



SUPPLEMENTARY MATERIAL TO
**One-pot green synthesis of isoxazol-5(4H)-one derivatives
using Dowex1-x8OH in water**

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CHARACTERIZATION DATA FOR COMPOUNDS **4a–i**

(Z)-4-Benzylidene-3-methylisoxazol-5(4H)-one (**4a**). Yellow crystals; m.p.: 140–142 °C (Lit.: 142–144 °C¹); FT-IR (KBr, cm⁻¹): 1732 (C=O), 1620, 1100, 1216, 879, 763; ¹H-NMR (300 MHz, CDCl₃, δ / ppm): 2.31 (3H, s, CH₃), 7.44 (1H, s, Ar-CH=), 7.49–7.59 (3H, m, Ar-H), 8.35 (2H, dd, J = 1.3 & 7.4 Hz, Ar-H); ¹³C-NMR (75 MHz, CDCl₃, δ / ppm): 11.63 (CH₃), 119.65 (C= isoxazolone ring), 129.03 (Ar-C), 130.47 (Ar-C), 132.29 (Ar-C), 134.01 (Ar-C), 149.98 (Ar-CH=), 161.16 (C=N), 167.88 (C=O).

(Z)-4-(4-Methoxybenzylidene)-3-methylisoxazol-5(4H)-one (**4b**). Yellow crystals; m.p.: 177–179 °C (Lit.: 177–178 °C¹); FT-IR (KBr, cm⁻¹): 1730 (C=O), 1590, 1267, 1018, 875, 775; ¹H-NMR (300 MHz, CDCl₃, δ / ppm): 2.28 (3H, s, CH₃), 3.92 (3H, s, OCH₃), 7.34 (1H, s, Ar-CH=), 7.00 (2H, d, J = 8.7 Hz, Ar-H), 8.44 (2H, d, J = 8.7 Hz, Ar-H); ¹³C-NMR (75 MHz, CDCl₃, δ / ppm): 11.63 (CH₃), 55.70 (OCH₃), 114.64 (C= isoxazolone ring), 116.31 (Ar-C), 125.82 (Ar-C), 136.96 (Ar-C), 149.35 (Ar-CH=), 161.29 (Ar-O), 164.60 (C=N), 168.77 (C=O).

(Z)-4-(2-Methoxybenzylidene)-3-methylisoxazol-5(4H)-one (**4c**). Yellow crystals; m.p.: 158–160 °C (Lit.: 159–160 °C²); FT-IR (KBr, cm⁻¹): 1732 (C=O), 1590, 1256, 1103, 887, 765; ¹H-NMR (300 MHz, CDCl₃, δ / ppm): 2.31 (3H, s, CH₃), 3.95 (3H, s, OCH₃), 6.96 (1H, d, J = 8.4 Hz, Ar-H), 7.09 (1H, t, J = 7.8 Hz, Ar-H), 7.56 (1H, t, J = 7.05 Hz, Ar-H), 8.06 (1H, s, Ar-CH=), 8.92 (1H, d, J = 8.1 Hz, Ar-H); ¹³C-NMR (75 MHz, CDCl₃, δ / ppm): 11.67 (CH₃), 55.47 (OCH₃), 110.70 (C= isoxazolone ring), 118.32 (Ar-C), 120.84 (Ar-C), 121.20 (Ar-C), 133.37 (Ar-C), 136.27 (Ar-C), 143.98 (Ar-CH=), 159.82 (C=N), 161.52 (C=O).

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(*Z*)-4-(3-Bromobenzylidene)-3-methylisoxazol-5(4H)-one (**4d**). Yellow crystals; m.p.: 141–143 °C (Lit.: 141–143 °C²); FT-IR (KBr, cm⁻¹): 1729 (C=O), 1544, 1217, 1123, 871, 775; ¹H-NMR (300 MHz, CDCl₃, δ / ppm): 2.31 (3H, *s*, CH₃), 7.35 (1H, *s*, Ar-CH=), 7.41 (1H, *t*, *J* = 8.1 Hz, Ar-H), 7.71 (1H, *d*, *J* = 7.80 Hz, Ar-H), 8.34 (1H, *d*, *J* = 7.8 Hz, Ar-H), 8.46 (1H, *s*, Ar-H); ¹³C-NMR (75 MHz, CDCl₃, δ / ppm): 11.60 (CH₃), 121.12 (Ar-Br), 122.89 (C= isoxazolone ring), 130.48 (Ar-C), 131.90 (Ar-C), 133.87 (Ar-C), 136.03 (Ar-C), 136.50 (Ar-C), 147.71 (Ar-CH=), 160.86 (C=N), 167.45 (C=O).

(*Z*)-4-(4-Fluorobenzylidene)-3-methylisoxazol-5(4H)-one (**4e**). Yellow crystals; m.p.: 154–156 °C (Lit.: 153–155 °C¹); FT-IR (KBr, cm⁻¹): 1768 (C=O), 1612, 1586, 1470, 1375, 1323, 1274; ¹H-NMR (300 MHz, CDCl₃, δ / ppm): 2.23 (3H, *s*, CH₃), 6.80 (2H, *d*, *J* = 8.1 Hz, Ar), 8.12 (1H, *s*, Ar-CH=), 8.80 (2H, *d*, *J* = 8.1 Hz, Ar-H); ¹³C-NMR (75 MHz, CDCl₃, δ / ppm): 12.01 (CH₃), 116.81 (C= isoxazolone ring), 119.63 (Ar-C), 129.28 (Ar-C), 137.06 (Ar-C), 148.20 (Ar-CH=), 160.86 (C=N), 161.42 (Ar-F), 168.43 (C=O).

(*Z*)-3-Methyl-4-(4-methylbenzylidene)isoxazol-5(4H)-one (**4f**). Lemon crystals; m.p.: 129–131 °C (Lit.: 126–127 °C³); FT-IR (KBr, cm⁻¹): 1731 (C=O), 1594, 1114, 873, 777; ¹H-NMR (300 MHz, CDCl₃, δ / ppm): 2.29 (3H, *s*, CH₃), 2.45 (3H, *s*, CH₃), 7.32 (2H, *d*, *J* = 7.8 Hz, Ar-H), 7.40 (1H, *s*, Ar-CH=), 8.29 (2H, *d*, *J* = 7.8 Hz, Ar-H); ¹³C-NMR (75 MHz, CDCl₃, δ / ppm): 11.65 (CH₃), 22.07 (CH₃), 118.40 (C= isoxazolone ring), 129.88 (Ar-C), 134.14 (Ar-C), 145.73 (Ar-C), 149.96 (Ar-CH=), 161.22 (C=N), 168.21 (C=O).

(*Z*)-4-(4-(Dimethylamino)benzylidene)-3-methylisoxazol-5(4H)-one (**4g**). Red crystals; m.p.: 206–209 °C (Lit.: 206–207 °C¹); FT-IR (KBr, cm⁻¹): 1714 (C=O), 1557, 1380, 1196, 1095, 867, 765; ¹H-NMR (300 MHz, CDCl₃, δ / ppm): 2.23 (3H, *s*, CH₃), 3.15 (6H, *s*, N(CH₃)₂), 6.71 (2H, *d*, *J* = 9 Hz, Ar-H), 7.27 (1H, *s*, Ar-CH=), 8.39 (2H, *d*, *J* = 9 Hz, Ar-H); ¹³C-NMR (75 MHz, CDCl₃, δ / ppm): 11.71 (CH₃), 40.10 ((CH₃)₂N), 111.07 (Ar-C), 111.50 (C= isoxazolone ring), 121.51 (Ar-C), 137.62 (Ar-C), 149.26 (Ar-C), 154.22 (Ar-CH=), 161.59 (C=N), 170.12 (C=O).

(*Z*)-3-Methyl-4-(2-thienylmethylidene)isoxazol-5(4H)-one (**4h**). Yellow–brown crystals; m.p.: 148–150 °C (Lit.: 148–149 °C¹); FT-IR (KBr, cm⁻¹): 1753 (C=O), 1612, 1415, 1125, 820; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 2.32 (3H, *s*, CH₃); 7.29 (1H, *dd*, *J* = 4.9 & 3.9 Hz, thienyl); 7.66 (*s*, 1H, vinyl); 7.96 (*d*, *J* = 5 Hz, 1H, ArH); 8.13 (*d*, *J* = 8.5 Hz, 1H, thienyl); ¹³C-NMR (126 MHz, DMSO-*d*₆, δ / ppm): 11.86 (CH₃), 114.98 (C= isoxazolone ring), 129.32 (thienyl), 136.87 (thienyl), 139.63 (thienyl), 139.98 (thienyl), 141.93 (thienyl-CH=), 161.1 (C=N), 169.1 (C=O).

3-Methyl-4-(3-phenyl-2-propen-1-ylidene)isoxazol-5(4H)-one (**4i**). Yellow crystals; m.p.: 180–182 °C (Lit.: 179–181 °C¹); FT-IR (KBr, cm⁻¹): 1733 (C=O), 1542, 1103, 993, 848, 753; ¹H-NMR (300 MHz, CDCl₃, δ / ppm): 2.25 (3H, *s*,

CH₃), 7.28–7.36 (2H, *m*, CH=CH), 7.36–7.47 (3H, *m*, 2×Ar-H & Ar-CH=), 7.64–7.66 (2H, *m*, Ar-H), 8.26–8.35 (1H, *m*, Ar-H); ¹³C-NMR (75 MHz, CDCl₃, δ / ppm): 11.19 (CH₃), 117.86 (C= isoxazolone ring), 121.34 (Ar-C), 122.38 (Ar-C), 129.31 (Ar-C), 131.53 (C=C), 134.96 (C=C), 147.53 (Ar-C), 151.45 (Ar-CH=), 159.89 (C=N), 168.99 (C=O).

CHARACTERIZATION DATA FOR COMPOUNDS 5a–e

(*Z*)-4-Benzylidene-3-phenylisoxazol-5(4H)-one (**5a**). Yellow crystals; m.p.: 192–194 °C (Lit.: 193–194 °C⁴); FT-IR (KBr, cm⁻¹): 1753 (C=O), 1610, 1110, 825; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 7.29 (2H, *dd*, *J* = 7.7 & 7.5 Hz, Ar-H); 7.76–7.66 (7H, *m*, vinyl, Ar-H), 8.37 (2H, *d*, *J* = 7.6 Hz, Ar-H); ¹³C-NMR (126 MHz, DMSO-*d*₆, δ / ppm): 119.25 (C= isoxazolone ring), 127.80 (Ar-C), 129.18 (Ar-C), 129.42 (Ar-C), 129.74 (Ar-C), 131.46 (Ar-C), 132.79 (Ar-C), 134.4 (Ar-C), 134.61 (Ar-C), 153.18 (Ar-CH=), 164.43 (C=N), 168.45 (C=O).

(*Z*)-4-(4-(Dimethylamino)benzylidene)-3-phenylisoxazol-5(4H)-one (**5b**). Red crystals; m.p.: 194–196 °C (Lit.: 194–196 °C⁵); FT-IR (KBr, cm⁻¹): 1729, 1636, 1085, 800; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 3.19 (6H, *s*, 2×CH₃); 6.74 (2H, *d*, *J* = 9.2 Hz, Ar-H); 7.41 (1H, *s*, vinyl); 7.57–7.63 (5H, *m*, Ar-H), 8.42 (2H, *d*, *J* = 8.1 Hz, Ar-H); ¹³C-NMR (126 MHz, DMSO-*d*₆, δ / ppm): 40.55 (2×CH₃), 110.42 (Ar-C), 111.94 (Ar-C), 122.18 (C= isoxazolone ring), 129.14 (Ar-C), 129.29 (Ar-C), 129.43 (Ar-C), 130.73 (Ar-C), 138.43 (Ar-C), 152.11 (Ar-C), 154.9 (Ar-CH=), 165.17 (C=N), 170.82 (C=O).

(*Z*)-4-(4-Methoxybenzylidene)-3-phenylisoxazol-5(4H)-one (**5c**). Yellow crystals; m.p.: 166–167 °C (Lit.: 166–167 °C⁴); FT-IR (KBr, cm⁻¹): 1753, 1612, 1200, 870; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 3.96 (3H, *s*, OCH₃); 7.04 (2H, *d*, *J* = 8.9 Hz, Ar-H); 7.56 (1H, *s*, vinyl); 7.70–7.64 (5H, *m*, Ar-H), 8.45 (2H, *d*, *J* = 8.9 Hz, Ar-H); ¹³C-NMR (126 MHz, DMSO-*d*₆, δ / ppm): 56.16 (OCH₃), 115.09 (Ar-C), 115.81 (Ar-C), 126.38 (C= isoxazolone ring), 128.22 (Ar-C), 129.21 (Ar-C), 129.63 (Ar-C), 131.22 (Ar-C), 137.69 (Ar-C), 152.52 (Ar-CH=), 164.74 (Ar-O), 165.26 (C=N), 169.41 (C=O).

(*Z*)-4-(4-Methylbenzylidene)-3-phenylisoxazol-5(4H)-one (**5d**). Yellow crystals; m.p.: 188–181 °C (Lit.: 189–191 °C⁵); FT-IR (KBr, cm⁻¹): 1750, 1635, 1090, 860; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 2.49 (3H, *s*, CH₃); 7.36 (2H, *d*, *J* = 8.2 Hz, Ar-H); 7.59–7.65 (6H, *m*, vinyl & Ar-H), 8.30 (2H, *d*, *J* = 8.2 Hz, Ar-H); ¹³C-NMR (126 MHz, DMSO-*d*₆, δ / ppm): 22.52 (CH₃), 118.0 (Ar-C), 128.0 (C= isoxazolone ring), 129.2 (Ar-C), 129.7 (Ar-C), 130.3 (Ar-C), 130.5 (Ar-C), 131.4 (Ar-C), 134.8 (Ar-C), 146.4 (Ar-C), 153.2 (Ar-CH=), 164.6 (C=N), 168.8 (C=O).

(*Z*)-3-Phenyl-4-(2-thienylmethylidene)isoxazol-5(4H)-one (**5e**). Yellow crystals; m.p.: 225–226 °C (Lit.: 226–228 °C⁵); FT-IR (KBr, cm⁻¹): 1753, 1612; ¹H-NMR (500 MHz, DMSO-*d*₆, δ / ppm): 7.31 (1H, *dd*, *J* = 4.6 & 4.5 Hz, thienyl);

7.59–7.66 (5H, *m*, Ar-H), 7.83 (1H, *s*, vinyl); 8.01 (1H, *d*, *J* = 5 Hz, thienyl); 8.11 (1H, *d*, *J* = 3.7 Hz, thienyl); ¹³C-NMR (126 MHz, DMSO-*d*₆, δ / ppm): 113.88 (Ar-C), 127.99 (C= isoxazolone ring), 128.95 (Ar-C), 129.33 (Ar-C), 129.77 (Ar-C), 131.38 (thienyl), 137.09 (thienyl), 140.65 (thienyl), 141.96 (thienyl), 142.50 (thienyl-CH=), 163.78 (C=N), 169.37 (C=O).

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