Supporting Information

for

A modular phosphate tether-mediated divergent strategy to complex polyols

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Experimental section
General Methods:  
Experimental Data:  

All reactions were carried out in an oven- or flame-dried glassware under argon atmosphere using standard gas-tight syringes, cannulae, and septa. Stirring was achieved with oven-dried magnetic stir bars. Et₂O, THF and CH₂Cl₂ were purified by passage through a purification system (Solv-Tek) employing activated Al₂O₃ (Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. Safe and Convenient Procedure for Solvent Purification Organometallics, 1996, 15, 1518–1520). Et₃N was purified by passage over basic alumina and stored over KOH. Butyllithium was purchased from Aldrich and titrated prior to use. All olefin metathesis catalysts were acquired from Materia and used without further purification. Flash column chromatography was performed with Sorbent Technologies (30930M-25, Silica Gel 60A, 40-63 µm) and thin layer chromatography was performed on silica gel 60F₂₅₄ plates (EM-5717, Merck). Deuterated solvents were purchased from Cambridge Isotope laboratories. ¹H and ¹³C NMR spectra were recorded in CDCl₃ (unless otherwise mentioned) on a Bruker DRX-500 spectrometer operating at 500 MHz, and 125 MHz, respectively and calibrated to the solvent peak. ³¹P NMR spectra was recorded on Bruker DRX-400 spectrometer operating at 162 MHz. High-resolution mass spectrometry (HRMS) was recorded on a LCT Premier Spectrometer (Micromass UK Limited) operating on ESI (MeOH). Observed rotations at 589 nm, were measured using AUTOPOL IV Model automatic polarimeter. IR was recorded on Shimadzu FTIR-8400S instrument.
General procedure for RCM/CM/hydrogenation (Procedure A)

To a stirring solution of triene ($S,S$) in freshly distilled, freeze-degas-thawed dichloroethane [1] (0.007 M) was added Hoveyda-Grubbs 2nd Gen. catalyst (HG-II) (3 mol %) and the reaction was refluxed for 2 hours. After completion of RCM (monitored by TLC), the solvent was removed under reduced pressure and olefin cross partner [3–5 equivalent with respect to the triene ($S,S$)] dissolved in freeze-degas-thawed CH$_2$Cl$_2$ (0.1 M) was introduced, followed by addition of HG-II (3–5 mol %). It should be noted that the use of dichloromethane was critical for successful cross-metathesis reaction in order to avoid the formation of isomerized ketone byproducts. Cross-metathesis (CM) reaction in dichloroethane provided the isomerized ketone byproduct (confirmed by $^1$H and $^{13}$C spectra) and the cross-metathesis product with 1:1 ratio both at 70 ºC and 90 ºC. The reaction was refluxed for an additional 2–3 hours upon which the reaction showed the CM product formation along with some amounts of RCM starting material ($S,S,S_P$). The reaction mixture was cooled to RT and o-nitrobenzenesulfonyl hydrazine (o-NBSH) (12 equiv.) and Et$_3$N (2 mL/g of o-NBSH) were added, upon which the reaction was stirred at RT overnight. The reaction mixture was quenched with sat. NaHCO$_3$ (1 mL), and diluted with CH$_2$Cl$_2$ (10 mL). The aqueous layer was washed with CH$_2$Cl$_2$ (3x5 mL) and the combined organic layers were dried (Na$_2$SO$_4$), concentrated under reduced pressure and purified using flash column chromatography.

General procedure for RCM/CM/hydrogenation (Procedure B)

To a stirring solution of triene ($S,S$) in freshly distilled, freeze-degas-thawed CH$_2$Cl$_2$ (0.007 M) was added HG-II (3 mol %) and the reaction was refluxed for 2 hours. After completion of RCM (monitored by TLC), the solvent was removed and olefin cross partner [3 equivalent with respect to the triene ($S,S$)] dissolved in freeze-degas-thawed CH$_2$Cl$_2$ (0.1 M) was introduced, followed by addition of HG-II (3 mol %). It should be noted that the use of dichloromethane was critical for successful CM reaction in order to avoid the formation of isomerized ketone byproducts. Cross-metathesis reaction in dichloroethane provided the isomerized ketone byproduct (confirmed by $^1$H and $^{13}$C spectra) and the CM product with 1:1 ratio both at 70 ºC and 90 ºC. The reaction was refluxed for an additional 2–3 hours upon which the reaction showed the CM product formation along with some amounts of RCM starting material ($S,S,S_P$). The reaction mixture was cooled to RT and o-nitrobenzenesulfonfyl hydrazine (o-NBSH) (12 equiv.) and Et$_3$N (2 mL/g of o-NBSH) were added, upon which the reaction was stirred at RT overnight. The

[1] In CH$_2$Cl$_2$, the RCM appeared to be slower in the presence of HG-II.
reaction mixture was quenched with sat. NaHCO₃ (1 mL), and diluted with CH₂Cl₂ (10 mL). The aqueous layer was washed with CH₂Cl₂ (3x5 mL) and the combined organic layers were dried (Na₂SO₄), concentrated under reduced pressure and purified using flash column chromatography.

**General procedure for RCM/CM/hydrogenation and subsequent reduction with LiAlH₄ (Procedure C)**

The above-mentioned procedure for one-pot RCM/CM/hydrogenation was followed and the crude product was purified using flash column chromatography [2]. To a stirring solution of the hydrogenated product in dry THF (0.5 M), LiAlH₄ (2–4 equiv.) was added portion wise at 0 ºC and the reaction was stirred at 0 ºC for 2 hours. After the completion of reduction, the reaction was quenched via slow sequential addition of H₂O (1 mL/g of LiAlH₄), 10% NaOH (1 mL/g of LiAlH₄), and H₂O (3 mL/g of LiAlH₄) [Feiser workup] [3], and the ice bath was removed and the reaction was stirred for 2 h. The reaction was filtered through Celite and washed with EtOAc (2x5 mL). The combined organic layers were concentrated under reduced pressure and purified using flash column chromatography.

**General procedure for RCM/CM/LiAlH₄ reduction (Procedure D)**

To a stirring solution of triene (S,S) in freshly distilled, freeze-degas-thawed dichloroethane (0.007 M) was added HG-II (3 mol %) and the reaction was refluxed for 2 hours. After completion of RCM (monitored by TLC), the solvent was removed and olefin cross partner [3–5 equivalent with respect to the triene (S,S)] dissolved in freeze-degas-thawed CH₂Cl₂ (0.1 M) was introduced, followed by addition of HG-II (3–6 mol %). The reaction was refluxed for an additional 2–3 hours upon which the reaction showed the CM product formation along with some amounts of RCM starting material (S,S,Sₚ). After the completion of CM, the solvent was evaporated under reduced pressure. The crude reaction mixture was then dissolved in dry THF (0.5 M) and cooled to 0 ºC. To this solution LiAlH₄ (4 equiv.) was added portion wise and the reaction was stirred at 0 ºC for 2 hours. After the completion of reduction, the reaction was quenched via slow sequential addition of H₂O (1 mL/g of LiAlH₄), 10% NaOH (1 mL/g of LiAlH₄), and H₂O (3 mL/g of LiAlH₄) [Feiser workup], and the ice bath was removed and the reaction was

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[2] Purification was necessary at this stage for subsequent successful LAH reduction.
stirred for 2 h. The reaction was filtered through Celite® and washed with EtOAc (2x5 mL). The combined organic layers were concentrated under reduced pressure and purified using flash column chromatography.

**General procedure for RCM/CM/LiAlH₄ reduction (Procedure E)**

To a stirring solution of triene (S,S) in freshly distilled, freeze-degas-thawed CH₂Cl₂ (0.007 M) was added HG-II (3 mol %) and the reaction was refluxed for 2 hours. After completion of RCM (monitored by TLC), the solvent was removed and olefin cross partner [3 equivalent with respect to the triene (S,S)] dissolved in freeze-degas-thawed CH₂Cl₂ (0.1 M) was introduced, followed by addition of Hoveyda-Grubbs 2nd Gen. catalyst (3 mol %). The reaction was refluxed for an additional 2–3 hours upon which the reaction showed the CM product formation along with some amounts of RCM starting material (S,S,S,P). After the completion of CM, the solvent was evaporated under reduced pressure. The crude reaction mixture was then dissolved in dry THF (0.5 M) and cooled to 0 ºC. To this solution LiAlH₄ (4 equiv.) was added portion wise and the reaction was stirred at 0 ºC for 2 hours. After the completion of reduction, the reaction was quenched via slow sequential addition of H₂O (1 mL/g of LiAlH₄), 10% NaOH (1 mL/g of LiAlH₄), and H₂O (3 mL/g of LiAlH₄) [Feiser workup], and the ice bath was removed and the reaction was stirred for 2 h. The reaction was filtered through Celite® and washed with EtOAc (2x5 mL). The combined organic layers were concentrated under reduced pressure and purified using flash column chromatography.

**General procedure for RCM/CM/ LiAlH₄ reduction and subsequent global hydrogenation (Procedure F)**

The above-mentioned procedure (D or E) was followed to obtain the reduced product and the crude product was dissolved in CH₂Cl₂ (0.1 M) and o-nitrobenzenesulfonyl hydrazine (o-NBSH) (20 equiv.) and Et₃N (2 mL/g of o-NBSH) were added, upon which the reaction was stirred at RT overnight. The reaction mixture was quenched with sat. NaHCO₃ (1 mL) and diluted with CH₂Cl₂ (10 mL). The aqueous layer was washed with CH₂Cl₂ (3x5 mL) and the combined organic layers were dried (Na₂SO₄), concentrated under reduced pressure and purified using flash column chromatography. At this stage, purification was performed twice in order to remove all the byproducts arising from the use of o-NBSH.
(4S,6S)-2-(((1S,2R)-1-(4-bromophenyl)-2-methylbut-3-en-1-yl)oxy)-4,6-divinyl-1,3,2-dioxaphosphinane 2-oxide (5)

Synthesized by following the literature precedence [4].

Yield: 75%

FTIR (neat): 2962, 2927, 2349, 1724, 1593, 1488, 1283, 1120, 1070, 997 cm⁻¹;

Optical Rotation: [α]D = +26.7 (c = 0.075, CHCl₃);

¹H NMR (500 MHz, CDCl₃) δ 7.45 (d, J = 8.4 Hz, 2H, aromatic), 7.17 (d, J = 8.4 Hz, 2H, aromatic), 6.00 (dddd, J = 17.1, 10.6, 5.5, 0.9 Hz, 1H, H₂C=CHCH(OP)CH₂), 5.68 (dddd, J = 17.3, 10.6, 5.4, 1.6 Hz, 1H, H₂C=CHCH(CH₃)CH(OP)Ar), 5.39–5.30 (m, 2H, H₂C=CHCH(OP)CH₂), 5.18–5.12 (m, 2H, H₂C=CHCH(OP)CH₂), 5.10 (d, J = 1.3 Hz, 1H, H₂C=CHCH(CH₃)CH(OP)Ar), 5.05 (dddd, J = 14.3, 6.8, 3.3, 1.4 Hz, 1H, H₂C=CHCH(OP)CH₂), 5.01–4.93 (m, 2H, H₂C=CHCH(OP)CH₂), 4.52–4.45 (m, 1H, H₂C=CHCH(OP)CH₂), 2.78–2.65 (m, 1H, H₂C=CHCH(CH₃)CH(OP)Ar), 2.09 (dddd, J = 14.7, 8.3, 5.0, 1.5 Hz, 1H, H₂C=CHCH(OP)CH₂), 1.93 (dddd, J = 14.8, 5.2, 3.5, 1.9 Hz, 1H, H₂C=CHCH(OP)CH₂), 1.13 (d, J = 6.8 Hz, 3H, H₂C=CHCH(C₃H₃)CH(OP)Ar).

¹³C NMR (126 MHz, CDCl₃) δ 137.9, 137.8 (d, J = 2.3 Hz), 135.2 (d, J = 3.0 Hz), 134.7 (d, J = 7.9 Hz), 131.3 (2C), 129.0 (2C), 122.2, 117.8, 117.6, 116.7, 83.5 (d, J = 6.2 Hz), 78.0 (d, J = 6.8 Hz), 75.7 (d, J = 6.0 Hz), 44.1 (d, J = 6.8 Hz), 35.0 (d, J = 7.4 Hz), 15.8.

³¹P NMR (162 MHz, CDCl₃) δ -6.85;

HRMS: cald. for C₁₉H₂₂BrO₄PNa (M+Na)⁺ 435.0337; found 435.0325 (TOF MS ES+).

(1S,3S,4R,7S,9R,Z)-9-(((S)-4-(benzoxyl)-3-hydroxybutyl)-3-(4-bromophenyl)-4-methyl-2,10,11-trioxa-1-phosphabicyclo[5.3.1]undec-5-ene 1-oxide (8)

Synthesized by following procedure B

Yield: 33% over 3 reactions (70% avg/rxn)

FTIR (neat): 3411, 2962, 2927, 2873, 1593, 1454, 1488, 1284, 1105, 1007, 979 cm⁻¹;

Optical Rotation: [α]D = -18.3 (c = 0.59, CHCl₃);

¹H NMR (500 MHz, CDCl₃) δ 7.39 (d, J = 13.6 Hz, 2H aromatic), 7.34–7.11 (m, 5H, aromatic), 7.03 (d, J = 13.6 Hz, 2H, aromatic), 5.47 (dt, J = 11.8, 1.8 Hz, 1H, CHOPCH=CHCHCH₂CHOPAr), 5.26–5.16 (m, 3H,

CH₂CHOPCH=CHCH₃CHOPAr), 4.70 (t, J = 11.5 Hz, 1H, CH₂CHOPCH₂CHOP), 4.46 (s, 2H, CH₂OCH₂Ph), 4.01–3.92 (m, 1H, CH=CHCH₃CHOPAr), 3.77–3.68 (m, 1H, CH₂CHOHCH₂OCH₂Ph), 3.41 (dd, J = 9.4, 3.2 Hz, 1H CHOHHOCH₂OCH₂Ph), 3.24 (dd, J = 9.5, 7.9 Hz, 1H, CHOHHOCH₂OCH₂Ph), 2.34 (s, 1H, OH), 2.15 (ddd, J = 14.4, 11.8, 6.1 Hz, 1H, CH₂CHOPCH₂CHOP), 1.89–1.76 (m, 2H, CHOHCH₂CH₂CHOPCH₂CHOP), 1.74–1.60 (m, 2H, CHCHOHCH₂CH₂CHOPCH₂CHOP), 1.42–1.34 (m, 1H, CHOHHOCH₂CH₂CHOPCH₂CHOP), 0.69 (d, J = 6.9 Hz, 3H, CH=CHCH₃CHOPAr);

¹³C NMR (126 MHz, CDCl₃) δ 137.7, 135.6 (d, JCP = 13.6 Hz), 132.0, 131.1 (2C), 130.4, 129.0 (2C), 128.5 (2C), 127.9, 127.8 (2C), 122.4, 79.1 (d, JCP = 4.0 Hz), 78.7 (d, JCP = 6.9 Hz), 78.5 (d, JCP = 7.4 Hz), 74.5, 73.4, 70.3, 36.4 (d, JCP = 6.5 Hz), 34.2, 32.6 (d, JCP = 9.3 Hz), 28.5, 16.9;

³¹P NMR (162 MHz, CDCl₃) δ -8.89;

HRMS: cald. for C₂₅H₃₀BrO₅PNa (M+Na)⁺ 559.0861; found 559.0865 (TOF MS ES+).

(1S,3S,6S,8R)-3-((benzyloxy)methyl)-8-((3R,4S)-4-(4-bromophenyl)-4-hydroxy-3-methylbutyl)-2,9,10-trioxa-1-phosphabicyclo[4.3.1]dec-4-ene 1-oxide (9):

![Phosphabicyclo[4.3.1]dec-4-ene 1-oxide](image)

Synthesized by following procedure A

Yield: 40% over 3 reactions (72% avg/rxn)

FTIR (neat): 3400, 2974, 2285, 1630, 1288, 1209, 1101, 977, 848 cm⁻¹;

Optical Rotation: [α]D = +17.34(c = 1, CHCl₃);

¹H NMR (500 MHz, CDCl₃) δ 7.50–7.43 (m, 2H, aromatic), 7.40–7.29 (m, 5H, aromatic), 7.21–7.16 (m, 2H, aromatic), 6.01 (ddd, J = 11.9, 3.0, 2.1 Hz, 1H, CH=CHCHOPCH₂OBn), 5.56 (ddd, J = 11.9, 3.9, 2.4 Hz, 1H, CH=CHCHOPCH₂OBn), 5.27 (ddd, J = 5.3, 5.3, 2.6, 2.6 Hz, 1H, CH=CHCHOPCH₂OBn), 5.18 (ddddd, JPPH = 24.6, JHH = 6.2, 4.1, 1.9 Hz, 1H, CH₂CHOPCH=CH), 4.65–4.58 (m, 2H, CH₂OCH₂Ph), 4.58–4.47 (m, 2H, 4-BrC₆H₄CHOH), 2.82–2.65 (m, 4H, CH₂CH₂CHOP), 3.71 (ddd, J = 10.3, 5.1, 1.2 Hz, 1H, CH₂OBn), 3.61 (dd, J = 10.2, 6.0 Hz, 1H, CH₂OBn), 3.21 (ddd, J = 14.7, 12.0, 6.2 Hz, 1H, CHOPCH₂CHOP), 1.88 (d, J = 3.5 Hz, 1H, OH), 1.87–1.71 (m, 2H, 4-BrC₆H₄CHOHCH(CH₃)CH₂, CH₂CHOPCH₂CHOP), 1.67 (ddd, J = 14.6, 3.5, 2.0 Hz, 1H, CHOPCH₂CHOP), 1.58–1.48 (m, 1H, CH₂CHOPCH₂CHOP), 1.48–1.40 (m, 1H, CH₂CH₂CHOPCH₂CHOP), 0.88 (d, J = 6.7 Hz, 3H, 4-BrC₆H₄CHOHCH(CH₃)CH₂);

¹³C NMR (126 MHz, CDCl₃) δ 142.4, 137.5, 131.3 (2C), 129.9, 129.6, 128.5 (2C), 128.0 (2C), 127.9, 127.7 (2C), 121.1, 77.3 (d, JCP = 6.8 Hz), 77.0, 76.8 (d, JCP = 7.2 Hz), 73.5, 72.2 (d, JCP = 6.1 Hz), 71.2 (d, JCP = 12.2 Hz), 39.9, 34.8 (d, JCP = 6.0 Hz), 33.5 (d, JCP = 9.3 Hz), 27.9, 14.0;

³¹P NMR (162 MHz, CDCl₃) δ -53.0;

HRMS: cald. for C₂₅H₃₀BrO₅P (M+Na)⁺ 559.0861; found (TOF MS ES⁺) 559.0856.
(1S,2R,5S,7R,10S,Z)-11-(benzzyloxy)-1-(4-bromophenyl)-2-methylundec-3-ene-1,5,7,10-tetraol (10)

![Chemical Structure Image]

Synthesized by following procedure C

**Yield:** 24% over 4 reactions (70% avg/rxn)

**FTIR** (neat): 3367, 3335, 3061, 2921, 2852, 1595, 1519, 1456, 1093, 1072, 1026, 910 cm⁻¹;

**Optical Rotation:** [α]D = +32.3 (c = 0.33, CHCl₃);

**¹H NMR** (500 MHz, CDCl₃) δ 7.46 (d, J = 7.8 Hz, 2H, aromatic), 7.38–7.28 (m, 5H, aromatic), 7.16 (d, J = 7.8 Hz, 2H, aromatic), 5.64 (dd, J = 8.5, 10.4 Hz, 1H, CHOHCH=CHCH₂CHOHAr), 5.03 (dd, J = 10.5, 10.6 Hz, 1H, CHOHCH=CHCH₂CHOHAr), 4.70 (dd, J = 7.3, 10.7 Hz, 1H, CHOHCH=CHCH₂CHOHAr), 4.60 (d, J = 4.6 Hz, 1H, CHOHCH=CHCH₂CHOHAr), 4.50 (s, 2H, CH₂OCH₂Ph), 4.06–3.93 (m, 1H, OH), 3.87 (m, 1H, CH₂CHOHCH₂CHOH), 3.71 (dd, J = 5.7, 5.7 Hz, 1H, CH₂CHOHCH₂OCH₂Ph), 3.51 (dd, J = 3.2, 9.4 Hz, 1H, CHOHCH₂OCH₂Ph), 3.37 (dd, J = 8.9, 8.9 Hz, 1H, CHOHCH₂OCH₂Ph), 3.07 (dq, J = 12.8, 6.4 Hz, 1H, CH=CHCH₂CHOHAr), 2.92 (s, 1H, OH), 1.78–1.44 (m, 8H, OH, CHOHCH₂CH₂CHOHCH₂CHOH), 0.95 (d, J = 6.8 Hz, 3H, CH=CHCH₂CHOHAr);

**¹³C NMR** (126 MHz, CDCl₃) δ 139.8, 137.9, 133.7, 133.4, 130.9(2C), 128.9(2C), 128.9(2C), 127.8(2C), 127.2, 121.5, 77.7, 74.4, 73.2, 70.3, 68.7, 64.3, 42.1, 38.2, 33.4, 29.8, 17.4;

**HRMS:** calcd. for C₂₅H₃₀BrO₅ (M+Na)⁺ 515.1409; found 515.1397 (TOF MS ES+).

(1S,2R,5R,7S,10S,Z)-11-(benzyloxy)-1-(4-bromophenyl)-2-methylundec-8-ene-1,5,7,10-tetraol (11)

![Chemical Structure Image]

Synthesized by following procedure C

**Yield:** 26% over 4 reactions (71% avg/rxn)

**FTIR** (neat): 3365, 3294, 2943, 2872, 2349, 2872, 1631, 1485, 1454, 1070, 1028, 827, 750, 698 cm⁻¹;

**Optical Rotation:** [α]D = +5.0 (c = 0.12, CHCl₃);

**¹H NMR** (500 MHz, CDCl₃) δ 7.50–7.44 (m, 2H, aromatic), 7.39–7.28 (m, 5H, aromatic), 7.22–7.17 (m, 2H, aromatic), 5.71 (dd, J = 11.5, 7.3, 1.4 Hz, 1H, CHOHCH=CHCH₂CHOHAr), 5.50 (ddd, J = 11.4, 7.3, 1.3 Hz, 1H, CHOHCH=CHCH₂CHOH₂OBn), 4.85–4.77 (m, 1H, CHOHCH=CHCH₂CHOH₂OBn), 4.75–4.69 (m, 1H, CHOHCH=CHCH₂CHOH₂OBn), 4.62–4.54 (m, 3H, 4-BrC₆H₄CHOHCH(CH₃)CH₂, CH₂OCH₂Ph), 3.88 (bs, 1H, OH), 3.76–3.68 (m, 1H, CH₂CHOHCH₂CHOHCH₂CH), 3.50 (dd, J = 9.4, 4.1 Hz, 1H, CH₂OCH₂Ph), 3.45 (dd, J = 9.4, 7.6 Hz, 1H, CH₂OCH₂Ph), 3.44–3.29 (m, 1H, 4-BrC₆H₄CHOHCH(CH₃)CH₂), 1.78–1.68 (m, 1H, OH), 1.67–1.62 (m, 2H, CH₂CHOHCH₂OH), 1.46–1.37 (m, 2H, CH₂CHOHCH₂CH₂OH), 1.26 (s, 4H, OH, CH₂CHOHCH₂CH₂OH), 0.88 (d, J = 6.8 Hz, 3H, 4-BrC₆H₄CHOHCH(CH₃)CH₂).
$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 142.5, 137.5, 136.5, 131.2 (2C), 129.2, 128.5 (2C), 128.0 (2C), 127.99, 127.9 (2C), 120.9, 76.9, 73.6, 73.5, 67.3, 66.2, 62.9, 42.6, 40.0, 28.9, 28.8, 14.3.

HRMS: cald. for C$_{25}$H$_{33}$BrO$_5$ (M+Na)$^+$ 515.1409; found (TOF MS ES+) 515.1398.

(1S,2R,3S,5S,7S,8E,10S)-11-(benzyloxy)-1-(4-bromophenyl)-2-methylundeca-3,8-diene-1,5,7,10-tetraol (12)

![Structural diagram]

Synthesized by following procedure E

Yield: 38% over 3 reactions (73% avg/rxn)

FTIR (neat): 3377, 3330, 2962, 2926, 2868, 1865, 1591, 1454, 1215, 1101, 1070, 1009, 976 cm$^{-1}$;

Optical Rotation: $[\alpha]_D$ = -38.8 (c = 0.45, CHCl$_3$);

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.45 (d, $J$ = 8.4 Hz, 2H, aromatic), 7.40–7.29 (m, 5H, aromatic), 7.15 (d, $J$ = 8.4 Hz, 2H, aromatic), 5.91 (ddd, $J$ = 15.6, 5.6, 1.4 Hz, 1H, CHOHCH=CHCH=CHCHOHCH$_2$CHOH), 5.74 (ddd, $J$ = 15.6, 5.8, 1.4 Hz, 1H, CHOHC=CHCHOHCH$_2$CHOH), 5.63 (ddd, $J$ = 10.9, 8.2, 0.9 Hz, 1H, CH$_2$CHOHCH=CHCHCH$_3$), 4.97 (td, $J$ = 10.6, 1.2 Hz, 1H, CHOHCH=CHCH=CHCHOHCH$_2$CHOH), 4.65 (ddd, $J$ = 11.4, 6.3, 2.2 Hz, 1H, CH$_2$CHOHCH=CHCHCH$_3$), 4.58 (d, $J$ = 2.9 Hz, 4H, OH, PhCH$_2$OCH$_2$CHOHCH=CHCHOHCH$_2$CHOH=CHCHCH$_3$CHOHAr), 4.48 (s, 1H PhCH$_2$OCH$_2$CHOHCH=CHCHOHCH$_2$CHOH), 4.40 (s, 1H PhCH$_2$OCH$_2$CHOHCH=CH), 3.56 (dd, $J$ = 9.6, 3.3 Hz, 1H, PhCH$_2$OCH$_2$CHOH), 3.38 (dd, $J$ = 9.6, 8.1 Hz, 1H, PhCH$_2$OCH$_2$CHOH), 3.13 (s, 1H, OH), 3.12–3.01 (m, 1H, CHOHC=CHCH=CHCHOHCH$_2$CHOH), 2.62 (s, 1H, OH), 1.87 (ddd, $J$ = 14.4, 8.7, 3.6 Hz, 1H, CH=CHCHOHCH$_2$CHOH), 1.70 (ddd, $J$ = 14.3, 7.5, 3.3 Hz, 1H, CH=CHCHOHCH$_2$CHOH), 1.61 (s, 1H, OH), 0.9 (s, 3H, CH=CHCH$_3$);

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 139.6, 137.7, 134.8, 133.8, 133.5, 130.9 (2C), 128.9 (2C), 128.5 (2C), 128.4, 127.90, 127.8 (2C), 121.6, 76.9, 74.1, 73.4, 70.7, 69.5, 64.1, 41.9, 38.2, 17.4;

HRMS: cald. for C$_{25}$H$_{31}$BrO$_5$ (M+Na)$^+$ 513.1253; found 513.1255 (TOF MS ES+).

(1S,2R,3E,5S,7S,8Z,10S)-11-(benzyloxy)-1-(4-bromophenyl)-2-methylundeca-3,8-diene-1,5,7,10-tetraol (13)

![Structural diagram]

Synthesized by following procedure D

Yield: 35% over 3 reactions (70% avg/rxn)

FTIR (neat): 3440, 3417, 3386, 2390, 1643, 1633, 1054, 698, 522 cm$^{-1}$;

Optical Rotation: $[\alpha]_D$ = - 4.81 (c = 0.22, CHCl$_3$)
1H NMR (500 MHz, CDCl3) δ 7.47–7.43 (m, 2H, aromatic), 7.37–7.29 (m, 5H, aromatic), 7.18–7.12 (m, 2H, aromatic), 5.68 (dd, J = 11.4, 7.6, 1.4 Hz, 1H, CHOCH=CH(CH2)7CHOHCH2OBn), 6.51 (dd, J = 15.6, 7.0, 1.0 Hz, 1H, CH=CH(CH2)7CHOHCH2CHOH), 5.53 (d, J = 6.2 Hz, 1H, CH=CH(CH2)7CHOHCH2CHOH), 5.48 (dd, J = 11.4, 7.4, 1.3 Hz, 1H, CH=CH(CH2)7CHOHCH2CHOH), 4.76 (dd, J = 7.9, 2.9 Hz, 1H, CHOCH=CH(CH2)7CHOHCH2OBn), 4.68 (dd, J = 7.5, 4.1, 1.4 Hz, 1H, CHOCH=CH(CH2)7CHOHCH2OBn), 4.59 (d, J = 5.1 Hz, 1H, 4-BrC6H4CHOHCH(CH3)2), 4.56 (bs, 2H, CH2OBn), 4.35 (dd, J = 6.6, 10.4 Hz, 1H, CH=CH(CH2)7CHOHCHOH), 3.55 (s, 1H, OH), 3.48 (dd, J = 9.5, 4.1 Hz, 1H, CH2OBn), 3.44 (dd, J = 9.5, 7.5 Hz, 1H, CH2OBn), 3.16 (s, 1H, OH), 2.90 (s, 1H, OH), 2.52 (dd, J = 12.6, 6.7 Hz, 1H, 4-BrC6H4CHOHCH(CH3)2), 2.46 (s, 1H, OH), 1.74 (ddd, J = 14.4, 8.7, 3.8 Hz, 1H, CHOCH(CH2)7CHOHCHOH), 1.61 (ddd, J = 14.3, 7.5, 3.3 Hz, 1H, CHOCH(CH2)7CHOHCHOH), 0.96 (d, J = 6.8 Hz, 3H, 4-BrC6H4CHOHCH(CH3)2).

13C NMR (126 MHz, CDCl3) δ 141.6, 137.5, 136.3, 133.8, 132.5, 132.4, 129.2, 128.5 (2C), 128.1 (2C), 127.9, 127.9 (2C), 121.0, 76.6, 73.6, 73.5, 70.1, 67.2, 65.7, 43.3, 42.8, 14.0.

HRMS: cald. for C25H31BrO5 (M+Na)+ 513.1253; found (TOF MS ES+) 513.1237.

(1S,2R,5R,7R,10S)-11-(benzylxoy)-1-(4-bromophenyl)-2-methylundecane-1,5,7,10-tetraol (14)

Synthesized by following procedure F

Yield: 26% over 4 reactions (72% avg/rxn)

FTIR (neat): 3377, 3330, 2962, 2926, 2868, 1865, 1454, 1215, 1070, 1009, 976 cm⁻¹;

Optical Rotation: [α]D = -11.4 (c = 0.285, CHCl3);

1H NMR (500 MHz, CDCl3) δ 7.46 (d, J = 8.3 Hz, 2H, aromatic), 7.38–7.28 (m, 5H, aromatic), 7.2 (d, J = 8.3 Hz, 2H, aromatic), 4.60 (d, J = 4.9 Hz, 1H, CH2CH2CH2CHOHAr), 4.56 (s, 2H, PhCH2OCH2CHOH), 4.06–3.93 (m, 1H, CH2CHOHCH2CHOH), 3.92–3.85 (m, 2H, PhCH2OCH2CHOHCH2CH2CHOHCH2CHOH), 3.50 (dd, J = 9.3, 3.5 Hz, 1H, PhCH2OCH2CHOH), 3.37 (m, 1H, PhCH2OCH2CHOH), 3.30–1.74 (m, 1H, CH2CH2CH2CHOHAr), 1.65–1.55 (m, 1H, OH, aliphatic), 1.46–1.37 (m, 2H, aliphatic), 1.28–1.21 (m, 1H, aliphatic), 0.95 (d, J = 6.8 Hz, 3H, CH2CH2CH2CHOHAr);

13C NMR (126 MHz, CDCl3) δ 142.6, 137.7, 131.2, 128.5(2C), 128.1(2C), 127.8(2C), 126.4(2C), 121.0, 77.1, 74.3, 73.4, 70.3, 69.4, 69.0, 42.4, 40.0, 34.8, 33.4, 29.0, 28.9, 14.3;

HRMS: cald. for C25H33BrO5 (M+Na)+ 517.1566; found 517.1584 (TOF MS ES+).

(1S,3S,7S,9R,Z)-9-((S)-4-benzyloxy-3-hydroxybutyl)-3-((benzylxoy)methyl)-2,11-trioxa-1-phosphabicyclo[5.3.1]undec-5-ene 1-oxide (15)

Synthesized by following procedure B
Yield: 40% over 3 reactions (72% avg/rxn)

FTIR (neat): 3413, 3028, 2923, 2858, 1679, 1452, 1363, 1288, 1101, 979 cm⁻¹;

Optical Rotation: [α]D = +7.6 (c = 0.41, CHCl₃);

¹H NMR (500 MHz, CDCl₃) δ 7.40–7.30 (m, 10H, aromatic), 5.69 (dt, J = 11.8, 8.8, 2.9 Hz, 1H, CHOPCH=CHCH₂CHOP), 5.45 (dt, J = 11.8, 1.7 Hz, 1H, CHOPCH=CHCH₂CHOP), 5.25 (dd, J = 25.0, 4.3 Hz, 1H, CH₂CHOPCH=CHCH₂CHOP), 4.76–4.65 (m, 1H, CH=CHCH₂CHOPCH₂OBn), 4.59–4.52 (m, 5H, OCH₂Ph, CHOPCH₂CHOPCH=CH), 3.79 (dd, J = 11.4, 8.9, 3.1 Hz, 1H, PhCH₂OCH₂CHOHCH₂), 3.51 (dd, J = 11.3, 9.7, 4.2 Hz, 2H, PhCH₂OCH₂CHOHCH₂CHOPCH=CHCH₂CHOPCH=CHCH₂CHOP), 3.43 (dd, J = 9.4, 7.4 Hz, 1H, CH=CHCH₂CHOPCH₂OBn), 3.36–3.29 (m, 2H, BnOCH₂CHOHCH₂CHOPCH=CHCH₂CHOPCH=CHCH₂CHOP), 2.36 (dd, J = 13.9, 8.5 Hz, 1H, CH=CHCH₂CHOPCH₂OBn), 2.14 (dd, J = 14.5, 11.7, 6.0 Hz, 1H, CHOPCH=CHCH₂CHOPCH=CH), 1.91–1.79 (m, 1H, CHOCH₂CH₂CHOPCH=CHCH₂CHOP), 1.78–1.65 (m, 3H, CHOCH₂CH₂CHOPCH=CHCH₂CHOP), 1.62 (s, 1H, OH), 1.44 (dddd, J = 15.2, 12.9, 7.5, 3.0 Hz, 1H, CHOCH₂CH₂CHOPCH=CHCH₂CHOP);

¹³C NMR (126 MHz, CDCl₃) δ 137.8, 137.7, 131.5, 128.5 (2C), 128.5 (2C), 127.9, 127.8, 127.7 (2C), 127.6 (2C), 125.03, 78.4 (d, JCP = 7.1 Hz), 78.1 (d, JCP = 7.2 Hz) 74.4, 73.4, 73.2, 72.3 (d, JCP = 4.9 Hz), 70.4 (d, JCP = 15.5 Hz), 70.2, 36.2 (d, JCP = 6.5 Hz), 32.4 (d, JCP = 8.8 Hz), 28.5, 27.4;

³¹P NMR (162 MHz, CDCl₃) δ -8.2;

HRMS: cald. for C₂₆H₃₅O₇P (M+Na)⁺ 511.1862; found 511.1855 (TOF MS ES+).

### (1S,3S,6S,8R)-8- ((S)-5-benzyloxy-4-hydroxypentyl)-3-((benzyloxy)methyl)-2,9,10-trioxa-1-phosphabicyclo[4.3.1]dec-4-ene 1-oxide (16)

![Chemical structure of 16](attachment:image)

Synthesized by following procedure A

Yield: 35% over 3 reactions (70% avg/rxn)

FTIR (neat): 3520, 3444, 2395, 1633, 1286, 1101, 973, 734 cm⁻¹;

Optical Rotation: [α]D = +21.57(c = 0.26, CHCl₃);

¹H NMR (500 MHz, CDCl₃) δ 7.42–7.28 (m, 10H, aromatic), 6.02 (ddd, J = 11.9, 3.0, 2.1 Hz, 1H, CH=CHCHOPCH₂OBn), 5.57 (ddd, J = 11.9, 3.9, 2.4 Hz, 1H, CH=CHCHOPCH₂OBn), 5.28 (ddddd, J = 5.3, 5.3, 2.7, 2.7, 2.7 Hz, 1H, CH=CHCHOPCH₂OBn), 5.19 (ddddd, Jₚₕ = 24.5, Jₜₘ = 6.2, 4.1, 1.8, 1.8 Hz, 1H, CH₂CHOPCH=CH), 4.66–4.52 (m, 5H, CH₂OCH₂Ph, CH₂CH₂CH₂CHOP), 3.84–3.77 (m, 1H, CH₂OCHOCH₂CH₂CHOP), 3.72 (dd, J = 10.2, 5.1, 1.1 Hz, 1H, CH₂OBn), 3.61 (dd, J = 10.2, 6.1 Hz, 1H, CH₂OBn), 3.50 (dd, J = 9.4, 3.1 Hz, 1H, CH₂OBn), 3.32 (dd, J = 9.4, 7.9 Hz, 1H, CH₂OBn), 2.34 (d, J = 3.4 Hz, 1H, BnOCH₂CHOHCH₂CH₂CHOP), 2.22–2.12 (m, 2H, CHOPCH₂CHOP), 1.83–1.55 (m, 4H, BnOCH₂CHOHCH₂CH₂CHOP), 1.52–1.37 (m, 2H, BnOCH₂CHOHCH₂CH₂CHOP);

¹³C NMR (126 MHz, CDCl₃) δ 137.8, 137.5, 129.9, 129.6, 128.5 (4C), 127.9, 127.8, 127.7 (4C), 77.3 (d, JCP = 6.7 Hz), 76.6 (d, JCP = 6.8 Hz), 74.4, 73.5, 73.4, 72.1 (d, JCP = 6.0 Hz), 71.2 (d, JCP = 12.2 Hz), 70.0, 35.4 (d, JCP = 9.3 Hz), 34.7 (d, JCP = 6.0 Hz), 32.4, 20.5;

³¹P NMR (162 MHz, CDCl₃) δ -5.46;
HRMS: calcd. for C_{26}H_{33}O_7PNa (M+Na)^+ 511.1862; found (TOF MS ES+) 511.1847.

(2S,5R,7S,11S,Z)-1,12-bis(benzyloxy)dodec-8-ene-2,5,7,11-tetraol (17)

Synthesized by following procedure C

Yield: 24% over 4 reactions (70% avg/rxn)

FTIR (neat): 3367, 2914, 2858, 2331, 1093, 1076, 698 cm^{-1};

Optical Rotation: \([\alpha]_D^\text{D} = +6.8 \quad (c = 0.44, \text{CHCl}_3);

^1H NMR (500 MHz, CDCl_3) δ 7.40–7.30 (m, 10H, aromatic), 5.70 (ddt, J = 11.0, 8.2, 1.4 Hz, 1H, CHOHC=CHCH=CHCHOH), 5.51 (dddd, J = 11.2, 5.1, 3.5 Hz 1H, CH=CHCH=CHCHOHCH=CHCHOH), 4.79–4.67 (m, 1H, CH=CHC=CHCHOH), 3.97–3.88 (m, 1H, CH=CHCH=CHCHOH), 3.88–3.79 (m, 1H, CH=CHCH=CHCHOH), 3.51 (m, 2H, CH2OC=CH2OBn), 3.45–3.33 (m, 2H, CH2OC=CH2OBn), 3.15 (bs, 1H, O), 2.90 (bs, 2H, OH), 2.43 (dddd, J = 14.5, 8.6, 5.1, 1.1 Hz, 1H, CH=CHCH=CHCHOHOBn), 2.30 (dddd, J = 14.2, 7.7, 6.5, 1.3 Hz, 1H, CH=CHCH=CHCHOHOBn), 1.77–1.67 (m, 4H, CH=CHCH=CHCHOH), 1.30 (s, 1H, OH);

^13C NMR (126 MHz, CDCl_3) δ 137.9, 137.7, 135.6, 128.5 (2C), 128.4 (2C), 127.9, 127.8 (3C), 127.7 (2C), 126.4, 74.4, 73.6, 73.5, 73.3, 70.4, 69.6, 68.9, 65.3, 42.5, 33.5, 31.2, 29.2;

HRMS: calcd. for C_{26}H_{36}O_6 (M+Na)^+ 467.2410; found 467.2390 (TOF MS ES+).

(2S,5S,7R,11S,Z)-1,12-bis(benzyloxy)dodec-3-ene-2,5,7,11-tetraol (18)

Synthesized by following procedure C

Yield: 23% over 4 reactions (69% avg/rxn)

FTIR (neat): 3400, 3290, 2943, 2350, 1631, 1480, 1070, 750, 698 cm^{-1};

Optical Rotation: \([\alpha]_D^\text{D} = +6.32(c = 0.19, \text{CHCl}_3);

^1H NMR (500 MHz, CDCl_3) δ 7.40–7.28 (m, 10H, aromatic), 5.70 (ddd, J = 11.5, 7.5, 1.4 Hz, 1H, CHOHC=CHCHOHCH2OBn), 5.48 (ddd, J = 11.4, 7.4, 1.4 Hz, 1H, CHOHC=CHCHOHCH2OBn), 4.80 (ddd, J = 10.4, 7.3, 3.4 Hz, 1H, CHOHC=CHCHOHCH2OBn), 4.75–4.67 (m, 1H, CH=CHCHOHCH2OBn), 4.59–4.53 (m, 4H, CH2OCH2Ph), 3.97–3.88 (m, 1H, CH2OCH2CHOHCH=CH), 3.88–3.79 (m, 1H,
BnOCH₂CHOHCH₂CH₂), 3.56–3.41 (m, 4H, CH₂OBn), 3.40–3.29 (m, 2H, BnOCH₂CHOHCH₂CH₂), 1.66 (ddd, J = 14.5, 7.5, 2.6 Hz, 1H, CHOHCH₂CHOH), 1.63–1.57 (m, 3H, CHOHCH₂CHOH, BnOCH₂CHOHCH₂CH₂, OH), 1.53–1.35 (m, 6H, BnOCH₂CHOHCH₂CH₂CHOH).

¹³C NMR (126 MHz, CDCl₃) δ 137.9, 137.6, 136.5, 129.2, 128.5 (2C), 128.46 (2C), 127.9, 127.85 (2C), 127.8, 127.7 (2C), 74.5, 73.6, 73.4, 73.3, 70.23, 68.8, 67.1, 66.1, 42.7, 37.2, 32.8, 21.5.

HRMS: cald. for C₂₆H₃₆O₆(M+Na)⁺ 467.2410; found (TOF MS ES+) 467.2413.

(2S,3E,5S,7S,8Z,11S)-1,12-bis(benzyloxy)dodeca-3,8-diene-2,5,7,11-tetraol (19)

Synthesized by following procedure E

Yield: 42% over 3 reactions (75% avg/rxn)

FTIR (neat): 3377, 3361, 3028, 2916, 2858, 1602, 1452, 1101 cm⁻¹; Optical Rotation: [α]D = +10.4 (c = 0.67, CHCl₃);

¹H NMR (500 MHz, CDCl₃) δ 7.40–7.29 (m, 10H, aromatic), 5.89 (ddd, J = 15.5, 5.4, 1.3 Hz, 1H, CHOHCH=CHCHOHCH₂), 5.77–5.64 (m, 2H, CH=CHCHOHCH₂), 5.53–5.48 (m, 1H, CHOHC=CHCHOHCH₂OBn), 4.71 (td, J = 2.1 Hz, 1H, CHOHCH=CHC₂H₂OCH₂), 4.57 (d, J = 2.1 Hz, 2H, CH₂OCH₂Ph), 4.55 (d, J = 3.8 Hz, 2H, CH₂OCH₂Ph), 4.46 (bs, 1H, CHOHCH=CHCHOHCH₂), 4.38 (bs, 1H, PhCH₂OCH₂CHOHCH=CHCHOH), 3.91 (ddt, J = 11.0, 6.7, 3.4 Hz, 1H, CH=CHCHOHCH₂OBn), 3.54 (ddd, J = 9.5, 3.4, 2.2 Hz, 1H, CH=CHCHOHCH₂OBn), 3.51 (dd, J = 9.5, 3.4 Hz, 1H, CH=CHCHOHCH₂OBn), 3.45–3.32 (m, 2H, CH₂OCH₂), 3.06 (s, 1H, OH), 2.82 (s, 1H, OH), 2.60 (s, 1H, OH) 2.42 (ddd, J = 13.9, 8.7, 5.1, 1.3 Hz, 1H, CH=CHCHOHCH₂OBn), 2.28 (ddd, J = 14.1, 7.6, 6.3, 1.3 Hz, 1H, CH=CHCHOHCH₂OBn), 1.85 (ddd, J = 14.3, 8.1, 3.6 Hz, 1H, CHOHCH=CHOHCH=CHCH₂), 1.70 (ddd, J = 14.3, 7.6, 3.7 Hz, 1H, CHOHCH=CHOHCH=CHCH₂), 1.64 (s, 1H, OH);

¹³C NMR (126 MHz, CDCl₃) δ 137.7, 137.6, 135.4, 134.9, 128.51 (2C), 128.5, 128.48 (2C), 128.4, 127.92, 127.89, 127.86, 127.8 (2C), 126.6, 74.1, 73.5, 73.4, 73.36, 70.7, 69.6, 69.5, 64.9, 42.4, 42.3, 31.1;

HRMS: cald. for C₂₆H₃₆O₆(M+Na)⁺ 465.2253; found 465.2241 (TOF MS ES+).

(2S,3Z,5S,7S,8E,11S)-1,12-bis(benzyloxy)dodeca-3,8-diene-2,5,7,11-tetraol (20)

Synthesized by following procedure D

Yield: 40% over 3 reactions (72% avg/rxn)

FTIR (neat): 3408, 3402, 3385, 2918, 2852, 2349, 1637, 1632, 1072, 1027, 972, 737, 698 cm⁻¹; Optical Rotation: [α]D = +5.92(c = 0.14, CHCl₃);
$^1$H NMR (500 MHz, CDCl$_3$) δ 7.42–7.28 (m, 10H, aromatic), 5.75–5.64 (m, 2H, CHOHCH=CHCHOHCH$_2$OBn), 5.62–5.53 (m, 1H, BnOCH$_2$CHOHCH=CHCHOH), 5.48 (ddd, $J = 11.3$, 7.4, 1.3 Hz, 1H, BnOCH$_2$CHOHCH=CHCHOH), 4.78 (ddd, $J = 7.8$, 7.8, 3.0 Hz, 1H, CHOHCH=CHCHOHCH$_2$OBn), 4.70 (ddd, $J = 7.5$, 7.5, 4.3, 1.4 Hz, 1H, CHOHCH$_2$CHOHCH=CHCHOHCH$_2$OBn), 4.59–4.52 (m, 4H, CH$_2$OCH$_2$Ph), 4.42–4.35 (m, 1H, CHOHCH$_2$CH=CHCHOH), 3.90–3.82 (m, 1H, BnOCH$_2$CHOHCH=CHCHOH), 3.54–3.40 (m, 4H, BnOCH$_2$CHOH), 3.36 (ddd, $J = 9.5$, 7.3 Hz, 2H, BnOCH$_2$CHOH), 2.30–2.13 (m, 4H, BnOCH$_2$CHOHCH=CHCHOH), 1.77 (ddd, $J = 14.3$, 8.6, 3.6 Hz, 1H, CHOHCH$_2$CHOH), 1.64 (ddd, $J = 14.4$, 7.7, 3.3 Hz, 1H, CHOHCH$_2$CHOH).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 137.8, 137.6, 136.3, 135.5, 129.3, 128.5 (2C), 128.4 (2C), 127.9, 127.85 (2C), 127.8, 127.7 (2C), 126.7, 77.2, 73.9, 73.6, 73.4, 73.3, 69.9, 67.1, 65.6, 42.6, 36.2;

HRMS: cald. for C$_{26}$H$_{34}$O$_6$ (M+Na)$^+$ 465.2253; found (TOF MS ES+) 465.2236.

(2S,5R,7R,11S)-1,12-bis(benzyloxy)dodecane-2,5,7,11-tetraol (21)

![Structure Image]

Synthesized by following procedure F

Yield: 34% over 4 reactions (77% avg/rxn)

FTIR (neat): 3283, 2943, 2394, 1454, 1093, 1076, 737 cm$^{-1}$;

Optical Rotation: $[\alpha]_D = +2.80$ (c = 0.25, CHCl$_3$);

$^1$H NMR (500 MHz, CDCl$_3$) δ 7.40–7.29 (m, 10H, aromatic), 4.60–4.52 (m, 4H, CH$_2$OCH$_2$Ph), 4.04–3.91 (m, 2H, CHOHCH$_2$CHOH), 3.92–3.76 (m, 2H, CHOHCH$_2$OBn), 3.51 (ddd, $J = 9.5$, 4.9, 3.2 Hz, 2H, BnOCH$_2$CHOH), 3.36 (ddd, $J = 17.2$, 9.4, 8.0 Hz, 2H, BnOCH$_2$CHOH), 2.80 (s, 1H, OH), 2.61 (s, 1H, OH), 2.40 (bs, 1H, OH), 1.70–1.37 (m, 13H, aliphatic, OH).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 137.9, 137.8, 128.5 (4C), 127.9, 127.8, 127.77 (2C), 127.75 (2C), 74.3, 74.5, 73.3 (2C), 70.4, 70.3, 69.3, 69.0, 42.3, 37.2, 33.5, 32.8, 29.1, 21.6.

HRMS: cald. for C$_{26}$H$_{38}$O$_6$ (M+Na)$^+$ 469.2566; found (TOF MS ES+) 469.2567.
(4S,6S)-2-(((1S,2R)-1-(4-bromophenyl)-2-methylbut-3-en-1-yl)oxy)-4,6-divinyl-1,3,2-dioxaphosphinane 2-oxide (5)
(1S,3S,4R,7S,9R,Z)-9-((S)-4-(benzoyloxy)-3-hydroxybutyl)-3-(4-bromophenyl)-4-methyl-2,10,11-trioxa-1-phosphabicyclo[5.3.1]undec-5-ene 1-oxide (8)
(1S,3S,6S,8R)-3-((benzyloxy)methyl)-8-((3R,4S)-4-(4-bromophenyl)-4-hydroxy-3-methylbutyl)-2,9,10-trioxa-1-phosphabicyclo[4.3.1]dec-4-ene 1-oxide (9)
(1S,2R,5S,7R,10S,Z)-11-(benzyloxy)-1-(4-bromophenyl)-2-methylundec-3-ene-1,5,7,10-tetraol (10)
(1S,2R,5R,7S,10S,Z)-11-(benzyloxy)-1-(4-bromophenyl)-2-methylundec-8-ene-1,5,7,10-tetraol (11)
(1S,2R,3Z,5S,7S,8E,10S)-11-(benzyloxy)-1-(4-bromophenyl)-2-methylundeca-3,8-diene-1,5,7,10-tetraol (12)
(1S,2R,3E,5S,7S,8Z,10S)-11-(benzyloxy)-1-(4-bromophenyl)-2-methylundeca-3,8-diene-1,5,7,10-tetraol (13)
(1S,2R,5R,7R,10S)-11-(benzyloxy)-1-(4-bromophenyl)-2-methylundecane-1,5,7,10-tetraol (14)
(1S,3S,7S,9R,Z)-9-((S)-4-(benzylloxy)-3-hydroxybutyl)-3-((benzylloxy)methyl)-2,10,11-trioxa-1-phosphabicyclo[5.3.1]undec-5-ene 1-oxide (15)
(1S,3S,6S,8R)-8-((S)-5-(benzyloxy)-4-hydroxypentyl)-3-((benzyloxy)methyl)-2,9,10-trioxa-1-phosphabicyclo[4.3.1]dec-4-ene 1-oxide (16)
(2S,5R,7S,11S,Z)-1,12-bis(benzyloxy)dodec-8-ene-2,5,7,11-tetraol (17)
(2S,5S,7R,11S,Z)-1,12-bis(benzyloxy)dodec-3-ene-2,5,7,11-tetraol (18)
(2S,3E,5S,7S,8Z,11S)-1,12-bis(benzyloxy)dodeca-3,8-diene-2,5,7,11-tetraol (19)
(2S,3Z,5S,7S,8E,11S)-1,12-bis(benzyloxy)dodeca-3,8-diene-2,5,7,11-tetraol (20)
(2S,5R,7R,11S)-1,12-bis(benzyloxy)dodecane-2,5,7,11-tetraol (21)