Electronic Supplementary Material (ESI) for Green Chemistry.

Electronic Supplementary Information

for

Very Simple one-pot Electrosynthesis of Nitrones starting from Nitro and Aldehyde Components

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1. General information

All reagents were used in analytical grades. Solvents were purified by standard methods. For electrochemical reactions boron-doped diamond (BDD) and glassy carbon electrodes were applied. BDD electrodes were obtained from CONDIAS GmbH, Itzehoe, Germany. BDD (15 µm diamond layer) on a silicon or niobium support (wafer) was used.

Compound **2c** was synthesized from the corresponding acid chloride, following the procedure described in the literature, using EtOH as alcohol.[1]

0.1 M AcOH / AcONa buffer solution at pH = 3.7 was prepared dissolving 1.03 mL of acetic acid (18 mmol) and 164 mg of sodium acetate (2 mmol) in 200 mL of distillated water.

NMR spectra: ^{1}H NMR and ^{13}C NMR spectra were recorded at 25 °C by using a Bruker Avance III HD 300 (300 MHz, 5 mm BBFO-SmartProbe with z gradient and ATM, SampleXPress 60 sample changer, Analytische Messtechnik, Karlsruhe, Germany). Chemical shifts (δ) are reported in parts per million (ppm) relative to traces of $CHCl_3$ (7.26 ppm) or DMSO (2.50 ppm) in the corresponding deuterated solvent.

Mass Spectrometry: Mass spectra and high resolution mass spectra were obtained by using a QTof Ultima 3 (Waters, Milford, Massachusetts) apparatus employing ESI^+.

Column chromatography was performed on silica gel 60 M (0.040-0.063 mm, Macherey-Nagel GmbH & Co, Düren, Germany) with a maximum pressure of 1.6 bar. As eluents mixtures of cyclohexane and ethyl acetate were used. Silica gel 60 sheets on aluminum (F254, Merck, Darmstadt, Germany) were used for thin layer chromatography.

Melting points were determined with a Melting Point Apparatus B-545 (Büchi, Flawil, Switzerland) and were uncorrected.

[1] J. R. Lawson, L. C. Wilkins and R. L. Melen, *Chem. Eur. J.*, 2017, **23**, 10997.

2. General procedure for the electrosynthesis of nitrones 3

Screening cell (beaker-type cell, 5 mL)

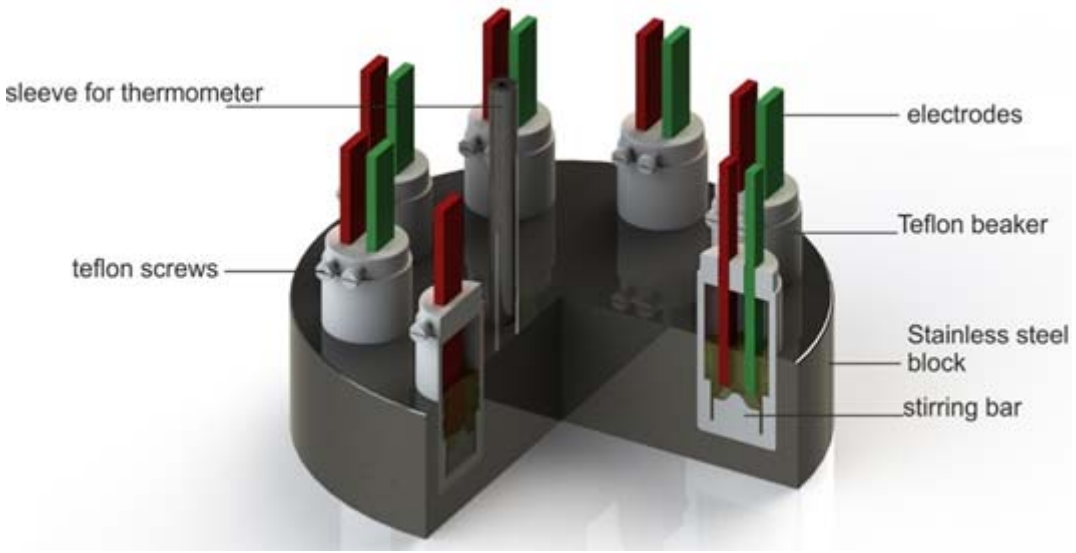


Figure 1. Schematic view of undivided screening cells of 5 mL in a screening arrangement.

A detailed description of this set up was published recently.[2] Dimensions of glassy carbon and BDD electrodes were 7.0 x 1.0 x 0.3 cm. Using a 5 mL reaction mixture, electrodes immersed 1.7 cm into solution and had upon immersion into the electrolyte an active surface of 1.7 cm^2 (1 x 1.7 cm).

Into an undivided beaker-type screening electrolysis cell equipped with a glassy carbon anode and a BDD cathode, EtOH (2.4 mL), buffered water at pH = 3.7 (2.4 mL), acetone (0.2 mL), NBu_4BF_4 (16.4 mg, 0.01 mmol), aldehyde **1** (0.6 mmol) and nitroderivative **2** (0.66 mmol) were added. A constant current electrolysis with a current density of 7 mA/cm^2 was performed at room temperature. After application of 290 C (4.5 F) the electrolysis was stopped.

The mixture was then transferred into a separatory funnel, where AcOEt (20 mL) and water (20 mL) were added. The aqueous phase was again extracted with AcOEt (20 mL), organic phases were washed with brine (20 ml) and dried over $MgSO_4$. The solution was concentrated under reduced pressure and the crude was purified under flash column chromatography to afford the corresponding nitrones **3**.

[2] C. Gütz, B. Klöckner and S. R. Waldvogel, *Org. Process Res. Dev.*, 2016, **20**, 26.

Beaker-type cell (25 mL)

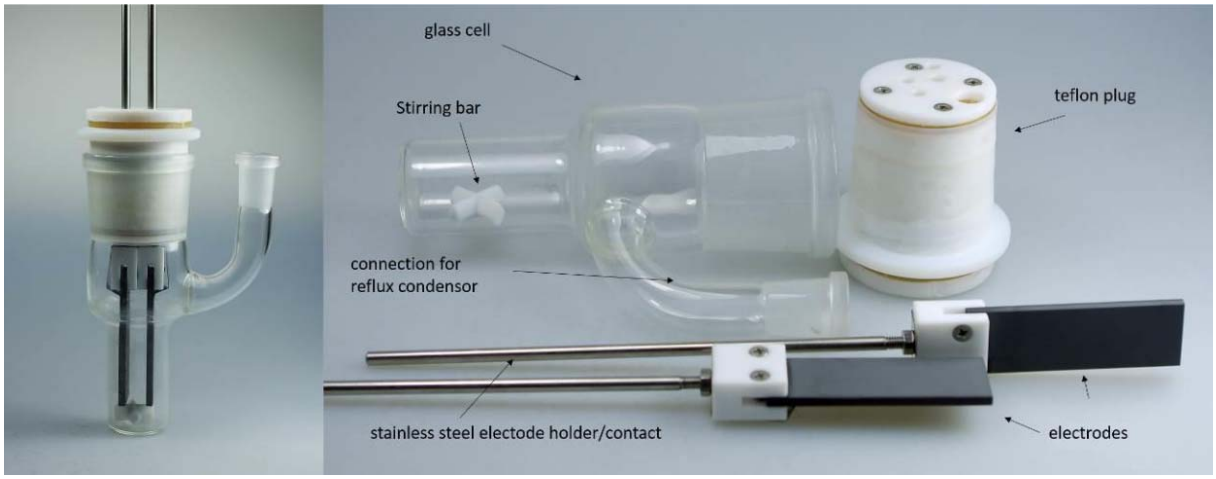


Figure 2. View of undivided Beaker-type cell of 25 mL.

The beaker-type cell (25 mL) consists of a simple glass beaker with or without cooling jacket and closed by a teflon plug. This cap allows precise arrangement of the electrodes. Dimensions of glassy carbon and BDD electrodes were 7.0 x 2.0 x 0.2 cm. The electrodes had upon immersion into the electrolyte an active surface of 7 cm^2 (2 x 3.5 cm).

The scale-up of nitrone **3ba** was carried out using this cell.

Into an undivided beaker-type screening electrolysis cell equipped with a glassy carbon anode and a BDD cathode, EtOH (12 mL), buffered water at pH = 3.7 (12 mL), acetone (1 mL), NBu_4BF_4 (83 mg, 0.25 mmol), cinnamaldehyde **1b** (471 µL, 3.75 mmol) and nitroderivative **2a** (565 mg, 4.12 mmol) were added. A constant current electrolysis with a current density of 7 mA/cm^2 was performed at room temperature. After application of 1787 C (4.5 F) the electrolysis was stopped.

The mixture was then transferred into a separatory funnel, where AcOEt (100 mL) and water (100 mL) were added. The aqueous phase was again extracted with AcOEt (100 mL), organic phases were washed with brine (100 mL) and dried over $MgSO_4$. The solution was concentrated under reduced pressure and the crude was purified under flash column chromatography (cyclohexane/EtOAc = 3:1 to 1:1) to afford the corresponding nitrone **3ba**.

Beaker-type cell (200 mL)

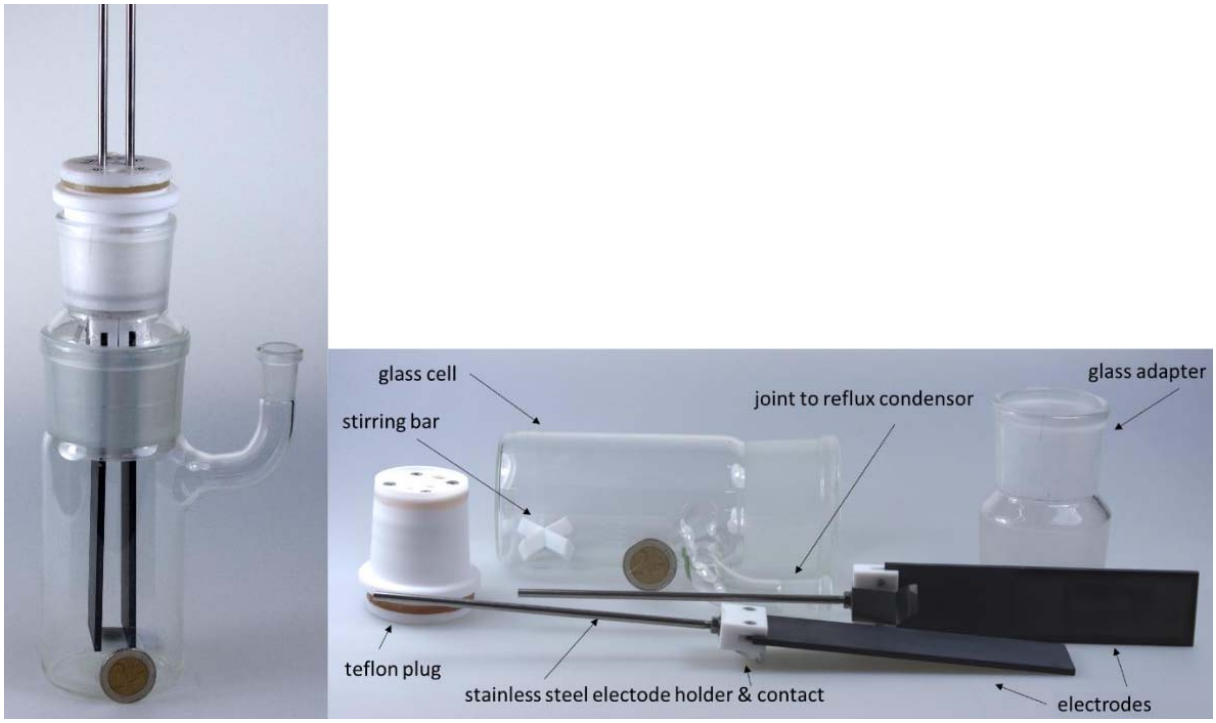


Figure 3. View of undivided Beaker-type cell of 200 mL.

The beaker-type cell (200 mL) consists of a simple glass beaker with or without cooling jacket and closed by a teflon plug. This cap allows precise arrangement of the electrodes. Dimensions of glassy carbon and BDD electrodes were 14 x 3.5 x 0.3 cm. The electrodes had upon immersion into the electrolyte an active surface of 17.5 cm^2 (3.5 x 5 cm).

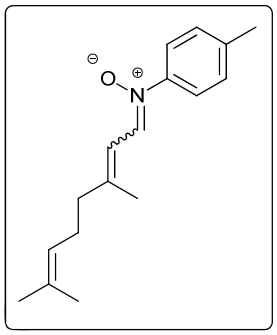
The scale-up of nitrone **3de** was carried out using this cell.

Into an undivided beaker-type screening electrolysis cell equipped with a glassy carbon anode and a BDD cathode, EtOH (96 mL), buffered water at pH = 3.7 (96 mL), acetone (8 mL), NBu_4BF_4 (658 mg, 2 mmol), aldehyde **1d** (3.63 mL, 24 mmol) and nitroderivative **2e** (2.71 mL, 26.4 mmol) were added. A constant current electrolysis with a current density of 7 mA/cm^2 was performed at room temperature. After application of 11450 C (4.5 F) the electrolysis was stopped.

The mixture was then transferred into a separatory funnel, where AcOEt (300 mL) and water (300 mL) were added. The aqueous phase was again extracted with AcOEt (300 mL), organic phases were washed with brine (300 mL) and dried over $MgSO_4$. The solution was concentrated under reduced pressure and the crude was purified under flash column chromatography (cyclohexane/EtOAc = 3:1) to afford the corresponding nitrone **3de**.

3. Characterization of nitrones 3

N-(3,7-dimethylocta-2,6-dien-1-ylidene)-4-methylaniline oxide (3aa)



Brown oil. Obtained from citral as a 3:1 mixture of *E:Z* isomers.

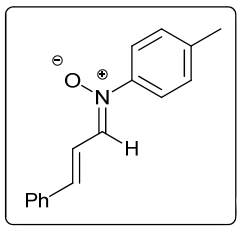
Purified by flash column chromatography (cyclohexane/EtOAc = 4:1). Yield: 71% (109 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.77 (d, *J* = 10.2 Hz, $1H_{minor}$), 7.75 (d, *J* = 10.2 Hz, $1H_{major}$), 7.63-7.55 (m, $2H_{major,}$ $2H_{minor}$), 7.23 (d, *J* = 8.1 Hz, $2H_{major,}$ $2H_{minor}$), 6.87 (d, *J* = 10.2 Hz, $1H_{major}$), 6.86 (d, *J* = 10.2 Hz, $1H_{minor}$), 5.16-5.07 (m, $1H_{major,}$ $1H_{minor}$), 2.40 (t, $3H_{major,}$ $3H_{minor}$), 2.34-2.14 (m, $4H_{major,}$ $4H_{minor}$), 2.02 (s, $3H_{major}$), 1.93 (s, $3H_{minor}$), 1.70 (s, $3H_{minor}$), 1.68 (s, $3H_{major}$), 1.63 (s, $3H_{minor}$), 1.61 (s, $3H_{major}$).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 152.06 (C), 145.52 (C), 139.88 (C), 133.26 (CH), 133.05 (C), 129.53 (CH), 123.16 (CH), 122.95 (CH), 121.37 (CH), 121.34 (CH), 117.66 (CH), 116.83 (CH), 40.67 (CH_2), 33.89 (CH_2), 26.64 (CH_2), 26.40 (CH_2), 25.71 (CH_3), 24.77 (CH_3), 21.15 (CH_3), 18.28 (CH_3), 17.77 (CH_3).

HRMS (ESI): calculated for $C_{17}H_{23}NO$ (M^+ + H): 258.1857; found: 258.1838.

(*Z*)-4-Methyl-*N*-((*E*)-3-phenylallylidene)aniline oxide (3ba)



Yellow solid (m.p = 149-151 °C). Purified by flash column chromatography (cyclohexane/EtOAc = 3:1 to 1:1). Yield: 72% (102 mg).

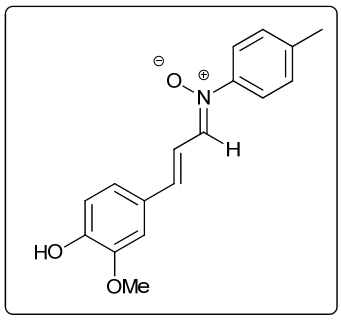
This nitrone was also synthesized in a 25 mL beaker-type cell (See experimental procedure). Yield: 56% (498 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.85 (d, *J* = 9.8 Hz, 1H), 7.76-7.63 (m, 3H), 7.60-7.54 (m, 2H), 7.41-7.32 (m, 3H), 7.26-7.21 (m, 2H), 7.15 (d, *J* = 15.8 Hz, 1H), 2.40 (s, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 145.01 (C), 140.33 (C), 140.00 (CH), 136.17 (CH), 136.13 (C), 129.63 (CH), 129.48 (CH), 128.91 (CH), 127.53 (CH), 121.18 (CH), 119.13 (CH), 21.18 (CH_3).

HRMS (ESI): calculated for $C_{16}H_{16}NO$ (M^+ + H): 237.1226; found: 237.1225.

(*Z*)-*N*-((*E*)-3-(4-hydroxy-3-methoxyphenyl)allylidene)4-methylaniline oxide (3ca)



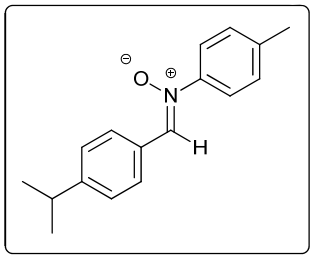
Orange solid (m.p = 68-70 °C). Purified by flash column chromatography (cyclohexane/EtOAc = 1:2 to 1:5). Yield: 66% (112 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.81 (d, *J* = 9.8 Hz, 1H), 7.64 (d, *J* = 8.7 Hz, 2H), 7.54 (dd, *J* = 16.0 and 9.8 Hz, 1H), 7.23 (d, *J* = 8.7 Hz, 2H), 7.14-7.10 (m, 1H), 7.07 (d, *J* = 16.0 Hz, 1H), 6.98 (dd, *J* = 8.1 and 1.6 Hz, 1H), 6.90 (d, *J* = 8.1 Hz, 1H), 3.87 (s, 3H), 2.40 (s, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 148.11 (C), 147.37 (C), 144.76 (C), 141.13 (CH), 140.15 (C), 137.07 (CH), 129.63 (C), 128.51 (CH), 123.16 (CH), 121.10 (CH), 116.58 (CH), 114.99 (CH), 108.38 (CH), 55.96 (CH_3), 21.16 (CH_3).

HRMS (ESI): calculated for $C_{17}H_{18}NO_3$ (M^+ + H): 284.1281; found: 284.1269.

(*Z*)-*N*-(4-isopropylbenzylidene)-4-methylaniline oxide (3da)



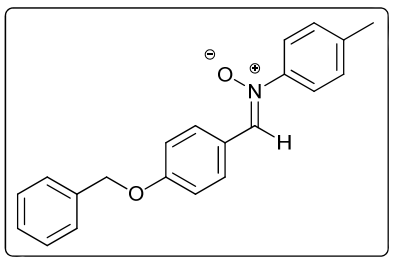
Pale brown solid (m.p = 83-85 °C). Purified by flash column chromatography (cyclohexane/EtOAc = 3:1). Yield: 51% (78 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.34 (d, *J* = 8.3 Hz, 2H), 7.88 (s, 1H), 7.67 (d, *J* = 8.3 Hz, 2H), 7.35 (s, *J* = 8.3 Hz, 2H), 7.26 (d, *J* = 8.3 Hz, 2H), 2.97 (hep, *J* = 6.9 Hz, 1H), 2.42 (s, 3H), 1.30 (d, *J* = 6.9 Hz, 6H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 152.24 (C), 146.78 (C), 140.00 (C), 134.20 (CH), 129.61 (CH), 129.26 (CH), 128.49 (C), 126.73 (CH), 121.47 (CH), 34.31 (CH), 23.73 (CH_3), 21.16 (CH_3).

HRMS (ESI): calculated for $C_{17}H_{19}NO$ (M^+ + H): 254.1539; found: 254.1534.

(*Z*)-*N*-(4-(benzyloxy)benzylidene)-4-methylaniline oxide (3ea)



Brown solid (m.p = 143-145°C). Purified by flash column chromatography (cyclohexane/EtOAc = 3:1 to 1:1). Yield: 48% (91 mg).

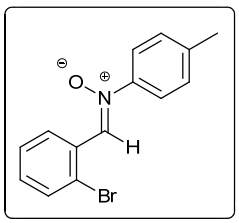
^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.41 (d, *J* = 8.9 Hz, 2H), 7.85 (s, 1H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.49-7.33 (m, 5H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.08 (d, *J* = 8.9 Hz, 2H), 5.15 (s, 2H), 1.43 (s,

3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 160.60 (C), 146.61 (C), 139.86 (C), 136.37 (C), 133.88 (CH), 131.16 (CH), 129.61 (CH), 128.67 (CH), 128.18 (CH), 127.54 (CH), 124.02 (C), 121.40 (CH), 114.89 (CH), 70.06 (CH_2), 21.17 (CH_3).

HRMS (ESI): calculated for $C_{21}H_{20}NO_2$ (M^+ + H): 318.1489; found: 318.1475.

((*Z*)-*N*-(2-bromobenzylidene)-4-methylaniline oxide (3fa)



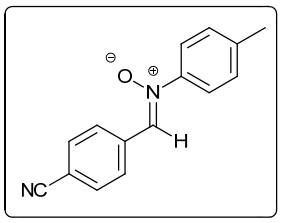
Brown solid (m.p = 71-73°C). Purified by flash column chromatography (cyclohexane/EtOAc = 6:1). Yield: 31% (53 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 9.52 (dd, *J* = 8.1 and 1.6 Hz, 1H), 8.42 (s, 1H), 7.73-7.65 (m, 3H), 7.47 (t, *J* = 7.5 Hz, 1H), 7.34-7.26 (m, 3H), 2.45 (s, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 147.16 (C), 140.56 (C), 132.96 (CH), 132.77 (CH), 131.68 (CH), 129.84 (C), 129.76 (CH), 129.52 (CH), 127.80 (CH), 124.11 (C), 121.57 (CH), 21.22 (CH_3).

HRMS (ESI): calculated for $C_{14}H_{13}NO_2Br$ (M^+ + H): 290.0175; found: 290.0183.

(*Z*)-*N*-(4-cyanobenzylidene)-4-methylaniline oxide (3ga)



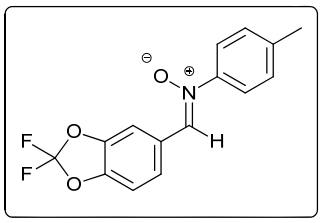
Brown solid (m.p = 145-147 °C). Purified by flash column chromatography (cyclohexane/EtOAc = 4:1 to 3:1). Yield: 21% (31 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.48 (d, *J* = 8.5 Hz, 2H), 8.00 (s, 1H), 7.71 (d, *J* = 8.5 Hz, 2H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H), 2.44 (s, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 146.58 (C), 141.06 (C), 134.56 (CH), 132.32 (CH), 132.28 (C) 129.82 (CH), 128.84 (CH), 121.44 (CH), 118.55 (C), 113.17 (C), 21.23 (CH_3).

HRMS (ESI): calculated for $C_{15}H_{13}N_2O$ (M^+ + H): 237.1022; found: 237.1025.

(*Z*)-*N*-((2,2-difluorobenzo[*d*][1,3]dioxol-5-yl)methylene)-4-methylaniline oxide (3ha)



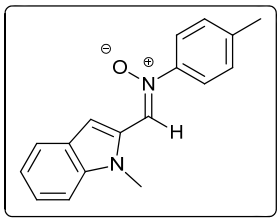
Pale brown solid (m.p = 161-163°C). Purified by flash column chromatography (cyclohexane/EtOAc = 3:1). Yield: 56% (98 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.70 (d, *J* = 1.6 Hz, 1H), 7.92 (s, 1H), 7.81 (dd, *J* = 8.5 and 1.6 Hz, 1H), 7.66 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 8.5 Hz, 1H), 2.44 (s, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 146.49 (C), 144.74 (C), 143.79 (C), 140.52 (C), 132.71 (CH), 131.68 (t, *J* = 256 Hz, CF_2), 129.74 (CH), 127.28 (C), 125.97 (CH), 121.37 (CH), 109.47 (CH), 21.17 (CH_3).

HRMS (ESI): calculated for $C_{15}H_{12}F_2NO_3$ (M^+ + H): 292.0780; found: 292.0785.

(*Z*)-4-methyl-*N*-((1-methyl-1*H*-indol-2-yl)methylene)aniline oxide (3ia)



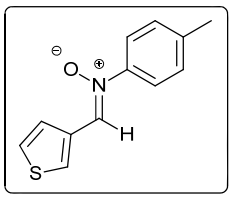
Yellowish solid (m.p = 165-167°C). Purified by flash column chromatography (cyclohexane/EtOAc = 1:1). In this case, only 80% of conversion was achieved. Yield: 60% (bsmr, 76 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.44 (s, 1H), 8.12 (s, 1H), 7.76 (d, *J* = 7.9 Hz, 1H), 7.70 (d, *J* = 8.5 Hz, 2H), 7.36-7.24 (m, 4H), 5.15 (ddd, *J* = 7.9, 6.1 and 1.7 Hz, 1H), 3.78 (s, 3H), 2.43 (s, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 146.29 (C), 140.10 (C), 138.69 (C), 130.39 (C), 129.70 (CH), 127.56 (C), 124.59 (CH), 123.49 (CH), 122.60 (CH), 121.14 (CH), 120.33 (CH), 109.10 (CH), 109.06 (CH), 29.90 (CH_3), 21.18 (CH_3).

HRMS (ESI): calculated for $C_{17}H_{17}N_2O$ (M^+ + H): 265.1335; found: 265.1329.

(*Z*)-4-methyl-*N*-(thiophen-3-ylmethylene)aniline oxide (3ja)



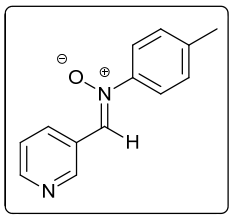
Brown solid (m.p = 106-108°C). Purified by flash column chromatography (cyclohexane/EtOAc = 3:1 to 1:1). Yield: 42% (55 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 9.14 (d, *J* = 2.9 Hz, 1H), 8.06 (s, 1H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.45 (dd, *J* = 5.0 and 1.1 Hz, 1H), 7.38 (dd, *J* = 5.0 and 2.9 Hz, 1H), 7.26 (d, *J* = 8.5 Hz, 2H), 2.42 (s, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 145.81 (C), 140.13 (C), 131.62 (C), 129.70 (CH), 129.67 (CH), 128.75 (CH), 128.14 (CH), 125.39 (CH), 121.24 (CH), 21.17 (CH_3).

HRMS (ESI): calculated for $C_{12}H_{12}NOS$ (M^+ + H): 218.0634; found: 218.0633.

(*Z*)-4-methyl-*N*-(pyridin-3-ylmethylene)aniline oxide (3ka)



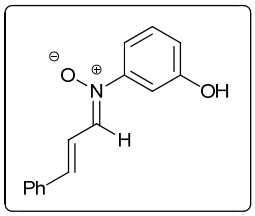
Brown solid (m.p = 126-128°C). Purified by flash column chromatography (cyclohexane/EtOAc = 1:2 to 1:5). Yield: 30% (38 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 9.21 (d, *J* = 8.1 Hz, 1H), 9.10 (s, 1H), 8.65 (d, *J* = 4.8 Hz, 1H), 7.98 (s, 1H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.43 (dd, J = 8.1 and 4.8 Hz), 7.30 (d, *J* = 8.4 Hz, 2H), 2.44 (s, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 150.77 (CH), 150.31 (CH), 146.45 (C), 140.76 (C), 134.79 (CH), 130.98 (CH), 129.77 (CH), 127.39 (C), 123.66 (CH), 121.38 (CH), 21.20 (CH_3).

HRMS (ESI): calculated for $C_{13}H_{13}N_2O$ (M^+ + H): 213.1022; found: 213.1029.

(*Z*)-3-hydroxy-*N*-((*E*)-3-phenylallylidene)aniline oxide (3bb)



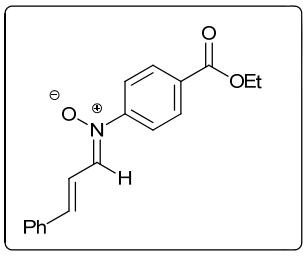
Brown solid (m.p = 116-118 °C). Purified by flash column chromatography (cyclohexane/EtOAc = 1:2). Yield: 61% (87 mg).

^{1}H NMR (DMSO, 300 MHz): δ 10.02 (bs, 1H), 8.37 (d, *J* = 9.1 Hz, 1H), 7.65-7.59 (m, 2H), 7.53 (dd, *J* = 16.1 and 9.1 Hz, 1H), 7.45-7.25 (m, 7H), 6.90 (dt, *J* = 6.6 and 2.5 Hz, 1H).

^{13}C NMR (DMSO, 75 MHz): δ 158.36 (C), 148.59 (C), 139.55 (CH), 136.62 (C), 135.71 (CH), 130.25 (CH), 129.71 (CH), 129.51 (CH), 127.71 (CH), 119.63 (CH), 117.31 (CH), 111.73 (CH), 108.78 (CH).

HRMS (ESI): calculated for $C_{15}H_{14}NO_2$ (M^+ + H): 240.1024; found: 240.1035.

(*Z*)-4-(ethoxycarbonyl)-*N*-((*E*)-3-phenylallylidene)aniline oxide (3bc)



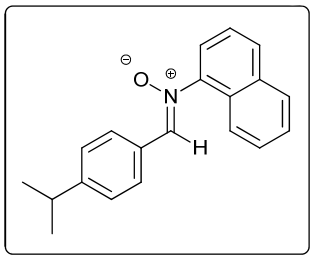
Pale brown solid (m.p = 119-121 °C). Purified by flash column chromatography (cyclohexane/EtOAc = 2:1 to 1:1). Yield: 46% (82 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.14 (d, *J* = 8.7 Hz, 2H), 7.94 (d, *J* = 9.5 Hz, 1H), 7.84 (d, *J* = 8.7 Hz, 2H), 7.70 (dd, *J* = 16.1 and 9.5 Hz, 1H), 7.60-7.53 (m, 2H), 7.47-7.34 (m, 3H), 7.22 (d, *J* = 16.1 Hz, 1H), 4.40 (q, *J* = 7.0 Hz, 2H), 1.42 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 165.31 (C), 150.09 (C), 141.40 (CH), 137.26 (CH), 135.87 (C), 131.81 (C), 130.57 (CH), 129.86 (CH), 128.98 (CH), 127.69 (CH), 121.34 (CH), 118.81 (CH), 61.48 (CH_2), 14.30 (CH_3).

HRMS (ESI): calculated for $C_{18}H_{18}NO_3$ (M^+ + H): 296.1281; found: 296.1279.

(*Z*)-*N*-(4-isopropylbenzylidene)naphthalen-1-amine oxide (3dd)



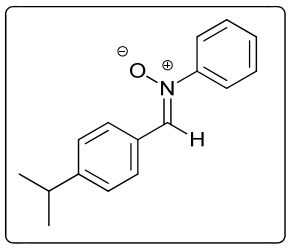
Reddish oil. Purified by flash column chromatography (cyclohexane/EtOAc = 5:1 to 3:1). Yield: 50% (86 mg).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.40 (d, *J* = 8.4 Hz, 2H), 8.21-8.14 (m, 1H), 7.98-7.90 (m, 2H), 7.79 (s, 1H), 7.64-7.48 (m, 4H), 7.41 (d, *J* = 8.4 Hz, 2H), 3.02 (hep, *J* = 6.9 Hz, 1H), 1.33 (d, *J* = 6.9 Hz, 6H).

^{13}C NMR ($CDCl_3$, 75 MHz): δ 152.59 (C), 145.92 (C), 138.85 (CH), 134.22 (C), 129.84 (CH), 129.25 (CH), 128.19 (C), 128.02 (CH), 127.72 (CH), 127.03 (CH), 126.97 (C), 126.84 (CH), 124.89 (CH), 122.89 (CH), 120.33 (CH), 34.39 (CH), 23.77 (CH_3).

HRMS (ESI): calculated for $C_{20}H_{20}NO$ (M^+ + H): 290.1539; found: 290.1533.

(*Z*)-*N*-(4-isopropylbenzylidene)aniline oxide (3de)



Brown solid (m.p = 81-83 °C). Purified by flash column chromatography (cyclohexane/EtOAc = 3:1). Yield: 48% (69 mg).

This nitrone was also synthesized in a 200 mL beaker-type cell (See experimental procedure). Yield: 43% (2.43g).

^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.35 (d, *J* = 8.4 Hz, 2H), 7.92 (s, 1H), 7.83-7.75 (m, 2H), 7.52-7.44 (m, 3H), 7.35 (d, *J* = 8.4 Hz, 2H), 2.99 (hep, *J* = 6.9 Hz, 1H), 1.30 (d, *J* = 6.9 Hz, 6H).

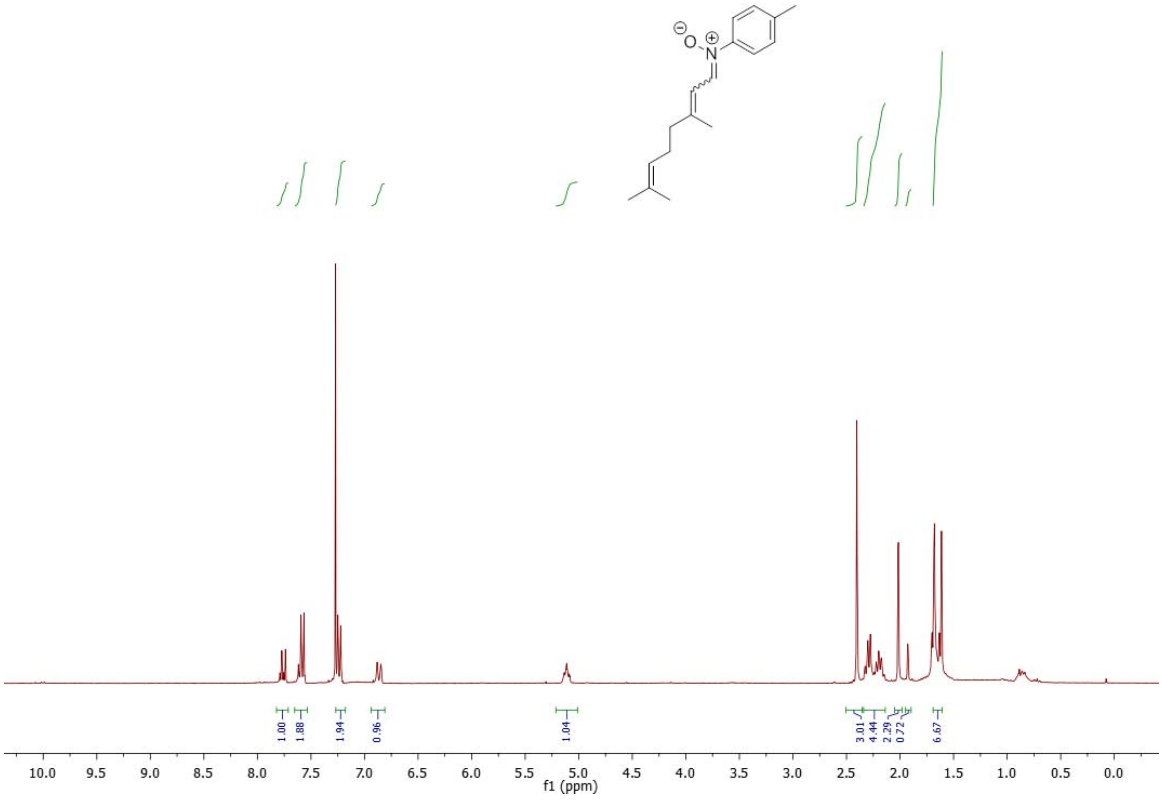
^{13}C NMR ($CDCl_3$, 75 MHz): δ 152.50 (C), 148.97 (C), 134.92 (CH), 129.81 (CH), 129.40 (CH), 129.14 (CH), 128.34 (C), 126.78 (CH), 121.76 (CH), 34.33 (CH), 23.72 (CH_3).

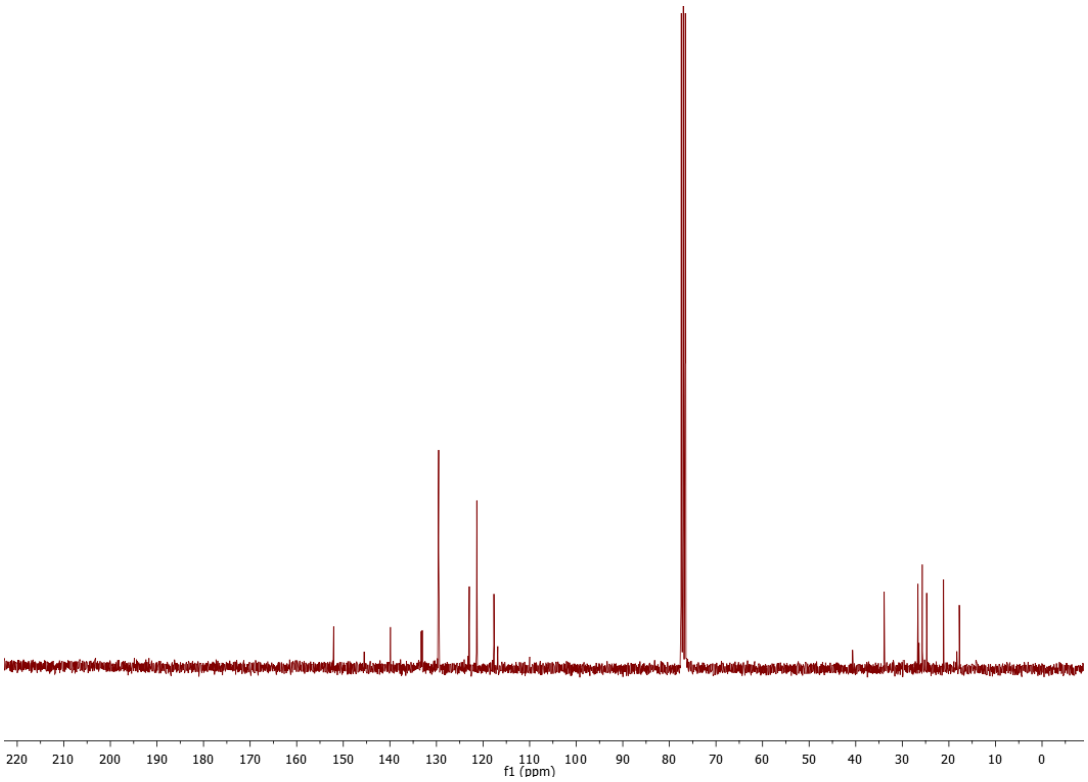
HRMS (ESI): calculated for $C_{16}H_{17}NONa$ (M^+ + Na): 254.1207; found: 262.1194.

4. ^{1}H and ^{13}C spectra of the compounds

***N*-(3,7-dimethylocta-2,6-dien-1-ylidene)-4-methylaniline oxide (*E* and *Z* isomers) (3aa)**

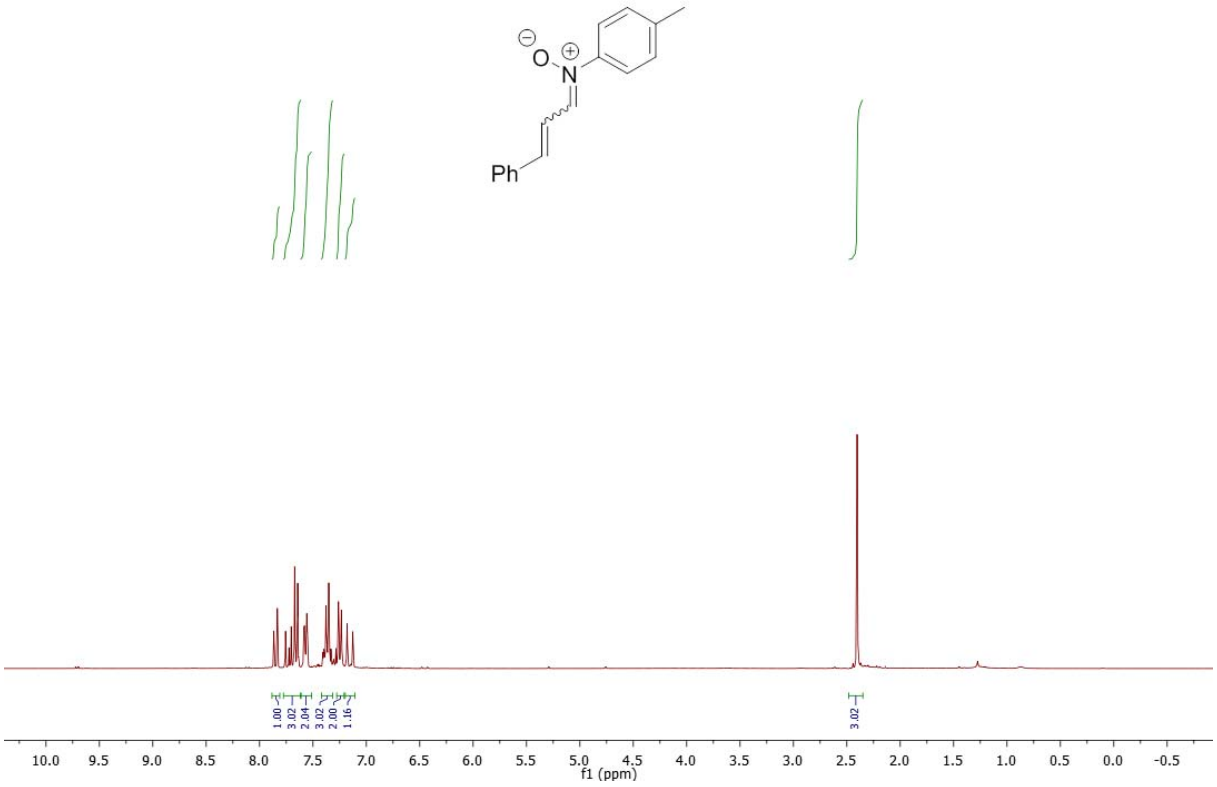
($CDCl_3$, 300 MHz)

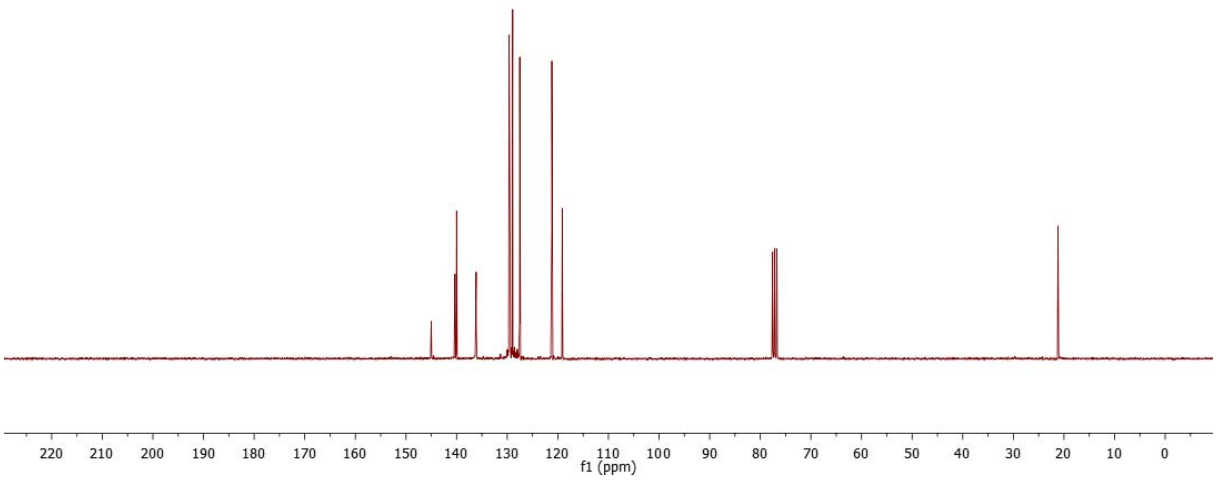




(*Z*)-4-Methyl-*N*-((*E*)-3-phenylallylidene)aniline oxide (3ba)

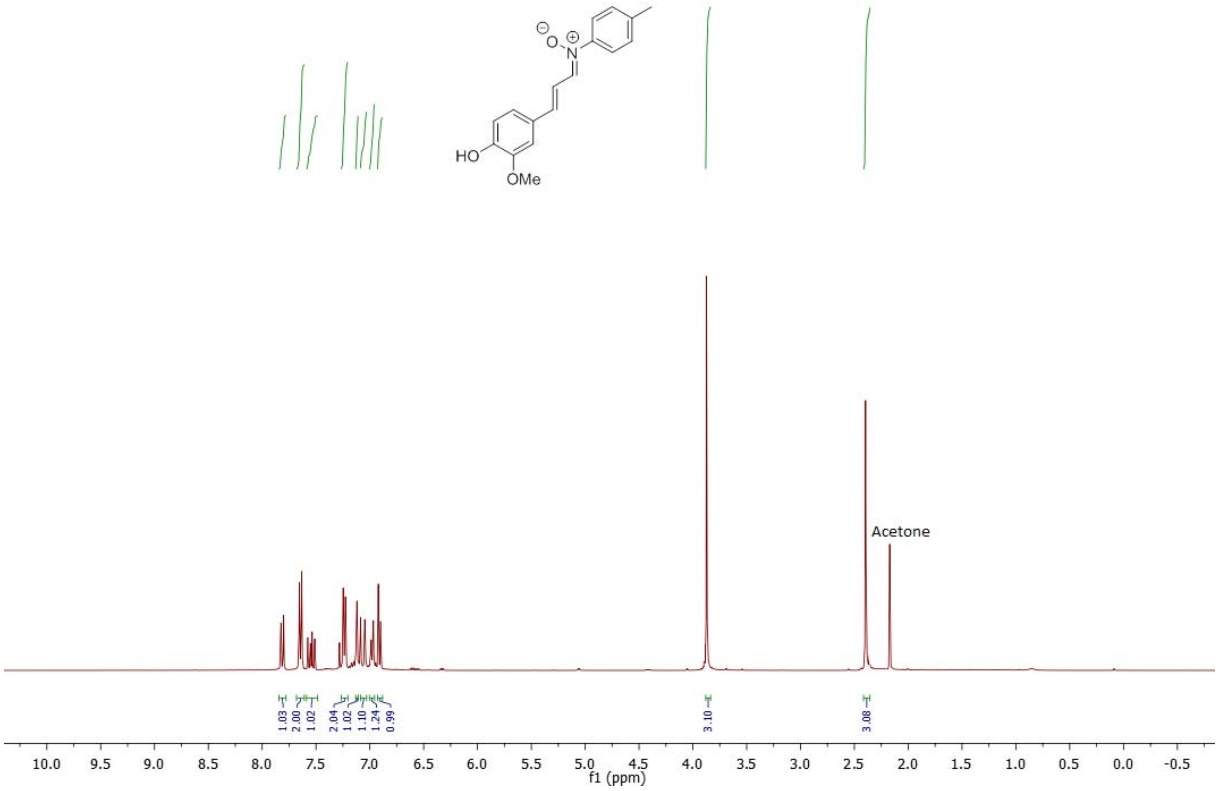
($CDCl_3$, 300 MHz)

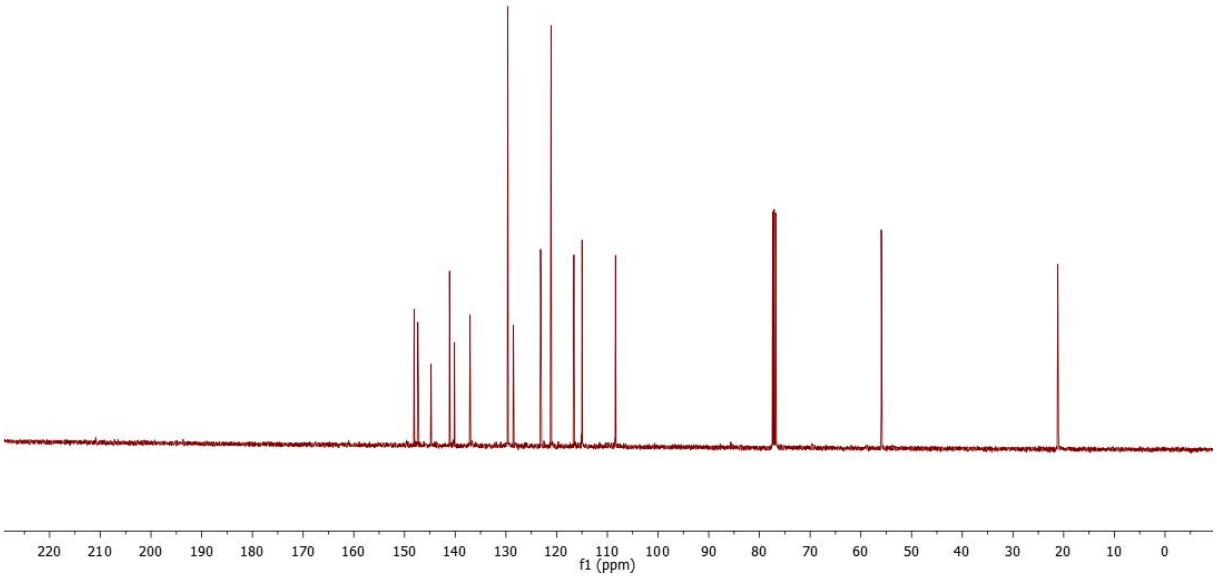




(*Z*)-*N*-((*E*)-3-(4-hydroxy-3-methoxyphenyl)allylidene)4-methylaniline oxide (3ca)

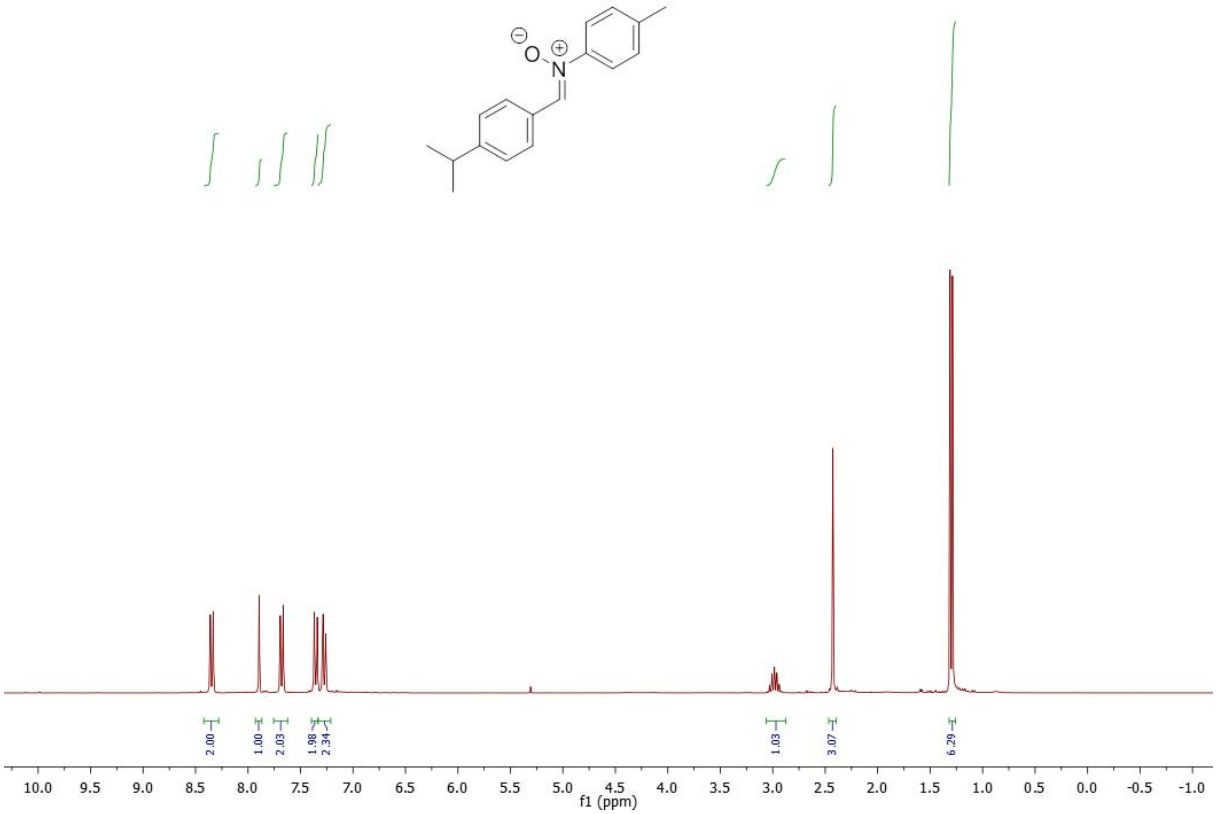
($CDCl_3$, 300 MHz)

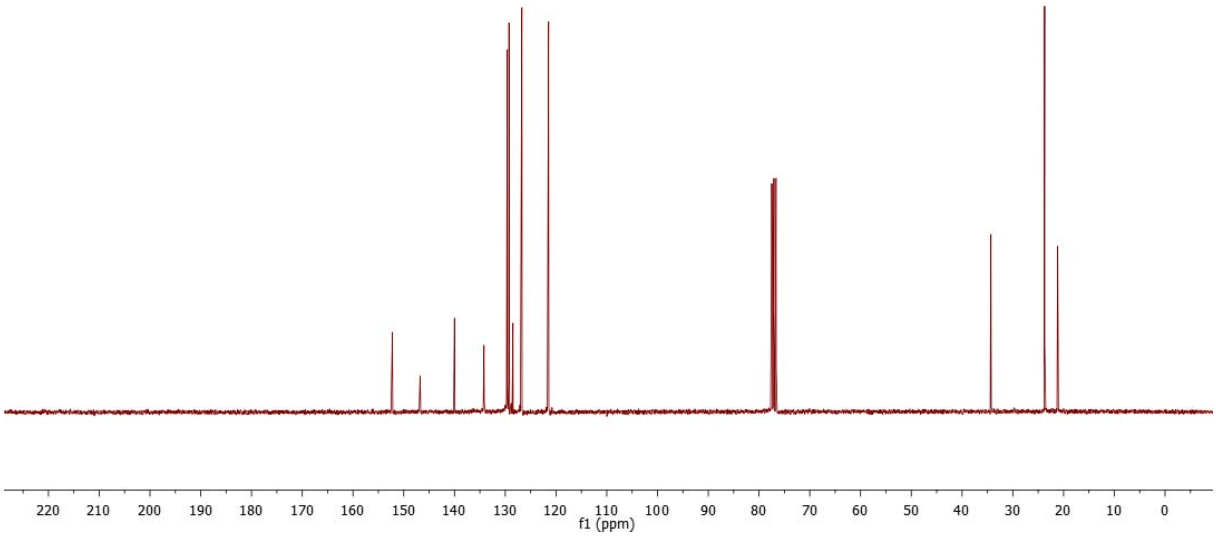




(*Z*)-*N*-(4-isopropylbenzylidene)-4-methylaniline oxide (3da)

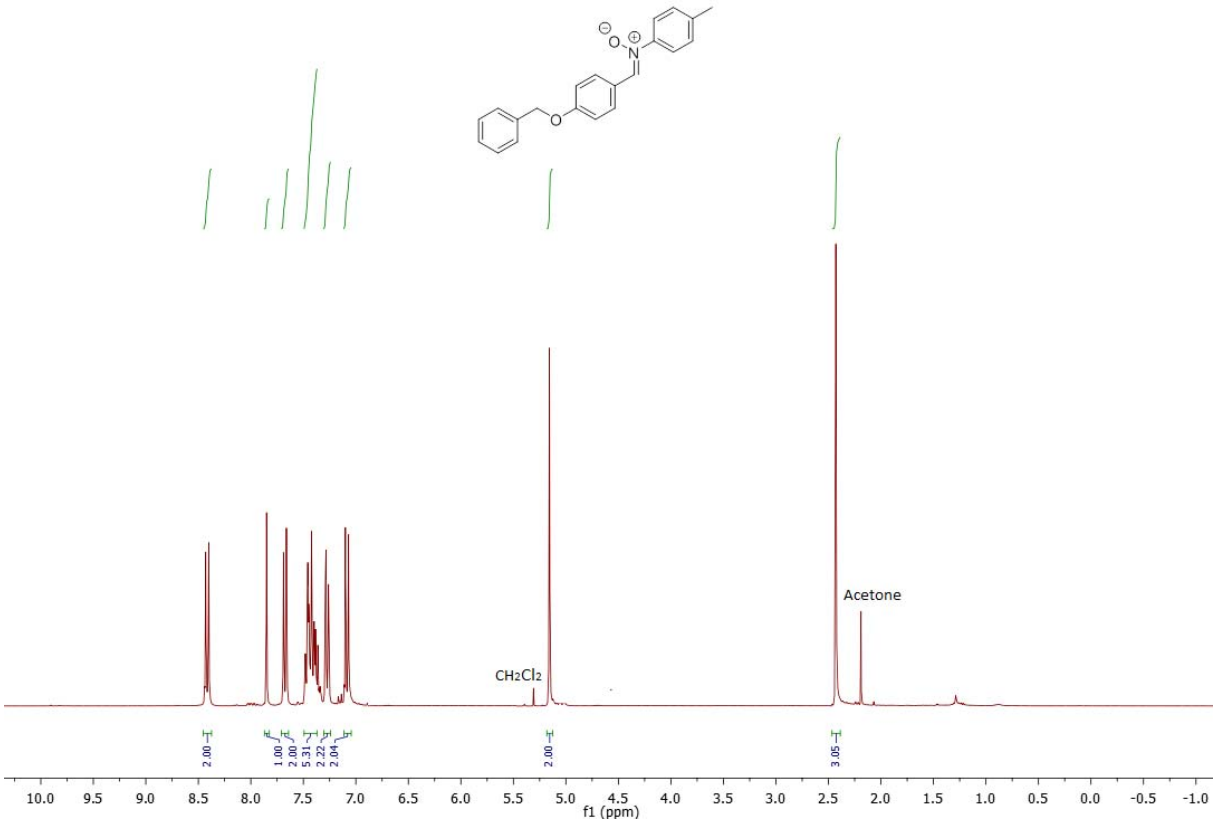
($CDCl_3$, 300 MHz)

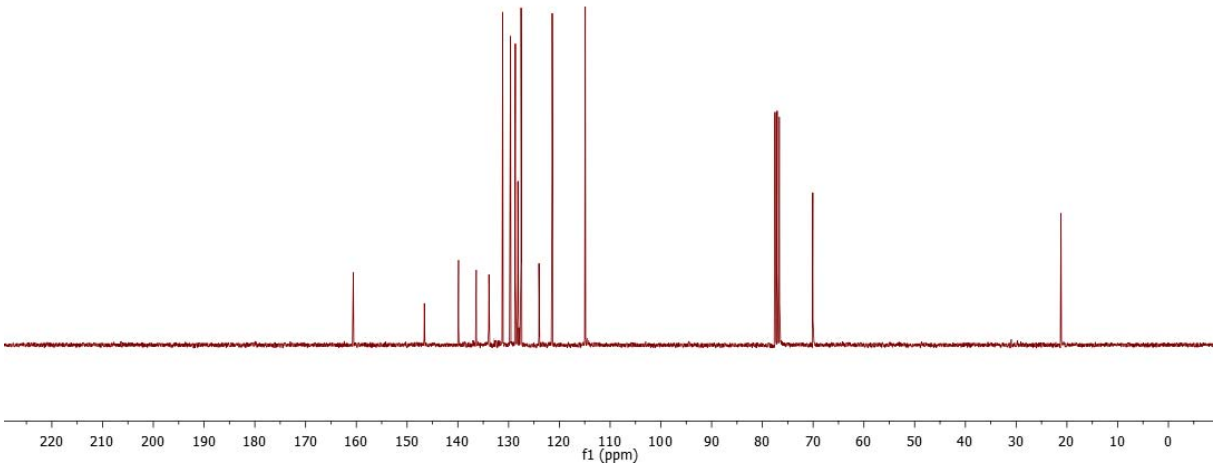




(*Z*)-*N*-(4-(benzyloxy)benzylidene)-4-methylaniline oxide (3ea)

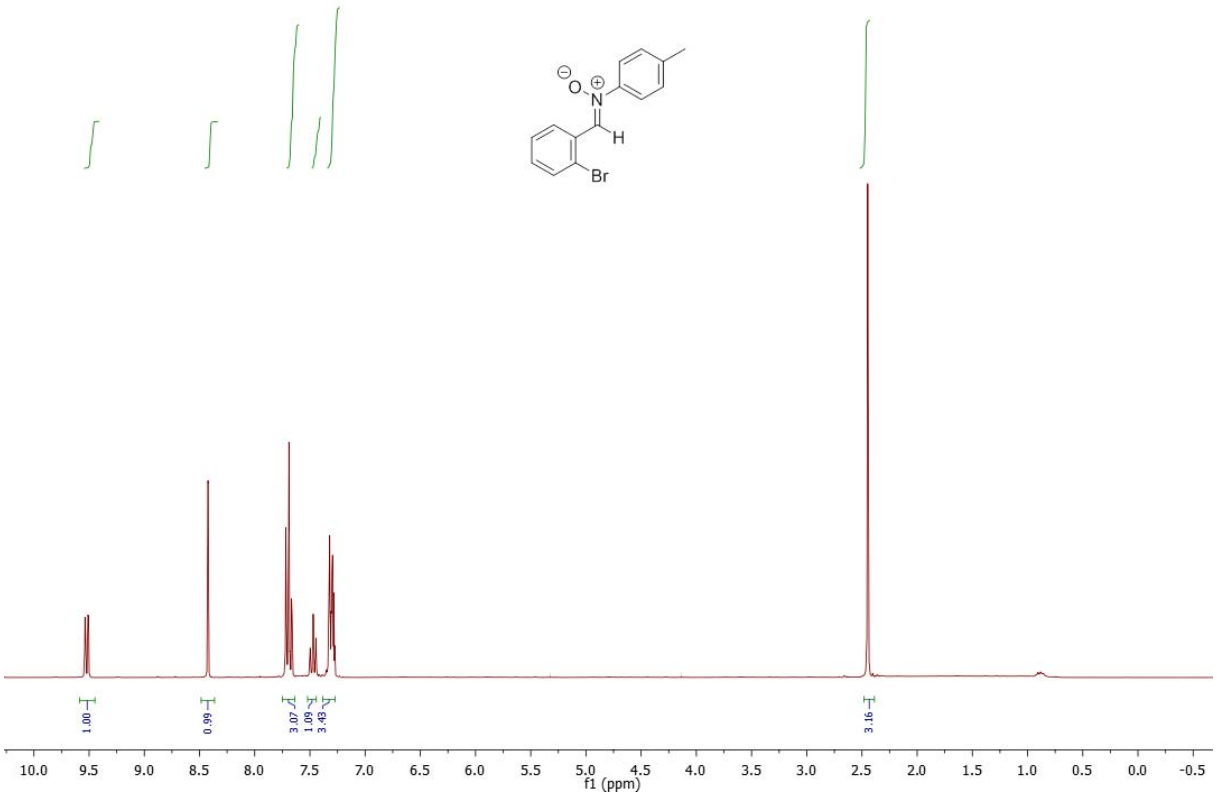
($CDCl_3$, 300 MHz)

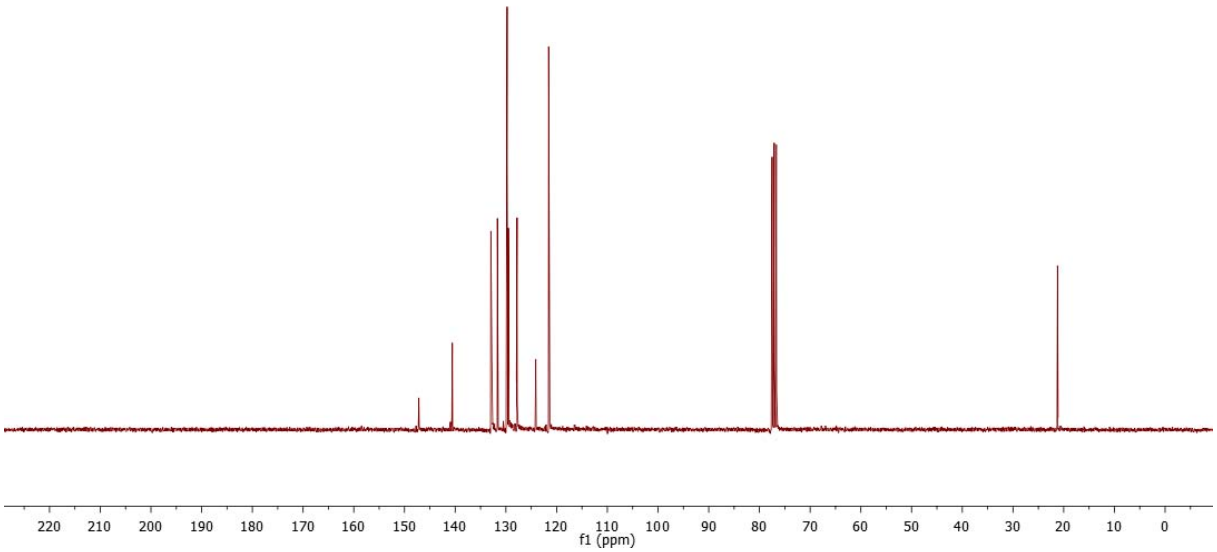




((*Z*)-*N*-(2-bromobenzylidene)-4-methylaniline oxide (3fa)

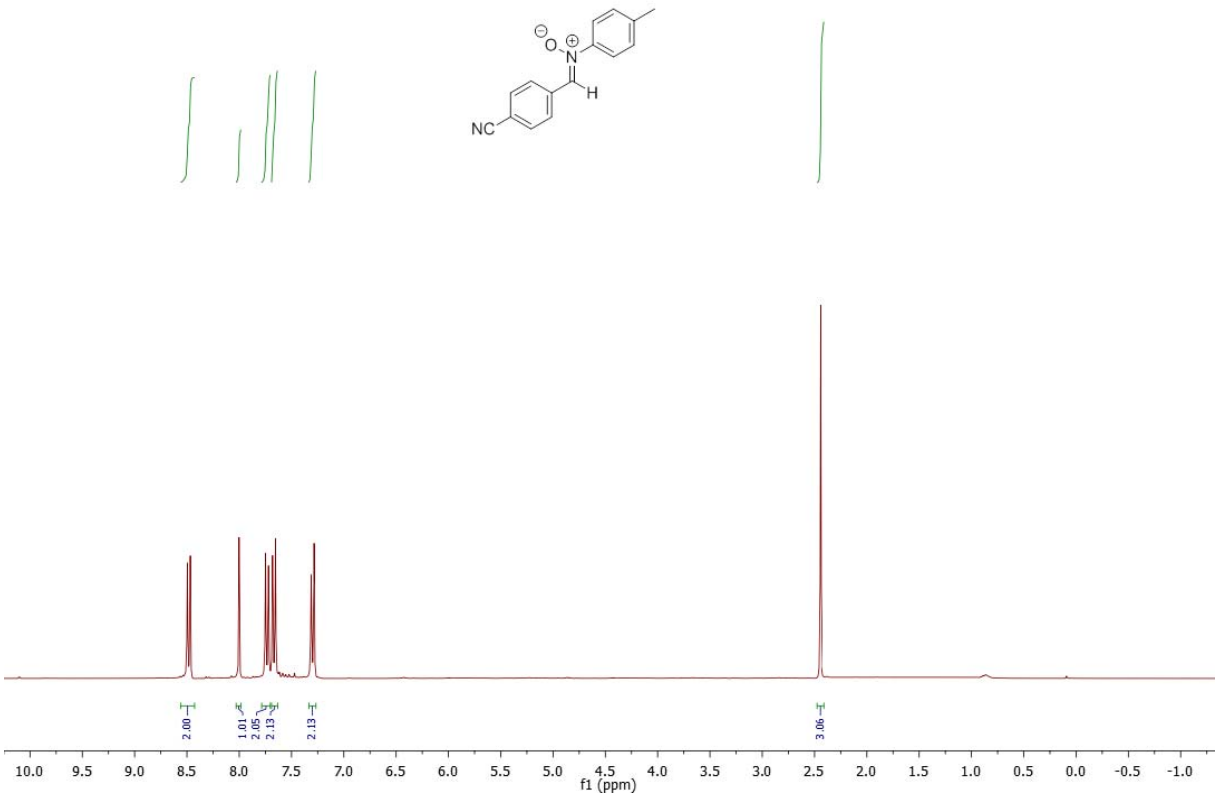
($CDCl_3$, 300 MHz)

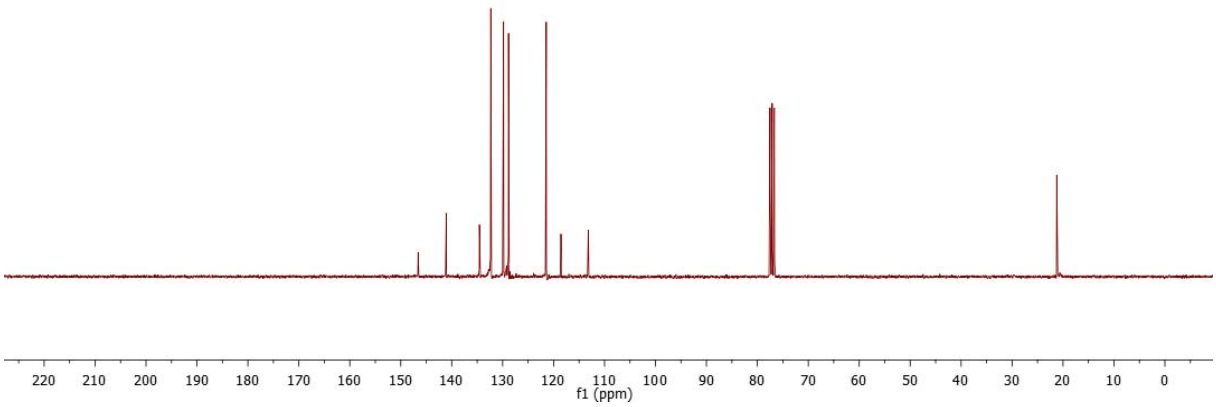




(*Z*)-*N*-(4-cyanobenzylidene)-4-methylaniline oxide (3ga)

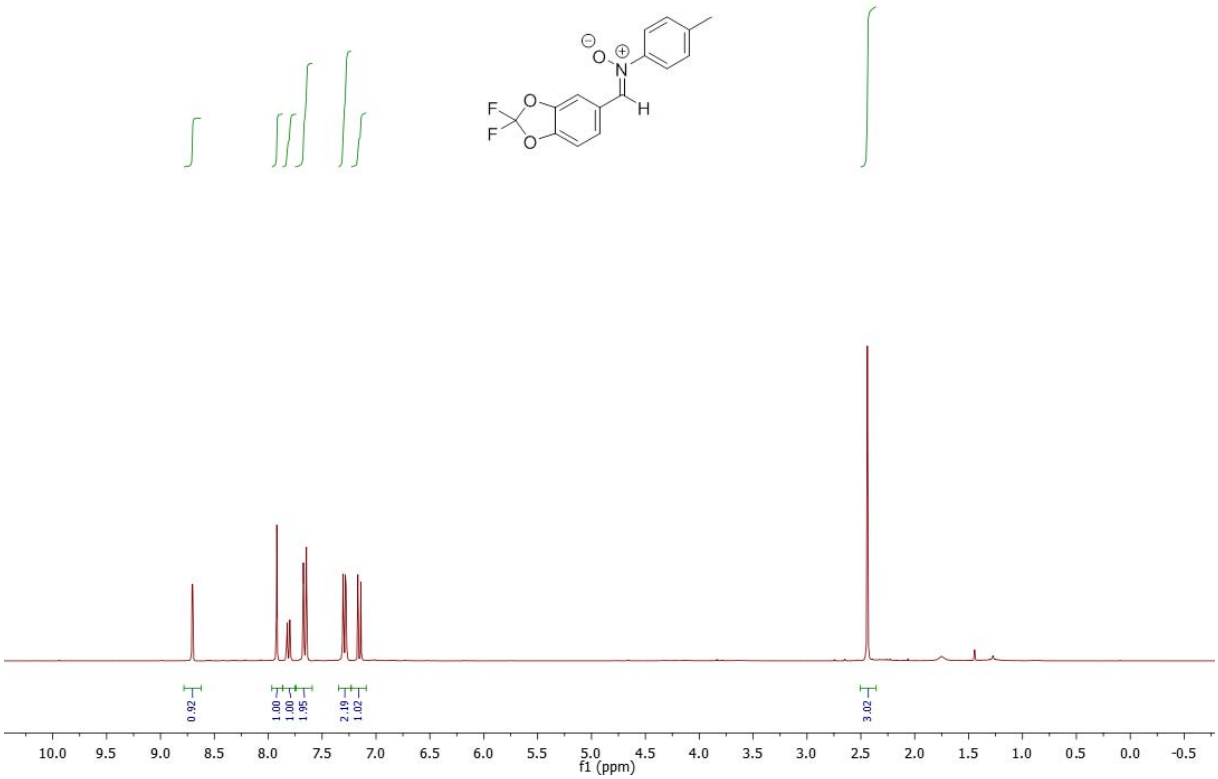
($CDCl_3$, 300 MHz)

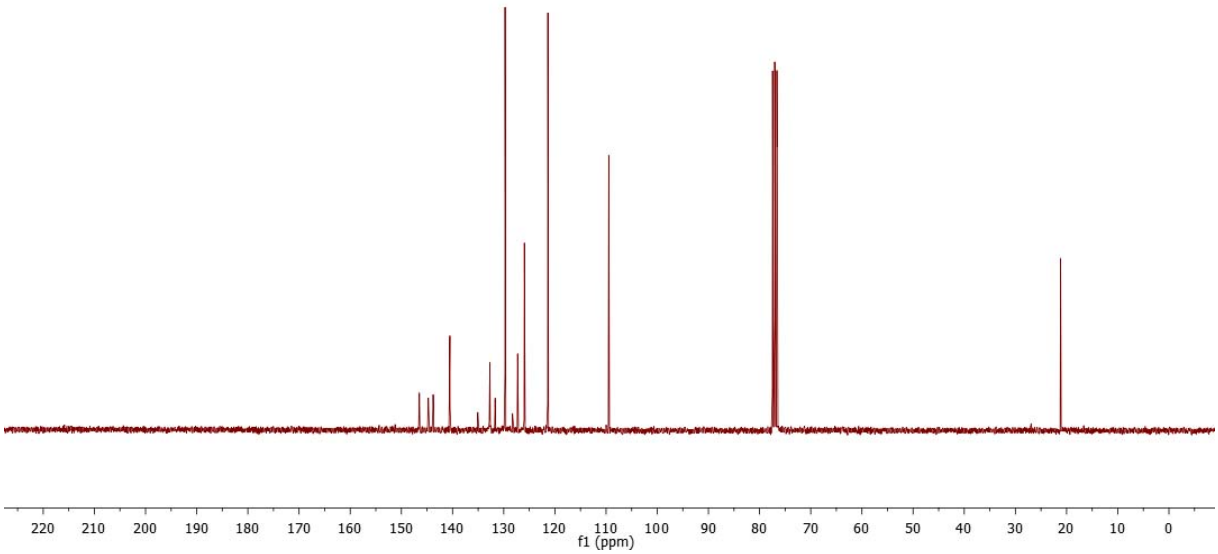




(*Z*)-*N*-((2,2-difluorobenzo[*d*][1,3]dioxol-5-yl)methylene)-4-methylaniline oxide (3ha)

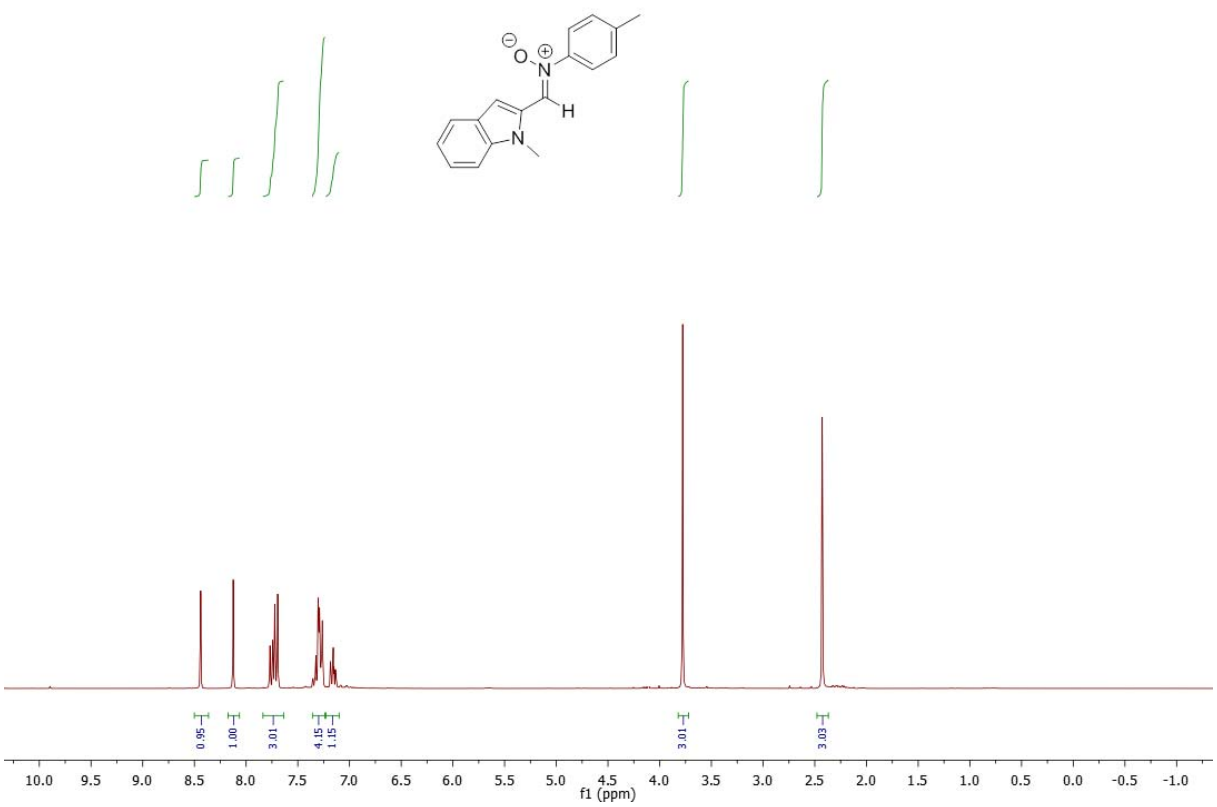
($CDCl_3$, 300 MHz)

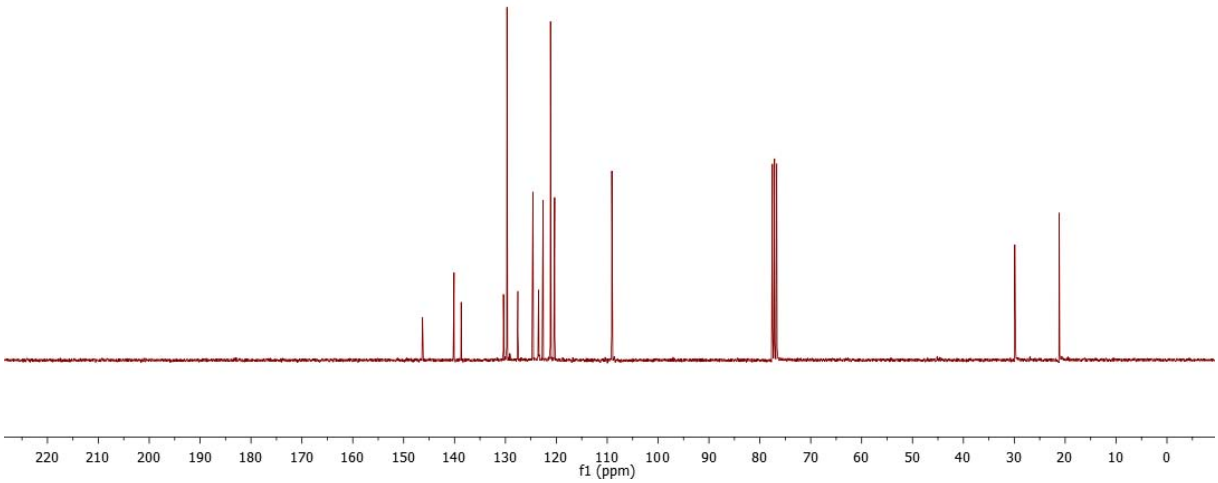




(*Z*)-4-methyl-*N*-((1-methyl-1*H*-indol-2-yl)methylene)aniline oxide (3ia)

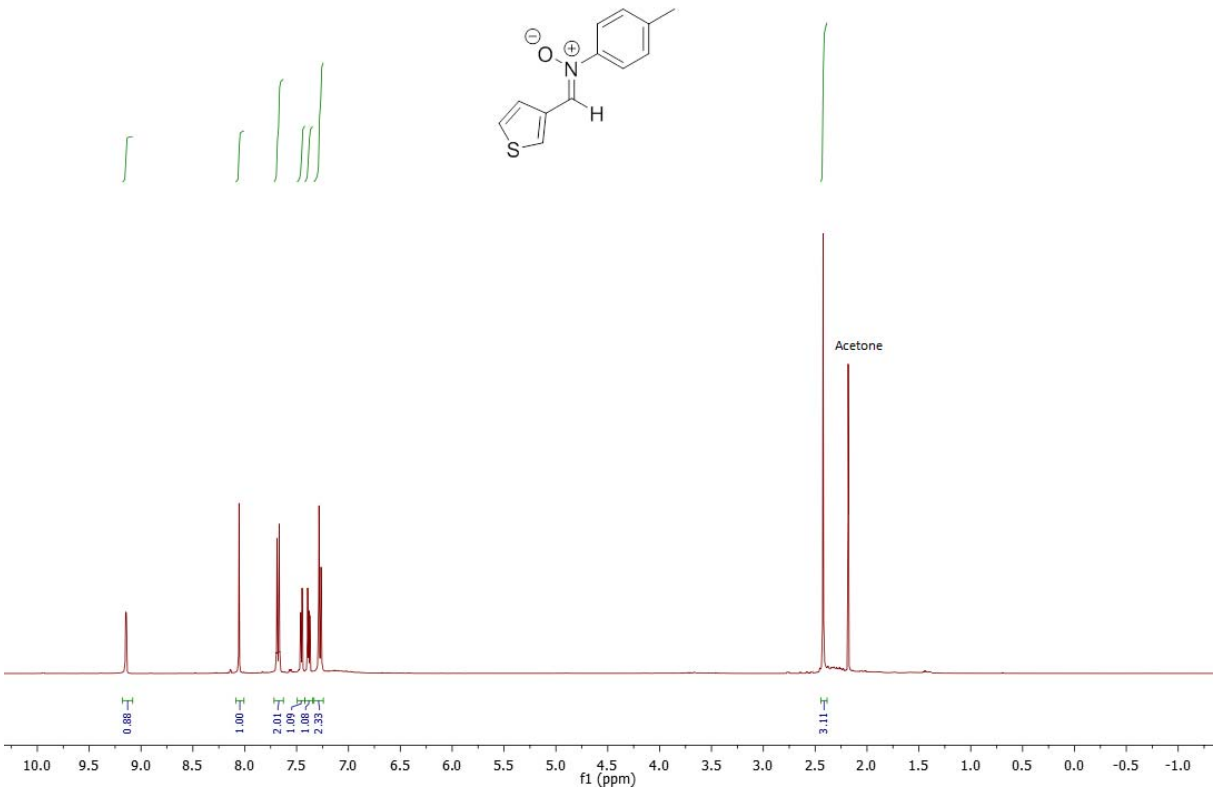
($CDCl_3$, 300 MHz)

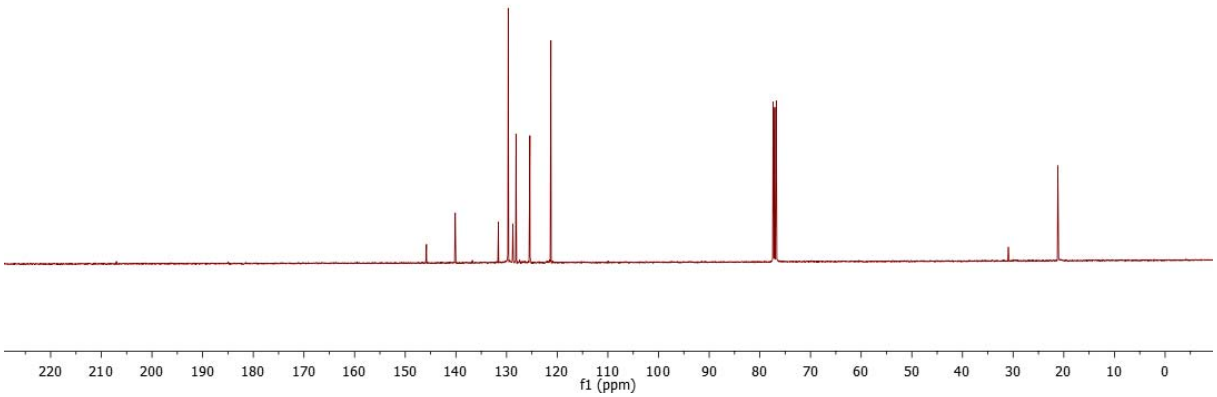




(*Z*)-4-methyl-*N*-(thiophen-3-ylmethylene)aniline oxide (3ja)

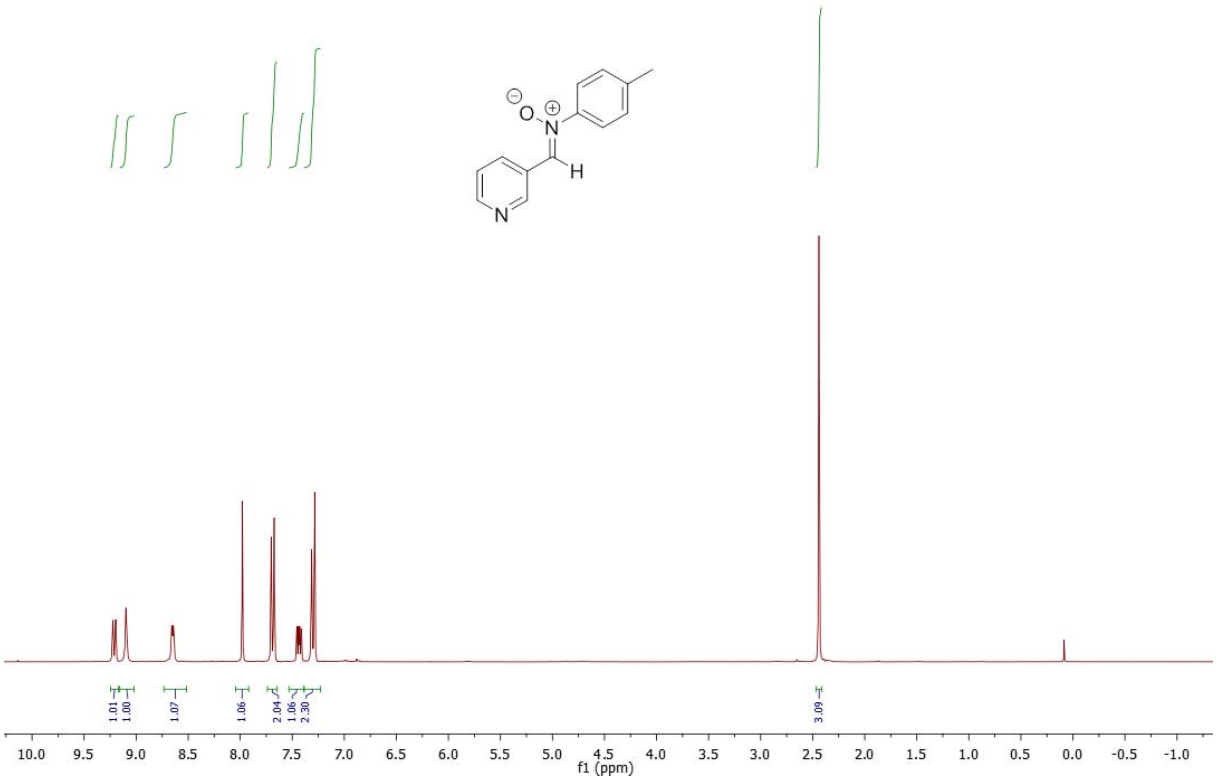
($CDCl_3$, 300 MHz)

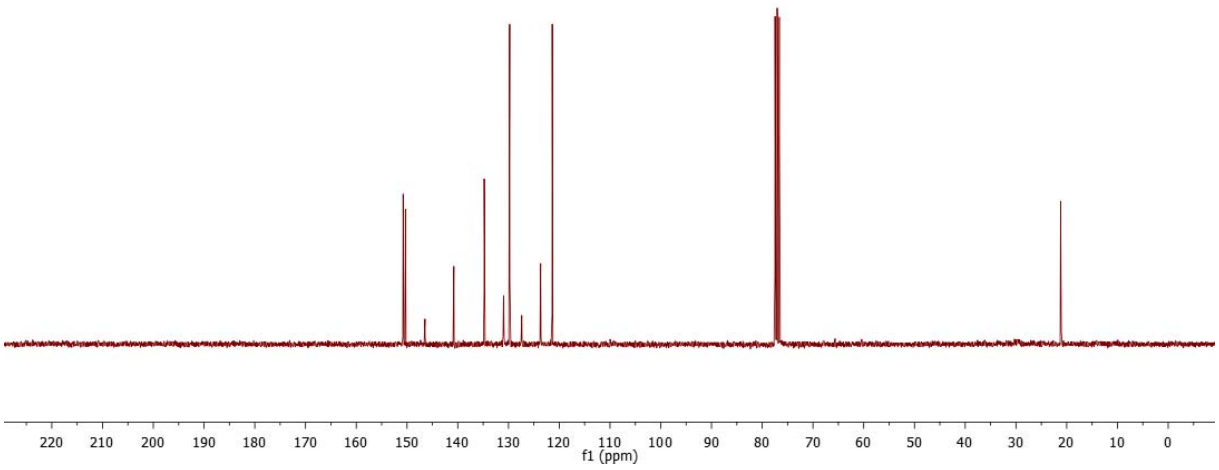




(*Z*)-4-methyl-*N*-(pyridin-3-ylmethylene)aniline oxide (3ka)

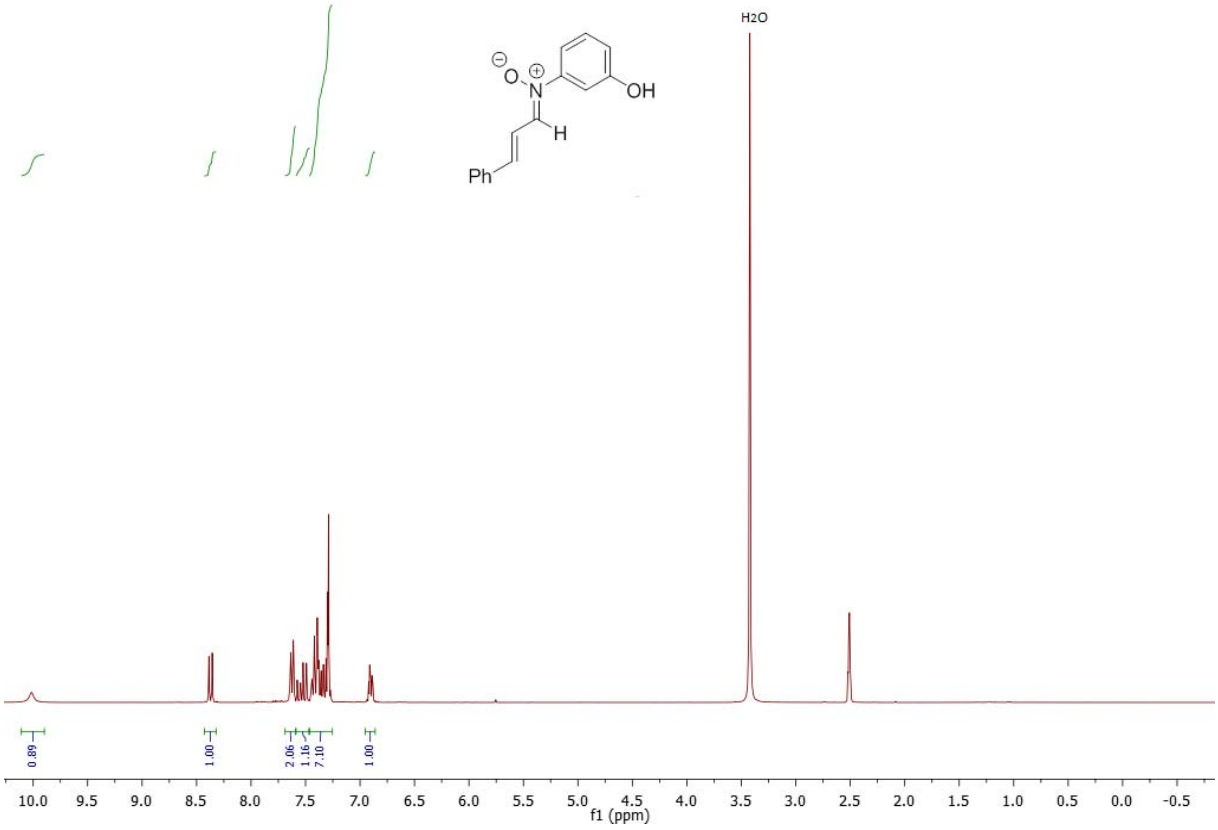
($CDCl_3$, 300 MHz)

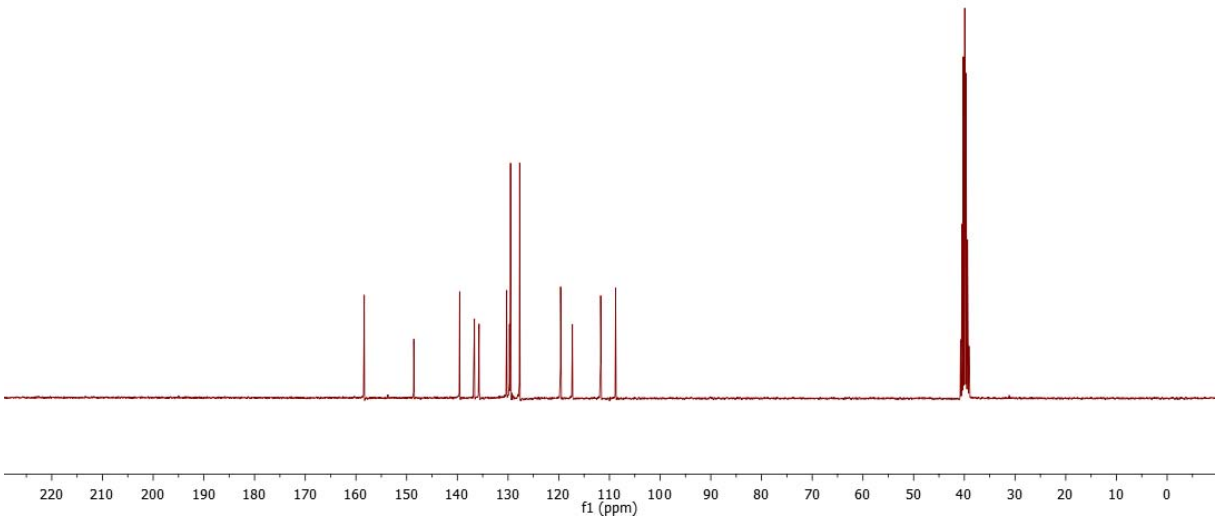




(*Z*)-3-hydroxy-*N*-((*E*)-3-phenylallylidene)aniline oxide (3bb)

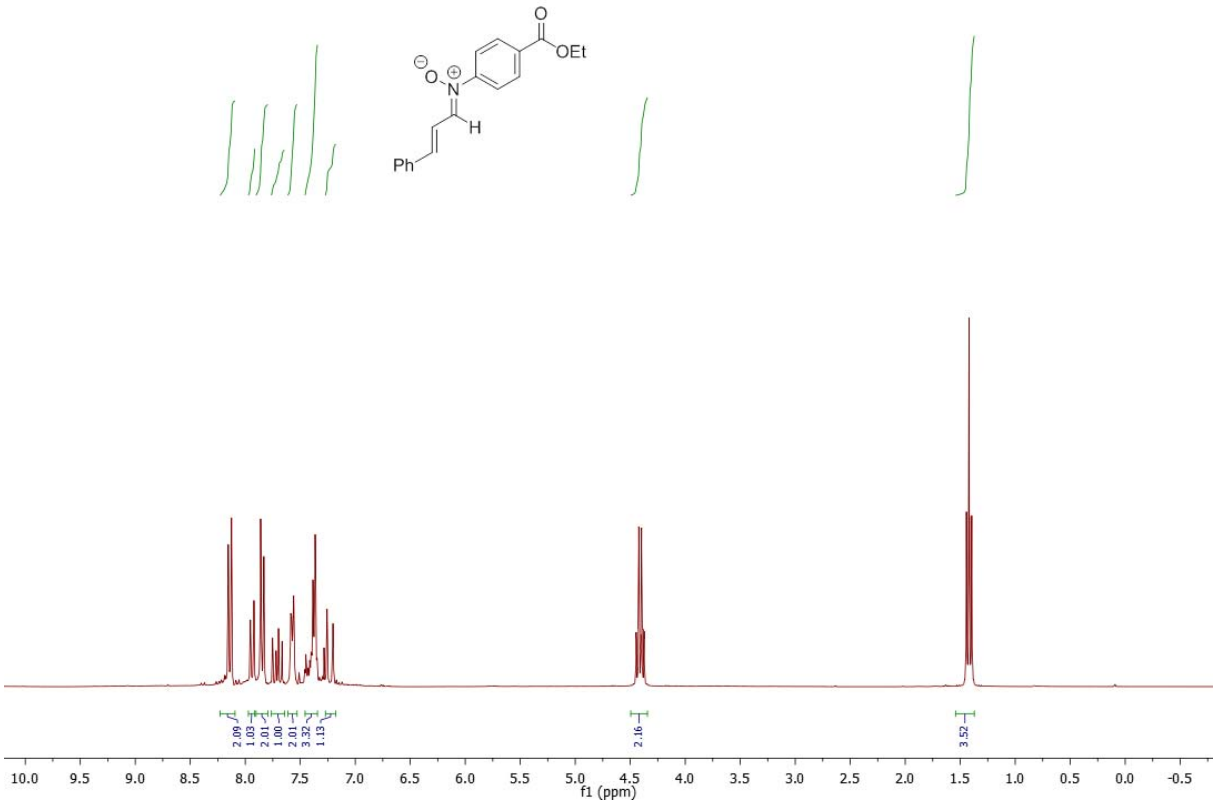
(DMSO, 300 MHz)

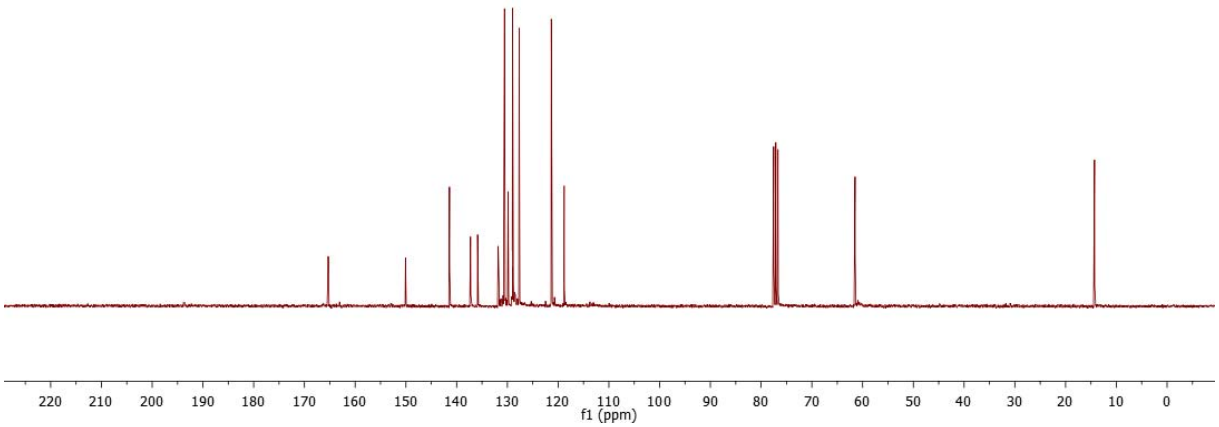




(*Z*)-4-(ethoxycarbonyl)-*N*-((*E*)-3-phenylallylidene)aniline oxide (3bc)

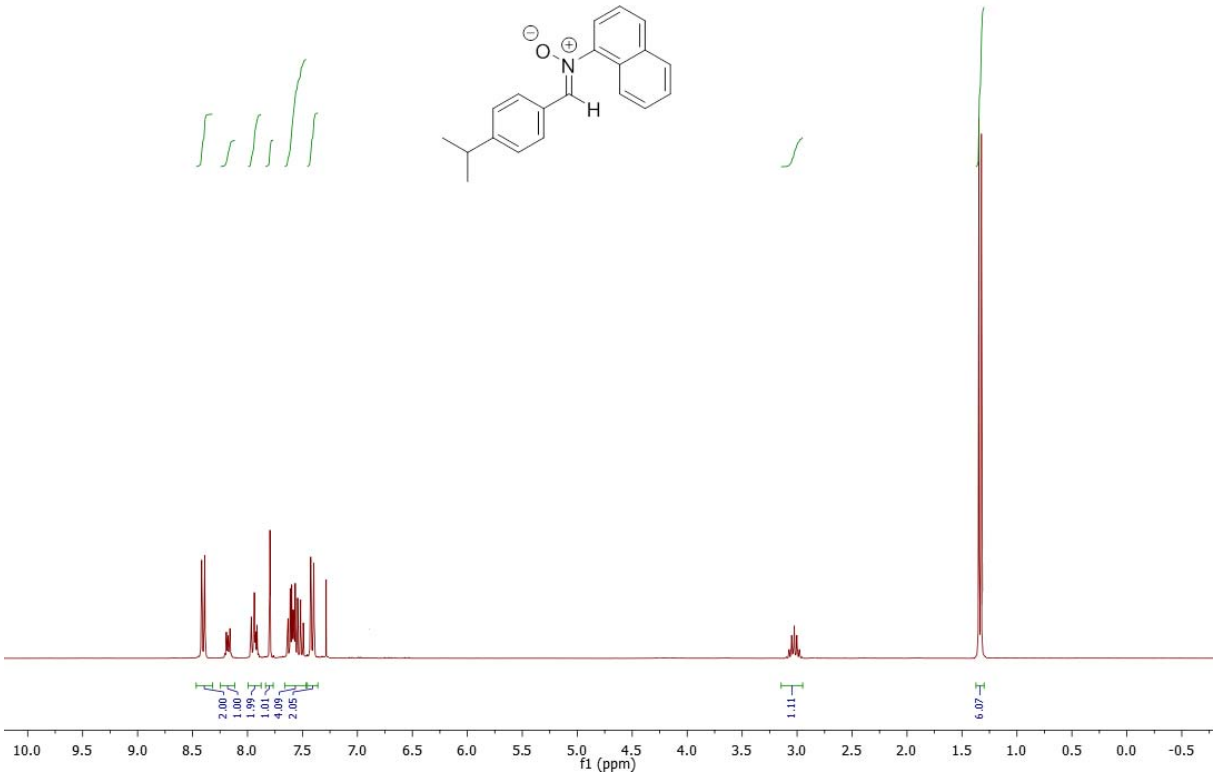
($CDCl_3$, 300 MHz)

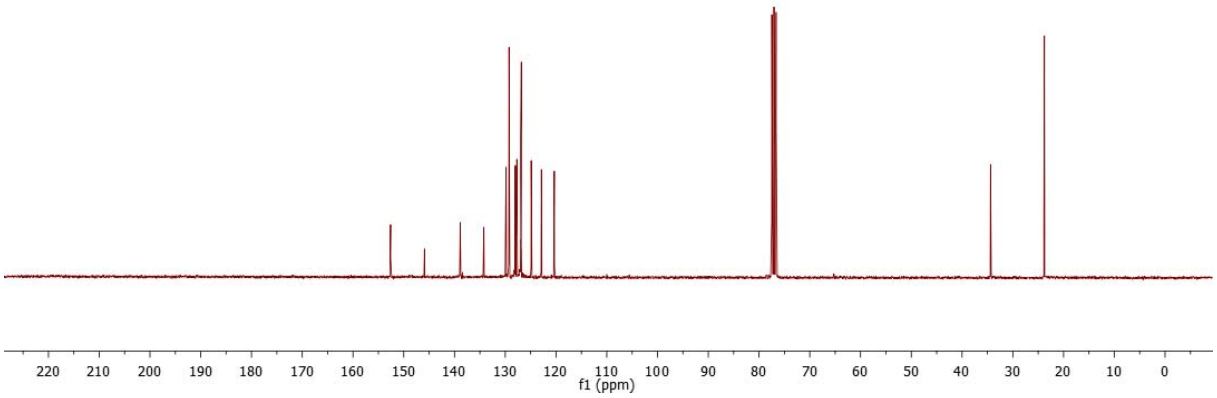




(*Z*)-*N*-(4-isopropylbenzylidene)naphthalen-1-amine oxide (3dd)

($CDCl_3$, 300 MHz)





(*Z*)-*N*-(4-isopropylbenzylidene)aniline oxide (3de)

($CDCl_3$, 300 MHz)