
Supplementary Materials

Morpholine Radical in the Electrochemical Reaction with Quinoline *N*-Oxide

Egor L. Dolengovski ¹, Tatyana V. Gryaznova ¹, Oleg G. Sinyashin ¹, Elena L. Gavrilova ², Kirill V. Kholin ² and Yulia H. Budnikova ^{1,*}

¹ Arbuzov Institute of Organic and Physical Chemistry, FRC Kazan Scientific Center, Russian Academy of Sciences, 8 Arbuzov Street, Kazan 420088, Russia; dolengovski@gmail.com (E.L.D.); tatyana@iopc.ru (T.V.G.); oleg@iopc.ru (O.G.S.)

² Organic Chemistry Department, Kazan National Research Technological University, 68 Karl Marx Street, Kazan 420015, Russia; gavrilova_elena_@mail.ru (E.L.G.); kholin06@mail.ru (K.V.K.)

* Correspondence: yulia@iopc.ru

General electrolysis procedure

The electrochemical cell equipped with a magnetic stir bar was loaded with 1.38 mmol quinoline *N*-oxide, 0.14 mmol copper acetate, 1.65 (or 4.14) mmol morpholine and 5 mmol K₃PO₄ in acetonitrile (20 ml) at 25°C under argon. The reaction mixture was stirred at room temperature. 2*F* electricity (the electrolysis time is 75 min), or 3*F* (115 min) or 4*F* (150 min) have been passed at 60 mA current (3 mA/cm² current density). At the end of electrolysis precipitation of K₃PO₄ was filtered out, the reaction mixture was evaporated on rotary evaporator, was washed with chloroform. The residue after removal of chloroform was purified by passing through chromatographic column with silica gel (hexane-ethyl acetate eluent).

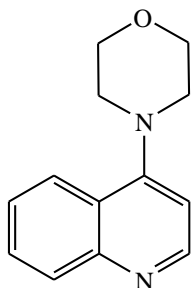
Table S1. Electrochemical cross-coupling products of morpholine and *N*-oxide, 60 mA.

No	N-oxide	Ratio morpholine: N-oxide	Catalyst	Solvent	Base	Number of electrons (Faradays)	Product (yield, %)
1	quinoline <i>N</i> -oxide	1.2:1	Cu(OAc) ₂	CH ₃ CN	-	2	5 (54)
2	quinoline <i>N</i> -oxide	1.2:1	Cu(OAc) ₂	CH ₃ CN	-	3	6 (31)
3	quinoline <i>N</i> -oxide	2.4:1	Cu(OAc) ₂	CH ₃ CN	-	4	4 (48)
4	quinoline <i>N</i> -oxide	1.2:1	Cu(OAc) ₂	CH ₃ CN	K ₃ PO ₄	2	5 (80)
5	quinoline <i>N</i> -oxide	1.2:1	Cu(OAc) ₂	CH ₃ CN	K ₃ PO ₄	3	6 (67)
6	quinoline <i>N</i> -oxide	3:1	Cu(OAc) ₂	CH ₃ CN	K ₃ PO ₄	4	4 (52)
7*	quinoline <i>N</i> -oxide	3:1	Cu(OAc) ₂	CH ₂ Cl ₂	K ₃ PO ₄	4	3 (64)
8	quinoline <i>N</i> -oxide	1.2:1	AgOAc	CH ₃ CN	-	2	5 (32)
9	quinoline <i>N</i> -oxide	1.2:1	AgOAc	CH ₃ CN	-	3	6 (24)
10	quinoline <i>N</i> -oxide	3:1	AgOAc	CH ₃ CN	-	4	4 (46)
11	<i>iso</i> -quinoline <i>N</i> -oxide	1.2:1	Cu(OAc) ₂	CH ₃ CN	-	3	8 (62)
12	pyridine- <i>N</i> -oxide	1.2:1	Cu(OAc) ₂	CH ₃ CN	-	3	not selectively
13	pyridine- <i>N</i> -oxide	1.2:1	Cu(OAc) ₂	CH ₂ Cl ₂	K ₃ PO ₄	3	traces

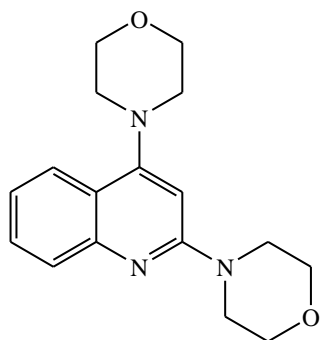
14	phenylpyridine <i>N</i> -oxide	1.2:1	Cu(OAc) ₂	CH ₃ CN	-	3	traces
15**	phenylpyridine <i>N</i> -oxide	1.2:1	Cu(OAc) ₂	CH ₃ CN	K ₃ PO ₄	3	9 (14)

* additive of Et₄NBF₄ (0.02M) was used for better electroconductivity.

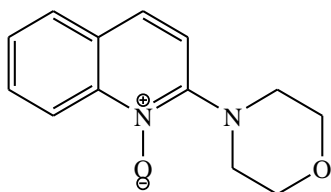
**by Spectrum ¹H NMR spectroscopy and mass-spectrum

4-(Quinolin-4-yl)morpholine (3) [1,2]

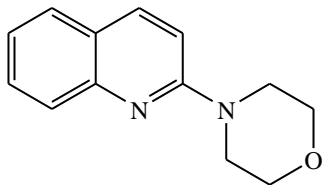
Yellow solid, m.p. 83–85 °C. Yield 0.19 g (64%). ^1H NMR (399.9 MHz, CDCl_3): δ 8.71 (d, J = 8.8 Hz, 1H), 8.06 (d, J = 8.0 Hz, 1H), 7.85 (d, J = 8.1 Hz, 1H), 7.62 (t, J = 6.9 Hz, 1H), 7.45 (t, J = 7.1 Hz, 1H), 7.29 (d, J = 6.4 Hz, 1H), 4.04 (t, J = 4.7 Hz, 4H), 3.23 (t, J = 4.8 Hz, 4H). ^{13}C NMR (100.6 MHz, CDCl_3), δ ppm: 158.14 (C_i), 149.36 (CH), 141.95 (C_i), 130.95 (CH), 129.25 (CH), 128.63 (CH), 127.29 (C_i), 126.66 (CH), 118.09 (CH), 66.30 (CH_2), 53.85 (CH_2). MS (ESI), m/z : 215.1 [$\text{M} + 1$] $^+$. Anal. calc. (%): C 72.87; H 6.59; N 13.07. $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}$. Found (%): C 72.69; H 6.35; N 13.12.

4,4'-(Quinoline-2,4-diyl)dimorpholine (4) [3]

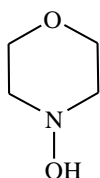
Yellow solid, m.p. 165–167 °C. Yield 0.21 g (52%). ^1H NMR (399.9 MHz, CDCl_3): δ 8.28 (d, J = 7.5 Hz, 1H), 8.08 (m, 2H), 7.79 (d, J = 6.9 Hz, 1H), 7.55 (t, J = 7.1 Hz, 1H), 3.71 (t, J = 4.5 Hz, 4H), 3.67 (t, J = 4.8 Hz, 4H), 3.59 (t, J = 4.8 Hz, 4H), 3.40 (t, J = 4.85 Hz, 4H). ^{13}C NMR (100.6 MHz, CDCl_3), δ ppm: 159.22 (C_i), 152.74 (C_i), 141.32 (C_i), 129.83 (CH), 128.86 (CH), 128.77 (CH), 123.98 (C_i), 123.37 (CH), 109.47 (CH), 61.32. (CH_2), 61.07 (CH_2), 52.80 (CH_2), 52.74 (CH_2). MS (ESI), m/z : 300.2 [$\text{M} + 1$] $^+$. Anal. calc. (%): C 68.20; H 7.07; N 14.04. $\text{C}_{17}\text{H}_{21}\text{N}_3\text{O}_2$. Found (%): C 68.01; H 6.85; N 14.14.

2-Morpholinoquinoline 1-oxide (5) [4,5]

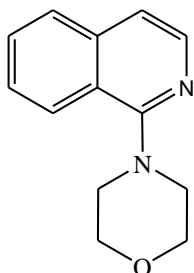
Yellow solid, m.p. 126–128 °C. Yield 0.25 g (80%). ^1H NMR (399.9 MHz, CDCl_3): δ 8.82 (d, J = 8.6 Hz, 1H), 8.39 (d, J = 6.1 Hz, 1H), 7.95 (d, J = 8.1 Hz, 1H), 7.86–7.82 (m, 2H), 7.55 (t, J = 8.1 Hz, 1H), 3.92 (t, J = 4.7 Hz, 4H), 3.56 (t, J = 4.7 Hz, 4H). ^{13}C NMR (100.6 MHz, CDCl_3), δ ppm: 152.19 (C_i), 141.92 (C_i), 130.91 (CH), 129.21 (CH), 128.59 (CH), 126.63 (CH), 124.13 (C_i), 120.12 (CH), 118.51 (CH), 68.44 (CH_2), 46.85 (CH_2). MS (ESI), m/z : 231.1 [$\text{M} + 1$] $^+$. Anal. calc. (%): C 67.81; H 6.13; N 12.17; $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}_2$; Found (%): C 67.92; H 6.25; N 12.12.

2-Morpholinoquinoline (6) [6]

Pale yellow solid, m.p. 87–89 °C. Yield 0.19g (67%). ^1H NMR (399.9 MHz, CDCl_3): δ 8.18 (d, J = 8.68 Hz, 1H), 7.99 (d, J = 6.0 Hz, 1H), 7.75 (d, J = 6.0 Hz, 1H), 7.53 (t, J = 7.9 Hz, 1H), 7.40 (d, J = 8.0 Hz, 1H), 6.92 (d, J = 7.6 Hz, 1H), 3.86 (t, J = 4.6 Hz, 4H), 3.68 (t, J = 4.6 Hz, 4H). ^{13}C NMR (100.6 MHz, CDCl_3), δ ppm: 154.14 (C_i), 141.90 (C_i), 136.06 (CH), 129.19 (CH), 128.57 (CH), 124.11 (C_i), 121.41 (CH), 120.10 (CH), 110.24 (CH), 66.43 (CH_2), 45.56 (CH_2). MS (ESI), m/z : 215.3 $[\text{M} + 1]^+$. Anal. calc. (%): C 72.87; H 6.59; N 13.07; $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}$; Found (%): C 72.76; H 6.48; N 13.10.

Morpholin-4-ol (7) [7]

^1H NMR (399.9 MHz, CDCl_3): δ 8.08 (s, 1H), 3.70 (t, J = 4.7 Hz, 2H), 3.66 (t, J = 4.6 Hz, 2H), 3.58 (t, J = 4.6 Hz, 4H), 3.68 (t, J = 4.7 Hz, 4H). MS (ESI), m/z : 104.1 $[\text{M} + 1]^+$. Anal. calc. (%): C 46.59; H 8.80; N 13.58. $\text{C}_4\text{H}_9\text{NO}_2$; Found (%): C 46.77; H 8.49; N 13.45.

4-(Isoquinolin-1-yl)morpholine (8) [8]

White solid, m.p. 85–88 °C. Yield 0.18g (62%). ^1H NMR (399.9 MHz, CDCl_3): δ 8.22 (d, J = 8.4Hz, 1H), 8.14 (t, J = 6.1 Hz, 1H), 7.94–7.88 (m, 2H), 7.78 (t, J = 7.3 Hz, 1H), 7.49 (d, J = 6.6 Hz, 1H), 4.03 (m, 4H), 3.88 (m, 4H). ^{13}C NMR (100.6 MHz, CDCl_3), δ ppm: 159.69 (C_i), 139.69 (C_i), 137.38 (CH), 130.13 (CH), 127.30 (CH), 126.28 (CH), 125.09 (C_i), 122.74 (CH), 117.05 (CH), 68.01 (CH_2), 52.35 (CH_2). MS (ESI), m/z : 215.1 $[\text{M} + 1]^+$. Anal. calc. (%): C 72.87; H 6.59; N 13.07; $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}$; Found (%): C 72.69; H 6.42; N 13.15.

References

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Copies of ^1H , ^{13}C NMR Spectra for the Products

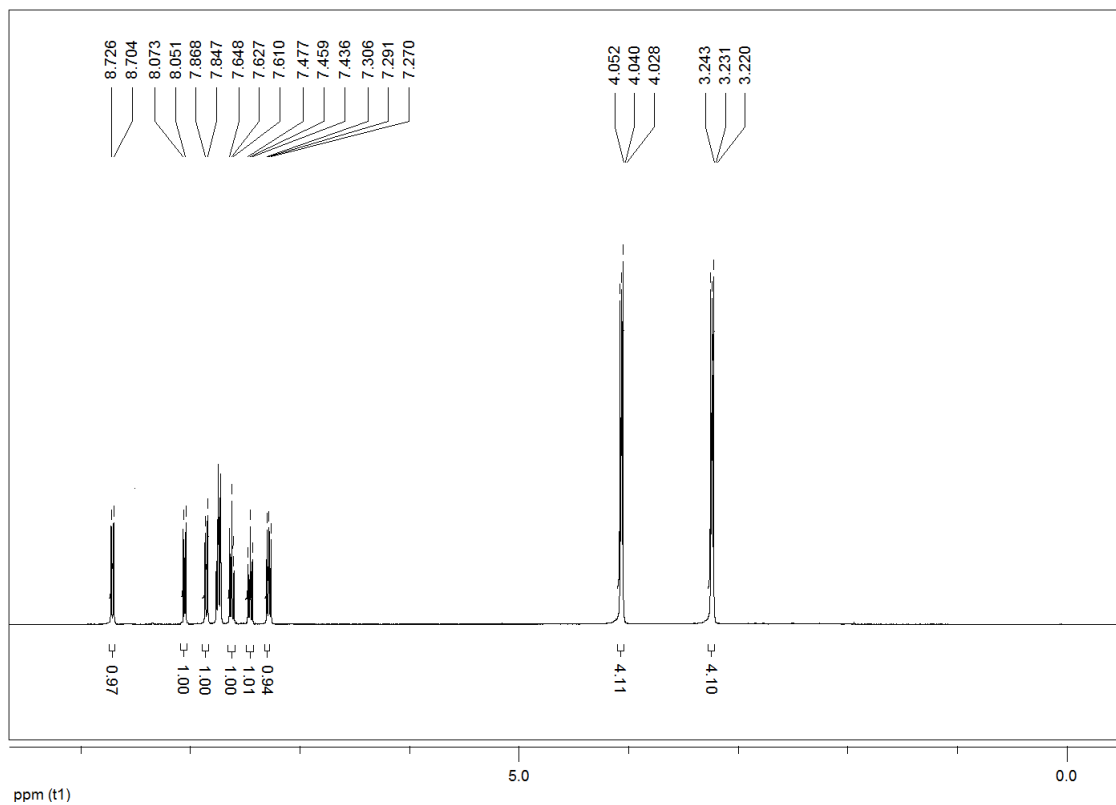


Figure S1. Spectrum ^1H NMR of 4-(Quinolin-4-yl)morpholine (3) in CDCl_3 .

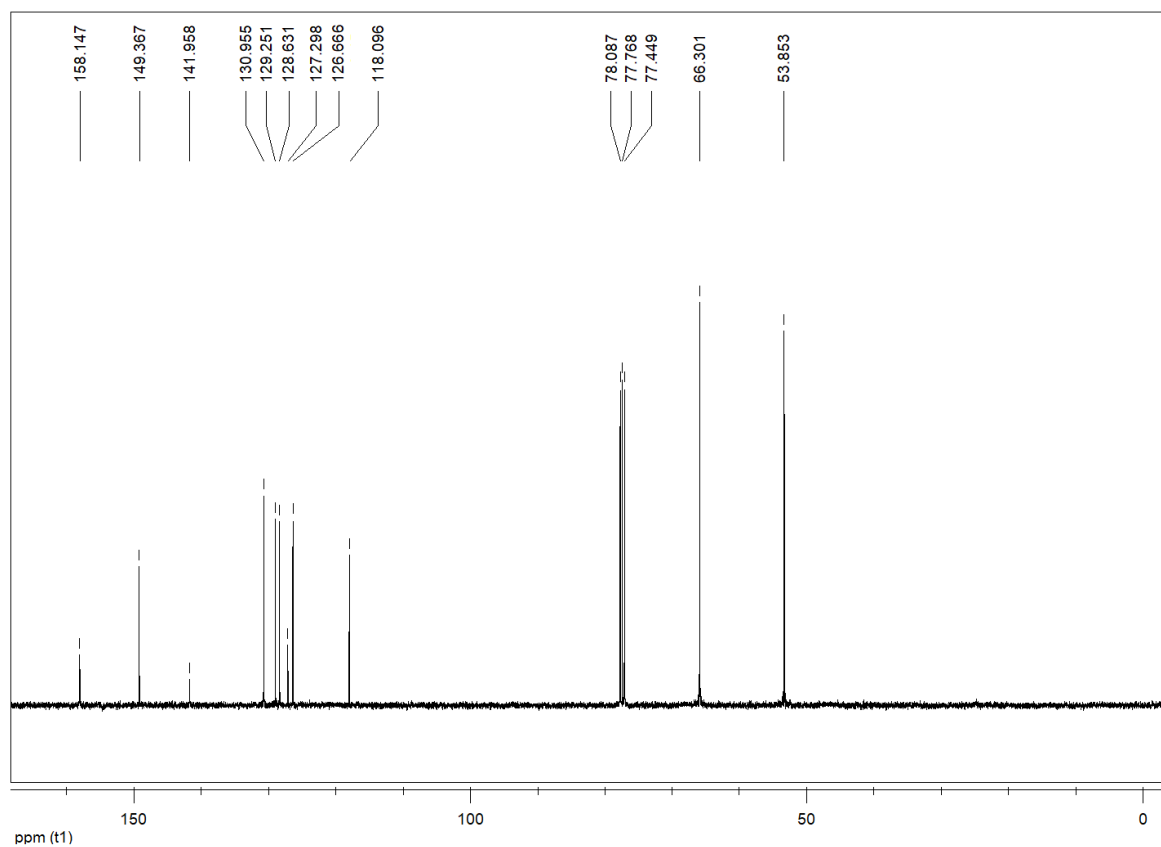


Figure S2. Spectrum ^{13}C NMR of 4-(Quinolin-4-yl)morpholine (3) in CDCl_3 .

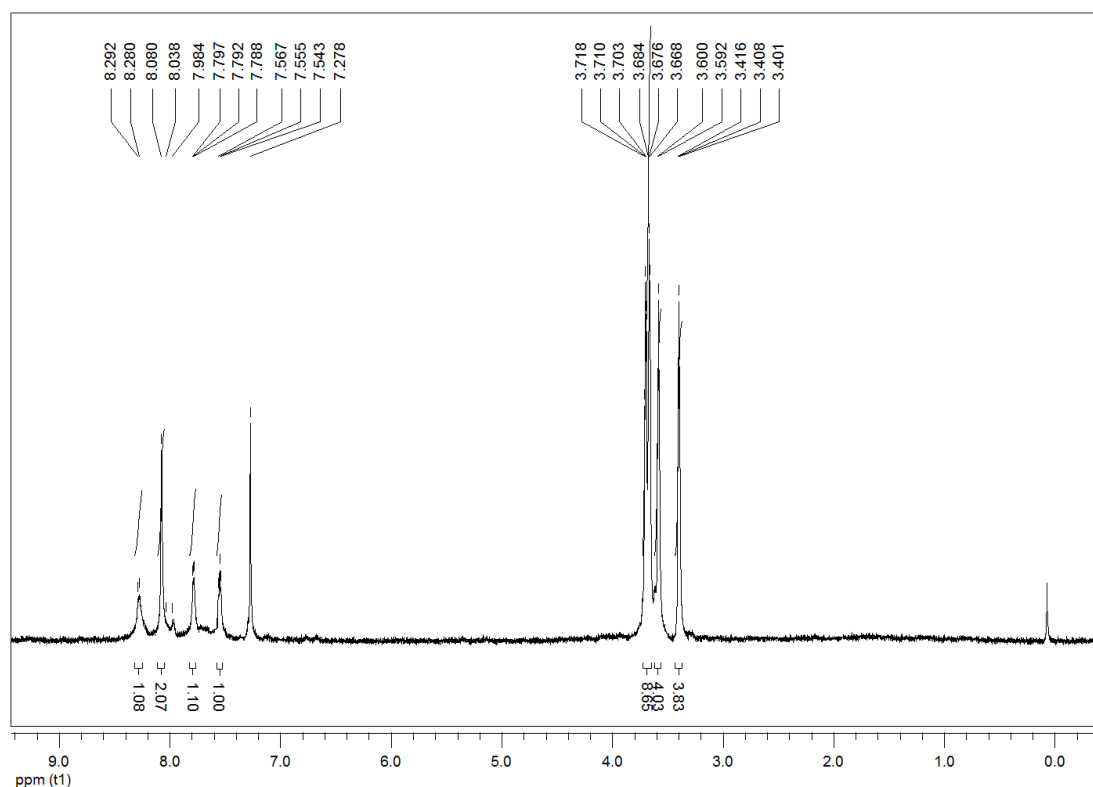


Figure S3. Spectrum ¹H NMR of 4,4'-(Quinoline-2,4-diyl)dimorpholine (4) in CDCl₃.

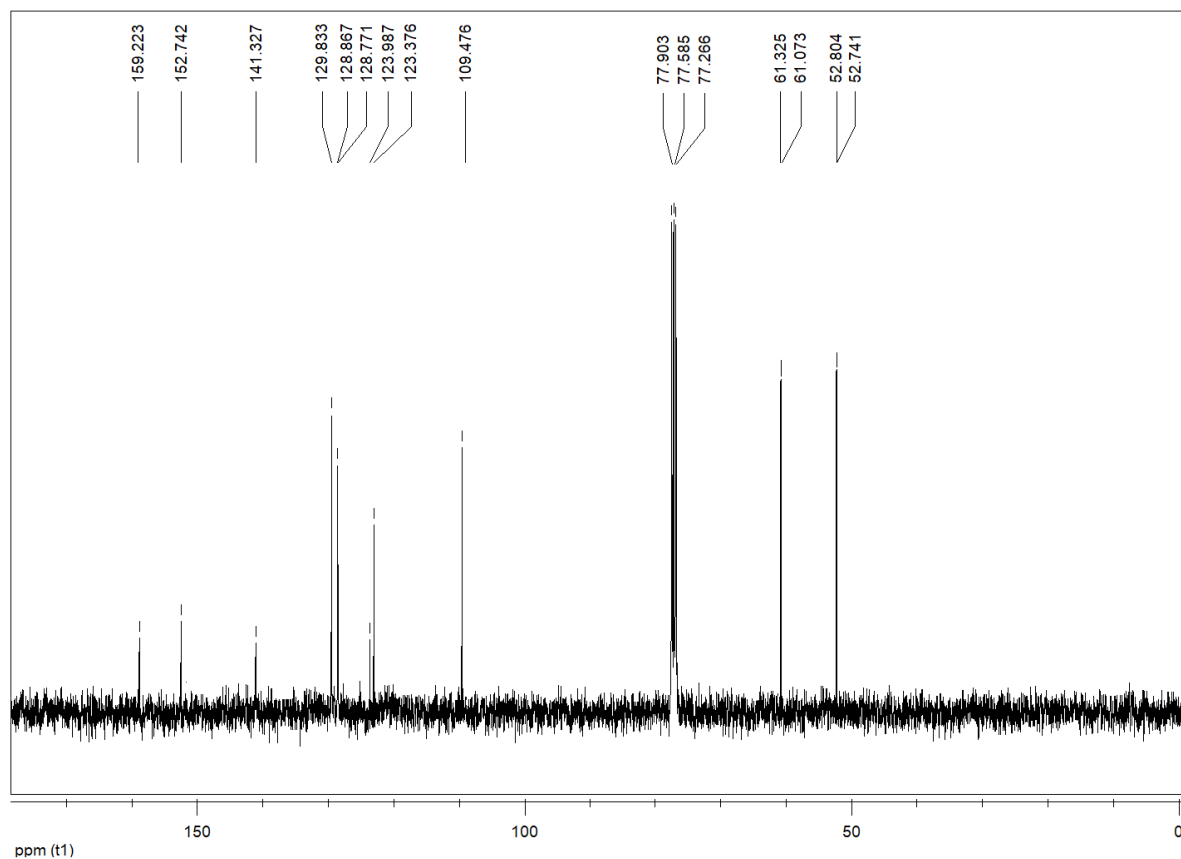


Figure S4. Spectrum ¹³C NMR of 4,4'-(Quinoline-2,4-diyl)dimorpholine (4) in CDCl₃.

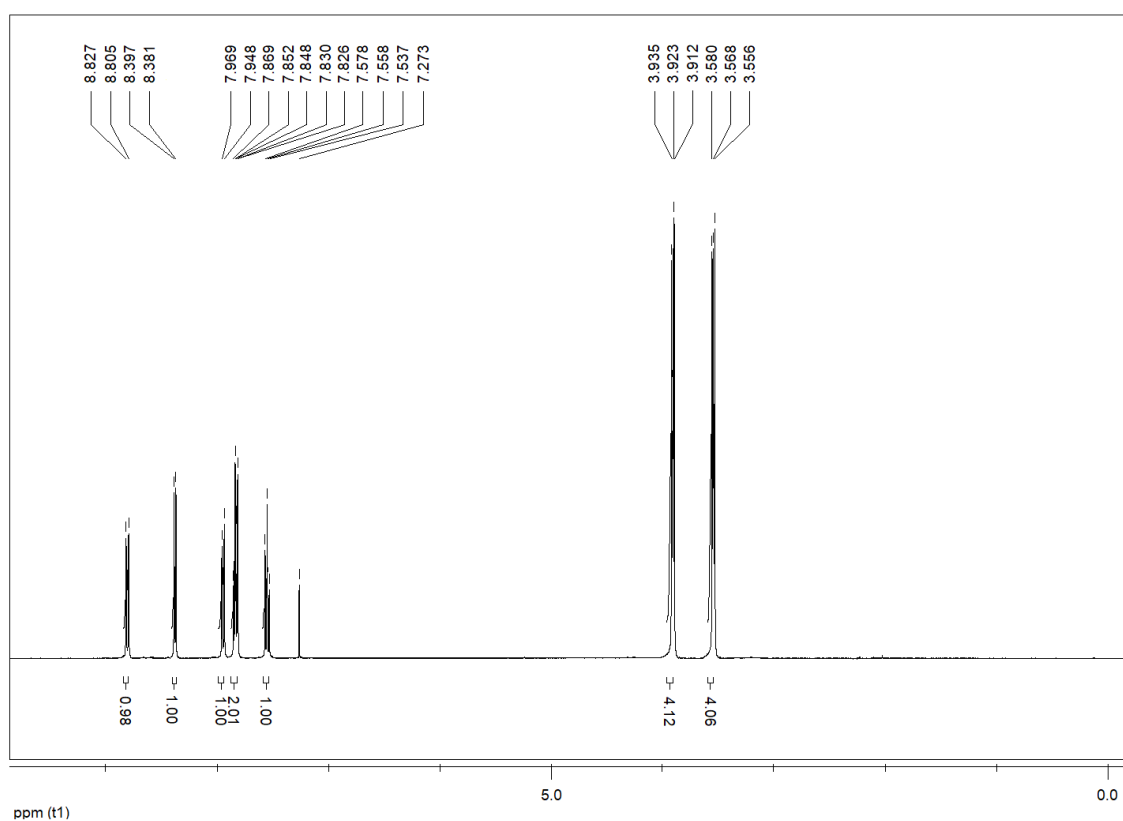


Figure S5. Spectrum ¹H NMR of 2-morpholinoquinoline 1-oxide (5) in CDCl₃.

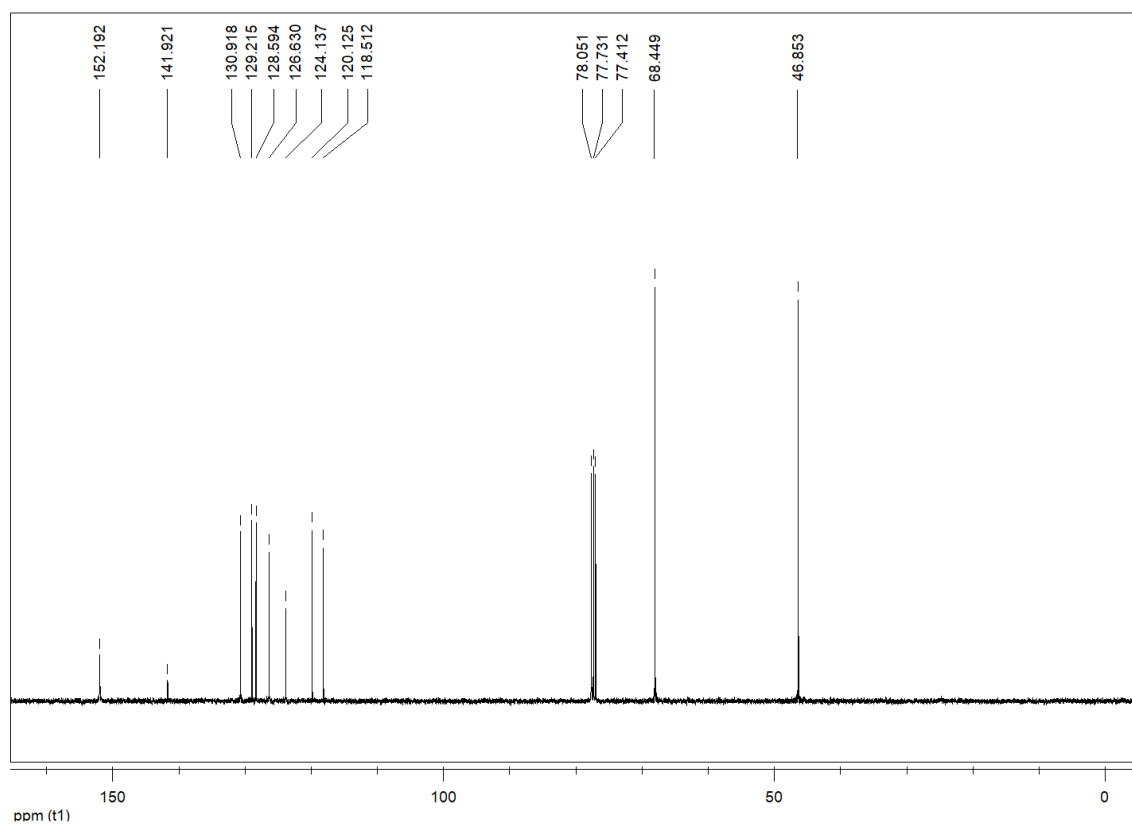


Figure S6. Spectrum ¹³C NMR of 2-morpholinoquinoline 1-oxide (5) in CDCl₃.

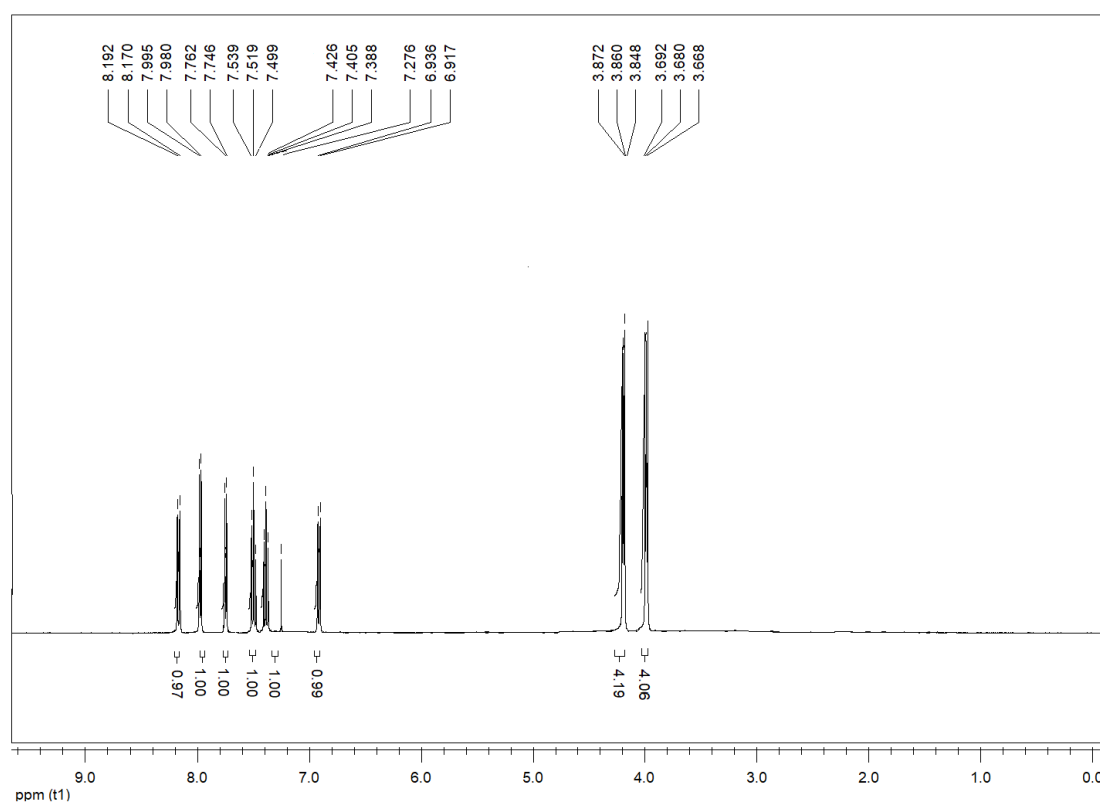


Figure S7. Spectrum ¹H NMR of 2-morpholinoquinoline (6) in CDCl₃.

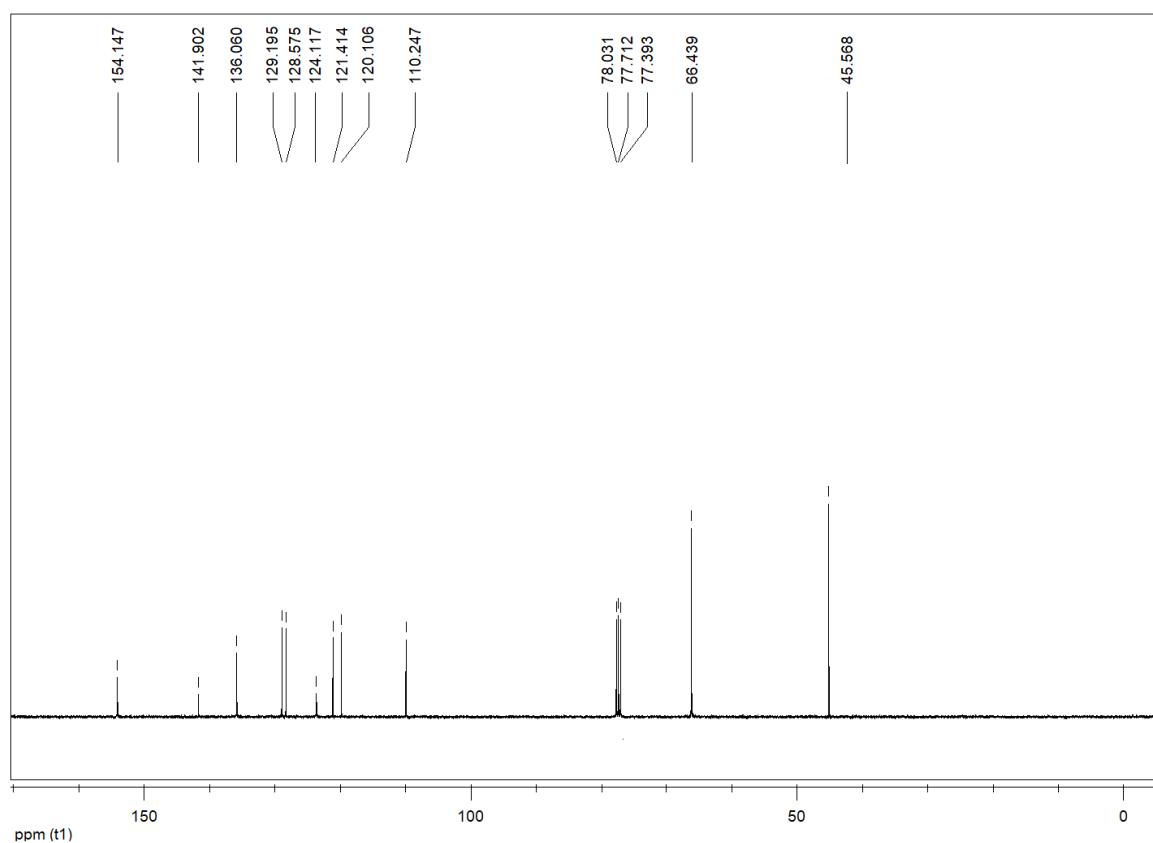


Figure S8. Spectrum ¹³C NMR of 2-morpholinoquinoline (6) in CDCl₃.

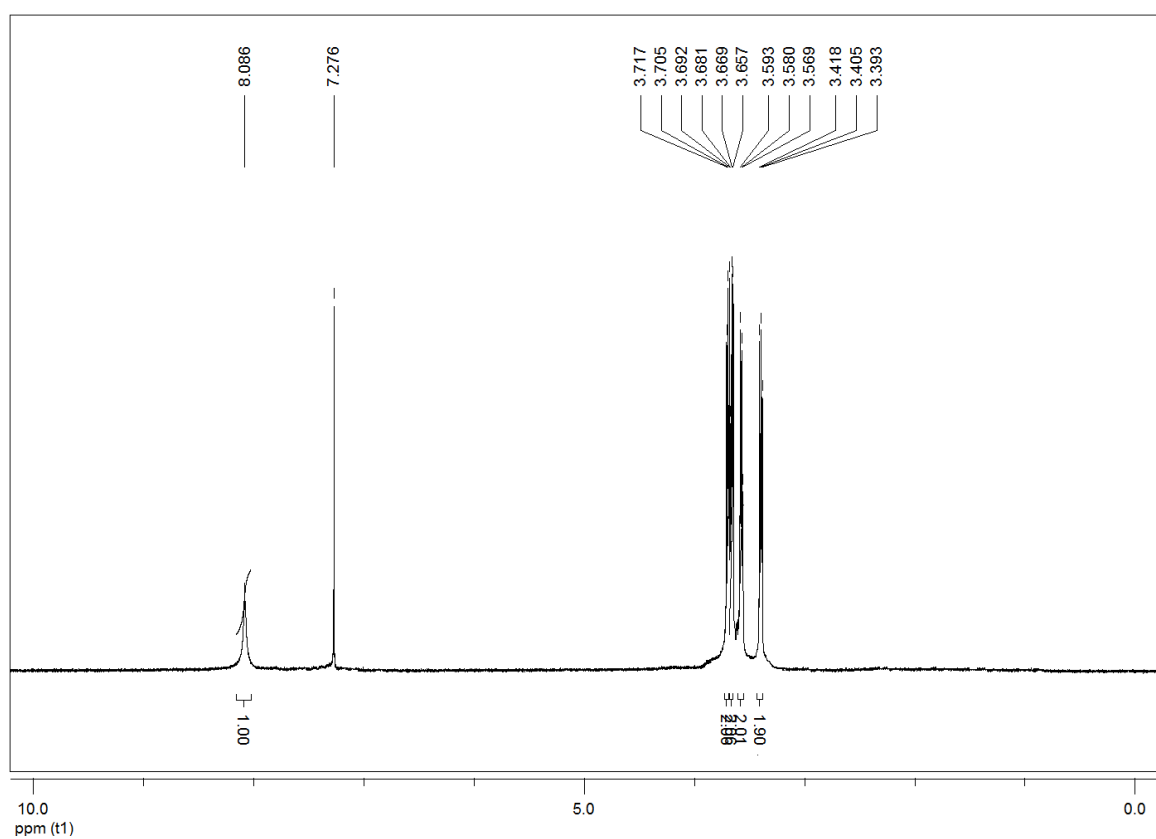


Figure S9. Spectrum ¹H NMR of morpholine-1-oxide (7) in CDCl₃.

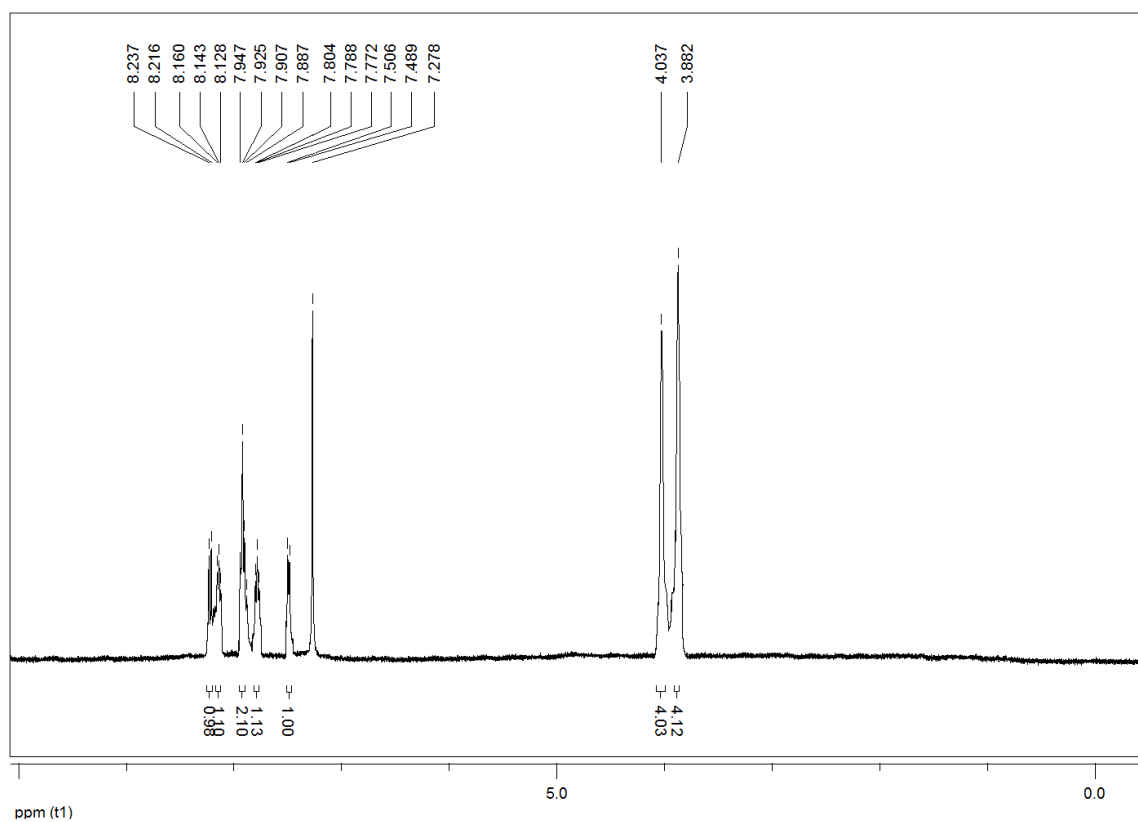


Figure S10. Spectrum ¹H NMR 4-(isoquinolin-1-yl)morpholine (8) in CDCl₃.

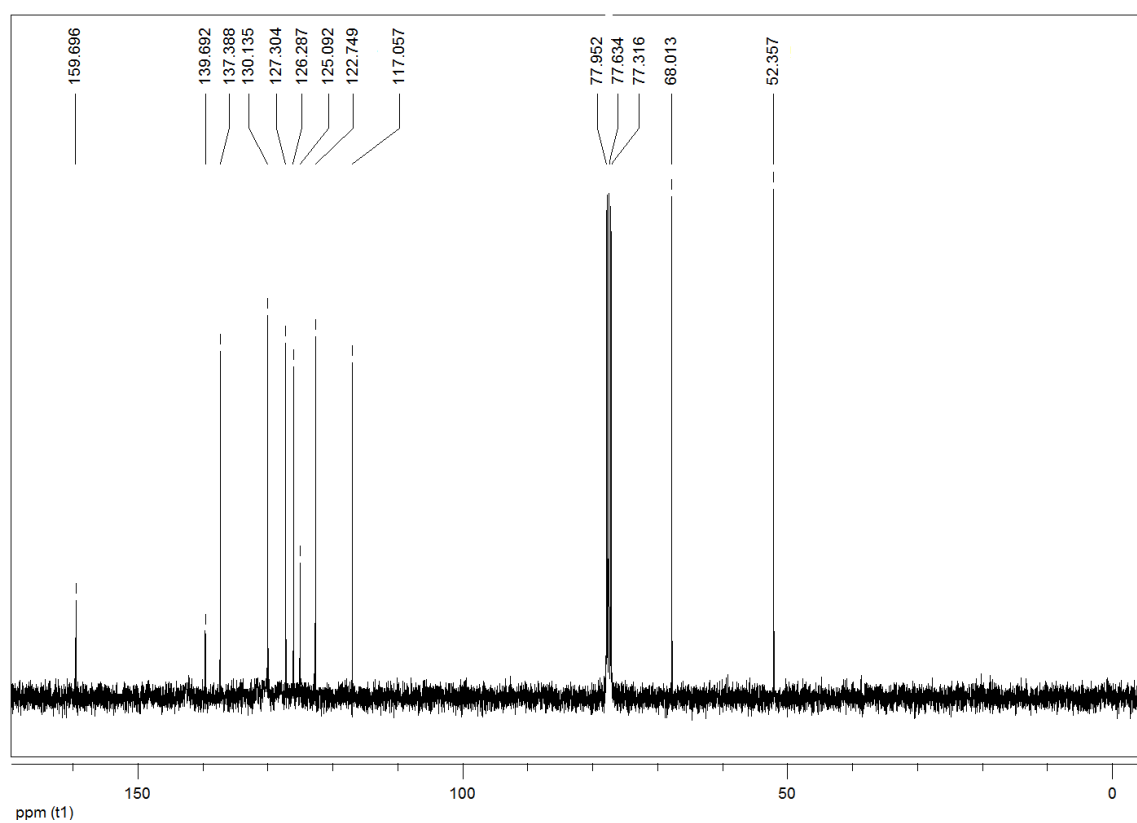


Figure S11. Spectrum ^{13}C NMR of 4-(isoquinolin-1-yl)morpholine (8) in CDCl_3 .