Convenient route to trisubstituted oxazoles *via* a copper-catalysed tandem oxidative cyclisation by oxygen oxidation

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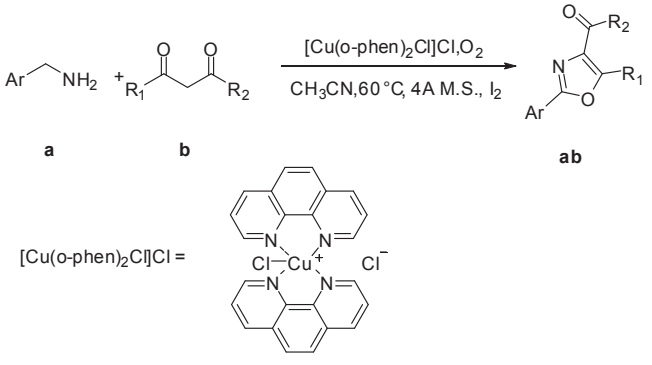
A novel copper-catalysed oxidative cyclisation has been developed for the synthesis of trisubstituted oxazoles, which is thought to proceed through cascade formation of C–N and C–O bonds by oxygen oxidation. The desired products can be obtained from readily available starting materials while avoiding hazardous materials. Therefore, a green synthetic method for the preparation of oxazoles has been found.

Keywords: copper, oxazoles, β-diketones, benzylamine, oxygen oxidation

The oxazole moiety, which has attracted increasing attention in both industrial and academic fields for decades, is a significant structure in numerous bioactive natural products. Furthermore, a large number of natural and synthetic compounds which contain the moiety exhibit significant biological activities such as anticancer, antifungal, antitumour, anti-inflammatory, and antiviral properties. For instance, leucamide A and its analogues, which are important anticancer reagents,[1] are mainly composed of several oxazole moieties. Moreover, oxazole derivatives can also be used as organic materials, such as a corrosion inhibitor.[2] Thus, various synthetic methodologies of oxazole derivatives have been developed, such as cyclodehydration of acyclic precursors[3–11] or oxidative functionalisation of alkynes.[12–14] Iodine catalysed C–H bond functionalisation has been extensively explored.[15,16] However, the development of simple and efficient methods for the preparation of trisubstituted oxazoles is still desirable. Wang[17] and Zhu[18] have reported the synthesis of polysubstituted oxazoles, but they used *tert*-butyl hydroperoxide (TBHP), which is a hazardous material. Narender has developed a route to utilise the iodine promoted C–H functionalisation for accessing poly-substituted oxazoles under metal and peroxide free conditions.[19] Copper, among the transition metals, is particularly attractive in organic synthesis because of its low price, slight toxicity, and environmentally benign features. There have been many reports on copper-catalysed oxidative C–H bond activation.[13,18] We now describe a copper-catalysed oxidative tandem cyclisation of 1, 3-dicarbonyl derivatives and benzylamines to form oxazoles by O_2 oxidation (Scheme 1).



Scheme 1 Synthesis of trisubstituted oxazoles.

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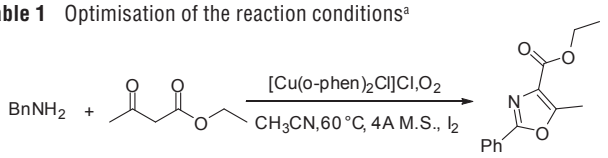
Results and discussion

To initiate our study, the reaction of ethyl acetoacetate with benzylamine was chosen as a model reaction in the presence of a copper source in CH_3CN at 40 °C. First, the model reaction was carried out with copper catalysts; the desired product, ethyl 5-methyl-2-phenyloxazole-4-carboxylate, was isolated in low yields or there was no reaction (Table 1, entries 1–5). Then we optimised the reaction conditions to increase the reaction yield. Changing the reaction temperature had a positive influence on this reaction, resulting in an increase in the yield to 63% (Table 1, entry 6). When the model reaction was carried out in the presence of O_2, the desired product **1ab** was isolated in 79% yield (Table 1, entry 7). Encouraged by this result, we have tried to raise the temperature to 80 °C or modulated the reaction time, but they had no positive influence (Table 1, entries 8–9). Following these results, the other solvents, such as toluene, THF, $CHCl_3$ and DMF, were used in place of CH_3CN for this reaction. CH_3CN gave the best result (Table 1, entries 7, 10–13).

Table 1 Optimisation of the reaction conditions[a]



1a **1b** **1ab**

Entry	Catalyst	Solvent	Temp./°C	Atmosphere	Yield/%[b]
1	$Cu(OAc)_2.H_2O$	CH_3CN40	Air	13	
2	$CuCl_2$	CH_3CN	40	Air	NR[c]
3	$Cu(OTf)_2$	CH_3CN	40	Air	0
4	$CuSO_4$	CH_3CN	40	Air	0
5	[Cu(*o*-phen)$_2$Cl]Cl	CH_3CN	40	Air	42
6	[Cu(*o*-phen)$_2$Cl]Cl	CH_3CN	60	Air	63
7	[Cu(*o*-phen)$_2$Cl]Cl	CH_3CN	60	O_2	79
8	[Cu(*o*-phen)$_2$Cl]Cl	CH_3CN	80	O_2	78
9[d]	[Cu(*o*-phen)$_2$Cl]Cl	CH_3CN	60	O_2	79
10	[Cu(*o*-phen)$_2$Cl]Cl	Toluene	60	O_2	0
11	[Cu(*o*-phen)$_2$Cl]Cl	THF	60	O_2	64
12	[Cu(*o*-phen)$_2$Cl]Cl	CH_3Cl	60	O_2	69
13	[Cu(*o*-phen)$_2$Cl]Cl	DMF	60	O_2	73
14[e]	[Cu(*o*-phen)$_2$Cl]Cl	CH_3CN	60	O_2	77

[a]Reaction conditions: benzylamine (1.5 equiv., 1.5 mmol), ethyl acetoacetate (1 equiv., 1 mmol), catalyst (0.1 mmol, 10 mmol%), 4 Å M.S. (0.1 g), I_2 (1.2 equiv., 1.2 mmol), solvent (5 mL). The reaction was performed for 24 h.

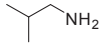
[b]Isolated yield.

[c]No reaction.

[d]The reaction was performed for 48 h.

[e]The addition of benzylamine was reduced to 1.1 equiv.

Table 2 Synthesis of oxazole derivatives[a]

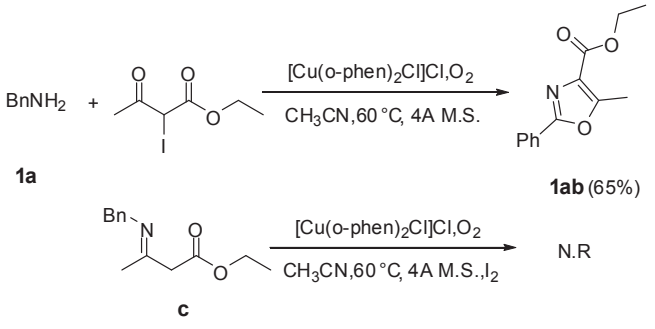
Entry	Ar	R_1	R_2	Time/h	Yield /%[b]
1	C_6H_5	CH_3	OEt	24	79
2	C_6H_5	CH_3	OMe	26	77
3	C_6H_5	CH_3	O*t*-Bu	25	80
4	C_6H_5	*n*-Pr	OEt	24	78
5	C_6H_5	*i*-Pr	OEt	24	75
6	C_6H_5	t-Bu	OEt	24	77
7	C_6H_5	C_6H_5	OEt	24	82
8	C_6H_5	4-ClC_6H_4	OEt	36	79
9	C_6H_5	C_6H_5	C_6H_5	24	73
10	C_6H_5	CH_3	CH3	24	70
11	4-CH_3 C_6H_4	CH_3	OEt	24	63
12	4-Cl C_6H_4	CH_3	OEt	28	72
13	4-F C_6H_4	CH_3	OEt	24	71
14	2-Cl C_6H_4	CH_3	OEt	28	64
15		CH_3	OEt	24	58
16		CH_3	OEt	48	0

[a]Reaction conditions: **a** (1.5 equiv., 1.5 mmol), **b** (1 equiv., 1 mmol), catalyst (0.1 mmol, 10 mmol%), 4 Å M.S.(0.1 g), I_2 (1.2 equiv., 1.2 mmol),solvent (5 mL).
[b]Isolated yield.

When toluene was used there was no reaction. This may be due to the catalyst having a low solubility in a non-polar solvent. If the addition of benzylamine was reduced to 1.1 equiv., it increased the yield (Table 1, entry 14) since the benzylamine can be oxidised by O_2.

Using the optimised conditions, we investigated the scope of this protocol between various 1,3-dicarbonyl compounds and different benzylamines. The experimental results are listed in Table 2. First, we examined the reaction with a series of 1,3-dicarbonyl compounds. When the ethyl acetoacetate was switched to methyl or *t*-butyl acetoacetate, a satisfactory yield was obtained (entries 1–3). This meant that steric effect and electronic effect had little influence on the reaction. Moreover, β-keto esters with different alkyl substituents can also react with benzylamine smoothly, furnishing the desired products in 75–82% yield (entries 4–8). When the reaction substrates were switched from β-keto esters to β-diketones, the reactions can be carried out smoothly to give the corresponding products with good yields (entries 9 and 10). Subsequently, different benzylamine derivatives were investigated as reaction substrates.

The results demonstrated that both electron-rich and electron-deficient benzylamines might be used. They react readily with β-keto esters to afford the oxazoles in 58–72% yield (entries 11–15). However, *iso*-butylamine failed to yield the desired product (entries 16).



Scheme 2

To explore the reaction process, the reaction of ethyl 2-iodo-3-oxobutanoate with benzyl amine was performed. It was found that the reaction can give the oxazole product with a yield of 65%, as shown in Scheme 2. When the reaction substrate was changed to substrate **c**,[20] no product was observed.

On the basis of the experimental results and the previous reports,[17] we proposed a mechanism, as shown in Scheme 3. First, **2** is formed from **1a** and **1b** in the presence of iodine. The oxidation of **2** and coordination of copper ions provides **3**, which undergoes an intramolecular cyclisation *via* an oxygen atom attacking the double bond giving the intermediate **4** in a tandem process.

Conclusions

In summary, we developed a facile and efficient oxidative synthesis of poly-substituted oxazoles from readily available starting materials. This transformation from benzylamine and β-diketone derivatives into oxazoles was achieved using a tandem oxidative cyclisation. In contrast to the traditional synthetic methods for oxazoles, this reaction avoided the use of hazardous materials. Therefore, this method is an attractive alternative to synthesise oxazoles. The mechanism and synthetic applications of this reaction are being studied further in our laboratory and the results will be reported in due course.

Experimental

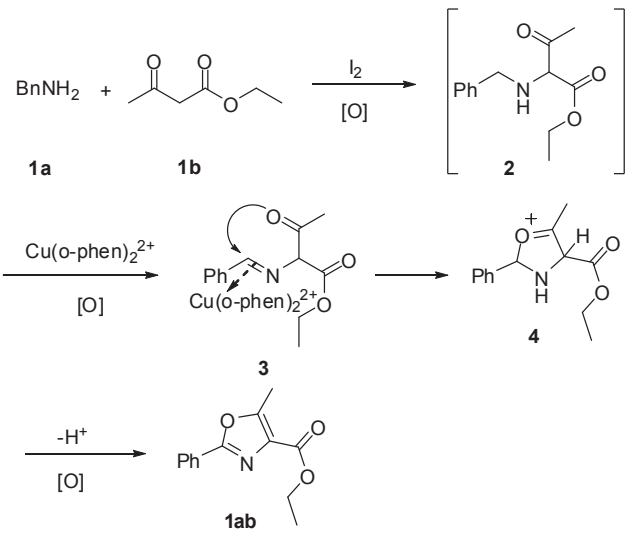
NMR spectra were performed on a Mercury 4N-PEG-300 (^{1}H: 300 MHz; ^{13}C: 75 MHz) spectrometer, using $CDCl_3$ as a solvent and TMS as the internal standard. IR spectra were recorded on Perkin-Elmer 2000 FTIR spectrometer. HRMS data were determined on a Bruker Daltonics APEXII 47e FT-ICR spectrometer. Melting points are uncorrected.

Synthesis of [Cu(o-phen)$_2$Cl]Cl [21]

The synthesis of [Cu(*o*-phen)$_2$Cl]Cl complex was carried out by addition of 1,10-phenatroline ligand (2 equiv.) in ethanol (10.0 mL) to $CuCl_2.2H_2O$ (1 equiv.) in ethanol (10.0 mL). The mixture was stirred for 1 h at 25 °C. The solvent was slowly evaporated at room temperature and the complex was collected and dried, yield: 91% as a green solid.

Typical procedure

Iodine (1.2 mmol), 1,3-dicarbonyl compounds (1 mmol), [Cu(*o*-phen)$_2$Cl]Cl (0.2 mmol), 4 Å M.S.(0.1 g) were successively added to a solution of benzylamine (1.5 mmol) derivatives in CH_3CN (5 mL).



Scheme 3 Possible mechanism for the formation of oxazoles.

The resulting solution was stirred at 60 °C for 24 h (the progress of the reaction was monitored by TLC). Then H_2O (5 mL) and EtOAc (15 mL) were added. The aqueous phase was extracted with EtOAc (3 × 15 mL). The combined organic phases were washed with brine, dried (anhyd. Na_2SO_4), filtered, and concentrated. The residue was purified by column chromatography to give ethyl 5-methyl-2-phenyloxazole-4-carboxylate (**1ab**); yield 182 mg (79%).

Ethyl 5-methyl-2-phenyloxazole-4-carboxylate: Colourless solid; m.p. 49–51 °C (lit.[3] 51–52 °C); IR (film)=2967, 1713, 1608, 1377, 1183, 1112, 712, 691 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.10–8.05 (m, 2H), 7.48–7.43 (m, 3H), 4.45 (q, *J*=7.2 Hz, 2H), 2.71 (s, 3H), 1.42 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 161.3, 158.5, 155.0, 129.6, 127.4, 127.2, 125.4, 125.3, 60.0, 14.2, 12.3; HRMS (ESI) calcd for $C_{13}H_{14}NO_3$ (M^++H): 232.0968; found: 232.0969; error: 0.4 ppm.

Methyl 5-methyl-2-phenyloxazole-4-carboxylate: Light yellow solid; m.p. 86–88 °C (lit.[6] 89–91 °C); IR (film)=2980, 1734, 1615, 1372, 1341, 1236, 1104, 783, 709, 691 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.10–8.05 (m, 2H), 7.50–7.45 (m, 3H), 3.94 (s, 3H), 2.70 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 161.5, 160.1, 158.6, 131.5, 127.7, 127.3, 125.7, 125.6, 61.3, 28.2, 21.2, 14.2, 13.5; HRMS (ESI) calcd for $C_{12}H_{12}NO_3$ (M^++H): 218.0812; found: 218.0812; error: 0 ppm.

tert-*Butyl 5-methyl-2-phenyloxazole-4-carboxylate*: Light yellow solid; m.p. 67–69 °C (lit.[17] 72–73 °C); IR (film)=2977, 1730, 1710, 1369, 1166, 1111, 710, 497 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.08–8.02 (m, 2 H),7.45–7.41 (m, 3 H), 2.66 (s, 3 H), 1.61 (s, 9 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 160.5, 158.4, 154.9, 131.0, 129.1, 129.3, 125.5, 125.3, 28.7, 12.2; HRMS (ESI) calcd for $C_{15}H_{18}NO_3$ (M^++H): 260.1281; found: 260.1283; error: 0.7 ppm.

Ethyl 2-phenyl-5-propyloxazole-4-carboxylate: Light yellow solid; m.p. 51–53 °C (lit.[17] 53–54 °C); IR (film)=2980, 1715, 1611, 1363, 1332, 1229, 1099, 775, 701, 682 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.12–8.01 (m, 2H), 7.48–7.42 (m, 3H), 4.41 (q, *J*=7.2 Hz, 2H), 3.08 (t, *J*=7.5 Hz, 2H), 1.84–1.72 (m, 2H), 1.41 (t, *J*=7.2 Hz, 3H), 1.2 (t, *J*=7.5 Hz, 3H).; ^{13}C NMR (75 MHz, $CDCl_3$): δ 160.5, 157.7, 157.5, 128.6, 126.4, 126.0, 124.2, 124.4, 59.1, 26.2, 19.9, 13.4, 12.7; HRMS (ESI) calcd for $C_{15}H_{18}NO_3$ (M^++H): 260.1281; found: 260.1281; error: 0 ppm.

Ethyl 5-iso-*propyl-2-phenyloxazole-4-carboxylate*: Light yellow solid; m.p. 56–58 °C (lit.[18] 57–58 °C); IR (film)=2972, 1718, 1605, 1438, 1363, 1239, 1053, 693 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.10–8.02 (m, 2 H), 7.50–7.40 (m, 3 H), 4.32 (q, *J*=7.2 Hz, 3 H), 3.89–3.76 (m, 1 H), 1.38 (t, *J*=7.2 Hz, 3 H), 1.35 (d, *J*=7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 160.1, 158.3, 155.1, 126.2, 124.3, 122.1, 122.5, 122.1, 57.9, 24.2, 19.6, 12.3; HRMS (ESI) calcd for $C_{15}H_{18}NO_3$ (M^++H): 260.1281; found: 260.1281; error: 0 ppm.

Ethyl 5-tert-*butyl-2-phenyloxazole-4-carboxylate*: Light yellow solid; m.p. 70–73 °C (lit.[17] 75–76 °C); IR (film)=2964, 1707, 1606, 1374, 1333, 1229, 1092, 707, 681 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.06–8.02 (m, 2H), 7.48–7.43 (m,3H), 4.40 (q, *J*=7.2 Hz, 2H), 1.51 (s, 9H), 1.45–1.39 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 164.7, 160.4, 155.7, 128.9, 126.8, 126.3, 124.7, 124.5, 59.0, 32.4, 26.0; HRMS (ESI) calcd for $C_{16}H_{20}NO_3$ (M^++H): 274.1438; found: 274.1441; error: 1.1 ppm.

Ethyl 2,5-diphenyloxazole-4-carboxylate: Light yellow solid; m.p. 84–86 °C (lit.[5] 86–87 °C); IR (film)=3060, 2979, 1718, 1489, 1317, 1214, 1092, 707, 386 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.19–8.04 (m, 4H),7.55–7.45 (m, 6H), 4.45 (q, *J*=7.2 Hz, 2H), 1.43 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 162.4, 160.1, 155.8, 132.9, 131.8, 131.9, 130.2, 129.1, 128.9, 127.8, 127.0, 125.9, 60.0, 15.8; HRMS (ESI) calcd for $C_{18}H_{16}NO_3$ (M^++H): 294.1125; found: 294.1125; error: 0 ppm.

Ethyl 5-(4-chlorophenyl)-2-phenyloxazole-4-carboxylate: White solid; m.p. 86–88 °C (lit.[12] 110–111 °C); IR (film)=2983, 1718, 14869, 1214, 1092, 753, 712 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.17–8.06 (m, 4H),7.53–7.41 (m, 5H), 4.46 (q, *J*=7.2 Hz, 2H), 1.43 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 162.2, 159.9, 153.9, 136.3, 131.2, 129.8, 128.8, 128.7, 128.6, 126.9, 126.2, 125.6, 61.6, 14.3; HRMS (ESI) calcd for $C_{18}H_{15}ClNO_3$ (M^++H): 328.0735; found: 328.0734; error: 0.3 ppm.

(2,5-Diphenyloxazol-4-yl)(phenyl) methanone: Light yellow solid; m.p. 82–84 °C (lit.[17] 79–80 °C); IR (film)=3041, 2904, 1638, 1536, 1464, 1339, 1202, 874, 691, 673 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.23–8.16 (m, 3H), 8.15–8.04 (m, 2H), 8.02–7.94 (m, 1H),7.66–7.54 (m, 1H), 7.52–7.40 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 187.7, 158.1, 153.5, 136.4, 132.2, 130.0, 129.5, 129.4, 128.6, 128.1, 127.3, 127.2, 126.8, 126.4, 125.7, 125.1; HRMS (ESI) calcd for $C_{22}H_{16}NO_2$ (M^++H): 326.1176; found: 326.1176; error: 0 ppm.

1-(5-Methyl-2-phenyloxazol-4-yl) ethanone: Colourless solid; m.p. 77–78 °C (lit.[17], 78–79 °C); IR (film)=2963, 1619, 1380, 1078, 693, 674 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.05–7.99 (m, 2H), 7.49–7.43 (m, 3H),2.69 (s, 3H), 2.60 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 196.0, 159.3, 155.0, 130.9, 129.4, 127.3, 126.9, 28.5, 12.9; HRMS (ESI) calcd for $C_{12}H_{12}NO_2$ (M^++H): 202.0863; found: 202.0863; error: 0 ppm.

Ethyl 5-methyl-2-p-*tolyloxazole-4-carboxylate*: Yellow solid; m.p. 65–67 °C (lit.[17] 67–68 °C); IR (film)=2988, 1712, 1612, 1486, 1192, 1007, 821, 719 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 7.95 (d, *J*=7.5 Hz, 2H), 7.28–7.22 (m, 2H), 4.43 (q, *J*=7.2 Hz, 2H), 2.68 (s, 3H), 2.38 (s, 3H), 1.40 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 162.0, 159.3, 155.3, 140.1, 128.9, 127.7, 127.6, 125.9, 60.4, 21.1, 13.9, 11.9; HRMS (ESI) calcd for $C_{14}H_{16}NO_3$ (M^++H): 246.1125; found: 246.1126; error: 0.4 ppm.

Ethyl 2-(4-chlorophenyl)-5-methyloxazole-4-carboxylate: Light yellow solid; m.p. 80–82 °C (lit.[18] 68–69 °C); IR (film)=2980, 1712, 1609, 1480, 1183, 1104, 836, 728 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.00 (d, *J*=7.2 Hz,2H), 7.38 (d, *J*=7.2 Hz, 2H), 4.42 (q, *J*=7.2 Hz, 2H), 2.71 (s, 3H), 1.43 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 161.4, 157.9, 155.5, 135.9, 128.7, 127.7, 126.9, 124.2, 60.2, 13.5, 12.3; HRMS (ESI) calcd for $C_{13}H_{13}ClNO_3$ (M^++H): 266.0578; found: 266.0578; error: 0 ppm.

Ethyl 2-(4-fluorophenyl)-5-methyloxazole-4-carboxylate: Yellow solid; m.p. 70–72 °C (lit.[17] 72–73 °C); IR (film)=3012, 2986, 1709, 1622, 1500, 1199, 820, 733 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 8.08–8.01 (m, 2H), 7.15–7.05 (m, 2H), 4.41 (q, *J*=7.2 Hz, 2H), 2.67 (s, 3H), 1.40 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.8, 162.5, 162.0, 157.8, 155.3, 135.9, 128.8, 128.7, 127.9, 127.7, 115.1, 114.8, 60.3, 60.2, 14.2, 12.1; HRMS (ESI) calcd for $C_{13}H_{13}FNO_3$ (M^++H): 250.0874; found: 250.0874; error: 0 ppm.

Ethyl 2-(2-chlorophenyl)-5-methyloxazole-4-carboxylate: Yellow solid; m.p. 63–65 °C (lit.[17] 65–66 °C); IR (film)=2979, 1732, 1614, 1433, 1227, 1109, 1078, 1030, 758 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$):δ 8.02–7.96 (m, 1H), 7.50–7.45 (m, 1H), 7.43–7.32 (m, 2H), 4.42 (q, *J*=7.2 Hz, 2H), 2.72 (s, 3H), 1.41 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 157.0, 155.9, 131.8, 130.6, 130.4, 130.0, 126.5, 125.8, 125.0, 60.0, 13.7, 12.1; HRMS (ESI) calcd for $C_{13}H_{13}ClNO_3$ (M^++H): 266.0578; found: 266.0576; error: 0.8 ppm.

Ethyl 2-(furan-2-yl)-5-methyloxazole-4-carboxylate: Light yellow solid; m.p. 76–78 °C (lit.[17] 75–76 °C); IR (film)=3111, 2923, 1709, 1607, 1372, 1106, 768 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.57–7.53 (m, 1H), 7.11–7.06 (m, 1H), 6.56–6.50 (m, 1H), 4.40 (q, *J*=7.2 Hz, 2H), 2.68 (s, 3H), 1.40 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 161.8, 155.0, 151.6, 143.9, 141.4, 127.9, 111.5, 111.1, 60.5, 13.7, 11.7; HRMS (ESI) calcd for $C_{11}H_{12}NO_4$ (M^++H): 222.0761; found: 222.0761; error: 0 ppm.

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