Supporting Information

Gold-Catalyzed One-Step Practical Synthesis of Oxetan-3-ones from Readily Available Propargylic Alcohols

Longwu Ye, Weimin He and Liming Zhang

Department of Chemistry and Biochemistry,
University of California, Santa Barbara, California, 93106

<table>
<thead>
<tr>
<th>Content</th>
<th>Page number</th>
</tr>
</thead>
<tbody>
<tr>
<td>General</td>
<td>2</td>
</tr>
<tr>
<td>Preparation of secondary propargyl Alcohols 1 or 5</td>
<td>2</td>
</tr>
<tr>
<td>Preparation of tertiary propargylic alcohols 7</td>
<td>7</td>
</tr>
<tr>
<td>Gold catalysis using 1 or 5 as substrates</td>
<td>11</td>
</tr>
<tr>
<td>Gold catalysis using 7 as substrates</td>
<td>16</td>
</tr>
<tr>
<td>$^1$H and $^{13}$C NMR spectra</td>
<td>23</td>
</tr>
</tbody>
</table>
General. Ethyl acetate (ACS grade), hexanes (ACS grade) and diethyl ether (ACS grade) were purchased from Fisher Scientific and used without further purification. Anhydrous 1, 2-dichloroethane (HPLC grade) was purified by distillation over calcium hydride. Anhydrous tetrahydrofuran was distilled from sodium benzophenone ketyl. Commercially available reagents were used without further purification. Reactions were monitored by thin layer chromatography (TLC) using silicycle pre-coated silica gel plates. Flash column chromatography was performed over silicycle silica gel (230-400 mesh).

$^1$H NMR and $^{13}$C NMR spectra were recorded on a Varian 500 MHz UNITY INOVA spectrometer and a Varian 400 MHz UNITY INOVA spectrometer using residue solvent peaks as internal standards. Infrared spectra were recorded with a Perkin Elmer FT-IR spectrum 2000 spectrometer and are reported in reciprocal centimeter (cm$^{-1}$). Mass spectra were recorded with Micromass QTOF2 Quadrupole/Time-of-Flight Tandem mass spectrometer using electron spray ionization.

General procedure A: preparation of secondary propargyl alcohols 1 and 5

A solution of $n$-butyllithium in hexanes (1.6 M, 5.3 mL, 8.5 mmol) was added dropwise to a solution of (trimethylsilyl)acetylene (8.0 mmol) in anhydrous THF (20 mL) at -78°C. After 15 min, a solution of an aldehyde (5.3 mmol) in anhydrous THF (20 mL) was added dropwise to the reaction mixture, and the resulting mixture was further stirred at the same temperature for 3 h. The reaction was quenched with saturated NH$_4$Cl solution, and the mixture was extracted four times with Et$_2$O. The combined organic layers were washed with brine, dried with anhydrous MgSO$_4$, and the solvents were evaporated under vacuum. The oily residue was purified by flash silica gel column chromatography (hexanes/EtOAc) to yield propargylic alcohol A.

A solution of tetrabutylammonium fluoride (1.0 M, 4.8 mL, 4.8 mmol) was added dropwise to a solution of propargylic alcohol A (4.0 mmol) in THF (35 mL) at 0°C and
the mixture was stirred at 0 °C for 15 min. After addition of an aqueous NH₄Cl solution, the mixture was extracted three times with Et₂O. The combined organic layers were washed with brine, dried with anhydrous MgSO₄, and the solvents was evaporated under vacuum. The oily residue was purified by flash silica gel column chromatography (hexanes/EtOAc ) to yield propargylic alcohols 1 or 5.

**Dec-1-yn-3-ol (1)**

![Dec-1-yn-3-ol](image)

Alcohol 1 was prepared in 77% yield (2 steps) by following General Procedure A. This compound is known and the spectroscopic data match those reported.¹ ¹H NMR (400 MHz, CDCl₃) δ 4.40 – 4.35 (m, 1H), 2.46 (d, 1H, J = 2.4 Hz), 1.78 (d, 1H, J = 6.0 Hz), 1.75 – 1.67 (m, 2H), 1.49 – 1.41 (m, 2H), 1.30 – 1.25 (m, 8H), 0.88 (t, 3H, J = 6.8 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 85.0, 72.8, 62.4, 37.7, 31.8, 29.2, 29.1, 25.0, 22.6, 14.1.

**1-Cyclohexylprop-2-yn-1-ol (5a)**

![1-Cyclohexylprop-2-yn-1-ol](image)

Alcohol 5a was prepared in 73% yield (2 steps) by following General Procedure A. This compound is known and the spectroscopic data match those reported.¹ ¹H NMR (400 MHz, CDCl₃) δ 4.19 – 4.14 (m, 1H), 2.46 (d, 1H, J = 2.0 Hz), 1.88 – 1.75 (m, 5H), 1.70 – 1.64 (m, 1H), 1.61 – 1.51 (m, 1H), 1.31 – 1.02 (m, 5H); ¹³C NMR (125 MHz, CDCl₃) δ 83.9, 73.6, 67.0, 43.9, 28.4, 27.9, 26.3, 25.8, 25.7.

**5-Phenylpent-1-yn-3-ol (5b)**
Alcohol $5b$ was prepared in 69% yield (2 steps) by following General Procedure A. This compound is known and the spectroscopic data match those reported. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.32 – 7.29 (m, 2H), 7.23 – 7.19 (m, 3H), 4.40 – 4.36 (m, 1H), 2.82 (t, 2H, $J = 8.0$ Hz), 2.52 (d, 1H, $J = 2.4$ Hz), 2.10 – 1.98 (m, 2H), 1.88 (d, 1H, $J = 5.2$ Hz); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 141.1, 128.5, 128.4, 126.0, 84.6, 73.3, 61.5, 39.0, 31.2.

**Oct-7-en-1-yn-3-ol (5c)**

Alcohol $5c$ was prepared in 63% yield (2 steps) by following General Procedure A. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 5.84 – 5.76 (m, 1H), 5.05 – 4.95 (m, 2H), 4.40 – 4.36 (m, 1H), 2.47 (d, 1H, $J = 2.0$ Hz), 2.12 – 2.07 (m, 2H), 1.93 – 1.90(bs, 1H), 1.78 – 1.69 (m, 2H), 1.60 – 1.53 (m, 2H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 138.3, 114.9, 84.8, 73.0, 62.1, 37.0, 33.2, 24.2; IR (neat): 3432(bs), 3302, 2935, 2862, 2114, 1643, 1439, 910; MS (ES$^+$) Calculated for [C$_8$H$_{13}$O]$^+$: 125.1; Found: 125.1.

**1-Phenylprop-2-yn-1-ol (5d)**

Alcohol $5d$ was prepared in 73% yield (2 steps) by following General Procedure A. This compound is known and the spectroscopic data match those reported. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.56 (d, 2H, $J = 6.0$ Hz), 7.42 – 7.33 (m, 3H), 5.48 (dd, 1H, $J = 5.2$ Hz, $J$
\( = 1.6 \text{ Hz}, 2.68 \text{ (d, 1H, } J = 1.6 \text{ Hz)}, 2.37 \text{ (d, 1H, } J = 5.2 \text{ Hz); }^{13}\text{C NMR (125 MHz, CDCl}_3\text{)} \delta 140.0, 128.7, 128.6, 126.6, 83.5, 74.9, 64.4.\)

6-Methoxymethoxyhex-1-yn-3-ol (5e)

Alcohol 5e was prepared in 70% yield (2 steps) by following General Procedure A. \(^1\text{H NMR (400 MHz, CDCl}_3\text{)} \delta 4.63 \text{ (s, 2H)}, 4.46 – 4.42 \text{ (m, 1H)}, 3.63 – 3.54 \text{ (m, 2H)}, 3.37 \text{ (s, 3H)}, 2.56 \text{ (d, 1H, } J = 6.0 \text{ Hz)}, 2.47 \text{ (d, 1H, } J = 2.4 \text{ Hz)}, 1.88 – 1.75 \text{ (m, 4H); }^{13}\text{C NMR (125 MHz, CDCl}_3\text{)} \delta 96.1, 84.8, 72.7, 67.2, 61.6, 55.1, 34.5, 25.2; \text{ IR (neat): 3429} (bs), 3290, 2939, 2114, 1732, 1446, 1041; \text{ Calculated for [C}_8\text{H}_{14}\text{NaO}_3\text{]+: 181.1; Found: 181.1.}\)

7-Bromohept-1-yn-3-ol (5h)

A solution of ethynylmagnesium bromide (24 mL) in THF (0.5 M, 12.0 mmol) was added dropwise over 5 min to a stirred solution of 5-bromopentanal (8.0 mmol) in anhydrous THF (20 mL) at 0 ºC under N\(_2\). The mixture was allowed to warm to room temperature and stirred for 30 min. Ether (80 mL) and saturated aqueous ammonium chloride solution (80 mL) were added, and the organic layer was then separated. The aqueous layer was extracted with ether (2 x 80 mL). The combined organic layers were then washed with brine (100 mL), dried and concentrated \textit{in vacuo} to afford alcohol 5h (90%) as a pale yellow oil. \(^1\text{H NMR (400 MHz, CDCl}_3\text{)} \delta 4.38 \text{ (dt, 1H, } J = 6.4 \text{ Hz, } J = 2.0 \text{ Hz)}, 3.41 \text{ (t, 2H, } J = 6.8 \text{ Hz)}, 2.48 \text{ (d, 1H, } J = 2.4 \text{ Hz)}, 2.06 \text{ (bs, 1H)}, 1.94 – 1.86 \text{ (m, 2H), 1.77 – 1.71} \text{ (m, 2H)}, 1.65 – 1.58 \text{ (m, 2H); }^{13}\text{C NMR (125 MHz, CDCl}_3\text{)} \delta 84.5, 73.2,
61.9, 36.5, 33.4, 32.3, 23.7; IR (neat): 3385(bs), 3294, 2939, 2866, 2114, 1624, 1442, 1254, 1053, 1011, 652; MS (ES+) Calculated for [C_{7}H_{11}BrNaO]^+: 213.0; Found: 213.0.

7-Azidohept-1-yn-3-ol (5g)

To a solution of 5h (1.15 g, 6.0 mmol) in DMF (20 mL) was added NaN₃ (1.18 g, 18 mmol) and NaI (0.60 g, 3.0 mmol) at room temperature. The resulting mixture was heated at 80 °C for 12 h and cooled to room temperature before the addition of water (20 mL) and diethyl ether (30 mL). The two layers were separated, and the aqueous layer was extracted with Et₂O (3 × 50 mL). The combined organic layers were washed with water (2 × 20 mL), brine (20 mL), dried with MgSO₄, and concentrated. The crude product was purified with flash silica gel column chromatography (eluents: hexanes : ethyl acetate = 5:1) to get 5g (0.55 g) in 60% yield. ¹H NMR (400 MHz, CDCl₃) δ 4.40 (dt, 1H, J = 6.4 Hz, J = 2.0 Hz), 3.30 (t, 2H, J = 6.8 Hz), 2.48 (d, 1H, J = 2.0 Hz), 1.83 – 1.52 (m, 7H); ¹³C NMR (125 MHz, CDCl₃) δ 84.6, 73.1, 61.8, 51.2, 36.9, 28.4, 22.2; IR (neat): 3386(bs), 3294, 2943, 2866, 2102, 1458, 1265, 1022, 660; MS (ES+) Calculated for [C₇H₁₁N₃NaO]^+: 176.1; Found: 176.1.

tert-Butyl (5-hydroxyhept-6-ynyl)carbamate (5f)

To a solution of 5g (0.400 g, 2.6 mmol) in Et₂O (30 mL) was added PPh₃ (0.818 g, 3.1 mmol) and H₂O (1.9 mL) at room temperature. The resulting mixture was stirred for 12 h. The reaction mixture was then treated with (Boc)₂O (4.19 g, 19.2 mmol) and stirred overnight. Water was then added, and the resulting two layers were separated. The
aqueous layer was extracted with Et₂O (3 × 30 mL), and the combined organic layers were washed with brine (30 mL), dried with MgSO₄, and concentrated. The crude product was purified with flash silica gel column chromatography (eluents: hexanes : ethyl acetate = 3:1) to give 5f (0.396 g) in 67% yield. ¹H NMR (500 MHz, CDCl₃) δ 4.66 (bs, 1H), 4.34 (dt, 1H, J = 6.5 Hz, J = 2.0 Hz), 3.10 (s, 2H), 2.75 (bs, 1H), 2.43 (d, 1H, J = 2.5 Hz), 1.73 – 1.68 (m, 2H), 1.51 – 1.44 (m, 4H), 1.41 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 156.1, 84.9, 79.1, 72.7, 61.8, 40.2, 37.0, 29.6, 28.3, 22.1; IR (neat): 3352(bs), 3305(bs), 2939, 2870, 2110, 1693, 1527, 1169; MS (ES⁺) Calculated for [C₁₂H₂₁NNaO₃]⁺: 250.1; Found: 250.1.

General procedure B: preparation of tertiary propargylic alcohols 7

A solution of n-butyllithium in hexanes (1.6 M, 33.0 mmol) was added dropwise to a solution of freshly distilled diisopropylamine (33.0 mmol) in dried THF (30 mL) at 0 °C. The solution was stirred for 1 h at 0 °C, then cooled to -78 °C. Ethyl propiolate (31.3 mmol) in dried THF (10 mL) was then added dropwise to the reaction mixture. After 1 h at the same temperature, ketone (62.6 mmol) was added, and the resulting mixture was stirred at -78 °C for 3 h. The reaction was quenched with saturated NH₄Cl solution, and the mixture was extracted four times with Et₂O. The combined organic layers were washed with brine, dried with anhydrous MgSO₄, and the solvents evaporated to dryness. The oily residue was purified by flash silica gel column chromatography (hexanes/EtOAc) to yield propargylic alcohol 7.

4-Hydroxy-4-methylpent-2-ynoic acid ethyl ester (7a)
Alcohol 7a was prepared in 90% yield (2 steps) by following General Procedure B. This compound is known and the spectroscopic data match those reported.\(^2\) \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 4.22 (q, 2H, \(J = 7.0\) Hz), 2.55 (s, 1H), 1.55 (s, 6H), 1.30 (t, 3H, \(J = 7.3\) Hz); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 153.6, 91.0, 74.0, 64.9, 62.1, 30.5, 13.9; IR (neat): 3413(bs), 2985, 2237, 1705, 1612, 1450, 1373, 1257, 1026, 949; MS (ES\(^+\)) Calculated for \([\text{C}_8\text{H}_{12}\text{NaO}_3]^+\): 179.1; Found: 179.1.

4-Hydroxy-4-methylhept-2-ynoic acid ethyl ester (7b)

\[
\begin{align*}
\text{OH} & \quad \text{CO}_2\text{Et} \\
7b
\end{align*}
\]

Alcohol 7b was prepared in 87% yield (2 steps) by following General Procedure B. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 4.21 (q, 2H, \(J = 7.0\) Hz), 2.32 (bs, 1H), 1.72 – 1.62 (m, 2H), 1.57 – 1.44 (m, 5H), 1.29 (t, 3H, \(J = 7.0\) Hz), 0.94 (t, 3H, \(J = 7.0\) Hz); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 153.6, 90.4, 75.1, 67.9, 62.0, 45.0, 28.9, 17.7, 14.0, 13.9; IR (neat): 3421(bs), 2966, 2885, 2233, 1709, 1462, 1369, 1250, 1030, 752; MS (ES\(^+\)) Calculated for \([\text{C}_{10}\text{H}_{16}\text{NaO}_3]^+\): 207.1; Found: 207.1.

4-Hydroxy-4,5-dimethylhex-2-ynoic acid ethyl ester (7c)

\[
\begin{align*}
\text{OH} & \quad \text{CO}_2\text{Et} \\
7c
\end{align*}
\]

Alcohol 7c was prepared in 88% yield (2 steps) by following General Procedure B. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 4.21 (q, 2H, \(J = 7.2\) Hz), 2.23 (bs, 1H), 1.86 (heptet, 1H, \(J = 7.2\) Hz), 1.48 (s, 3H), 1.30 (t, 3H, \(J = 7.2\) Hz), 1.04 (d, 3H, \(J = 7.2\) Hz), 1.02 (d, 3H, \(J = 6.8\) Hz); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 153.6, 89.9, 75.9, 71.5, 62.1, 38.6, 26.3, 17.6,
17.2, 14.0; IR (neat): 3406(bs), 2935, 2862, 2233, 1712, 1458, 1250, 1026; MS (ES+) 
Calculated for [C_{10}H_{16}NaO_{3}]^{+}: 207.1; Found: 207.1.

(1-Hydroxycyclopentyl)propynoic acid ethyl ester (7d)

![Chemical structure of 7d]

Alcohol 7d was prepared in 91% yield (2 steps) by following General Procedure B. This compound is known and the spectroscopic data match those reported.\(^2\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 4.23 (q, 2H, \(J = 7.2\) Hz), 2.06 – 1.94 (m, 5H), 1.90 – 1.74 (m, 4H), 1.31 (t, 3H, \(J = 7.2\) Hz); \(^1\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 153.6, 90.3, 75.2, 74.0, 62.1, 42.1, 23.5, 14.0.

(1-Hydroxycyclohexyl)propynoic acid ethyl ester (7e)

![Chemical structure of 7e]

Alcohol 7e was prepared in 90% yield (2 steps) by following General Procedure B. This compound is known and the spectroscopic data match those reported.\(^2\) \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 4.23 (q, 2H, \(J = 7.2\) Hz), 2.40 (bs, 1H), 2.00 – 1.94 (m, 2H), 1.74 – 1.51 (m, 8H), 1.31 (t, 3H, \(J = 7.2\) Hz); \(^1\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 153.6, 90.3, 76.0, 68.4, 62.1, 39.0, 24.9, 22.8, 14.0.

(1-Hydroxycycloheptyl)propynoic acid ethyl ester (7f)
Alcohol 7f was prepared in 82% yield (2 steps) by following General Procedure B. \( ^1 \text{H} \) NMR (400 MHz, CDCl\(_3\)) \( \delta \) 4.23 (q, 2H, \( J = 7.2 \text{ Hz} \)), 2.10 – 2.03 (m, 3H), 1.92 – 1.86 (m, 2H), 1.69 – 1.55 (m, 8H), 1.31 (t, 3H, \( J = 7.2 \text{ Hz} \)); \( ^{13} \text{C} \) NMR (125 MHz, CDCl\(_3\)) \( \delta \) 153.7, 91.6, 75.1, 71.5, 62.1, 42.2, 28.1, 21.9, 14.0; IR (neat): 3406(bs), 2935, 2862, 2233, 1712, 1458, 1250, 1026; Calculated for [C\(_{12}\)H\(_{18}\)NaO\(_3\)]\(^+\): 233.1; Found: 233.1.

\((2\text{-Hydroxyadamantan-2-yl})\text{propynoic acid ethyl ester (7g)}\)

Alcohol 7g was prepared in 85% yield (2 steps) by following General Procedure B. \( ^1 \text{H} \) NMR (500 MHz, CDCl\(_3\)) \( \delta \) 4.23 (q, 2H, \( J = 7.0 \text{ Hz} \)), 2.25 (s, 1H), 2.16 (d, 2H, \( J = 13.0 \text{ Hz} \)), 2.09 (d, 2H, \( J = 13.0 \text{ Hz} \)), 2.02 (bs, 2H), 1.85 – 1.74 (m, 4H), 1.70 (s, 2H), 1.59 – 1.55 (m, 2H), 1.30 (t, 3H, \( J = 7.2 \text{ Hz} \)); \( ^{13} \text{C} \) NMR (125 MHz, CDCl\(_3\)) \( \delta \) 153.7, 91.0, 72.5, 62.0, 38.1, 37.3, 35.2, 31.2, 26.6, 26.5, 14.0; IR (neat): 3429(bs), 2920, 2854, 2225, 1701, 1250, 1022; MS (ES\(^+\)) Calculated for [C\(_{15}\)H\(_{20}\)NaO\(_3\)]\(^+\): 271.1; Found: 271.2.

\((4\text{-Benzyloxymethoxy-1-hydroxycyclohexyl})\text{propynoic acid ethyl ester (7h)}\)

Alcohol 7h (dr: 10/1) was prepared in 78% yield (2 steps) by following General Procedure B. \( ^1 \text{H} \) NMR (500 MHz, CDCl\(_3\)) \( \delta \) 7.39 – 7.27 (m, 5H), 4.80 (s, 2H), 4.62 (s,
2H), 4.23 (q, 2H, \( J = 7.2 \) Hz), 3.76 (bs, 1H), 2.33 (bs, 1H), 2.03 – 1.95 (m, 2H), 1.87 – 1.76 (m, 6H), 1.31 (t, 3H, \( J = 7.2 \) Hz); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \( \delta \) 153.5, 137.8, 128.4, 127.8, 127.7, 92.7, 89.8, 75.6, 71.4, 69.5, 67.3, 62.1, 35.0, 27.6, 14.0; IR (neat): 3417(bs), 2943, 2889, 2229, 1709, 1454, 1369, 1250, 1041, 744; MS (ES\(^+\)) Calculated for [C\(_{19}\)H\(_{24}\)NaO\(_5\)]\(^+\): 355.2; Found: 355.2.

Procedure for the preparation of chiral 5-phenyl-pent-1-yn-3-ol

This chiral alcohol was prepared in 36% overall yield by reacting (trimethylsilyl)acetylene with 3-phenyl-propionaldehyde (according to Carreira’s method\(^3\)) and subsequently removing TMS group. This compound is known and the spectroscopic data match those reported.\(^4\) Its enantiomeric access (80%) was determined by chiral HPLC [Chiralpak IA Column, 2/98 iPrOH /hexane, 0.7 mL/min, 254 nm; TR = 27.83 min (major), 26.62 min (minor)]. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.32 – 7.19 (m, 5H), 4.41 – 4.36 (m, 1H), 2.82 (t, 2H, \( J = 8.0 \) Hz), 2.52 (d, 1H, \( J = 2.0 \) Hz), 2.10 – 2.02 (m, 2H), 1.99 (d, 1H, \( J = 5.6 \) Hz).

General procedure C: 3-Methoxycarbonyl-5-bromopyridine N-oxide (4, 0.60 mmol), Tf\(_2\)NH (1.8 mL, 0.20 M in DCE), and (2-biphenyl)Cy\(_2\)PAuNTf\(_2\) (12.5 mg, 0.015 mmol) were added sequentially to a solution of secondary propargyl alcohol 1 or 5 (0.30 mmol) in DCE (4.2 mL) at room temperature. The reaction mixture was stirred at rt and the progress of the reaction was monitored by TLC. The reaction typically took 3 – 4 h. Upon completion, the reaction was treated with saturated aqueous NaHCO\(_3\) (15 mL), and the resulting solution was extracted with DCM (2 × 30 mL). The combined organic layers
were dried with MgSO₄. The mixture was concentrated and the residue was purified by silica gel flash chromatography (eluent: hexanes/ethyl acetate) to afford desired products 2 or 6.

**2-Heptyloxetan-3-one (2)**

![2-Heptyloxetan-3-one (2)](image)

Compound 2 was prepared in 71% yield using 4 as oxidant according to General Procedure C. The reaction time was 3 h. ¹H NMR (500 MHz, CDCl₃) δ 5.56 – 5.45 (m, 1H), 5.28 (d, 1H, J = 15.0 Hz), 5.22 (dd, 1H, J = 4.5, 15.0 Hz), 1.88 – 1.77 (m, 2H), 1.52 – 1.40 (m, 2H), 1.39 – 1.22 (m, 8H), 0.88 (t, 3H, J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 203.6, 104.0, 88.6, 31.7, 31.3, 29.2, 29.0, 24.1, 22.6, 14.0; IR (neat): 2931, 2862, 1824, 1462, 1261, 1034, 960, 802; MS (ES⁺) Calculated for [C₁₀H₁₈NaO₂]⁺: 193.1; Found: 193.1.

**Methanesulfonic acid 3-hydroxy-2-oxodecyl ester 3**

![Methanesulfonic acid 3-hydroxy-2-oxodecyl ester 3](image)

Compound 3 was isolated in 9.2% yield (4 as oxidant, 1.2 equiv MsOH as the acid and PPh₃AuNTf₂ as the gold catalyst) according to General Procedure C (entry 7, table 1). The reaction time was 6 h. ¹H NMR (500 MHz, CDCl₃) δ 5.05 (dd, 2H, J = 36.0 Hz, J = 17.0 Hz), 4.33 – 4.30 (m, 1H), 3.20 (s, 3H), 2.84 (d, 1H, J = 5.0 Hz), 1.83 – 1.76 (m, 1H), 1.65 – 1.56 (m, 1H), 1.47 – 1.27 (m, 10H), 0.88 (t, 3H, J = 7.2 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 204.9, 75.3, 69.8, 38.9, 33.8, 31.7, 29.2, 29.0, 24.6, 22.6, 14.0; IR (neat): 3464(bs), 2924, 2854, 2256, 1732, 1335, 1173, 1026, 733; MS (ES⁺) Calculated for [C₁₁H₂₂NaO₅S]⁺: 289.1; Found: 289.1.
2-Cyclohexyloxetan-3-one (6a)

![Cyclohexyloxetan-3-one (6a)](image)

Compound 6a was prepared in 73% yield using 4 as oxidant according to General Procedure C. The reaction time was 3 h. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 5.24 – 5.21 (m, 2H), 5.14 (dd, 1H, $J$ = 15.0 Hz, $J$ = 4.5 Hz), 1.87 – 1.66 (m, 6H), 1.29 – 1.07 (m, 5H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 203.5, 107.8, 88.7, 40.1, 27.5, 27.4, 26.1, 25.5, 25.4; IR (neat): 2931, 2854, 1824, 1446, 1045, 960; MS (ES$^+$) Calculated for [C$_9$H$_{14}$KO$_2$]$^+$: 193.1; Found: 193.1.

2-Phenethyloxetan-3-one (6b)

![Phenethyloxetan-3-one (6b)](image)

Compound 6b was prepared in 80% yield using 4 as oxidant according to General Procedure C. The reaction time was 4 h. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.32 – 7.29 (m, 2H), 7.23 – 7.20 (m, 3H), 5.48 – 5.44 (m, 1H), 5.31 (d, 1H, $J$ = 15.0 Hz), 5.26 (dd, 1H, $J$ = 3.5, 15.0 Hz), 2.85 – 2.75 (m, 2H), 2.23 – 2.10 (m, 2H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 203.1, 140.3, 128.5, 128.4, 126.3, 102.8, 88.8, 32.8, 30.1; IR (neat): 2924, 2866, 1817, 1454, 1068, 957, 698; Calculated for [C$_{11}$H$_{12}$NaO$_2$]$^+$: 199.1; Found: 199.1.

2-Pent-4-enyl-oxetan-3-one (6c)

![Pent-4-enyl-oxetan-3-one (6c)](image)
Compound 6c was prepared in 65% yield using 4 as oxidant according to General Procedure C. The reaction time was 3 h. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 5.82 – 5.74 (m, 1H), 5.47 (dd, 1H, $J$ = 10.5 Hz, $J$ = 6.5 Hz), 5.28 (d, 1H, $J$ = 15.0 Hz), 5.22 (dd, 1H, $J$ = 4.0, 15.0 Hz), 5.04 – 4.97 (m, 2H), 2.13 – 2.08 (m, 2H), 1.88 – 1.81 (m, 2H), 1.63 – 1.50 (m, 2H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 203.3, 137.8, 115.2, 103.7, 88.7, 33.2, 30.6, 23.3; IR (neat): 2931, 2862, 1817, 1439, 1049, 957; MS (ES$^+$) Calculated for [C$_8$H$_{12}$KO$_2$]$^+$: 179.1; Found: 179.1.

2-Phenyloxetan-3-one (6d)

![6d]

Compound 6d was prepared in 57% yield using 2-bromopyridine N-oxide as oxidant and MsOH (1.2 equiv) as additive according to General Procedure C. The reaction time was 4 h. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.45 – 7.36 (m, 5H), 6.39 (d, 1H, $J$ = 4.4 Hz), 5.53 (d, 1H, $J$ = 14.8 Hz), 5.47 (dd, 1H, $J$ = 4.4, 14.8 Hz); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 199.3, 130.4, 128.9, 128.7, 125.2, 104.3, 90.1; IR (neat): 2931, 2854, 2364, 1828, 1732, 1643, 1404, 957, 698; MS (ES$^+$) Calculated for [C$_9$H$_9$O$_2$]$^+$: 149.1; Found: 149.0.

2-(3-Methoxymethoxypropyl)oxetan-3-one (6e)

![6e]

Compound 6e was prepared in 65% yield using 4 as oxidant according to General Procedure C. The reaction time was 3 h. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.50 (dd, 1H, $J$ = 10.8 Hz, $J$ = 6.4 Hz), 5.29 (d, 1H, $J$ = 15.2 Hz), 5.24 (dd, 1H, $J$ = 4.0, 15.2 Hz), 4.61 (s, 2H), 3.56 (t, 2H, $J$ = 6.4 Hz), 3.35 (s, 3H), 1.96 (dd, 2H, $J$ = 14.4 Hz, $J$ = 6.8 Hz), 1.82 – 1.71 (m, 2H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 203.0, 103.5, 96.4, 88.8, 66.9, 55.2, 28.2,
24.4; IR (neat): 2935, 1824, 1439, 1041, 957; Calculated for [C₈H₁₅O₄]⁺: 175.1; Found: 175.1.

[4-(3-Oxooxetan-2-yl)butyl]carbamic acid tert-butyl ester (6f)

![Structural formula of 6f]

Compound 6f was prepared in 60% yield using 4 as oxidant at − 20 ºC according to General Procedure C. The reaction time was 16 h. ¹H NMR (400 MHz, CDCl₃) δ 5.48 – 5.43 (m, 1H), 5.28 (d, 1H, J = 14.8 Hz), 5.24 (dd, 1H, J = 4.0, 14.8 Hz), 4.54 (bs, 1H), 3.17 – 3.10 (m, 2H), 1.88 – 1.82 (m, 2H), 1.55 – 1.46 (m, 4H), 1.43 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 203.1, 155.9, 103.6, 88.8, 79.1, 40.2, 30.8, 29.8, 28.4, 21.4; IR (neat): 3356(bs), 2978, 2935, 2866, 1817, 1701, 1527, 1250, 1173, 957; MS (ES⁺) Calculated for [C₁₂H₂₁NNaO₄]⁺: 266.1; Found: 266.2.

2-(4-Azidobutyl)oxetan-3-one (6g)

![Structural formula of 6g]

Compound 6g was prepared in 81% yield using 4 as oxidant according to General Procedure C. The reaction time was 3 h. ¹H NMR (400 MHz, CDCl₃) δ 5.50 – 5.44 (m, 1H), 5.30 (d, 1H, J = 15.2 Hz), 5.22 (dd, 1H, J = 4.4, 15.2 Hz), 3.29 (t, 2H, J = 6.4 Hz), 1.90 – 1.83 (m, 2H), 1.68 – 1.49 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 202.9, 103.4, 88.9, 51.1, 30.7, 28.5, 21.5; IR (neat): 2939, 2870, 2102, 1817, 1261, 960; MS (ES⁺) Calculated for [C₇H₁₁N₃NaO₂]⁺: 192.1; Found: 192.1.

2-(4-Bromo-butyl)-oxetan-3-one (6h)
Compound **6h** was prepared in 70% yield using 4 as oxidant according to General Procedure C. The reaction time was 3 h. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.49 – 5.45 (m, 1H), 5.30 (d, 1H, $J = 14.8$ Hz), 5.23 (dd, 1H, $J = 4.4, 14.8$ Hz), 3.41 (t, 2H, $J = 6.4$ Hz), 1.95 – 1.82 (m, 4H), 1.71 – 1.56 (m, 2H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 202.9, 103.4, 88.9, 33.0, 32.3, 30.3, 22.9; IR (neat): 2935, 2870, 1817, 1439, 1273, 1030, 957; MS (ES$^+$) Calculated for [C$_7$H$_{11}$BrNaO$_2$]$^+$: 229.0; Found: 229.0.

**(2S)-2-phenethyloxetan-3-one**

[Diagram of the compound]

This chiral compound was prepared in 83% yield using 4 as oxidant according to General Procedure C. The reaction time was 4 h. $\left[\alpha\right]_{D}^{20} = -39.5^\circ$ (c = 0.50, CHCl$_3$). 81% ee [determined by HPLC: Chiralpak IA Column, 1/99 iPrOH/hexane, 0.6 mL/min, 254 nm; TR = 15.09 min (major), 16.59 min (minor)]. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.32 – 7.19 (m, 5H), 5.49 – 5.45 (m, 1H), 5.48 – 5.44 (m, 1H), 5.31 (d, 1H, $J = 15.0$ Hz), 2.84 – 2.76 (m, 2H), 2.24 – 2.10 (m, 2H).

**General Procedure D:** 4-Acetylpyridine $N$-oxide (0.60 mmol), Tf$_2$NH (1.8 mL, 0.20 M in DCE), and IPrAuNTf$_2$ (13.9 mg, 0.015 mmol) were added sequentially to a solution of tertiary propargyl alcohol 7 (0.30 mmol) in DCE (1.2 mL) at room temperature. The
reaction mixture was stirred at 40 °C or 60 °C, and the progress of the reaction was monitored by TLC. The reaction typically took 20 – 24 h. Upon completion, the reaction was treated with saturated aqueous NaHCO₃ (15 mL), and the resulting solution was extracted with DCM (2 × 30 mL). The combined organic layers were dried with MgSO₄. The mixture was concentrated and the residue was purified by silica gel flash chromatography (eluent: hexanes/ethyl acetate) to afford desired products 8.

4,4-Dimethyl-3-oxooxetane-2-carboxylic acid ethyl ester (8a)

[Chemical structure image]

Compound 8a was prepared in 82% yield according to General Procedure D. The reaction time was 20 h. ¹H NMR (400 MHz, CDCl₃) δ 5.73 (s, 1H), 4.36 – 4.23 (m, 2H), 1.59 (s, 3H), 1.56 (s, 3H), 1.32 (t, 3H, J = 7.2 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 197.9, 165.1, 108.6, 93.6, 62.2, 22.9, 22.5, 14.1; IR (neat): 2985, 2931, 1836, 1747, 1466, 1369, 1215, 1030; MS (ES⁺) Calculated for [C₈H₁₂NaO₄]⁺: 195.1; Found: 195.1.

4-Methyl-3-oxo-4-propyl-oxetane-2-carboxylic acid ethyl ester (8b)

[Chemical structure image]

Compound 8b (dr: 1.8/1) was prepared in 78% yield according to General Procedure D. The reaction time was 24 h. ¹H NMR (500 MHz, CDCl₃) δ 5.70 (s, 0.4H), 5.63 (s, 0.6H), 4.33 – 4.23 (m, 2H), 1.84 – 1.76 (m, 2H), 1.63 – 1.53 (m, 4H), 1.44 – 1.30 (m, 4H), 0.97 – 0.92 (m, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 198.4, 198.2, 165.1, 164.6, 111.7, 111.0, 93.9, 93.2, 62.2, 62.1, 38.8, 38.2, 21.1, 21.0, 17.0, 16.8, 14.2, 14.1; IR (neat): 2970, 1824, 1743, 1462, 1200, 1014; Calculated for [C₁₀H₁₆NaO₄]⁺: 223.1; Found: 223.1.
4-Isopropyl-4-methyl-3-oxo-oxetane-2-carboxylic acid ethyl ester (8c)

![Structure of 8c]

Compound 8c (dr: 2/1) was prepared in 72% yield according to General Procedure D. The reaction time was 24 h. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 5.70 (s, 0.33H), 5.60 (s, 0.67H), 4.36 – 4.18 (m, 2H), 2.22 (heptet, 0.33H, $J$ = 8.5 Hz), 2.09 (heptet, 0.67H, $J$ = 8.5 Hz), 1.53 (s, 2H), 1.48 (s, 1H), 1.35 – 1.26 (m, 3H), 1.06 – 0.98 (m, 6H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 198.4, 198.3, 165.1, 164.5, 114.3, 113.4, 93.9, 92.9, 62.2, 62.0, 34.0, 33.1, 17.8, 17.3, 16.7, 16.6, 16.5, 16.3, 14.2, 14.0; IR (neat): 2978, 1820, 1743, 1458, 1203, 1014; Calculated for [C$_{10}$H$_{16}$NaO$_4$]$^+$: 223.1; Found: 223.1.

3-Oxo-1-oxa-spiro[3.4]octane-2-carboxylic acid ethyl ester (8d)

![Structure of 8d]

Compound 8d was prepared in 80% yield according to General Procedure D. The reaction time was 20 h. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.65 (s, 1H), 4.34 – 4.24 (m, 2H), 2.38 – 2.31 (m, 1H), 2.16 (t, 2H, $J$ = 7.6 Hz), 2.13 – 2.05 (m, 1H), 1.86 – 1.80 (m, 2H), 1.71 – 1.61 (m, 2H), 1.30 (t, 3H, $J$ = 7.2 Hz); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 199.5, 165.0, 117.4, 94.1, 62.1, 36.4, 36.2, 24.8, 24.7, 14.1; IR (neat): 2966, 2877, 1828, 1747, 1446, 1203, 1022, 987; Calculated for [C$_{10}$H$_{14}$NaO$_4$]$^+$: 221.1; Found: 221.1.

3-Oxo-1-oxa-spiro[3.5]nonane-2-carboxylic acid ethyl ester (8e)

![Structure of 8e]
Compound 8e was prepared in 83% yield according to General Procedure D. The reaction time was 20 h. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.65 (s, 1H), 4.34–4.21 (m, 2H), 2.05–1.97 (m, 2H), 1.94–1.80 (m, 2H), 1.78–1.68 (m, 2H), 1.67–1.54 (m, 2H), 1.50–1.34 (m, 2H), 1.30 (t, 3H, $J = 7.2$ Hz); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 198.1, 165.2, 110.7, 92.7, 62.1, 32.6, 32.1, 24.6, 22.2, 22.0, 14.1; IR (neat): 2943, 2862, 1824, 1747, 1446, 1196, 1007; Calculated for [C$_{11}$H$_{16}$NaO$_4$]$^+$: 235.1; Found: 235.1.

3-Oxo-1-oxa-spiro[3.6]decane-2-carboxylic acid ethyl ester (8f)

![8f](image)

Compound 8f was prepared in 75% yield according to General Procedure D. The reaction time was 24 h. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.65 (s, 1H), 4.35–4.21 (m, 2H), 2.26–2.20 (m, 1H), 2.16–2.02 (m, 3H), 1.76–1.52 (m, 8H), 1.31 (t, 3H, $J = 7.2$ Hz); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 198.6, 165.3, 114.0, 93.0, 62.1, 35.1, 34.6, 28.9, 28.8, 22.0, 14.1; IR (neat): 2931, 2862, 1824, 1747, 1454, 1308, 1265, 1196, 1011, 960; MS (ES$^+$) Calculated for [C$_{12}$H$_{18}$NaO$_4$]$^+$: 249.1; Found: 249.1.

Compound 8g

![8g](image)

Compound 8g was prepared in 92% yield according to General Procedure D. The reaction time was 22 h. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 5.61 (s, 1H), 4.33–4.21 (m, 2H), 2.42 (s, 1H), 2.35 (s, 1H), 2.16 (d, 1H, $J = 12.0$ Hz), 2.07 (d, 1H, $J = 12.0$ Hz), 1.98 (t, 2H, $J = 14.0$ Hz), 1.85–1.76 (m, 4H), 1.72 (s, 2H), 1.68–1.63 (m, 2H), 1.30 (t, 3H, $J = 7.2$ Hz); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 197.8, 165.4, 114.6, 92.3, 62.0, 36.3, 34.2, 34.1,
33.9, 33.6, 32.1, 32.0, 26.4, 26.1, 14.1; IR (neat): 2916, 2854, 1817, 1747, 1446, 1308, 1196, 1030, 984; MS (ES^+) Calculated for [C_{15}H_{21}O_{4}]^+: 265.1; Found: 265.2.

7-Benzyloxy methoxy-3-oxo-1-oxa-spiro[3.5]nonane-2-carboxylic acid ethyl ester (8h)

\[
\text{BOMO} \quad \text{O} \quad \text{CO}_2\text{Et}
\]

8h

Compound 8h (dr: 10/1) was prepared in 74% yield according to General Procedure D. The reaction time was 24 h. Major isomer: \(^1\text{H} \text{NMR (500 MHz, CDCl}_3\) \(\delta\) 7.38 – 7.28 (m, 5H), 5.69 (s, 1H), 4.81 (s, 2H), 4.62 (s, 2H), 4.33 – 4.25 (m, 2H), 3.75 – 3.71 (m, 1H), 2.28 – 2.14 (m, 2H), 1.96 – 1.82 (m, 6H), 1.32 (t, 3H, \(J = 7.2\) Hz); \(^{13}\text{C} \text{NMR (125 MHz, CDCl}_3\) \(\delta\) 198.0, 165.1, 137.8, 128.4, 127.8, 127.7, 109.6, 93.0, 92.9, 71.5, 69.5, 62.2, 29.0, 28.5, 27.4, 27.3, 14.1; IR (neat): 2943, 1805, 1743, 1454, 1377, 1269, 1192, 1038; Calculated for [C_{19}H_{24}NaO_{6}]^+: 371.1; Found: 371.1.

Preparation of 3-(benzylamino)oxetane-3-carbonitrile (9)

\[
\text{CN} \quad \text{NHBn}
\]

9

3-Methoxy-5-bromopyridine N-oxide (1.0 mmol), Tf\(_2\)NH (3.0 mL, 0.20 M in DCE), and (2-biphenyl)Cy\(_2\)PAuNTf\(_2\) (20.7 mg, 0.025 mmol) were added sequentially to a solution of propargyl alcohol (28.0 mg, 0.50 mmol) in DCE (7.0 mL) at room temperature. The reaction mixture was stirred at rt for 0.5 h (\(^1\text{H} \text{NMR monitoring of this reaction progress was carried out under the same conditions except that CD}_2\text{Cl}_2\) instead of DCE was used solvent). Then, benzylamine (73 mg, 0.73 mmol) and trimethylacetonitrile (79 mg, 0.73 mmol) were added at rt. After stirring for 8 h, the mixture was concentrated and the residue was purified by chromatography on silica gel (eluent: hexanes/ethyl acetate) to afford the desired product. This compound is known and the spectroscopic data match
those reported.\textsuperscript{5} \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.41 – 7.29 (m, 5H), 4.85 (d, 2H, \(J = 6.8\) Hz), 4.47 (d, 2H, \(J = 6.8\) Hz), 3.86 (s, 2H), 1.93 (bs, 1H); \textsuperscript{13}C NMR (125 MHz, CDCl\textsubscript{3}) \(\delta\) 137.6, 128.7, 128.5, 127.9, 118.8, 79.2, 53.8, 49.5; IR (neat): 3313 (bs), 2954, 2877, 2229, 1454, 1219, 987, 744, 698; MS (ES\textsuperscript{+}) Calculated for [C\textsubscript{11}H\textsubscript{12}N\textsubscript{2}NaO]\textsuperscript{+}: 211.1; Found: 211.1.

**Preparation of ethyl 2-(oxetan-3-ylidene)acetate**

![Diagram of ethyl 2-(oxetan-3-ylidene)acetate](image)

3-Methoxy-5-bromopyridine \(N\)-oxide (1.0 mmol), Tf\textsubscript{2}NH (3.0 mL, 0.20 M in DCE), and (2-biphenyl)Cy\textsubscript{2}PAuNTf\textsubscript{2} (20.7 mg, 0.025 mmol) were added sequentially to a solution of propargyl alcohol (28.0 mg, 0.50 mmol) in DCE (7.0 mL) at room temperature. The reaction mixture was stirred at rt for 0.5 h. Then, the reaction was treated with saturated aqueous NaHCO\textsubscript{3} (5 mL) and the resulting solution was extracted with DCM (1 × 10 mL) and the combined organic layers were dried with MgSO\textsubscript{4}. The mixture was filtrated and the residue was used directly for the next step.

To the above solution was added carboethoxymethylene triphenylphosphorane (192 mg, 0.55 mmol, 1.5 equiv) at rt. After stirring for 5 h, the mixture was concentrated and the residue was purified by silica gel flash chromatography (eluent: hexanes/ethyl acetate) to afford the desired product. This compound is known and the spectroscopic data match those reported.\textsuperscript{6} \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 5.63 – 5.61 (m, 1H), 5.50 – 5.48 (m, 2H), 5.30 – 5.28 (m, 2H), 4.15 (q, 2H, \(J = 7.2\) Hz), 1.26 (t, 3H, \(J = 7.2\) Hz); \textsuperscript{13}C NMR (125 MHz, CDCl\textsubscript{3}) \(\delta\) 165.2, 159.1, 111.1, 81.0, 78.4, 60.3, 14.2; IR (neat): 2985, 2931, 2862, 1724, 1446, 1342, 1261, 1207, 1099, 1034, 960, 864, 833; MS (ES\textsuperscript{+}) Calculated for [C\textsubscript{7}H\textsubscript{10}NaO\textsubscript{3}]\textsuperscript{+}: 165.1; Found: 165.1.

**Reference:**

Gold-Catalyzed Synthesis of Oxetan-3-ones
Ye, L.; He, W.; Zhang, L.

MeCH\(_2\)CH=CH$_2$
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-24
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-26
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-27
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-31

PS

\[ \text{HO} \]

\[ \text{C} \equiv \text{C} \]

\[ \text{CH}_3 \]
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-32
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-36
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-40
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-44
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-48
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-50
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-52
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-53
Gold-Catalyzed Synthesis of Oxetan-3-ones
Ye, L.; He, W.; Zhang, L.

SI-55
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-56
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-58
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-61
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-67
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones
Ye, L.; He, W.; Zhang, L.

[Chemical structure image]
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-72
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-73
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-75
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-79
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-80
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-81
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-82
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-86
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-91
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-92
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-93
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

SI-97
Ph
\[ \text{5b} \]

**<Chromatogram>**

<table>
<thead>
<tr>
<th>Peak#</th>
<th>Ret. Time</th>
<th>Area</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>26.441</td>
<td>1382759</td>
<td>50.028</td>
</tr>
<tr>
<td>2</td>
<td>27.840</td>
<td>1381195</td>
<td>49.972</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>2763954</td>
<td>100.000</td>
</tr>
</tbody>
</table>

**<Chromatogram>**

<table>
<thead>
<tr>
<th>Peak#</th>
<th>Ret. Time</th>
<th>Area</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>26.615</td>
<td>422444</td>
<td>9.865</td>
</tr>
<tr>
<td>2</td>
<td>27.839</td>
<td>3859983</td>
<td>90.135</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>4282427</td>
<td>100.000</td>
</tr>
</tbody>
</table>
Gold-Catalyzed Synthesis of Oxetan-3-ones

Ye, L.; He, W.; Zhang, L.

<Chromatogram>

![Chromatogram Image]

**Peak Table**

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Ret. Time</th>
<th>Area</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>14.934</td>
<td>724498</td>
<td>49.678</td>
</tr>
<tr>
<td>2</td>
<td>16.305</td>
<td>733876</td>
<td>50.322</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>1458374</td>
<td>100.000</td>
</tr>
</tbody>
</table>

<Chromatogram>

![Chromatogram Image]

**Peak Table**

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Ret. Time</th>
<th>Area</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15.090</td>
<td>1377288</td>
<td>90.354</td>
</tr>
<tr>
<td>2</td>
<td>16.590</td>
<td>147039</td>
<td>9.646</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>1524327</td>
<td>100.000</td>
</tr>
</tbody>
</table>