Supplementary Information

Silver-Mediated Radical Cyclization: Construction of $\Delta^2$-Isoxazolines from $\alpha$-Halo Ketoximes and 1,3-Dicarbonyl Compounds

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(A) General Experimental Procedure

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Substrates 1a-1k were prepared according to literature procedures.\[1\]

(b) General Procedures for Silver-Mediated Synthesis of Isoxazolines 3:

Conditions A: To a Schlenk tube were added $\alpha$-halo ketoxime 1 (0.3 mmol), 1,3-dicarbonyl compound 2 (2a-2e) (0.6 mmol), Ag$_2$CO$_3$ (0.6 mmol), K$_2$CO$_3$ (0.3 mmol) and DMA (3 mL). Then the tube was charged with argon, and was stirred at 50°C for about 20 h until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was washed with brine. The aqueous phase was re-extracted with ethyl acetate. The combined organic extracts were dried over Na$_2$SO$_4$, concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate = 20:1) to afford the desired product 3.

Conditions B: To a Schlenk tube were added $\alpha$-halo ketoxime 1 (0.3 mmol), 1,3-dicarbonyl compound 2 (2f-2q) (0.6 mmol), Ag$_2$CO$_3$ (0.6 mmol), Cs$_2$CO$_3$ (0.3 mmol) and DMA (3 mL). Then the tube was charged with argon, and was stirred at 50°C for about 20 h until complete consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was washed with brine. The aqueous phase was re-extracted with ethyl acetate. The combined organic extracts were dried over Na$_2$SO$_4$, concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate = 20:1) to afford the desired product 3.
(c) Table S1. Screening Optimizing Reaction Conditions

<table>
<thead>
<tr>
<th>Entry</th>
<th>[M] [equiv]</th>
<th>Base [equiv]</th>
<th>Solvent</th>
<th>Isolated Yield [%]</th>
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<tr>
<td>1</td>
<td>Ag₂CO₃ (0.1)</td>
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<td>——</td>
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<td>K₂CO₃ (1)</td>
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</table>

*a Reaction conditions: 1a (0.3 mmol), 2a (2 equiv), [M], base, and DMA (N,N-diethylacetamide, 3 mL) at room temperature under argon. ** At 50 °C.

(d) Scheme S1  Base-Mediated the Reaction of 2-Chloro-1-phenylethanone

Oxime (1a) with 1,3-Dicarboxyl compounds (2)

As shown in Scheme S1, the reaction of 2-chloro-1-phenylethanone oxime (1a) with 1,3-dicarboxyl compounds 2 in the presence of bases alone were performed to increasing the length of the carbon chain. The results in Table 1 indicated that only 15% yield of 5-(acetoxyimino)-5-phenylpentan-2-one (4aa) was isolated from 2-chloro-1-phenylethanone oxime (1a) and pentane-2,4-dione (2a) in the presence of 1 equiv K₂CO₃ (entry 5; Table 1). Thus, the effect of bases was screened, and the results demonstrated that Na₂CO₃ was a preferred base in view of the yield, and the amount of Na₂CO₃ was found to affect the yield (Eq 1): While the reaction afforded the desired product 4aa in 40% yield at a loading of 1 equiv Na₂CO₃, the yield was increased from 40% to 60% at 2 equiv Na₂CO₃ and to 58% using 3 equiv Na₂CO₃. 1,3-Diketones 2c and 2d, containing a or two phenyl groups adjacent to the carbonyl group, provided the corresponding products 4ac and 4ad in excellent yields. Using 3-keto ester 2o, however, only nucleophilic replacement product 5ao was obtained in the presence of Na₂CO₃ or Cs₂CO₃ (Eq 2).
(e) Scheme S2  Control Experiments

To elucidate the mechanism, some control experiments were carried out (Scheme S2). The results in Eq 3 showed that the reactivity of substrate 5ao was rather lower using Ag$_2$CO$_3$ alone. To our delight, substrate 5ao could be readily converted to isoxazoline 3aa in the presence of both Ag$_2$CO$_3$ and K$_2$CO$_3$ (94% yield). These suggest that substrate 5ao may be an intermediate for this Ag-mediated transformation. Subsequently, two radical inhibitors, BHT (2,6-di-tert-butyl-4-methylphenol) and hydroquinone, were added to this Ag-mediated reaction (Eq 3): a stoichiometric amount of BHT (2 equiv) or hydroquinone (2 equiv) resulted in no conversion of substrate 5ao. Identical results were observed from the reaction substrate 1a and diketone 2a in the presence of either BHT or hydroquinone (Eq 4). These results imply that this Ag-mediated transformation includes a radical process, and the generation of a radical at the 2 position of 1,3-dicarbonyl compounds can be triggered by Ag$_2$CO$_3$. 8

Notably, the silver salts were recovered and reused among this current reaction (Eq 5). 8e Excess Ag$_2$CO$_3$ and all silver species were filtrated after the reaction and in turn treated with nitric acid and Na$_2$CO$_3$ to recover fresh Ag$_2$CO$_3$. Interestingly, the fresh Ag$_2$CO$_3$ was also efficient for this current reaction without loss of activity (Eq 5).
Analytical data for 3-5

1,1’-(3-phenyl-4,5-dihydroisoxazole-5,5-diyl)diethanone (3aa):

White solid, mp 78.3-79.6 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) δ:
7.67-7.64 (m, 2H), 7.47-7.39 (m, 3H), 3.78 (s, 2H), 2.36 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ: 202.4, 156.6, 131.0, 128.9, 127.7, 126.9, 97.5, 40.1, 25.9; IR (KBr, cm$^{-1}$): 1719, 1689; LRMS (EI, 70 eV) m/z (%): 231 (M$^+$, 4), 189 (100), 160 (21), 118 (85); HRMS m/z (ESI) calcd for C$_{13}$H$_{14}$NO$_3$ (M+H)$^+$ 232.0968, found 232.0964.

1,1’-(3-phenyl-4,5-dihydroisoxazole-5,5-diyl)dipropan-1-one (3ab):

White solid, mp 89.0-89.9 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) δ:
7.67-7.64 (m, 2H), 7.46-7.38 (m, 3H), 3.78 (s, 2H), 2.36 (s, 6H), 2.84-2.64 (m, 4H), 1.09 (t, $J = 7.2$ Hz, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ: 205.4, 156.6, 130.9, 128.9, 127.9, 126.9, 97.7, 40.7, 31.7, 7.3; IR (KBr, cm$^{-1}$): 1720, 1693; LRMS (EI, 70 eV) m/z (%): 259 (M$^+$, 1), 203 (20), 146 (18), 57 (100); HRMS m/z (ESI) calcd for C$_{15}$H$_{18}$NO$_3$ (M+H)$^+$ 260.1281, found 260.1282.

5-benzoyl-3-phenyl-4,5-dihydroisoxazole-5-carbonitrile (3ae):
White solid, mp 102.2-103.5 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 8.28-8.26 (m, 2H), 7.71-7.68 (m, 3H), 7.56 (t, $J = 8.0$ Hz, 2H), 7.52-7.43 (m, 3H), 4.65 (d, $J = 17.2$ Hz, 1H), 3.92 (d, $J = 17.6$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 185.2, 156.8, 135.1, 131.4, 130.7, 128.9, 127.2, 126.9, 116.9, 81.5, 43.7; IR (KBr, cm$^{-1}$): 1691; LRMS (EI, 70 eV) $m/z$ (%): 276 (M$^+$, 1), 249 (2), 221 (2), 105 (100); HRMS $m/z$ (ESI) calcd for C$_{17}$H$_{13}$N$_2$O$_2$ (M+H)$^+$ 277.0972, found 277.0983.

![Structure of 5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid methyl ester (3af):](image1)

5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid methyl ester (3af):

Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.67-7.64 (m, 2H), 7.46-7.38 (m, 3H), 3.94-3.83 (m, 5H), 2.42 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 202.4, 167.9, 156.5, 130.8, 128.8, 127.8, 126.9, 92.6, 53.6, 40.9, 25.8; IR (KBr, cm$^{-1}$): 1751, 1695; LRMS (EI, 70 eV) $m/z$ (%): 247 (M$^+$, 4), 188 (46), 177 (36), 144 (100); HRMS $m/z$ (ESI) calcd for C$_{13}$H$_{14}$NO$_4$ (M+H)$^+$ 248.0917, found 248.0909.

![Structure of 5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid ethyl ester (3ag):](image2)

5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid ethyl ester (3ag):

Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.67-7.64 (m, 2H), 7.45-7.38 (m, 3H), 4.32-4.27 (m, 2H), 3.94-3.81 (m, 2H), 2.42 (s, 3H), 1.31 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 202.5, 167.3, 156.5, 130.8, 128.8, 127.9, 126.9, 92.7, 62.9, 40.8, 25.8, 13.9; IR (KBr, cm$^{-1}$): 1749, 1698; LRMS (EI, 70 eV) $m/z$ (%): 261
(M⁺, 3), 176 (7), 162 (14), 144 (100); HRMS m/z (ESI) calcd for C₁₄H₁₆NO₄ (M+H)⁺ 262.1074, found 262.1083.

5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid isopropyl ester (3ah):

Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ: 7.66-7.64 (m, 2H), 7.44-7.37 (m, 3H), 5.15-5.08 (m, 1H), 3.93-3.77 (m, 2H), 2.41 (s, 3H), 1.29 (t, J = 6.0 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ: 202.4, 166.8, 156.4, 130.7, 128.8, 128.0, 126.9, 92.8, 71.0, 40.6, 25.7, 21.4 (2C); IR (KBr, cm⁻¹): 1754, 1693; LRMS (EI, 70 eV) m/z (%): 275 (M⁺, 3), 188 (50), 160 (18), 144 (100); HRMS m/z (ESI) calcd for C₁₅H₁₈NO₄ (M+H)⁺ 276.1230, found 276.1243.

5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid benzyl ester (3ai):

Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ: 7.67-7.64 (m, 2H), 7.44-7.37 (m, 3H), 5.27 (s, 2H), 3.96-3.82 (m, 2H), 2.38 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 202.2, 167.2, 156.5, 134.5, 130.8, 128.8, 128.6 (2C), 128.3, 128.2, 126.9, 92.7, 68.3, 40.8, 25.8; IR (KBr, cm⁻¹): 1746, 1688; LRMS (EI, 70 eV) m/z (%): 323 (M⁺, 1), 236 (8), 188 (15), 91 (100); HRMS m/z (ESI) calcd for C₁₉H₁₈NO₄ (M+H)⁺ 324.1230, found 324.1256.
5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid 2-methoxyethyl ester (3aj):

Colorless oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 7.65-7.63 (m, 2H), 7.44-7.36 (m, 3H), 4.38-4.36 (m, 2H), 3.97-3.78 (m, 2H), 3.60-3.58 (m, 2H), 3.33 (s, 3H), 2.41 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\): 201.6, 167.4, 156.4, 130.8, 128.8, 127.9, 126.9, 92.5, 69.7, 65.4, 58.8, 40.6, 25.6; IR (KBr, cm\(^{-1}\)): 1756, 1696; LRMS (EI, 70 eV) \(m/z\) (%): 291 (M\(^+\), 1), 221 (5), 188 (64), 144 (100); HRMS \(m/z\) (ESI) calcd for C\(_{15}\)H\(_{18}\)NO\(_5\) (M+H\(^+\)) 292.1179, found 292.1190.

5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid allyl ester (3ak):

Colorless oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 7.66-7.64 (m, 2H), 7.45-7.37 (m, 3H), 5.95-5.85 (m, 1H), 5.37-5.26 (m, 2H), 4.72 (d, \(J = 5.6\) Hz, 2H), 3.94-3.83 (m, 2H), 2.42 (s, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\): 202.4, 167.0, 156.5, 130.8, 130.6, 128.8, 127.9, 126.9, 119.6, 92.7, 67.1, 40.8, 25.8; IR (KBr, cm\(^{-1}\)): 1749, 1690; LRMS (EI, 70 eV) \(m/z\) (%): 273 (M\(^+\), 3), 202(6), 188 (50), 144 (100); HRMS \(m/z\) (ESI) calcd for C\(_{15}\)H\(_{16}\)NO\(_4\) (M+H\(^+\)) 274.1074, found 274.1082.
3-phenyl-5-propionyl-4,5-dihydroisoxazole-5-carboxylic acid methyl ester (3al):

Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.65-7.64 (m, 2H), 7.45-7.37 (m, 3H), 3.89 (s, 2H), 3.83 (s, 3H), 2.85-2.80 (m, 2H), 1.09 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 205.4, 168.0, 156.5, 130.8, 128.8, 127.9, 126.9, 92.6, 53.6, 41.3, 31.5, 7.2; IR (KBr, cm$^{-1}$): 1756, 1694; LRMS (EI, 70 eV) $m/z$ (%): 261 (M$^+$, 1), 202 (10), 144 (16), 57 (100); HRMS $m/z$ (ESI) calcd for C$_{14}$H$_{16}$NO$_4$ (M+H)$^+$ 262.1074, found 262.1083.

5-butyryl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid ethyl ester (3am):

Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.67-7.65 (m, 2H), 7.45-7.38 (m, 3H), 4.31-4.26 (m, 2H), 3.93-3.82 (m, 2H), 2.77 (t, $J = 7.2$ Hz, 2H), 1.67-1.61 (m, 2H), 1.30 (t, $J = 7.2$ Hz, 3H), 0.92 (t, $J = 7.6$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 204.8, 167.5, 156.5, 130.8, 128.8, 128.0, 126.9, 92.8, 62.9, 41.0, 39.9, 16.6, 13.9, 13.4; IR (KBr, cm$^{-1}$): 1750, 1689; LRMS (EI, 70 eV) $m/z$ (%): 289 (M$^+$, 1), 216 (12), 144 (16), 71 (100); HRMS $m/z$ (ESI) calcd for C$_{16}$H$_{20}$NO$_4$ (M+H)$^+$ 290.1387, found 290.1396.

5-isobutyryl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid methyl ester (3an):
Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.68-7.65 (m, 2H), 7.46-7.38 (m, 3H), 3.89 (s, 2H), 3.83 (s, 3H), 3.32-3.25 (m, 1H), 1.18 (d, $J = 6.8$ Hz, 3H), 1.11 (d, $J = 6.8$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 208.9, 168.2, 156.5, 130.8, 128.8, 127.9, 127.0, 92.7, 53.5, 41.8, 36.8, 18.9, 18.7; IR (KBr, cm$^{-1}$): 1752, 1697; LRMS (EI, 70 eV) m/z (%): 275 (M$^+$, 1), 232 (42), 172 (70), 71 (100); HRMS m/z (ESI) calcd for C$_{15}$H$_{18}$NO$_4$ (M+H)$^+$ 276.1230, found 276.1242.

5-benzoyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid ethyl ester (3ao):$^{[2]}$

Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 8.14-8.12 (m, 2H), 7.71-7.69 (m, 2H), 7.61-7.57 (m, 1H), 7.49-7.38 (m, 5H), 4.56 (d, $J = 17.6$ Hz, 1H), 4.26-4.21 (m, 2H), 3.72 (d, $J = 17.6$ Hz, 1H), 1.15 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 189.9, 169.0, 156.1, 133.9, 133.3, 130.7, 130.0, 128.7, 128.6, 128.0, 127.0, 91.9, 62.7, 42.1, 13.7; LRMS (EI, 70 eV) m/z (%): 323 (M$^+$, 1), 250 (88), 190 (18), 105 (100).

5-acetyl-N,3-diphenyl-4,5-dihydroisoxazole-5-carboxamide (3ap):

Pale yellow solid, mp 149.2-150.7 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 8.76 (s, 1H), 7.67-.7.60 (m, 4H), 7.48-7.34 (m, 5H), 7.17 (t, $J = 7.6$ Hz, 1H), 4.29 (d, $J = 17.6$ Hz, 1H), 3.72 (d, $J = 18$ Hz, 1H), 2.41 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 197.6, 166.3, 158.0, 136.4, 131.2, 129.1, 128.9, 127.1, 125.3, 119.9, 93.1,
40.8, 25.3; IR (KBr, cm\(^{-1}\)): 1730, 1681; LRMS (EI, 70 eV) m/z (%): 308 (M\(^+\), 1), 207 (18), 188 (89), 146 (100); HRMS m/z (ESI) calcd for C\(_{18}\)H\(_{17}\)N\(_2\)O\(_3\) (M+H\(^+\)) 309.1234, found 309.1230.

1,1'-(3-\(p\)-tolyl-4,5-dihydroisoxazole-5,5-diyl)diethanone (3ca):

White solid, mp 74.6-75.9 °C (uncorrected); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\):
7.54 (d, \(J = 6.4\) Hz, 2H), 7.21 (d, \(J = 6.8\) Hz, 2H), 3.75 (s, 2H), 2.37 (s, 3H), 2.35 (s, 6H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\): 202.5, 156.5, 141.3, 129.5, 126.9, 124.9, 97.4, 40.2, 25.7, 21.4; IR (KBr, cm\(^{-1}\)): 1720, 1687; LRMS (EI, 70 eV) m/z (%): 245 (M\(^+\), 12), 203 (75), 160 (52), 132 (100); HRMS m/z (ESI) calcd for C\(_{14}\)H\(_{16}\)NO\(_3\) (M+H\(^+\)) 246.1125, found 246.1142.

1,1'-(3-(4-methoxyphenyl)-4,5-dihydroisoxazole-5,5-diyl)diethanone (3da):

White solid, mp 88.9-90.1 °C (uncorrected); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\):
7.60-7.56 (m, 2H), 6.93-6.89 (m, 2H), 3.82 (s, 3H), 3.73 (s, 2H), 2.34 (s, 6H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\): 202.6, 161.6, 156.1, 128.6, 120.2, 114.2, 97.3, 55.3, 40.3, 25.8; IR (KBr, cm\(^{-1}\)): 1717, 1690; LRMS (EI, 70 eV) m/z (%): 261 (M\(^+\), 29), 219 (42), 176 (49), 148 (100); HRMS m/z (ESI) calcd for C\(_{14}\)H\(_{16}\)NO\(_4\) (M+H\(^+\)) 262.1074, found 262.1083.
1,1'-((3-(4-chlorophenyl)-4,5-dihydroisoxazole-5,5-diyl)dianethanone (3ea):

White solid, mp 78.4-79.6 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.58 (d, $J = 8.4$ Hz, 2H), 7.38 (d, $J = 8.4$ Hz, 2H), 3.73 (s, 2H), 2.35 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 202.1, 155.7, 137.0, 129.2, 128.2, 126.3, 97.8, 39.8, 25.8; IR (KBr, cm$^{-1}$): 1719, 1688; LRMS (EI, 70 eV) $m/z$ (%): 267 (M$^+$+2, 3), 265 (M$^+$, 9), 222 (100), 180 (63); HRMS $m/z$ (ESI) calcd for C$_{13}$H$_{13}$ClNO$_3$ (M+H)$^+$ 266.0578, found 266.0586.

1,1'-((3-(4-bromophenyl)-4,5-dihydroisoxazole-5,5-diyl)dianethanone (3fa):

White solid, mp 97.1-98.1 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.62-7.47 (m, 4H), 3.73 (s, 2H), 2.35 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 202.1, 155.8, 132.1, 128.3, 126.7, 125.3, 97.8, 39.8, 25.8; IR (KBr, cm$^{-1}$): 1718, 1688; LRMS (EI, 70 eV) $m/z$ (%): 311 (M$^+$+2, 10), 309 (M$^+$, 11), 267 (100), 226 (47); HRMS $m/z$ (ESI) calcd for C$_{13}$H$_{13}$BrNO$_3$ (M+H)$^+$ 310.0073, found 310.0076.

4-(5,5-diacetyl-4,5-dihydroisoxazol-3-yl)benzonitrile (3ga):

White solid, mp 128.5-129.5 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.77-7.69 (m, 4H), 3.76 (s, 2H), 2.36 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 201.6,
1,1’-(3-(4-nitrophenyl)-4,5-dihydroisoxazole-5,5-diyl)diethanone (3ha):

White solid; mp 138.0-139.7 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) δ:

8.24 (d, $J$ = 8.8 Hz, 2H), 7.83 (d, $J$ = 8.8 Hz, 2H), 3.80 (s, 2H), 2.36 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ: 201.5, 155.1, 148.8, 133.8, 127.8, 124.0, 98.4, 39.4, 25.8; IR (KBr, cm$^{-1}$): 1722, 1687; LRMS (EI, 70 eV) m/z (%): 276 (M$^+$, 1), 234 (100), 206 (43), 191 (44); HRMS m/z (ESI) calcd for C$_{13}$H$_{13}$N$_2$O$_5$ (M+H)$^+$ 277.0819, found 277.0823.

1,1’-(3-(thiophen-3-yl)-4,5-dihydroisoxazole-5,5-diyl)diethanone (3ia):

White solid; mp 109.6-110.4 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) δ:

7.53-7.52 (m, 1H), 7.46-7.44 (m, 1H), 7.39-7.37 (m, 1H), 3.74 (s, 2H), 2.35 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ: 202.3, 152.6, 129.4, 127.3, 127.0, 125.4, 97.2, 40.7, 25.8; IR (KBr, cm$^{-1}$): 1717, 1686; LRMS (EI, 70 eV) m/z (%): 237 (M$^+$, 10), 195 (100), 166 (16), 152 (55); HRMS m/z (ESI) calcd for C$_{11}$H$_{12}$NO$_3$S (M+H)$^+$ 238.0532, found 238.0538.
(E)-5-(acetoxyimino)-5-phenylpentan-2-one (4aa):

Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.67 (d, $J$ = 7.2 Hz, 2H), 7.44-7.38! (m, 3H), 3.09 (t, $J$ = 8.0 Hz, 2H), 2.68 (t, $J$ = 8.0 Hz, 2H), 2.25 (s, 3H), 2.14 (s, 3H);
$^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 206.1, 168.9, 165.3, 133.3, 130.7, 128.7, 127.2, 39.7, 29.8, 22.1, 19.8; IR (KBr, cm$^{-1}$): 1763, 1698; HRMS m/z (ESI) calcd for C$_{13}$H$_{16}$NO$_3$ (M+H)$^+$ 234.1125, found 234.1127.

(E)-4-(benzoyloxyimino)-1,4-diphenylbutan-1-one (4ac):

White solid, mp 92.3-93.4 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 8.09-8.07 (m, 2H), 7.94-7.91 (m, 2H), 7.85-7.83 (m, 2H), 7.61-7.54 (m, 2H), 7.50-7.41 (m, 7H), 3.46-3.42 (m, 2H), 3.34-3.30 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 197.6, 166.5, 163.7, 136.1, 133.5, 133.4, 130.9, 129.6, 128.8 (2C), 128.7, 128.6, 128.0, 127.3, 35.2, 22.9; IR (KBr, cm$^{-1}$): 1727, 1677; HRMS m/z (ESI) calcd for C$_{23}$H$_{20}$NO$_3$ (M+H)$^+$ 358.1438, found 358.1434.
(E)-4-(acetoxyimino)-1,4-diphenylbutan-1-one (4ad):

White solid, mp 80.4-81.6 °C (uncorrected); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 7.92 (d, $J = 7.6$ Hz, 2H), 7.75 (d, $J = 6.8$ Hz, 2H), 7.55 (t, $J = 7.6$ Hz, 1H), 7.46-7.38 (m, 5H), 3.30-3.20 (m, 4H), 2.23 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 197.5, 168.7, 165.4, 136.2, 133.4, 133.3, 130.7, 128.7, 128.6, 127.9, 127.2, 35.1, 22.7, 19.7; IR (KBr, cm$^{-1}$): 1758, 1681; HRMS m/z (ESI) calcd for C$_{18}$H$_{18}$NO$_3$ (M+H)$^+$ 296.1281, found 296.1273.

(E)-ethyl 2-benzoyl-4-(hydroxyimino)-4-phenylbutanoate (5ao):

Colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$: 9.63 (s, 1H), 7.94 (d, $J = 7.6$ Hz, 2H), 7.59-7.52 (m, 3H), 7.43-7.32 (m, 5H), 4.96 (t, $J = 7.6$ Hz, 1H), 3.98-3.92 (m, 2H), 3.51-3.40 (m, 2H), 1.01 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$: 194.6, 169.3, 157.0, 135.8, 135.2, 133.6, 129.4, 128.6 (2C), 128.5, 126.6, 61.6, 50.5, 26.4, 13.6.

(C) Reference

(D) Spectra

1,1'-(3-phenyl-4,5-dihydroisoxazole-5,5-diyl)diethanone (3aa):
1,1'-(3-phenyl-4,5-dihydroisoxazole-5,5-diyl)dipropan-1-one (3ab):
5-benzoyl-3-phenyl-4,5-dihydroisoxazole-5-carbonitrile (3ae):
5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid methyl ester (3af):
5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid ethyl ester (3ag):
5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid isopropyl ester (3ah):
5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid benzyl ester (3ai):
5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid 2-methoxyethyl ester

(3aj):
5-acetyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid allyl ester (3ak):
3-phenyl-5-propionyl-4,5-dihydroisoxazole-5-carboxylic acid methyl ester (3al):
5-butyryl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid ethyl ester (3am):
5-isobutyryl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid methyl ester (3an):
5-benzoyl-3-phenyl-4,5-dihydroisoxazole-5-carboxylic acid ethyl ester (3ao):
5-acetyl-N,3-diphenyl-4,5-dihydroisoxazole-5-carboxamide (3ap):
1,1'-(3-\(p\)-tolyl-4,5-dihydroisoxazole-5,5-diyl)diethanone (3ca):
1,1'-(3-(4-methoxyphenyl)-4,5-dihydroisoxazole-5,5-diyl)diethanone (3da):
1,1'-((3-(4-chlorophenyl)-4,5-dihydroisoxazole-5,5-diyl)diethanone (3ea):
1,1'-[(3-(4-bromophenyl)-5,5-dihydroisoxazole-4,5-diyl)diethanone (3fa): 

![chemical structure image]

![NMR spectra image]

![chemical structure image]
4-(5,5-diacetyl-4,5-dihydroisoxazol-3-yl)benzonitrile (3ga):
1,1'-(3-(4-nitrophosphyl)-4,5-dihydroisoxazole-5,5-diyl)diethanone (3ha):
1,1’-(3-(thiophen-3-yl)-4,5-dihydroisoxazole-5,5-diyl)diethanone (3ia):
(E)-5-(acetoxyimino)-5-phenylpentan-2-one (4aa):
(E)-4-(benzoyloxyimino)-1,4-diphenylbutan-1-one (4ac):
(E)-4-(acetoxyimino)-1,4-diphenylbutan-1-one (4ad):
(E)-ethyl 2-benzoyl-4-(hydroxyimino)-4-phenylbutanoate (5ao):