Supporting Information

Acid-catalyzed Transacetalization from Glycol to Pinacol Acetals

Yvonne Seeleib, Gregor Nemecek, Dominik Pfaff, Bastian Süveges, and Joachim Podlech*

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Experimental Procedures

General. Flash column chromatography¹ was carried out using *Merck* SiO₂ 60 (230–400 mesh), and thin layer chromatography (TLC) was carried out using commercially available *Merck* F₂₅₄ precoated sheets. ¹H and ¹³C NMR spectra were recorded on a *Bruker* AVANCE 300 or on an AM-400 spectrometer. Chemical shifts are given in ppm and were referenced using residual signals of the solvent as internal standard (¹H: CHCl₃, 7.26; ¹³C: CDCl₃, 77.16). IR spectra were recorded on a *Bruker* IFS-88 spectrometer. Mass spectra were recorded on a *Finnigan* MAT-90 mass spectrometer.

General procedure (GP). Pinacol (6 eq.) is placed in a flask, evacuated and flushed with Ar (4×). TFA (5 mL / 6 mmol pinacol) is added and the solution is stirred until pinacol is completely dissolved. The solution is cooled (0 °C) and the glycol acetal (1 eq.) is added. After stirring at 0 °C for 30 min, the mixture is carefully dissolved in an excess of a satd. aq. NaHCO₃ soln and extracted with EtOAc (3×50 mL). The combined organic layers are washed with brine (3×50 mL), dried (Na₂SO₄) and concentrated. The crude product is purified by flash column chromatography (silica gel).

4,4,5,5-Tetramethyl-2-phenyl-1,3-dioxolane (2).² 2-Phenyl-1,3-dioxolane (1) and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc/Et₃N, 250:10:1) afforded **2** (286 mg, 68%) as a colorless oil. *R*_f = 0.75 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm⁻¹) = 2976 (m), 1451 (w), 1366 (m), 1216 (m), 1151 (s), 1061 (m), 991 (m). ¹H NMR (400 MHz, CDCl₃): δ = 1.28 (s, 6 H, CH₃), 1.34 (s, 6 H, CH₃), 5.99 (s, 1 H, 2-H), 7.32–7.39 (m, 3 H, Ph), 7.49–7.51 (m, 2 H, Ph). ¹³C NMR (100 MHz, CDCl₃): δ = 22.2 (CH₃), 24.5 (CH₃), 82.8 (C), 100.0 (CH), 126.4 (CH), 128.4 (CH), 128.7 (CH), 139.8 (C). MS (EI, 20 °C): *m/z* (%) = 206 (82) [M⁺], 205 (90) [M–H]⁺, 148 (39), 105 (100), 90 (38), 84 (50), 77 (38). HRMS (¹²C₁₃¹H₁₈¹⁶O₂, EI): calcd. 206.1307 amu; found 206.1302 amu.

2-(3-Chloropropyl)-4,4,5,5-tetramethyl-1,3-dioxolane (17). 2-(3-Chloropropyl)-1,3-dioxolane (3) and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc, 25:1) afforded **17** (324 mg, 77%) as a colorless oil. *R*_f = 0.54 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm⁻¹) = 2976 (w), 2870 (w), 1366 (m), 1128 (s), 650 (w). ¹H NMR (400 MHz, CDCl₃): δ = 1.19 (s, 12 H, CH₃), 1.69–1.76 (m, 2 H, 1'-H₂), 1.84–1.94 (m, 2 H, 2'-H₂), 3.57 (t, ³*J* = 6.7 Hz, 2 H, 3'-H₂), 5.05 (t, ³*J* = 5.0 Hz, 1 H, 2-H). ¹³C NMR (100 MHz, CDCl₃): δ = 22.2 (CH₃), 24.4 (CH₃), 27.8 (CH₂), 33.6 (CH₂), 45.1 (CH₂), 82.0 (C), 100.3 (CH). MS (EI, 20 °C): *m/z* (%) = 205 (3) [M–H]⁺, 129 (82), 101 (71), 85 (35), 83 (100), 59 (33), 57 (28).

¹ Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923-2925.

² Kulesza, J.; Kowalski, A. *Zesz. Nauk. - Politech. Lodz., Chem. Spozyw.* **1963**, *8*, 27-36; *Chem. Abstr.* **1965**, 63:31510.

2-(2-Bromoethyl)-4,4,5,5-tetramethyl-1,3-dioxolane (18).³ 2-(2-Bromoethyl)-1,3-dioxolane (**4**) and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc, 25:1) afforded **18** (316 mg, 63%) as a colorless liquid. R_f = 0.66 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 2975 (m), 1366 (m), 1124 (s), 519 (w). ^1H NMR (400 MHz, CDCl_3): δ = 1.18 (s, 6 H, CH_3), 1.19 (s, 6 H, CH_3), 2.13 (td, 3J = 7.2, 5.0 Hz, 2 H, $1'\text{-H}_2$), 3.45 (t, 3J = 7.2 Hz, 2 H, $2'\text{-H}_2$), 5.14 (t, 3J = 4.9, 1 H, 2-H). ^{13}C NMR (100 MHz, CDCl_3): δ = 22.2 (CH_3), 24.3 (CH_3), 27.9 (CH_2), 39.6 (CH_2), 82.2 (C), 98.9 (CH). MS (EI, 25 °C): m/z (%) = 237 (4) [M^+], 235 (4) [M-H^+], 129 (100), 101 (87), 85 (86), 83 (87). HRMS ($^{12}\text{C}_9^1\text{H}_{16}^{79}\text{Br}^{16}\text{O}_2$, EI): calcd. 235.0328 amu; found 235.0328 amu.

4-Iodo-5-methyl-5-((4,4,5,5-tetramethyl-1,3-dioxolan-2-yl)-methyl)furan-2(5H)-one (19). 4-Iodo-5-methyl-5-((1,3-dioxolan-2-yl)-methyl)furan-2(5H)-one (**7**)⁴ and pinacol were reacted according to the GP. In this case an extended reaction time of 20 h was necessary. Chromatography (hexanes/EtOAc, 25:1) afforded **19** (112 mg, 95%) as a white solid. R_f = 0.35 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3093 (w), 2971 (w), 2884 (w), 1723 (s), 1579 (m), 1115 (m), 956 (m), 711 (m). ^1H NMR (400 MHz, CDCl_3): δ = 1.12 (s, 3 H, CH_3), 1.14 (s, 3H, CH_3), 1.17 (s, 3H, CH_3), 1.18 (s, 3 H, CH_3), 1.50 (s, 3 H, 5- CH_3), 2.00 (dd, 3J = 14.5, 5.2 Hz, 1 H, CH_aH_b), 2.17 (dd, 3J = 14.6, 5.0 Hz, 1 H, CH_aH_b), 5.07 (t, 3J = 5.1 Hz, 1 H, $2'\text{-H}$), 6.40 (s, 1 H, 3-H). ^{13}C NMR (100 MHz, CDCl_3): δ = 22.1 (CH_3), 24.1 (CH_3), 25.4 (CH_3), 44.0 (CH_2), 82.2 (C), 90.5 (C), 96.8 (CH), 129.5 (CH), 132.4 (C), 170.8 (C). MS (EI, 50 °C): m/z (%) = 365 (1) [M-H^+], 129 (100), 101 (33), 85 (52). HRMS ($^{12}\text{C}_{13}^1\text{H}_{19}^{16}\text{O}_4^{127}\text{I}_1$, EI): calcd. 366.0328 amu; found 366.0326 amu.

2-Methyl-1-(4,4,5,5-tetramethyl-1,3-dioxolan-2-yl)but-3-yn-2-ol (20). 2-Methyl-1-(1,3-dioxolan-2-yl)but-3-yn-2-ol (**8**)⁴ and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc, 25:1) afforded **20** (248 mg, 72%) as a light brown liquid. R_f = 0.36 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 3293 (w), 2981 (w), 1368 (m), 1155 (s). ^1H NMR (400 MHz, CDCl_3): δ = 1.21 – 1.23 (m, 12 H, CH_3), 1.52 (s, 3 H, CH_3), 1.80 (dd 3J = 13.9, 8.6 Hz, 1 H, CH_aH_b), 2.10 (dd, 3J = 13.9, 2.9 Hz, 1 H, CH_aH_b), 2.48 (s, 1 H, 4-H), 5.49 (dd, 3J = 8.6, 2.9 Hz, 1 H, $2'\text{-H}$), 5.56 (bs, 1 H, OH). ^{13}C NMR (100 MHz, CDCl_3): δ = 22.0 (CH_3), 22.2 (CH_3), 23.7 (CH_3), 24.6 (CH_3), 30.5 (CH_3), 47.6 (CH_2), 65.9 (C), 72.2 (CH), 82.1 (C), 83.2 (C), 86.3 (C), 99.2 (CH). MS (EI, 30 °C): m/z (%) = 211 (1) [M-H^+], 129 (100), 101 (63), 85 (63), 83 (22), 82 (62), 69 (26). HRMS ($^{12}\text{C}_{12}^1\text{H}_{20}^{16}\text{O}_3$, EI): calcd. 212.1412 amu; found 212.1409 amu.

³ Stach, L. J.; Hotz, R. D.; Richter, S. B. (US 4113464), Velsicol Chemical Company, 1978.

⁴ a) Nemecek, G.; Thomas, R.; Goesmann, H.; Feldmann, C.; Podlech, J. *Eur. J. Org. Chem.* **2013**, 6420-6432; b) Nemecek, G. *Strukturaufklärung und Totalsynthese von Toxinen aus Alternaria tenuis*. Ph. D. Thesis, Karlsruher Institut für Technologie (KIT), 2013.

2-(3-Bromophenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (21). 2-(3-Bromophenyl)-1,3-dioxolane (**9**) and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc, 25:1) afforded **21** (1367 mg, 92%) as a white solid. R_f = 0.58 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 2981 (w), 1714 (m), 1290 (s), 1119 (m), 746 (m). ^1H NMR (400 MHz, CDCl_3): δ = 1.28 (s, 6 H, CH_3), 1.35 (s, 6 H, CH_3), 5.96 (s, 1 H, 2-H), 7.27 (dd, 3J = 14.3, 6.5 Hz, 1 H, Ar), 7.46 (dd, 3J = 14.5, 7.8 Hz, 2 H, Ar), 7.67 (s, 1 H, Ar). ^{13}C NMR (100 MHz, CDCl_3): δ = 22.0 (CH_3), 24.1 (CH_3), 82.8 (C), 98.7 (CH), 122.2 (C), 124.7 (CH), 129.1 (CH), 129.7 (CH), 131.4 (CH), 142.1 (C). MS (EI, 30 °C): m/z (%) = 286 (96) [M^+], 284 (96) [M^+], 228 (30), 226 (32), 185 (89), 183 (81), 84 (100). HRMS ($^{12}\text{C}_{13}\text{H}_{17}^{16}\text{O}_2^{79}\text{Br}$, EI): calcd. 284.0412 amu; found 284.0407 amu.

2-(4-Fluorophenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (22).⁵ 2-(4-Fluorophenyl)-1,3-dioxolane (**10**) and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc, 25:1) afforded **22** (257 mg, 62%) as a colorless oil. R_f = 0.54 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 2977 (w), 1509 (m), 1222 (s), 1148 (s), 1070 (s), 828 (m). ^1H NMR (400 MHz, CDCl_3): δ = 1.27 (s, 6 H, CH_3), 1.32 (s, 6 H, CH_3), 5.95 (s, 1 H, 2-H), 7.04 (ddd, 3J = 9.5, 5.8, 2.4, 2 H, Ph), 7.44–7.49 (m, 2 H, Ph). ^{13}C NMR (100 MHz, CDCl_3): δ = 22.3 (CH_3), 25.0 (CH_3), 82.9 (C), 99.5 (CH), 115.3 (CH), 128.3 (CH), 135.8 (C), 163.1 (C). MS (EI, 40 °C): m/z (%) = 224 (3) [M^+], 223 (45) [$\text{M}-\text{H}^+$], 123 (100), 83 (24), 59 (10). HRMS ($^{12}\text{C}_{13}\text{H}_{16}^{16}\text{O}_2^{19}\text{F}$, EI): calcd. 223.1129 amu; found 223.1130 amu.

2-(Furan-2-yl)-4,4,5,5-tetramethyl-1,3-dioxolane (23).⁶ 2-(Furan-2-yl)-1,3-dioxolane (**11**) and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc, 25:1) afforded **23** (232 mg, 56%) as a colorless oil. R_f = 0.59 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 2976 (w), 1368 (m), 1151 (s), 1067 (s), 987 (m), 739 (m). ^1H NMR (400 MHz, CDCl_3): δ = 1.28 (s, 6 H, CH_3), 1.31 (s, 6 H, CH_3), 5.95 (s, 1 H, 2-H), 6.33 (dd, 3J = 3.2, 1.8 Hz, 1 H, 3'-H), 6.43 (d, 3J = 3.3 Hz, 1 H, 4'-H), 7.40–7.41 (m, 1 H, 5'-H). ^{13}C NMR (100 MHz, CDCl_3): δ = 21.9 (CH_3), 23.7 (CH_3), 82.8 (C), 94.2 (CH), 108.6 (CH), 110.1 (CH), 143.1 (CH), 152.2 (C). MS (EI, 70 °C): m/z (%) = 196 (39) [M^+], 195 (26) [M^+], 138 (38), 129 (43), 101 (37), 97 (60), 59 (100). HRMS ($^{12}\text{C}_{11}\text{H}_{16}^{16}\text{O}_3$, EI): calcd. 196.1099 amu; found 196.1093 amu.

2-(2-Bromo-5-hexyloxyphenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (24). 2-(5-(Hexyloxy)-2-bromophenyl)-1,3-dioxolane (**12**) and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc, 25:1) afforded **24** (293 mg, 83%) as a colorless oil. R_f = 0.62 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 2931 (s), 1573 (m), 1293 (s), 1155 (s), 1080 (s),

⁵ Pourjavadi, A. *Iran. J. Sci. Technol.* **1998**, 22, 135-150; *Chem. Abstr.* **1998**, 129: 148643.

⁶ Kosulina, T. P.; Kul'nevich, V. G. *Khim. Geterotsikl. Soedin.* **1990**, 992-993; *Chem. Heterocycl. Compd.* **1990**, 26, 831-832.

1006 (s). ^1H NMR (400 MHz, CDCl_3): δ = 0.90 (t, 3J = 6.7 Hz, 3 H, 6'-H₃), 1.30 (s, 6 H, CH₃), 1.31–1.34 (m, 4 H, 5'-H₂, 4'-H₂), 1.35 (s, 6 H, CH₃), 1.40–1.47 (m, 2 H, 3'-H₂), 1.72–1.80 (m, 2 H, 2'-H₂), 3.93 (t, 3J = 6.5 Hz, 2 H, 1'-H₂), 5.96 (s, 1 H, 2-H), 6.62 (dd, 3J = 8.6, 3.1 Hz, 1 H, Ar), 7.21 (d, 3J = 3.1 Hz, 1 H, Ar), 7.65 (d, 3J = 8.6 Hz, 1 H, Ar). ^{13}C NMR (100 MHz, CDCl_3): δ = 14.2 (CH₃), 22.3 (CH₃), 22.7 (CH₂), 24.5 (CH₃), 25.8 (CH₂), 29.2 (CH₂), 31.7 (CH₂), 68.4 (CH₂), 83.0 (C), 98.9 (CH), 113.3 (C), 114.0 (CH), 116.7 (CH), 133.6 (CH), 139.0 (C), 158.6 (C). MS (EI, 100 °C): m/z (%) = 386 (85) [M^+], 384 (80) [M^+], 326 (36), 284 (40), 283 (34), 225 (35), 129 (36), 101 (35), 84 (100). HRMS ($^{12}\text{C}_{19}\text{H}_{29}^{16}\text{O}_3^{79}\text{Br}$, EI): calcd. 384.1300 amu; found 384.1292 amu.

2-(5-(Hexyloxy)-2-iodophenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (25). 2-(5-(Hexyloxy)-2-iodophenyl)-1,3-dioxolane (**13**) and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc, 25:1) afforded **25** (345 mg, 81%) as a light yellow solid. R_f = 0.72 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 2929 (m), 1565 (w), 1376 (m), 1291 (m), 1147 (m), 1075 (m), 890 (m), 811 (m). ^1H NMR (400 MHz, CDCl_3): δ = 0.90 (t, 3J = 6.7 Hz, 3 H, 6'-H₃), 1.30 (s, 6 H, CH₃), 1.31–1.34 (m, 4 H, 5'-H₂, 4'-H₂), 1.35 (s, 6 H, CH₃), 1.40–1.47 (m, 2 H, 3'-H₂), 1.72–1.80 (m, 2 H, 2'-H₂), 3.93 (t, 3J = 6.5 Hz, 2 H, 1'-H₂), 5.96 (s, 1 H, 2-H), 6.62 (dd, 3J = 8.6, 3.1 Hz, 1 H, Ar), 7.21 (d, 3J = 3.1 Hz, 1 H, Ar), 7.65 (d, 3J = 8.6 Hz, 1 H, Ar). ^{13}C NMR (100 MHz, CDCl_3): δ = 14.5 (CH₃), 22.6 (CH₃), 23.0 (CH₂), 24.9 (CH₃), 26.1 (CH₂), 29.5 (CH₂), 32.0 (CH₂), 68.6 (CH₂), 83.4 (C), 86.1 (C), 102.8 (CH), 114.5 (CH), 117.7 (CH), 140.4 (CH), 141.9 (C), 160.0 (C). MS (EI, 70 °C): m/z (%) = 433 (20) [M^+], 432 (100) [M^+], 374 (19), 331 (35), 332 (11), 273 (27), 129 (24), 101 (17), 84 (32), 83 (31). HRMS ($^{12}\text{C}_{19}\text{H}_{29}^{16}\text{O}_3^{127}\text{I}$, EI): calcd. 432.1161 amu; found 432.1159 amu.

2-(Anthracen-9-yl)-4,4,5,5-tetramethyl-1,3-dioxolane (26). 2-(Anthracen-9-yl)-1,3-dioxolane (**14**) and pinacol were reacted according to the GP. Chromatography (hexanes/EtOAc, 25:1) afforded **26** (81 mg, 20%) as an orange solid. R_f = 0.59 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ (cm^{-1}) = 2974 (w), 1144 (w), 1101 (m), 731 (m). ^1H NMR (400 MHz, CDCl_3): δ = 1.49 (s, 6 H, CH₃), 1.52 (s, 6 H, CH₃), 7.30 (s, 1 H, CH), 7.41–7.53 (m, 4 H, Ar), 7.99 (d, 3J = 7.9 Hz, 2 H, Ar), 8.46 (s, 1 H, Ar), 8.94 (d, 3J = 9.1 Hz, 2 H, Ar). ^{13}C NMR (100 MHz, CDCl_3): δ = 23.1 (CH₃), 25.1 (CH₃), 82.7 (C), 98.4 (CH), 124.8 (CH), 125.2 (CH), 126.0 (CH), 126.8 (C), 129.2 (CH), 129.9 (CH), 130.5 (C), 131.7 (C). MS (EI, 100 °C): m/z (%) = 306 (39) [M^+], 190 (15), 189 (24), 177 (16), 178 (100), 179 (35), 84 (20). HRMS ($^{12}\text{C}_{21}\text{H}_{22}^{16}\text{O}_2$, EI): calcd. 306.1620 amu; found 306.1613 amu.

3-Bromobenzaldehyde (27); Deprotection of Pinacol Acetals. H₂O (2 mL) was placed in a flask, TfOH (2 mL) was carefully added, and the mixture was cooled to 0 °C. 2-(3-Bromophenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (**21**) (55.4 mg, 0.29 mmol) was added and the

mixture was stirred 2.5 h at 0 °C. Satd. NaHCO₃ soln (60 mL) was added and the mixture was extracted with EtOAc (3x50 mL). The combined organic layers were washed with brine (3x50 mL), dried (Na₂SO₄) and concentrated. The crude product was purified by flash column chromatography (silica gel, hexanes/EtOAc, 25:1) to yield aldehyde **27** (25.4 mg, 70%) as a colorless solid. *R*_f = 0.45 (hexanes/EtOAc, 3:1). ¹H NMR (300 MHz, CDCl₃): δ = 7.43 (t, ³*J* = 7.8 Hz, 1 H, Ar), 7.73–7.84 (m, 2 H, Ar), 8.02 (t, ³*J* = 1.7 Hz, 1 H, Ar), 9.96 (s, 1 H, CHO). These spectroscopic data are in full agreement with published data.⁷

⁷ Jiang, M.; Shi, M. *J. Org. Chem.* **2009**, 74, 2516-2520.

^1H and ^{13}C NMR Spectra**1) 4,4,5,5-Tetramethyl-2-phenyl-1,3-dioxolane (2)**

2) 2-(3-Chloropropyl)-4,4,5,5-tetramethyl-1,3-dioxolane (**17**)

3) 2-(2-Bromoethyl)-4,4,5,5-tetramethyl-1,3-dioxolane (**18**)

4) 4-Iodo-5-methyl-5-((4,4,5,5-tetramethyl-1,3-dioxolan-2-yl)-methyl)furan-2(5*H*)-one (**19**)

5) 2-Methyl-1-(4,4,5,5-tetramethyl-1,3-dioxolan-2-yl)but-3-yn-2-ol (**20**)

6) 2-(3-Bromophenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (**21**)

7) 2-(4-Fluorophenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (**22**)

8) 2-(Furan-2-yl)-4,4,5,5-tetramethyl-1,3-dioxolane (**23**)

9) 2-(2-Bromo-5-hexyloxyphenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (**24**)

10) 2-(5-(Hexyloxy)-2-iodophenyl)-4,4,5,5-tetramethyl-1,3-dioxolane (**25**)

11) 2-(Anthracen-9-yl)-4,4,5,5-tetramethyl-1,3-dioxolane (**26**)

12) 3-Bromobenzaldehyde (27)