

Supporting Information for

KO^tBu-promoted oxidative dimerizations of 2-methylquinolines to 2-alkenyl bisquinolines with molecular oxygen

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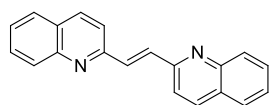
huiqiaowang@163.com

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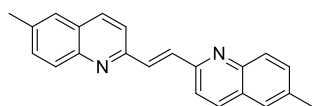
Characterization data of the products

(*E*)-1,2-di(quinolin-2-yl)ethene (2a)¹



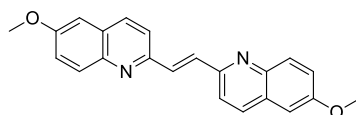
Yellow solid, m.p. 191-192 °C. ¹H NMR (300 MHz, CDCl₃): δ 8.19 (d, *J* = 9 Hz, 2H), 8.12 (d, *J* = 9 Hz, 2H), 7.95 (s, 2H), 7.85-7.81 (m, 4H), 7.76-7.71 (m, 2H), 7.56-7.51 (m, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 155.6, 148.5, 136.7, 134.8, 130.0, 129.6, 127.8, 127.7, 126.8, 119.7. HRMS calcd for C₂₀H₁₄N₂: 282.1157, Found: 282.1161.

(*E*)-1,2-bis(6-methylquinolin-2-yl)ethene (2b)



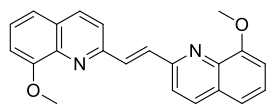
Yellow solid, m.p. 213-214 °C. ¹H NMR (300 MHz, CDCl₃): δ 8.09 (d, *J* = 9 Hz, 2H), 8.00 (d, *J* = 9 Hz, 2H), 7.97 (s, 2H), 7.79 (d, *J* = 9 Hz, 2H), 7.56 (d, *J* = 9 Hz, 4H), 2.55 (s, 6H). ¹³C NMR (75 MHz, CDCl₃): δ 154.7, 146.9, 136.6, 135.8, 134.3, 132.1, 129.1, 127.7, 126.5, 119.5, 21.6. HRMS calcd for C₂₂H₁₈N₂: 310.1470, Found: 310.1473.

(*E*)-1,2-bis(6-methoxyquinolin-2-yl)ethene (2c)



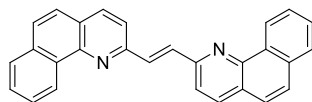
Yellow solid, m.p. 214-216 °C. ¹H NMR (300 MHz, CDCl₃): δ 8.08-7.99 (m, 4H), 7.85 (s, 2H), 7.78 (d, *J* = 8.4 Hz, 2H), 7.40-7.36 (m, 2 H), 7.08 (d, *J* = 3 Hz, 2H), 3.95 (s, 6H). ¹³C NMR (75 MHz, CDCl₃): δ 158.1, 153.2, 143.9, 135.7, 133.5, 130.2, 128.7, 122.7, 119.4, 105.4, 55.6. HRMS calcd for C₂₂H₁₈N₂O₂: 342.1368, Found: 342.1369.

(*E*)-1,2-bis(8-methoxyquinolin-2-yl)ethene (2d)



Yellow solid, m.p. 197-198 °C. ¹H NMR (300 MHz, CDCl₃): δ 8.16 (d, *J* = 8.4 Hz, 2H), 7.95 (d, *J* = 9 Hz, 4 H), 7.48-7.38 (m, 4H), 7.07 (d, *J* = 7.2 Hz, 2H), 4.13 (s, 6H). ¹³C NMR (75 MHz, CDCl₃): δ 155.5, 154.9, 140.2, 136.6, 135.2, 128.8, 126.9, 119.6, 119.3, 108.2, 56.2. HRMS calcd for C₂₂H₁₈N₂O₂: 342.1368, Found: 342.1371.

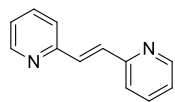
(*E*)-1,2-bis(benzo[*h*]quinolin-2-yl)ethene (2e)



Yellow solid, m.p. 222-223 °C. ¹H NMR (300 MHz, CDCl₃): δ 9.54 (d, *J* = 8.1 Hz, 1H), 9.36 (t, *J* = 3 Hz, 1H), 8.26-8.21 (m, 2H), 8.18 (s, 1H), 7.93-7.58 (m, 13H). ¹³C NMR (75 MHz, CDCl₃): δ 154.1,

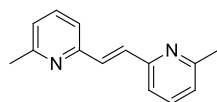
152.9, 145.5, 135.7, 135.2, 132.9, 132.8, 130.6, 130.3, 127.3, 127.2, 126.9, 126.8, 126.7, 126.6, 126.0, 125.9, 125.1, 124.7, 124.1, 124.0, 123.8, 123.6, 120.1, 116.7. HRMS calcd for C₂₈H₁₈N₂: 382.1470, Found: 382.1473.

(E)-1,2-di(pyridin-2-yl)ethene (2f)²



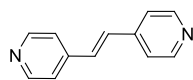
White solid, m.p. 115-116 °C. ¹H NMR (300 MHz, CDCl₃): δ 8.63 (d, *J* = 4.5 Hz, 2 H), 7.70-7.65 (m, 4H), 7.43 (d, *J* = 8.8 Hz, 2H), 7.20-7.16 (m, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 155.2, 149.9, 136.7, 131.9, 123.4, 122.7. HRMS calcd for C₁₂H₁₀N₂: 182.0844, Found: 182.0848.

(E)-1,2-bis(6-methylpyridin-2-yl)ethane (2g)



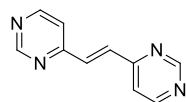
Yellow solid, m.p. 107-109 °C. ¹H NMR (300 MHz, CDCl₃): δ 7.64 (s, 2H), 7.56 (t, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 2.59 (s, 6H). ¹³C NMR (75 MHz, CDCl₃): δ 158.4, 154.8, 136.8, 132.1, 122.3, 119.8, 24.7. HRMS calcd for C₁₄H₁₄N₂: 210.1157, Found: 210.1153.

(E)-1,2-di(pyridin-4-yl)ethene (2h)³



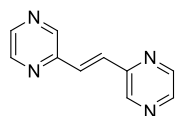
White solid, m.p. 146-147 °C. ¹H NMR (300 MHz, CDCl₃): δ 8.61 (t, *J* = 4.8 Hz, 4H), 7.38-7.36 (m, 4H), 7.19 (s, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 150.4, 143.4, 130.5, 121.2. HRMS calcd for C₁₂H₁₀N₂: 182.0844, Found: 182.0846.

(E)-1,2-di(pyrimidin-4-yl)ethane (2i)



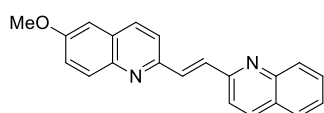
Yellow solid, m.p. 180-181 °C. ¹H NMR (300 MHz, CDCl₃): δ 8.69 (s, 2H), 8.61 (s, 2H), 8.50 (d, *J* = 1.5 Hz, 2H), 7.83 (s, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 150.2, 144.9, 144.8, 144.1, 130.3. HRMS calcd for C₁₀H₈N₄: 184.0749, Found: 184.0751.

(E)-1,2-di(pyrazin-2-yl)ethane (2j)⁴



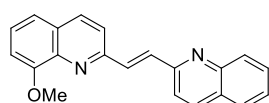
Yellow solid, m.p. 189-190 °C. ¹H NMR (300 MHz, CDCl₃): δ 8.69 (s, 2 H), 8.60 (s, 2 H), 8.49 (s, 2 H), 7.83 (s, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 150.2, 144.9, 144.8, 144.1, 130.3. HRMS calcd for C₁₀H₈N₄: 184.0749, Found: 184.0751.

(E)-6-methoxy-2-(2-(quinolin-2-yl)vinyl)quinoline (3)



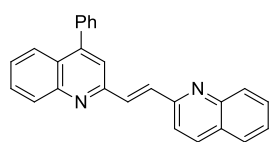
White solid, m.p. 165-166 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.11 (t, *J* = 9.0 Hz, 2H), 8.01 (t, *J* = 8.1 Hz, 2H), 7.87 (d, *J* = 4.1 Hz, 2H), 7.84 – 7.64 (m, 4H), 7.50 (t, *J* = 7.4 Hz, 1H), 7.37 (dd, *J* = 9.2, 2.6 Hz, 1H), 7.03 (d, *J* = 2.4 Hz, 1H), 3.91 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 158.1, 155.7, 153.1, 148.4, 144.4, 136.6, 135.3, 134.8, 133.5, 131.0, 129.9, 129.5, 128.8, 127.7, 127.6, 126.6, 122.6, 119.9, 119.5, 105.3, 55.7. HRMS calcd for C₂₁H₁₆N₂O: 312.1263, Found: 312.1266.

(E)-8-methoxy-2-(2-(quinolin-2-yl)vinyl)quinoline (4)



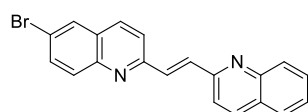
White solid, m.p. 155-156 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.14 – 8.00 (m, 3H), 7.95 (d, *J* = 16.6 Hz, 1H), 7.87 – 7.75 (m, 3H), 7.72 (d, *J* = 8.1 Hz, 1H), 7.64 (dd, *J* = 8.2, 7.1 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.9 Hz, 1H), 7.31 (d, *J* = 8.1 Hz, 1H), 6.99 (d, *J* = 7.5 Hz, 1H), 4.05 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 155.7, 155.4, 154.6, 148.3, 140.2, 136.6, 136.5, 135.4, 134.4, 129.8, 129.4, 128.7, 127.6, 127.5, 126.9, 126.8, 126.7, 119.6, 119.5, 119.2, 108.1, 56.2. HRMS calcd for C₂₁H₁₆N₂O: 312.1263, Found: 312.1265.

(E)-4-phenyl-2-(2-(quinolin-2-yl)vinyl)quinoline (5)



Pale yellow solid, m.p. 179-180 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.11 (d, *J* = 8.5 Hz, 2H), 8.02 (d, *J* = 8.5 Hz, 1H), 7.90 (s, 2H), 7.84 (d, *J* = 8.4 Hz, 1H), 7.78 – 7.69 (m, 3H), 7.65 (q, *J* = 8.0 Hz, 2H), 7.49 – 7.39 (m, 7H). ¹³C NMR (100 MHz, CDCl₃) δ 155.5, 155.0, 149.0, 148.9, 148.4, 138.2, 136.6, 134.9, 134.7, 129.9, 129.8, 129.7, 129.6, 128.7, 128.6, 127.8, 127.6, 126.8, 126.7, 126.4, 125.9, 119.9, 119.6. HRMS calcd for C₂₆H₁₈N₂: 358.1470, Found: 358.1473.

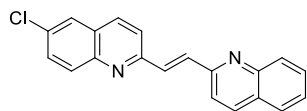
(E)-6-bromo-2-(2-(quinolin-2-yl)vinyl)quinoline (6)



Pale yellow solid, m.p. 236-237 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.18 (d, *J* = 8.5 Hz, 1H), 8.09 (dd, *J* = 16.6, 8.5 Hz, 2H), 7.98 – 7.92 (m, 4H), 7.86 – 7.76 (m, 4H), 7.73 (t, *J* = 7.4 Hz, 1H), 7.53 (t, *J* = 7.4 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 155.9, 155.3, 148.5, 147.0, 136.7, 135.6, 135.2, 134.2,

133.4, 131.3, 130.1, 129.7, 129.6, 128.8, 127.8, 127.7, 126.9, 120.7, 120.6, 119.8. HRMS calcd for C₂₀H₁₃BrN₂: 360.0262, Found: 360.0266.

(E)-6-chloro-2-(2-(quinolin-2-yl)vinyl)quinoline (7)



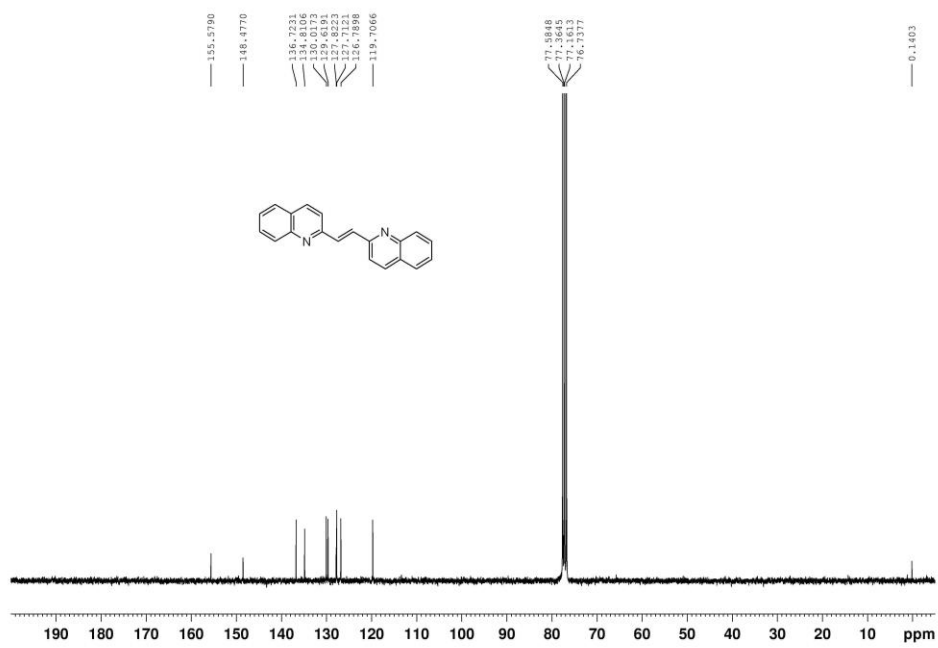
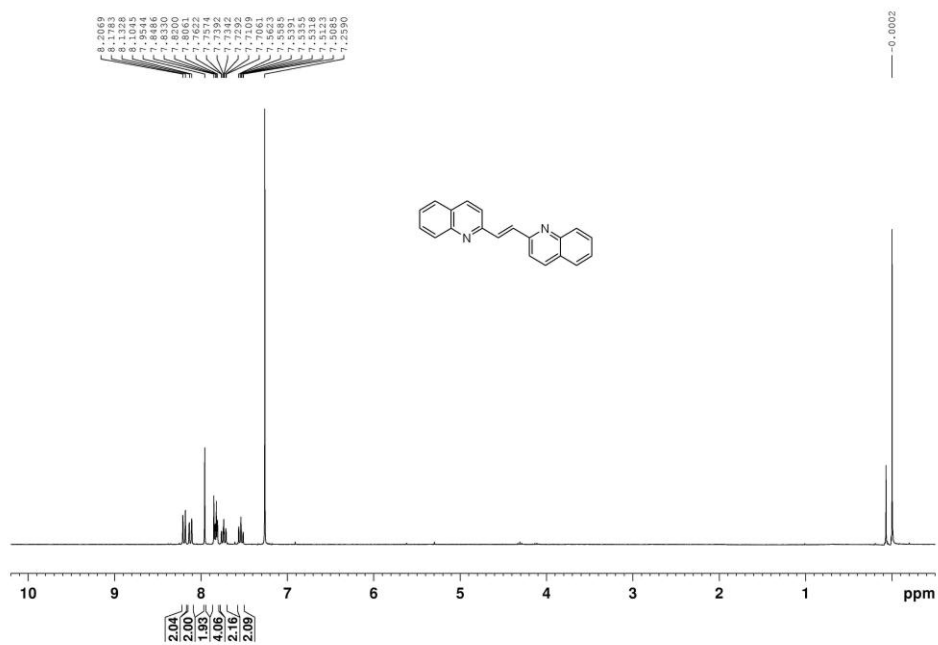
Pale yellow solid, m.p. 210-211 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.17 (d, *J* = 8.5 Hz, 1H), 8.13 – 8.02 (m, 3H), 7.92 (d, *J* = 5.0 Hz, 2H), 7.86 – 7.76 (m, 4H), 7.76 – 7.70 (m, 1H), 7.65 (d, *J* = 8.9 Hz, 1H), 7.53 (t, *J* = 7.3 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 155.8, 155.3, 148.5, 146.8, 136.7, 135.7, 135.1, 134.2, 132.4, 131.2, 130.9, 130.0, 129.6, 128.3, 127.8, 127.7, 126.8, 126.4, 120.7, 119.8. HRMS calcd for C₂₀H₁₃ClN₂: 316.0767, Found: 316.0771.

References

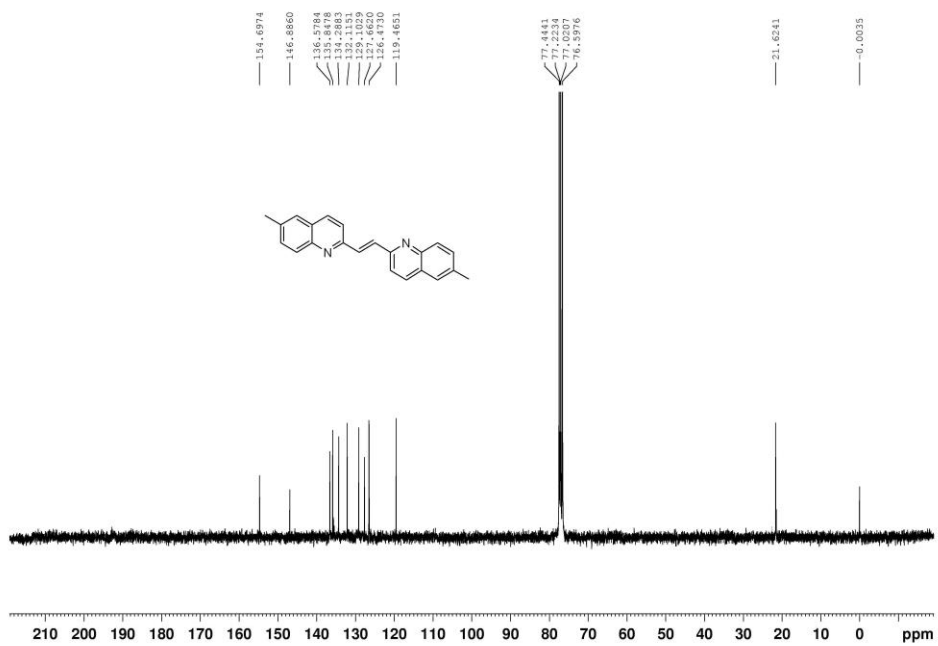
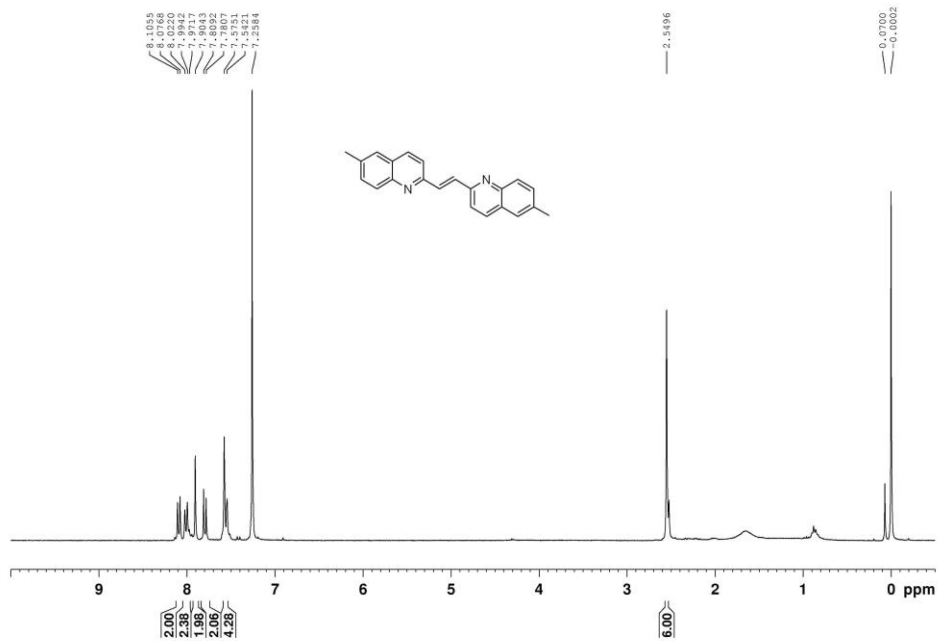
- [1] (a) M. A. Fakhfakh, A. Fournet, E. Prina, J.-F. Mouscadet, X. Franck, R. Hocquemiller, B. Figadère, *Bioorg. Med. Chem.* **2003**, *11*, 5013-5023; (b) L. He, J.-Q. Wang, Y. Gong, Y.-M. Liu, Y. Cao, H.-Y. He, K.-N. Fan, *Angew. Chem. Int. Ed.* **2011**, *50*, 10216-10220.
- [2] C. S. Marvel, A. T. Tweedie, J. Economy, *J. Org. Chem.* **1956**, *21*, 1420-1422.
- [3] E. Gleich, Z. Warnket, *Phosphorus Sulfur* **1991**, *55*, 9-17.
- [4] J. Vansant, G. Smets, J. P. Declercq, G. Germain, M. Van Meerssche, *J. Org. Chem.* **1980**, *45*, 1557-1565.

Copies of NMR spectra of the products

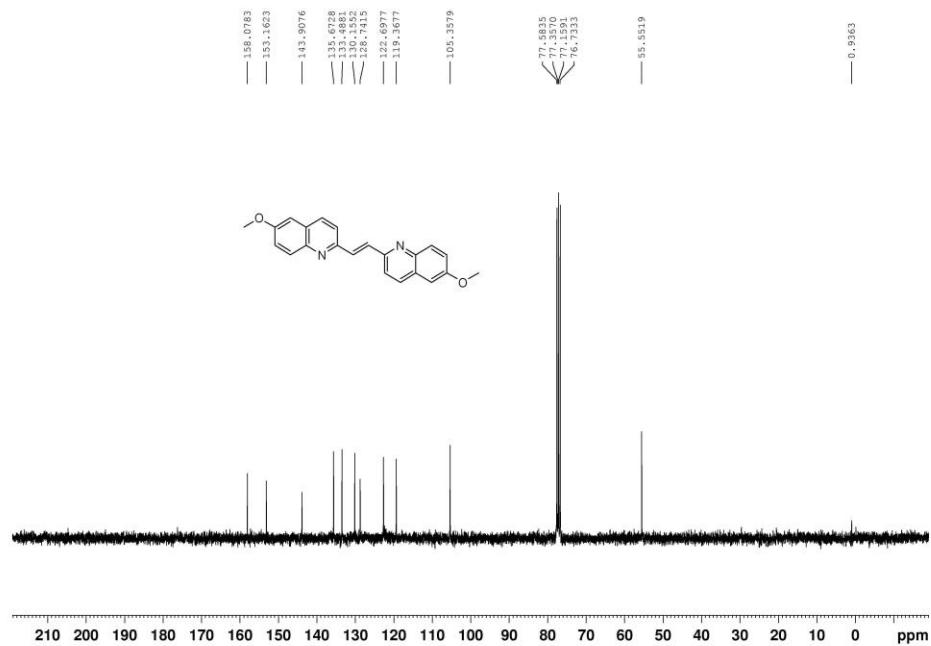
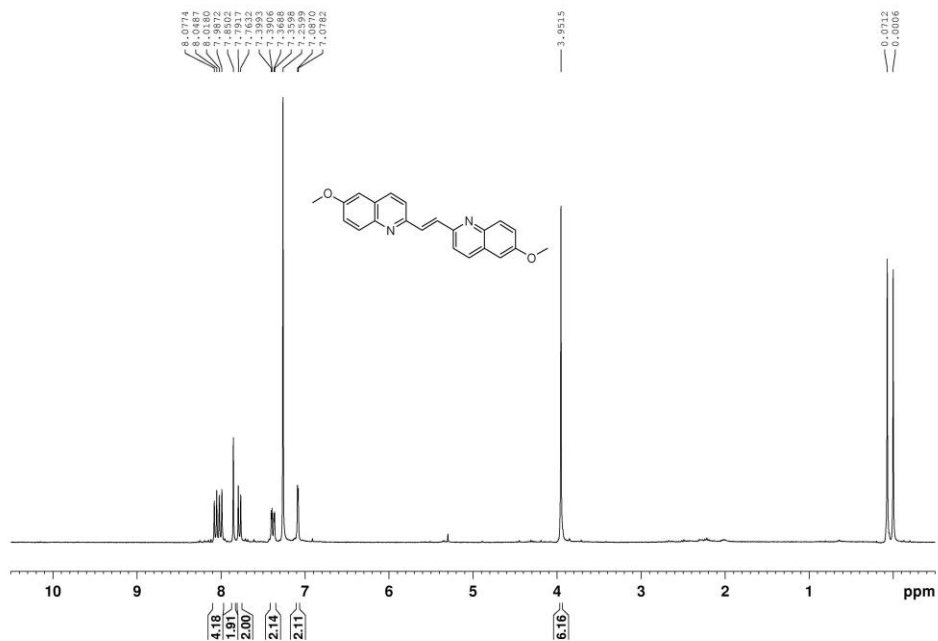
(*E*)-1,2-di(quinolin-2-yl)ethene (2a)



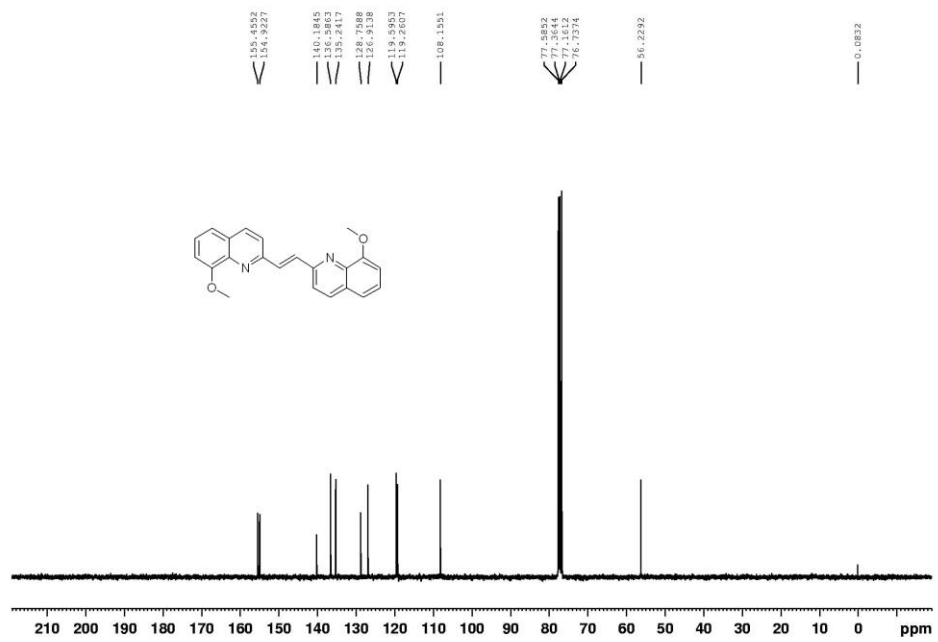
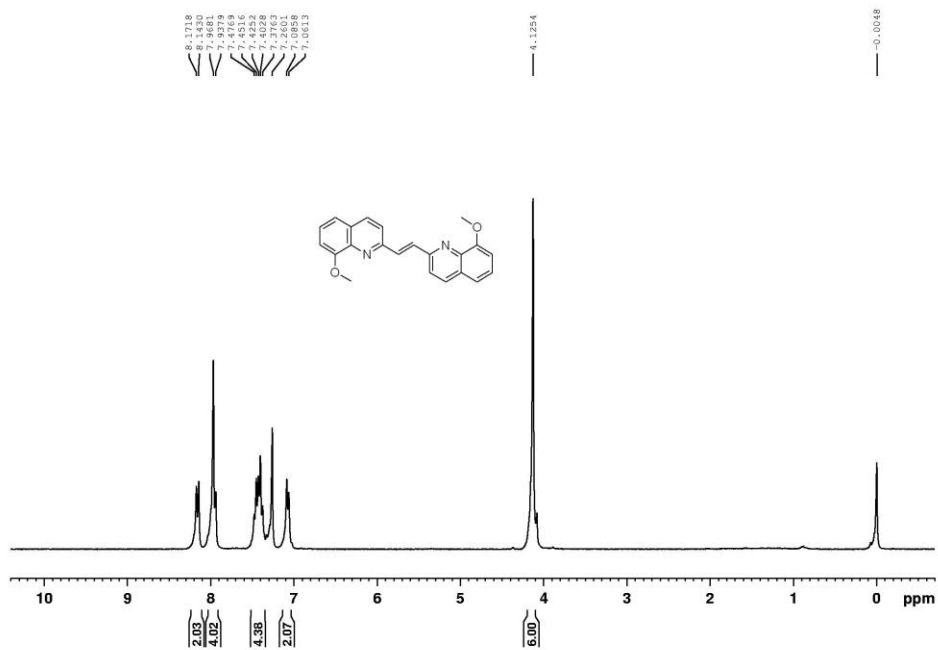
(E)-1,2-bis(6-methylquinolin-2-yl)ethene (2b)



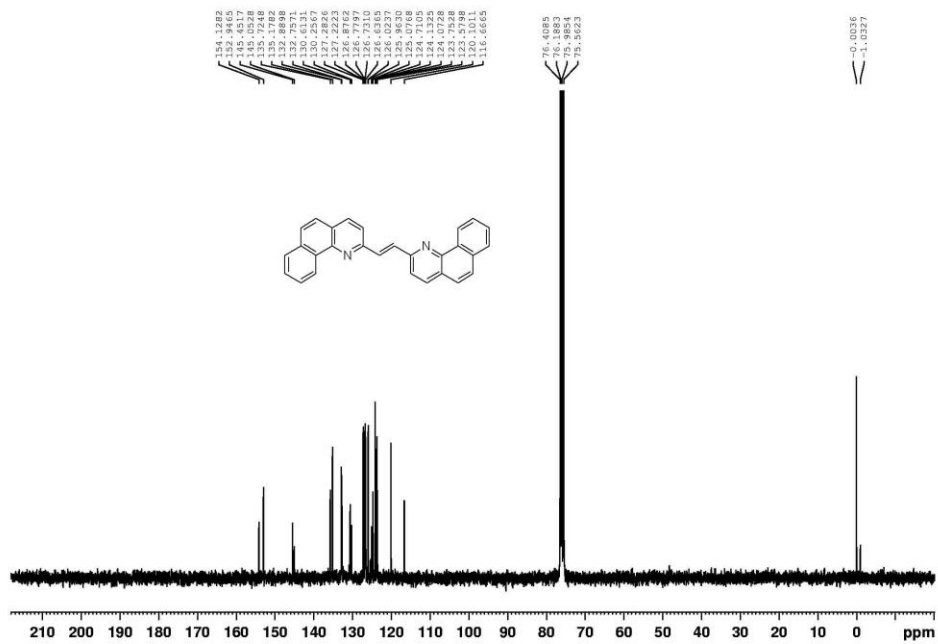
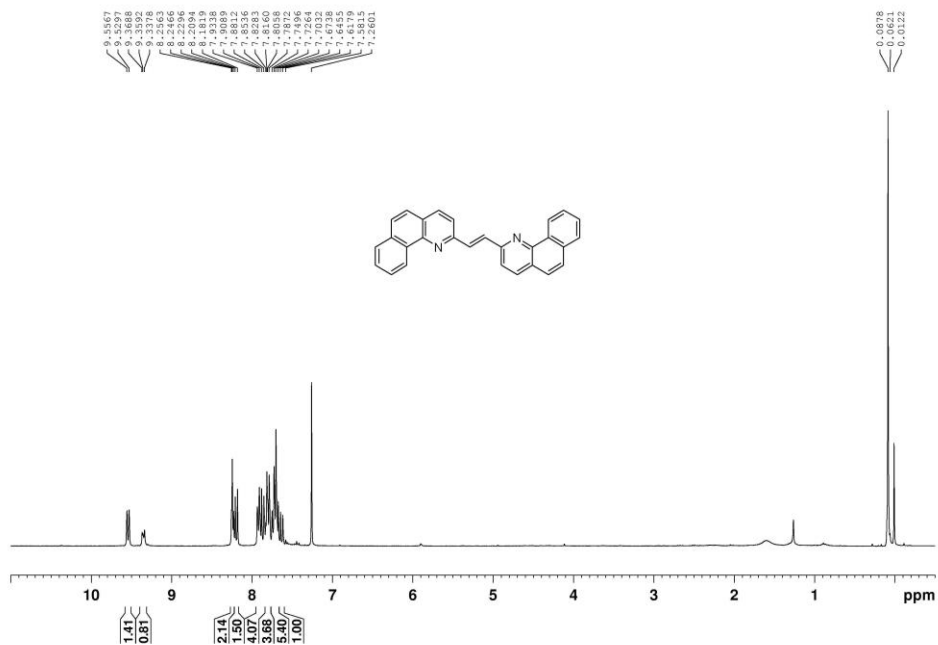
(E)-1,2-bis(6-methoxyquinolin-2-yl)ethene (2c)



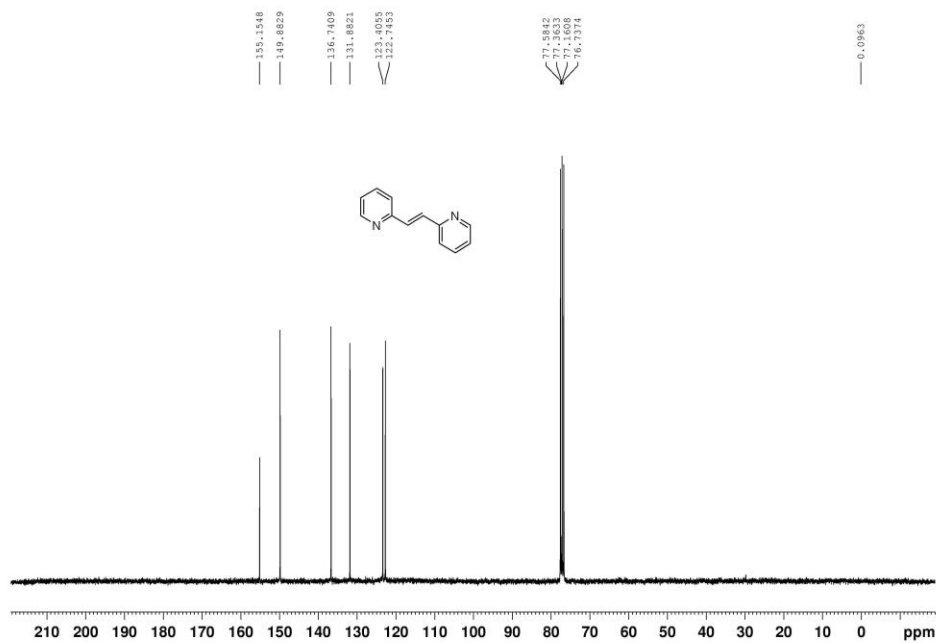
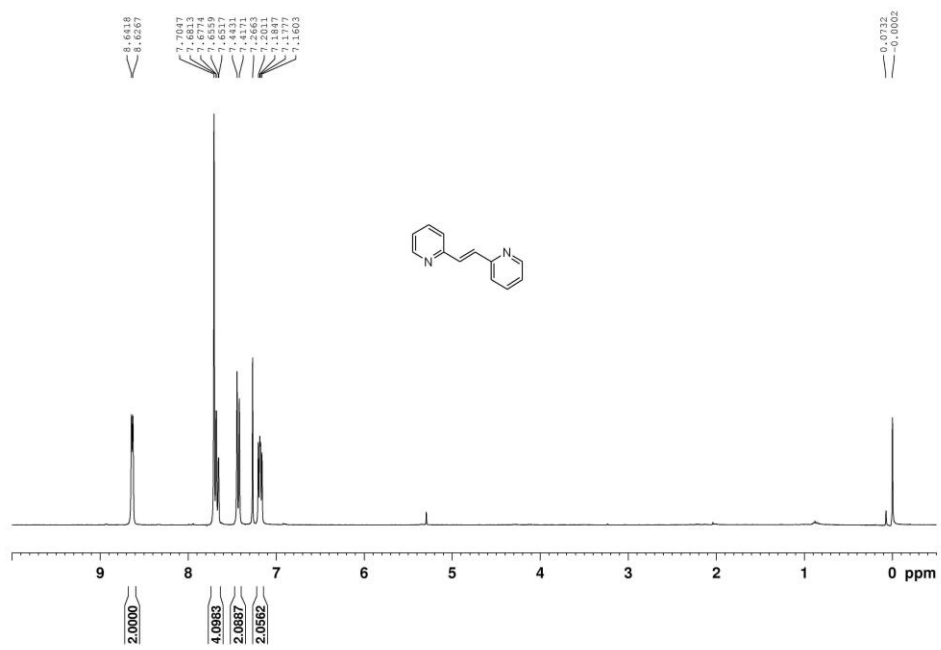
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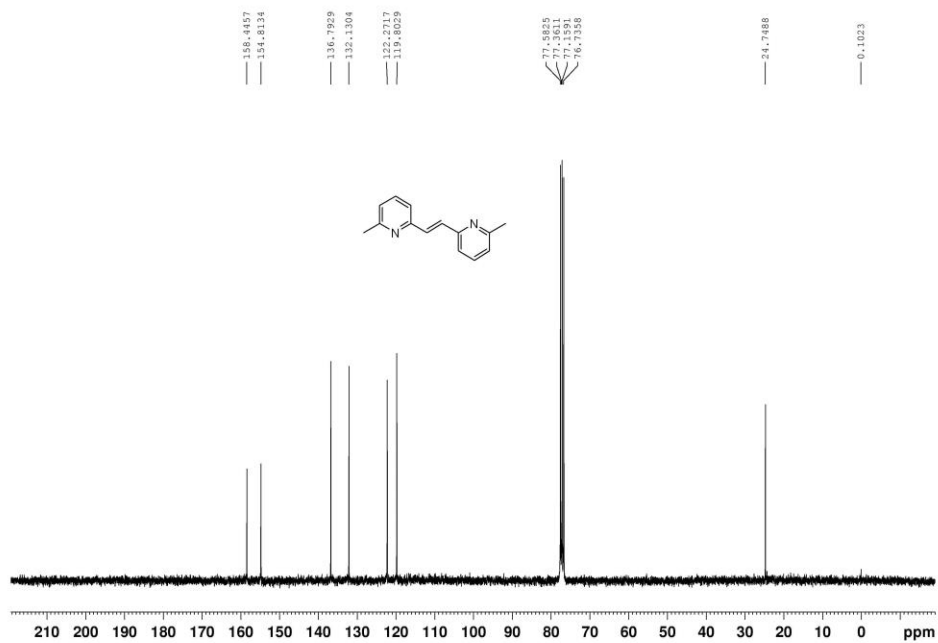
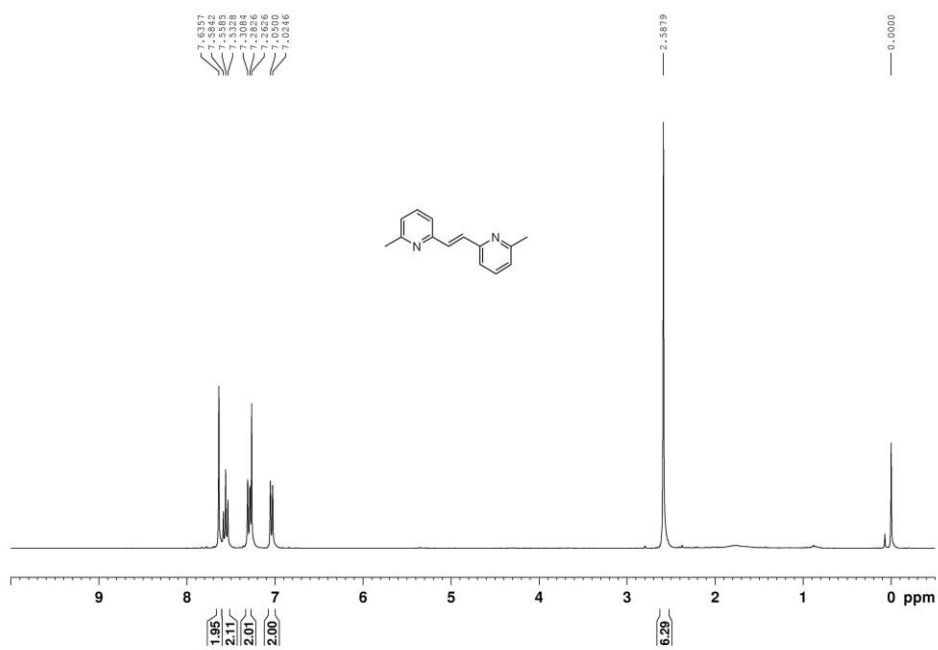
(E)-1,2-bis(benzo[h]quinolin-2-yl)ethene (2e)



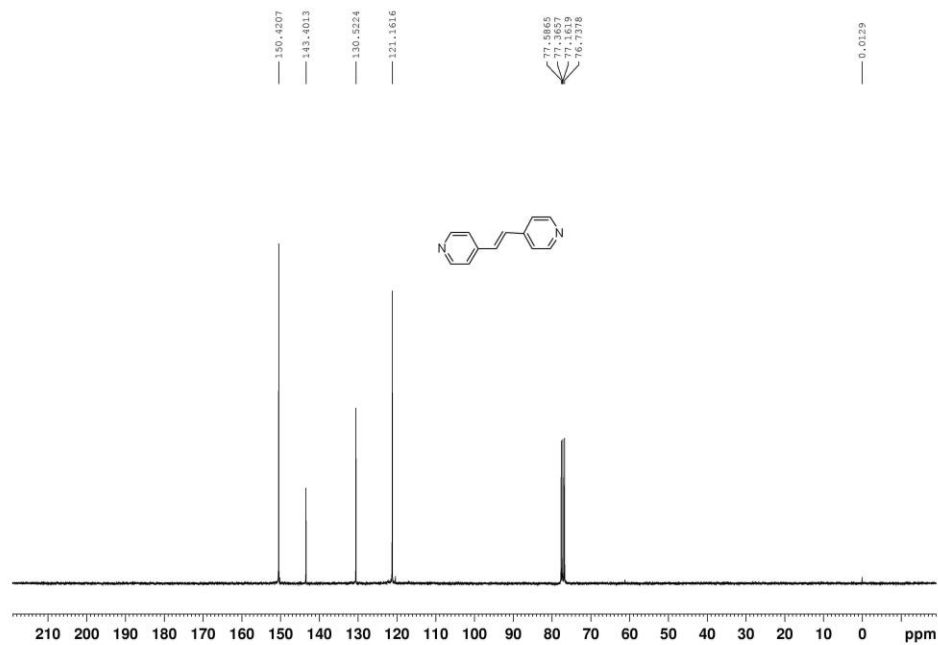
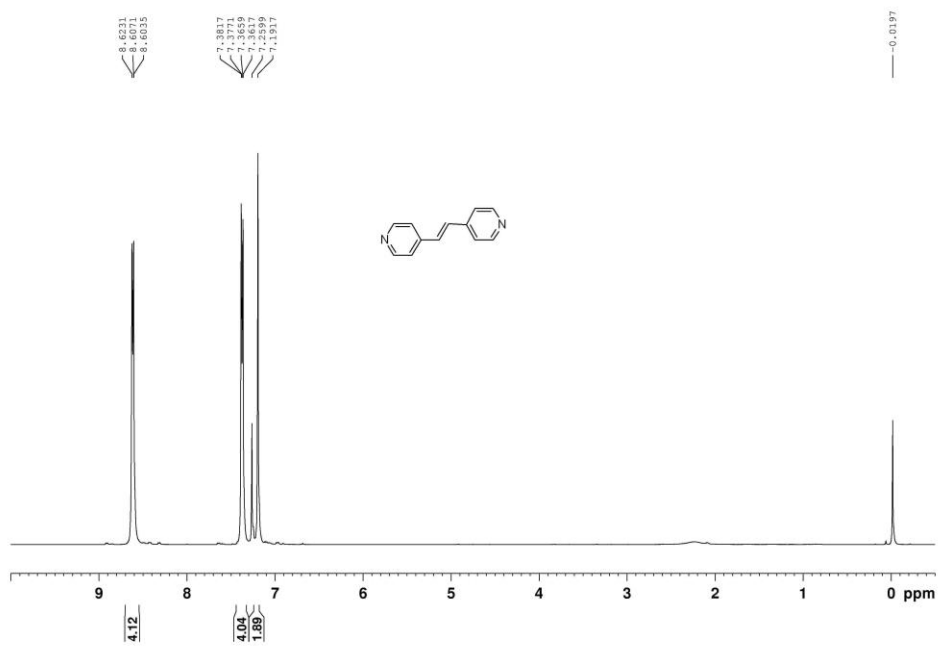
(E)-1,2-di(pyridin-2-yl)ethene (2f)



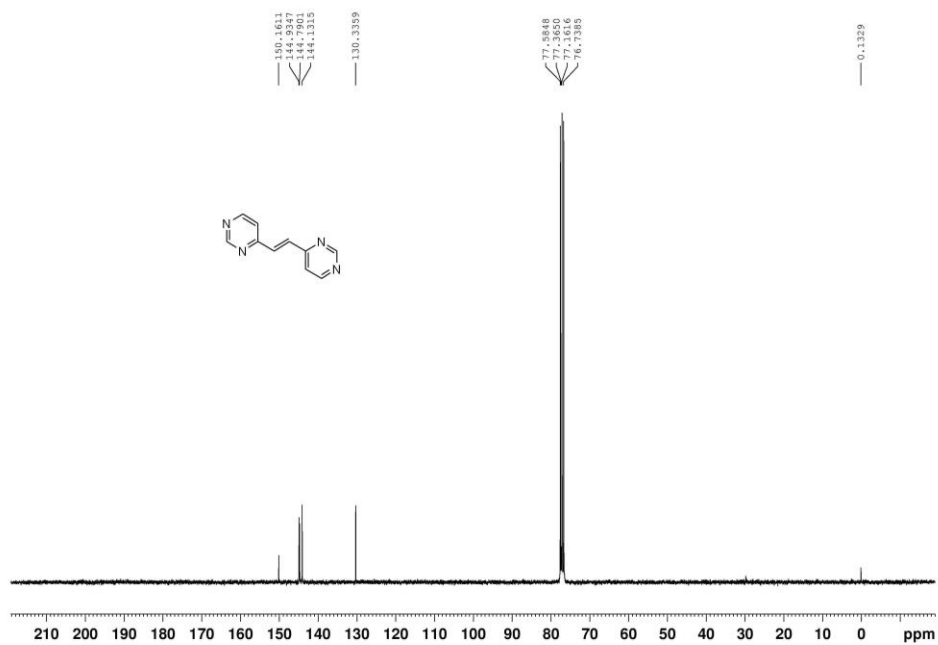
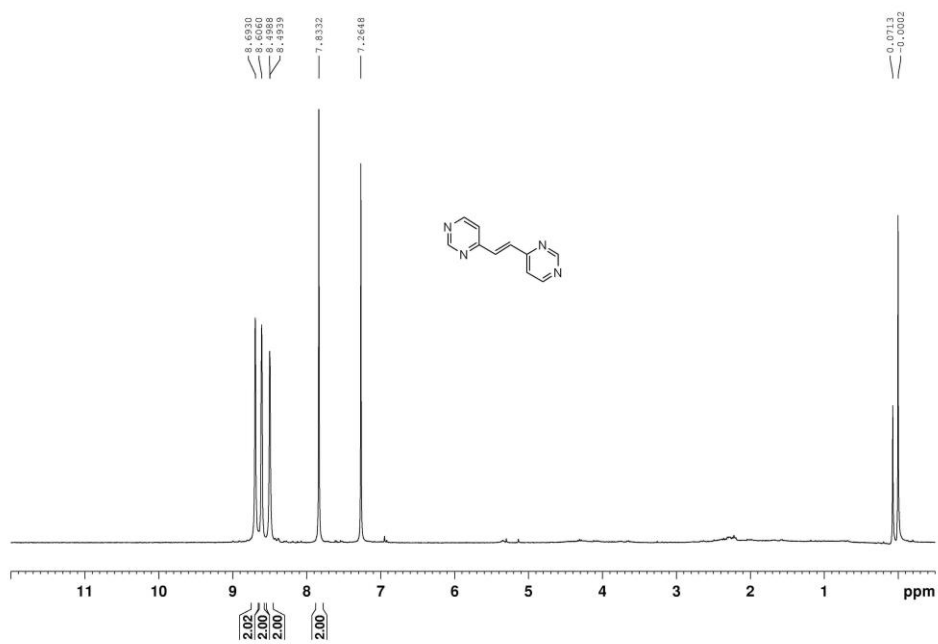
(E)-1,2-bis(6-methylpyridin-2-yl)ethane (2g)



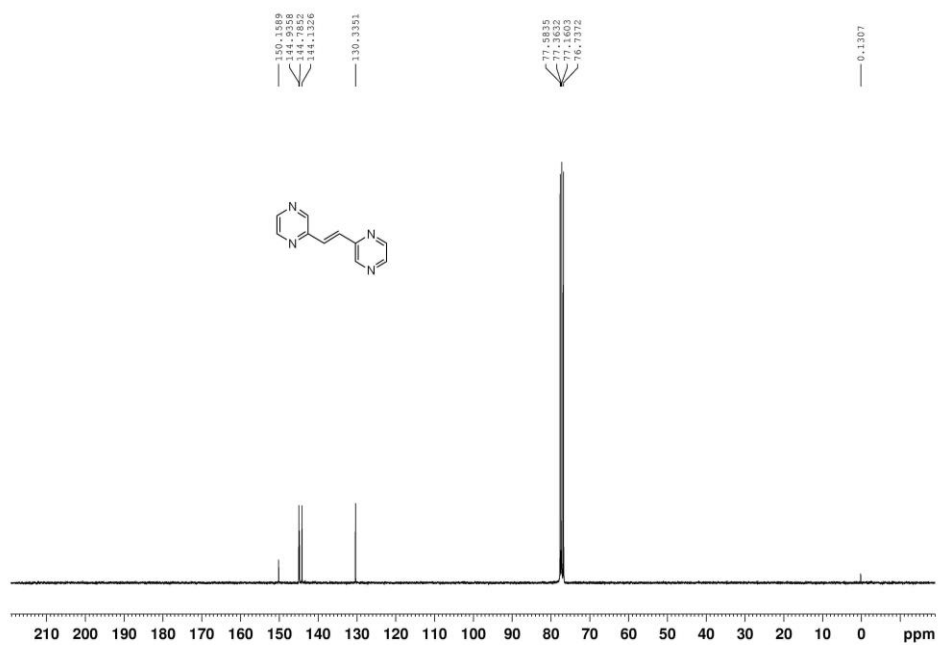
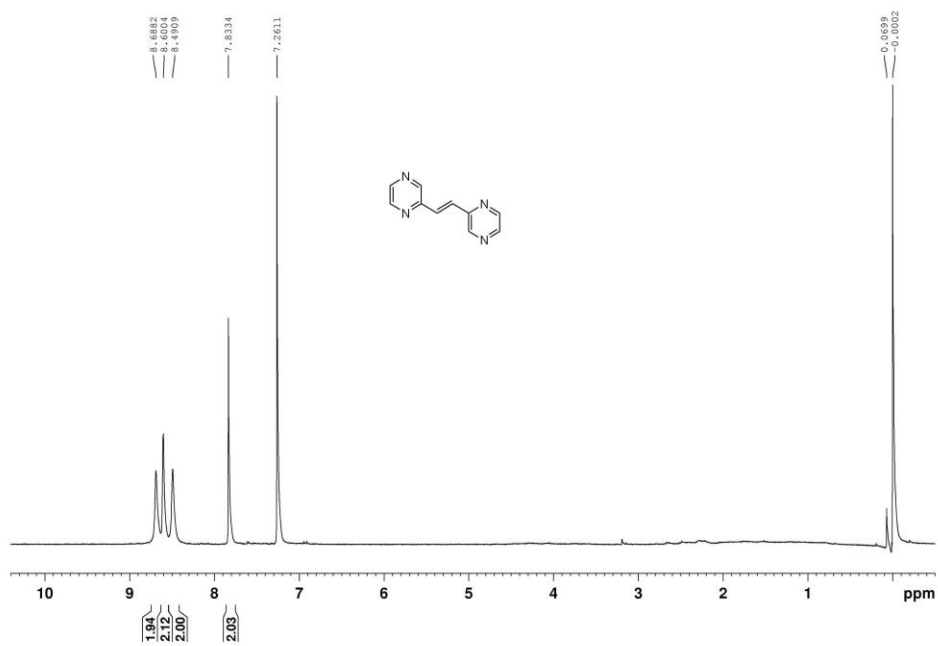
(E)-1,2-di(pyridin-4-yl)ethane (2h)



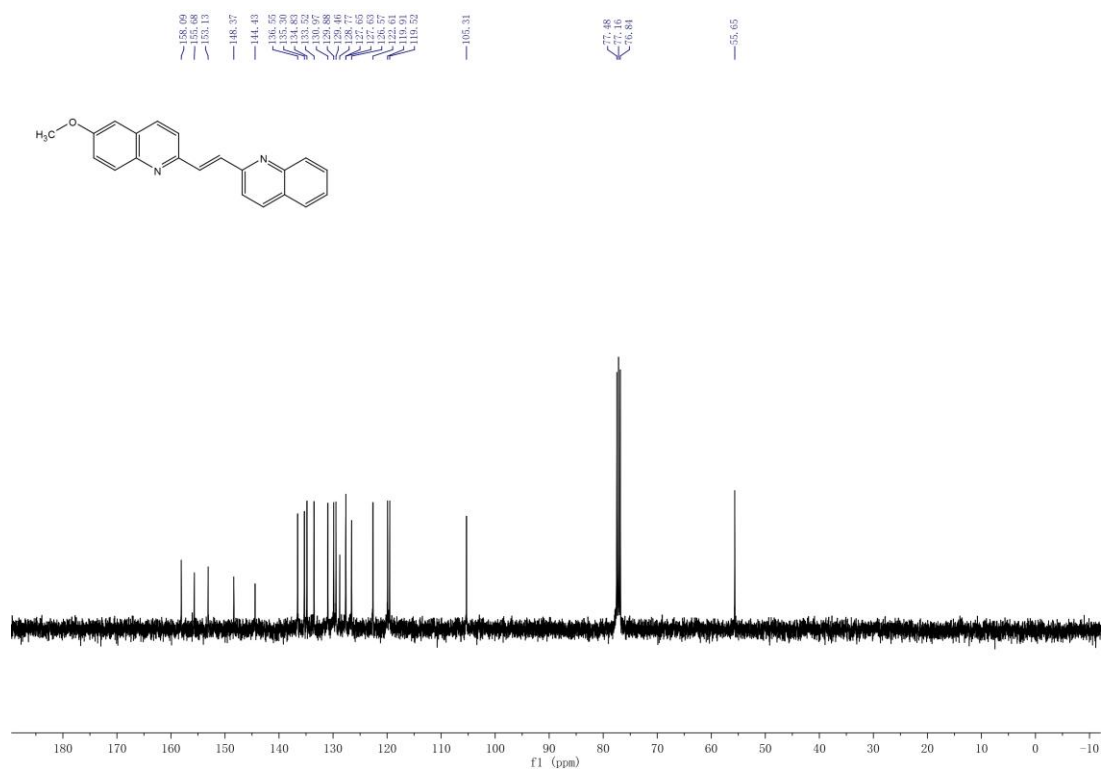
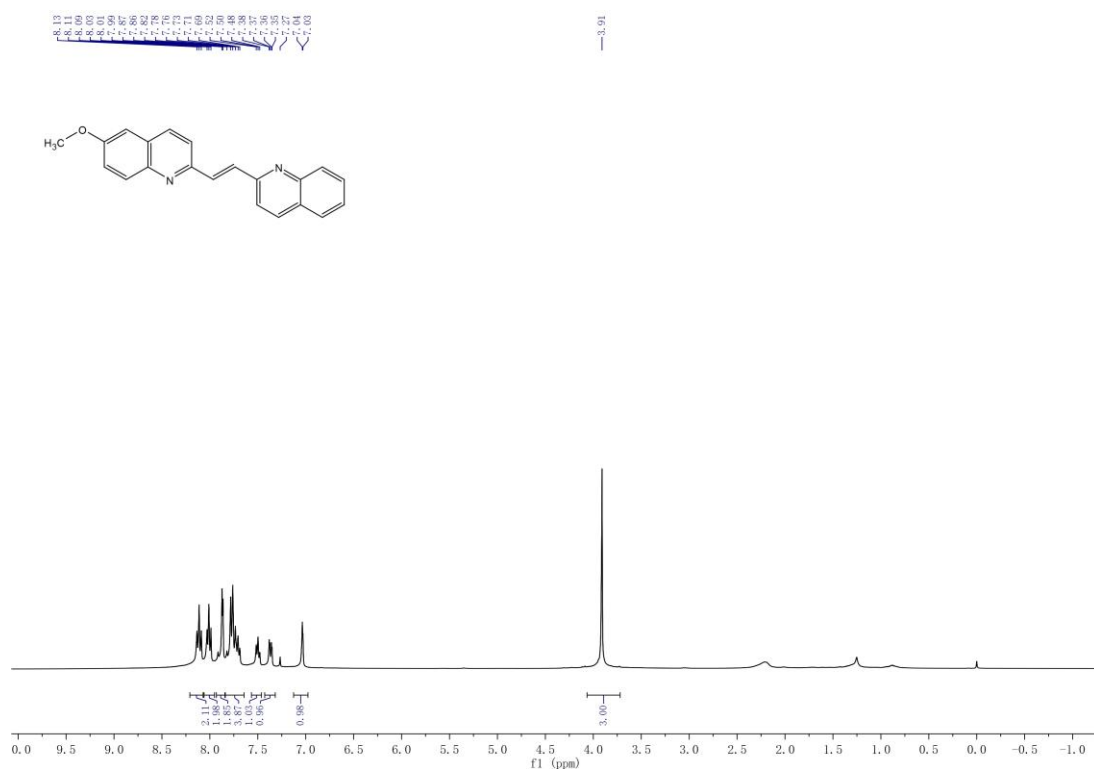
(E)-1,2-di(pyrimidin-4-yl)ethane (2i)



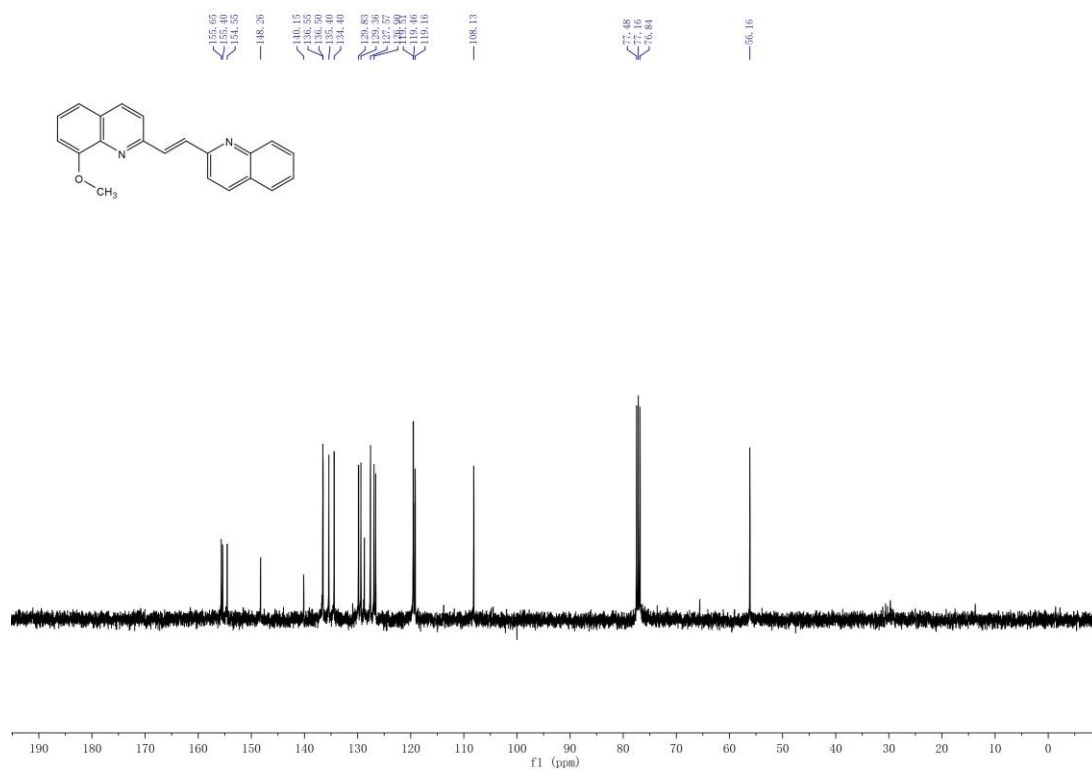
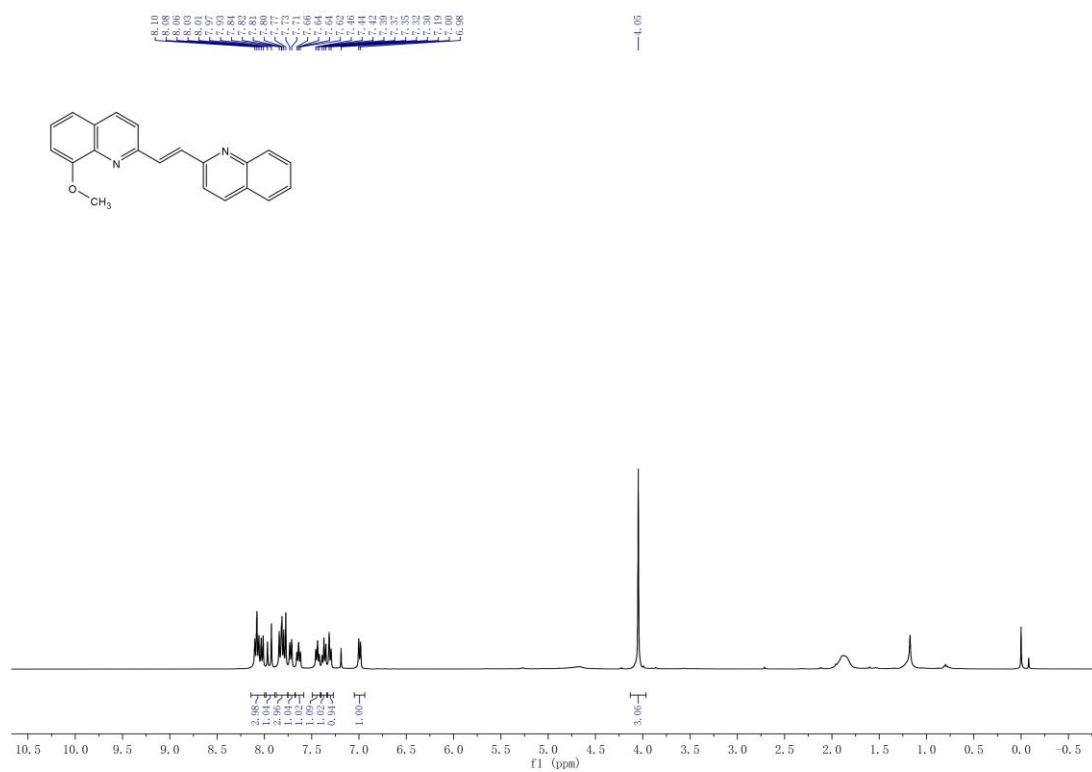
(E)-1,2-di(pyrazin-2-yl)ethane (2j)



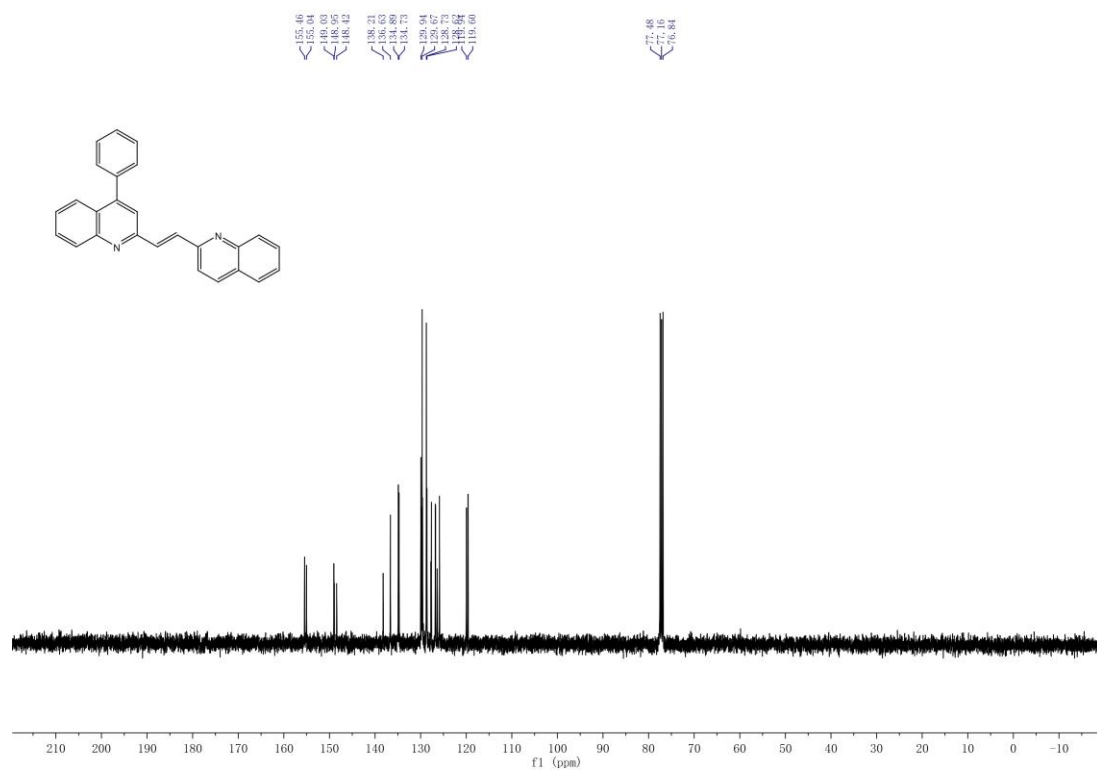
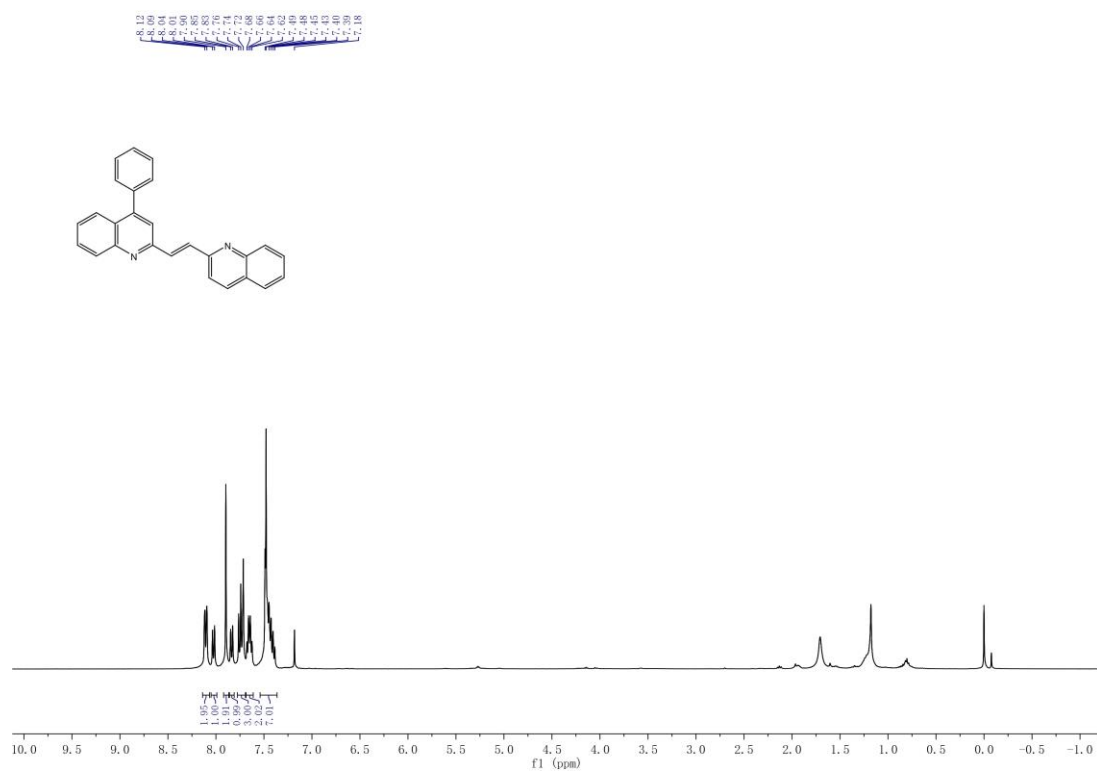
(E)-6-methoxy-2-(2-(quinolin-2-yl)vinyl)quinoline (3)



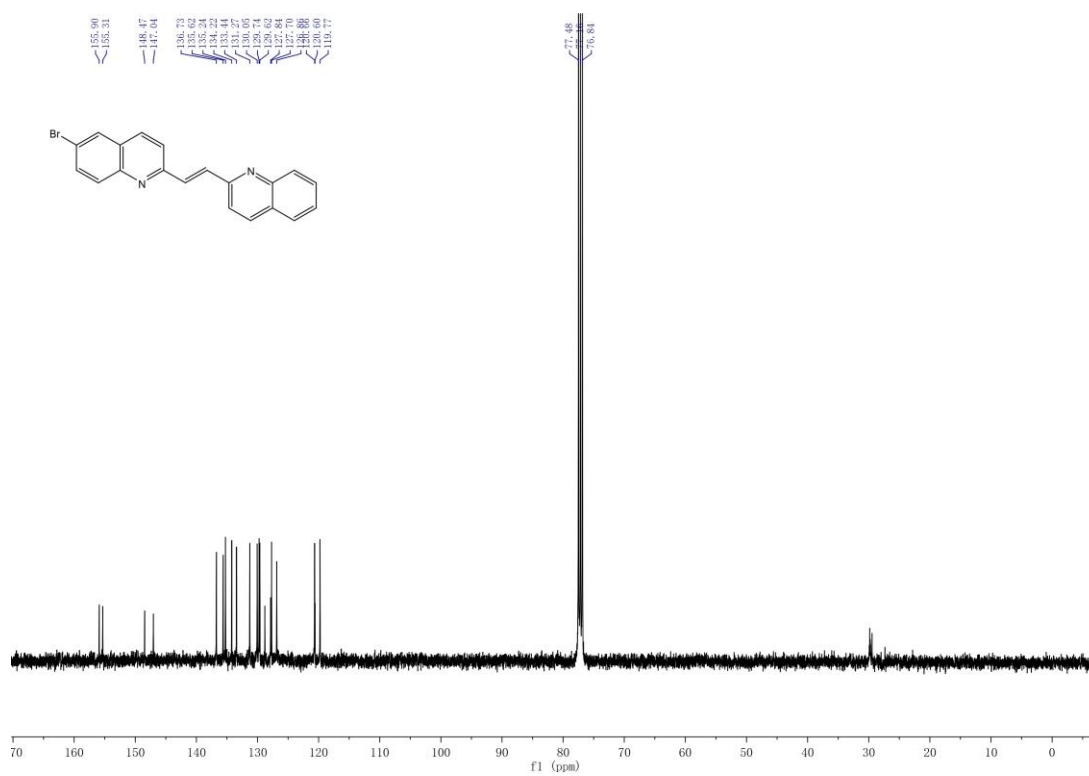
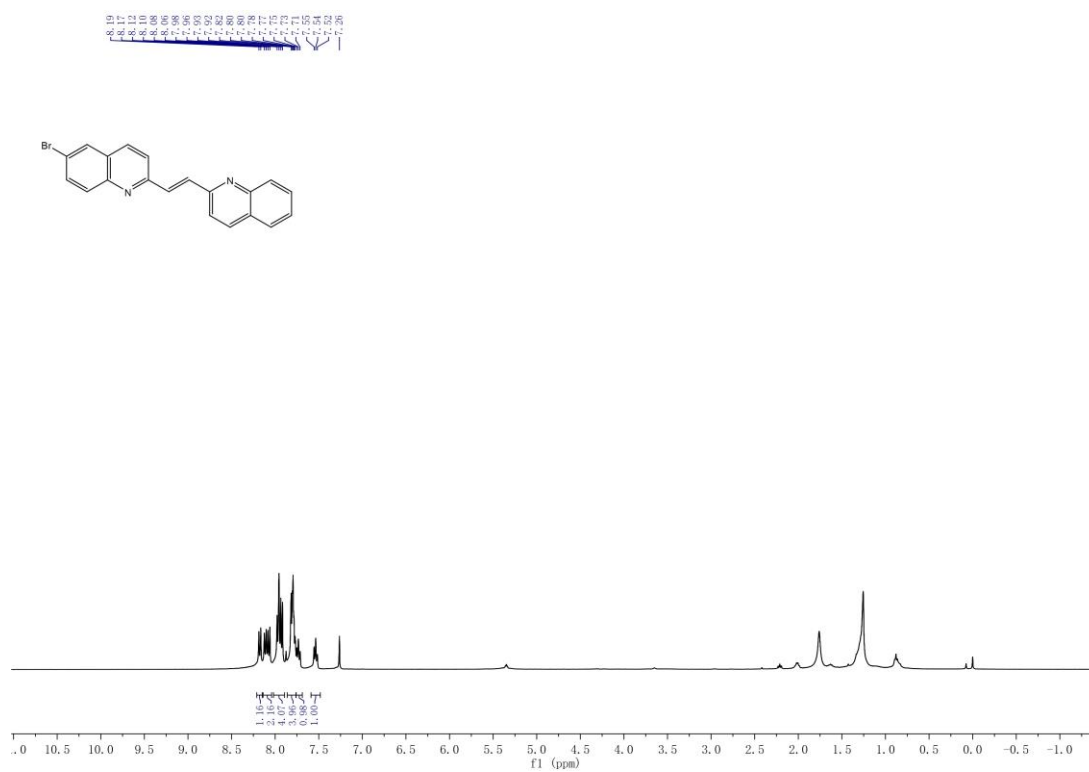
(E)-8-methoxy-2-(2-(quinolin-2-yl)vinyl)quinoline (4)



(E)-4-phenyl-2-(2-(quinolin-2-yl)vinyl)quinoline (5)



(E)-6-bromo-2-(2-(quinolin-2-yl)vinyl)quinoline (6)



(E)-6-chloro-2-(2-(quinolin-2-yl)vinyl)quinoline (7)

