Supporting Information
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A Rapid and Diastereoselective Synthesis of 2-Deoxy-2-iodo-α-glycosides and its Mechanism for Diastereoselectivity

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1. General Information.

NMR spectra were recorded with 400 or 600 MHz spectrometer for $^1$H NMR and 100 or 151 MHz for $^{13}$C{$^1$H} NMR using TMS as an internal standard. Chemical shifts ($\delta$) are reported relative to TMS ($^1$H) or CDCl$_3$ ($^{13}$C) and multiplicities are reported as follows: s (singlet); brs (broad singlet); d (doublet); t (triplet); q (quartet); dd (doublets of doublet); ddd (doublets of doublets of doublet); td (triplet of doublet); m (multiplets); ddt (doublet of doublet of triplet) and etc.. High resolution mass spectra (HRMS) were recorded on a QTOF mass analyzer with electrospray ionization (ESI).

2. Experimental data for compounds 2a-m

**General Procedures for Preparation of 2-Deoxy-2-iodo-α-glycosides:**

To a solution of glycal (1 mmol), alcohol (10 mmol) and PhI(OAc)$_2$ (1.2 mmol) in CH$_3$CN (4 mL) was added I$_2$ (0.6 mmol), the mixture was stirred at rt for 15 min. After addition of ethyl acetate (50 mL) to reaction mixture, the organic phase was washed with saturated Na$_2$S$_2$O$_3$, water and brine, dried over anhydrous Na$_2$SO$_4$ and concentrated. The residue was further purified by column chromatography to afford final product.

**Cyclohexyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-α-D-mannopyranoside (2a):** 438.5 mg, yield: 88%, colorless syrup. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.37 (t, $J$ = 9.7 Hz, 1H), 5.32 (s, 1H), 4.69 (dd, $J$ = 9.4, 4.3 Hz, 1H), 4.51 (dd, $J$ = 4.2, 0.9 Hz, 1H), 4.26 – 4.16 (m, 2H), 4.16 – 4.09 (m, 1H), 3.60 (ddd, $J$ = 13.1, 9.1, 3.8 Hz, 1H), 2.12 (s, 3H), 2.10 (s, 3H), 2.07 (s, 3H), 1.92 – 1.84 (m, 2H), 1.78 – 1.72 (m, 2H), 1.59 – 1.50 (m, 1H), 1.48 – 1.37 (m, 1H), 1.36 – 1.20 (m, 4H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 170.69, 169.87, 169.56, 99.59, 76.92, 69.20, 69.15, 67.86, 62.38, 33.19, 31.59, 30.70, 25.45, 24.10, 23.84, 20.98, 20.73, 20.68.
**Methyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-D-mannopyranoside (2b)**: 364.8 mg (α/β:4/1), yield: 85%, colorless syrup. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.39 (t, $J$ = 9.7 Hz, 1H), 5.31 (dd, $J$ = 11.1, 9.1 Hz, 0.25H), 5.10 (s, 1H), 4.99 (t, $J$ = 9.6 Hz, 0.25H), 4.65 (dd, $J$ = 9.5, 4.4 Hz, 1H), 4.51 (d, $J$ = 4.4 Hz, 1H), 4.02 (ddd, $J$ = 9.9, 4.8, 2.4 Hz, 1H), 3.90 (dd, $J$ = 11.2, 9.0 Hz, 0.25H), 3.76 (ddd, $J$ = 9.9, 4.6, 2.3 Hz, 0.25H), 3.59 (s, 0.75H), 3.43 (s, 3H), 2.14 (s, 3H), 2.11 (s, 0.75H), 2.10 (s, 3H), 2.07 (s, 3H), 2.06 (s, 0.75H), 2.03 (s, 0.75H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 170.69, 169.83, 169.49, 102.32, 69.03, 67.54, 62.27, 55.43, 29.27, 20.93, 20.74, 20.65.

**Ethyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-α-D-mannopyranoside (2c)**: 409.6 mg, yield: 92%, white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.39 (t, $J$ = 9.7 Hz, 1H), 5.20 (s, 1H), 4.68 (dd, $J$ = 9.5, 4.4 Hz, 1H), 4.54 (dd, $J$ = 4.3, 0.9 Hz, 1H), 4.25 (dd, $J$ = 12.2, 4.9 Hz, 1H), 4.16 (dd, $J$ = 12.2, 2.4 Hz, 1H), 4.05 (ddd, $J$ = 9.9, 4.7, 2.4 Hz, 1H), 3.75 (dq, $J$ = 9.7, 7.1 Hz, 1H), 3.56 (dq, $J$ = 9.7, 7.1 Hz, 1H), 2.13 (s, 3H), 2.10 (s, 3H), 2.07 (s, 3H), 1.25 (t, $J$ = 7.1 Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 170.71, 169.86, 169.51, 101.11, 69.12, 69.07, 67.68, 64.11, 62.30, 29.82, 20.97, 20.75, 20.67, 14.98.

**Propyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-D-mannopyranoside (2d)**: 412.8 mg, yield: 90%, white solid. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.37 (t, $J$ = 9.7 Hz, 1H), 5.18 (s, 1H), 4.66 (dd, $J$ = 9.4, 4.4 Hz, 1H), 4.54 (d, $J$ = 4.2 Hz, 1H), 4.23 (dd, $J$ = 12.2, 4.9 Hz, 1H), 4.16 (dd, $J$ = 12.2, 2.3 Hz, 1H), 4.03 (ddd, $J$ = 9.9, 4.7, 2.4 Hz, 1H), 3.63 (dt, $J$ = 9.4, 6.7 Hz, 1H), 3.44 (dt, $J$ = 9.5, 6.6 Hz, 1H), 2.13 (s, 3H), 2.10 (s, 3H), 2.06 (s, 3H), 1.69 – 1.59 (m, 2H), 0.95 (t, $J$ = 7.4 Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 170.63, 169.80, 169.47, 101.23, 70.19, 69.10, 69.03, 67.60, 62.26, 29.80, 22.62, 20.94, 20.72, 20.65, 10.59.
\( i \)-Propyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-\( \alpha \)-D-mannopyranoside (2e): \(^2\) 429.8 mg, yield: 98%, colorless syrup. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 5.37 (t, \( J = 9.7 \) Hz, 1H), 5.27 (s, 1H), 4.66 (dd, \( J = 9.4, 4.3 \) Hz, 1H), 4.48 (dd, \( J = 4.3, 1.2 \) Hz, 1H), 4.23 (dd, \( J = 12.2, 4.9 \) Hz, 1H), 4.17 – 4.06 (m, 2H), 3.92 (dt, \( J = 12.3, 6.2 \) Hz, 1H), 2.11 (s, 3H), 2.09 (s, 3H), 2.06 (s, 3H), 1.23 (d, \( J = 6.2 \) Hz, 3H), 1.17 (d, \( J = 6.1 \) Hz, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \( \delta \) 170.55, 169.72, 169.42, 99.58, 71.03, 69.04, 67.71, 62.27, 30.57, 23.02, 21.59, 20.91, 20.66, 20.61.

Butyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-\( \alpha \)-D-mannopyranoside (2f): \(^2\) 399.6 mg, yield: 85%, colorless syrup. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 5.38 (t, \( J = 9.7 \) Hz, 1H), 5.18 (s, 1H), 4.66 (dd, \( J = 9.5, 4.4 \) Hz, 1H), 4.57 – 4.52 (m, 1H), 4.24 (dd, \( J = 12.2, 4.9 \) Hz, 1H), 4.16 (dd, \( J = 12.2, 2.3 \) Hz, 1H), 4.03 (dd, \( J = 9.9, 4.8, 2.4 \) Hz, 1H), 3.69 (dt, \( J = 9.6, 6.7 \) Hz, 1H), 3.48 (dt, \( J = 9.6, 6.5 \) Hz, 1H), 2.13 (s, 3H), 2.10 (s, 3H), 2.07 (s, 3H), 1.65 – 1.55 (m, 2H), 1.46 – 1.34 (m, 2H), 0.95 (t, \( J = 7.4 \) Hz, 3H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \( \delta \) 170.69, 169.85, 169.51, 101.30, 69.15, 69.09, 68.41, 67.65, 62.32, 31.41, 29.80, 20.96, 20.74, 20.67, 19.30, 13.79.

\( i \)-Butyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-\( \alpha \)-D-mannopyranoside (2g): 376.4 mg, yield: 80%, colorless syrup. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 5.38 (t, \( J = 9.7 \) Hz, 1H), 5.17 (s, 1H), 4.66 (dd, \( J = 9.4, 4.4 \) Hz, 1H), 4.56 (d, \( J = 4.3 \) Hz, 1H), 4.23 (dd, \( J = 12.2, 4.9 \) Hz, 1H), 4.17 (dd, \( J = 12.2, 2.4 \) Hz, 1H), 4.02 (ddd, \( J = 9.9, 4.7, 2.4 \) Hz, 1H), 3.45 (dd, \( J = 9.2, 6.9 \) Hz, 1H), 3.25 (dd, \( J = 9.3, 6.4 \) Hz, 1H), 2.13 (s, 3H), 2.10 (s, 3H), 2.07 (s, 3H), 1.90 (td, \( J = 13.3, 6.6 \) Hz, 1H), 0.95 (dd, \( J = 6.7, 3.5 \) Hz, 6H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \( \delta \) 170.66, 169.83, 169.51, 101.40, 75.17, 69.18, 69.10, 67.62, 62.29, 29.73, 28.26, 20.95, 20.72, 20.65, 19.30, 19.22; HRMS calcd for C\(_{16}\)H\(_{25}\)O\(_8\)INa [M+Na\(^+\)]: 495.0486, found: 495.0495.
\[\text{-Butyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-\(\alpha\)-D-mannopyranoside (2h)}: \text{431.6 mg, yield: 94\%, light yellow solid.} \]
\[^1\text{H NMR (400 MHz, CDCl}_3\text{) }\delta \text{ 5.42 (s, 1H), 5.37 (t, }J=9.6\text{ Hz, 1H), 4.70 (dd, }J=9.4, 4.2\text{ Hz, 1H), 4.42 (dd, }J=4.2, 1.1\text{ Hz, 1H), 4.27 – 4.19 (m, 2H), 4.10 (q, }J=5.0\text{ Hz, 1H), 2.11 (s, 3H), 2.10 (s, 3H), 2.07 (s, 3H), 1.28 (s, 9H).} \]
\[^{13}\text{C NMR (101 MHz, CDCl}_3\text{) }\delta \text{ 170.63, 169.84, 169.51, 96.14, 76.82, 69.12, 68.68, 67.89, 62.41, 32.03, 28.36, 20.97, 20.70, 20.66.}\]

\[\text{Menthyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-\(\alpha\)-D-mannopyranoside (2i): 455.7 mg, yield: 82\%, white solid.} \]
\[^1\text{H NMR (400 MHz, CDCl}_3\text{) }\delta \text{ 5.35 (t, }J=9.3\text{ Hz, 1H), 5.26 (s, 1H), 4.67 (dd, }J=9.4, 4.3\text{ Hz, 1H), 4.53 (dd, }J=4.2, 1.2\text{ Hz, 1H), 4.25 – 4.12 (m, 3H), 3.38 (td, }J=10.7, 4.3\text{ Hz, 1H), 2.12 (s, 3H), 2.10 (s, 3H), 2.07 (s, 3H), 1.69 – 1.61 (m, 2H), 1.44 – 1.26 (m, 3H), 1.10 – 0.82 (m, 10H), 0.78 (d, }J=7.0\text{ Hz, 3H).} \]
\[^{13}\text{C NMR (101 MHz, CDCl}_3\text{) }\delta \text{ 170.69, 169.91, 169.55, 103.05, 82.79, 69.33, 69.14, 67.89, 62.60, 48.27, 42.49, 34.17, 31.62, 30.29, 29.69, 25.97, 23.34, 22.29, 20.96, 20.92, 20.76, 20.68, 16.34.}\]

\[\text{-Propyl 3,4-di-O-acetyl-2-deoxy-2-iodo-D-lyxopyranoside (2j): 316.2 mg (\(\alpha/\beta\):4/3), yield: 82\%, colorless syrup.} \]
\[^1\text{H NMR (400 MHz, CDCl}_3\text{) }\delta \text{ 5.30 (dd, }J=11.0, 9.1\text{ Hz, 1H), 5.01 – 4.93 (m, 2.5H), 4.93 – 4.85 (m, 1H), 4.63 (d, }J=8.7\text{ Hz, 1H), 4.43 (dd, }J=4.9, 3.4\text{ Hz, 0.75H), 4.12 (dd, }J=11.5, 5.5\text{ Hz, 1H), 4.03 – 3.78 (m, 4H), 3.37 (dd, }J=11.5, 10.3\text{ Hz, 1H), 2.15 (s, 2.25H), 2.11 (s, 3H), 2.09 (s, 2.25H), 2.02 (s, 3H), 1.26 – 1.23 (m, 8.25H), 1.19 (d, }J=6.1\text{ Hz, 2.25H).} \]
\[^{13}\text{C NMR (101 MHz, CDCl}_3\text{) }\delta \text{ 169.73, 169.55, 169.32, 102.15, 99.12, 75.04, 72.85, 71.41, 70.17, 69.72, 68.30, 62.87, 61.82, 30.11, 29.49, 26.87, 23.28, 23.21, 21.54, 20.95, 20.87, 20.70.} \]
\[^{\text{HRMS ealed for C}_{12}\text{H}_{19}\text{O}_{6}\text{INa [M+Na}^+\text{]: 409.0119, found: 409.0111.}\]

\[\text{-Propyl 3,4-di-O-acetyl-2-deoxy-2-iodo-\(\alpha\)-D-arabinopyranoside (2k): 337.2 mg, yield: 87\%, white solid.} \]
\[^1\text{H NMR (400 MHz, CDCl}_3\text{) }\delta \text{ 5.53 (s, 1H), 5.19 – 5.04 (m, 1H), 4.87 (d, }J=7.5\text{ Hz, 1.19 (d, }J=6.1\text{ Hz, 2.25H).} \]
\[^{13}\text{C NMR (101 MHz, CDCl}_3\text{) }\delta \text{ 169.73, 169.55, 169.32, 102.15, 99.12, 75.04, 72.85, 71.41, 70.17, 69.72, 68.30, 62.87, 61.82, 30.11, 29.49, 26.87, 23.28, 23.21, 21.54, 20.95, 20.87, 20.70.} \]
\[^{\text{HRMS ealed for C}_{12}\text{H}_{19}\text{O}_{6}\text{INa [M+Na}^+\text{]: 409.0119, found: 409.0111.}\]

\[\text{-Propyl 3,4-di-O-acetyl-2-deoxy-2-iodo-\(\alpha\)-D-arabinopyranoside (2l): 337.2 mg, yield: 87\%, white solid.} \]
\[^1\text{H NMR (400 MHz, CDCl}_3\text{) }\delta \text{ 5.53 (s, 1H), 5.19 – 5.04 (m, 1H), 4.87 (d, }J=7.5\text{ Hz, 1.19 (d, }J=6.1\text{ Hz, 2.25H).} \]
\[^{13}\text{C NMR (101 MHz, CDCl}_3\text{) }\delta \text{ 169.73, 169.55, 169.32, 102.15, 99.12, 75.04, 72.85, 71.41, 70.17, 69.72, 68.30, 62.87, 61.82, 30.11, 29.49, 26.87, 23.28, 23.21, 21.54, 20.95, 20.87, 20.70.} \]
\[^{\text{HRMS ealed for C}_{12}\text{H}_{19}\text{O}_{6}\text{INa [M+Na}^+\text{]: 409.0119, found: 409.0111.}\]

\[\text{-Propyl 3,4-di-O-acetyl-2-deoxy-2-iodo-\(\alpha\)-D-arabinopyranoside (2l): 337.2 mg, yield: 87\%, white solid.} \]
\[^1\text{H NMR (400 MHz, CDCl}_3\text{) }\delta \text{ 5.53 (s, 1H), 5.19 – 5.04 (m, 1H), 4.87 (d, }J=7.5\text{ Hz, 1.19 (d, }J=6.1\text{ Hz, 2.25H).} \]
\[^{13}\text{C NMR (101 MHz, CDCl}_3\text{) }\delta \text{ 169.73, 169.55, 169.32, 102.15, 99.12, 75.04, 72.85, 71.41, 70.17, 69.72, 68.30, 62.87, 61.82, 30.11, 29.49, 26.87, 23.28, 23.21, 21.54, 20.95, 20.87, 20.70.} \]
\[^{\text{HRMS ealed for C}_{12}\text{H}_{19}\text{O}_{6}\text{INa [M+Na}^+\text{]: 409.0119, found: 409.0111.}\]
Hz, 1H), 4.16 (dd, J = 7.5, 3.2 Hz, 1H), 4.02 – 3.90 (m, 2H), 3.81 (dd, J = 11.3, 9.4 Hz, 1H), 2.20
(s, 3H), 2.03 (s, 3H), 1.24 (dd, J = 8.1, 6.3 Hz, 6H). 13C NMR (101 MHz, CDCl3) δ 169.68,
169.54, 99.64, 72.41, 70.23, 66.67, 61.76, 27.59, 23.29, 21.65, 20.80, 20.71. HRMS calcd for
C12H20O6IH [M+H+]: 387.0299, found: 387.0302.

i-Propyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-α-D-talopyranoside (2l): 437.0 mg, yield: 95%,
white solid. 1H NMR (400 MHz, CDCl3) δ 5.42 (s, 1H), 5.41 – 5.38 (m, 1H), 4.98 – 4.93 (m, 1H),
4.38 (td, J = 6.5, 1.7 Hz, 1H), 4.24 (d, J = 5.1 Hz, 1H), 4.19 (dd, J = 6.6, 3.2 Hz, 1H), 3.94 (dt, J =
12.3, 6.1 Hz, 1H), 2.20 (s, 3H), 2.09 (s, 3H), 2.06 (s, 3H), 1.24 (d, J = 6.2 Hz, 3H), 1.19 (d, J = 6.1
Hz, 3H). 13C NMR (101 MHz, CDCl3) δ 170.45, 170.10, 169.54, 101.05, 71.05, 66.70, 65.55,
65.38, 62.11, 23.02, 22.50, 21.69, 21.00, 20.89, 20.66. HRMS calcd for C15H23O8INa [M+Na+]:
481.0330, found: 481.0338.

i-Propyl 3,6-di-O-acetyl-2-deoxy-2-iodo-4-O-(2,3,4,6-tetra-O-acetyl-α-D-glucopyranosyl)-α-D-mannopyranoside (2m): 542 mg, yield: 73%, white solid. 1H NMR (400 MHz, CDCl3) δ 5.55 (d, J = 3.9
Hz, 1H), 5.41 (t, J = 10.0 Hz, 1H), 5.22 (s, 1H), 5.08 (t, J = 9.8 Hz, 1H), 4.91 (dd, J = 10.4, 3.9 Hz,
1H), 4.55 (dd, J = 7.7, 4.2 Hz, 1H), 4.51 – 4.47 (m, 1H), 4.43 (d, J = 12.1 Hz, 1H), 4.27 (ddd, J =
21.5, 12.3, 3.8 Hz, 2H), 4.17 – 4.00 (m, 4H), 3.92 (td, J = 12.4, 6.2 Hz, 1H), 2.15 (s, 3H), 2.11 (s,
6H), 2.04 (s, 6H), 2.02 (s, 3H), 1.28 (d, J = 6.2 Hz, 3H), 1.19 (d, J = 6.1 Hz, 3H). 13C NMR (101
MHz, CDCl3) δ 170.41, 170.34, 170.05, 169.89, 169.65, 169.28, 99.36, 95.73, 72.46, 72.35, 71.26,
70.02, 69.46, 68.95, 68.34, 67.91, 62.98, 61.35, 30.34, 23.03, 21.62, 21.13, 20.70, 20.56, 20.51,
3. $^1$H NMR and $^{13}$C NMR spectra for 2a-m

Cyclohexyl 3,4,6-tri-$O$-acetyl-2-deoxy-2-iodo-$\alpha$-$D$-mannopyranoside (2a)
Methyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-\(\alpha\)-mannopyranoside (2b)
Ethyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-α-D-mannopyranoside (2c)
Propyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-D-mannopyranoside (2d)
\[ \textit{i-Propyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-\textalpha-D-mannopyranoside (2e)} \]
Butyl 3,4,6-tri-O-acetyl-2-deoxy-2-ido-α-D-mannopyranoside (2f)
i-Butyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-α-D-mannopyranoside (2g)

$^1$H NMR (400 MHz, CDCl$_3$)

$^13$C NMR (101 MHz, CDCl$_3$)
$t$-Butyl 3,4,6-tri-$O$-acetyl-2-deoxy-2-iodo-$\alpha$-$D$-mannopyranoside (2h)

$^1$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (101 MHz, CDCl$_3$)
Mentyl 3,4,6-tri-\(O\)-acetyl-2-deoxy-2-iodo-\(\alpha\)-\(D\)-mannopyranoside (2i)
$i$-Propyl 3,4-di-$O$-acetyl-2-deoxy-2-ido-$d$-lyxopyranoside (2j)

$\text{H NMR (400 MHz, CDCl}_3$)

$\text{C NMR (101 MHz, CDCl}_3$)
i-Propyl 3,4-di-O-acetyl-2-deoxy-2-iodo-α-D-arabinopyranoside (2k)

$^1$H NMR (400 MHz, CDCl$_3$)

$^{13}$C NMR (101 MHz, CDCl$_3$)
i-Propyl 3,4,6-tri-O-acetyl-2-deoxy-2-iodo-α-D-talopyranoside (2l)
i-Propyl
3,6-di-O-acetyl-2-deoxy-2-iodo-4-O-(2,3,4,6-tetra-O-acetyl-α-D-glucopyranosyl)-α-D-mannopyranoside (2m)
4. Reference
