl₃

Universidad Autonoma del Estado de Mexico
Facultad de Quimica
Centro de Investigación en Química Sustentable
DCG-AP
13C RMN
CDCl₃

Compound 2

CCIOS
DGL_A 1H RMN
500 MHz CDCl3
Sample ID: s_20110210_07
File: s_20110210_07\data/cdc13_01.fid
Pulse Sequence: s2pul

CCIOS
DGL_A 13C RMN
125 MHz CDCl3
Sample ID: s_20110210_07
File: s_20110210_07\data/cdc13_02.fid
Pulse Sequence: s2pul

Compound 3a

```

CQIQS
DQC_DP
500MHz CDC13 1H RMN

Sample ID: s_20110210_05
File: s_20110210_05/data/cdc13_01.fid

Pulse Sequence: zgpg30

```

```
CCIOS
DGC_DP 13C RMN
125 MHz CDC13
Sample ID: s_20110210_05
File: s_20110210_05/data/cdc13_02.fid
Pulse Sequence: s2nul
```

Compound 3

CC105
 DDC_0 1H RMN
 500 MHz CDCl3
 Sample ID: s_20110210_06
 File: s_20110210_06\data/cdc13_01.fid
 Pulse Sequence: s2pu1

CC105
 DDC_0 13C RMN
 125 MHz CDCl3
 Sample ID: s_20110210_06
 File: s_20110210_06\data/cdc13_02.fid
 Pulse Sequence: s2pu1

Compound 4a

Compound 4b

CCIQS UAEM UNAM DCS
Dr. Carlos Gonzalez/ Carlos Gonzalez G.
Clave: CAGG-0
1H RMN CDCl3 500 MHz
Varian NMR System 500 MHz
No. Reg. 020
11/01/2013

File: xp

Pulse Sequence: s2pu1

CCIQS UAEM UNAM DCS
Dr. Carlos Gonzalez/ Carlos Gonzalez G.
Clave: CAGG-0
13C RMN CDCl3 125 MHz
Varian NMR System 500 MHz
No. Reg. 020
11/01/2013

File: home/DavidC2/vnmrsys/data/DrCarlosG/2013/020-13C.fid

Pulse Sequence: szpu1

Compound 4

Compound 5a

13C
CCIQS UAEM-UNAM NZ
Dr. Carlos Gonzalez/Luis B.
Clave: LJDS 02
CDCl₃
1H
Bruker-Avance de 300MHz
No. reg. 1567
23-11-2011

13C
CCIQS UAEM-UNAM NZ
Dr. Carlos Gonzalez/Luis B.
Clave: LJDS 02
CDCl₃
13C
Bruker-Avance de 300MHz
No. reg. 1567
23-11-2011

Compound 5b

631-11
CCIOS UAEM-UNAM, NZ

Dr. Carlos Gozalez/Carlos A.

Clave: CAGG-C

CDCI3

 $1H$

Varian-NMRSystem 500MHz

no. reg. 1631

8-12-2011

163.88	166.58	165.36	163.94	162.63	161.41	160.70	159.56	157.55	157.53	157.52	157.51
CC	CI	IO	UA	IM	UN	AM	NZ				

~~Dr. Carlos Gozalez/Carlos A.~~

Clave: CAGG-C

CDC13

13C

Varian-NMRSystem 500MHz

no reg 1631

8-12-2011

Compound 5

¹³C
 CCIQS UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Luis B.
 Clave: PD 005
 CDCl₃
¹H
 Bruker-Avance de 300MHz
 No. reg. 996
 3-08-2011

¹³C
 CCIQS UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Luis B.
 Clave: PD 005
 CDCl₃
¹³C
 Bruker-Avance de 300MHz
 No. reg. 996
 3-08-2011

Compound 6a

13C
CCIQS UAEM-UNAM NZ
Dr. Carlos Gniz./Luis B.
Clave: PD 10
CDCl₃
1H
Bruker-Avance de 300MHz
No. reg. 1029
24-08-2011

13C
CCIQS UAEM-UNAM NZ
Dr. Carlos Gniz./Luis B.
Clave: PD 10
CDCl₃
13C
Bruker-Avance de 300MHz
No. reg. 1029
24-08-2011

Compound 6b

¹³C
CCIQS UAEM-UNAM N2
Dr. Carlos Gonzalez/Luis B.
Clave: LJDS 17
CDCl₃
¹H
Bruker-Avance de 300MHz
No. reg. 1566
23-11-2011

13C
CCIQSUAEM-UNAM
Dr. Carlos Gonzalez/Luis B.
Clave: LJDS 17
CDCl3
13C
Bruker-Avance de 300MHz
No. reg. 1566
23-11-2011

Compound 6c

CCIOS UAEM UNAM DCS
Dr. Carlos Gonzalez/ Carlos Gonzalez G.
Clave: CAGG-F
1H RMN CDCl3 500 MHz
Varian NMR System 500 MHz
No. Reg. 021
11/01/2013

File: home/DavidC2/vnmrsys/data/DrCarlosG/2013/022-1H.fid

Pulse Sequence: s2pu1

CCIOS UAEM UNAM DCS
Dr. Carlos Gonzalez/ Carlos Gonzalez G.
Clave: CAGG-F
13C RMN CDCl3 125 MHz
Varian NMR System 500 MHz
No. Reg. 021
11/01/2013

File: home/DavidC2/vnmrsys/data/DrCarlosG/2013/022-13C.fid

Pulse Sequence: s2pu1

Compound 7b

13C
 CCIQS UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Luis B.
 Clave: LJDS 08
 CDCl₃
 1H
 Bruker-Avance de 300MHz
 No. reg. 1479
 10-11-2011

13C
 CCIQS UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Luis B.
 Clave: LJDS 08
 CDCl₃
 13C
 Bruker-Avance de 300MHz
 No. reg. 1479
 10-11-2011

Carpeta A-1
 CCIQS UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Carlos A.
 Clave: CACG-B
 CDCl₃
 1H
 Bruker-Avance de 300MHz
 No. reg. 1630
 8-12-2011

Carpeta A-1
 CCIQS UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Carlos A.
 Clave: CACG-B
 CDCl₃
 13C
 Bruker-Avance de 300MHz
 No. reg. 1630
 8-12-2011

Compound 8a

Compound 8b

Compound 8c

Compound 8d

Compound 9a

Compound 9b

Compound 11a

CCIQS UAEM-UNAM NZ
Academico Dr. C. Gonzalez
Equipo Bruker Avance III 300MHz
Experimento 13C
No. reg. 980
Disolvente CDCl₃
Clave DGC B

Compound 12a

13C
CCIQS UAEM-UNAM NZ
Dr. Carlos Gonzalez/Luis B.
Clave: PD 01
CDCl₃
1H
Bruker-Avance de 300MHz
No. reg. 988
2-08-2011

13C
CCIQS UAEM-UNAM NZ
Dr. Carlos Gonzalez/Luis B.
Clave: PD 01
CDCl₃
13C
Bruker-Avance de 300MHz
No. reg. 988
2-08-2011

Compound 12

13C
CCIQS UAEM-UNAM NZ
Dr. Carlos Gnz./Luis B.
Clave: PD 09
CDCl₃+DMSO
1H
Bruker-Avance de 300MHz
No. reg. 1035
24-08-2011

13C
CCIQS UAEM-UNAM NZ
Dr. Carlos Gnz./Luis B.
Clave: PD 09
CDCl₃+DMSO
13C
Bruker-Avance de 300MHz
No. reg. 1035
24-08-2011

Compound 13a

13C
CCIQS UAEM-UNAM N2
Dr. Carlos Gn2./Luis B.
Clave: PD 08
CDCl3
1H
Bruker-Avance de 300MHz
No. reg. 1027
24-08-2011

13C
CCIQS UAEM-UNAM N2
Dr. Carlos Gn2./Luis B.
Clave: PD 08
CDCl3
13C
Bruker-Avance de 300MHz
No. reg. 1027
24-08-2011

Compound 13

CCIQS-UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Luis B.
 Clave: PD 01
 CDCl₃
 1H
 Bruker-Avance de 300MHz
 No. reg. 993
 3-08-2011

CCIQS-UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Luis B.
 Clave: PD 01
 CDCl₃
 13C
 Bruker-Avance de 300MHz
 No. reg. 993
 3-08-2011

Compound 14

Carpeta A-1
 CCIQS UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Carlos A.
 Clave: CAGG-A
 CDCl₃
 1H
 Bruker-Avance de 300MHz
 No. reg. 1629
 8-12-2011

Carpeta A-1
 CCIQS UAEM-UNAM NZ
 Dr. Carlos Gonzalez/Carlos A.
 Clave: CAGG-A
 CDCl₃
 13C
 Bruker-Avance de 300MHz
 No. reg. 1629
 8-12-2011

Compound 15

CCIOS UAEM UNAM DCB
Dr. Carlos Gonzalez/ Carlos Gonzalez G.
Clave: CAGG-E
1H RMN CDCl3 500 MHz
Varian NMR System 500 MHz
No. Reg. 021
11/01/2013

File: home/DavidC2/vnmrsys/data/DrCarlosG/2013/021-1H.fid
Pulse Sequence: s2pul

CCIOS UAEM UNAM DCB
Dr. Carlos Gonzalez/ Carlos Gonzalez G.
Clave: CAGG-E
13C RMN CDCl3 125 MHz
Varian NMR System 500 MHz
No. Reg. 021
11/01/2013

File: home/DavidC2/vnmrsys/data/DrCarlosG/2013/021-13C.fid
Pulse Sequence: s2pul