Stereoselective Access to Z- and E-Macrocycles by Ruthenium-Catalyzed Z-Selective Ring-Closing Metathesis and Ethenolysis

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General methods

All reactions were carried out in dry glassware under an argon atmosphere using standard Schlenk line techniques or in a Vacuum Atmospheres Glovebox under a nitrogen atmosphere unless otherwise specified. All solvents were purified by passage through solvent purification columns, with the exception of anhydrous 1,2-dichloroethane which was used as received from Sigma Aldrich, and further degassed with argon. Ruthenium complex 2 was obtained from Materia Inc. 1H-NMR spectra were acquired at 500 MHz and 13C-NMR spectra at 125 MHz as CDCl3 solutions. Quantitative 13C measurements were acquired at 125 MHz (decoupled, without NOE, 13 second delay time). All HRMS were by positive-ion EI or FAB.

But-3-en-1-yl undec-10-enoate (1a)

\[
\begin{align*}
\text{H}_8 & \quad \text{O} \\
\text{O} & \quad \text{C} \\
\text{H}_2 & \quad \text{C} \\
\text{C} & \quad \text{C} \\
\end{align*}
\]

10-Undecenoyl chloride (4.5 mL, 21 mmol) was dissolved in CH2Cl2 (40 mL), and pyridine (1.7 mL, 21 mmol), then 3-butanol (1.6 mL, 19 mmol), were added dropwise at 0 °C. The solution was warmed to room temperature, and stirred for four hours. The mixture was then washed sequentially with 1M HCl (aq.), saturated NaHCO3 (aq.), brine, dried with Na2SO4, and the solvent was removed in vacuo. Flash chromatography of the residue (SiO2, using 5% EtOAc in hexanes) provided 1a (4.4 g, 88%) as a colourless oil; 1H NMR δ 5.79 (2H, m), 5.11 (1H, m), 5.07 (1H, m), 4.99 (1H, m), 4.93 (1H, m), 4.12 (2H, t, J = 6.7 Hz), 2.38 (2H, m), 2.29 (2H, t, J = 7.6 Hz), 2.03 (2H, m), 1.61 (2H, m), 1.26-1.40 (10H, m); 13C NMR δ 173.8, 139.2, 134.1, 117.1, 114.2, 63.3, 34.3, 33.8, 33.1, 29.3, 29.2, 29.1 (2C), 28.9, 25.0; HRMS (EI) 238.1932, [C15H26O2]+ requires 238.1933.

But-3-en-1-yl dec-9-enoate (4a)

\[
\begin{align*}
\text{H}_7 & \quad \text{O} \\
\text{O} & \quad \text{C} \\
\text{H}_2 & \quad \text{C} \\
\text{C} & \quad \text{C} \\
\end{align*}
\]

Oxalyl chloride (2.1 mL, 25 mmol) was added dropwise to a solution of 9-decenoic acid (3.9 mL, 21 mmol) and pyridine (0.20 mL, 2.1 mmol) in CH2Cl2 (100 mL), and the solution was let to stir for fifteen hours, then concentrated. The residue was dissolved in CH2Cl2 (40 mL), and pyridine (1.7 mL, 21 mmol),

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then 3-butenol (1.6 mL, 19 mmol), were added dropwise at 0 °C. The solution was warmed to room temperature, and stirred for four hours. The mixture was then washed sequentially with 1M HCl (aq.), saturated NaHCO₃ (aq.), brine, dried with Na₂SO₄, and the solvent was removed in vacuo. Flash chromatography of the residue (SiO₂, using 5% EtOAc in hexanes) provided 4a (4.0 g, 85%) as a colourless oil; ¹H NMR δ 5.79 (2H, m), 5.11(1H), 5.07 (1H, m), 4.99 (1H, m), 4.93 (1H, m), 4.13 (2H, t, J = 7.1 Hz), 2.38 (2H, m), 2.30 (2H, t, J = 7.5 Hz), 2.04 (2H, m), 1.62 (2H, m), 1.22-1.41 (8H, m); ¹³C NMR δ 173.8, 139.1, 134.1, 117.1, 114.2, 63.3, 34.3, 33.8, 33.1, 29.1 (2C), 28.9 (2C), 25.0; HRMS (EI) 224.1773, [C₁₄H₂₄O₂]⁺ requires 224.1776.

Undec-10-en-1-yl hex-5-enoate (5a)

![5a]

According to the procedure for compound 4a, 5-hexenoic acid (1.65 mL, 14 mmol) was reacted with oxalyl chloride (1.4 mL, 17 mmol) and pyridine (0.12 mL, 1.4 mmol), then 10-undecenol (2.6 mL, 13 mmol) and pyridine (1.2 mL, 14 mmol), to provide 5a (0.86 g, 23%) as a colourless oil; ¹H NMR δ 5.80 (2H, m), 5.01 (2H, m), 4.96 (2H, m), 4.06 (2H, t, J = 6.8 Hz), 2.31 (2H, t, J = 7.6 Hz), 2.09 (2H, m), 2.04 (2H, m), 1.73 (2H, m), 1.61 (2H, m), 1.24-1.41 (12H, m); ¹³C NMR δ 173.7, 139.2, 137.7, 115.3, 114.1, 64.5, 33.8, 33.6, 33.1, 29.5, 29.4, 29.2, 29.1, 28.9, 28.7, 25.9, 24.1; HRMS (EI) 266.2245, [C₁₇H₃₀O₂]⁺ requires 266.2246.

Dec-9-en-1-yl undec-10-enoate (6a)

![6a]

According to the procedure for compound 1a, 10-undecenoyl chloride (4.5 mL, 21 mmol) was reacted with 9-decenol (3.4 mL, 19 mmol) and pyridine (1.7 mL, 21 mmol), to provide 6a (6.4 g, 94%) as a colourless oil; ¹H NMR δ 5.81 (2H, m), 4.99 (2H, m), 4.93 (2H, m), 4.05 (2H, t, J = 6.7 Hz), 2.29 (2H, t, J = 7.5 Hz), 2.04 (2H, m), 1.61 (2H, m), 1.25-1.41 (20H, m); ¹³C NMR δ 174.0, 139.2, 139.1, 114.2, 114.1, 64.4, 34.4, 33.8 (2C), 29.4, 29.3, 29.2 (2C), 29.1 (2C), 29.0, 28.9 (2C), 28.7, 25.9, 25.0; HRMS (EI) 322.2884, [C₂₁H₃₈O₂]⁺ requires 322.2872.
Dec-9-en-1-yl oct-7-enoate (7a)

According to the procedure for compound 4a, 7-octenoic acid (3.2 mL, 21 mmol) was reacted with oxalyl chloride (2.1 mL, 25 mmol) and pyridine (0.20 mL, 2.1 mmol), then 9-decenol (3.4 mL, 19 mmol) and pyridine (1.7 mL, 21 mmol), to provide 7a (4.8 g, 81%) as a colourless oil; $^1$H NMR $\delta$ 5.80 (2H, m), 4.99 (2H, m), 4.93 (2H, m), 4.06 (2H, $J = 6.7$ Hz), 2.29 (2H, $J = 7.5$ Hz), 2.04 (2H, m), 1.62 (2H, m), 1.26-1.44 (14H, m); $^{13}$C NMR $\delta$ 173.9, 139.1, 138.8, 114.4, 114.2, 64.4, 34.3, 33.8, 33.6, 29.4, 29.2, 29.0, 28.9, 28.7, 28.6, 28.5, 25.9, 24.9; HRMS (EI) 280.2406, [C$_{18}$H$_{32}$O$_2$]$^+$ requires 280.2402.

9-Iodonon-1-ene (16)

Methanesulponyl chloride (3.3 mL, 42 mmol) was added dropwise to a 0 °C solution of 8-nonenol (5.9 mL, 35 mmol) and triethylamine (5.9 mL, 42 mmol) in CH$_2$Cl$_2$ (35 mL). The mixture was let to stir at room temperature for two hours, and was then washed sequentially with 1M HCl (aq.) (x2), saturated NaHCO$_3$ (aq.), brine, and dried with Na$_2$SO$_4$. The solvent was removed in vacuo, the residue was taken in acetone (125 mL), and NaI (13 g, 88 mmol) was added. The mixture was stirred at room temperature for 24 hours, then concentrated. Flash chromatography of the residue (SiO$_2$, using hexanes) provided 16 (6.9 g, 78%) as a colourless oil; $^1$H NMR $\delta$ 5.81 (1H, m), 4.99 (1H, m), 4.93 (1H, m), 3.19 (2H, $J = 7.0$ Hz), 2.04 (2H, m), 1.82 (2H, m), 1.39 (4H, m), 1.31 (4H, m); $^{13}$C NMR $\delta$ 139.0, 114.3, 33.7, 33.5, 30.5, 28.9, 28.8, 28.4, 7.3; HRMS (EI) 252.0373, [C$_9$H$_{17}$I]$^+$ requires 252.0375.

Nonadeca-1,18-dien-10-ol (10a)

A solution of iodide 16 (6.9 g, 27 mmol) in Et$_2$O (50 mL) was cooled to -78 °C, and tert-butyl lithium
(32 mL, 1.7M in pentanes) was added dropwise. The solution was warmed to room temperature over 1 hour, then re-cooled to -78 °C, and 9-decenal\(^2\) (3.9 g, 25 mmol) was added dropwise. The solution was warmed to room temperature over one hour, then washed with saturated NaHCO\(_3\) (aq.), then brine, dried with Na\(_2\)SO\(_4\), and concentrated. The residue was taken in hexanes, and recrystallized at -20 °C to provide 10a (5.0 g, 71%) as a colourless solid; \(^1\)H NMR δ 5.81 (2H, m), 4.99 (2H, m), 4.93 (2H, m), 3.58 (1H, m), 2.04 (4H, m), 1.25-1.48 (25H, m); \(^13\)C NMR δ 139.2 (2C), 114.1 (2C), 72.0, 37.5 (2C), 33.8 (2C), 29.6 (2C), 29.5 (2C), 28.9 (2C), 25.6 (2C); HRMS (EI) 280.2759, [C\(_{19}\)H\(_{36}\)O\(^+\)] requires 280.2766.

tert-Butyldimethyl(nonadeca-1,18-dien-10-yloxy)silane (11a)

![11a](image)

tert-Butyldimethylsilyl chloride (0.18 g, 1.2 mmol) was added to a solution of alcohol 10a (0.30 g, 1.1 mmol), imidazole (0.14 g, 2.1 mmol), and 4-dimethylaminopyridine (0.012 g, 0.10 mmol) in CH\(_2\)Cl\(_2\) (5 mL), and the solution was let to stir for 24 hours at room temperature. The solution was washed with saturated NaHCO\(_3\) (aq.), brine, and dried with Na\(_2\)SO\(_4\). The solvent was removed \textit{in vacuo}, and flash chromatography of the residue (SiO\(_2\), using pentanes) provided 11a (0.42 g, 98%) as a colourless oil; \(^1\)H NMR δ 5.81 (2H, m), 4.99 (2H, m), 4.93 (2H, m), 3.61 (1H, m), 2.04 (4H, m), 1.38 (8H, m), 1.28 (16H, m), 0.88 (9H, s), 0.03 (6H, s); \(^13\)C NMR δ 139.2 (2C), 114.1 (2C), 72.4, 37.1 (2C), 33.8 (2C), 29.8 (2C), 29.5 (2C), 29.1 (2C), 28.9 (2C), 26.0 (3C), 25.3 (2C), 18.2, -4.4 (2C); HRMS (EI) 393.3545, [C\(_{25}\)H\(_{49}\)OSi-H\(^+\)] requires 393.3553.

Nonadeca-1,18-dien-10-yl acetate (12a)

![12a](image)

Acetic anhydride (0.13 mL, 1.4 mmol) was added to a solution of alcohol 10a (0.19 g, 0.68 mmol) and pyridine (0.070 mL, 0.87 mmol) in CH\(_2\)Cl\(_2\) (1.5 mL), and the solution was let to stir for 17 hours at room temperature. The mixture was diluted with diethyl ether, washed with saturated NaHCO\(_3\) (aq.), brine, and dried with Na\(_2\)SO\(_4\). The solvent was removed \textit{in vacuo}, and flash chromatography of the residue

(SiO₂, using 10% Et₂O in pentanes) provided 12a (0.21 g, 95%) as a colourless oil; ^1H NMR δ 5.80 (2H, m), 4.99 (2H, m), 4.92 (2H, m), 4.85 (1H, m), 2.03 (3H, s), 2.03 (4H, m), 1.49 (4H, m), 1.36 (4H, m), 1.22-1.31 (16H, m); ^13C NMR δ 170.9, 139.2 (2C), 114.2 (2C), 74.4, 34.1 (2C), 33.8 (2C), 29.5 (2C), 29.4 (2C), 29.0 (2C), 28.9 (2C), 25.3 (2C), 21.3 (2C); HRMS (FAB) 321.2794, [C21H₃₈O₂-H]^+ requires 321.2794.

Nonadeca-1,18-dien-10-one (8a)

[Tetrapropylammonium perruthenate (0.21 g, 0.60 mmol) was added to a solution of alcohol 8a (3.5 g, 12 mmol) and N-methylmorpholine N-oxide (2.1 g, 18 mmol) in CH₂Cl₂ (25 mL). The mixture was let to stir at room temperature for one hour, then loaded directly onto a silica gel column. Purification by flash chromatography (using 10% EtOAc in hexanes) provided 8a (3.2 g, 97%) as a colourless solid; ^1H NMR δ 5.80 (2H, m), 4.98 (2H, m), 4.93 (2H, m), 2.38 (4H, t, J = 7.5 Hz), 2.03 (4H, m), 1.56 (4H, m), 1.37 (4H, m), 2.91 (12H, m); ^13C NMR δ 211.7, 139.1 (2C), 114.2 (2C), 42.8 (2C), 33.8 (2C), 29.3 (2C), 29.2 (2C), 28.9 (4C), 23.9 (2C); HRMS (EI) 278.2607, [C₁₉H₃₄O]^+ requires 278.2610.

2,2-Di(non-8-en-1-yl)-1,3-dioxolane (9a)

[para-Toluene sulphonic acid (0.010 g, 0.055 mmol) was added to a solution of ketone 9a (0.30 g, 1.1 mmol) and ethylene glycol (0.60 mL, 11 mmol) in benzene (10 mL), and refluxed using a Dean-Stark apparatus for 21 hours. The solution was then cooled, diluted with Et₂O, washed sequentially with 10% NaOH (aq.), saturated NaHCO₃ (aq.), then brine, dried with Na₂SO₄, and concentrated. In order to facilitate separation of 9a from unreacted 8a, the residue was dissolved in MeOH (5 mL), and stirred with NaBH₄ (10 mg) for ca. 30 minutes. The mixture was then diluted with Et₂O, washed with brine, then dried with Na₂SO₄. The solvent was removed in vacuo, and flash chromatography of the residue (SiO₂, using 2% Et₂O in pentanes) provided 9a (0.30 g, 86%) as a colourless oil; ^1H NMR δ 5.81 (2H, m), 4.99 (2H,
m), 4.92 (2H, m), 3.92 (4H, s), 2.04 (4H, m), 1.59 (4H, m), 1.25-1.40 (20H, m); $^{13}$C NMR δ 139.2 (2C), 114.1 (2C), 111.9, 64.9 (2C), 37.1 (2C), 33.8 (2C), 29.9 (2C), 29.5 (2C), 29.1 (2C), 28.9 (2C), 24.9 (2C); HRMS (EI) 322.2862, [C$_{21}$H$_{38}$O$_2$]$^+$ requires 322.2872.

$N$-(Dec-9-en-1-yl)hex-5-enamide (13a)

5-Hexenoic acid (1.5 mL, 13 mmol), then triethylamine (3.3 mL, 24 mmol), were added to a solution of 9-decenamine$^3$ (1.9 g, 12 mmol), 1-hydroxybenzotriazole hydrate (2.0 g, 13 mmol), and $N$-ethyl-$N'$-(3-dimethylaminopropyl)carbodiimide hydrochloride (2.5 g, 13 mmol) in CH$_2$Cl$_2$ (60 mL), and the mixture was stirred at room temperature overnight. The solution was diluted with EtOAc, and washed sequentially with 0.5M citric acid ($aq.$), saturated NaHCO$_3$, then brine, dried with Na$_2$SO$_4$, and concentrated. Purification by flash chromatography (using a gradient of CH$_2$Cl$_2$ to 5% MeOH in CH$_2$Cl$_2$) provided 13a (2.6 g, 87%) as a pale yellow oil; $^1$H NMR δ 5.79 (2H, m), 5.47 (1H, br s), 4.98 (4H, m), 3.23 (2H, m), 2.16 (2H, t, $J$ = 7.5 Hz), 2.09 (2H, m), 2.03 (2H, m), 1.74 (2H, m), 1.48 (2H, m), 1.37 (2H, m), 1.29 (8H, m); $^{13}$C NMR δ 172.7, 139.2, 138.0, 115.3, 114.2, 39.5, 36.0, 33.8, 33.2, 29.7, 29.4, 29.2, 29.0, 28.9, 26.9, 24.8; HRMS (EI) 251.2240, [C$_{16}$H$_{29}$ON]$^+$ requires 251.2249.

tert-Butyl dec-9-en-1-yl(hex-5-enoyl)carbamate (14a)

A solution of amide 13a (1.0 g, 4.0 mmol) in THF (15 mL) was cooled to -78 °C, and $n$-butyl lithium (15 mL, 2.5M in hexanes) was added dropwise. After 30 minutes of stirring, a solution of di-tert-butyl dicarbonate (0.89 g, 4.1 mmol) in THF (4 mL) was added dropwise, and the mixture was let to warm to 0 °C over two hours. The solution was then diluted with Et$_2$O, washed with saturated NH$_4$Cl ($aq.$), dried with Na$_2$SO$_4$, and concentrated. Purification by flash chromatography (using 2% Et$_2$O in pentanes) provided 13a (1.1 g, 79%) as a colourless oil; $^1$H NMR δ 5.79 (2H, m), 4.96 (4H, m), 3.62 (2H, t, $J$ = 7.5

Hz), 2.81 (2H, t, \(J = 7.5\) Hz), 2.08 (2H, m), 2.01 (2H, m), 1.72 (2H, m), 1.51 (9H, s), 1.48 (2H, m), 1.35 (2H, m), 1.26 (8H, m); \(^{13}\)C NMR \(\delta\) 175.8, 153.4, 139.2, 138.3, 114.9, 114.1, 82.6, 44.5, 37.7, 33.8, 33.2, 29.4, 29.3, 29.0, 28.9, 28.7, 28.1 (3C), 27.4, 26.9, 24.4; HRMS (EI) 351.2783, \([\text{C}_{21}\text{H}_{37}\text{O}_3\text{N}]^+\) requires 351.2773.

**General procedure 1: Z-selective macrocyclizations catalyzed by Ru-complex 2**

In a glovebox, a 500 mL Strauss flask was charged with a solution of diene (1 equiv, *ca.* 0.45 mmol) in dichloroethane (5 mM, 90 mL), and a solution of 2 (7.5 mol%) dissolved in dichloroethane (1 mL) was added. The flask was sealed, brought out of the glovebox, and subjected to a single freeze/pump/thaw/cycle. The flask was kept under a static vacuum of 20 mtorr, and heated at 60 °C. After 24 hours (except for Z-8 which was quenched after 8 hours), the mixture was cooled, quenched with excess ethyl vinyl ether, and concentrated. Flash chromatography of the residue (SiO\(_2\), using 2% Et\(_2\)O in pentanes for compounds 1, 4–9, 12, and 14, 10% Et\(_2\)O in pentanes for compound 10, pentanes for compound 11, and 66% Et\(_2\)O in pentanes for compound 13) provided the product.

**Z-Oxacyclotetradec-11-en-2-one (1)**

According to General Procedure 1, diene 1a (0.11 g, 0.46 mmol) was reacted with 2 (0.022 g, 0.035 mmol) to provide Z-1 (0.056 g, 58% yield, 85% Z as determined by \(^1\)H-NMR) as a colourless oil; \(^1\)H NMR \(\delta\) 5.54 (1H, m), 5.38 (1H, m), 4.23 (2H, t, \(J = 5.3\) Hz), 2.41 (2H, m), 2.35 (2H, t, \(J = 6.3\) Hz), 2.03 (2H, m), 1.64 (2H, m), 1.24-1.39 (10H, m); \(^{13}\)C NMR \(\delta\) 174.0, 132.3, 127.1, 63.7, 33.3, 27.7, 27.5, 26.1, 26.0, 25.5, 25.4, 25.2, 23.5; HRMS (EI) 210.1623, \([\text{C}_{13}\text{H}_{22}\text{O}_2]^+\) requires 210.1620.
**Z-Oxacyclotridec-10-en-2-one (4)**

According to *General Procedure 1*, diene 4a (0.10 g, 0.45 mmol) was reacted with 2 (0.021 g, 0.033 mmol) to provide **Z-4** (0.035 g, 40% yield, 86% Z as determined by $^1$H-NMR) as a colourless oil; $^1$H NMR $\delta$ 5.40 (2H, m), 4.23 (2H, $t$, $J$ = 4.5 Hz), 2.43 (2H, m), 2.28 (2H, $t$, $J$ = 6.0 Hz), 2.09 (2H, m), 1.67 (2H, m), 1.49 (2H, m), 1.38 (2H, m), 1.27 (2H, m), 1.21 (2H, m); $^{13}$C NMR $\delta$ 174.7, 132.3, 127.1, 64.2, 35.4, 29.7, 27.5, 27.3, 26.0, 25.9, 24.6, 23.5; HRMS (EI) 196.1424, [C$_{12}$H$_{20}$O$_2$]$^+$ requires 196.1463.

**Z-Oxacyclohexadec-6-en-2-one (5)**

According to *General Procedure 1*, diene 5a (0.11 g, 0.43 mmol) was reacted with 2 (0.022 g, 0.035 mmol) to provide **Z-5** (79 mg, 77% yield, 84% Z as determined by $^1$H-NMR) as a colourless oil; $^1$H NMR $\delta$ 5.35 (2H, m), 4.14 (2H, $t$, $J$ = 5.5 Hz), 2.35 (2H, $t$, $J$ = 6.6 Hz), 2.02-2.12 (4H, m), 1.61-1.74 (4H, m), 1.28-1.44 (12H, m); $^{13}$C NMR $\delta$ 174.0, 131.2, 129.0, 64.5, 34.3, 28.0, 27.9, 27.4, 27.0, 26.9, 26.8, 26.6, 26.2, 25.6, 25.5; HRMS (EI) 238.1933, [C$_{15}$H$_{26}$O$_2$]$^+$ requires 238.1943.
Z-Oxacycloicos-11-en-2-one (6)

According to General Procedure 1, diene 6a (0.14 g, 0.43 mmol) was reacted with 2 (0.021 g, 0.033 mmol) to provide Z-6 (0.98 g, 75% yield, 94% Z as determined by quantitative $^{13}$C-NMR) as a colourless oil; $^1$H NMR $\delta$ 5.35 (2H, m), 4.12 (2H, t, $J = 5.8$ Hz), 2.31 (2H, t, $J = 6.9$ Hz), 2.03 (4H, m), 1.63 (4H, m), 1.22-1.43 (20H, m); $^{13}$C NMR $\delta$ 174.0, 130.1, 130.0, 64.3, 34.8, 29.3, 29.1, 29.0, 28.8 (2C), 28.7 (2C), 28.5, 28.4, 28.1, 26.5, 26.4, 26.3, 25.1; HRMS (EI) 294.2552, [C$_{19}$H$_{34}$O$_{2}$]$^+$ requires 294.2559.

Z-Oxacycloheptadec-8-en-2-one (7)

According to General Procedure 1, diene 7a (0.13 g, 0.46 mmol) was reacted with 2 (0.022 g, 0.035 mmol) to provide Z-7 (0.085 g, 71% yield, 89% Z as determined by quantitative $^{13}$C-NMR) as a colourless oil; $^1$H NMR $\delta$ 5.32 (2H, m), 4.14 (2H, t, $J = 5.4$ Hz), 2.33 (2H, t, $J = 6.5$ Hz), 2.04 (4H, m), 1.63 (4H, m), 1.21-1.43 (14H, m); $^{13}$C NMR $\delta$ 173.9, 130.2, 130.1, 63.7, 34.6, 29.4, 28.8, 28.7, 28.5 (2C), 28.4, 27.7, 27.0, 26.8, 25.3 (2C); HRMS (EI) 252.2089, [C$_{16}$H$_{28}$O$_{2}$]$^+$ requires 252.2100.
Z-Cycloheptadec-9-enone (8)

According to General Procedure 1, diene 8a (0.12 g, 0.43 mmol) was reacted with 2 (0.021 g, 0.033 mmol) for 8 hours to provide Z-8 (0.068 g, 62% yield, 50% Z as determined by 1H-NMR) as a colourless solid; 1H NMR δ 5.34 (2H, m), 2.39 (4H, t, J = 6.7 Hz), 2.01 (4H, m), 1.61 (4H, m), 1.21-1.39 (16H, m); 13C NMR δ 212.95, 130.2 (2C), 42.5 (2C), 29.0 (2C), 28.6 (2C), 28.2 (4C), 26.7 (2C), 23.9 (2C); HRMS (EI) 250.2299, [C_{17}H_{30}O]^{+} requires 250.2297.

Z-1,4-Dioxaspiro[4.16]henicos-13-ene (9)

According to General Procedure 1, diene 9a (0.14 g, 0.43 mmol) was reacted with 2 (0.021 g, 0.033 mmol) to provide Z-9 (0.075 g, 60% yield, 85% Z as determined by quantitative 13C-NMR) as a colourless oil; 1H NMR δ 5.33 (2H, m), 3.91 (4H, s), 2.05 (4H, m), 1.57 (4H, m), 1.24-1.38 (20H, m); 13C NMR δ 130.1 (2C), 112.2, 64.3 (2C), 35.7 (2C), 29.2 (2C), 28.8 (2C), 27.8 (2C), 27.7 (2C), 27.1 (2C), 22.8 (2C); HRMS (EI) 294.2545, [C_{19}H_{34}O_{2}]^{+} requires 294.2559.
**Z-Cycloheptadec-9-enol (10)**

According to *General Procedure 1*, diene 10a (0.12 g, 0.43 mmol) was reacted with 2 (0.021 g, 0.033 mmol) to provide **Z-10** (0.062 g, 56% yield, 65% Z as determined by quantitative $^{13}$C-NMR) as a colourless solid; $^1$H NMR $\delta$ 5.34 (2H, m), 3.72 (1H, m), 2.04 (4H, m), 1.50 (4H, m), 1.22-1.40 (21H, m); $^{13}$C NMR $\delta$ 130.2 (2C), 70.4, 35.6 (2C), 29.0 (2C), 28.2 (2C), 28.0 (2C), 27.9 (2C), 26.8 (2C), 23.5 (2C); HRMS (EI) 252.2451, [$\text{C}_{17}\text{H}_{32}\text{O}]^{+}$ requires 252.2453.

**Z-tert-Butyl(cycloheptadec-9-en-1-yloxy)dimethylsilane (11)**

According to *General Procedure 1*, diene 11a (0.17 g, 0.43 mmol) was reacted with 2 (0.021 g, 0.033 mmol) to provide **Z-11** (0.090 g, 56% yield, 75% Z as determined by quantitative $^{13}$C-NMR) and recovered 11a (0.022 g, 13%) as an inseperable mixture; for **Z-11**: $^1$H NMR $\delta$ 5.34 (2H, m), 3.68 (1H, m), 2.04 (4H, m), 1.18-1.53 (24H, m), 0.88 (9H, s), 0.03 (6H, s); $^{13}$C NMR $\delta$ 130.2 (2C), 71.3, 35.9 (2C), 29.1 (2C), 28.5 (2C), 28.1 (2C), 27.9 (2C), 26.9 (2C), 25.9 (3C), 23.4 (2C), 18.2, 4.5 (2C); HRMS (EI) 365.3252, [$\text{C}_{23}\text{H}_{46}\text{OSi}]^{+}$ requires 365.3240.
**Z-Cycloheptadec-9-en-1-yl acetate (12)**

According to *General Procedure 1*, diene 12a (0.14 g, 0.43 mmol) was reacted with 2 (0.022 g, 0.035 mmol) to provide **Z-12** (0.074 g, 59% yield, 75% Z as determined by quantitative $^{13}$C-NMR) as a colourless oil; $^1$H NMR $\delta$ 5.34 (2H, m), 4.86 (1H, m), 2.03 (4H, m), 2.01 (3H, s), 1.55 (4H, m), 1.22-1.40 (20H, m); $^{13}$C NMR $\delta$ 170.8, 130.2, 73.5, 32.2 (2C), 29.1 (2C), 28.2 (2C), 27.8 (2C), 26.8 (2C), 23.4 (2C), 21.4 (2C); HRMS (FAB) 295.2633, [C$_{19}$H$_{34}$O$_2$+H]$^+$ requires 295.2637.

**Z-Azacyclopentadec-6-en-2-one (13)**

According to *General Procedure 1*, diene 13a (0.11 g, 0.44 mmol) was reacted with 2 (0.022 g, 0.035 mmol) to provide **(Z)-13** (0.029 g, 30% yield, 84% Z as determined by $^1$H-NMR) as a colourless solid; $^1$H NMR $\delta$ 5.41 (2H, m), 5.28 (1H, m), 3.32 (2H, m), 2.02 (2H, t, $J$ = 6.1 Hz), 2.13 (2H, m), 1.99 (2H, m), 1.75 (2H, m), 1.52 (2H, m), 1.25-1.39 (10H, m); $^{13}$C NMR $\delta$ 172.7, 131.2, 129.4, 38.9, 35.3, 28.4, 27.9, 27.3, 26.9, 26.7 (2C), 25.8, 25.4, 25.3; HRMS (EI) 223.1930, [C$_{14}$H$_{25}$ON]$^+$ requires 223.1936.
**Z-tert-Butyl 2-oxoazacyclopentadec-6-ene-1-carboxylate (14)**

![Z-14](image)

According to General Procedure 1, diene 14a (0.15 g, 0.43 mmol) was reacted with 2 (0.022 g, 0.035 mmol) to provide Z-14 (0.082 g, 59% yield, 83% Z as determined by ¹H-NMR) as a colourless oil; ¹H NMR δ 5.37 (1H, m), 5.27 (1H, m), 3.83 (2H, t, J = 5.8 Hz), 2.86 (2H, t, J = 5.8 Hz), 2.16 (2H, m), 1.96 (2H, m), 1.75 (2H, m), 1.58 (2H, m), 1.51 (9H, m), 1.20-1.35 (10H, m); ¹³C NMR δ 176.0, 153.6, 131.3, 129.6, 82.5, 43.9, 36.2, 28.4, 28.1 (3C), 28.0, 27.6, 27.1, 27.0, 26.3, 25.9, 25.8, 24.4; HRMS (EI) 323.2447, [C₁⁹H₃₃O₃N]⁺ requires 323.2460.

**General procedure 2: Synthesis of E-enriched macrocycles⁴**

A solution of diene (1 equiv, 1.8 mmol) and benzylidene-bis(tricyclohexylphosphine)dichlororuthenium (0.074 g, 0.090 mmol) in CH₂Cl₂ (6 mM, 300 mL) was refluxed for six hours, then quenched with ethyl vinyl ether, and concentrated. Flash chromatography of the residue (SiO₂, using 2% Et₂O in pentanes for compounds 6 and 8, 10% Et₂O in pentanes for compound 10, and 66% Et₂O in pentanes for compound 13) provided the product.

**E-Oxacyclocicos-11-en-2-one (6)**

![E-6](image)

According to General Procedure 2, diene 6a provided E-6 (0.31 g, 58% yield, 69% E as determined by quantitative ¹³C-NMR) as a colourless oil; ¹H NMR δ 5.32 (m, 2H), 4.09 (t, J = 5.6 Hz, 3C), 28.0, 27.6, 27.1, 27.0, 26.3, 25.9, 25.8, 24.4; HRMS (EI) 323.2447, [C₁⁹H₃₃O₃N]⁺ requires 323.2460.

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2H), 2.31 (t, \( J = 6.7 \) Hz, 2H), 2.01 (m, 4H), 1.63 (m, 4H), 1.26-1.41 (m, 20H); \(^{13}\text{C}\) NMR \( \delta \) 174.2, 130.8, 130.8, 64.4, 34.2, 32.0, 31.9, 29.4, 29.2, 28.9 (2C), 28.8 (2C), 28.7, 28.3, 27.7, 27.6, 26.2, 25.1.

**E-Cycloheptadec-9-enone (8)**

\[\text{E-8}\]

According to General Procedure 2, diene 8a provided **E-8** (0.20 g, 44% yield, 80% \( E \) as determined by \(^1\text{H}-\text{NMR}) as a colourless solid; \(^1\text{H}\) NMR \( \delta \) 5.31 (m, 2H), 2.37 (t, \( J = 7.1 \) Hz, 4H), 2.01 (m, 4H) 1.60 (m, 4H), 1.22-1.37 (m, 16H); \(^{13}\text{C}\) NMR \( \delta \) 213.4, 131.2 (2C), 42.6 (2C), 32.1 (2C), 29.0 (2C), 28.9 (2C), 28.5 (2C), 27.6 (2C), 24.2 (2C).

**E-Cycloheptadec-9-enol (10)**

\[\text{E-10}\]

According to General Procedure 2, diene 10a provided **E-10** (0.18 g, 40% yield, 80% \( E \) as determined by quantitative \(^{13}\text{C}-\text{NMR}) as a colourless solid; \(^1\text{H}\) NMR \( \delta \) 5.34 (m, 2H), 3.71 (m, 1H), 2.02 (m, 4H), 1.50 (m, 4H), 1.23-1.36 (m, 20H); \(^{13}\text{C}\) NMR \( \delta \) 131.0 (2C), 71.4, 35.6 (2C), 32.4 (2C), 29.2 (2C), 28.7 (2C), 28.2 (2C), 27.4 (2C), 22.9 (2C).
**E-Azacyclopendec-6-en-2-one (13)**

![13](image)

Similar to **General Procedure 2**, except after 6 hours a second aliquot of 5 mol% bis(tricyclohexylphosphine)benzylidene ruthenium(IV) was added and the solution refluxed for an additional six hours, diene **13a** provided **E-13** (0.13 g, 33% yield, 55% E as determined by 1H-NMR) as a colourless solid. 1H NMR δ 5.36 (m, 2H), 5.32 (1H, overlapped), 3.27 (q, J = 5.7 Hz, 2H), 2.21 (t, J = 6.3 Hz, 2H), 2.13 (m, 2H), 2.01 (m, 2H), 1.78 (m, 2H), 1.45 (m, 2H), 1.34 (m, 8H), 1.26 (m, 2H); 13C NMR δ 172.9, 130.4, 130.1, 38.9, 34.6, 31.5 (2C), 27.8, 27.6, 26.4, 26.2, 25.2, 23.8, 23.4.

**General procedure 3: Z-selective ethenolysis of E-dominant macrocycles**

A solution of E-enriched macrocycle (1 equiv.) in THF (1M) was prepared in a 4 mL vial in a glovebox and sealed with a septum cap. Catalyst **2** (2 mol %) was added as a solution in a minimal amount of THF. The sealed vial was removed from the glovebox and stirred under an ethylene atmosphere. The reaction was heated (35 °C for **6** and **9**, 40 °C for **12**, 75 °C for **7**) for 2 hours, then quenched with ethyl vinyl ether and concentrated. Flash chromatography of the residue (SiO₂, using 2% Et₂O in pentanes for compounds **6** and **7**, 10% Et₂O in pentanes for compound **9**, and 66% Et₂O in pentanes for compound **12**) provided the product as the pure E-isomer. Isolated yields of the pure E-macrocycles and recovered diene were calculated based on the assumption that only the Z-isomer underwent ethenolysis and that it reacted completely.
**E-Oxacycloicos-11-en-2-one (6)**

According to General Procedure 3, macrocycle 6 (97 mg, 0.33 mmol, 69% E) was reacted with 2 (4.2 mg, 7.0 μmol) and provided the pure E-isomer of 6 (47 mg, 69% yield) and diene 6a (27 mg, 81% yield) as colourless oils; $^1$H NMR δ 5.32 (m, 2H), 4.09 (t, $J = 5.6$ Hz, 2H), 2.31 (t, $J = 6.7$ Hz, 2H), 2.01 (m, 4H), 1.63 (m, 4H), 1.26-1.41 (m, 20H); $^{13}$C NMR δ 174.2, 130.8, 130.8, 64.4, 34.2, 32.0, 31.9, 29.4, 29.2, 28.9 (2C), 28.8 (2C), 28.7, 28.3, 27.7, 27.6, 26.2, 25.1. HRMS (EI) 294.2549, [C$_{19}$H$_{34}$O$_2$]$^+$ requires 294.2559.

**E-Cycloheptadec-9-enone (8)**

According to General Procedure 3, macrocycle 8 (156 mg, 0.62 mmol, 80% E) was reacted with 2 (7.9 mg, 12 μmol) and provided the pure E-isomer of 8 (50 mg, 40% yield) and diene 8a (16 mg, 46% yield) as colourless solids; $^1$H NMR δ 5.31 (m, 2H), 2.37 (t, $J = 7.1$ Hz, 4H), 2.01 (m, 4H) 1.60 (m, 4H), 1.22-1.37 (m, 16H); $^{13}$C NMR δ 213.4, 131.2, 42.6, 32.1, 29.0, 28.9, 28.5, 27.6, 24.2. HRMS (FAB) 251.2372, [C$_{17}$H$_{30}$O+H]$^+$ requires 251.2375.
**E-Cycloheptadec-9-enol (10)**

![E-10](image)

According to General Procedure 3, macrocycle 10 (108 mg, 0.43 mmol, 80% E) was reacted with 2 (5.4 mg, 9.0 μmol) and provided the pure E-isomer of 10 (68 mg, 78% yield) and diene 10a (19 mg, 79% yield) as colourless solids; ¹H NMR δ 5.34 (m, 2H), 3.71 (m, 1H), 2.02 (m, 4H), 1.50 (m, 4H), 1.23-1.36 (m, 20H); ¹³C NMR δ 131.0 (2C), 71.4, 35.6 (2C), 32.4 (2C), 29.2 (2C), 28.7 (2C), 28.2 (2C), 27.4 (2C), 22.9 (2C). HRMS (FAB) 251.2371, [C₁₇H₃₂O₂-H]+ requires 251.2375.

**E-Azacyclopentadec-6-en-2-one (13)**

![E-13](image)

According to General Procedure 3, macrocycle 13 (51 mg, 0.22 mmol, 55% E) was reacted with 2 (2.8 mg, 4.4 μmol) and provided the pure E-isomer of 13 (21 mg, 75% yield) as a colourless solid, and diene 13a (21 mg, 86% yield) as a pale yellow oil; ¹H NMR δ 5.36 (m, 2H), 5.32 (1H, overlapped), 3.27 (q, J = 5.7 Hz, 2H), 2.21 (t, J = 6.3 Hz, 2H), 2.13 (m, 2H), 2.01 (m, 2H), 1.78 (m, 2H), 1.45 (m, 2H), 1.34 (m, 8H), 1.26 (m, 2H); ¹³C NMR δ 172.9, 130.4, 130.1, 38.9, 34.6, 31.5 (2C), 27.8, 27.6, 26.4, 26.2, 25.2, 23.8, 23.4. HRMS (FAB) 224.2014, [C₁₄H₂₅NO+H]+ requires 224.2014.
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of but-3-en-1-yl undec-10-enoate (1a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of but-3-en-1-yl undec-10-enoate (1a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of but-3-en-1-yl dec-9-enoate (4a)
$^{13}\text{C NMR (CDCl}_3, 125 \text{ MHz)}$ spectrum of but-3-en-1-yl dec-9-enoate (4a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of undec-10-en-1-yl hex-5-enoate (5a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of undec-10-en-1-yl hex-5-enoate (5a)
$^1$H NMR ($\text{CDCl}_3$, 500 MHz) spectrum of dec-9-en-1-yl undec-10-enoate (6a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of dec-9-en-1-yl undec-10-enoate (6a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of dec-9-en-1-yl oct-7-enoate (7a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of dec-9-en-1-yl oct-7-enolate (7a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of nonadeca-1,18-dien-10-one (8a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of nonadeca-1,18-dien-10-one (8a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of 2,2-di(non-8-en-1-yl)-1,3-dioxolane (9a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of 2,2-di(non-8-en-1-yl)-1,3-dioxolane (9a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of nonadeca-1,18-dien-10-ol (10a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of nonadeca-1,18-dien-10-ol (10a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of tert-butyldimethyl(nonadeca-1,18-dien-10-yloxy)silane (11a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of tert-butylidimethyl(nonadeca-1,18-dien-10-yloxy)silane (11a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of nonadeca-1,18-dien-10-yl acetate (12a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of nonadeca-1,18-dien-10-yl acetate (12a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of $N$-(dec-9-en-1-yl)hex-5-enamide (13a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of $N$-(dec-9-en-1-yl)hex-5-enamide (13a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of tert-butyl dec-9-en-1-yl(hex-5-enoyl)carbamate (14a)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of tert-butyl dec-9-en-1-yl(hex-5-enoyl)carbamate (14a)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-oxacyclotetradec-11-en-2-one (1)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of Z-oxacyclotetradec-11-en-2-one (1)
HSQC spectrum of Z-oxacyclotetradec-11-en-2-one (1)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-oxacyclotridec-10-en-2-one (4)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of Z-oxacyclotridec-10-en-2-one (4)
HSQC spectrum of Z-oxacyclotridec-10-en-2-one (4)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-oxacyclohexadec-6-en-2-one (5)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of Z-oxacyclohexadec-6-en-2-one (5)

Z-5
HSQC spectrum of Z- oxacyclohexadec-6-en-2-one (5)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-oxacycloicos-11-en-2-one (6)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of Z-oxacycloicos-11-en-2-one (6)
HSQC spectrum of Z-oxacycloicos-11-en-2-one (6)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-oxacycloheptadec-8-en-2-one (7)
$^{13}\text{C NMR (CDCl}_3, 125 \text{ MHz) spectrum of Z-oxacycloheptadec-8-en-2-one (7)}$
HSQC spectrum of Z-oxacycloheptadec-8-en-2-one (7)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-cycloheptadec-9-enone (8)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of Z-cycloheptadec-9-enone (8)
HSQC spectrum of $Z$-cycloheptadec-9-enone (8)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-1,4-dioxaspiro[4.16]henicos-13-ene (9)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of Z-1,4-dioxaspiro[4.16]henicos-13-ene (9)
HSQC spectrum of Z-1,4-dioxaspiro[4.16]henicos-13-ene (9)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-cycloheptadec-9-enol (10)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of Z-cycloheptadec-9-enol (10)
HSQC spectrum of Z-cycloheptadec-9-enol (10)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-tert-butyl(cycloheptadec-9-en-1-yloxy)dimethylsilane (11)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of $Z$-tert-butyl(cycloheptadec-9-en-1-yloxy)dimethylsilane (11)
HSQC spectrum of Z-tert-butyl(cycloheptadec-9-en-1-yloxy)dimethylsilane (11)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-cycloheptadec-9-en-1-yl acetate (12)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of Z-cycloheptadec-9-en-1-yl acetate (12)
HSQC spectrum of Z-cycloheptadec-9-en-1-yl acetate (12)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-azacyclopentadec-6-en-2-one (13)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of Z-azacyclopentadec-6-en-2-one (13)
HSQC spectrum of Z-azacyclopentadec-6-en-2-one (13)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of Z-tert-butyl 2-oxoazacyclopentadec-6-ene-1-carboxylate (14)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of $Z$-tert-butyl 2-oxoazacyclopentadec-6-ene-1-carboxylate (14)
HSQC spectrum of Z-tert-butyl 2-oxoazacyclopentadec-6-ene-1-carboxylate (14)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of $E$-oxacycloicos-11-en-2-one (6)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of $E$-oxacycloicos-11-en-2-one (6)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of E-cycloheptadec-9-enone (8)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of E-cycloheptadec-9-enone (8)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of $E$-cycloheptadec-9-enol (10)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of $E$-cycloheptadec-9-enol (10)
$^{1}$H NMR (CDCl$_3$, 500 MHz) spectrum of $E$-azacyclopentadec-6-en-2-one (13)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of $E$-azacyclopentadec-6-en-2-one (13)
$^1$H NMR (CDCl$_3$, 500 MHz) spectrum of 9-iodonon-1-ene (16)
$^{13}$C NMR (CDCl$_3$, 125 MHz) spectrum of 9-iodonon-1-ene (16)