Supporting information

Efficient Synthesis of Fused Perhydrofuro [2, 3-b] pyrans (and furans) by Ring Opening of 1, 2-Cyclopropanated Sugar Derivatives

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Contents:

1. General experimental procedure and spectral data	S2- S15
2. ^1H and ^{13}C spectra of all new compounds	S16- S36

Experimental Procedure:

Physical properties and spectral measurements: All glassware were oven dried before starting a reaction. N-Iodosuccinamide was purchased from Lancaster India and used as such without crystallization. Solvents like THF, CH₃CN, and Et₂O were purified as mentioned in “Purification of Laboratory Chemicals” By Perrin & Armarego, Pergamon Press, Third Edition 1988. ¹H and ¹³C NMR spectra were recorded on 300 MHz NMR (300 MHz, ¹H and 75 MHz, ¹³C) or 400 MHz NMR (400 MHz, ¹H and 100 MHz, ¹³C) spectrometers. ¹H-¹H COSEY and ¹H-¹H NOESY were recorded on a 500 MHz NMR spectrometer. Chemical shifts (δ) are reported in parts per million downfield from the internal reference, tetramethylsilane for ¹H and CDCl₃ for ¹³C.

General procedure for the reduction of cyclopropylcarboxylates using LiAlH₄:

To a solution of lithium aluminium hydride (0.018 g, 0.5mmol) in Et₂O (10 ml) was slowly added a solution of appropriate cyclopropylcarboxylate ester (0.5 mmol) in Et₂O (10 ml) at 0 °C under nitrogen. After 1h the reaction mixture was quenched with water. The solution was then filtered under vacuum through a Celite pad and the organic layer was separated, and dried over anhydrous Na₂SO₄. The filtrate was concentrated and the crude product was purified by flash chromatography on silica gel (230-400 mesh) column using ethyl acetate and petroleum ether of appropriate composition to obtain the corresponding cyclopropyl methanols in good yield.

Compound 3:

Physical appearance	colorless gummy solid
Yield:	0.195 g, 90 %
$[\alpha]_D^{23}$	+ 36 ($c = 2.0$, CHCl ₃)
R _f (AcOEt: pet. ether 1:4)	0.12
IR (Neat) cm ⁻¹ :	3164, 2867, 1496, 1454, 1193, 737, 698.

^1H NMR (CDCl_3 , 300 MHz): δ 7.38-7.22 (m, 15H), 4.95-4.78 (m, 9H), 3.76 (td, $J_1 = 3.6$ Hz, $J_2 = 6.3$ Hz, 1H), 3.69 - 3.58 (m, 2H), 3.53 - 3.33 (m, 8H), 1.67(bs, 1H), 1.37(dt, $J_1 = 2.1$ Hz, $J_2 = 5.7$ Hz, 1H), 0.96-0.91 (m, 1H).

^{13}C NMR (CDCl_3 , 75 MHz): δ 128.4, 128.36, 128.32, 128.09, 127.93, 127.75, 127.7, 127.65, 127.62, 78.8, 76.4, 76.3, 73.4, 73.2, 71.3, 69.7, 63.1, 53.7, 26.4, 19.8.

High resolution MS: Calculated for $\text{C}_{29}\text{H}_{32}\text{O}_5 + \text{Na}$: 483.2147,
Found: 483.2143

Compound 7:

Physical appearance	colorless gummy solid
Yield	0.176 g, 72 %
$[\alpha]_D^{23}$	+10 ($c = 1.0$, CHCl_3)
R_f (AcOEt: pet. ether 1:4)	0.25
IR (Neat) cm^{-1} :	3087, 2927, 2855, 1466, 1454, 1295, 1193, 774, 697.
^1H NMR (CDCl_3 , 300 MHz):	δ 7.41 - 7.26 (m, 10H), 4.73 (each d, 4H, benzyl), 3.80(dd, $J_1 = 4.5$ Hz, $J_2 = 11.1$ Hz, 1H), 3.72 (dd, $J_1 = 3.3$ Hz, $J_2 = 11.1$ Hz, 2H), 3.64-3.60 (m, 3H), 3.46 (dd, $J_1 = 2.4$ Hz, $J_2 = 7.2$ Hz, 2H), 3.40-3.36 (m, 1H), 1.35-1.25 (m, 2H), 0.98 (s, 9H), 0.06 (s, 3H), 0.04 (s, 3H)
^{13}C NMR (CDCl_3 , 75 Hz):	138.5, 138.3, 128.4, 128.3, 127.9, 127.78, 127.7, 127.6, 79.4, 78.7, 76.2, 73.2, 71.4, 63.4, 54.1, 27.1, 25.9, 20.2, 18.2, -5.3, -5.4
High resolution MS:	Calculated for $\text{C}_{28}\text{H}_{40}\text{O}_5\text{Si} + \text{Na}$: 631.2856, Found: 631.2849

Compound 10:

Physical appearance	colorless oil
Yield:	0.159 g, 94 %
$[\alpha]_D^{24}$	- 11.7 ($c = 1.0$, CHCl_3)
R_f (AcOEt: pet. ether 1:4)	0.1
IR (Neat) cm^{-1} :	3401, 2872, 1496, 1454, 1093, 910, 736, 698.
^1H NMR (CDCl_3 , 300 MHz):	7.38-7.26(m, 10H), 4.77 (d, 2H, benzyl), 4.52 (d, 2H, benzyl), 3.69 (dd, $J_1 = 2.7$ Hz, $J_2 = 6.6$ Hz, 1H), 3.65 (d, $J = 6.6$ Hz, 1H), 3.38 (d, $J = 6.9$ Hz, 3H), 3.22 (t, $J = 6.3$ Hz, 1H), 2.16 (bs, 1H), 1.34 (dq, $J_1 = 1.8$ Hz, $J_2 = 5.7$ Hz, 1H), 1.27 (d, $J = 6.6$ Hz, 3H), 0.95 (dt, $J_1 = 2.7$ Hz, $J_2 = 6.3$ Hz, 1H).
^{13}C NMR (CDCl_3 , 75 MHz):	δ 138.3, 138.31, 128.4, 128.3, 128.2, 127.9, 127.8, 127.78, 127.71, 127.6, 83.3, 80.8, 78.4, 73.4, 71.7, 71.3, 63.1, 53.8, 25.0, 19.1, 18.5.
High resolution MS:	Calculated for $\text{C}_{22}\text{H}_{26}\text{O}_4 + \text{Na}$: 377.1729, Found: 377.1750

Compound 13:

Physical appearance	colorless gummy solid
Yield:	0.195 g, 90 %
R_f (AcOEt: pet. ether 3:7)	0.15
IR (Neat) cm^{-1} :	3159, 2858, 1488, 1450, 1156, 735, 696.

^1H NMR (CDCl_3 , 300 MHz):	δ 7.35 - 7.24 (m, 15H), 4.84 - 4.22 (each d, 6H, benzyl), 3.72 (d, J = 6.3 Hz, 2H), 3.66 (q, J = 2.7 Hz, 2H), 3.56 (d, J = 8.1 Hz, 1H), 3.47 - 3.31 (m, 3H), 1.83 (bs, 1H), 1.13 (dd, J_1 = 3 Hz, J_2 = 6 Hz, 1H), 1.06 (ddd, J_1 = 2.4 Hz, J_2 = 6.9 Hz, J_3 = 13.2 Hz, 1H).
^{13}C NMR (CDCl_3 , 75 Hz):	δ 138.5, 138.3, 138.1, 128.36, 128.31, 128.24, 127.80, 127.57, 127.47, 74.9, 73.3, 73.1, 71.2, 69.1, 63.4, 53.2, 26.4, 19.1
High resolution MS:	Calculated for $\text{C}_{29}\text{H}_{32}\text{O}_5 + \text{Na}$: 483.2147, Found: 483.2141

Compound 20:

Physical appearance	colorless gummy solid
Yield:	0.157 g, 98 %
$[\alpha]_D^{22}$	- 5.0 (c = 1.0, CHCl_3)
R_f (AcOEt: pet. ether 2:3)	0.25
IR (Neat) cm^{-1} :	3170, 2875, 1496, 1097, 735, 695
^1H NMR (CDCl_3 , 300 MHz):	7.35 - 7.26 (m, 5H), 4.64 (AB, 2H), 4.35 (q, J = 6.3 Hz, 1H), 4.14 (d, J = 4.8 Hz, 1H), 4.05 (dd, J_1 = 2.1 Hz, J_2 = 6.6 Hz, 1H), 3.97 - 3.92 (m, 2H), 3.70 (td, J_1 = 5.4 Hz, J_2 = 1.5 Hz, 1H), 3.44 (dd, J_1 = 6.9, J_2 = 2.11 Hz, 1H), 3.32 (dd, J_1 = 6.9 Hz, J_2 = 11.4 Hz, 1H), 1.65 (t, J = 6.9 Hz, 1H), 1.41 (m, 4H), 1.35(s, 3H).
^{13}C NMR (CDCl_3 , 75 Hz):	δ 138.08, 128.2, 127.6, 127.5, 108.5, 82.2, 79.5, 72.9, 72.0, 66.6, 61.9, 30.8, 26.5, 25.2, 24.0, 21.3
High resolution MS:	Calculated for $\text{C}_{18}\text{H}_{24}\text{O}_5 + \text{Na}$: 343.1521, Found: 343.1520.

General procedure for NIS- mediated ring opening of cyclopropyl methanols:

To a stirred solution of the appropriate cyclopropyl methanol (0.5 mmol) in dichloromethane (7ml) under nitrogen was added NIS (0.111 g, 0.5 mmol) and the stirring was continued for 36 h. Removal of the solvent under reduced pressure followed by flash chromatographic purification on silica gel (230-400 mesh) column using ethyl acetate and petroleum ether of appropriate composition gave the corresponding iodides in good yield.

Compound 4:

Physical appearance	pale pink gummy solid
Yield	0.186 g, 62 %
$[\alpha]_D^{23}$	- 13.1 ($c = 1.0$, CHCl_3)
R_f (AcOEt: pet. ether 1:9)	0.55
IR (Neat) cm^{-1} :	2921, 2851, 1496, 1454, 1362, 1215, 736, 697
^1H NMR (CDCl_3 , 300 MHz):	δ 7.37-7.18 (m, 15H), 5.70 (d, $J = 5.1$ Hz, 1H), 4.76 (d, AB, 1H), 4.65- 4.60 (m, 1H), 4.50 (AB, 2H), 4.41 - 4.36 (m, 1H), 4.10 - 4.01 (m, 2H), 3.82 -3.64 (m, 4H), 3.50 (t, $J = 6.3$ Hz, 1H), 2.65 - 2.60 (m, 1H).
^{13}C NMR (CDCl_3 , 75 MHz):	δ 137.9, 137.8, 137.6, 128.5, 128.4, 128.3, 128.0, 127.9, 127.8, 127.6, 100.3, 76.5, 76.4, 75.8, 73.5, 73.4, 73.2, 71.7, 68.6, 53.4, 18.9.
High resolution MS:	Calculated for $\text{C}_{29}\text{H}_{31}\text{IO}_5 + \text{Na}$: 609.1114, Found: 609.1112

Compound 8:

Physical appearance	colorless oil
Yield	0.177 g, 57 %
R _f (AcOEt: pet. ether 1:9)	0.64
IR (Neat) cm ⁻¹ :	2925, 2855, 1456, 1454, 1215, 735, 697.
¹ H NMR (CDCl ₃ , 400 MHz):	δ 7.45-7.28 (m, 10H), 5.68 (d, <i>J</i> = 5.2 Hz, 2H), 4.80, 4.65 (each d, 4H, benzyl), 4.39 (dd, <i>J</i> ₁ = 4.8 Hz, <i>J</i> ₂ = 10Hz, 1H), 3.82 (m, 2H), 3.69 (m, 2H), 3.48 (d, <i>J</i> = 6.4 Hz, 1H), 2.50 (m, 1H), 0.92 (s, 9H), 0.09 (s, 6H).
¹³ C NMR (CDCl ₃ , 100 Hz):	138.1, 138.0, 128.5, 128.0, 100.3, 76.2, 75.8, 73.7, 73.3, 62.2, 53.9, 25.9, 19.4, -5.3.
High resolution MS:	Calculated for C ₂₈ H ₃₉ IO ₅ Si + Na: 633.1490, Found: 633.1492.

Compound 11:

Physical appearance	colorless oil
Yield:	0.137 g, 58 %
R _f (AcOEt: pet. ether 1:9):	0.45
IR (Neat) cm ⁻¹ :	2852, 1463, 1377, 1101, 801, 738, 698.
¹ H NMR (CDCl ₃ , 400 MHz):	δ 7.29-7.52 (m, 10H), 5.62 (d, <i>J</i> = 5.2 Hz, 1H), 4.76 (d, 2H, benzyl), 4.65 (d, 1H, benzyl), 4.54 (d, 1H, benzyl), 4.39 (dd, <i>J</i> ₁ = 5.2 Hz, <i>J</i> ₂ = 8.4 Hz, 1H), 4.09 - 4.02 (m, 2H), 3.77 (dq, <i>J</i> ₁ = 1.6 Hz, <i>J</i> ₂ = 6 Hz, 1H), 3.49 (t, <i>J</i> = 6 Hz, 1H), 3.25 (dd, <i>J</i> ₁ = 6 Hz, <i>J</i> ₂ = 8.2 Hz, 1H), 2.61 (dd, <i>J</i> ₁ = 5.2 Hz, <i>J</i> ₂ = 11.3 Hz, 1H), 1.32 (d, <i>J</i> = 8.4 Hz, 3H).

^{13}C NMR (CDCl_3 , 100 Hz): δ 137.87, 137.69, 128.8, 128.6, 128.5, 128.14, 128.04, 127.9, 100.0, 82.1, 77.32, 76.68, 75.82, 73.79, 73.2, 68.2, 53.4, 19.2, 18.2

High resolution MS: Calculated for $\text{C}_{22}\text{H}_{25}\text{IO}_4 + \text{Na}$: 503.0695,
Found: 503.0693

Compound 14:

14

Physical appearance: pale pink oil

Yield: 0.187 g, 66 %

R_f (AcOEt: pet. ether 1:9): 0.45

IR (Neat) cm^{-1} : 2860, 1480, 1451, 1362, 1210, 735, 694.

^1H NMR (CDCl_3 , 300 MHz): δ 7.44-7.25 (m, 15 H), 5.81 (d, $J = 4.8$ Hz, 1H), 4.86, 4.71, 4.62, 4.46, 4.34 (each d, 6H, benzyl), 4.18 - 4.14 (m, 2H), 3.95 - 3.91 (m, 2H), 3.67-3.59 (m, 2H), 3.15 (dd, $J_1 = 1.05$ Hz, $J_2 = 10.35$ Hz, 1H), 2.82 - 2.77 (m, 1H).

^{13}C NMR (CDCl_3 , 75 Hz): δ 138.21, 137.7, 137.2, 128.6, 128.4, 128.3, 128.2, 128.16, 128.11, 127.9, 127.8, 127.7, 101.2, 77.4, 75.6, 74.1, 73.5, 71.6, 70.8, 69.1, 68.2, 49.8, 20.7.

High resolution MS: Calculated for $\text{C}_{29}\text{H}_{31}\text{IO}_5 + \text{Na}$: 609.1114,
Found: 609.1112

Compound 21:

21

Physical appearance	pale pink oil
Yield:	0.149 g, 65 %
R _f (AcOEt: pet. ether 1:4)	0.55
IR (Neat) cm ⁻¹ :	2876, 1455, 1097, 735, 693
¹ H NMR (CDCl ₃ , 300 MHz):	δ 7.37- 7.26 (m, 5 H) 5.88 (d, <i>J</i> = 5.4 Hz, 1H), 4.66, 4.56 (each d, 2H, benzyl), 4.35 (td, <i>J</i> ₁ = 1.9 Hz, <i>J</i> ₂ = 5.7 Hz, 1H), 4.19 (dd, <i>J</i> ₁ = 5.7 Hz, <i>J</i> ₂ = 9.9 Hz, 1H), 4.10 (dd, <i>J</i> ₁ = 6 Hz, <i>J</i> ₂ = 8.7 Hz, 1H), 4.03 - 3.90 (m, 4H), 3.84 (dd, <i>J</i> ₁ = 6 Hz, <i>J</i> ₂ = 12 Hz, 1H), 3.25 (dd, <i>J</i> = 5.7 Hz, 1H), 1.40 (s, 3H), 1.37 (s, 3H).
¹³ C NMR (CDCl ₃ , 75 Hz):	δ 137.2, 128.4, 127.9, 127.8, 109.0, 108.1, 82.6, 81.6, 77.4, 76.5, 72.6, 71.5, 67.1, 61.9, 26.7, 25.3, 16.7
High resolution MS:	Calculated for C ₁₈ H ₂₃ IO ₅ + Na: 469.0488. Found: 469.0479.

Procedure for the synthesis of furofuryl glycal **22**:

DBU (0.04 g, 0.26 mmol) was added to a solution of **21** (0.012 g, 0.026 mmol) in THF (2 ml) under argon. The stirring was continued until the reaction mixture showed the absence of starting material on TLC (~14hr). The reaction mixture was diluted with Et₂O (10 ml) and water (5 ml). The organic layer separated and aqueous layer was extracted twice with Et₂O. The combined organic layer was dried over anhydrous Na₂SO₄ and concentrated, and the residue was purified by flash chromatography using 10 % petroleum ether: ethyl acetate as a solvent to get compound **22** (0.0066 g, 78% yield) as a colorless oil.

Compound **22**:

Physical appearance	colorless oil
Yield:	0.0066 g, 78 %
R _f (AcOEt: pet. ether 1:9)	0.55

^1H NMR (CDCl_3 , 400 MHz):	δ 7.36-7.26 (m, 5H), 6.45 (t, $J = 2.6$ Hz, 1H), 6.16 (d, $J = 6$ Hz, 1H), 4.79 (t, $J = 2.6$ Hz, 1H), 4.65 (AB, 2H), 4.39 (m, 1H), 4.13(dd, 1H), 4.02 (dd, $J_1 = 6$ Hz, $J_2 = 11.5$ Hz, 1H), 3.97 (dd, $J_1 = 3$ Hz, $J_2 = 7.5$ Hz, 1H), 3.92 (d, $J = 3$ Hz, 1H), 3.59 (qd, $J_1 = 2.3$ Hz, $J_2 = 2.3$ Hz, 1H), 1.41 (s, 3H), 1.38 (s, 3H).
^{13}C NMR (CDCl_3 , 100 Hz):	δ 147.7, 137.9, 128.4, 127.7, 127.5, 108.9, 108.90, 98.7, 82.3, 76.6, 72.7, 71.9, 67.4, 53.3, 26.7, 25.4.
High resolution MS:	Calculated for $\text{C}_{18}\text{H}_{22}\text{O}_5 + \text{Na}$: 341.1365, Found: 341.1360.

General procedure for the hydrolysis of cyclopropyl carboxylate esters using LiOH:

A solution of LiOH (0.024 g, 1 mmol) and appropriate cyclopropyl ester (0.5 mmol) in MeOH/THF 1:2 (10 ml) was stirred at 25 °C, until TLC indicated complete conversion of the ester (*ca.* 24 h). After completion of the reaction, the solvent was removed *in vacuo* and the residue was diluted with water (15 ml) and extracted once with Et_2O (5 ml). The aq. phase was acidified with 1 N HCl and the extracted with Et_2O (2×10 ml). The organic phase was dried (Na_2SO_4) and filtered. The filtrate was evaporated to yield the corresponding crude carboxylic acid which was pure enough to use as such in the next reaction.

Compound 23:

Physical appearance	colorless gummy solid
Yield:	0.234 g, 99 %
R_f (AcOEt: pet. ether 3:7)	0.15 (streak)
IR (Neat) cm^{-1} :	3395, 2870, 1692, 1454, 737, 696.
^1H NMR (CDCl_3 , 300 MHz):	δ 7.32 - 7.21 (m, 15H), 4.71, 4.59, 4.56 (each d, 6H, benzyl), 4.01 (d, $J = 7.5$ Hz, 1H), 3.78 (d, $J = 6$ Hz, 1H), 3.68 (dd, $J_1 = 5.4$ Hz, $J_2 = 8.1$ Hz, 2H), 3.59(dd, J_1

	= 6 Hz, $J_2 = 6.3$ Hz, 2H), 1.98 (d, $J = 5.7$ Hz, 1H), 1.84 (d, $J = 6.6$ Hz, 1H).
^{13}C NMR (CDCl_3 , 75 Hz):	δ 177.0, 137.8, 137.83, 137.5, 128.3, 128.2, 127.7, 127.73, 127.65, 127.60, 127.5, 77.4, 76.1, 74.2, 73.25, 73.2, 71.37, 68.9, 58.08, 25.2, 24.2.
High resolution MS:	Calculated for $\text{C}_{29}\text{H}_{30}\text{O}_6 + \text{Na}$: 497.1940, Found: 497.1939.

Compound **28**:

Physical appearance	colorless gummy solid
Yield:	0.092 g, 99 %
$[\alpha]_{\text{D}}^{23}$	-4.3 ($c = 0.42$, CHCl_3)
R_f (AcOEt: pet. ether 2:3)	0.25 (streak)
IR (Neat) cm^{-1} :	3367, 2870, 1689, 1454, 1097, 737, 696.
^1H NMR (CDCl_3 , 400 MHz):	δ 7.30 - 7.20 (m, 5H), 4.70 (d, $J = 8.7$ Hz, 1H), 4.59 (d, $J = 9$ Hz, 1H), 4.41 - 4.31 (m, 1H), 4.20 (d, $J = 3.6$ Hz, 1H), 4.06 (td, $J_1 = 1.5$ Hz, $J_2 = 4.8$ Hz, 1H), 3.95 (td, $J_1 = 1.8$ Hz, $J_2 = 4.5$ Hz, 1H), 3.70 (t, $J = 4.2$ Hz, 1H), 1.4 - 2.38 (m, 1H), 1.88 (d, $J = 2.4$ Hz, 1H), 1.41 (s, 3H), 1.36 (s, 3H).
^{13}C NMR (CDCl_3 , 100 Hz):	δ 176.4, 137.5, 128.4, 127.9, 127.7, 108.8, 82.0, 78.7, 72.6, 66.5, 65.6, 29.1, 26.6, 25.2, 21.1.
High resolution MS:	Calculated for $\text{C}_{18}\text{H}_{22}\text{O}_6 + \text{Na}$: 357.1314, Found: 357.1310.

General procedure for NIS- mediated ring opening reaction of cyclopropane carboxylic acids:

To mixture of an appropriate cyclopropyl carboxylic acid (0.5 mmol) and 4 °A MS (0.050 g) in acetonitrile (7 ml) under nitrogen was added NIS (0.112 g , 0.5 mmol) and the stirring was continued until the reaction mixture showed the absence of starting material on TLC(~10 hr). Removal of the solvent under reduced pressure followed by flash chromatographic purification on silica gel (230-400 mesh) column using ethyl acetate and petroleum ether of appropriate composition gave the corresponding iodo- lactones in excellent yield.

Compound 5:

Physical appearance	pale violet oil
Yield:	0.268 g, 92 %
R _f (AcOEt: pet. ether 1:9)	0.43
IR (Neat) cm ⁻¹ :	2826, 1776, 1496, 1456, 735, 698
¹ H NMR (CDCl ₃ , 300 MHz):	δ 7.47 - 7.17 (m, 15H), 5.99 (d, <i>J</i> = 4.2 Hz, 1H), 4.85, 4.74, 4.61, 4.58, 4.56, 4.52 (each d, 6H, benzyl), 4.04 (d, <i>J</i> = 1.2 Hz, 1H), 3.82 - 3.77 (m, 3H), 3.68 (d, <i>J</i> = 4.8 Hz, 1H), 3.31-3.26 (m, 1H), 2.64 (ddd, <i>J</i> ₁ = 1.2 Hz, <i>J</i> ₂ = 4.5 Hz, <i>J</i> ₃ = 9 Hz, 1H).
¹³ C NMR (CDCl ₃ , 75 Hz):	δ 170, 137.5, 137.4, 137.2, 128.7, 128.4, 127.4, 1127.9, 127.94, 127.85, 127.80, 100.9, 78.7, 76.3, 74.8, 74.5, 73.5, 73.3, 67.6, 52.4, 12.8.
High resolution MS:	Calculated for C ₂₉ H ₂₉ IO ₆ + Na: 623.0907, Found: 623.0904

Compound 25: **25**

Physical appearance

pale violet oil

Yield:

0.177 g, 89 %

 R_f (AcOEt: pet. ether 1:4)

0.71

 $[\alpha]_D^{24}$ + 20.8 ($c = 1.0$, CHCl_3)IR (Neat) cm^{-1} :

2936, 1774, 1496, 1454, 1162, 1097, 907, 750, 698.

 ^1H NMR (CDCl_3 , 300 MHz):

δ 7.42 - 7.27 (m, 10H), 5.90 (d, $J = 4.2$ Hz, 1H), 4.83 , 4.61 (each d, 4H, benzyl), 4.04 (d, $J = 0.9$ Hz, 1H), 3.78 (qd, $J_1 = 6.9$ Hz , $J_2 = 1.2$ Hz, 1H), 3.30-3.22 (m, 2H), 2.63 (qd, $J_1 = 1.5$ Hz, $J_2 = 4.8$ Hz, 1H), 1.32 (d, $J = 8$ Hz, 3H).

 ^{13}C NMR (CDCl_3 , 75 Hz):

δ 171.0, 137.0, 128.7, 128.5, 127.4, 128.08, 128.01, 127.09, 100.6, 81.9, 78.6, 77.4, 77.0, 76.5, 74.9, 74.8, 70.1, 52.6, 17.4, 12.9.

High resolution MS:

Calculated for $\text{C}_{22}\text{H}_{23}\text{IO}_5 + \text{Na}$: 517.0488,

Found: 517.0480.

Compound 27: **27**

Physical appearance

pale violet oil

Yield:

0.172 g, 92 %

 R_f (AcOEt: pet. ether 1:9)

0.65

IR (Neat) cm^{-1} :

2875, 1774, 1456, 1097, 735, 695.

 ^1H NMR (CDCl_3 , 400 MHz):

δ 7.39-7.26 (m, 5H), 6.00(d, $J = 2.4$ Hz, 1H), 4.67 (d, $J = 6.9$ Hz, 1H), 4.4 (dd, $J_1 = 5.1$ Hz, $J_2 = 1.2$ Hz, 1H), 3.60 (ddd, $J_1 = 2.4$ Hz, $J_2 = 4.5$ Hz, $J_3 = 4.7$ Hz, 2H),

	3.6 (dt, $J_1 = 1.2$ Hz, $J_2 = 6.9$ Hz, 1H), 3.34-3.29 (m, 1H), 2.52-2.49 (m, 1H), 2.12-2.2.02 (m, 1H).1.35 (m, 1H)
^{13}C NMR (CDCl_3 , 100 Hz):	δ 171.5, 137.0, 128.6, 128.2, 127.7, 101.5, 73.8, 70.7, 61.0, 51.3, 28.1, 13.7.
High resolution MS:	Calculated for $\text{C}_{14}\text{H}_{15}\text{IO}_4 + \text{Na}$: 396.9913 Found: 396.9911

Compound 29:

Physical appearance	pale violet oil
Yield:	0.161 g, 83 %
$[\alpha]_D^{23}$	-6.1 ($c = 1.0$, CHCl_3)
R_f (AcOEt: pet. ether 1:4)	0.65
IR (Neat) cm^{-1} :	2924, 2854, 1775, 1469, 1335, 1171, 1335, 738, 698
^1H NMR (CDCl_3 , 300 MHz):	δ 7.42 - 7.26 (m, 5H), (d, $J = 5.4$ Hz, 1H), 4.62 (d, 2H, benzyl), 4.41 (dd, $J = 6.06$ Hz, 1H), 4.37 (d, $J = 3.03$ Hz, 1H), 4.11 (dd, $J_1 = 6$ Hz, $J_2 = 9$ Hz, 1H), 4.03 - 3.95 (m, 3H), 3.41 (t, $J = 4.5$ Hz, 1H), 1.40 (s, 3H), 1.37 (s, 3H).
^{13}C NMR (CDCl_3 , 75 Hz):	δ 172.8, 136.6, 128.6, 128.3, 127.9, 109.3, 106.2, 82.2, 72.3, 72.0, 66.9, 59.1, 26.7, 25.2, 7.9
High resolution MS:	Calculated for $\text{C}_{18}\text{H}_{21}\text{IO}_6 + \text{Na}$: 483.0281, Found: 483.0280.

Compound 31:

Physical appearance

pale pink oil

Yield:

0.042 g, 83 %

R_f (AcOEt: pet. ether 1:19)

0.42

IR (Neat) cm⁻¹:

2853, 1798, 1598, 1463, 1427, 1113, 739, 701.

¹H NMR (CDCl₃, 400 MHz):

δ 7.58 - 7.19 (m, 30H), 5.71 (d, *J* = 5.6 Hz, 1H), 5.29 (d, *J* = 10 Hz, 1H), 4.17 (s, 1H), 3.87 - 3.71 (m, 3H), 3.31 (dd, *J*₁ = 2.8 Hz, *J*₂ = 11.1 Hz), 2.58 (td, *J*₁ = 4.2 Hz, *J*₂ = 1.5 Hz, 1H), 0.098 (s, 9H), 0.93 (s, 9H), 0.77 (s, 9H).

¹³C NMR (CDCl₃, 100 Hz):

δ 173.0, 135.8, 135.7, 135.69, 135.62, 133.2, 130.18, 130.13, 130.08, 130.07, 129.61, 129.59, 128.0, 127.9, 127.8, 127.66, 127.63, 97.5, 78.7, 77.3, 69.9, 69.1, 62.7, 51.1, 29.7, 26.9, 26.76, 26.72, 19.1, 18.89, 18.87, 9.41.

High resolution MS:

Calculated for C₅₆H₆₅IO₆Si + K: 1083.2771

Found: 1083.2850

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

H1-H1 COESY
NMR (CDCl₃, 500 MHz)

H1-H1 NOESY
NMR (CDCl₃, 500 MHz)

^1H NMR (CDCl_3 , 400 MHz)

^{13}C NMR (CDCl_3 , 100 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 400 MHz)

^{13}C NMR (CDCl_3 , 100 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 400 MHz)

^{13}C NMR (CDCl_3 , 100 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

¹H NMR (CDCl₃, 300 MHz) ^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 300 MHz)

^{13}C NMR (CDCl_3 , 75 MHz)

^1H NMR (CDCl_3 , 400 MHz)

^{13}C NMR (CDCl_3 , 100 MHz)

¹H NMR (CDCl₃, 300 MHz)

¹³C NMR (CDCl₃, 75 MHz)