

Supporting Information

Recyclable Anhydride Catalyst for H₂O₂ Oxidation: N-oxidation of Pyridine Derivatives

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¹H NMR and ¹³C NMR of N-Oxide products:

2-carboxypyridine N-Oxide ($C_6H_5NO_3$)

δ ¹H NMR (500 MHz, DMSO): 7.88-7.95 (2H, m, Ar-H), 8.30-8.32 (1H, dd, J=2.5 Hz, 7.5 Hz, Ar-H), 8.73-8.74 (1H, m, Ar-H) ppm.

δ ¹³C NMR (125 MHz, DMSO): 128.6, 130.1, 132.8, 135.9, 139.0, 160.9 ppm.

2-Chloropyridine N-Oxide (C_5H_4ClNO)

δ ¹H NMR (500 MHz, CDCl₃): 7.28-7.32 (2H, m, Ar-H), 7.55-7.58 (1H, m, Ar-H), 8.40-8.41 (1H, m, Ar-H) ppm.

δ ¹³C NMR (125 MHz, CDCl₃ w/ DMSO): 123.8, 126.0, 126.9, 140.3, 141.5 ppm.

2-Methylpyridine N-Oxide

δ ¹H NMR (500 MHz, CDCl₃): 2.53 (3H, s, -CH₃), 7.20-7.32 (3H, m, Ar-H), 8.29-8.30 (1H, d, J = 5.5 Hz, Ar-H)

δ ¹³C NMR (125 MHz, CDCl₃): 17.3, 123.2, 125.5, 126.1, 138.8, 148.5 ppm.

3-Bromopyridine N-Oxide

δ ¹H NMR (500 MHz, CDCl₃): 7.21-7.24 (1H, dd, J=6.6, 8.2 Hz, Ar-H), 7.45-7.47 (1H, dq, J=0.8, 8.3 Hz, Ar-H), 8.19-8.21 (1H, dq, J=0.8, 6.5 Hz, Ar-H), 8.39-8.40 (1H, t, J=1.5 Hz, Ar-H) ppm.

δ ¹³C NMR (125 MHz, CDCl₃): 120.2, 125.9, 128.7, 137.7, 140.3 ppm.

4-Methylpyridine N-Oxide

δ ^1H NMR (500 MHz, CDCl_3): 2.37 (3H, s, -CH₃), 7.12 (2H, s, Ar-H), 8.13 (2H, s, Ar-H) ppm.

δ ^{13}C NMR (125 MHz, CDCl_3): 20.1, 126.6, 138.0, 138.4 ppm.

Nicotinic N-Oxide

δ ^1H NMR (500 MHz, DMSO): 7.53-7.55 (1H, dd, $J=6.8, 7.5$ Hz, Ar-H), 7.76-7.78 (1H, d, $J=8$ Hz, Ar-H), 8.42-8.44 (1H, dd, $J=0.7, 6.4$ Hz, Ar-H), 8.48 (1H, s, Ar-H) ppm.

δ ^{13}C NMR (125 MHz, DMSO): 126.1, 127.2, 131.1, 139.4, 142.6, 164.7 ppm.

Pyridine N-Oxide

δ ^1H NMR (500 MHz, CDCl_3): 7.35-7.37 (3H, m, Ar-H), 8.25-8.27 (2H, m, Ar-H) ppm.

δ ^{13}C NMR (125 MHz, CDCl_3): 125.3, 125.5, 138.5 ppm.

Quinoline N-Oxide

δ ^1H NMR (500 MHz, CDCl_3): 7.29-7.32 (1H, dd, $J=6.0, 8.5$ Hz, Ar-H), 7.62-7.65 (1H, t, $J=7.5$ Hz, Ar-H), 7.74-7.77 (2H, m, Ar-H), 7.86-7.87 (1H, d, $J=8.5$ Hz, Ar-H), 8.54-8.56 (1H, d, $J=6$ Hz, Ar-H), 8.73-8.75 (1H, d, $J=9$ Hz, Ar-H) ppm.

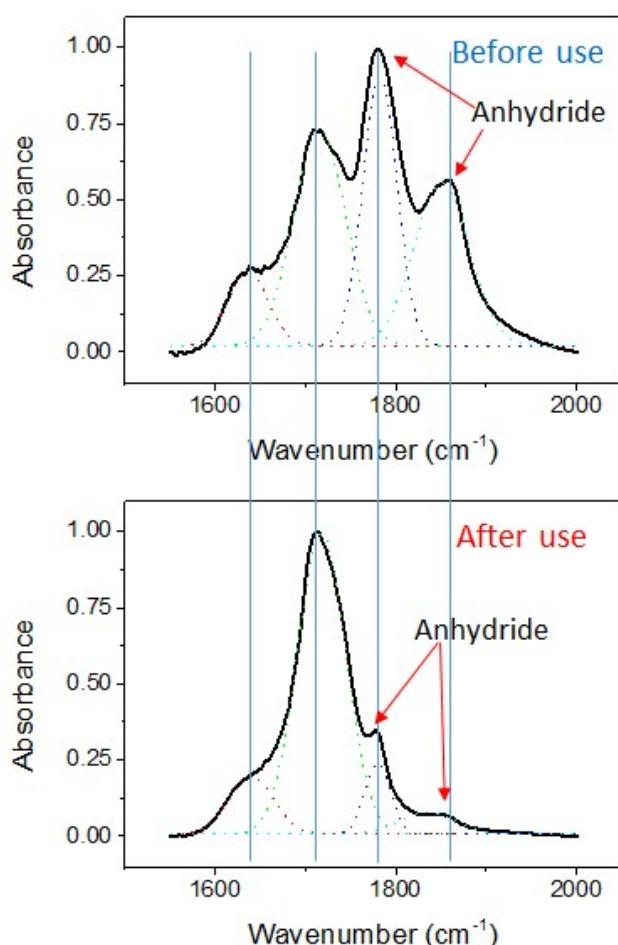
δ ^{13}C NMR (125 MHz, CDCl_3): 119.4, 120.7, 126.2, 127.9, 128.5, 130.2, 130.3, 135.4, 141.1 ppm.

Zinc Pyrithione

δ ^1H NMR (500 MHz, DMSO): 6.99-7.02 (1H, td, $J=2\text{Hz}$, 7Hz, Ar-H), 7.23-7.26 (1H, m, Ar-H), 7.60-7.62 (1H x 2, dd, $J=1.5$, 8.5 Hz, Ar-H), 8.42-8.44 (1H x 2, dd, $J=1.0$, 6.5 Hz, Ar-H) ppm.

δ ^{13}C NMR (125 MHz, DMSO): 117.9, 128.4, 129.3, 137.3, 159.3 ppm.

Figures:



Wavenumber (cm^{-1})	Area change (normalized based on 1637 cm^{-1})	
	Before	After
1637	0.38	0.38
1716	1.00	1.81
1780	0.86	0.29 (34% of 0.86)
1859	0.84 (1.0)	0.23 (27% of 0.84)

Figure S1. IR spectrum of Od-MA before and after use in oxidation reaction.

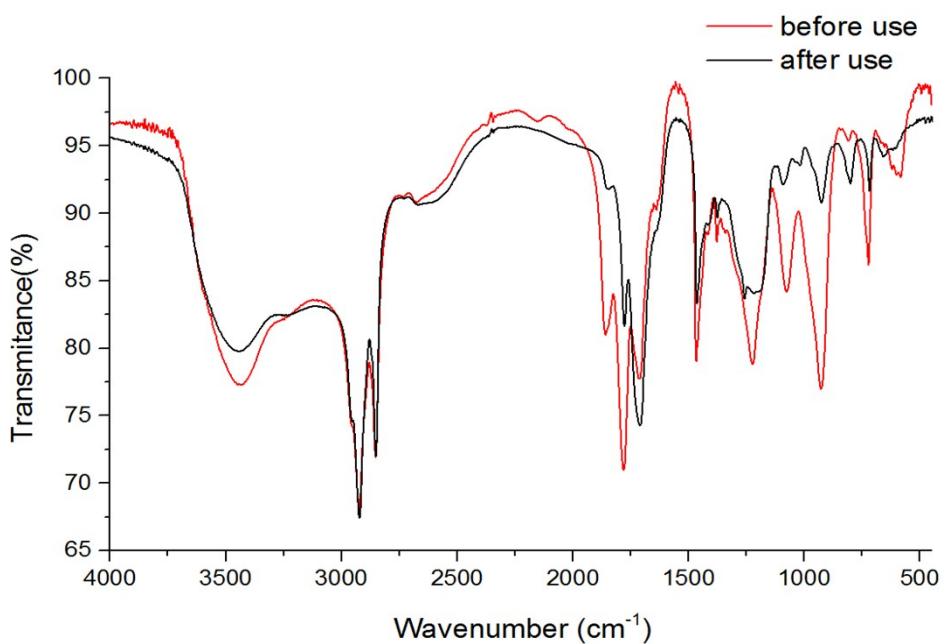


Figure S2. IR spectrum of Od-MA before and after use in oxidation reaction.

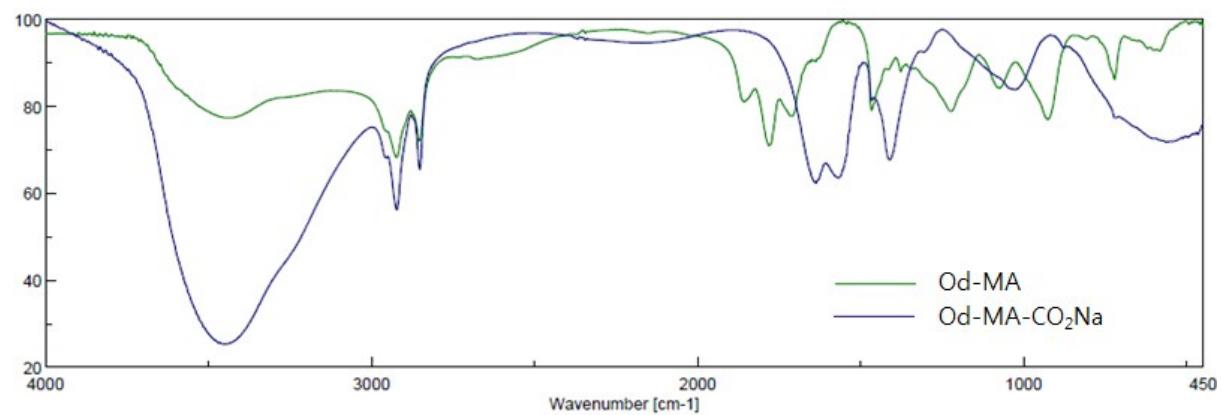


Figure S3. IR spectrum of Od-MA- CO_2Na .

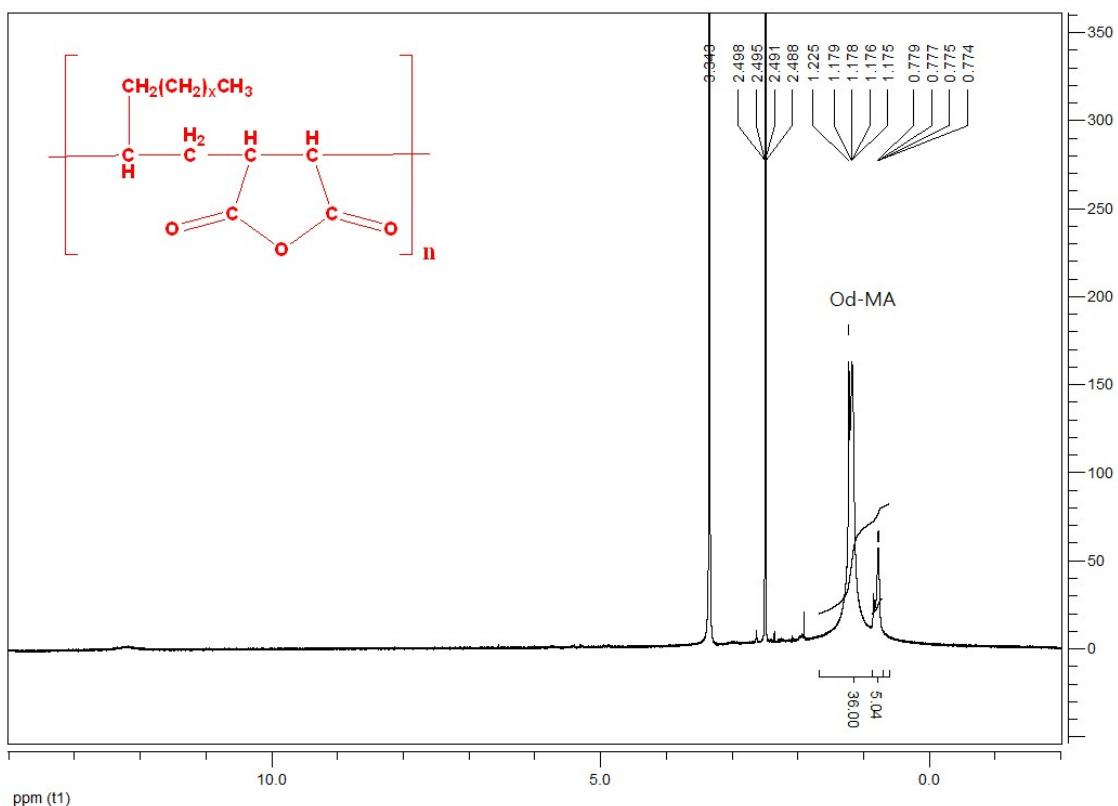


Figure S4. ^1H NMR spectrum of fresh Od-MA.

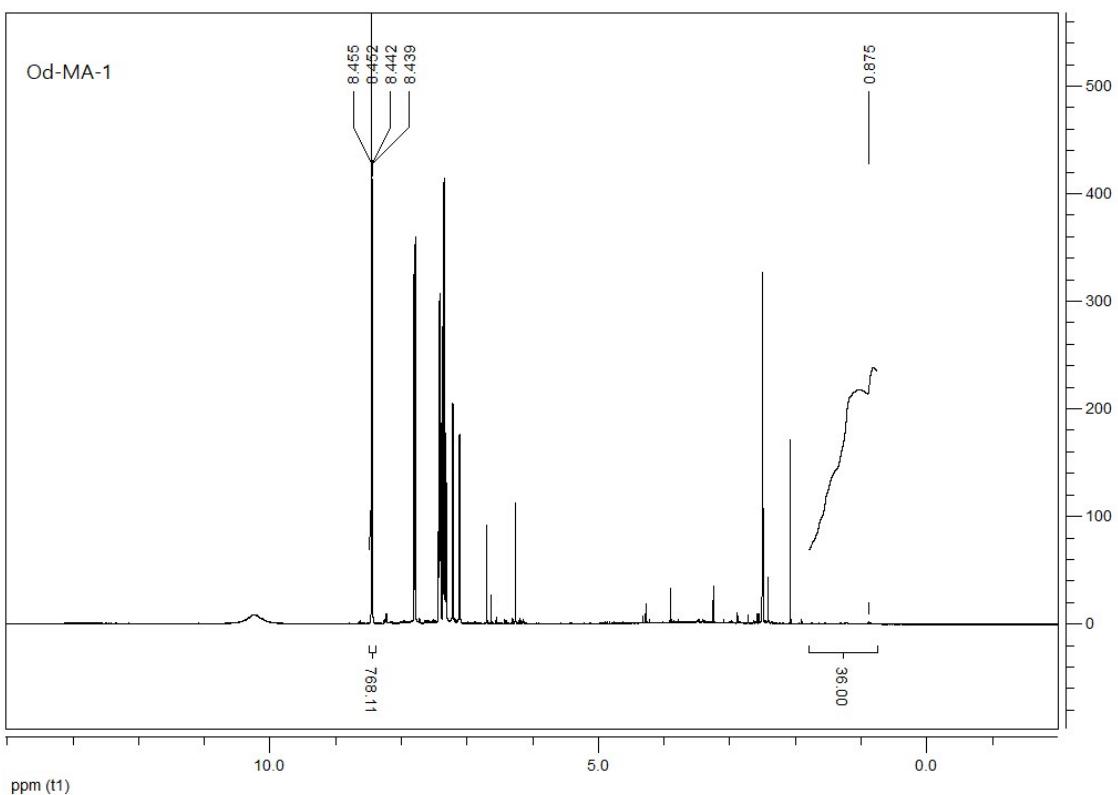


Figure S5. ^1H NMR spectrum of aqueous layer after 1st oxidation reaction using Od-MA.

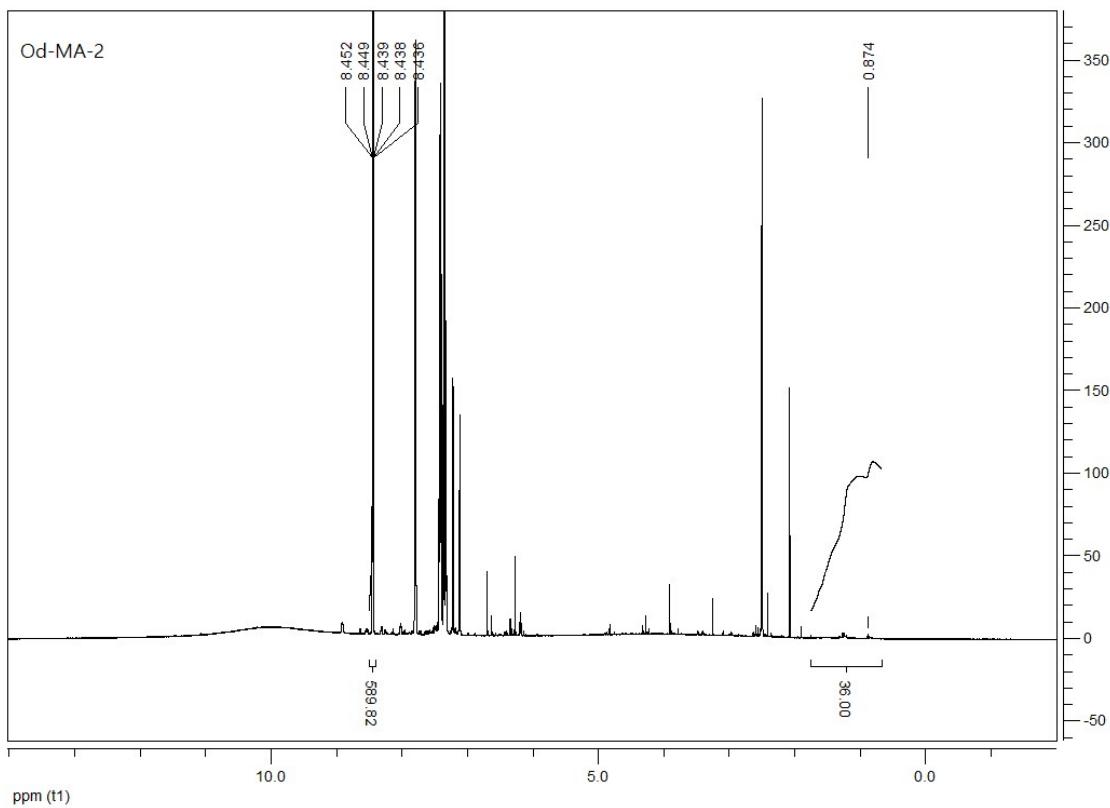


Figure S6. ^1H NMR spectrum of aqueous layer after 2nd oxidation reaction using Od-MA.

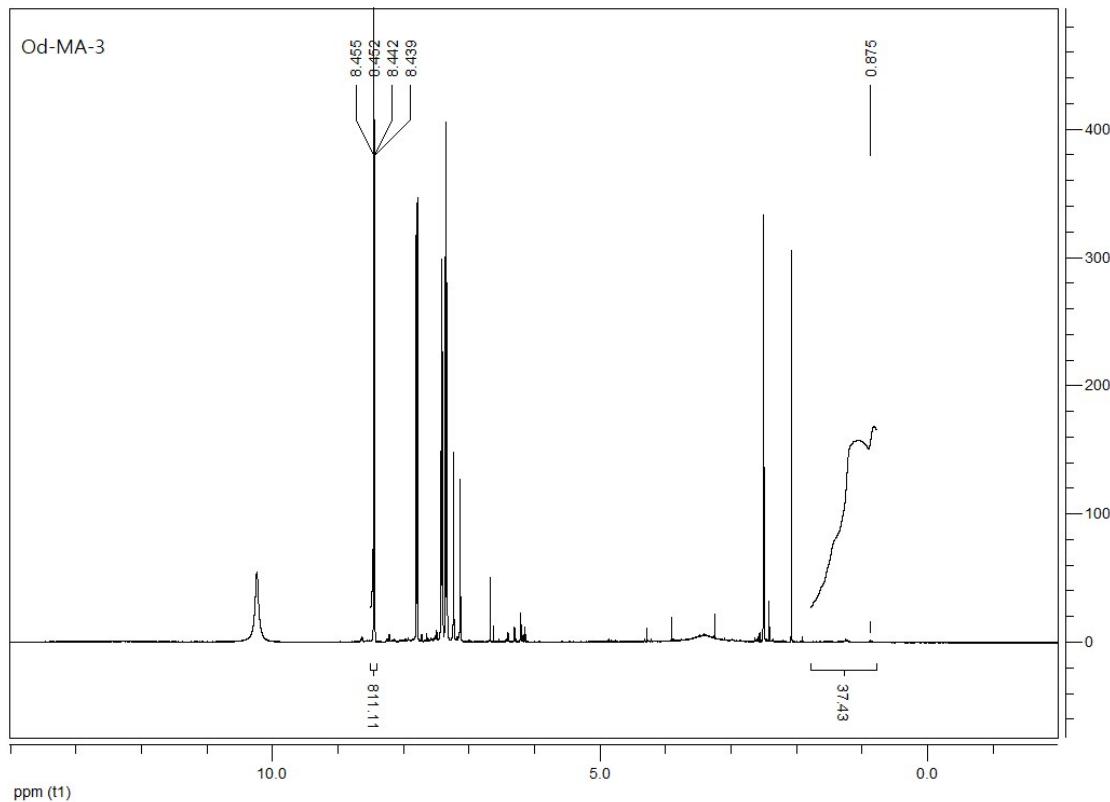


Figure S7. ^1H NMR spectrum of aqueous layer after 3rd oxidation reaction using Od-MA.

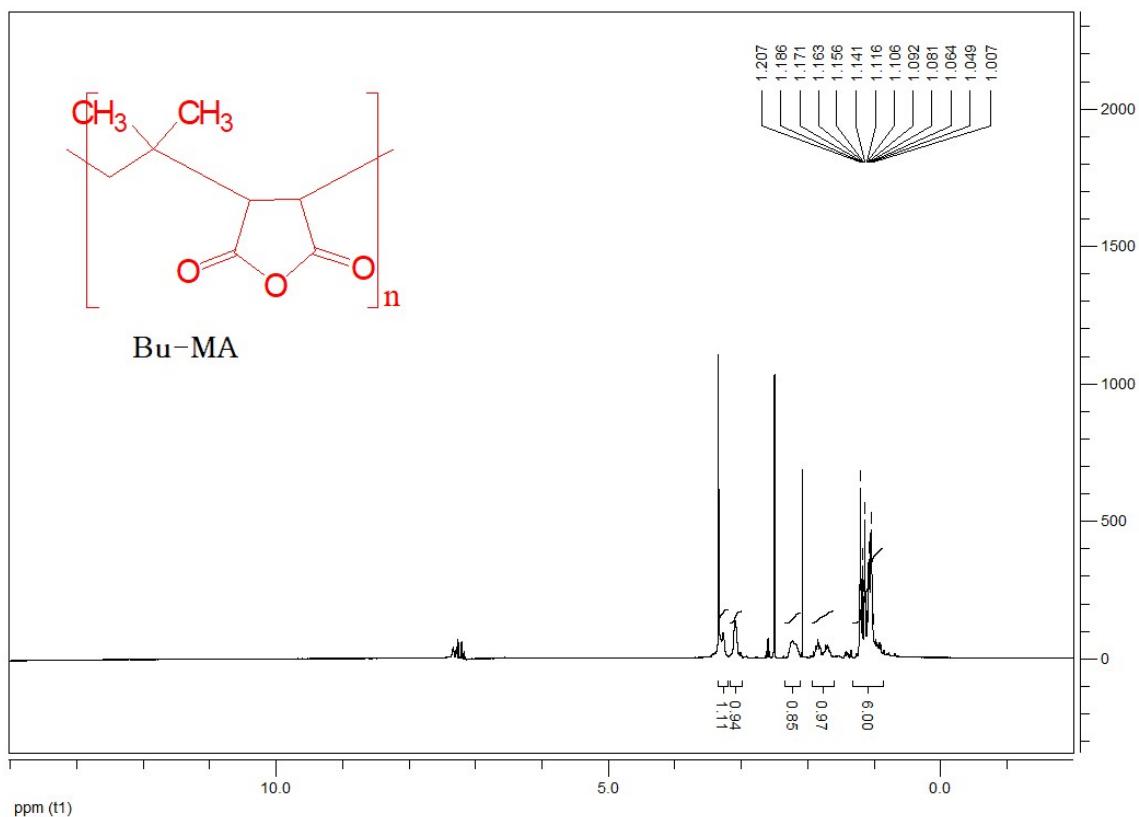


Figure S8. ^1H NMR (DMSO- d_6) of fresh Bu-MA.

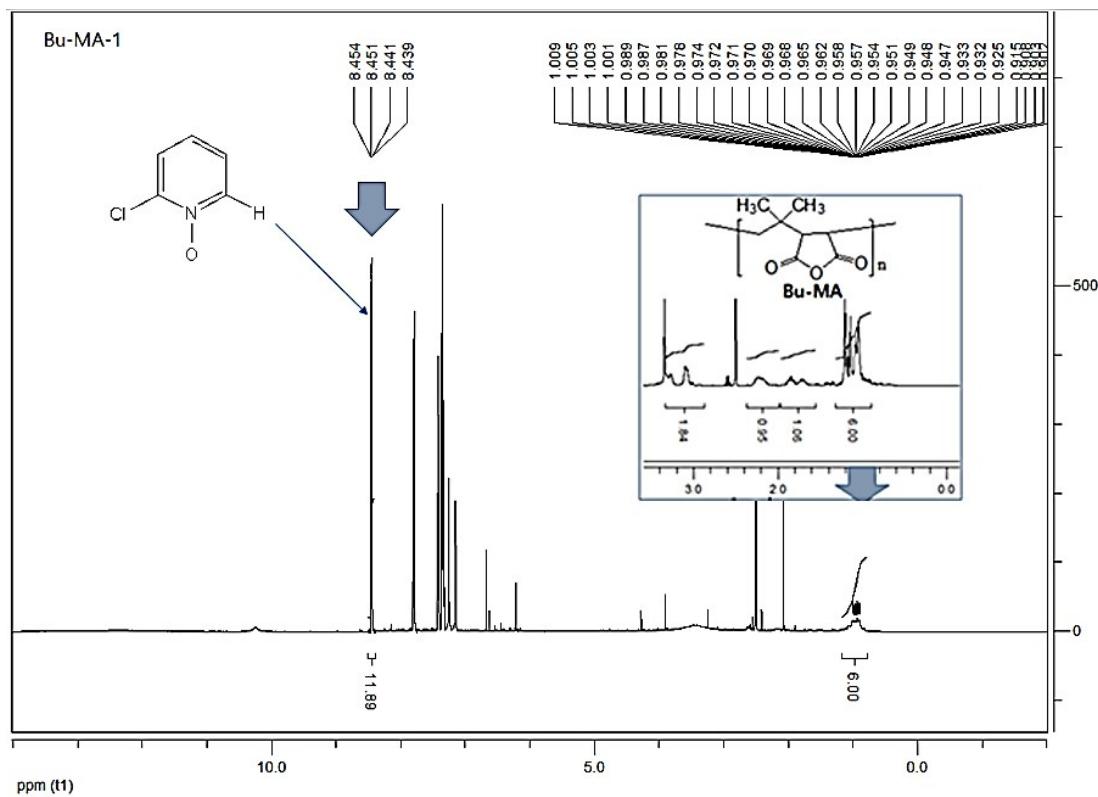


Figure S9. ^1H NMR spectrum of aqueous layer after 1st oxidation reaction using Bu-MA.

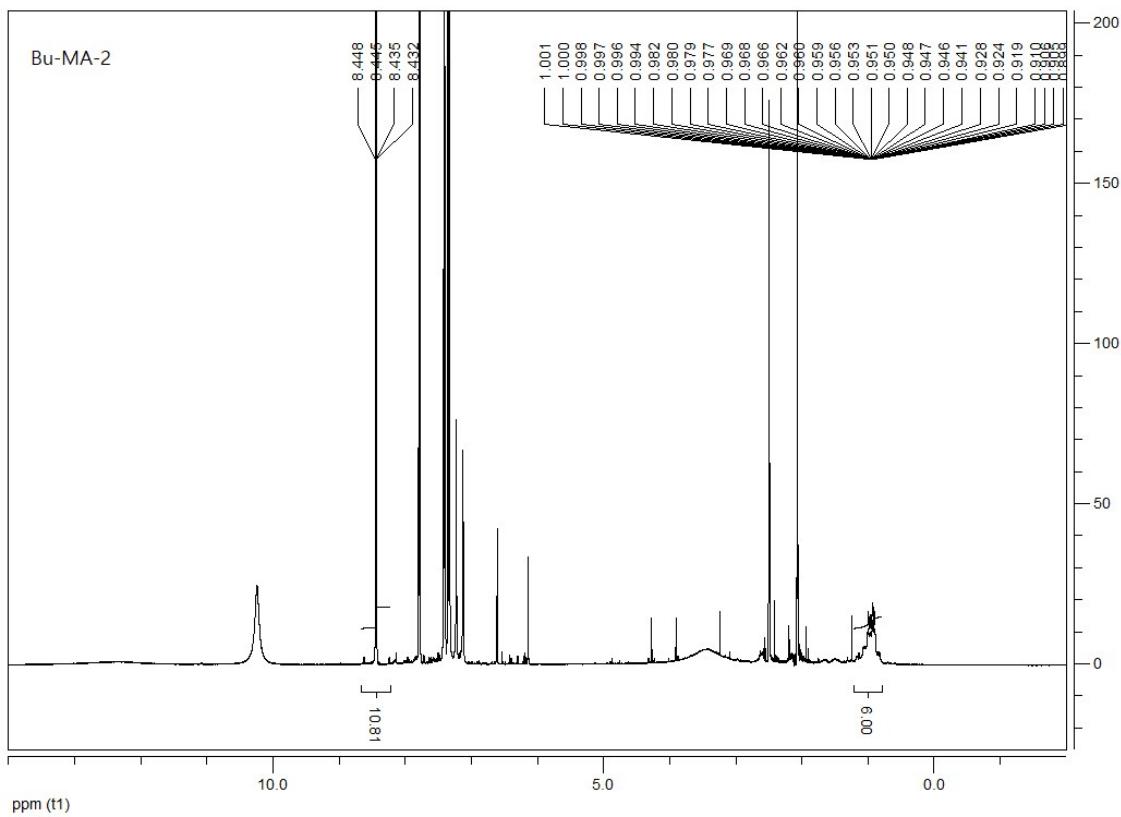


Figure S10. ¹H NMR spectrum of aqueous layer after 2nd oxidation reaction using Bu-MA.

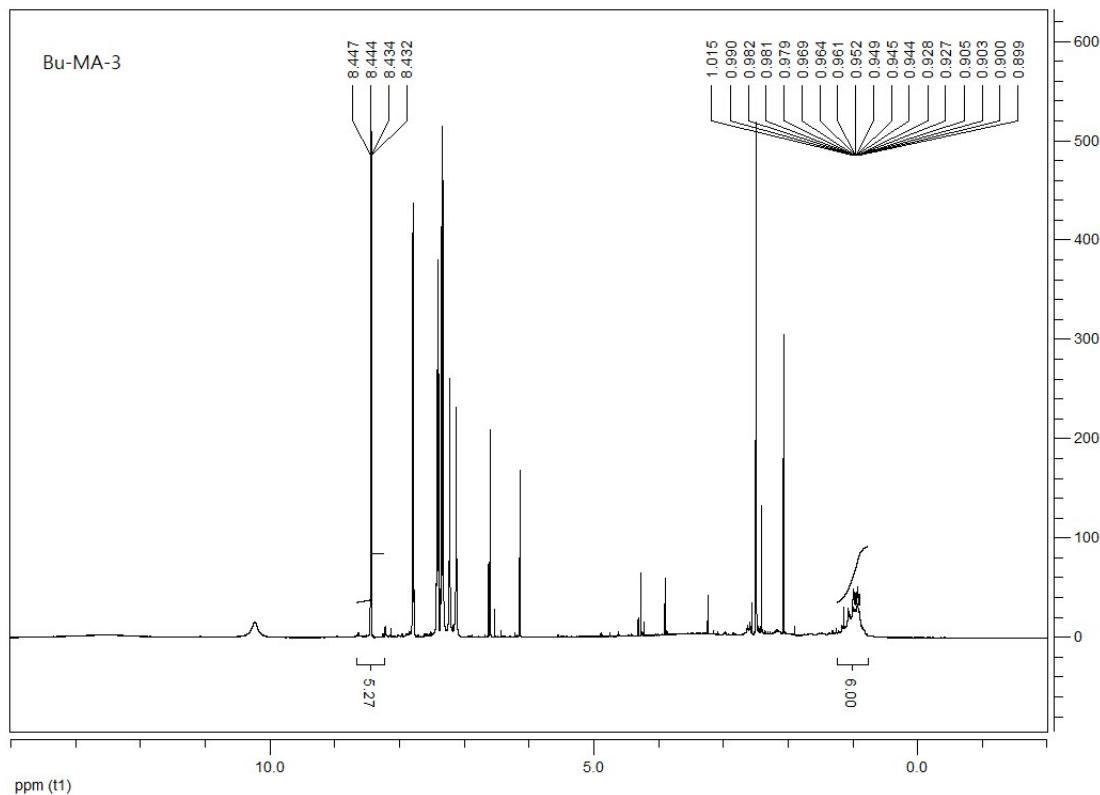


Figure S11. ¹H NMR spectrum of aqueous layer after 3rd oxidation reaction using Bu-MA.

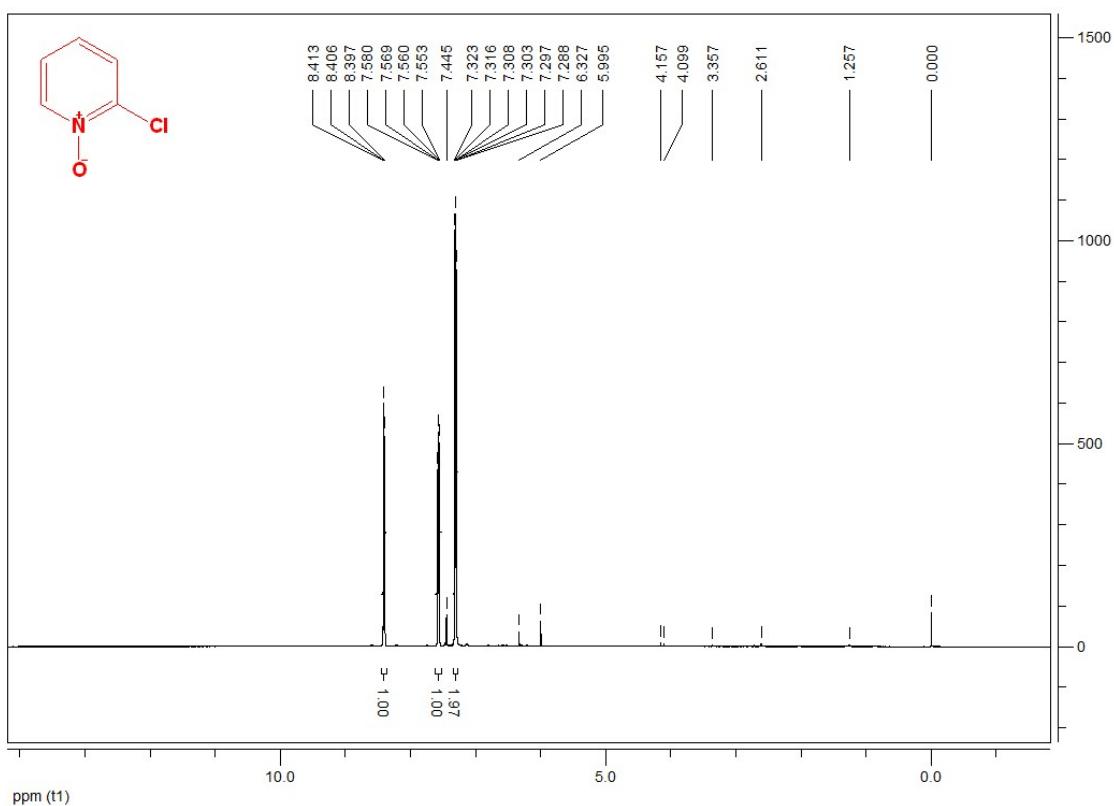


Figure S12. ¹H NMR spectrum of 2-Chloropyridine N-oxide.

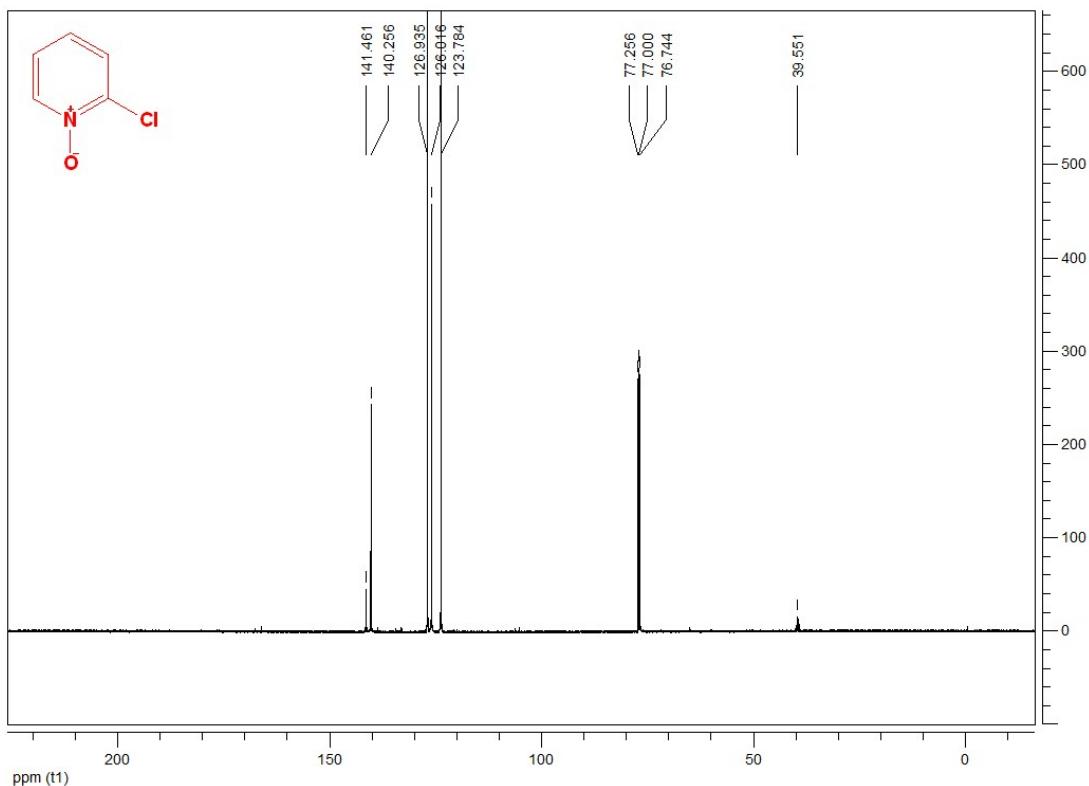


Figure S13. ¹³C NMR of 2-Chloropyridine N-oxide.

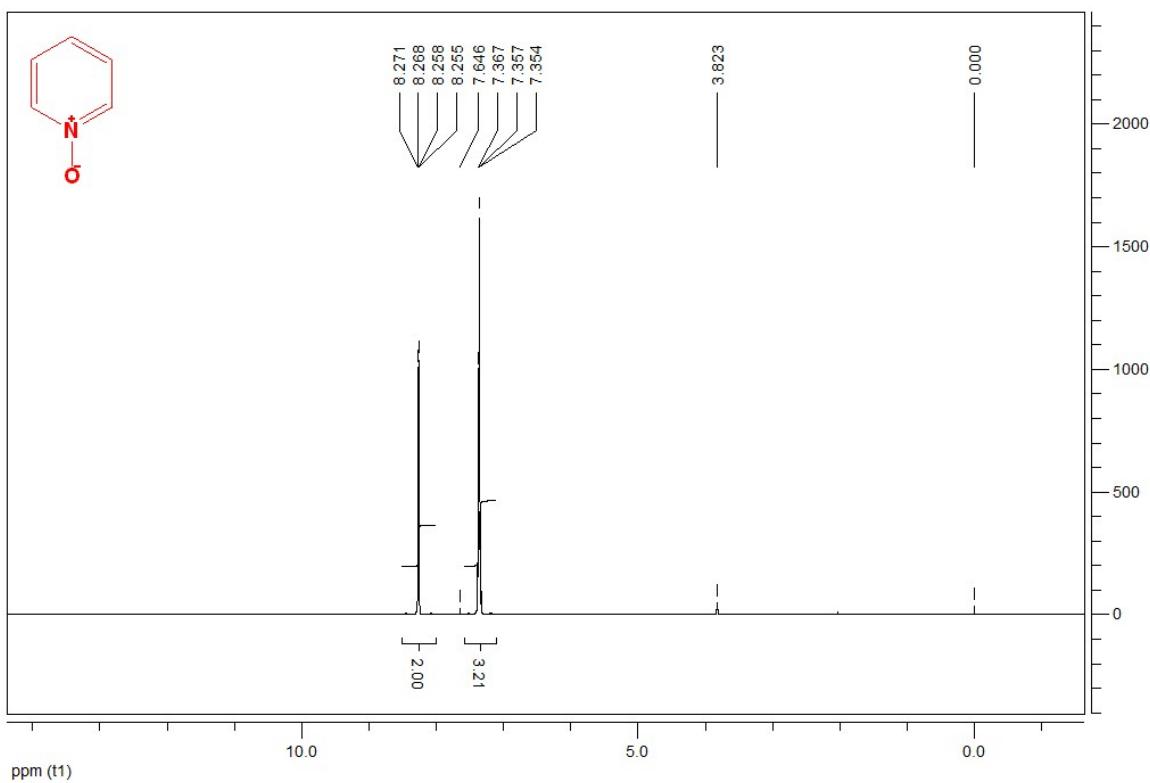


Figure S14. ¹H NMR spectrum of Pyridine N-Oxide.

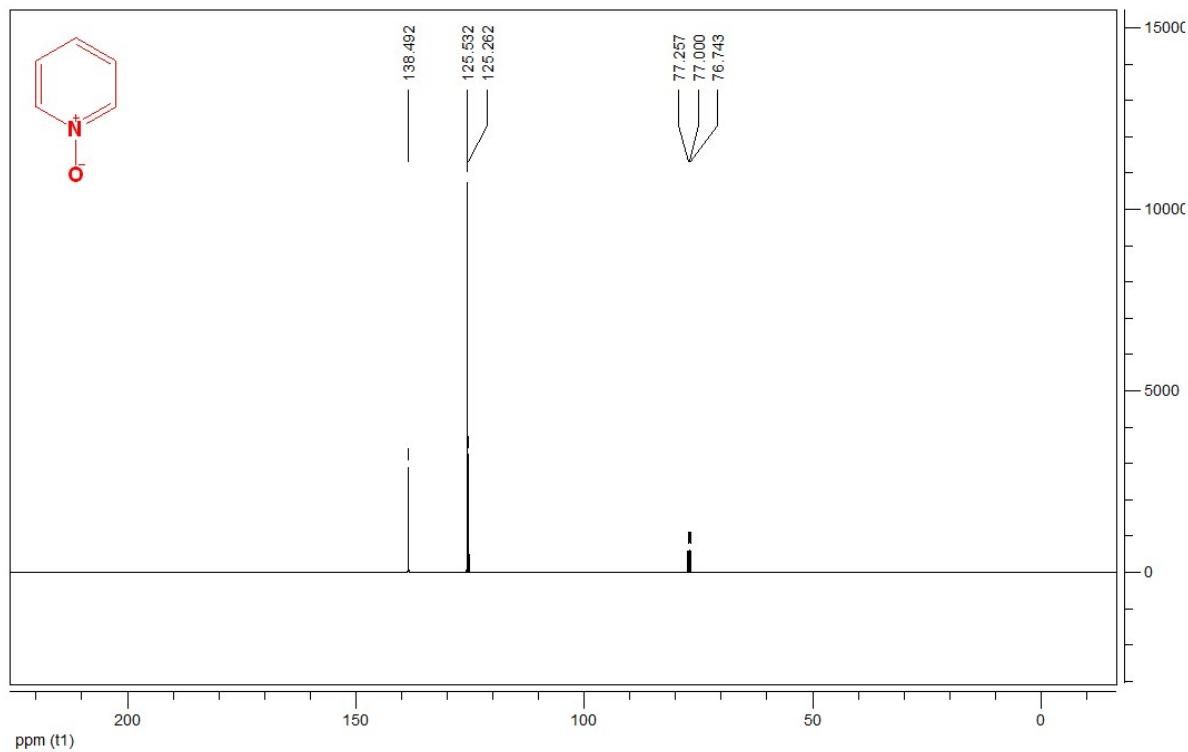


Figure S15. ¹³C NMR spectrum of Pyridine N-Oxide.

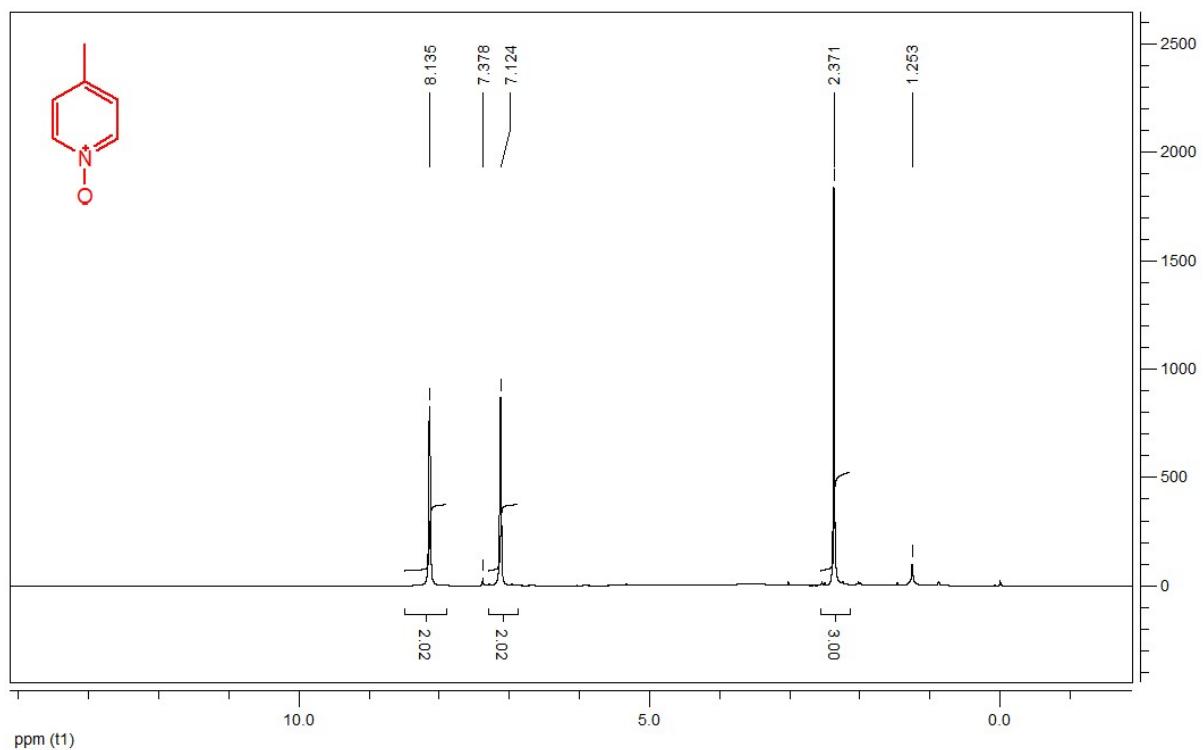


Figure S16. ¹H NMR spectrum of 4-Methylpyridine N-Oxide.

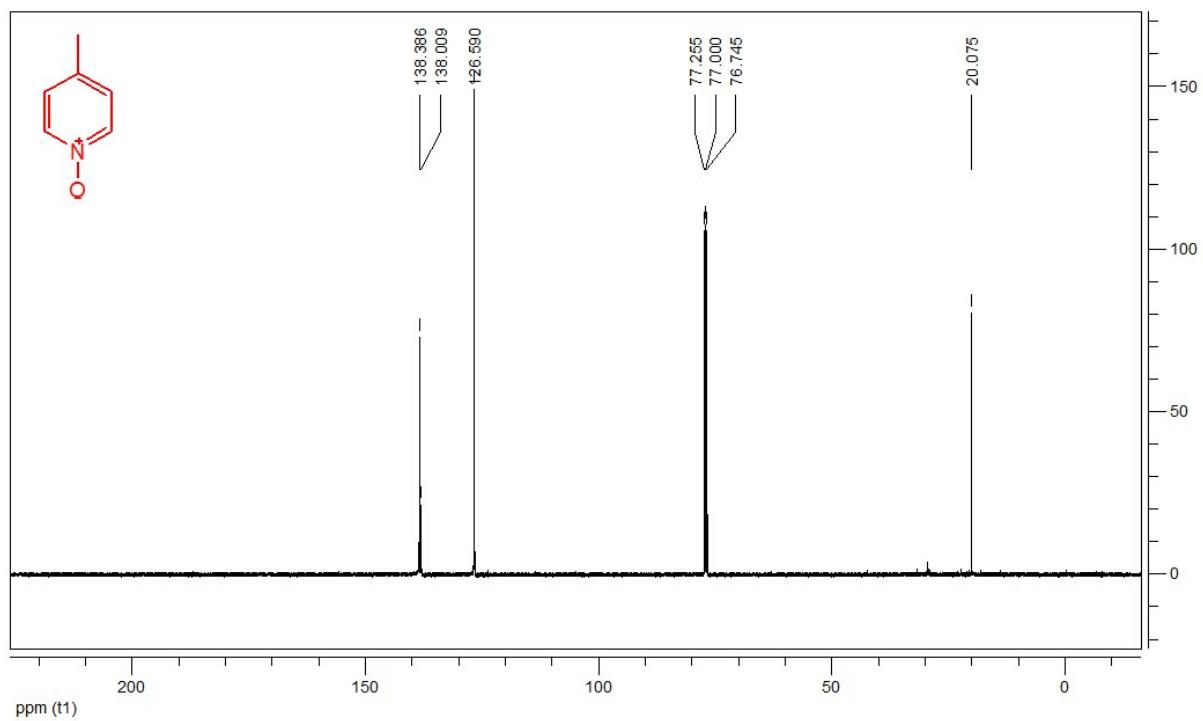


Figure S17. ¹³C NMR spectrum of 4-Methylpyridine N-Oxide.

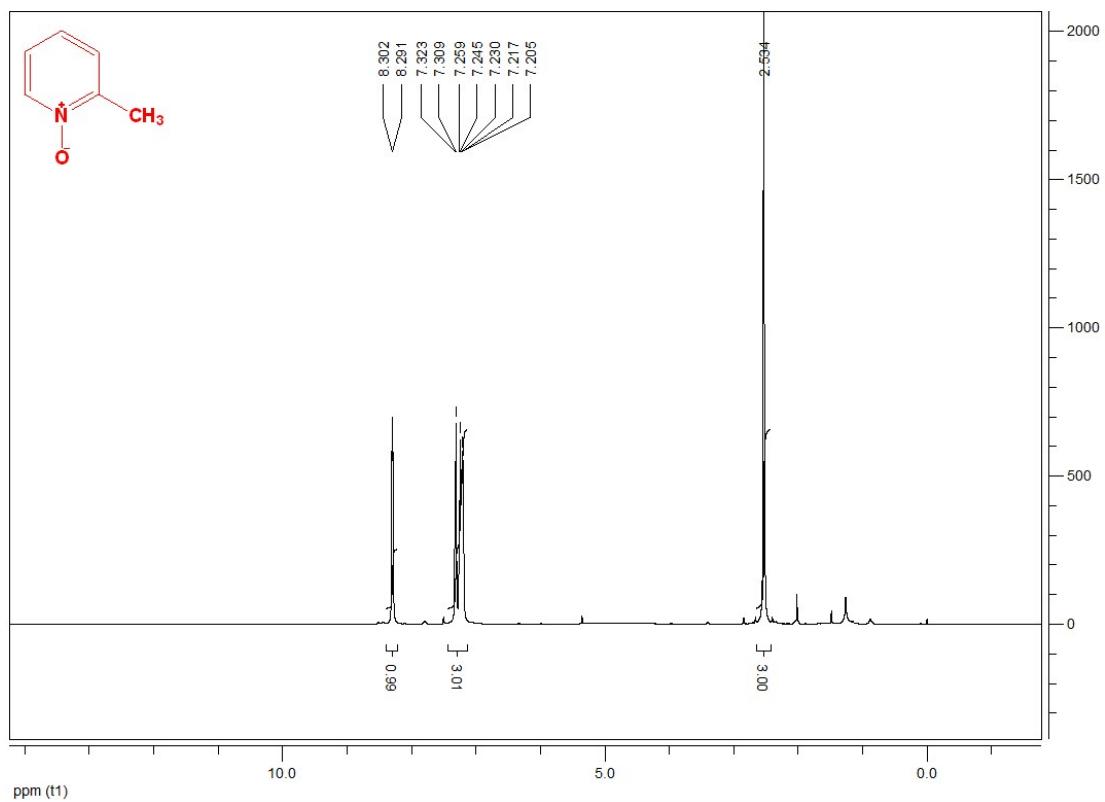


Figure S18. ¹H NMR spectrum of 2-Methylpyridine N-Oxide.

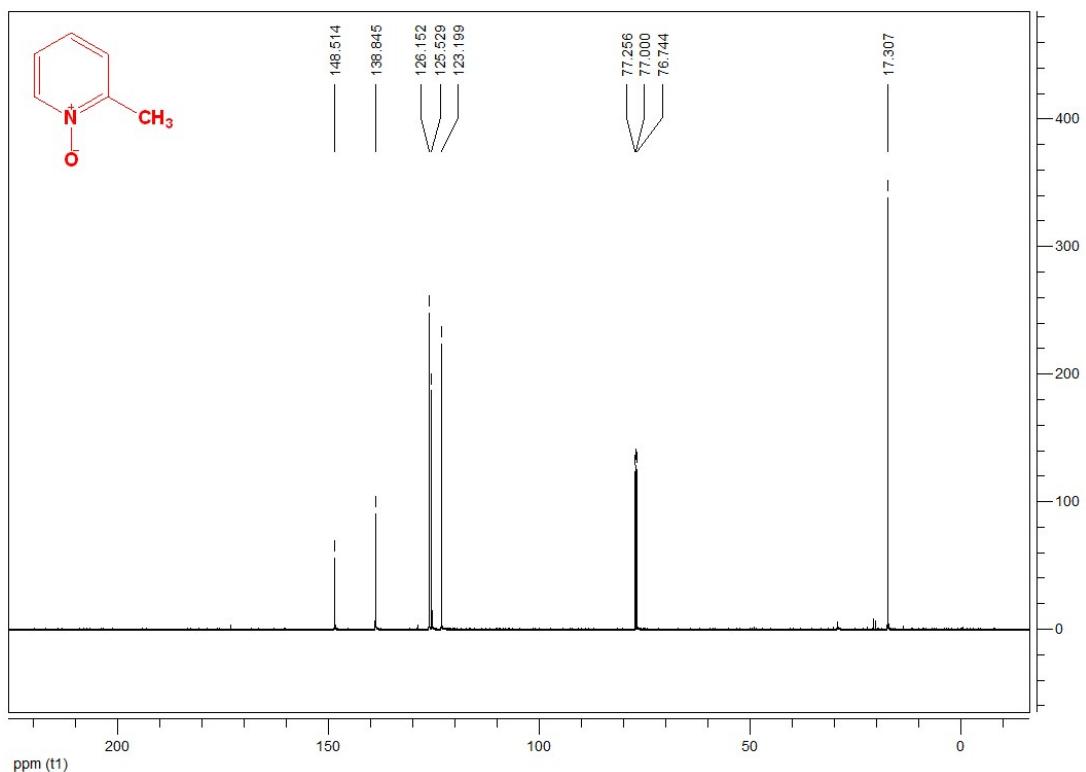


Figure S19. ¹³C NMR spectrum of 2-Methylpyridine N-Oxide.

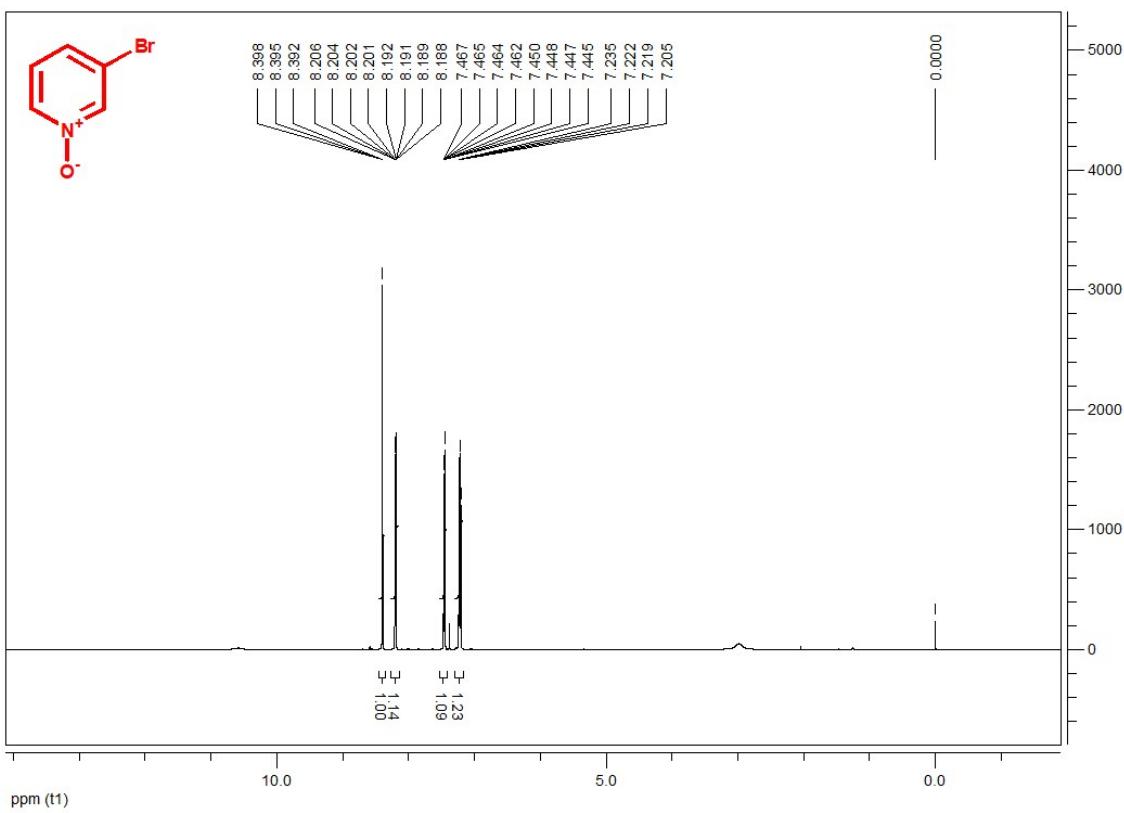


Figure S20. ¹H NMR spectrum of 3-Bromopyridine N-Oxide.

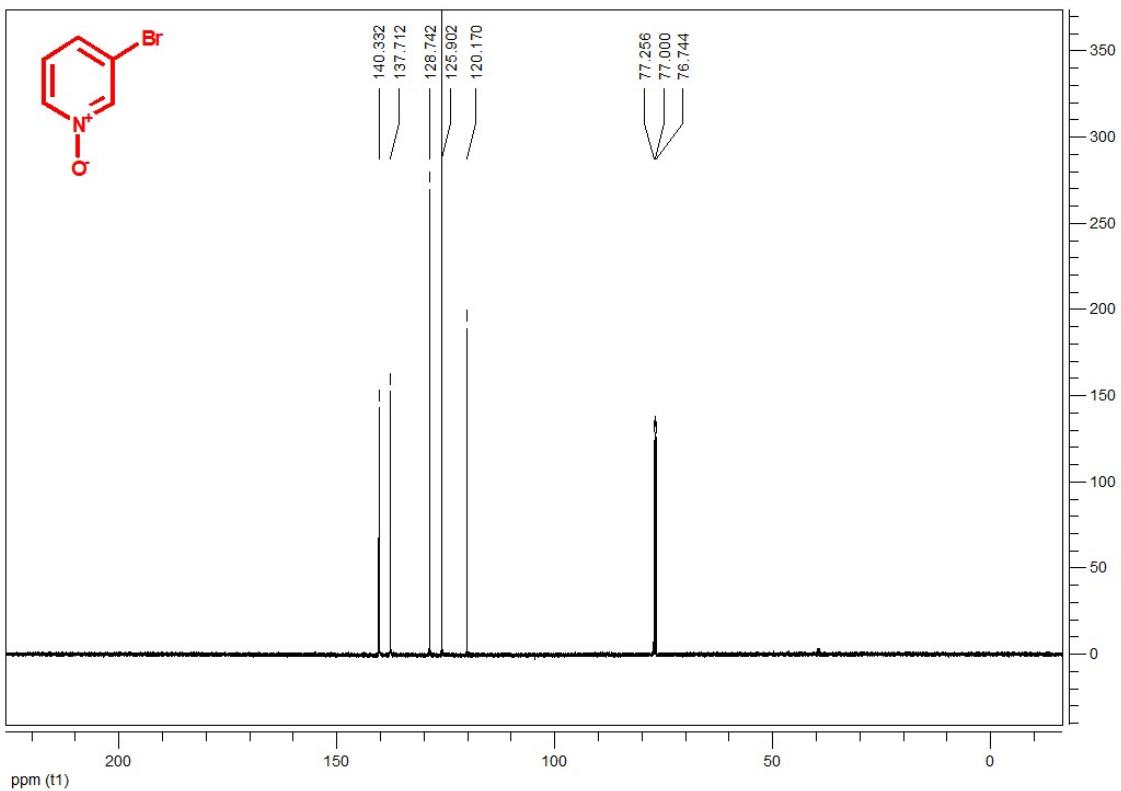


Figure S21. ¹³C NMR spectrum of 3-Bromopyridine N-Oxide.

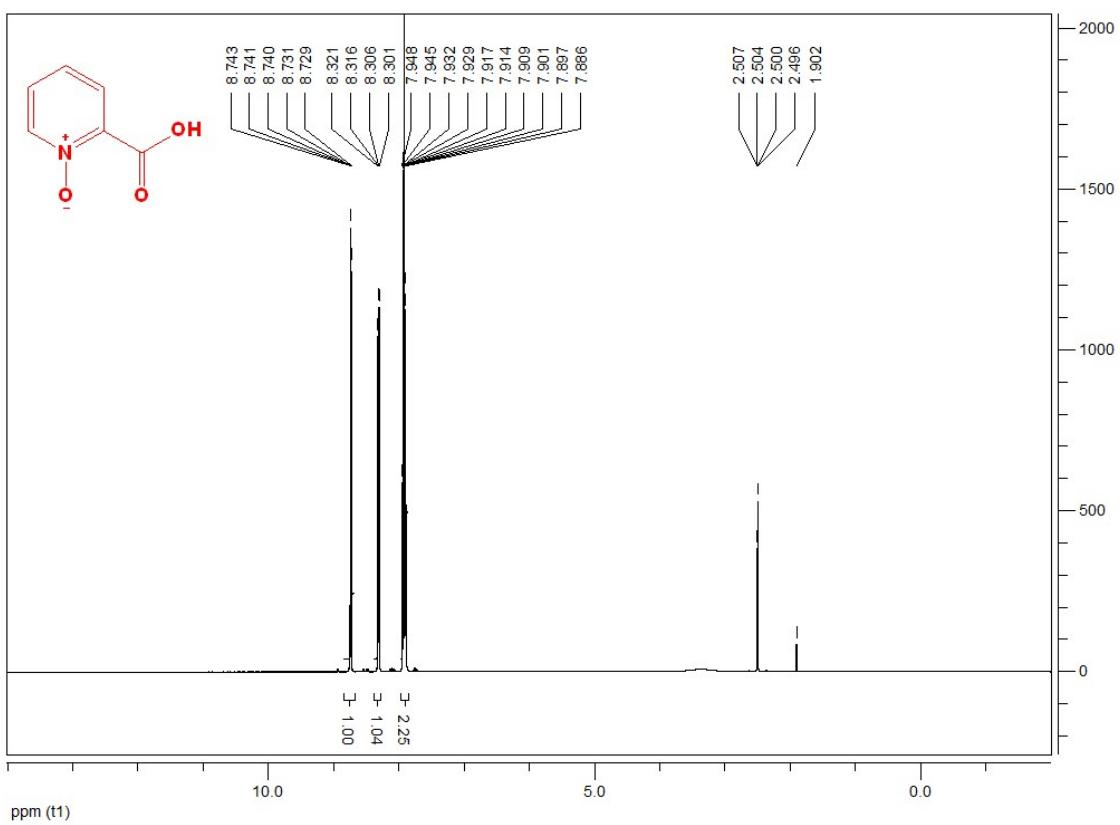


Figure S22. ¹H NMR spectrum of 2-Carboxypyridine N-Oxide.

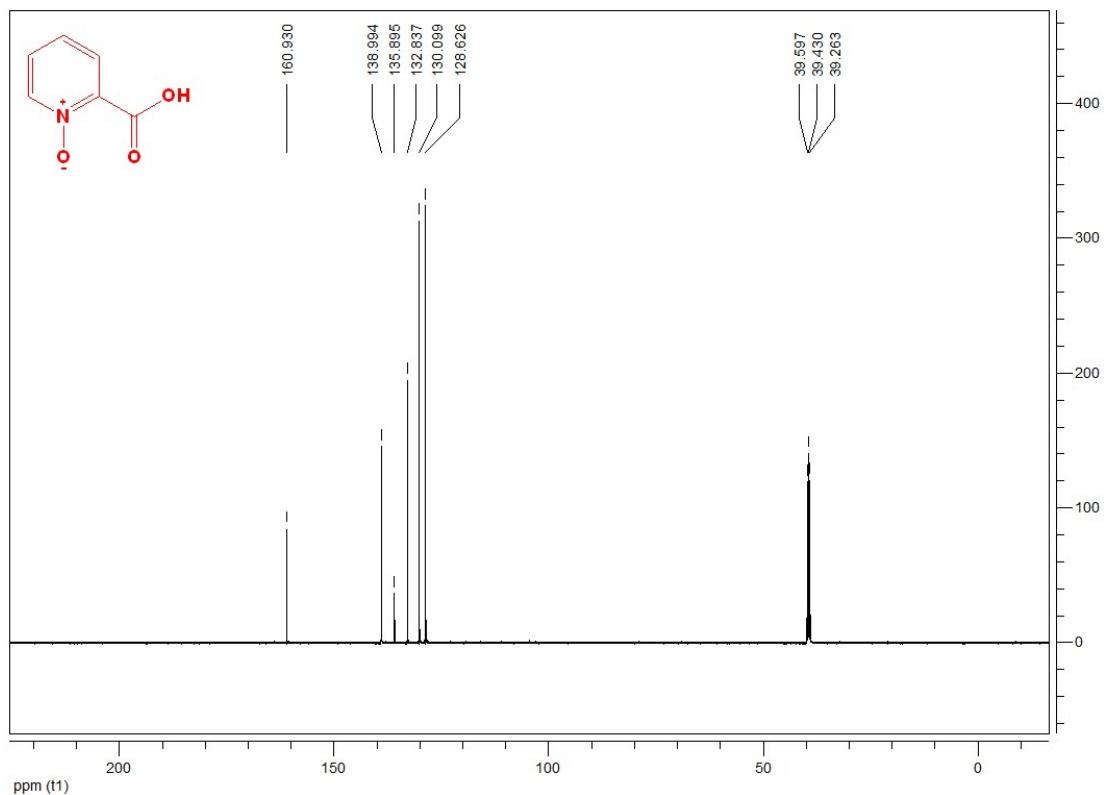


Figure S23. ¹³C NMR spectrum of 2-Carboxypyridine N-Oxide.

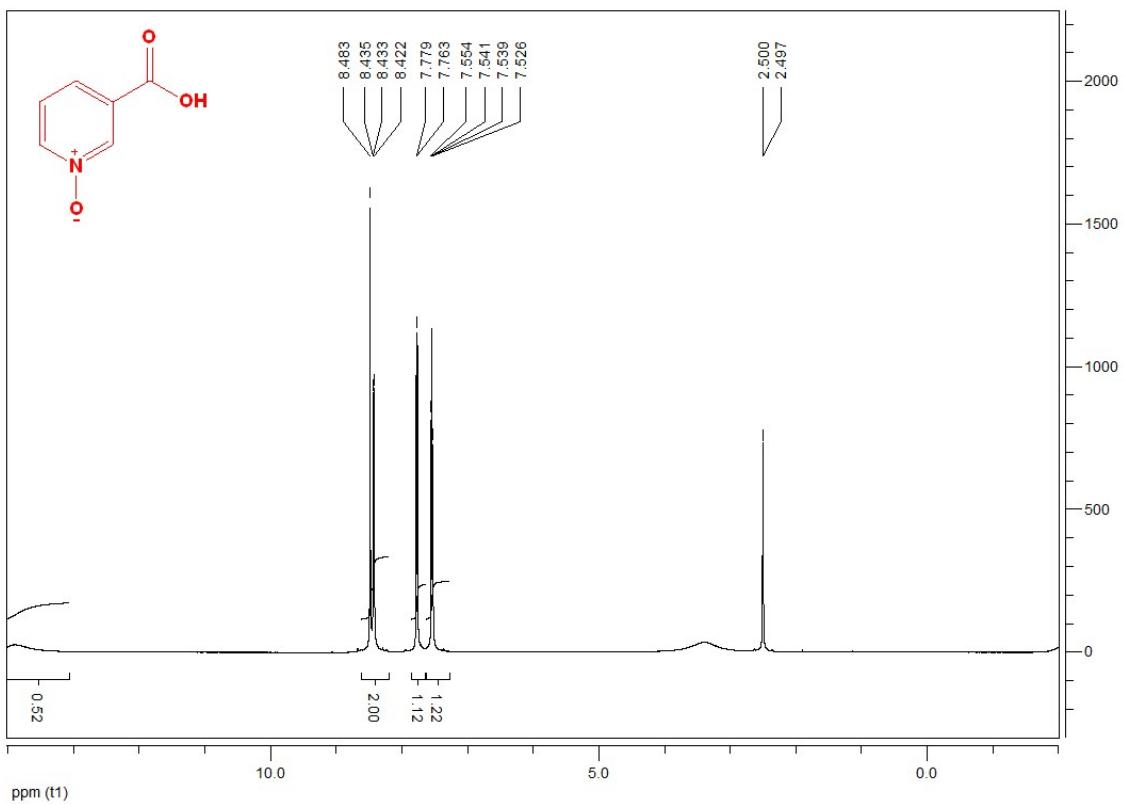


Figure S24. ¹H NMR spectrum of Nicotinic N-Oxide.

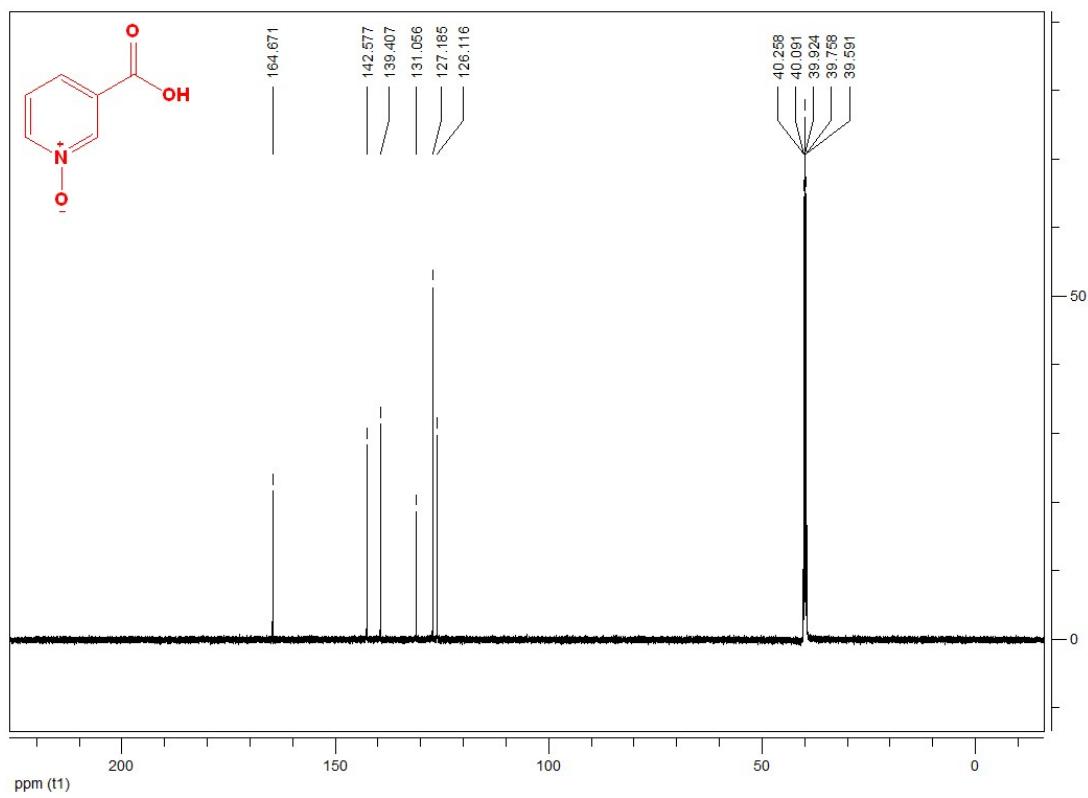


Figure S25. ¹³C NMR spectrum of Nicotinic N-Oxide.

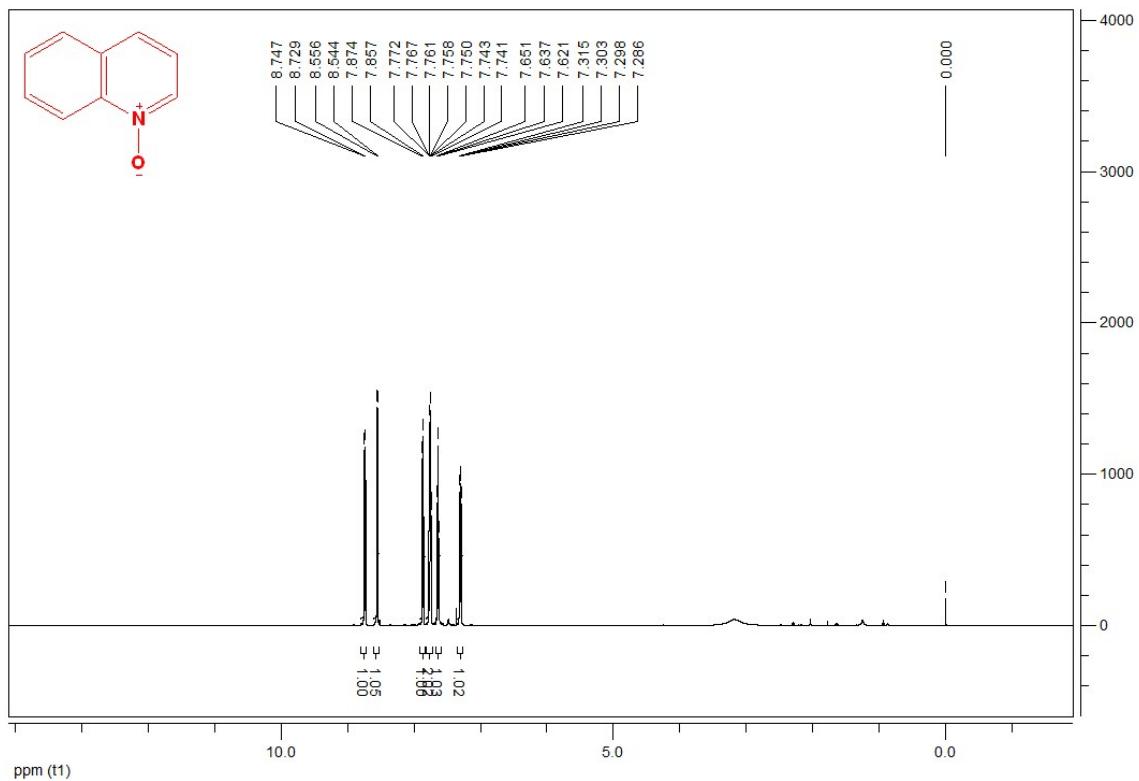


Figure S26. ^1H NMR spectrum of Quinoline N-Oxide.

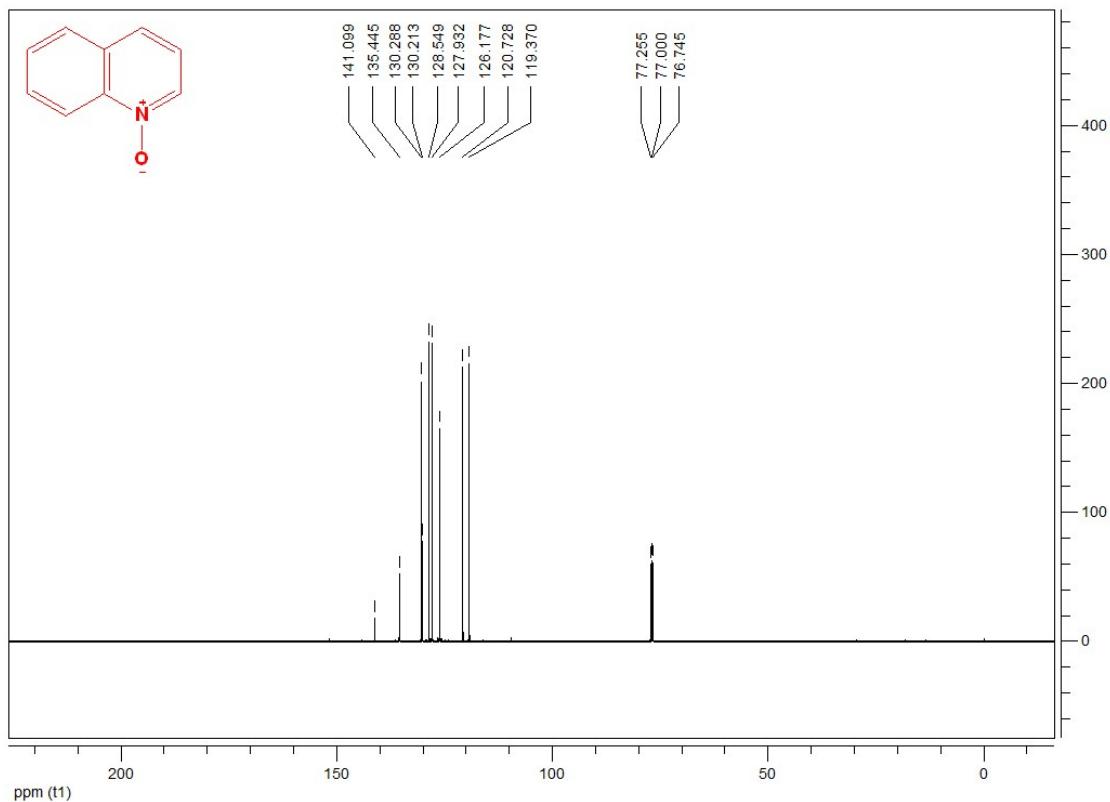


Figure S27. ^{13}C NMR spectrum of Quinoline N-Oxide.

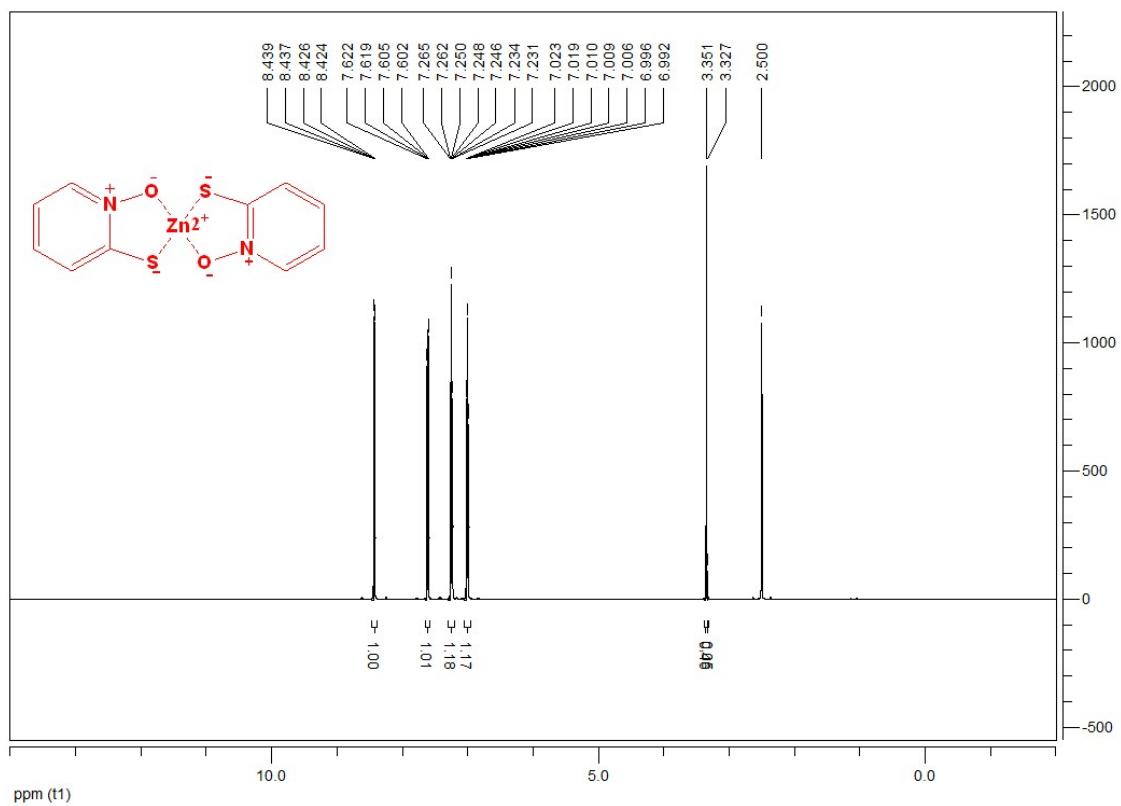


Figure S28. ¹H NMR spectrum of Zinc Pyrithione.

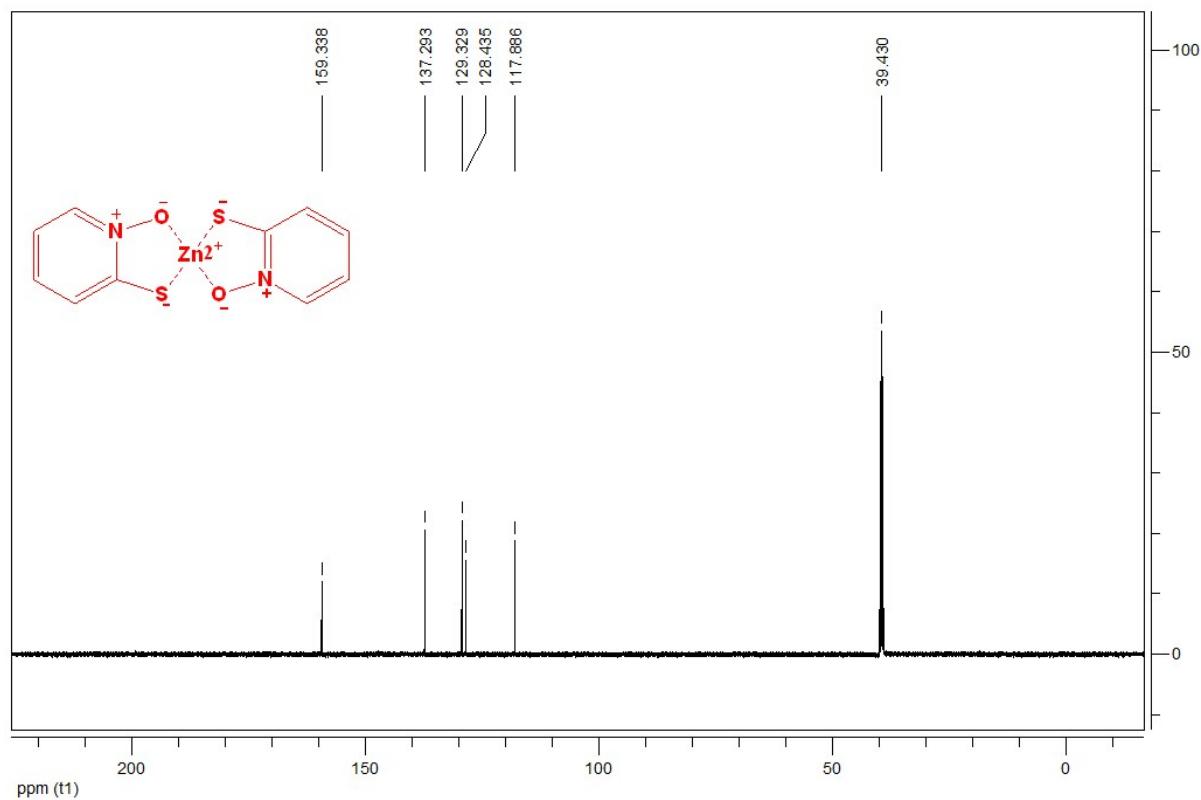


Figure S29. ¹³C NMR spectrum of Zinc Pyrithione.