Preparation of 2-(1-Chloroalkyl)-4,5-dimethyloxazoles and (*E*)-2-Alkenyl-4,5-dimethyloxazoles

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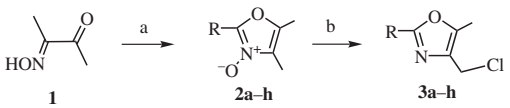
Abstract: 2-(1-Chloroalkyl)-4,5-dimethyloxazoles were prepared in 84–91% yields by simultaneous reduction and regioselective chlorination of the corresponding unstable *N*-oxides with $POCl_3$ or $SOCl_2$. Dehydrochlorination of 2-(1-chloroalkyl)-4,5-dimethyloxazoles with potassium hydroxide afforded (*E*)-2-alkenyl-4,5-dimethyloxazoles in 71–90% yields.

Key words: oxazole, chlorination, regioselectivity, dehydrochlorination

Oxazoles have attracted great interest due to their occurrence as subunits of various biologically active natural products and some drug molecules, as well as their applications as valuable precursors in many useful synthetic transformations.[1,2] There are some synthetic methods for 2,4,5-trisubstituted oxazoles,[3,4] but there is no report on the synthesis of 2-(1-chloroalkyl)-4,5-dimethyloxazoles **5** and (*E*)-2-alkenyl-4,5-dimethyloxazoles **6**. Here, we report a practical and convenient synthesis of **5** and **6**.

According to the procedure reported,[5,6] several 4-chloromethyl-2-aryl-5-methyloxazoles **3** were prepared from the *N*-oxides **2** with $POCl_3$ or $SOCl_2$ in chloroform at reflux (Scheme 1, Table 1). Compounds **2** could be obtained by condensation of aromatic aldehyde and 2,3-butanedione monooxime **1** in acetic acid saturated by anhydrous hydrochloride gas.[5,6]



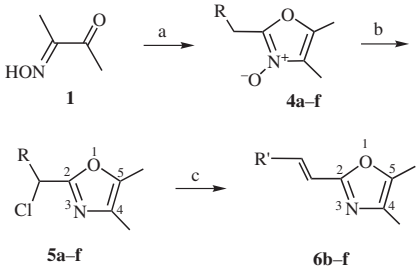
Scheme 1 *Reagents and conditions*: (a) anhyd HCl (g), ArCHO, AcOH; (b) $POCl_3$ (or $SOCl_2$), $CHCl_3$, reflux.

While attempting to prepare 2-alkyl-4-chloromethyl-5-methyloxazoles following the above-mentioned procedure, we observed the chlorination of the corresponding *N*-oxides by $POCl_3$ or $SOCl_2$ at 2-methylene rather than 4-methyl (Scheme 2) position. This phenomenon may be due to the activity difference between 2-methylene and 4-methyl (Scheme 2) groups. To investigate the scope and efficiency of the reaction, compounds **5a–f** were synthesized. Dehydrochlorination of **5b–f** with potassium hydroxide afforded **6b–f** (Table 2). The structure of **5b** was determined by 2D NMR experiments (Figure 1).

Table 1 Synthesis of **3a–h**

R	Products	Isolated yield (%)[a]
Phenyl	**3a**	94 (91)
4-Methoxyphenyl	**3b**	88 (85)
3,4-Dimethoxyphenyl	**3c**	86 (83)
3,4,5-Trimethoxyphenyl	**3d**	83 (80)
4-Chlorophenyl	**3e**	89 (86)
4-Bromophenyl	**3f**	79 (78)
4-Nitrophenyl	**3g**	91 (89)
Furan-2-yl	**3h**	85 (84)

[a] The numbers in parentheses are the yields obtained when $SOCl_2$ was used.



Scheme 2 *Reagents and conditions*: (a) anhyd HCl (g), RCH_2CHO, AcOH; (b) $POCl_3$ or $SOCl_2$, $CHCl_3$, reflux; (c) KOH, C_2H_5OH, reflux.

In conclusion, a simple and efficient procedure to synthesize 2-(1-chloroalkyl)-4,5-dimethyloxazoles and (*E*)-2-alkenyl-4,5-dimethyloxazoles was developed.

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Table 2 Synthesis of **5a–f** and **6b–f**

Y		Isolated yield (%)[a]
Chlorophenylmethyl	**5a**	88 (86)
1-Chloro-2-phenylethyl	**5b**	91 (88)
1-Chloropropyl	**5c**	87 (81)
2-Chloropropan-2-yl	**5d**	90
1-Chloropentyl	**5e**	87
1-Chlorooctyl	**5f**	84
Styryl	**6b**	90
Propenyl	**6c**	73
Isopropenyl	**6d**	71
Pentenyl	**6e**	80
Octenyl	**6f**	87

[a] The numbers in parentheses are the yields obtained when $SOCl_2$ was used.

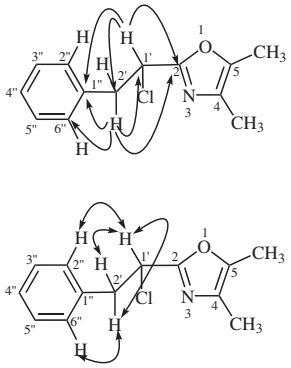

Figure 1 Major HMBC (→) and NOESY (↔) correlations in compound **5b**.

All starting compounds were used as received from commercial sources without further purification. Petroleum ether (PE) used had the boiling range 60–90 °C. Melting points were determined on a XRC-1 micromelting point apparatus and are uncorrected. Column chromatography was carried out on silica gel (200–300 mesh, Qingdao Haiyang Chemical Co. Ltd.). NMR spectra were recorded on a Bruker Avance 600 spectrometer with TMS as internal standard. MS spectra measurements were carried out on a Finnigan LCQ^{DECA} mass spectrometer (ESI–MS) and a BioTOF-Q mass spectrometer (HR–ESI–MS). IR spectra were recorded on a Perkin-Elmer spectrum one FT-IR spectrometer (KBr disc).

Synthesis of 3a–h and 5a–f; General Procedure

A solution of 2,3-butanedione monooxime[7] (0.40 g, 5.0 mmol) and aldehyde (5.3 mmol) in AcOH (20 mL) was cooled to 0–5 °C. Anhyd HCl (g) was bubbled into the reaction mixture for 0.5 h. The mixture was stirred at the same temperature for 0.5 h and diluted with Et_2O (40 mL). The precipitate was filtered and washed with Et_2O (2 × 10 mL), then dissolved in H_2O (20 mL) and concd NH_4OH (20 mL). This solution was extracted with $CHCl_3$. The combined organic layer was washed with H_2O and brine, and dried over $MgSO_4$. The solvent was removed in vacuo to give the unstable *N*-oxides. To a solution of the unstable *N*-oxides (4 mmol) in $CHCl_3$ (20 mL) was added dropwise $POCl_3$ or $SOCl_2$ (0.4 mL, 5 mmol). The mixture was stirred at reflux for 0.5 h, then cooled to r.t., and washed with H_2O (3 × 20 mL). The combined aqueous layer was re-extracted with $CHCl_3$ (2 × 30 mL). The combined organic layer was dried over $MgSO_4$, and the solvent was removed in vacuo to give the crude products, which were purified by column chromatography or by recrystallization.

Synthesis of 6b–f; General Procedure

The mixture of **5b–f** (2.0 mmol) in anhyd EtOH (20 mL) and anhyd KOH (2.2 mmol) was stirred at reflux for 2 h and then cooled to r.t. The solvent was removed in vacuo to give the residue, which was washed with Et_2O (3 × 20 mL). The combined organic layer was washed with H_2O and dried over $MgSO_4$. Then the solvent was removed in vacuo to give the crude products, which were purified by column chromatography.

4-Chloromethyl-5-methyl-2-phenyloxazole (3a)

Yield: 0.78 g (94%); colorless columnar crystals [PE–acetone, 30:1]; mp 77.8–78.3 °C.

IR (KBr): 2924, 1633, 1558, 1490, 1448, 1259, 1112, 1063, 774, 700 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.43 (s, 3 H), 4.56 (s, 2 H), 7.26 (t, *J* = 7.8 Hz, 1 H), 7.30 (t, *J* = 7.2 Hz, 2 H), 7.42 (d, *J* = 7.5 Hz, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 160.1, 146.6, 132.9, 130.3, 128.7, 127.2, 126.2, 37.3, 10.4.

ESI–MS (+ve mode): *m*/*z* (%) = 230 (82) $[M + Na]^+$, 246 (15) $[M + K]^+$.

HRMS (ESI, +ve mode): *m*/*z* $[M + H]^+$ calcd for $C_{11}H_{11}ClNO$: 208.0524; found: 208.0521.

4-Choromethyl-2-(4-methoxyphenyl)-5-methyloxazole (3b)

Yield: 0.83 g (88%); colorless cubic crystals [PE–acetone, 30:1]; mp 83.9–84.2 °C.

IR (KBr): 2964, 1617, 1590, 1499, 1447, 1251, 1165, 1024, 832, 740, 698 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.40 (s, 3 H), 3.85 (s, 3 H), 4.54 (s, 2 H), 6.94 (d, *J* = 8.8 Hz, 2 H), 7.93 (d, *J* = 8.6 Hz, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 161.2, 160.2, 145.9, 132.6, 127.8, 120.1, 114.1, 55.4, 37.4, 10.3.

ESI–MS (+ve mode): *m*/*z* (%) = 238 (70) $[M + H]^+$, 260 (10) $[M + Na]^+$.

HRMS (ESI, +ve mode): *m*/*z* $[M + H]^+$ calcd for $C_{12}H_{13}ClNO_2$: 238.0635; found: 238.0629.

4-Chloromethyl-2-(3,4-dimethoxyphenyl)-5-methyloxazole (3c)

Yield: 0.92 g (86%); colorless needles [PE–acetone, 20:1]; mp 113.8–114.3 °C.

IR (KBr): 2962, 1607, 1568, 1513, 1463, 1251, 1138, 1023, 858, 770, 714 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.42 (s, 3 H), 3.93 (s, 3 H), 3.97 (s, 3 H), 4.54 (s, 2 H), 6.91 (d, *J* = 8.3 Hz, 1 H), 7.52 (d, *J* = 1.7 Hz, 1 H), 7.57 (dd, *J* = 1.7, 8.3 Hz, 1 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 160.1, 150.9, 149.1, 146.1, 132.6, 120.2, 119.4, 111.0, 109.0, 56.0, 37.4, 10.4.

ESI–MS (+ve mode): *m/z* (%) = 268 (71) $[M + H]^+$, 290 (60) $[M + Na]^+$.

HR–MS (ESI, +ve mode): *m/z* $[M + H]^+$ calcd for $C_{13}H_{15}ClNO_3$: 268.0739; found: 268.0735.

4-Chloromethyl-2-(3,4,5-trimethoxyphenyl)-5-methyloxazole (3d)
Yield: 0.98 g (83%); colorless cubic crystals [PE–acetone, 15:1]; mp 113.9–114.6 °C.

IR (KBr): 2924, 1630, 1560, 1502, 1418, 1241, 1127, 1004, 747, 779, 714 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.44 (s, 3 H), 3.90 (s, 3 H), 3.94 (s, 6 H), 4.56 (s, 2 H), 7.25 (s, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 159.9, 153.5, 146.5, 140.0, 132.9, 122.6, 103.4, 61.0, 56.3, 37.3, 10.4.

ESI–MS (+ve mode): *m/z* (%) = 298 (84) $[M + H]^+$, 320 (72) $[M + Na]^+$, 337 (15) $[M + K]^+$.

HRMS (ESI, +ve mode): *m/z* $[M + H]^+$ calcd for $C_{14}H_{17}ClNO_4$: 298.0841; found: 298.0834.

4-Chloromethyl-2-(4-chlorophenyl)-5-methyloxazole (3e)
Yield: 0.86 g (89%); colorless cubic crystals [PE–acetone, 30:1]; mp 92.3–92.9 °C.

IR (KBr): 2962, 1634, 1605, 1549, 1485, 1260, 1118, 1090, 834, 716, 691 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.42 (s, 3 H), 4.54 (s, 2 H), 7.41 (d, *J* = 8.5 Hz, 2 H), 7.93 (d, *J* = 8.5 Hz, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 159.1, 146.8, 136.4, 133.1, 129.0, 127.4, 125.7, 37.1, 10.4.

ESI–MS (+ve mode): *m/z* (%) = 243 (74) $[M + H]^+$, 265 (20) $[M + Na]^+$.

HRMS (ESI, +ve mode): *m/z* $[M + H]^+$ calcd for $C_{11}H_{10}Cl_2NO$: 243.0134; found: 243.0125.

2-(4-Bromophenyl)-4-chloromethyl-5-methyloxazole (3f)
Yield: 0.91 g (79%); colorless cubic crystals [PE–acetone, 20:1]; mp 57–58 °C.

IR (KBr): 2970, 1633, 1599, 1481, 1402, 1258, 1117, 1006, 843, 718, 700 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.44 (s, 3 H), 4.53 (s, 2 H), 7.57 (d, *J* = 8.5 Hz, 2 H), 7.87 (d, *J* = 8.5 Hz, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 159.3, 146.6, 133.2, 132.0, 127.8, 126.1, 124.7, 37.4, 10.7.

ESI–MS (+ve mode): *m/z* (%) = 286 (80) $[M + H]^+$, 308 (24) $[M + Na]^+$.

4-Chloromethyl-5-methyl-2-(4-nitrophenyl)oxazole (3g)
Yield: 0.92 g (91%); yellow columnar crystals [PE–acetone, 25:1]; mp 107.4–107.9 °C.

IR (KBr): 3088, 2975, 2925, 1629, 1603, 1561, 1520, 1485, 1353, 1333, 1108, 1063, 864, 852, 710 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.48 (s, 3 H), 4.57 (s, 2 H), 8.18 (d, *J* = 8.6 Hz, 2 H), 8.31 (d, *J* = 8.6 Hz, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 157.9, 148.6, 148.3, 134.1, 132.6, 126.9, 124.2, 36.8, 10.5.

ESI–MS (+ve mode): *m/z* (%) = 253 (85) $[M + H]^+$.

HRMS (ESI, +ve mode): *m/z* $[M + H]^+$ calcd for $C_{11}H_{10}ClN_2O_3$: 253.0312; found: 253.0316.

4-Chloromethyl-2-(furan-2-yl)-5-methyloxazole (3h)
Yield: 0.67 g (85%); colorless needles [PE–acetone, 40:1]; mp 43–45 °C.

IR (KBr): 3106, 2970, 1634, 1620, 1546, 1495, 1436, 1305, 1264, 1157, 1025, 954, 893, 764, 774, 747, 706 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.42 (s, 3 H), 4.53 (s, 2 H), 6.53 (dd, *J* = 1.7, 3.4 Hz, 1 H), 6.98 (d, *J* = 3.4 Hz, 1 H), 7.54 (t, *J* = 0.8 Hz, 1 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 152.7, 146.1, 144.3, 142.6, 132.7, 111.8, 111.2, 37.0, 10.3.

ESI–MS (+ve mode): *m/z* (%) = 220 (75) $[M + Na]^+$, 162 (79) $[M – Cl]^+$.

HRMS (ESI, +ve mode): *m/z* $[M + Na]^+$ calcd for $C_9H_8ClNO_2Na$: 220.0138; found: 220.0136.

2-(Phenylchloromethyl)-4,5-dimethyloxazole (5a)
Yield: 0.78 g (88%); colorless oil.

IR (KBr): 2928, 2852, 1625, 1559, 1490, 1441, 1254, 1110, 1030, 770, 690 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.36 (s, 3 H), 2.38 (s, 3 H), 6.21 (s, 1 H), 7.28 (d, *J* = 7.8 Hz, 2 H), 7.30 (t, *J* = 7.3 Hz, 1 H), 7.33 (t, *J* = 7.6 Hz, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 159.4, 138.7, 136.2, 129.7, 128.3, 127.6, 126.2, 62.8, 12.1, 10.3.

HRMS (ESI, +ve mode): *m/z* $[M + H]^+$ calcd for $C_{12}H_{13}ClNO$: 222.0618; found: 222.0612.

2-(1-Chloro-2-phenylethyl)-4,5-dimethyloxazole (5b)
Yield: 0.86 g (91%); yellow oil.

IR (KBr): 2965, 1630, 1551, 1497, 1443, 1260, 1110, 1060, 771, 710 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.06 (s, 3 H), 2.34 (s, 3 H), 3.39 (dd, *J* = 7.1, 14.1 Hz, 1 H), 3.58 (dd, *J* = 8.2, 14.1 Hz, 1 H), 5.05 (t, *J* = 7.6 Hz, 1 H), 7.20 (d, *J* = 7.4 Hz, 2 H), 7.24 (t, *J* = 7.1 Hz, 1 H), 7.28 (t, *J* = 7.5 Hz, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 158.4, 144.4, 136.5, 131.2, 129.3, 128.6, 127.2, 53.8, 42.2, 11.1, 10.0.

HRMS (ESI, +ve mode): *m/z* $[M + H]^+$ calcd for $C_{13}H_{15}ClNO$: 236.0837; found: 236.0831.

2-(1-Chloropentyl)-4,5-dimethyloxazole (5c)
Yield: 0.60 g (87%); colorless oil.

IR (KBr): 2967, 2862, 1667, 1455, 1384 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 1.05 (t, *J* = 7.3 Hz, 3 H), 2.04 (s, 3 H), 2.18 (s, 3 H), 2.20 (m, 2 H), 4.79 (t, *J* = 7.1 Hz, 1 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 159.2, 144.2, 130.9, 55.0, 37.3, 13.7, 10.1, 9.9.

HRMS (ESI, +ve mode): *m/z* $[M + H]^+$ calcd for $C_8H_{13}ClNO$: 174.0686; found: 174.0683.

2-(2-Chloropropan-2-yl)-4,5-dimethyloxazole (5d)
Yield: 0.64 g (90%), colorless oil.

IR (KBr): 2957, 2868, 1661, 1445, 1380 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 1.98 (s, 6 H), 2.20 (s, 3 H), 2.39 (s, 3 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 167.4, 145.8, 130.8, 58.2, 29.3, 10.1.

HRMS (ESI, +ve mode): *m/z* $[M + H]^+$ calcd for $C_8H_{13}ClNO$: 174.0686; found: 174.0683.

2-(1-Chloropentyl)-4,5-dimethyloxazole (5e)
Yield: 0.70 g (87%); colorless oil.

IR (KBr): 2959, 2874, 1645, 1455, 1196 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 0.92 (t, *J* = 7.1 Hz, 3 H), 1.24–1.56 (m, 6 H), 2.08 (s, 3 H), 2.27 (s, 3 H), 4.86 (t, *J* = 5.1 Hz, 1 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 159.4, 144.3, 130.9, 53.5, 35.8, 28.6, 21.9, 13.9, 11.0, 10.0.

HRMS (ESI, +ve mode): *m*/*z* $[M + H]^+$ calcd for $C_{10}H_{17}ClNO$: 202.1017; found: 202.1015.

2-(1-Chlorooctyl)-4,5-dimethyloxazole (5f)
Yield: 0.82 g (84%); colorless oil.

IR (KBr): 2982, 2857, 1669, 1450, 1378 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 0.89 (t, *J* = 6.7 Hz, 3 H), 1.20–1.54 (m, 12 H), 2.08 (s, 3 H), 2.26 (s, 3 H), 4.85 (t, *J* = 5.1 Hz, 1 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 160.4, 144.8, 131.0, 53.5, 38.3, 31.6, 29.0, 28.8, 25.2, 23.6, 14.3, 10.1, 9.8.

HRMS (ESI, +ve mode): *m*/*z* $[M + H]^+$ calcd for $C_{13}H_{23}ClNO$: 244.1466; found: 244.1461.

(*E*)-4,5-Dimethyl-2-styryloxazole (6b)
Yield: 0.36 g (90%); white solid.

IR (KBr): 3025, 2963, 16370, 1550, 1493, 1440, 1260, 1110, 1060 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 2.08 (s, 3 H), 2.26 (s, 3 H), 6.90 (d, *J* = 16.2 Hz, 1 H), 6.98 (d, *J* = 16.2 Hz, 1 H), 7.18 (t, *J* = 7.6 Hz, 2 H), 7.28 (t, *J* = 7.3 Hz, 1 H), 7.37 (d, *J* = 7.8 Hz, 2 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 155.1, 139.6, 136.2, 133.8, 128.8, 127.8, 126.9, 124.9, 124.1, 11.3, 10.7.

HRMS (ESI, +ve mode): *m*/*z* $[M + H]^+$ calcd for $C_{13}H_{14}NO$: 200.1062; found: 200.1068.

(*E*)-4,5-Dimethyl-2-propenyloxazole (6c)
Yield: 0.20 g (73%); light-yellow oil.

IR (KBr): 3052, 2980, 2831, 16651, 1441, 1380 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 1.78 (d, *J* = 15.6 Hz, 3 H), 2.12 (s, 3 H), 2.41 (s, 3 H), 6.10 (dq, *J* = 6.9, 16.2 Hz, 1 H), 6.42 (d, *J* = 16.2 Hz, 1 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 158.8, 142.3, 131.3, 131.0, 130.0, 15.1, 9.8.

HRMS (ESI, +ve mode): *m*/*z* $[M + H]^+$ calcd for $C_8H_{12}NO$: 138.0934; found: 138.0975.

4,5-Dimethyl-2-isopropenyloxazole (6d)
Yield: 0.19 g (71%); light-yellow oil.

IR (KBr): 3075, 2928, 2830, 1668, 1440, 1375, 1118 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 1.76 (s, 3 H), 2.10 (s, 3 H), 2.31 (s, 3 H), 4.93 (d, *J* = 7.1 Hz, 1 H), 5.31 (d, *J* = 7.1 Hz, 1 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 156.8, 145.8, 139.6, 126.7, 119.3, 22.5, 11.7, 10.1.

HRMS (ESI, +ve mode): *m*/*z*: $[M + H]^+$ calcd for $C_8H_{12}NO$: 138.0934; found: 138.0906.

(*E*)-4,5-Dimethyl-2-pentenyloxazole (6e)
Yield: 0.26 g (80%); yellow oil.

IR (KBr): 3028, 2962, 2853, 1660, 1439, 1105 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 0.93 (t, *J* = 6.0 Hz, 3 H), 1.42 (m, 2 H), 1.90 (m, 2 H), 2.15 (s, 3 H), 2.39 (s, 3 H), 6.12 (dt, *J* = 7.1, 15.9 Hz, 1 H), 6.46 (d, *J* = 15.9 Hz, 1 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 159.8, 138.1, 131.5, 130.0, 126.7, 36.2, 21.5, 14.1, 9.9.

HRMS (ESI, +ve mode): *m*/*z* $[M + H]^+$ calcd for $C_{10}H_{16}NO$: 166.1213; found: 166.1216.

(*E*)-4,5-Dimethyl-2-octenyloxazole (6f)
Yield: 0.36 g (87%); yellow oil.

IR (KBr): 3034, 2958, 2872, 1644, 1455, 1094 cm^{-1}.

^1H NMR (600 MHz, $CDCl_3$): δ = 0.89 (t, *J* = 6.2 Hz, 3 H), 1.29–1.98 (m, 10 H), 2.10 (s, 3 H), 2.31 (s, 3 H), 6.12 (dt, *J* = 7.2, 16.1 Hz, 1 H), 6.58 (d, *J* = 16.1 Hz, 1 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 156.3, 139.3, 130.6, 126.1, 123.8, 35.6, 30.7, 29.6, 29.0, 23.5, 15.1, 10.0, 9.6.

HRMS (ESI, +ve mode): *m*/*z* $[M + H]^+$ calcd for $C_{13}H_{22}NO$: 208.1712; found: 208.1702.

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