Supporting Information

Formal [5 + 2] Cycloaddition of Vinylethylene Carbonates with Oxazol-5-(4H)-ones for Synthesis of 3,4-Dihydrooxepin-2(7H)-ones

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1. General Information

Proton (\(^{1}\)H) and carbon (\(^{13}\)C) NMR spectra were recorded on 400 MHz instrument (400 MHz for \(^{1}\)H NMR, 100 MHz for \(^{13}\)C NMR) and calibrated using tetramethylsilane (TMS) as internal reference. High resolution mass spectra (HRMS) were recorded under electrospray ionization (ESI) conditions. The melting point of compounds was determined by a melting point instrument. Thin layer chromatography (TLC) was carried out on 0.25 mm SDS silica gel coated glass plates (60F254). Eluted plates were visualized using a 254 nm UV lamp. Unless otherwise indicated, all reagents were commercially available and used without further purification. All solvents were distilled from the appropriate drying agents immediately before using. Substituted vinylene carbonates 1a-1j were synthesized according to the reported procedures. Oxazol-5-(4H)-ones 2a-2j were prepared according to literature procedures.

2. General Procedure for [5 + 2] Cycloaddition

A mixture of vinylene carbonates 1 (1.0 equiv, 0.1 mmol), oxazol-5-(4H)-ones 2 (1.0 equiv, 0.1 mmol), Pd\(_2\) (2.5 mol%), PPh\(_3\) (10.0 mol%) and TMSCl (1.0 eq) in THF (1.0 mL) was stirred at rt until the reaction was completed as indicated by TLC plate. The reaction mixture was concentrated under reduced pressure and the resulted crude product was purified by flash column chromatography on silica gel (petroleum ether / ethyl acetate/CH\(_2\)Cl\(_2\) = 10:1:10) to afford products 3 (67-99% yields).


A mixture of vinylene carbonate 1a (1.0 equiv, 0.1 mmol), oxazol-5-(4H)-one 2a (1.0 equiv, 0.1 mmol), Pd precatalyst (2.5 mol%), chiral ligand (10.0 mol%) and Et\(_3\)N (1.0 equiv, 0.1mmol) was stirred in THF (1.0 mL) at rt until the reaction was completed indicated by TLC plate. The reaction mixture was concentrated under reduced pressure and the resulted crude product was purified by flash column chromatography on silica gel (petroleum ether / ethyl acetate/CH\(_2\)Cl\(_2\) = 10:1:10) to afford product 3aa.

Table S1 Screening of Pd precatalysts

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(S,S,S)-L3

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**Table S2** Screening of chiral ligands

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<th>Entry</th>
<th>Ligand</th>
<th>Time (h)</th>
<th>Yield&lt;sup&gt;b&lt;/sup&gt; (%)</th>
<th>ee&lt;sup&gt;c&lt;/sup&gt;</th>
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<td>6</td>
<td>L6</td>
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<td>7</td>
<td>L7</td>
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<sup>a</sup> Reactions were carried out with 1a (0.1 mmol), 2a (0.1 mmol), [Pd] (2.5 mol%), L3 (10.0 mol%), Et<sub>3</sub>N (0.1 mmol) in THF (1.0 mL) at rt. <sup>b</sup> Isolated yield. <sup>c</sup> Determined by chiral HPLC analysis.

4. Screening of Solvents

A mixture of vinylene carbonate 1a (1.0 equiv, 0.1 mmol), oxazol-5-(4H)-one 2a (1.0 equiv, 0.1 mmol), Pd<sub>2</sub>(dba)<sub>3</sub> (2.5 mol%), PPh<sub>3</sub> (10.0 mol%) and Et<sub>3</sub>N (1.0 equiv, 0.1 mmol) in the specified solvent (1.0 mL) was stirred at rt until the reaction was completed indicated by TLC plate. The reaction mixture was concentrated under reduced pressure and the resulted crude products were purified by flash column chromatography on silica gel (petroleum ether / ethyl acetate/CH<sub>2</sub>Cl<sub>2</sub> = 10:1:10) to afford product 3aa.
Table S3 Screening of Solvents

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<td>2</td>
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<td>3</td>
<td>Toluene</td>
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<td>4</td>
<td>EtOH</td>
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<td>1,2-DCE</td>
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<tr>
<td>6</td>
<td>CHCl₃</td>
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</tr>
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aReactions were carried out with 1a (0.1 mmol), 2a (0.1 mmol), Pd₂dba₃ (2.5 mol%), PPh₃ (10.0 mol%), Et₃N (0.1mmol) in the specified solvent (1.0 mL) at rt. bIsolated yield.

5. Extension of Reaction Scope in the Presence of Et₃N

A mixture of vinylethylene carbonates 1 (1.0 equiv, 0.1 mmol), oxazol-5-(4H)-ones 2 (1.0 equiv, 0.1 mmol), Pd₂dba₃ (2.5 mol%), PPh₃ (10.0 mol%) and Et₃N (0.1mmol) in THF (1.0 mL) was stirred at room temperature. The reaction mixture was concentrated under reduced pressure and the crude products were purified by flash column chromatography on silica gel (petroleum ether / ethyl acetate/CH₂Cl₂ = 10:1:10) to afford products 3.

Table S4 Extension of reaction scope

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<td>2f (3,4-di-ClC₆H₃, Bn)</td>
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<td>2h (Ph, H)</td>
<td>3ah</td>
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<td>nr³</td>
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<td>2a (Ph, Bn)</td>
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6. Characterization

N-(3-benzyl-2-oxo-6-phenyl-2,3,4,7-tetrahydrooxepin-3-yl)benzamide (3aa): White solid, yield: 31.8 mg, 80%; M.P. = 172.8-173.9 °C; 'H NMR (400 MHz, CDCl3): δ 7.74 (d, J = 7.6 Hz, 2H), 7.55 (t, J = 6.8 Hz, 1H), 7.46-7.43 (m, 2H), 7.35-7.28 (m, 8H), 7.21 (d, J = 7.2 Hz, 2H), 6.71 (s, 1H), 6.08-6.06 (m, 1H), 5.47 (d, J = 15.6 Hz, 1H), 5.02 (d, J = 15.6 Hz, 1H), 3.67 (d, J = 14.0 Hz, 1H), 3.44 (d, J = 14.0 Hz, 1H), 3.13 (dd, J = 17.6, 5.6 Hz, 1H), 2.90 (d, J = 17.6 Hz, 1H) ppm; 13C NMR (100 MHz, CDCl3): δ 172.5, 167.0, 140.1, 139.6, 135.3, 133.3, 132.2, 130.8, 128.8, 128.7, 128.6, 128.1, 127.4, 127.0, 126.0, 125.9, 69.0, 63.2, 41.2, 33.5 ppm; HRMS (ESI) calculated for C_{26}H_{23}NO_{3}[M + H]^+: 398.1751, found 398.1750.

N-(3-benzyl-2-oxo-6-phenyl-2,3,4,7-tetrahydrooxepin-3-yl)4-methylbenzamide (3ab): White solid, yield: 27.5 mg, 67%; M.P. = 163.5-163.9 °C; 'H NMR (400 MHz, CDCl3): δ 7.65 (d, J = 8.0 Hz, 2H), 7.35-7.30 (m, 6H), 7.28-7.18 (m, 6H), 6.71 (s, 1H), 6.03 (d, J = 4.4 Hz, 1H), 5.45 (d, J = 15.6 Hz, 1H), 4.95 (d, J = 15.2 Hz, 1H), 3.69 (d, J = 14.0 Hz, 1H), 3.37 (d, J = 14.0 Hz, 1H), 3.08 (dd, J = 17.6, 5.6 Hz, 1H), 2.85 (d, J = 18.0 Hz, 1H), 2.41 (s, 3H) ppm; 13C NMR (100 MHz, CDCl3): δ 172.3, 167.0, 142.8, 139.8, 139.7, 135.5, 130.9, 129.5, 128.7, 128.6, 128.0, 127.3, 127.1, 126.1, 125.8, 68.9, 62.7, 41.3, 33.7, 21.5 ppm; HRMS (ESI) calculated for C_{27}H_{26}NO_{3}[M + H]^+: 412.1907, found 412.1911.

N-(3-benzyl-2-oxo-6-phenyl-2,3,4,7-tetrahydrooxepin-3-yl)4-bromobenzamide (3ac): White solid, yield: 36.5 mg, 77%; M.P. = 157.0-157.5 °C; 'H NMR (400 MHz, CDCl3): δ 7.58 (s, 4H), 7.37-7.28 (m, 8H), 7.18 (d, J = 6.4 Hz, 2H), 6.72 (s, 1H), 6.10 (t, J = 5.2 Hz, 1H), 5.43 (d, J = 15.2 Hz, 1H), 5.05 (d, J = 15.2 Hz, 1H), 3.62 (d, J = 14.0 Hz, 1H), 3.35 (d, J = 14.0 Hz, 1H), 3.16 (dd, J = 17.6, 5.6 Hz, 1H), 2.92 (dd, J = 17.2, 4.4 Hz, ppm; 13C NMR (100 MHz, CDCl3): δ 172.5, 166.0, 140.1, 139.4, 135.2, 132.2, 132.1, 130.6, 128.8, 128.7, 128.6, 128.1, 127.5, 127.0, 126.1, 126.0, 69.0, 63.7, 41.1, 33.4 ppm; HRMS (ESI) calculated for C_{26}H_{23}BrNO_{3} [M + H]^+: 476.0856, found 476.0856.

N-(3-benzyl-2-oxo-6-phenyl-2,3,4,7-tetrahydrooxepin-3-yl)4-chlorobenzamide (3ad): White solid, yield: 38.8 mg, 90%; M.P. = 104.8-105.6 °C; 'H NMR (400 MHz, CDCl3): δ 7.66 (d, J = 8.4 Hz, 2H), 7.41 (d, J = 8.4 Hz, 2H), 7.36-7.28 (m, 8H), 7.19 (d, J = 7.2 Hz, 2H), 6.72 (s, 1H), 6.10 (t, J = 5.2 Hz, 1H), 5.43 (d, J = 15.2 Hz, 1H), 5.05 (d, J = 15.6 Hz, 1H), 3.62 (d, J = 14.0 Hz, 1H), 3.46 (d, J = 14.4 Hz, 1H), 3.16 (dd, J = 17.6, 6.0 Hz, 1H), 2.92 (dd, J = 17.2, 4.0 Hz, 1H) ppm; 13C NMR (100 MHz,
CDCl₃): δ 172.6, 165.9, 140.2, 139.4, 138.5, 135.2, 131.8, 130.6, 129.1, 128.7, 128.6, 128.5, 128.1, 127.5, 126.1, 126.0, 69.0, 63.7, 41.1, 33.4 ppm; HRMS (ESI) calculated for C₂₆H₂₃ClNO₃ [M + H]⁺: 432.1361, found 432.1358.

N-(3-benzyl-2-oxo-6-phenyl-2,3,4,7-tetrahydroxepin-3-yl)-3-bromobenzamide (3a): White solid, yield: 33.4 mg, 70%; M.P. = 178.6-178.9 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.87 (s, 1H), 7.65 (d, J = 7.6 Hz, 1H), 7.62 (d, J = 7.6 Hz, 1H), 7.35-7.28 (m, 9H), 7.19 (d, J = 7.2 Hz, 2H), 6.78 (s, 1H), 6.09 (t, J = 4.0 Hz, 1H), 5.42 (d, J = 15.6 Hz, 1H), 5.03 (d, J = 15.2 Hz, 1H), 3.62 (d, J = 14.0 Hz, 1H), 3.44 (d, J = 14.0 Hz, 1H), 3.15 (dd, J = 17.6, 5.6 Hz, 1H), 2.91 (d, J = 14.4 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 172.5, 165.6, 140.0, 139.4, 135.4, 135.2, 135.1, 130.7, 130.4, 130.3, 128.8, 128.7, 128.1, 127.5, 126.1, 126.0, 125.5, 123.0, 69.0, 63.7, 41.1, 33.4 ppm; HRMS (ESI) calculated for C₂₆H₂₃BrNO₃ [M + H]⁺: 476.0856, found 476.0858.

N-(3-benzyl-2-oxo-6-phenyl-2,3,4,7-tetrahydroxepin-3-yl)-3,4-dichlorobenzamide (3a): White solid, yield: 32.0 mg, 69%; M.P. = 171.5-172.1 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.80 (s, 1H), 7.50 (s, 2H), 7.35-7.28 (m, 8H), 7.18 (d, J = 6.4 Hz, 2H), 6.83 (s, 1H), 6.11 (s, 1H), 5.39 (d, J = 15.2 Hz, 1H), 5.04 (d, J = 15.2 Hz, 1H), 3.59 (d, J = 14.0 Hz, 1H), 3.45 (d, J = 14.0 Hz, 1H), 3.17 (dd, J = 17.2, 5.2 Hz, 1H), 2.93 (d, J = 14.8 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 172.5, 164.9, 139.9, 139.3, 136.7, 135.1, 133.4, 133.3, 130.8, 130.6, 129.3, 128.7, 127.6, 126.2, 126.1, 126.0, 69.1, 64.0, 41.1, 33.3 ppm; HRMS (ESI) calculated for C₂₆H₂₃Cl₂NO₃ [M + H]⁺: 466.0971, found 466.0971.

N-(3-benzyl-6-(4-bromophenyl)-2-oxo-2,3,4,7-tetrahydroxepin-3-yl)benzamide (3b): White solid, yield: 43.0 mg, 91%; M.P. = 190.8-191.4 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.73 (d, J = 7.6 Hz, 2H), 7.55 (t, J = 7.2 Hz, 1H), 7.45 (t, J = 8.0 Hz, 4H), 7.32-7.28 (m, 3H), 7.20-7.13 (m, 4H), 6.72 (s, 1H), 6.07 (s, 1H), 5.45 (d, J = 15.2 Hz, 1H), 4.92 (d, J = 15.2 Hz, 1H), 3.62 (d, J = 14.0 Hz, 1H), 3.40 (d, J = 14.0 Hz, 1H), 3.13 (dd, J = 17.6, 5.2 Hz, 1H), 2.84 (d, J = 15.6 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 172.4, 167.0, 139.1, 138.4, 135.2, 133.2, 132.3, 131.8, 130.8, 128.8, 128.7, 127.6, 127.5, 127.1, 126.9, 122.1, 68.4, 63.1, 41.5, 33.5 ppm; HRMS (ESI) calculated for C₂₆H₂₃BrNO₃ [M + H]⁺: 476.0856, found 476.0854.

N-(3-benzyl-6-(4-chlorophenyl)-2-oxo-2,3,4,7-tetrahydroxepin-3-yl)benzamide (3c): White solid, yield: 43.0 mg, 99%; M.P. = 202.0-202.9 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.73 (d, J = 7.6 Hz, 2H), 7.56-7.53 (m, 1H), 7.44 (t, J = 7.2 Hz, 2H), 7.30-7.29 (m, 5H), 7.21-7.19 (m, 4H), 6.74 (s, 1H), 6.06 (s, 1H), 5.45 (d, J = 15.2 Hz, 1H), 4.92 (d, J = 15.2 Hz, 1H), 3.62 (d, J = 14.0 Hz, 1H), 3.39 (d, J = 14.0 Hz, 1H), 3.14 (dd, J = 17.6, 4.8 Hz, 1H), 2.85 (d, J = 16.8 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 172.4, 167.0, 139.0, 138.0, 135.2, 134.0, 133.2, 132.3, 130.8, 128.9, 128.8, 128.7, 127.5, 127.3, 127.1, 126.8, 68.5, 63.1, 41.5, 33.5 ppm; HRMS (ESI) calculated for C₂₆H₂₃ClNO₃ [M + H]⁺: 432.1361, found 432.1356.

N-(3-benzyl-6-(4-fluorophenyl)-2-oxo-2,3,4,7-tetrahydroxepin-3-yl)benzamide (3d): White solid, yield: 35.0 mg, 84%; M.P. = 194.5-195.0 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.74 (d, J = 7.2 Hz, 2H), 7.56-7.53 (m, 1H), 7.44 (t, J = 7.2 Hz, 2H), 7.32-7.28 (m, 3H), 7.26-7.23 (m, 2H), 7.20 (d, J = 4.0 Hz, 2H), 7.02 (t, J = 8.0 Hz, 2H), 6.74 (s, 1H), 6.02 (s, 1H), 5.45 (d, J = 15.2 Hz, 1H), 4.94 (d, J = 15.2 Hz, 1H),
N-(3-benzyl-2-oxo-6-(p-tolyl)-2,3,4,7-tetrahydrooxepin-3-yl)benzamide (3ea): White solid, yield: 28.0 mg, 68%; M.P. = 193.3-194.1 °C; 1H NMR (400 MHz, CDCl3): δ 7.74 (d, J = 7.6 Hz, 2H), 7.54 (t, J = 7.2 Hz, 1H), 7.44 (t, J = 7.2 Hz, 2H), 7.33-7.28 (m, 3H), 7.21-7.14 (m, 6H), 6.72 (s, 1H), 6.04 (s, 1H), 5.44 (d, J = 15.2 Hz, 1H), 5.00 (d, J = 15.2 Hz, 1H), 3.67 (d, J = 14.0 Hz, 1H), 3.42 (d, J = 14.0 Hz, 1H), 3.11 (dd, J = 17.6, 5.2 Hz, 1H), 2.88 (d, J = 16.4 Hz, 1H), 2.35 (s, 3H) ppm; 13C NMR (100 MHz, CDCl3): δ 172.5, 167.0, 139.8, 138.0, 136.7, 135.4, 133.3, 132.2, 130.8, 129.4, 128.8, 128.6, 127.4, 127.1, 125.9, 125.0, 69.0, 63.1, 41.2, 33.5, 21.1 ppm; HRMS (ESI) calculated for C29H25FNO5 [M + H]+: 416.1656, found 416.1657.

N-(3-benzyl-6-(4-methoxyphosphonyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)benzamide (3fa): White solid, yield: 35.0 mg, 82%; M.P. = 199.2-200.3 °C; 1H NMR (400 MHz, CDCl3): δ 7.73 (d, J = 7.6 Hz, 2H), 7.54 (t, J = 7.2 Hz, 1H), 7.44 (t, J = 7.2 Hz, 2H), 7.32-7.28 (m, 3H), 7.23-7.19 (m, 4H), 6.87 (d, J = 8.4 Hz, 2H), 6.73 (s, 1H), 5.99 (s, 1H), 5.43 (d, J = 15.2 Hz, 1H), 4.98 (d, J = 15.2 Hz, 1H), 3.80 (s, 3H), 3.66 (d, J = 14.0 Hz, 1H), 3.41 (d, J = 14.0 Hz, 1H), 3.10 (dd, J = 17.6, 5.6 Hz, 1H), 2.86 (d, J = 15.6 Hz, 1H) ppm; 13C NMR (100 MHz, CDCl3): δ 172.5, 167.0, 159.6, 139.5, 135.4, 133.3, 132.2, 132.0, 130.8, 128.8, 128.6, 127.4, 127.2, 127.1, 124.4, 114.1, 69.0, 63.1, 55.3, 41.2, 33.5 ppm; HRMS (ESI) calculated for C27H26NO5 [M + H]+: 428.1856, found 428.1850.

N-(3-benzyl-6-(3-chlorophenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)benzamide (3ga): White solid, yield: 36.0 mg, 84%; M.P. = 202.0-202.1 °C; 1H NMR (400 MHz, CDCl3): δ 7.73 (d, J = 7.6 Hz, 2H), 7.55 (t, J = 7.2 Hz, 1H), 7.47-7.43 (m, 2H), 7.33-7.28 (m, 4H), 7.27-7.16 (m, 5H), 6.71 (s, 1H), 6.10 (s, 1H), 5.46 (d, J = 14.8 Hz, 1H), 4.95 (d, J = 15.2 Hz, 1H), 3.62 (d, J = 13.6 Hz, 1H), 3.43 (d, J = 13.6 Hz, 1H), 3.17 (dd, J = 17.2, 5.2 Hz, 1H), 2.87 (d, J = 14.0 Hz, 1H) ppm; 13C NMR (100 MHz, CDCl3): δ 172.4, 167.0, 141.3, 139.1, 135.1, 134.7, 133.2, 132.3, 130.8, 129.9, 128.8, 128.7, 128.1, 127.6, 127.5, 127.1, 126.3, 124.1, 68.4, 63.3, 41.6, 33.5 ppm; HRMS (ESI) calculated for C26H25ClNO5 [M + H]+: 432.1361, found 432.1356.

N-(3-benzyl-6-(4-fluorophenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-4-methylbenzamide (3db): White solid, yield: 30.0 mg, 70%; M.P. = 196.8-197.4 °C; 1H NMR (400 MHz, CDCl3): δ 7.63 (d, J = 7.6 Hz, 2H), 7.32-7.28 (m, 3H), 7.26-7.18 (m, 6H), 7.02 (t, J = 8.4 Hz, 2H), 6.60 (s, 1H), 6.01 (t, J = 4.0 Hz, 1H), 5.44 (d, J = 15.2 Hz, 1H), 4.95 (d, J = 15.2 Hz, 1H), 3.64 (d, J = 14.0 Hz, 1H), 3.42 (d, J = 14.0 Hz, 1H), 3.11 (dd, J = 17.6, 6.0 Hz, 1H), 2.88-2.84 (m, 1H), 2.41 (s, 3H) ppm; 13C NMR (100 MHz, CDCl3): δ 172.4, 166.9, 162.5 (d, J = 246.2 Hz), 142.8, 139.2, 135.7 (d, J = 3.2 Hz), 135.3, 130.8, 130.4, 129.5, 128.6, 127.7 (d, J = 8.0 Hz), 127.4, 127.1, 126.1, 115.6 (d, J = 21.5 Hz), 68.8, 63.0, 41.4, 33.5, 21.5 ppm; HRMS (ESI) calculated for C27H25FNO5 [M + H]+: 430.1813, found 430.1806.
N-(3-benzyl-6-(4-fluorophenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-4-bromobenzamide (3dc): White solid, yield: 47.0 mg, 95%; M.P. = 199.1-199.5 °C; ^1H NMR (400 MHz, CDCl3): δ 7.58 (s, 4H), 7.32-7.29 (m, 3H), 7.28-7.17 (m, 4H), 7.03 (t, J = 8.4 Hz, 2H), 6.73 (s, 1H), 6.05 (t, J = 5.2 Hz, 1H), 5.41 (d, J = 15.2 Hz, 1H), 4.99 (d, J = 14.8 Hz, 1H), 3.58 (d, J = 14.0 Hz, 1H), 3.45 (d, J = 14.0 Hz, 1H), 3.17 (dd, J = 17.2, 5.6 Hz, 1H), 2.89 (dd, J = 17.2, 4.4 Hz, 1H) ppm; ^13C NMR (100 MHz, CDCl3): δ 172.5, 166.0, 162.6 (d, J = 246.5 Hz), 139.3, 135.5 (d, J = 3.2 Hz), 135.1, 132.2, 132.1, 130.6, 128.7, 128.6, 127.7 (d, J = 8.1 Hz), 127.5, 127.0, 126.3, 115.6 (d, J = 21.4 Hz), 68.9, 63.7, 41.3, 33.3 ppm; HRMS (ESI) calculated for C_{26}H_{22}BrFNO_3 [M + H]^+: 494.0762, found 494.0766.

N-(3-benzyl-6-(4-fluorophenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-4-chlorobenzamide (3dd): White solid, yield: 38.0 mg, 85%; M.P. = 199.8-200.4 °C; ^1H NMR (400 MHz, CDCl3): δ 7.66 (d, J = 8.4 Hz, 2H), 7.42 (d, J = 8.4 Hz, 2H), 7.34-7.29 (m, 3H), 7.28-7.17 (m, 4H), 7.04 (t, J = 8.4 Hz, 2H), 6.71 (s, 1H), 6.05 (t, J = 5.2 Hz, 1H), 5.42 (d, J = 15.2 Hz, 1H), 5.00 (d, J = 15.2 Hz, 1H), 3.59 (d, J = 14.0 Hz, 1H), 3.45 (d, J = 14.0 Hz, 1H), 3.17 (dd, J = 17.6, 6.0 Hz, 1H), 2.89 (dd, J = 17.6, 5.2 Hz, 1H) ppm; ^13C NMR (100 MHz, CDCl3): δ 172.6, 165.9, 162.6 (d, J = 246.5 Hz), 139.4, 138.6, 135.4 (d, J = 3.2 Hz), 135.1, 131.8, 130.6, 129.1, 128.7, 128.4, 127.7 (d, J = 8.1 Hz), 127.5, 126.3, 115.7 (d, J = 21.4 Hz), 68.9, 63.8, 41.3, 33.3 ppm; HRMS (ESI) calculated for C_{26}H_{22}ClFNO_3 [M + H]^+: 450.1267, found 450.1270.

N-(3-benzyl-6-(4-fluorophenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-3-bromobenzamide (3de): White solid, yield: 38.0 mg, 77%; M.P. = 181.6-182.4 °C; ^1H NMR (400 MHz, CDCl3): δ 7.86 (s, 1H), 7.65 (d, J = 8.0 Hz, 1H), 7.61 (d, J = 7.6 Hz, 1H), 7.34-7.29 (m, 4H), 7.27-7.23 (m, 2H), 7.18 (d, J = 7.2 Hz, 2H), 7.03 (t, J = 8.4 Hz, 2H), 6.77 (s, 1H), 6.05 (t, J = 5.2 Hz, 1H), 5.40 (d, J = 15.2 Hz, 1H), 4.97 (d, J = 15.2 Hz, 1H), 3.59 (d, J = 14.0 Hz, 1H), 3.44 (d, J = 14.0 Hz, 1H), 3.16 (dd, J = 17.6, 6.0 Hz, 1H), 2.89 (dd, J = 17.2, 4.4 Hz, 1H) ppm; ^13C NMR (100 MHz, CDCl3): δ 172.5, 165.6, 162.6 (d, J = 246.3 Hz), 139.2, 135.5 (d, J = 3.3 Hz), 135.4, 135.1, 135.0, 130.6, 130.4, 130.3, 128.7, 127.7 (d, J = 8.0 Hz), 127.5, 126.3, 125.5, 123.0, 115.6 (d, J = 21.4 Hz), 68.9, 63.8, 41.3, 33.4 ppm; HRMS (ESI) calculated for C_{26}H_{22}BrFNO_3 [M + H]^+: 494.0762, found 494.0766.

N-(3-benzyl-2-oxo-6-(p-tolyl)-2,3,4,7-tetrahydrooxepin-3-yl)-4-methylbenzamide (3eb): White solid, yield: 30.0 mg, 71%; M.P. = 187.3-187.9 °C; ^1H NMR (400 MHz, CDCl3): δ 7.64 (d, J = 8.0 Hz, 2H), 7.32-7.28 (m, 3H), 7.24 (d, J = 7.6 Hz, 2H), 7.20-7.14 (m, 6H), 6.63 (s, 1H), 6.01 (s, 1H), 5.43 (d, J = 15.2 Hz, 1H), 4.98 (d, J = 15.6 Hz, 1H), 3.69 (d, J = 14.0 Hz, 1H), 3.38 (d, J = 14.0 Hz, 1H), 3.06 (dd, J = 17.6, 5.2 Hz, 1H), 2.85 (d, J = 16.4 Hz, 1H), 2.41 (s, 3H), 2.35 (s, 3H) ppm; ^13C NMR (100 MHz, CDCl3): δ 172.4, 166.9, 142.8, 139.6, 138.0, 136.8, 135.5, 130.9, 130.4, 129.5, 129.4, 128.6, 127.3, 127.1, 125.9, 124.9, 69.0, 62.8, 41.2, 33.6, 21.5, 21.1 ppm; HRMS (ESI) calculated for C_{26}H_{22}NO_3 [M + H]^+: 426.2064, found 426.2056.

N-(3-benzyl-2-oxo-6-(p-tolyl)-2,3,4,7-tetrahydrooxepin-3-yl)-4-bromobenzamide (3ec): White solid, yield: 46.0 mg, 94%; M.P. = 209.0-209.9 °C; ^1H NMR (400 MHz, CDCl3): δ 7.57 (s, 4H), 7.32-7.28 (m, 3H), 7.18-7.14 (m, 6H), 6.74 (s, 1H), 6.06 (t, J = 5.2 Hz, 1H), 5.40 (d, J = 15.2 Hz, 1H), 5.02 (d, J = 15.2 Hz, 1H), 3.62 (d, J = 14.0 Hz, 1H), 3.43 (d, J = 14.0 Hz, 1H), 3.13 (dd, J = 17.6, 5.6 Hz, 1H),
2.92-2.88 (m, 1H), 2.35 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): δ 172.6, 166.0, 139.9, 138.1, 136.5, 135.2, 132.3, 132.0, 130.7, 129.4, 128.7, 128.6, 127.5, 127.0, 125.9, 125.2, 69.1, 63.6, 41.0, 33.3, 21.1 ppm; HRMS (ESI) calculated for C$_{27}$H$_{33}$BrNO$_3$ [M + H]: 490.1012, found 490.1010.

N-(3-benzyl-2-oxo-6-(p-tolyl)-2,3,4,7-tetrahydrooxepin-3-yl)-4-chlorobenamide (3ed): White solid, yield: 35.0 mg, 79%; M.P. = 204.7-205.0 °C; $^1$H NMR (400 MHz, CDCl$_3$):
δ 7.66 (d, J = 8.4 Hz, 2H), 7.41 (d, J = 8.4 Hz, 2H), 7.33-7.28 (m, 3H), 7.18-7.15 (m, 6H), 6.72 (s, 1H), 6.06 (t, J = 5.2 Hz, 1H), 5.40 (d, J = 15.2 Hz, 1H), 5.03 (d, J = 15.2 Hz, 1H), 3.62 (d, J = 14.0 Hz, 1H), 3.43 (d, J = 14.0 Hz, 1H), 3.13 (dd, J = 17.6, 6.0 Hz, 1H), 2.90 (dd, J = 17.2, 4.0 Hz, 1H), 2.36 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): δ 172.6, 165.9, 139.9, 138.5, 131.8, 130.7, 129.1, 128.7, 128.5, 127.5, 125.9, 125.2, 69.1, 63.6, 41.0, 33.3, 21.1 ppm; HRMS (ESI) calculated for C$_{27}$H$_{27}$ClNO$_3$ [M + H]: 446.1517, found 446.1509.

N-(3-benzyl-2-oxo-6-(p-tolyl)-2,3,4,7-tetrahydrooxepin-3-yl)-3-bromobenamide (3ee): White solid, yield: 37.5 mg, 77%; M.P. = 190.0-190.3 °C; $^1$H NMR (400 MHz, CDCl$_3$):
δ 7.87 (s, 1H), 7.65 (d, J = 7.6 Hz, 1H), 7.61 (d, J = 7.6 Hz, 1H), 7.34-7.28 (m, 4H), 7.20-7.15 (m, 6H), 6.74 (s, 1H), 6.07 (t, J = 5.2 Hz, 1H), 5.39 (d, J = 15.2 Hz, 1H), 5.04 (d, J = 15.2 Hz, 1H), 3.62 (d, J = 14.4 Hz, 1H), 3.45 (d, J = 14.0 Hz, 1H), 3.14 (dd, J = 17.6, 6.0 Hz, 1H), 2.92 (dd, J = 17.6, 4.8 Hz, 1H), 2.36 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): δ 172.5, 165.5, 139.9, 138.1, 136.5, 135.5, 135.2, 135.1, 130.7, 130.4, 130.3, 129.4, 128.7, 127.5, 125.9, 125.2, 123.0, 69.1, 63.8, 41.0, 33.3, 21.1 ppm; HRMS (ESI) calculated for C$_{27}$H$_{27}$ClNO$_3$ [M + H]: 490.1012, found 490.1012.

N-(3-benzyl-6-(4-methoxyphenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-4-methylbenzamide (3fb):
White solid, yield: 31.0 mg, 70%; M.P. = 184.6-185.5 °C; $^1$H NMR (400 MHz, CDCl$_3$):
δ 7.63 (d, J = 7.6 Hz, 2H), 7.32-7.28 (m, 3H), 7.25-7.18 (m, 6H), 6.87 (d, J = 8.4 Hz, 2H), 6.63 (s, 1H), 5.96 (s, 1H), 5.42 (d, J = 15.2 Hz, 1H), 4.97 (d, J = 15.6 Hz, 1H), 3.81 (s, 3H), 3.67 (d, J = 14.0 Hz, 1H), 3.39 (d, J = 14.0 Hz, 1H), 3.06 (dd, J = 17.6, 5.2 Hz, 1H), 2.84 (d, J = 17.2 Hz, 1H), 2.41 (s, 3H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): δ 172.5, 166.9, 159.5, 142.8, 139.4, 135.5, 132.1, 130.9, 130.4, 129.5, 128.6, 127.3, 127.2, 127.1, 124.2, 114.1, 69.0, 62.8, 55.3, 41.2, 33.5, 21.5 ppm; HRMS (ESI) calculated for C$_{28}$H$_{29}$NO$_4$ [M + H]: 442.1313, found 442.2001.

N-(3-benzyl-6-(4-methoxyphenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-4-bromobenamide (3fc):
White solid, yield: 49.6 mg, 98%; M.P. = 139.0-139.4 °C; $^1$H NMR (400 MHz, CDCl$_3$): δ 7.57 (s, 4H), 7.32-7.28 (m, 3H), 7.23-7.17 (m, 4H), 6.87 (d, J = 8.4 Hz, 2H), 6.74 (s, 1H), 6.00 (t, J = 5.2 Hz, 1H), 5.39 (d, J = 15.2 Hz, 1H), 5.01 (d, J = 15.6 Hz, 1H), 3.81 (s, 3H), 3.60 (d, J = 14.0 Hz, 1H), 3.42 (d, J = 14.0 Hz, 1H), 3.13 (dd, J = 17.6, 5.6 Hz, 1H), 2.87 (dd, J = 17.2, 4.4 Hz, 1H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): δ 172.6, 166.0, 159.6, 139.5, 135.0, 132.2, 132.0, 131.8, 130.7, 128.7, 128.6, 127.5, 127.2, 127.0, 124.4, 114.1, 69.0, 63.6, 55.4, 41.1, 33.3 ppm; HRMS (ESI) calculated for C$_{28}$H$_{29}$BrNO$_4$ [M + H]: 506.0961, found 506.0965.

N-(3-benzyl-6-(4-methoxyphenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-4-chlorobenamide (3fd):
White solid, yield: 46.0 mg, 99%; M.P. = 157.2-157.7 °C; $^1$H NMR (400 MHz, CDCl$_3$): δ 7.65 (d, J = 8.4 Hz, 2H), 7.40 (d, J = 8.4 Hz, 2H), 7.34-7.28 (m, 3H), 7.23-7.17 (m, 4H), 6.87 (d, J = 8.8 Hz, 2H), 6.75 (s, 1H), 6.00 (t, J = 4.8 Hz, 1H), 5.39 (d, J = 15.2 Hz, 1H), 5.00 (d, J = 15.2 Hz, 1H), 3.81 (s,
N-(3-benzyl-6-(4-methoxyphenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-3-bromobenzamide (3fe):
White solid, yield: 49.0 mg, 97%; M.P. = 160.6-161.1 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.86 (s, 1H), 7.65 (d, J = 8.0 Hz, 1H), 7.60 (d, J = 7.6 Hz, 1H), 7.35-7.29 (m, 4H), 7.25-7.18 (m, 4H), 6.88 (d, J = 8.8 Hz, 2H), 6.72 (s, 1H), 6.02 (t, J = 5.2 Hz, 1H), 5.39 (d, J = 15.2 Hz, 1H), 5.03 (d, J = 15.2 Hz, 1H), 3.82 (s, 3H), 3.61 (d, J = 14.0 Hz, 1H), 3.46 (d, J = 14.0 Hz, 1H), 3.14 (dd, J = 17.2, 6.0 Hz, 1H), 2.90 (dd, J = 17.6, 4.4 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 172.6, 165.5, 159.6, 139.6, 135.5, 135.2, 135.1, 131.8, 130.6, 130.4, 130.3, 128.7, 127.5, 127.2, 125.5, 124.5, 123.0, 114.1, 69.1, 63.8, 55.3, 41.1, 33.3 ppm; HRMS (ESI) calculated for C₂₇H₂₃BrNO₃ [M + H]^⁺: 506.0961, found 506.0953.

N-(3-benzyl-6-(4-bromophenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-3-bromobenzamide (3be):
White solid, yield: 44.0 mg, 79%; M.P. = 200.5-201.4 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.86 (s, 1H), 7.66 (d, J = 6.4 Hz, 1H), 7.61 (d, J = 6.0 Hz, 1H), 7.47 (d, J = 6.8 Hz, 2H), 7.31 (s, 4H), 7.16 (d, J = 8.4 Hz, 4H), 6.73 (s, 1H), 6.11 (s, 1H), 5.40 (d, J = 14.8 Hz, 1H), 4.98 (d, J = 15.2 Hz, 1H), 3.57 (d, J = 13.2 Hz, 1H), 3.46 (d, J = 13.6 Hz, 1H), 3.18 (d, J = 14.0 Hz, 1H), 2.90 (d, J = 15.6 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 172.5, 165.6, 139.2, 138.2, 135.4, 135.1, 135.0, 131.9, 130.6, 130.3, 128.8, 127.6, 127.5, 127.1, 125.5, 123.0, 122.2, 68.6, 63.9, 41.4, 33.4 ppm; HRMS (ESI) calculated for C₂₉H₂₅BrNO₃ [M + H]^⁺: 553.9961, found 553.9961.

N-(3-benzyl-6-(4-chlorophenyl)-2-oxo-2,3,4,7-tetrahydrooxepin-3-yl)-3-bromobenzamide (3ce):
White solid, yield: 38.0 mg, 75%; M.P. = 206.2-206.5 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.86 (s, 1H), 7.66 (d, J = 7.6 Hz, 1H), 7.61 (d, J = 7.6 Hz, 1H), 7.33-7.28 (m, 6H), 7.23-7.18 (m, 4H), 6.73 (s, 1H), 6.10 (t, J = 4.8 Hz, 1H), 5.41 (d, J = 15.2 Hz, 1H), 4.99 (d, J = 15.2 Hz, 1H), 3.58 (d, J = 14.0 Hz, 1H), 3.46 (d, J = 14.0 Hz, 1H), 3.19 (dd, J = 17.2, 6.0 Hz, 1H), 2.91 (dd, J = 16.8, 4.4 Hz, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 172.5, 165.6, 139.2, 137.7, 135.4, 135.1, 135.0, 134.1, 130.6, 130.3, 128.9, 128.8, 127.6, 127.2, 127.0, 125.5, 123.0, 68.7, 64.0, 41.4, 33.3 ppm; HRMS (ESI) calculated for C₂₉H₂₅BrClNO₃ [M + H]^⁺: 510.0466, found 510.0458.
7. $^1$H and $^{13}$C NMR spectra
3de

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S26
8. Copy of HPLC Spectra of Compound 3aa

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9. X-Ray Crystal Data of Compound 3ad

Figure S1 X-ray single crystal structure of 3ad (with thermal ellipsoids shown at the 50% probability level)

Identification code 3ad

Empirical formula C_{26}H_{22}ClNO_3

Formula weight 431.89

Temperature 133.15 K

Crystal system, space group Triclinic, P-1

Unit cell dimensions

\begin{align*}
    a &= 9.588(4) \text{ Å} \\
    \alpha &= 88.60(2) \text{ deg.} \\
    b &= 14.091(7) \text{ Å} \\
    \beta &= 78.027(16) \text{ deg.} \\
    c &= 15.915(7) \text{ Å} \\
    \gamma &= 88.20(3) \text{ deg.}
\end{align*}

Volume 2102.0(16) Å^3

Z, Calculated density 4, 1.365 g/cm^3

Absorption coefficient 0.211 mm^-1

F(000) 904.0

Crystal size 0.2 × 0.18 × 0.14 mm^3

Radiation MoKα (λ = 0.71073)

Theta range for data collection 6.028 to 50.038 deg.

Index ranges -11 ≤ h ≤ 11, -16 ≤ k ≤ 16, -18 ≤ l ≤ 18
Reflections collected / unique independent reflections 20434 / 7111 [R_int = 0.0834, R_sigma = 0.0770]

Data / restraints / parameters 7111 / 2 / 567

Goodness-of-fit on F^2 1.112

Final R indices [I>2sigma(I)] R_1 = 0.0868, wR_2 = 0.2250

R indices (all data) R_1 = 0.1070, wR_2 = 0.2348

Largest diff. peak and hole 0.70/-0.33 eÅ^-3

10. References
