

Supporting Information

Facile One-Pot Direct Arylation and Alkylation of Nitropyridine N-oxides with Grignard Reagents

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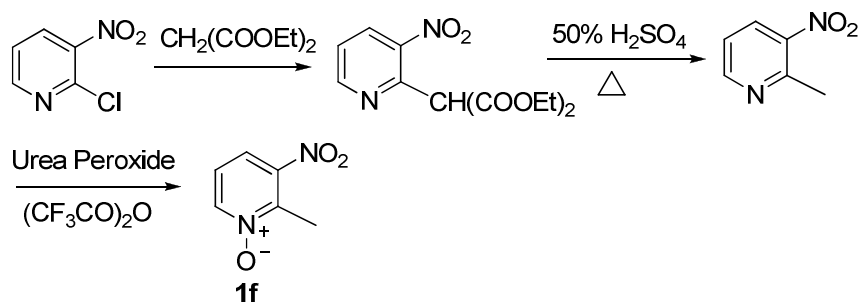
General

All NMR spectra were collected using a 400 MHz or 500 MHz Bruker NMR (100 MHz or 125 MHz for ^{13}C spectroscopy) and all spectra recorded in CDCl_3 with tetramethylsilane as an internal standard unless otherwise noted. EI mass spectra were recorded on TRACE MS spectrometer. Infrared data were acquired using an AVATAR 360 FT-IR spectrophotometer. Melting points recorded on a TECH X-4 microscopic instrument and are uncorrected.

All reagents and solvents used for Grignard reagents and reactions were freshly dehydrated before use. The corresponding glassware was oven dried ($120\text{ }^\circ\text{C}$) and cooled under a stream of argon gas. Functionalized Grignard reagents such as 2-(methoxycarbonyl)phenyl magnesium chloride, 2-cyanophenyl magnesium chloride and heteroaromatic Grignard reagents such as 2- or 3-pyridinyl magnesium chloride were prepared via iodine or bromine-magnesium exchange using *i*-PrMgCl·LiCl according to the Knochel's method¹ and titrated before use.² Other Grignard reagents were purchased or prepared according to standard procedure and titrated before use.² 4-Nitropyridine N-oxides were prepared via nitration of the corresponding N-oxides according to the literature.³

Typical procedure: A dry argon-flushed 25-mL flask, equipped with a magnetic stirrer and a septum, was charged with 2-methyl-4-nitropyridine N-oxide (**1a**, 500 mg, 3.2 mmol). Dry THF (80 mL) was added, the mixture was cooled to $-60\text{ }^\circ\text{C}$, and PhMgBr (**2a**, 3.3 mL, 1.2 M in THF, 4.0 mmol) was then added dropwise. The addition was complete after 1-1.5 h (checked by TLC). DDQ (900 mg, 4.0 mmol) was added. The mixture was then allowed to come to room temperature and stirred for 4 h. The reaction was quenched with a 20% solution of Na_2CO_3 (20 mL) and THF was removed by distillation in vacuo. The aqueous phase was extracted with CH_2Cl_2 ($5 \times 40\text{ mL}$), and the organic fractions were dried (Na_2SO_4), and concentrated in vacuo. The crude residue was purified by flash chromatography (petroleum ether/ethyl acetate = 5:1; $R_f = 0.25$) to yield the oxide **3aa** as a yellow solid (677 mg, 92%).

Preparation of 3-nitro-2-picoline N-oxide (**1f**) and 3-nitro-6-picoline N-oxide (**1g**)

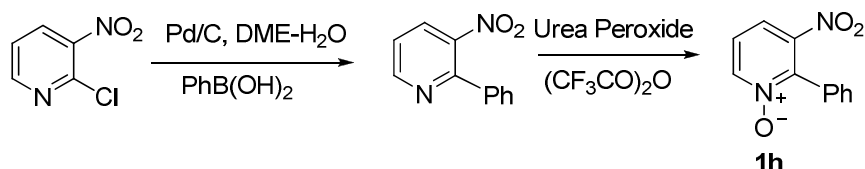


3-Nitro-2-picoline (1.4g, 0.01 mol) [prepared from 2-chloro-3-nitropyridine according to the reported method⁴ (as illustrated above) and was used without purification] was

dissolved in CH_2Cl_2 (30 mL) and urea peroxide (2.0g, 0.02 mol) was added. The mixture was stirred and cooled to $0 \sim 5^\circ\text{C}$. To the mixture $(\text{CF}_3\text{CO})_2\text{O}$ (5 mL) was added dropwise. After stirring at that temperature for 30 min, the mixture was allowed to warm to room temperature and stirred until the oxidation was completed. The reaction was quenched with an aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$ to destroy any residual peroxides before being poured into a saturated aqueous solution of NaHCO_3 and extracted with CH_2Cl_2 . The combined organic layers were dried over Na_2SO_4 . Concentration by rotary evaporation afforded the crude product. Purification flash column chromatography over silica (ethyl acetate = 5:1; $R_f = 0.31$) afforded 3-nitro-2-picoline N-oxide (**1f**) in a 76% yield.

3-Nitro-6-picoline N-oxide (**1g**) was prepared in a similar manner from 2-chloro-5-nitropyridine.

Preparation of 3-nitro-2-phenylpyridine N-oxide (**1h**) and 2-phenyl-5-nitropyridine N-oxide (**1i**)



(1) 3-Nitro-2-phenylpyridine

According to reported procedure:⁵ 2-Chloro-3-nitropyridine (5 g, 0.032 mol) and phenylboronic acid (6.0 g, 0.049 mol) were dissolved in a mixture of DME (120 mL) and an aqueous solution of Na_2CO_3 (2.0 M, 60 mL). Pd-C (1.9g, 10%) was added. The mixture was stirred at 80°C for 9h. It was filtered and extracted with ethyl acetate. The combined organic layers was washed with saturated aqueous solution of Na_2CO_3 and dried over Na_2SO_4 . After concentration by rotary evaporation the residue was used directly in next step.

(2) 3-Nitro-2-phenylpyridine (**1h**)

The residue was dissolved in CH_2Cl_2 (200 mL) and urea peroxide (6.0g, 0.064 mol) was added. The mixture was stirred and cooled to $0 \sim 5^\circ\text{C}$. To the mixture $(\text{CF}_3\text{CO})_2\text{O}$ (15 mL) was added dropwise. After stirring at that temperature for 30 min, the mixture was allowed to warm to room temperature and stirred until the oxidation was completed. The reaction was quenched with an aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$ to destroy any residual peroxides before being poured into a saturated aqueous solution of NaHCO_3 and extracted with CH_2Cl_2 . The combined organic layers were dried over Na_2SO_4 . After concentration by rotary evaporation the residue was purified by flash column chromatography over silica (petroleum ether/ethyl acetate = 2:1; $R_f = 0.30$) to afford 3-nitro-2-phenylpyridine N-oxide (**1h**) in an overall yield of 62%.

Preparation of Emoxipin (Scheme 4)

(1) 2-Ethyl-6-methyl-3-nitropyridine 1-oxide (**3gm**)

Under argon, a four-necked flask equipped with a magnetic stirrer was charged with 6-methyl-3-nitropyridine 1-oxide (**1g**) (500 mg, 3.2 mmol) and 80 mL of THF. The

mixture was cooled to -60 °C, and EtMgBr (**2m**) (4.0 mmol) was added slowly with a syringe while the reaction temperature was kept under -60 °C. The mixture was stirred at -60 °C until starting material was consumed (monitored by TLC). DDQ (900 mg, 4.0 mmol) was added. The mixture was then allowed to come to room temperature and stirred for 4 h. The reaction was quenched with a 20% solution of Na₂CO₃ (20 mL) and THF was removed by rotary evaporation in vacuo. The aqueous phase was extracted with CH₂Cl₂ (5 × 40 mL), and the organic fractions were dried (Na₂SO₄), and concentrated in vacuo. The crude residue was purified by flash chromatography (ethyl acetate/ethanol = 5:1; R_f = 0.35) to yield the oxide **3gm** (542 mg, 92%).

(2) 2-Ethyl-6-methylpyridin-3-amine (**5**)

A four-necked flask equipped with a magnetic stirrer was charged with **3gm** (182 mg, 1 mmol) and 50 mL of acetic acid. Iron powder (0.18g, 3.2mol) was added and the mixture was heated at 110 °C with stirring for 1h. Another portion of iron powder (0.18g, 3.2mol) was added and the mixture was heated at 110 °C with stirring for 4 h. A grey pasty mixture was formed. It was cooled to room temperature and was then made alkaline with 10% sodium hydroxide. The mixture was extracted with ether (5 × 50 mL), and the organic fractions were dried (Na₂SO₄). The product was afforded in a yield of 98% (133mg) after concentration by rotary evaporation.

(3) 2-Ethyl-6-methylpyridin-3-ol (**Emoxipin**)

A four-necked flask equipped with a magnetic stirrer was charged with **5** (110 mg, 0.81 mmol), and 5% sulfuric acid (5.0 mL). The mixture was cooled to 0 °C with stirring and a solution of sodium nitrite (0.3 g) in 2.3 mL of water was added dropwise at 0 –5°C. The solution was maintained at 0 °C for an additional 30 min and transferred into an additional funnel maintained at 0 °C with external cooling, and added dropwise into boiling 5% aqueous sulfuric acid (5 mL) over a period of 10 min. The resultant solution was refluxed for an additional 15 min, cooled to 0 °C, neutralized with 40% aqueous sodium hydroxide, and saturated by the addition of solid sodium chloride. The product was extracted with methylene chloride. The organic extract was dried over anhydrous sodium sulfate, filtered, and concentrated by rotary evaporation to yield 100 mg (90%) of 2-ethyl-6-methylpyridin-3-ol.

Preparation of Caerulomycin A and E (Scheme 5)

(1) 2-Methyl-4-nitro-6-(pyridin-2-yl)pyridine 1-oxide (**3at**)

Under argon, a four-necked flask equipped with a magnetic stirrer was charged with 2-methyl-4-nitropyridine 1-oxide (**1a**) (500 mg, 3.2 mmol) and 80 mL of THF. The mixture was cooled to -60 °C, and 2-PyMgCl·LiCl (prepared from 2-bromopyridine and *i*-PrMgCl·LiCl according to the Knochel's method¹ and titrated before use.²) (4.0 mmol) was added slowly with a syringe while the reaction temperature was kept under -60 °C. The mixture was stirred at -60 °C until the starting material was consumed (monitored by TLC). DDQ (900 mg, 4.0 mmol) was added. The mixture was then allowed to come to room temperature and stirred for 4 h. The reaction was quenched with a 20% solution of Na₂CO₃ (20 mL) and THF was removed by rotary evaporation in vacuo. The aqueous phase was extracted with CH₂Cl₂ (5 × 40 mL), and

the organic fractions were dried (Na_2SO_4), and concentrated in vacuo. The crude residue was purified by flash chromatography (petroleum ether/ethyl acetate = 5:1; R_f = 0.32) to yield the oxide **3at** (436 mg, 59%).

(2) 4-Methoxy-2-methyl-6-(pyridin-2-yl)pyridine 1-oxide (**6**)

A four-necked flask equipped with a magnetic stirrer was charged with **3at** (540 mg, 2.3 mmol) and 25 mL of CH_3OH . The mixture was heated to 60 °C with stirring and a solution of CH_3ONa in CH_3OH (8.1 mL, 2.58 mmol) was added dropwise. It was stirred at 60 °C until compound **3at** was consumed (monitored by TLC). The mixture was cooled to room temperature and 10 mL of water was added. After removal of methanol by rotary evaporation, the aqueous solution was extracted with CH_2Cl_2 (3 × 40 mL), and the organic fractions were dried (Na_2SO_4), and concentrated in vacuo. The product was given in a yield of 95% (480 mg) and used in next step without purification.

(3) (4-methoxy-2,2'-bipyridin-6-yl)methanol (**7**)

A four-necked flask equipped with a magnetic stirrer was charged with **6** (540 mg, 2.5 mmol) and 10 mL of acetic anhydride. The mixture was stirred at 110 °C until the oxide **6** was consumed (monitored by TLC). It was cooled to room temperature and was then made alkaline with a 20% solution of Na_2CO_3 . The mixture was extracted with CH_2Cl_2 (3 × 40 mL), and the organic fractions were dried (Na_2SO_4) and concentrated in vacuo. The residue was dissolved in a solution of 1 mL concentrated HCl in 8 mL H_2O , and stirred at 70 °C for 2h. After cooled to room temperature, the mixture was made alkaline with a 20% solution of Na_2CO_3 and then extracted with CH_2Cl_2 (3 × 40 mL). The organic fractions were dried (Na_2SO_4) and concentrated in vacuo. The crude residue was purified by flash chromatography (petroleum ether /ethyl acetate = 1:1; R_f = 0.37) to yield **7** (510 mg, 95%).

(4) Caerulomycin E

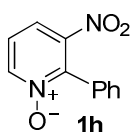
A flask equipped with a magnetic stirrer was charged with **7** (110 mg, 0.5 mmol), MnO_2 (440 mg, 5 mmol) and 10 mL of CHCl_3 . The suspension was stirred at room temperature until **7** was consumed (monitored by TLC). The mixture was filtered through a short silica gel pad and the filtrate was collected and concentrated by rotary evaporation. The product (petroleum ether/ethyl acetate = 1:1; R_f = 0.61) was obtained in a yield of 97% (104 mg).

(5) Caerulomycin A

To a stirred solution of **7** (120 mg 0.56 mmol) in 10 mL CHCl_3 , MnO_2 (500mg, 5.7 mmol) was added. The mixture was stirred at room temperature until the oxidation was completed (monitored by TLC). The mixture was filtered through a short silica gel pad and the filtrate was collected and concentrated. The residue was dissolved in 10 mL ethanol. Hydroxylammonium chloride (200 mg, 2.8 mol) and pyridine (0.20 mL, 2.4 mmol) were added to the solution and the resulting mixture was heated to reflux until the reaction was completed (monitored by TLC). The solution was then concentrated to afford the crude product which was purified by flash column chromatography over silica (petroleum ether/ethyl acetate = 1:1; R_f = 0.22) to give a white solid in a 87% yield.

Compound characterization

2-Phenyl-3-nitropyridine N-oxide (1h)



Yellow solid, m.p. 144-146 °C, R_f = 0.30 (petroleum ether/ethyl acetate = 2:1).

IR (cm^{-1} , KBr): 3048, 3022, 1534, 1347, 1260, 1011, 823, 734, 696.

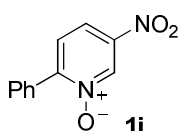
^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.31 (d, J = 6.5 Hz, 1H), 7.47 (d, J = 8.3 Hz, 1H), 7.32-7.31 (m, 3H), 7.25-7.19 (m, 3H).

^{13}C NMR (100MHz, CDCl_3) δ (ppm): 149.5, 145.5, 142.9, 130.6, 128.9, 128.8, 127.4, 124.2, 119.7.

MS (EI): m/z (%) 216 (M, 45), 215 (M-1, 70), 171 (40), 169 (100), 138 (35).

Anal. Calcd for $\text{C}_{11}\text{H}_8\text{N}_2\text{O}_3$: C, 61.11; H, 3.73; N, 12.96; Found: C, 60.96; H, 3.54; N, 12.74.

6-Phenyl-3-nitropyridine N-oxide (1i)



Yellow solid, m.p. 180-182 °C, R_f = 0.59 (petroleum ether/ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3131, 3059, 1561, 1519, 1374, 1350, 1258, 1245, 1014, 818, 734.

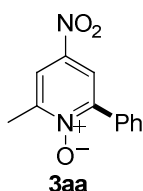
^1H NMR (400 MHz, CDCl_3) δ (ppm): 9.18 (d, J = 2.0 Hz, 1H), 8.08 - 8.05 (dd, J = 2.0 and 8.8 Hz, 1H), 7.88-7.86 (m, 2H), 7.64 (d, J = 2.9 Hz, 1H), 7.55-7.54 (m, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 154.4, 145.1, 136.9, 131.1, 130.6, 129.3, 128.6, 126.8, 119.4.

MS (EI): m/z (%) 217 (M+1, 15), 216 (M, 25), 215 (M-1, 30), 139 (40), 137 (100), 77 (45), 63 (40), 51 (60).

Anal. Calcd for $\text{C}_{11}\text{H}_8\text{N}_2\text{O}_3$: C, 61.11; H, 3.73; N, 12.96; Found: C, 60.94; H, 4.00; N, 12.84.

2-Methyl-4-nitro-6-phenylpyridine 1-oxide (3aa)



Yellow solid, m.p. 182-184 °C; R_f = 0.25 (petroleum ether/ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3061, 2922, 1590, 1572, 1458, 1233, 1028, 804, 756, 693.

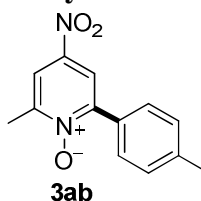
^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.11(d, J = 3.0Hz, 1H), 8.03 (d, J = 2.9 Hz, 1H), 7.72-7.70 (m, 2H), 7.45 (d, J = 3.9 Hz, 3H), 2.55 (s, 3H);

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 151.5, 150.3, 141.0, 131.5, 130.4, 129.2, 128.5, 118.9, 118.5, 18.8.

MS (EI): m/z (%) 231 (M+1, 10), 230 (M, 15), 183 (18), 126 (40), 115 (60), 77 (75), 63 (100).

Anal. Calcd for $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3$: C, 62.60; H, 4.38; N, 12.17; Found: C, 62.65; H, 4.11; N, 11.89.

2-Methyl-4-nitro-6-(*p*-tolyl)pyridine 1-oxide (3ab)



Pale yellow solid, m.p. 215-216 °C; R_f = 0.23 (petroleum ether/ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3065, 2919, 1608, 1527, 1272, 1101, 726.

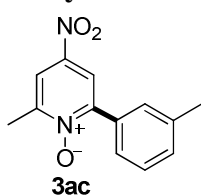
^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.09 (d, J = 3.2 Hz, 1H), 8.00 (d, J = 3.1 Hz, 1H), 7.63 (d, J = 8.2 Hz, 2H), 7.24 (d, J = 8.0 Hz, 2H), 2.54 (s, 3H), 2.36 (s, 3H).

^{13}C NMR (100MHz, CDCl_3) δ (ppm): 151.4, 150.4, 140.9, 140.8, 129.1, 129.0, 128.5, 118.7, 118.2, 21.4, 18.8.

MS (EI): m/z (%) 245 (M+1, 30), 244 (M, 100), 243 (M-1, 95), 215 (40), 197 (85), 128 (85), 115 (90), 91 (65).

Anal. Calcd for $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_3$: C, 63.93; H, 4.95; N, 11.47. Found: C, 63.58; H, 4.57; N, 11.30.

2-Methyl-4-nitro-6-(*m*-tolyl)pyridine 1-oxide (3ac)



Pale yellow solid, m.p. 189-190 °C; R_f = 0.25 (petroleum ether/ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3067, 1602, 1526, 1332, 1273, 1101, 953, 909, 804, 791, 731.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.09 (d, J = 3.2 Hz, 1H), 8.02 (d, J = 3.2 Hz, 1H), 7.53 (s, 1H), 7.48 (d, J = 8.0 Hz, 1H),

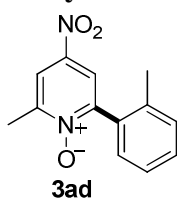
7.33 (t, J = 7.6 Hz, 1H), 7.25 (d, J = 7.6 Hz, 1H), 2.55 (s, 3H), 2.36 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 151.4, 150.6, 141.0, 138.3, 131.3, 131.2, 129.7, 128.4, 126.3, 118.9, 118.5, 21.4, 18.9.

MS (EI): m/z (%) 245 (M+1, 10), 244 (M, 70), 228 (40), 215 (70), 198 (45), 197 (100).

Anal. Calcd for $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_3$: C, 63.93; H, 4.95; N, 11.47; Found: C, 63.95; H, 4.58; N, 11.19.

2-Methyl-4-nitro-6-(*o*-tolyl)pyridine 1-oxide (3ad)



Pale yellow solid, m.p. 139-140 °C; R_f = 0.21 (petroleum ether/ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3075, 2958, 1607, 1528, 1336, 1279, 1103, 902, 738.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.08 (d, J = 3.1 Hz, 1H), 7.99 (d, J = 3.2 Hz, 1H), 7.36-7.32 (m, 1H), 7.27-7.22 (m, 2H),

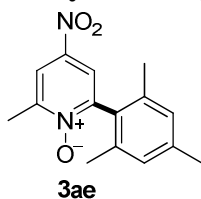
7.18-7.16 (m, 1H), 2.54 (s, 3H), 2.12 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 151.8, 151.0, 140.4, 137.7, 131.7, 130.3, 130.1, 129.3, 126.1, 119.5, 119.1, 19.5, 18.7.

MS (EI): m/z (%) 245 (M+1, 5), 244 (M, 25%), 227 (60), 196 (35), 181 (100).

Anal. Calcd for $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_3$: C, 63.93; H, 4.95; N, 11.47; Found: C, 64.11; H, 4.36; N, 11.17.

2-Mesityl-6-methyl-4-nitropyridine 1-oxide (3ae)



Pale yellow solid, m.p. 92-93 °C, R_f = 0.32 (petroleum ether/ethyl acetate = 5:1).

IR (cm⁻¹, KBr): 3089, 2958, 2916, 2850, 1609, 1523, 1330, 1275, 1103, 850, 712.

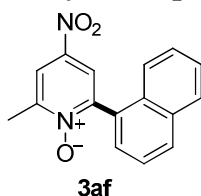
¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.08 (d, J = 3.2 Hz, 1H), 7.92 (d, J = 3.2 Hz, 1H), 6.91 (s, 2H), 2.54 (s, 3H), 2.27 (s, 3H), 1.96 (s, 6H).

¹³C NMR (100MHz, CDCl₃) δ (ppm): 150.02, 150.0, 139.3, 138.7, 135.4, 127.6, 127.5, 118.8, 117.9, 20.2, 18.5, 17.7.

MS (EI): m/z (%) 273 (M+1, 30), 272 (M, 45), 256 (80), 255 (60), 208 (100), 114 (35), 90 (30).

Anal. Calcd for C₁₅H₁₆N₂O₃: C, 66.16; H, 5.92; N, 10.29; Found: C, 65.91; H, 5.65; N, 10.02.

2-Methyl-6-(naphthalen-1-yl)-4-nitropyridine 1-oxide (3af)



Pale yellow solid, m.p. 223-225 °C, R_f = 0.29 (petroleum ether/ethyl acetate = 5:1).

IR (cm⁻¹, KBr): 3083, 2911, 1522, 1331, 1283, 1217, 1094, 891, 795.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.25-8.22 (dd, J = 2.9 and 11.2 Hz, 2H), 8.02 (d, J = 8.2 Hz, 1H), 7.95 (d, J = 8.1 Hz, 1H),

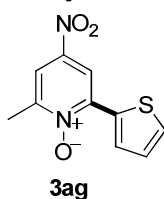
7.62-7.48 (m, 4H), 7.36 (d, J = 8.2 Hz, 1H), 2.66 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ (ppm): 151.3, 150.8, 140.6, 133.4, 130.9, 130.6, 129.8, 128.8, 127.9, 127.2, 126.5, 125.3, 124.6, 120.4, 119.4, 18.7.

MS (EI): m/z (%) 280 (M, 45), 279 (M-1, 40), 252 (50), 204 (40), 191 (35), 165 (100), 163 (75), 115 (50).

Anal. Calcd for C₁₆H₁₂N₂O₃: C, 68.56; H, 4.32; N, 9.99; Found: C, 68.27; H, 4.56; N, 9.82.

2-Methyl-4-nitro-6-(thiophen-2-yl)pyridine 1-oxide (3ag)



Pale yellow solid, m.p. 212-214 °C, R_f = 0.33 (petroleum ether/ethyl acetate = 5:1).

IR (cm⁻¹, KBr): 3078, 1621, 1524, 1414, 1376, 1338, 1270, 1102, 741.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.62 (d, J = 2.9 Hz, 1H), 7.94-7.92 (dd, J = 0.8 and 4.1 Hz, 1H), 7.90 (d, J = 2.8 Hz,

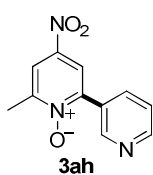
1H), 7.61-7.59 (dd, J = 0.7 and 5.0 Hz, 1H), 7.24-7.21 (m, 1H), 2.61 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ (ppm): 150.8, 143.9, 141.7, 131.9, 130.7, 128.8, 126.7, 115.5, 114.2, 18.7.

MS (EI): m/z (%) 237 (M+1, 20), 236 (M, 60), 219 (25), 148 (50), 132 (60), 110 (85), 68 (100), 66 (70).

Anal. Calcd for C₁₀H₈N₂O₃S: C, 50.84; H, 3.41; N, 11.86; Found: C, 50.59; H, 3.58; N, 11.61.

2-Methyl-4-nitro-6-(pyridin-3-yl)pyridine 1-oxide (3ah)



White solid, m.p. 190-191 °C, R_f = 0.16 (petroleum ether/ ethyl acetate = 2:1).

IR (cm^{-1} , KBr): 3063, 3013, 1589, 1527, 1491, 1343, 1282, 1032, 753;

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.97 (s, 1H), 8.76 (d, J = 3.2 Hz, 1H), 8.27 (d, J = 8.0 Hz, 1H), 8.23 (d, J = 3.0 Hz, 1H),

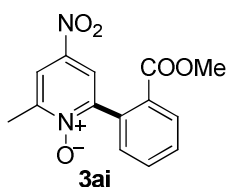
8.17 (d, J = 3.0 Hz, 1H), 7.51-7.47 (dd, J = 4.9 and 7.8 Hz, 1H), 2.64 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 151.7, 151.1, 149.5, 147.3, 141.0, 136.9, 127.8, 123.1, 119.4, 118.7, 18.8

MS (EI): m/z (%) 232 (M+1, 15), 231 (M, 25), 230 (M-1, 45), 116 (30), 103 (25), 89 (30), 74 (50), 63 (95), 51 (100).

Anal. Calcd for $\text{C}_{11}\text{H}_9\text{N}_3\text{O}_3$: C, 57.14; H, 3.92; N, 18.17; Found: C, 56.87; H, 3.97; N, 18.09.

2-(2-(Methoxycarbonyl)phenyl)-6-methyl-4-nitropyridine 1-oxide(3ai)



Pale yellow solid, m.p. 232-233 °C, R_f = 0.33 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3078, 2946, 1721, 1527, 1450, 1433, 1336, 1275, 1127, 1099, 952, 901, 734.

^1H NMR(400 MHz, CDCl_3) δ (ppm): 8.06 (d, J = 3.1 Hz, 1H), 8.02 (d, J = 3.0 Hz, 1H), 8.00 (d, J = 1.1 Hz, 1H), 7.61 (dd, J =

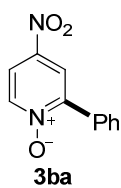
1.2 and 8.00 Hz, 1H), 7.53 (dd, J = 1.1 and 8.0 Hz, 1H), 7.31-7.29 (m, 1H), 3.70 (s, 3H), 2.50 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 166.4, 151.5, 150.4, 141.1, 132.7, 132.5, 130.9, 130.4, 130.2, 130.1, 119.1, 118.1, 52.4, 18.5.

MS (EI): m/z (%) 289 (M+1, 15), 288 (M, 5), 257 (25), 241 (15), 230 (35), 229 (75), 183 (100).

Anal. Calcd for $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_5$: C, 58.33; H, 4.20; N, 9.72.; Found: C, 58.10; H, 4.35; N, 9.55.

4-Nitro-2-phenylpyridine 1-oxide (3ba)



Pale yellow solid, m.p. 135-136 °C, R_f = 0.19 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3036, 1582, 1516, 1339, 1275, 1236, 1102, 915, 711.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.37 (d, J = 7.2 Hz, 1H), 8.30 (d, J = 3.2 Hz, 1H), 8.05-8.03 (dd, J = 3.2 and 7.2 Hz, 1H), 7.92-

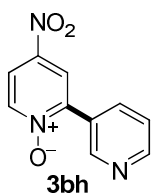
7.80 (m, 2H), 7.54-7.52 (m, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 150.4, 142.1, 141.4, 130.8, 130.6, 129.1, 128.7, 121.6, 118.5.

MS (EI): m/z (%) 217 (M+1, 10); 216 (M, 40%), 215 (M-1, 50), 199 (55), 168 (60), 154 (65), 127 (100), 115 (80).

Anal. Calcd for $\text{C}_{11}\text{H}_8\text{N}_2\text{O}_3$: C, 61.11; H, 3.73; N, 12.96; Found: C, 60.99; H, 3.56; N, 12.81.

4-Nitro-2-(pyridin-3-yl)pyridine 1-oxide (3bh)



White solid, m.p. 204-206 °C, R_f = 0.15 (petroleum ether/ ethyl acetate = 2:1).

IR (cm^{-1} , KBr): 3039, 1578, 1538, 1519, 1348, 1283, 1253, 1027, 750, 703.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.92 (s, 1H), 8.70 (d, J = 3.8 Hz, 1H), 8.35 (d, J = 7.2 Hz, 1H), 8.28 (d, J = 2.8 Hz, 1H), 8.23 (d,

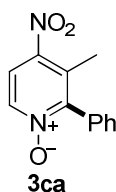
J = 7.9 Hz, 1H), 8.07-8.04 (dd, J = 2.9 and 7.0 Hz, 1H), 7.45-7.42 (dd, J = 4.9 and 7.6 Hz, 1H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 151.4, 149.3, 147.5, 142.1, 141.4, 136.8, 127.0, 123.2, 121.4, 191.4.

MS (EI): m/z (%) 218 (M+1, 10), 217 (M, 15%), 216 (M-1, 20), 116 (30), 87 (50), 76 (55), 74 (75), 63 (100), 62 (85).

Anal. Calcd for $\text{C}_{10}\text{H}_7\text{N}_3\text{O}_3$: C, 55.30; H, 3.25; N, 19.35; Found: C, 55.03; H, 3.35; N, 19.42.

3-Methyl-4-nitro-2-phenylpyridine 1-oxide (3ca)



Pale yellow solid, m.p. 169-170 °C, R_f = 0.26 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3082, 1589, 1514, 1343, 1295, 1249, 1073, 827, 722, 698.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.30 (d, J = 7.2 Hz, 1H), 7.93 (d, J = 7.2 Hz, 1H), 7.59-7.51 (m, 3H), 7.33-7.30 (m, 2H),

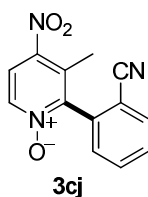
2.34 (s, 3H).

^{13}C NMR (100MHz, CDCl_3) δ (ppm): 152.2, 144.3, 138.1, 131.5, 131.0, 129.8, 129.2, 129.1, 120.1, 17.6.

MS (EI): m/z (%) 230 (M, 30%), 229 (M-1, 85), 213 (25), 184 (75), 183 (100), 167 (65), 154 (90).

Anal. Calcd for $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3$: C, 62.60; H, 4.38; N, 12.17; Found: C, 62.34; H, 4.59; N, 11.89.

2-(2-Cyanophenyl)-3-methyl-4-nitropyridine 1-oxide (3cj)



Pale yellow solid, m.p. 178-179 °C, R_f = 0.51 (ethyl acetate).

IR (cm^{-1} , KBr): 3066, 2985, 2228, 1587, 1571, 1519, 1463, 1431, 1346, 1294, 1280, 1246, 1070, 1049, 853.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.26 (d, J = 7.2 Hz, 1H), 8.00 (d, J = 7.2 Hz, 1H), 7.83 (d, J = 7.8 Hz, 1H), 7.77-7.73 (dd,

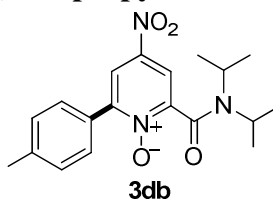
J = 7.7 and 1.1 Hz, 1H), 7.62-7.58 (dd, J = 7.7 and 0.7 Hz, 1H), 7.37 (d, J = 7.7 Hz, 1H), 2.31 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 148.6, 143.9, 138.4, 134.8, 133.7, 132.2, 130.5, 130.4, 121.6, 116.4, 113.4, 17.3.

MS(EI): m/z (%) 255 (M, 75%), 254 (M-1, 75), 229 (70), 207 (80), 242 (65), 180 (75), 139 (85), 63 (100).

Anal. Calcd for $\text{C}_{13}\text{H}_9\text{N}_3\text{O}_3$: C, 61.18; H, 3.55; N, 16.46. Found: C, 61.00; H, 3.33; N, 16.43.

2-(Diisopropylcarbamoyl)-4-nitro-6-p-tolylpyridine 1-oxide (3db)



Pale yellow solid, m.p. 180-183 °C, R_f = 0.53 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3094, 2973, 2927, 1647, 1521, 1509, 1474, 1331, 1276, 1201, 1113, 819.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.17 (d, J = 3.2 Hz, 1H), 7.92 (d, J = 3.3 Hz, 1H), 7.67 (d, J = 8.2 Hz, 2H),

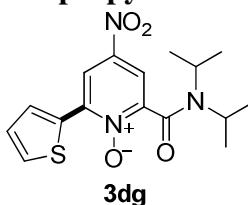
7.25 (d, J = 8.1 Hz, 2H), 3.55-3.48 (m, 1H), 3.45-3.39 (m, 1H), 2.36 (s, 3H), 1.52 (d, J = 6.8 Hz, 3H), 1.50 (d, J = 6.8 Hz, 3H), 1.24 (d, J = 6.6 Hz, 3H), 1.10 (d, J = 6.6 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 160.2, 150.6, 148.4, 141.7, 141.4, 129.3, 129.1, 127.6, 120.2, 116.0, 51.5, 46.5, 21.5, 21.1, 21.0, 20.3, 19.8.

MS (EI): m/z (%) 358 (M+1, 5%), 340 (10), 183 (15), 119 (45), 100 (60), 69 (65), 57 (75), 55 (100).

Anal. Calcd for $\text{C}_{19}\text{H}_{23}\text{N}_3\text{O}_4$: C, 63.85; H, 6.49; N, 11.76; Found: C, 63.75; H, 6.58; N, 11.49.

2-(Diisopropylcarbamoyl)-4-nitro-6-(thiophen-2-yl)pyridine 1-oxide (3dg)



Pale yellow solid, m.p. 235-239 °C, R_f = 0.53 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3098, 2977, 2938, 1642, 1530, 1443, 1331, 1281, 1201, 883, 738.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.64 (d, J = 3.0 Hz, 1H), 7.95-7.93 (dd, J = 4.00 and 0.7 Hz, 1H), 7.82 (d, J =

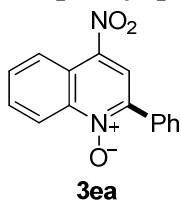
3.0 Hz, 1H), 7.60 (d, J = 5.0 Hz, 1H), 7.22-7.20 (m, 1H), 3.59-3.49 (m, 1H), 3.43-3.34 (m, 1H), 1.57 (d, J = 6.8 Hz, 3H), 1.50 (d, J = 6.8 Hz, 3H), 1.27 (d, J = 6.6 Hz, 3H), 1.08 (d, J = 6.6 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 159.9, 147.8, 144.2, 142.2, 132.2, 130.0, 129.1, 126.7, 115.8, 113.4, 51.6, 46.6, 21.0, 20.9, 20.3, 19.9.

MS (EI): m/z (%) 350 (M+1, 5%), 332 (10), 158 (20), 111 (100), 69 (25).

Anal. Calcd for $\text{C}_{16}\text{H}_{19}\text{N}_3\text{O}_4\text{S}$: C, 55.00; H, 5.48; N, 12.03; Found: C, 54.92; H, 5.56; N, 11.98.

4-Nitro-2-phenylquinoline 1-oxide (3ea)⁶



Pale yellow solid, m.p. 137-138 °C (lit.⁶ 135-137 °C); R_f = 0.32 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3077, 1521, 1503, 1492, 1298, 766, 740, 687.

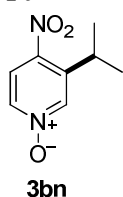
^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.90-8.84 (m, 2H), 8.46 (s, 1H), 7.98-7.96 (m, 2H), 7.94-7.86 (m, 2H),

7.61-7.53 (m, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 144.1, 143.8, 139.6, 131.6, 130.5, 129.3, 128.7, 124.5, 121.4, 121.1, 120.8.

MS (EI): m/z (%) 266 (M, 80), 265 (M-1, 70), 219 (85), 191 (100), 165 (55), 75 (83), 51 (65).

3-Isopropyl-4-nitropyridine 1-oxide (3bn)⁷



Pale yellow solid, m.p. 136-139 °C (lit.⁷ 138-139 °C); R_f = 0.12 (petroleum ether/ ethyl acetate = 1:1).

IR (cm⁻¹, KBr): 3118, 2948, 1602, 1567, 1518, 1338, 1290, 1243, 1071, 648.

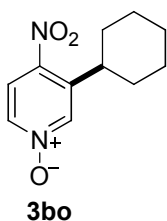
¹H NMR (500 MHz, CDCl₃) δ (ppm): 8.18 (d, J = 1.8 Hz, 1H), 8.03-8.00 (dd, J = 7.0 and 1.9 Hz, 1H), 7.78 (d, J =

7.1 Hz, 1H), 3.66-3.59 (m, 1H), 1.26 (d, J = 6.8 Hz, 1H).

¹³C NMR (125 MHz, CDCl₃) δ (ppm): 143.2, 142.3, 139.3, 137.4, 121.9, 27.7, 22.7.

MS (EI): m/z (%) 183 (M+1, 35), 182 (M, 100), 148 (40), 85 (65), 71 (70), 57 (75).

3-Cyclohexyl-4-nitropyridine 1-oxide (3bo)



Pale yellow solid, m.p. 123-126 °C, R_f = 0.13 (petroleum ether/ ethyl acetate = 1:1).

IR (cm⁻¹, KBr): 2925, 2852, 1604, 1564, 1516, 1448, 1333, 1301, 1253, 1229, 1079, 799.

¹H NMR (500 MHz, CDCl₃) δ (ppm): 8.27(d, J = 1.5 Hz, 1H), 8.12-8.10 (dd, J = 7.1 and 1.4 Hz, 1H), 7.85 (d, J = 7.1 Hz, 1H), 3.30-3.29 (m, 1H), 1.98-1.80 (m, 5H),

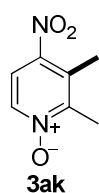
1.48-1.27 (m, 5H).

¹³C NMR (125 MHz, CDCl₃) δ (ppm): 143.4, 141.1, 139.7, 137.3, 121.9, 37.7, 33.3, 26.4, 25.7.

MS (EI): m/z (%) 223 (M+1, 30), 222 (M, 100), 205 (75), 188 (50), 65 (55), 63 (70), 53 (80).

Anal. Calcd for C₁₁H₁₄N₂O₃: C, 59.45; H, 6.35; N, 12.60; Found: C, 59.30; H, 6.25; N, 12.46.

2,3-Dimethyl-4-nitropyridine 1-oxide (3ak)⁸

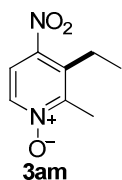


Pale yellow solid, m.p. 88-90 °C (lit.⁸ 91-93 °C), R_f = 0.11 (petroleum ether/ ethyl acetate = 1:1).

IR (cm⁻¹, KBr): 3101, 2925, 2850, 1521, 1458, 1344, 1282, 1213, 1089.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.14 (d, J = 7.1 Hz, 1H), 7.64 (d, J = 7.1 Hz, 1H), 2.52 (s, 3H), 2.50 (s, 3H).

3-Ethyl-2-methyl-4-nitropyridine 1-oxide (3am)



Pale yellow solid, m.p. 88-90 °C, R_f = 0.13 (petroleum ether/ ethyl acetate = 1:1).

IR (cm⁻¹, KBr): 3073, 2969, 1593, 1571, 1521, 1479, 1343, 1281, 1045, 698.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.14 (d, J = 7.1 Hz, 1H), 7.62 (d, J = 7.1 Hz, 1H), 2.86-2.81 (dd, J = 7.0 and

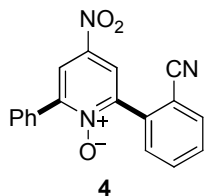
1.9 Hz, 2H), 2.51 (s, 3H), 1.25 (t, J = 7.2 Hz, 3H).

¹³C NMR (125 MHz, CDCl₃) δ (ppm): 150.9, 144.2, 137.5, 135.3, 118.5, 23.00, 14.1, 13.5.

MS (EI): m/z (%) 183 (M+1, 15), 182 (M, 20), 121 (30), 118 (85), 116 (65), 92 (90), 50 (100).

Anal. Calcd for $C_8H_{10}N_2O_3$: C, 52.74; H, 5.53; N, 15.38; Found: C, 52.67; H, 5.26; N, 15.42.

2-(2-Cyanophenyl)-4-nitro-6-phenylpyridine 1-oxide (4) (Scheme 2)



Pale yellow solid, m.p. 138-141 °C, R_f = 0.23 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3089, 2971, 2230, 1523, 1498, 1333, 1293, 1268, 1112, 898, 777, 718.

1H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.32 (d, J = 3.2 Hz, 1H), 8.18 (d, J = 3.2 Hz, 1H), 7.81-7.69 (m, 4H), 7.61-7.56

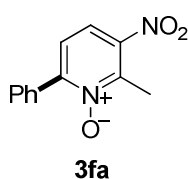
(m, 2H), 7.47-7.46 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 151.4, 148.5, 141.1, 135.1, 133.2, 133.1, 130.9, 130.8, 130.6, 130.5, 129.3, 128.6, 121.3, 120.3, 117.0, 113.4.

MS (EI): m/z (%) 317 (M, 45), 316 (M-1, 100), 300 (60), 270 (80), 242 (85), 241 (25), 102 (45).

Anal. Calcd for $C_{18}H_{11}N_3O_3$: C, 68.14; H, 3.49; N, 13.24; Found: C, 68.21; H, 3.56; N, 13.22.

2-Methyl-3-nitro-6-phenylpyridine 1-oxide (3fa)



Pale yellow solid, m.p. 148-150 °C, R_f = 0.55 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3051, 3014, 1531, 1355, 1256, 1164, 1048, 813.

1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.81-7.79 (m, 2H), 7.77 (d, J = 8.9 Hz, 1H), 7.52-7.51 (m, 3H), 7.46 (d, J = 8.8

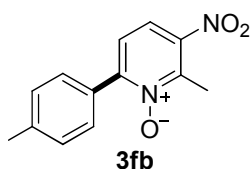
Hz, 1H), 2.80 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 152.7, 147.5, 147.2, 131.8, 130.5, 129.3, 128.4, 123.4, 119.3, 14.7.

MS (EI): m/z (%) 231 (M+1, 15), 230 (M, 90), 229 (M-1, 100), 214 (35), 201 (40), 183 (45), 154 (35), 77 (15).

Anal. Calcd for $C_{12}H_{10}N_2O_3$: C, 62.60; H, 4.38; N, 12.17. Found: C, 62.65; H, 4.11; N, 11.96.

2-Methyl-3-nitro-6-p-tolylpyridine 1-oxide (3fb)



Pale yellow solid, m.p. 152-153 °C, R_f = 0.62 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3056, 3025, 1610, 1524, 1350, 1253, 808, 749.

1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.75-7.72 (m, 3H), 7.44 (d, J = 8.8 Hz, 1H), 7.31 (d, J = 8.0 Hz, 2H), 2.79 (s,

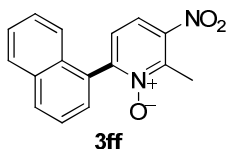
3H), 2.43 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 152.8, 147.2, 140.9, 129.3, 129.2, 129.1, 128.9, 123.2, 119.3, 21.5, 14.8.

MS (EI): m/z (%) 245 (M+1, 15), 244 (M, 50), 243 (M-1, 65), 197 (60), 151 (85), 115 (95), 91 (60), 63 (100).

Anal. Calcd for C₁₃H₁₂N₂O₃: C, 63.93; H, 4.95; N, 11.47; Found: C, 63.75; H, 5.12; N, 11.19.

2-Methyl-6-(naphthalen-1-yl)-3-nitropyridine 1-oxide (3ff)



Pale yellow solid, m.p. 125-127 °C; R_f = 0.65 (petroleum ether/ ethyl acetate = 1:3).

IR (cm⁻¹, KBr): 3044, 2911, 2844, 1557, 1524, 1349, 1043, 821, 772.

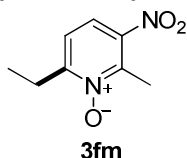
¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.94 (d, *J* = 8.2 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.74 (d, *J* = 8.6 Hz, 1H), 7.53-7.40 (m, 5H), 7.30 (d, *J* = 8.2 Hz, 1H), 2.75 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ (ppm): 153.4, 148.2, 147.1, 133.4, 130.4, 130.2, 128.8, 127.8, 127.2, 126.5, 125.3, 124.9, 124.8, 118.9, 14.7.

MS (EI): *m/z* (%) 280 (M, 10), 252 (15), 204 (35), 163 (50), 152 (85), 51 (100).

Anal. Calcd for C₁₆H₁₂N₂O₃: C, 68.56; H, 4.32; N, 9.99; Found: C, 68.76; H, 4.57; N, 9.79.

6-Ethyl-2-methyl-3-nitropyridine 1-oxide (3fm)



Pale yellow solid, m.p. 58-59 °C, R_f = 0.32 (petroleum ether/ ethyl acetate = 2:1).

IR (cm⁻¹, KBr): 2965, 2927, 1531, 1352, 1264, 1047, 817.

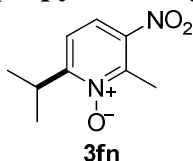
¹H NMR (400MHz, CDCl₃) δ (ppm): 7.64 (d, *J* = 8.7 Hz, 1H), 7.21 (d, *J* = 8.6 Hz, 1H), 2.93 (q, *J* = 7.3 Hz, 2H), 2.70 (s, 3H), 1.28 (t, *J* = 7.4 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) δ (ppm): 158.1, 146.8, 146.2, 120.4, 119.3, 24.8, 14.6, 10.2.

MS (EI): *m/z* (%) 183 (M+1, 15), 182 (M, 65), 165 (40), 119 (100), 117 (35).

Anal. Calcd for C₈H₁₀N₂O₃: C, 52.74; H, 5.53; N, 15.38. Found: C, 52.62; H, 5.26; N, 15.47.

6-Isopropyl-2-methyl-3-nitropyridine 1-oxide (3fn)



Yellow liquid, R_f = 0.33 (petroleum ether/ ethyl acetate = 2:1).

IR (cm⁻¹, KBr): 3074, 2968, 2875, 1562, 1532, 1357, 1260, 1047, 814.

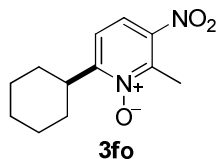
¹H NMR (400MHz, CDCl₃) δ (ppm): 7.63 (d, *J* = 8.8 Hz, 1H), 7.21 (d, *J* = 8.8 Hz, 1H), 3.75-3.68 (m, 1H), 2.68 (s, 3H), 1.25 (d, *J* = 7.0 Hz, 6H).

¹³C NMR (100MHz, CDCl₃) δ (ppm): 162.0, 146.7, 146.2, 119.2, 119.0, 28.7, 19.9, 14.6.

MS (EI): *m/z* (%) 196 (M, 5), 149 (15), 133 (55), 91 (65), 77 (85), 57 (80), 55 (100).

Anal. Calcd for C₉H₁₂N₂O₃: C, 55.09; H, 6.16; N, 14.28; Found: C, 55.49; H, 6.56; N, 13.85.

6-Cyclohexyl-2-methyl-3-nitropyridine 1-oxide (3fo)



Pale yellow solid, m.p. 80-83 °C, R_f = 0.31 (petroleum ether/ ethyl acetate = 2:1).

IR (cm^{-1} , KBr): 2928, 2853, 1532, 1449, 1350, 1278, 1252, 1047, 820.

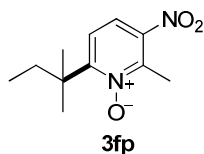
^1H NMR (400MHz, CDCl_3) δ (ppm): 7.68 (d, J = 8.8 Hz, 1H), 7.23 (d, J = 8.8 Hz, 1H), 3.56-3.50 (m, 1H), 2.75 (s, 3H), 2.06 (d, J = 12.5 Hz, 2H), 1.90-1.81 (m, 3H), 1.58-1.47 (m, 2H), 11.34-1.24 (m, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 161.0, 146.6, 146.2, 119.6, 119.4, 38.4, 30.6, 26.2, 26.1, 14.8.

MS (EI): m/z (%) 237 (M+1, 90), 236 (M, 65), 219 (100), 181 (45), 173 (85), 144 (30).

Anal. Calcd for $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_3$: C, 61.00; H, 6.83; N, 11.86; Found: C 61.25; H, 7.02; N, 11.57.

2-Methyl-3-nitro-6-tert-pentylpyridine 1-oxide (3fp)



Pale yellow liquid, R_f = 0.33 (petroleum ether/ ethyl acetate = 2:1).

IR (cm^{-1} , KBr): 2963, 2922, 2877, 1531, 1349, 1266, 1234, 1947, 822.

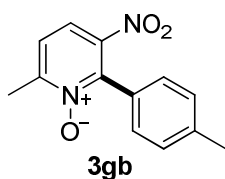
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.55 (d, J = 9.1 Hz, 1H), 7.26 (d, J = 9.1 Hz, 1H), 2.63 (s, 3H), 2.05 (q, J = 7.6 Hz, 2H), 1.40 (s, 6H), 0.57 (t, J = 7.5 Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 161.2, 147.2, 147.0, 121.4, 118.4, 40.9, 29.9, 25.5, 14.7, 14.6, 9.6.

MS (ESI): m/z (%) 225 (M+1, 100).

Anal. Calcd for $\text{C}_{11}\text{H}_{16}\text{N}_2\text{O}_3$: C, 58.91; H, 7.19; N, 12.49; Found: C, 59.12; H, 7.54; N, 12.96.

6-Methyl-3-nitro-2-p-tolylpyridine 1-oxide (3gb)



Pale yellow solid, m.p. 106-108 °C, R_f = 0.59 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3074, 3014, 2918, 1533, 1475, 1374, 1345, 1271, 1004, 815, 539.

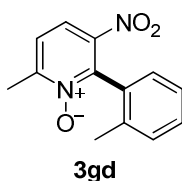
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.53 (d, J = 8.5 Hz, 1H), 7.31 (d, J = 8.6 Hz, 1H), 7.26-7.22 (m, 4H), 2.52 (s, 3H), 2.34 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 153.7, 147.5, 145.5, 140.4, 129.5, 128.8, 125.4, 124.1, 118.5, 21.5, 18.7.

MS (EI): m/z (%) 245 (M+1, 35), 244 (M, 55), 243 (M-1, 100), 199 (40), 115 (50), 77 (35).

Anal. Calcd for $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_3$: C, 63.93; H, 4.95; N, 11.47; Found: C, 63.86; H, 4.87; N, 11.58.

6-Methyl-3-nitro-2-o-tolylpyridine 1-oxide (3gd)



Yellow solid, m.p. 125-128 °C, R_f = 0.61 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3389, 2923, 1533, 1471, 1347, 1270, 817.

^1H NMR (400MHz, CDCl_3) δ (ppm): 7.59 (d, J = 8.6 Hz, 1H), 7.36 (d, J = 8.6 Hz, 1H), 7.31 (d, J = 7.3 Hz, 1H), 7.28 (t, J = 7.3 Hz, 1H), 7.19 (t, J = 7.6 Hz, 1H), 7.01 (d, J =

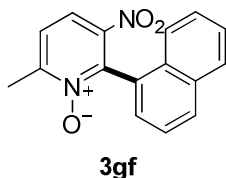
7.5 Hz, 1H), 2.53 (s, 3H), 2.12 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 153.9, 147.4, 146.3, 137.9, 130.2, 130.1, 128.8, 127.9, 126.2, 124.4, 118.3, 19.2, 18.6.

MS (EI): m/z (%) 245 (M+1, 30), 227 (75), 213 (50), 197 (45), 181 (100), 115 (80).

Anal. Calcd for $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_3$: C, 63.93; H, 4.95; N, 11.47; Found: C, 63.74; H, 4.66; N, 11.25.

6-Methyl-2-(naphthalen-1-yl)-3-nitropyridine 1-oxide (3gf)



Pale yellow solid, m.p. 208-210 °C, R_f = 0.63 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3074, 3000, 1557, 1530, 1347, 1269, 1037, 818.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.92 (d, J = 8.3 Hz, 1H), 7.85 (d, J = 8.0 Hz, 1H), 7.66 (d, J = 8.6 Hz, 1H),

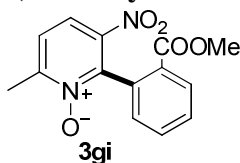
7.49-7.37 (m, 4H), 7.32-7.30 (dd, J = 0.7 and 7.1 Hz, 1H), 7.26 (d, J = 8.2 Hz, 1H), 2.53 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 151.7, 145.4, 142.8, 130.9, 128.4, 126.3, 124.7, 124.5, 123.9, 122.8, 122.2, 121.5, 116.0, 16.1.

MS (EI): m/z (%) 280 (M, 5), 234 (15), 175 (25), 163 (65), 115 (60), 74 (65), 63 (95), 51 (100).

Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_3$: C, 68.56; H, 4.32; N, 9.99; Found: C, 68.28; H, 4.06; N, 9.76.

2-(2-(Methoxycarbonyl)phenyl)-6-methyl-3-nitropyridine 1-oxide (3gi)



Pale yellow solid, m.p. 175-177 °C, R_f = 0.62 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3066, 3000, 2948, 1724, 1528, 1450, 1349, 1292, 1270, 1127, 1077, 814;

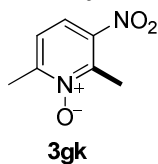
^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.15 (d, J = 7.8 Hz, 1H), 7.81 (d, J = 8.6 Hz, 1H), 7.61 (t, J = 7.5 Hz, 1H), 7.52 (t, J = 7.6 Hz, 1H), 7.38 (d, J = 8.7 Hz, 1H), 7.16 (d, J = 7.5 Hz, 1H), 3.70 (s, 3H), 2.52 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 165.9, 154.1, 147.3, 145.4, 133.3, 131.6, 130.9, 129.8, 129.5, 128.9, 124.0, 119.6, 52.3; 18.6.

MS (EI): m/z (%) 289 (M+1, 2), 185 (35), 142 (50), 130 (65), 75 (60), 63 (85), 51 (100).

Anal. Calcd for $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_5$: C, 58.33; H, 4.20; N, 9.72; Found: C, 58.04; H, 4.44; N, 9.98.

2,6-dimethyl-3-nitropyridine 1-oxide (3gk)⁹

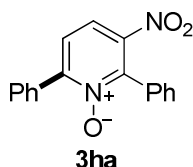


Pale yellow solid, m.p. 99-101 °C (lit.⁹ 101-103 °C), R_f = 0.29 (petroleum ether/ ethyl acetate = 2:1).

IR (cm⁻¹, KBr): 3079, 2922, 1562, 1532, 1447, 1353, 1257, 1054, 813.

¹H NMR(400 MHz, CDCl₃) δ (ppm): 7.67(d, J = 8.6 Hz, 1H), 7.31 (d, J = 8.6 Hz, 1H), 2.78 (s, 3H), 2.60 (s, 3H).

3-Nitro-2,6-diphenylpyridine 1-oxide (3ha)



Yellow solid, m.p. 230-231 °C, R_f = 0.52 (petroleum ether/ ethyl acetate = 5:1).

IR (cm⁻¹, KBr): 3059, 1531, 1346, 1306, 1259, 1017, 760, 700.

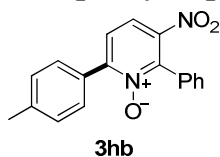
¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.79-7.77 (m, 2H), 7.67 (d, J = 8.7Hz, 1H), 7.52 (d, J = 8.7 Hz, 1H), 7.45-7.41 (m, 8H);

¹³C NMR (100 MHz, CDCl₃) δ (ppm): 153.1, 147.7, 146.2, 131.4, 130.6, 130.3, 129.5, 129.0, 128.8, 128.4, 128.3, 125.4, 119.2.

MS (EI): m/z (%) 293 (M+1, 20), 292 (M, 65), 291 (M-1, 90), 246 (55), 245 (90), 115 (90), 101 (100), 81 (90).

Anal. Calcd for C₁₇H₁₂N₂O₃: C, 69.86; H, 4.14; N, 9.58; Found: C, 69.75; H, 4.10; N, 9.65.

3-Nitro-2-phenyl-6-p-tolylpyridine 1-oxide (3hb)



Pale yellow solid, m.p. 225-230 °C, R_f = 0.55 (petroleum ether/ ethyl acetate = 5:1).

IR (cm⁻¹, KBr): 3074, 2925, 1528, 1357, 1280, 1263, 811, 703.

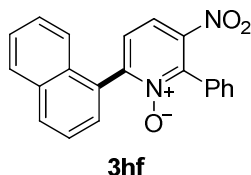
¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.71 (d, J = 8.2 Hz, 2H), 7.66 (d, J = 8.7 Hz, 1H), 7.51 (d, J = 8.8 Hz, 1H), 7.44-7.40 (m, 5H), 7.22 (d, J = 8.1 Hz, 2H), 2.34 (s, 3H).

¹³C NMR (100 MHz, CDCl₃) δ (ppm): 153.1, 147.5, 146.2, 141.0, 130.1, 129.4, 128.7, 128.6, 125.1, 118.9, 118.8, 21.5.

MS (EI): m/z (%) 307 (M+1, 10), 306 (M, 30), 305 (M-1, 35), 259 (40), 109 (50), 97 (75), 95 (90), 55 (100).

Anal. Calcd for C₁₈H₁₄N₂O₃: C, 70.58; H, 4.61; N, 9.15; Found: C, 70.35; H, 4.71; N, 8.97.

6-(Naphthalen-1-yl)-3-nitro-2-phenylpyridine 1-oxide (3hf)



Pale yellow solid, m.p. 177-178 °C, R_f = 0.59 (petroleum ether/ ethyl acetate = 5:1).

IR (cm⁻¹, KBr): 3060, 1537, 1353, 1259, 989, 818, 779, 699.

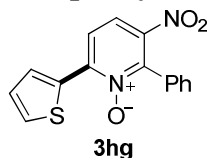
¹H NMR (400 MHz, CDCl₃) δ (ppm): 8.02-7.99 (m, 1H), 7.94-7.91 (m, 1H), 7.75 (d, J = 8.6 Hz, 1H), 7.60-7.48 (m, 11H).

¹³C NMR (100 MHz, CDCl₃) δ (ppm): 153.7, 148.5, 145.9, 133.4, 130.8, 130.5, 129.9, 129.2, 128.7, 128.1, 127.9, 127.2, 126.8, 126.4, 125.2, 124.8, 118.4.

MS (EI): m/z (%) 342 (M, 5), 190 (15), 152 (25), 77 (40), 55 (45), 49 (100).

Anal. Calcd for $C_{21}H_{14}N_2O_3$: C, 73.68; H, 4.12; N, 8.18; Found: C, 73.79; H, 4.40; N, 8.25.

3-Nitro-2-phenyl-6-(thiophen-2-yl)pyridine 1-oxide (3hg)



Yellow solid, m.p. 206-211 °C, R_f = 0.61 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3100, 3064, 1557, 1523, 1343, 1267, 1240, 1066, 823.

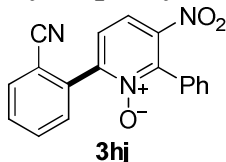
1H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.10 (d, J = 9.2 Hz, 1H), 8.03-8.01 (dd, J = 0.6 and 4.1 Hz, 1H), 7.85 (d, J = 9.2 Hz, 1H), 7.69-7.67 (m, 1H), 7.57-7.54 (m, 3H), 7.49-7.46 (m, 2H), 7.30 (t, J = 4.3 Hz, 1H);

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 146.6, 146.1, 144.9, 132.9, 130.8, 130.3, 129.5, 128.9, 128.8, 128.5, 126.7, 120.2, 119.8.

MS (EI): m/z (%) 299 (M+1, 5), 298 (M, 10), 297 (M-1, 10), 221 (25), 126 (40), 121 (75), 77 (80), 69 (90), 63 (100).

Anal. Calcd for $C_{15}H_{10}N_2O_3S$: C, 60.39; H, 3.38; N, 9.39. Found: C, 60.54; H, 3.15; N, 9.54.

6-(2-Cyanophenyl)-3-nitro-2-phenylpyridine 1-oxide (3hj)



Pale yellow solid, m.p. 68-77 °C, R_f = 0.63 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3067, 2959, 2227, 1532, 1443, 1352, 1313, 1267, 1013, 831, 757.

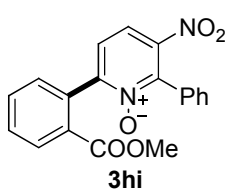
1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.76 (d, J = 7.7 Hz, 1H), 7.70 (d, J = 8.6 Hz, 1H), 7.66 (d, J = 4.0 Hz, 2H), 7.55-7.52 (m, 2H), 7.41-7.45 (m, 5H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 149.9, 148.9, 146.4, 135.1, 133.2, 132.7, 130.7, 130.4, 129.0, 128.9, 127.7, 126.1, 118.7, 117.1, 113.1.

MS (EI): m/z (%) 318 (M+1, 3), 317 (M, 5), 316 (M-1, 10), 272 (30), 242(40), 140 (55), 105 (70), 80 (95), 77 (100).

Anal. Calcd for $C_{18}H_{11}N_3O_3$: C, 68.14; H, 3.49; N, 13.24; Found: C, 68.34; H, 3.68; N, 13.50.

6-(2-(Methoxycarbonyl)phenyl)-3-nitro-2-phenylpyridine 1-oxide (3hi)



Pale yellow solid, m.p. 190-195 °C, R_f = 0.59 (petroleum ether/ ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3089, 3007, 2948, 1709, 1530, 1449, 1354, 1302, 1287, 1129, 823.

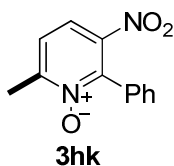
1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.97 (d, J = 7.7 Hz, 1H), 7.70 (d, J = 8.6 Hz, 1H), 7.59 (t, J = 7.5 Hz, 1H), 7.49 (t, J = 7.6 Hz, 1H), 7.43-7.35 (m, 6H), 7.31 (d, J = 7.6 Hz, 1H), 3.72 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 166.4, 154.6, 148.0, 145.4, 132.7, 132.5, 131.0, 130.3, 130.2, 130.1, 129.9, 129.0, 128.4, 124.2, 119.2, 52.5.

MS (EI): m/z (%) 351 (M+1, 2), 291 (10), 245 (30), 126 (50), 102 (65), 77 (95), 76 (100).

Anal. Calcd for C₁₉H₁₄N₂O₅: C, 65.14; H, 4.03; N, 8.00; Found: C, 65.35; H, 4.31; N, 7.78.

6-Methyl-3-nitro-2-phenylpyridine 1-oxide (3hk)



Yellow solid, m.p. 135-137 °C, R_f = 0.58 (petroleum ether/ethyl acetate = 1:3).

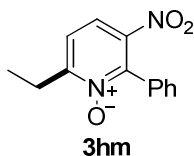
IR (cm⁻¹, KBr): 3060, 2923, 2856, 1562, 1530, 1363, 1349, 1264, 1201, 812, 699.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.61 (d, *J* = 8.6 Hz, 1H), 7.50-7.49 (m, 3H), 7.43-7.39 (m, 3H), 2.58 (s, 3H);
¹³C NMR (100 MHz, CDCl₃) δ (ppm): 153.9, 147.5, 145.4, 130.2, 129.0, 128.8, 128.6, 124.4, 118.5, 18.7.

MS (EI): *m/z* (%) 231 (M+1, 55), 230 (M, 40), 229 (M-1, 55), 185 (45), 115 (65), 81 (85), 77 (75), 53 (100).

Anal. Calcd for C₁₂H₁₀N₂O₃: C, 62.60; H, 4.38; N, 12.17; Found: C, 62.43; H, 4.09; N, 12.34.

6-Ethyl-3-nitro-2-phenylpyridine 1-oxide (3hm)



Yellow solid, m.p. 97-99 °C, R_f = 0.62 (petroleum ether/ethyl acetate = 1:3).

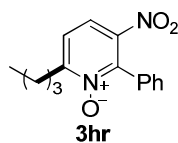
IR (cm⁻¹, KBr): 3072, 2936, 2868, 1531, 1475, 1451, 1358, 1282, 1240, 1009, 812.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.60 (d, *J* = 8.6 Hz, 1H), 7.44-7.41 (m, 3H), 7.36-7.31 (m, 3H), 2.93 (q, *J* = 7.4 Hz, 2H), 1.30 (t, *J* = 7.4 Hz, 3H).
¹³C NMR (100 MHz, CDCl₃) δ (ppm): 158.5, 147.2, 145.3, 130.1, 128.9, 128.8, 128.6, 122.3, 118.6, 24.6, 10.2.

MS (EI): *m/z* (%) 244 (M, 3), 184 (15), 126 (35), 115 (55), 77 (75), 63 (70), 51 (100).

Anal. Calcd for C₁₃H₁₂N₂O₃: C, 63.93; H, 4.95; N, 11.47; Found: C, 63.65; H, 4.71; N, 11.76.

6-Butyl-3-nitro-2-phenylpyridine 1-oxide (3hr)



Pale yellow solid, m.p. 105-107 °C, R_f = 0.63 (petroleum ether/ethyl acetate = 1:3).

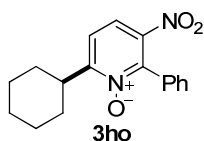
IR (cm⁻¹, KBr): 3059, 2956, 2927, 2873, 1535, 1355, 1266, 816, 766, 699.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.58 (d, *J* = 8.6 Hz, 1H), 7.42 (m, 3H), 7.36 (m, 2H), 7.31 (d, *J* = 8.6 Hz, 1H), 2.90 (t, *J* = 7.7 Hz, 2H), 1.71-1.63 (m, 2H), 1.45-1.37 (m, 2H), 0.91 (t, *J* = 7.2 Hz, 3H);
¹³C NMR (100 MHz, CDCl₃) δ (ppm): 157.5, 147.1, 145.4, 130.1, 128.9, 128.8, 128.6, 123.3, 118.7, 31.2, 27.8, 22.6, 13.8.

MS (EI): *m/z* (%) 272 (M, 10), 243 (20), 230 (100).

Anal. Calcd for C₁₅H₁₆N₂O₃: C, 66.16; H, 5.92; N, 10.29; Found: C, 66.25; H, 5.63; N, 10.03.

6-Cyclohexyl-3-nitro-2-phenylpyridine 1-oxide (3ho)



Pale yellow solid, m.p. 128-130 °C, R_f = 0.59 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3062, 2926, 2852, 1536, 1447, 1352, 1278, 1253, 1166, 982, 818, 695.

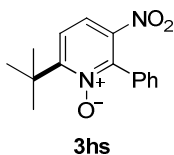
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.68 (d, J = 8.7 Hz, 1H), 7.50 (m, 3H), 7.43 (m, 2H), 7.36 (d, J = 8.7 Hz, 1H), 3.49 (t, J = 11.3 Hz, 1H), 2.11 (d, J = 11.8 Hz, 2H), 1.90 (d, J = 12.9 Hz, 2H), 1.82 (d, J = 12.9 Hz, 1H), 1.53-1.44 (m, 2H), 1.36-1.28 (m, 3H);

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 161.4, 146.9, 145.3, 130.0, 128.9, 128.8, 128.7, 121.3, 118.9, 38.4, 30.5, 26.2, 26.1.

MS (EI): m/z (%) 299 (M+1, 12), 298 (M, 22), 282 (24), 281 (32), 243 (20), 234 (32).

Anal. Calcd for $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_3$: C, 68.44; H, 6.08; N, 9.39; Found: C, 68.46; H, 6.24; N, 9.09.

6-tert-Butyl-3-nitro-2-phenylpyridine 1-oxide (3hs)



Pale yellow solid, m.p. 181-183 °C, R_f = 0.66 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3113, 2973, 2916, 2874, 1536, 1361, 1341, 1260, 1143, 1066, 984, 834, 819, 699.

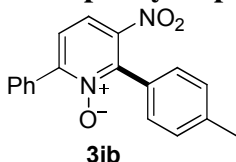
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.55 (d, J = 8.9 Hz, 1H), 7.45-7.41 (m, 4H), 7.36-7.34 (m, 2H), 1.46 (s, 9H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 162.7, 147.5, 146.5, 130.1, 129.0, 128.8, 122.2, 118.5, 37.2, 26.8.

MS (ESI): m/z (%) 273 (M+1, 100%).

Anal. Calcd for $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_3$: C, 66.16; H, 5.92; N, 10.29. Found: C, 66.37; H, 5.56; N, 10.47.

3-Nitro-6-phenyl-2-p-tolylpyridine 1-oxide (3ib)



Pale yellow solid, m.p. 205-207 °C, R_f = 0.59 (petroleum ether/ ethyl acetate = 5:1).

R (cm^{-1} , KBr): 3118, 3103, 3059, 1528, 1352, 1263, 815, 750, 694.

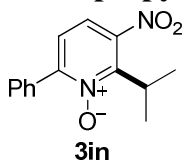
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.79-7.76 (m, 2H), 7.63 (d, J = 8.7 Hz, 1H), 7.49 (d, J = 8.7 Hz, 1H), 7.43-7.40 (m, 3H), 7.31 (d, J = 8.2 Hz, 2H), 7.24 (d, J = 8.0 Hz, 2H), 2.34 (s, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 152.9, 147.8, 146.3, 140.6, 131.6, 130.5, 129.5, 128.9, 125.3, 125.2, 118.9, 21.6.

MS (EI): m/z (%) 307 (M+1, 10), 306 (M, 45), 305 (M-1, 80), 261 (60), 259 (75), 115 (85), 95 (90), 63 (100).

Anal. Calcd for $\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_3$: C, 70.58; H, 4.61; N, 9.15; Found: C, 70.69; H, 4.31; N, 9.42.

3-Nitro-2-isopropyl-6-phenylpyridine 1-oxide (3in)



Pale yellow solid, m.p. 148-153 °C, R_f = 0.62 (petroleum ether/ ethyl acetate = 1:3).

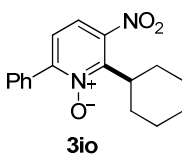
IR (cm^{-1} , KBr): 3008, 2977, 2943, 1557, 1526, 1347, 1259, 1060, 829, 779.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.71-7.68 (m, 2H), 7.46-7.39 (m, 4H), 7.33 (d, J = 8.6 Hz, 1H), 3.71-3.64 (m, 1H), 1.42 (d, J = 7.0 Hz, 6H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 152.7, 151.9, 148.5, 132.0, 130.3, 129.3, 128.4, 123.9, 118.6, 29.3, 17.2.

MS (EI): m/z (%) 258 (M, 2), 240 (10), 169 (15), 127 (35), 115 (100), 102 (60), 77 (75).
Anal. Calcd for $\text{C}_{14}\text{H}_{14}\text{N}_2\text{O}_3$: C, 65.11; H, 5.46; N, 10.85; Found: C, 65.32; H, 5.76; N, 10.98.

2-Cyclohexyl-3-nitro-6-phenylpyridine 1-oxide (3io)



Yellow solid, m.p. 156-158 °C, R_f = 0.65 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3068, 2971, 1557, 1530, 1351, 1276, 830, 815.

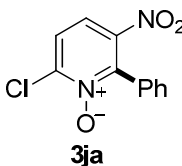
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.68-7.65 (m, 2H), 7.44-7.41 (m, 3H), 7.34-7.28 (m, 2H), 3.35 (s, 1H), 2.15 (s, 2H), 1.79-1.64 (m, 5H), 1.37-1.21 (m, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 152.6, 150.9, 148.8, 132.2, 130.2, 129.3, 128.4, 123.9, 118.4, 40.0, 26.5, 25.6.

MS (EI): m/z (%) 299 (M+1, 5), 298 (M, 2), 281 (20), 169 (30), 154 (55), 115 (100), 102 (75), 77 (95).

Anal. Calcd for $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_3$: C, 68.44; H, 6.08; N, 9.39; Found: C, 68.16; H, 6.27; N, 9.65.

6-Chloro-3-nitro-2-phenylpyridine 1-oxide (3ja)



Yellow solid, m.p. 163-164 °C, R_f = 0.56 (petroleum ether/ ethyl acetate = 1:3).

IR (cm^{-1} , KBr): 3108, 3078, 2922, 1547, 1529, 1461, 1443, 1347, 1262, 1150, 1064, 698.

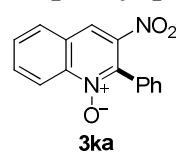
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.66 (d, J = 8.9 Hz, 1H), 7.61 (d, J = 9.0 Hz, 1H), 7.53-7.51 (m, 3H), 7.44-7.43 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 147.2, 146.9, 146.5, 130.8, 128.9, 127.6, 125.1, 118.7.

MS (ESI): m/z (%) 250.9 (M+1, 100).

Anal. Calcd for $\text{C}_{11}\text{H}_7\text{ClN}_2\text{O}_3$: C, 52.71; H, 2.82; N, 11.18; Found: C, 52.44; H, 2.53; N, 11.43.

3-nitro-2-phenylquinoline 1-oxide (3ka)



Pale yellow solid, m.p. 196-197 °C, R_f = 0.18 (petroleum ether/ ethyl acetate = 2:1).

IR (cm^{-1} , KBr): 3065, 1592, 1560, 1532, 1485, 1350, 1326, 1313, 1117, 889, 777, 753.

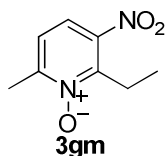
^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.11 (d, $J = 8.6$ Hz, 1H), 8.07 (s, 1H), 7.85 (d, $J = 8.1$ Hz, 1H), 7.76 (t, $J = 7.4$ Hz, 1H), 7.62 (t, $J = 7.5$ Hz, 1H), 7.34 (s, 5H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 145.6, 143.6, 133.3, 133.2, 130.4, 129.6, 129.1, 128.8, 126.8, 120.8, 119.6.

MS (EI): m/z (%) 267 (M+1, 15), 266 (M, 60), 265 (M-1, 75), 221 (50), 190 (55), 163 (45), 75 (65), 53 (100).

Anal. Calcd for $\text{C}_{15}\text{H}_{10}\text{N}_2\text{O}_3$: C, 67.67; H, 3.79; N, 10.52; Found: C, 67.55; H, 3.98; N, 10.48.

2-Ethyl-6-methyl-3-nitropyridine 1-oxide (3gm) (Scheme 4)



Yellow solid, m.p. 58-59 °C, $R_f = 0.35$ (ethyl acetate/ethanol = 5:1).

IR (cm^{-1} , KBr): 3076, 2979, 2941, 1563, 1532, 1352, 1265, 1078, 1038, 819.

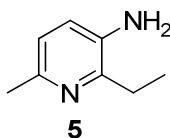
^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.56 (d, $J = 8.6$ Hz, 1H), 7.22 (d, $J = 8.6$ Hz, 1H), 3.11 (q, $J = 7.2$ Hz, 2H), 2.52 (s, 3H), 1.31 (t, $J = 7.3$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 153.6, 150.4, 146.9, 122.6, 119.0, 21.7, 18.8, 10.2.

MS (ESI): m/z (%) 183 (M+1, 100).

Anal. Calcd for $\text{C}_8\text{H}_{10}\text{N}_2\text{O}_3$: C, 52.74; H, 5.53; N, 15.38; Found: C, 52.47; H, 5.76; N, 15.14.

2-Ethyl-6-methylpyridin-3-amine (5) (Scheme 4)



Pale white solid, m.p. 108-111 °C, $R_f = 0.21$ (ethyl acetate/ethanol = 1:1).

IR (cm^{-1} , KBr): 3433, 3317, 3195, 2966, 2930, 2872, 1630, 1578, 1478, 1296, 1276, 1151, 1046, 834.

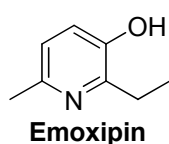
^1H NMR (400 MHz, CDCl_3) δ (ppm): 6.80 (d, $J = 7.9$ Hz, 1H), 6.75 (d, $J = 8.0$ Hz, 1H), 3.43 (s, 1H), 2.65 (q, $J = 7.5$ Hz, 2H), 2.37 (s, 3H), 1.21 (t, $J = 7.6$ Hz, 3H).

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 147.6, 147.5, 137.1, 123.1, 121.3, 26.9, 23.3, 11.6.

MS (ESI): m/z (%) 137 (M+1, 100).

Anal. Calcd for $\text{C}_8\text{H}_{12}\text{N}_2$: C, 70.55; H, 8.88; N, 20.57; Found: C, 70.76; H, 8.98; N, 20.94.

2-Ethyl-6-methylpyridin-3-ol (Emoxipin)¹⁰ (Scheme 4)

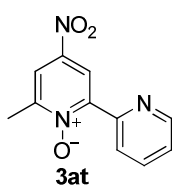


Pale white solid, m.p. 169-170 °C (lit.¹⁰ 168-169 °C), $R_f = 0.25$ (ethyl acetate/ethanol = 1:1)..

IR (cm^{-1} , KBr): 2968, 2936, 2876, 2569, 1819, 1580, 1499, 1454, 1425, 1351, 1271, 1228, 1164, 1130.

^1H NMR (400 MHz, D_2O) δ (ppm): 7.10 (d, $J = 8.6$ Hz, 1H), 6.96 (d, $J = 8.6$ Hz, 1H), 2.60 (q, $J = 7.6$ Hz, 2H), 2.25 (s, 3H), 1.00 (t, $J = 7.6$ Hz, 3H).

2-Methyl-4-nitro-6-(pyridin-2-yl)pyridine 1-oxide¹¹ (3at) (Scheme 5)

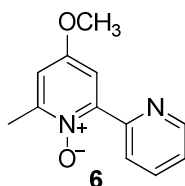


Yellow solid, m.p. 174–175 °C (lit.¹¹ 174–175 °C), R_f = 0.32 (petroleum ether/ethyl acetate = 5:1).

IR(cm^{-1} , KBr): 3079, 1526, 1452, 1336, 1273, 1090, 741.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.90 (d, J = 3.20 Hz, 1H), 8.75–8.70 (m, 2H), 8.05 (d, J = 3.12, 1H), 7.81–7.77 (m, 1H), 7.35–7.32 (m, 1H), 2.56 (s, 3H).

4-Methoxy-2-methyl-6-(pyridin-2-yl)pyridine 1-oxide¹¹ (6) (Scheme 5)

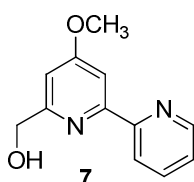


White solid, m.p. 89–90 °C (lit.¹¹ 88–89 °C), R_f = 0.38 (petroleum ether/ethyl acetate = 5:1).

IR (cm^{-1} , KBr): 3073, 2922, 1464, 1428, 1400, 1373, 1244, 853, 781.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.89 (d, J = 8.1 Hz, 1H), 8.64–8.63 (m, 1H), 7.76–7.72 (m, 1H), 7.53 (d, J = 3.6 Hz, 1H), 7.28–7.25 (m, 1H), 6.80 (d, J = 3.3 Hz, 1H), 3.84 (s, 3H), 2.52 (s, 3H).

(4-Methoxy-2,2'-bipyridin-6-yl)methanol¹¹ (7) (Scheme 5)

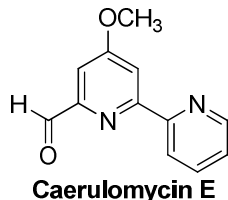


White solid, m.p. 63–64 °C (lit.¹¹ 63–64 °C). R_f = 0.35 (petroleum ether/ethyl acetate = 1:1).

IR (cm^{-1} , KBr): 3422, 3270, 2934, 1603, 1586, 1570, 1463, 1430, 1052, 793.

^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.59 (d, J = 4.4 Hz, 1H), 8.33 (d, J = 8.0 Hz, 1H), 7.82 (s, 1H), 7.75–7.71 (m, 1H), 7.25–7.22 (m, 1H), 6.69 (s, 1H), 4.69 (s, 2H), 3.87 (s, 3H).

4-Methoxy-2,2'-bipyridine-6-carbaldehyde¹¹ (Caerulomycin E) (Scheme 5)



Caerulomycin E

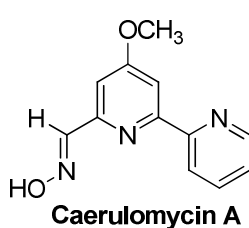
White solid, m.p. 81–82 °C (lit.¹¹ 80–81), R_f = 0.60

(petroleum ether/ethyl acetate = 1:1).

IR (cm^{-1} , KBr): 2922, 2852, 1711, 1603, 1586, 1464, 1433, 1379, 1221, 1049, 936;

^1H NMR (400 MHz, CDCl_3) δ (ppm): 10.06 (s, 1H), 8.64 (d, J = 4.8 Hz, 1H), 8.49 (d, J = 8.0 Hz, 1H), 8.15 (s, 1H), 7.82 (t, J = 7.7 Hz, 1H), 7.43 (d, J = 2.5 Hz, 1H), 7.31 (t, J = 5.8 Hz, 1H), 3.94 (s, 3H).

(E)-4-methoxy-2,2'-bipyridine-6-carbaldehyde oxime¹¹ (Caerulomycin A) (Scheme 5)



Caerulomycin A

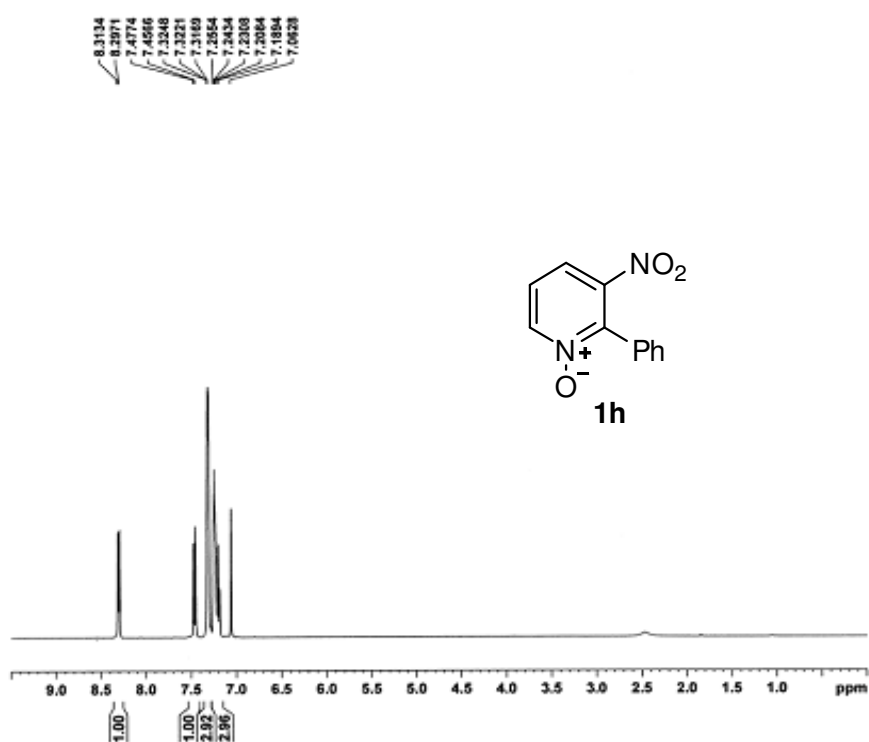
White solid, m.p. 172–174 °C (lit.¹¹ 172–173 °C), R_f = 0.20 (petroleum ether/ethyl acetate = 1:1).

IR (cm^{-1} , KBr): 3192, 3076, 2923, 2851, 2766, 1589, 1573, 1430, 1360, 1168, 981.

^1H NMR (400 MHz, CD_3SOCD_3) δ (ppm): 12.09 (s, 1H), 8.81 (d, J = 4.0 Hz, 1H), 8.59 (d, J = 7.7 Hz, 1H), 8.25–8.21 (m, 2H), 8.07 (s, 1H), 7.80–7.73 (m, 1H), 7.44 (s, 1H), 4.02 (s, 3H).

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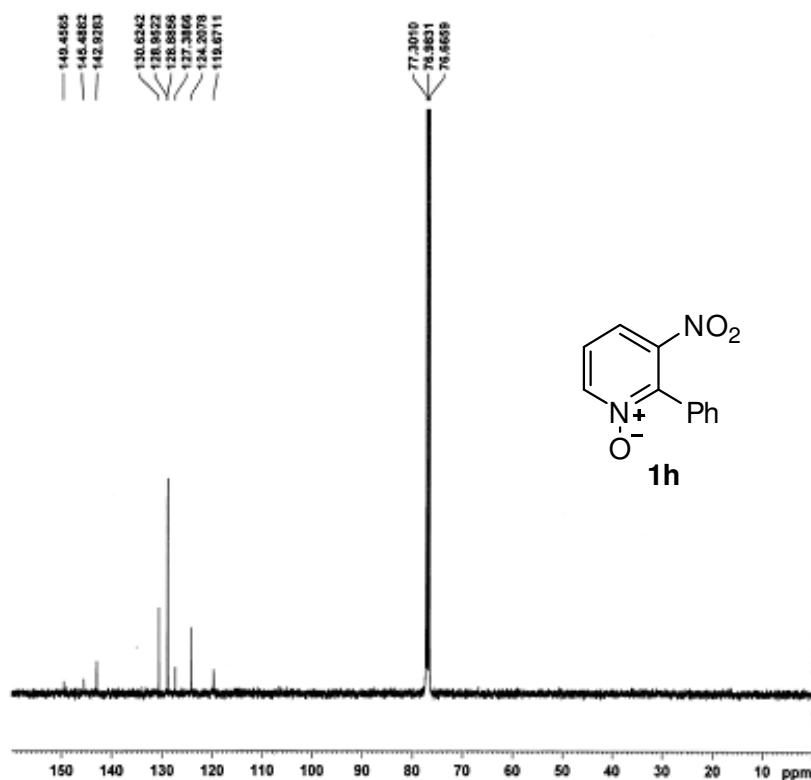
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FIDRES     0.125483 Hz
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DE         6.50 usec
TE         293.1 K
D1         1.0000000 sec
TDO        1

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WDW        EM
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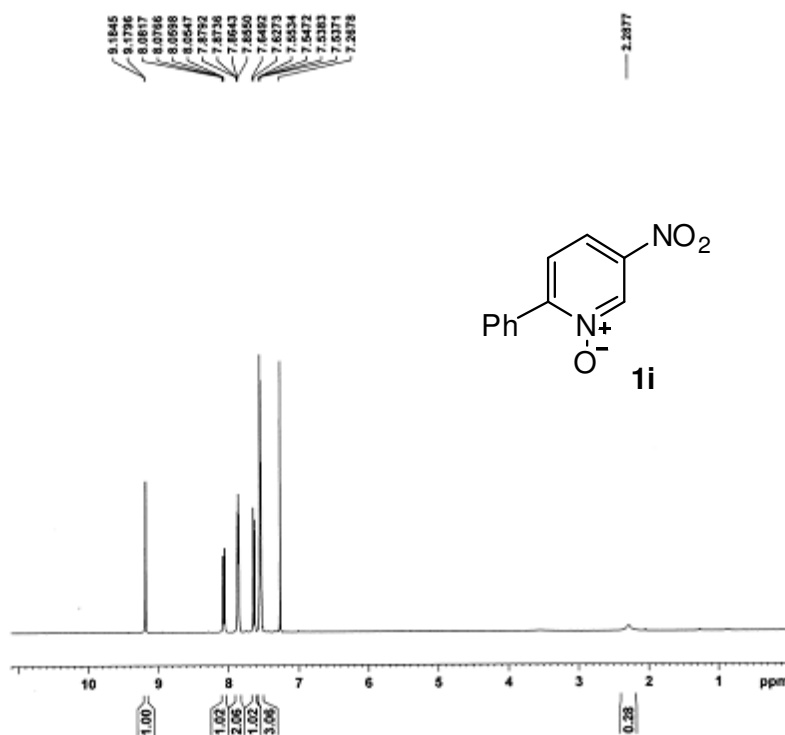
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PL12W      0.35276794 W
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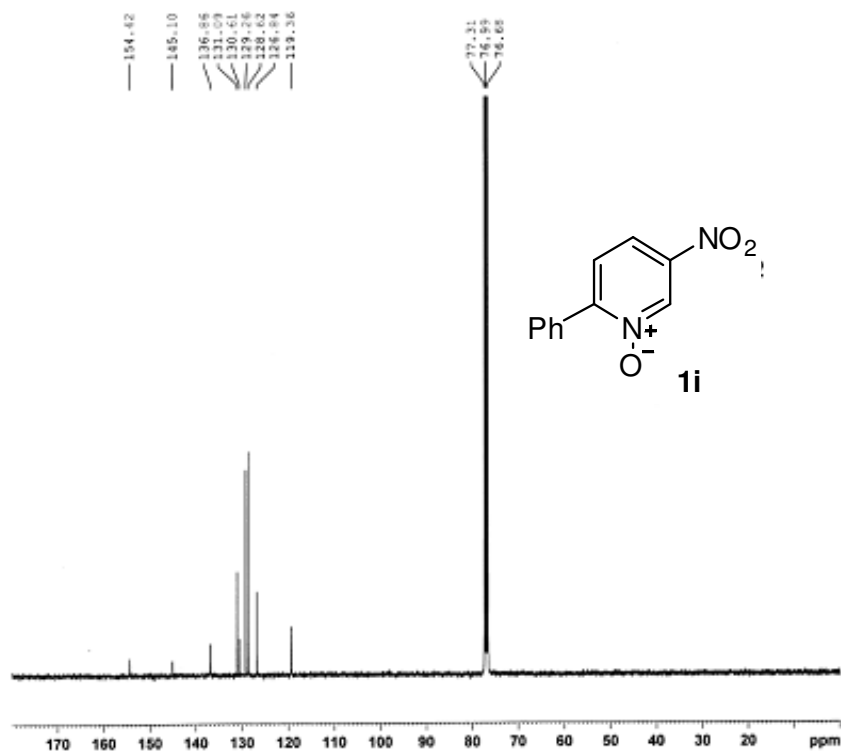


BRUKER

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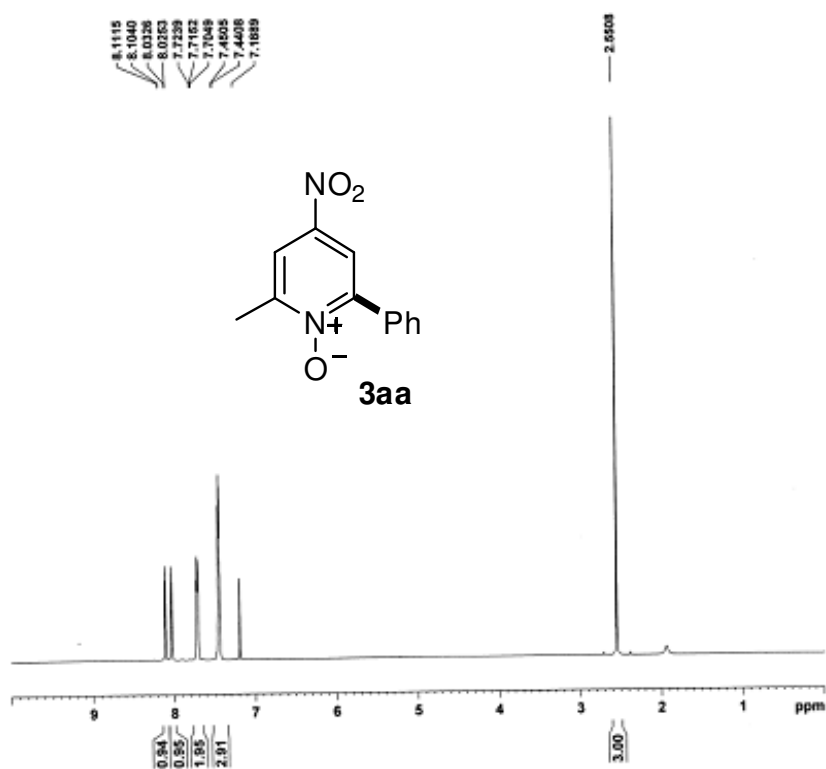
BRUKER

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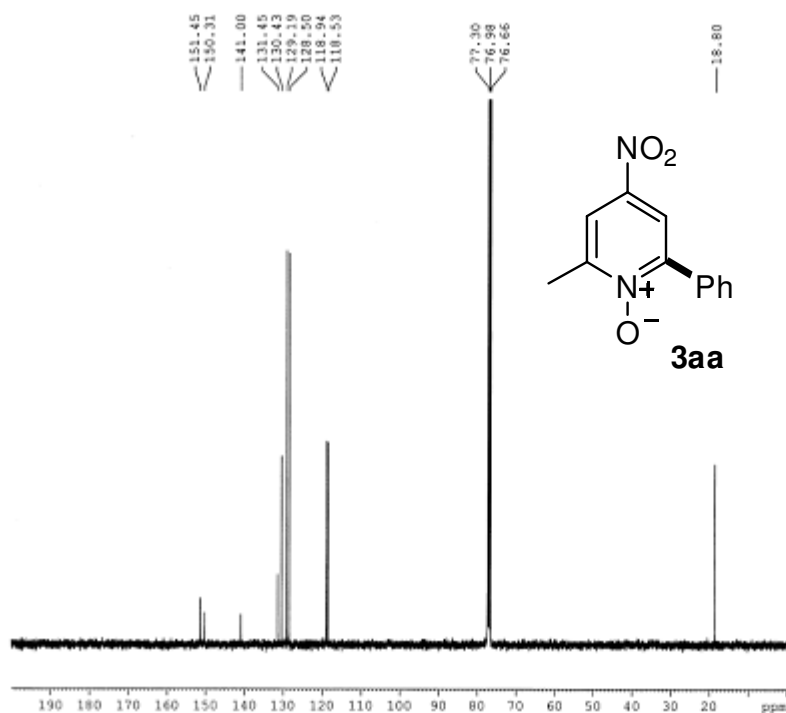
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PL12       14.26 dB
PL13       14.46 dB
PL2W       13.18669796 W
PL12W      0.39216794 W
PL13W      0.37529048 W
SFO2       400.1716007 MHz
SI         32768
SF         100.6278270 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```



NAME ZF6-4
 EXPNO 1
 PROCNO 1
 Date_ 20090604
 Time 16.00
 INSTRUM spect
 PROBRD 5 mm PABBO 88-
 PULPROG zgpg30
 TD 45536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWS 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 203
 TM 60.800 usec
 DE 6.50 usec
 TE 299.4 K
 D1 1.00000000 sec
 TDO 1

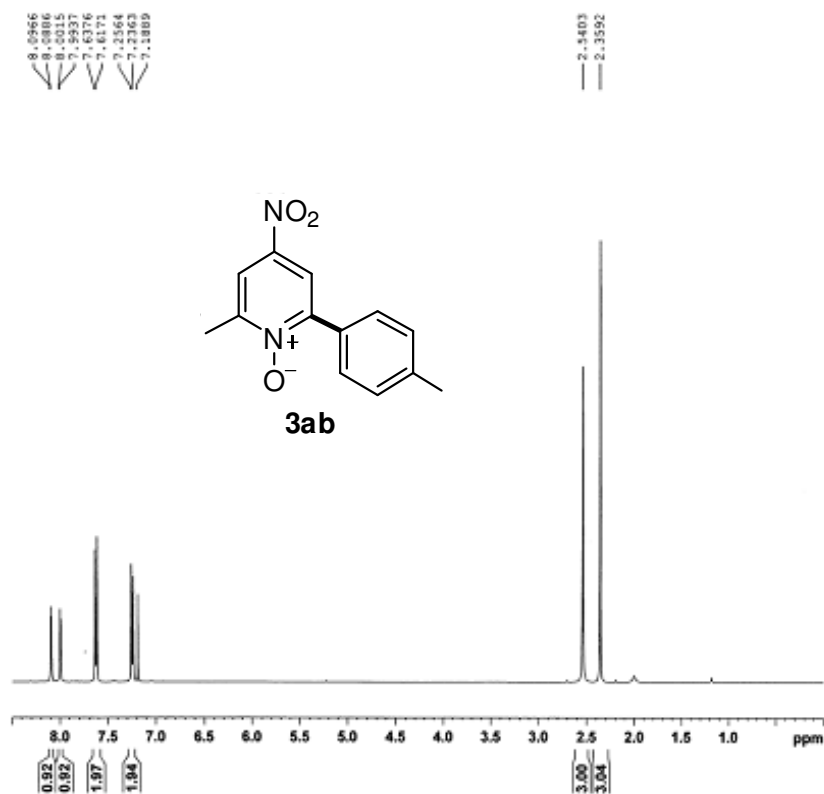
===== CHANNEL f1 =====
 NUC1 1H
 P1 13.80 usec
 PL1 -1.00 dB
 PL1W 13.18669796 W
 SFO1 400.1724712 MHz
 SI 32768
 SF 400.1700314 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



NAME ZF6-4-C13
 EXPNO 1
 PROCNO 1
 Date_ 20090604
 Time 20.33
 INSTRUM spect
 PROBRD 5 mm PABBO 88-
 PULPROG zgpg30
 TD 45536
 SOLVENT CDCl3
 NS 1804
 DS 4
 SWS 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 203
 TM 20.800 usec
 DE 6.50 usec
 TE 300.7 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.50 usec
 PL1 -2.00 dB
 PL1W 57.32743073 W
 SFO1 100.6328888 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 14.26 dB
 PL13 14.46 dB
 PL2W 13.18669796 W
 PL12W 0.39276794 W
 PL13W 0.37509048 W
 SFO2 400.1716007 MHz
 SI 32768
 SF 100.6228270 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

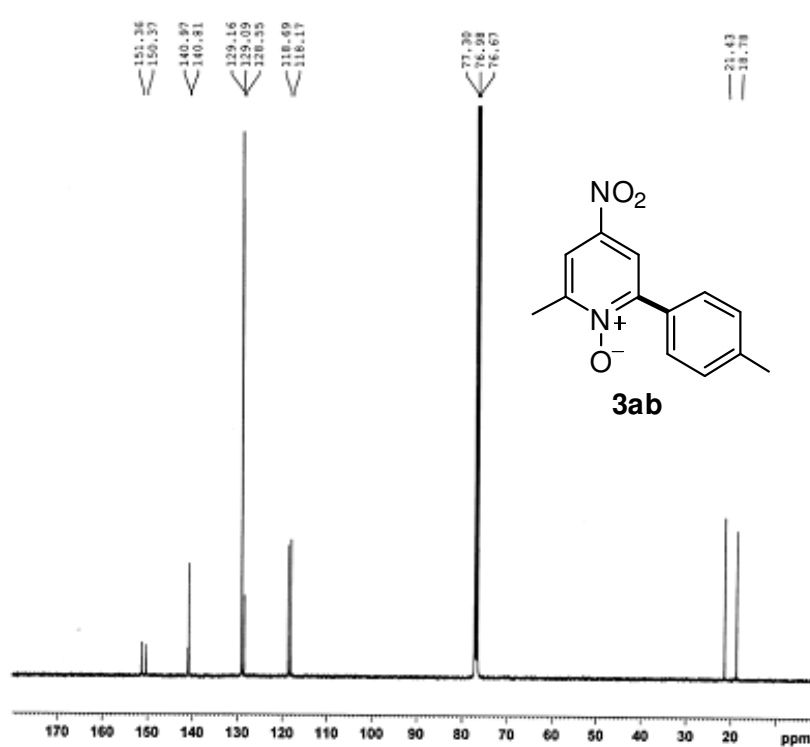


```

NAME      2F4-44-a
EXPNO     1
PROCNO    1
Date_     20080902
Time      9.26
INSTRUM   spect
PROBHD    5 mm F4BBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9446387 sec
RG         203
DW         40.800 usec
DE         6.50 usec
TE         297.7 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.80 usec
PL1        -1.00 dB
PL1W       13.18469796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1700314 MHz
WDW         EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00

```



```

NAME      5F4-44-a-Cl3
EXPNO     1
PROCNO    1
Date_     20080902
Time      22.38
INSTRUM   spect
PROBHD    5 mm F4BBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         8000
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
DW         20.800 usec
DE         6.50 usec
TE         299.9 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

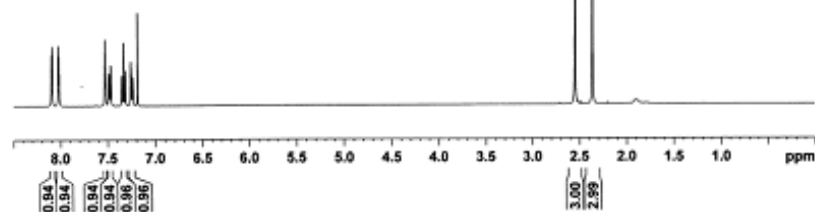
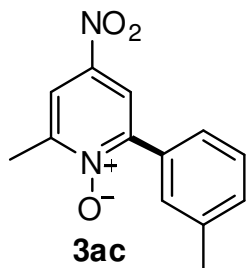
===== CHANNEL f1 =====
NUC1       13C
P1         8.50 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1       100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        -1.00 dB
PL12       14.26 dB
PL13       14.46 dB
PL1W       13.18669796 W
PL2W       0.39276794 W
PL13W      0.37529048 W
SFO2       400.1716007 MHz
SI         32768
SF         100.6228270 MHz
WDW         RM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40

```

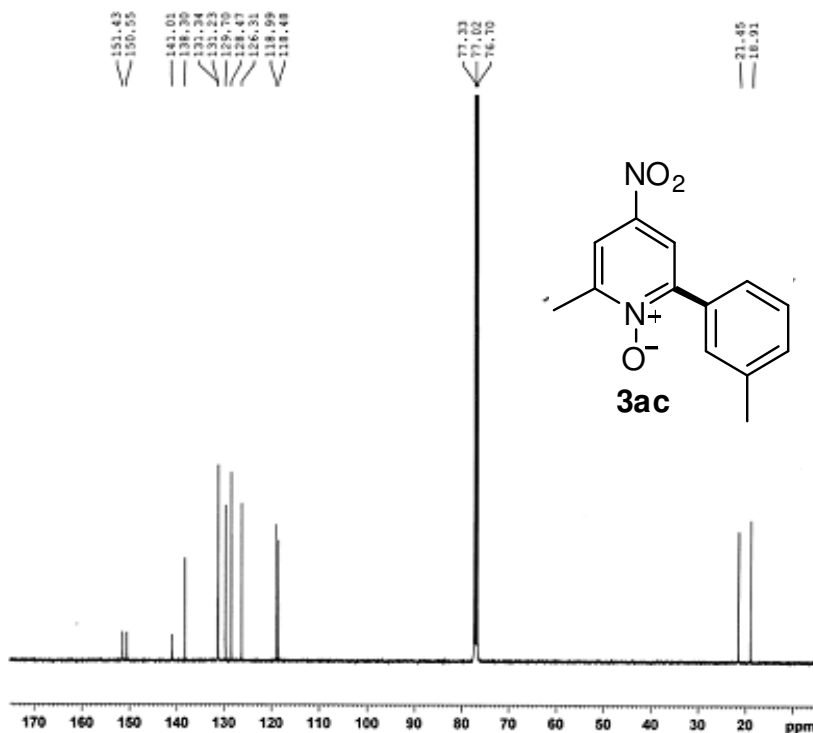
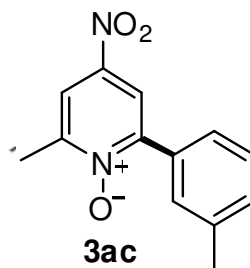
8.0054
8.0877
8.0230
8.0151
7.5284
7.4902
7.4512
7.3519
7.3328
7.3136
7.2566
7.2376
7.2898

2.5480
2.3634



NAME zft-48
EXPNO 1
PROCNO 1
Date_ 20080908
Time 9.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DM 60.800 usec
DE 6.50 usec
TE 297.5 K
D1 1.0000000 sec
TD0 1

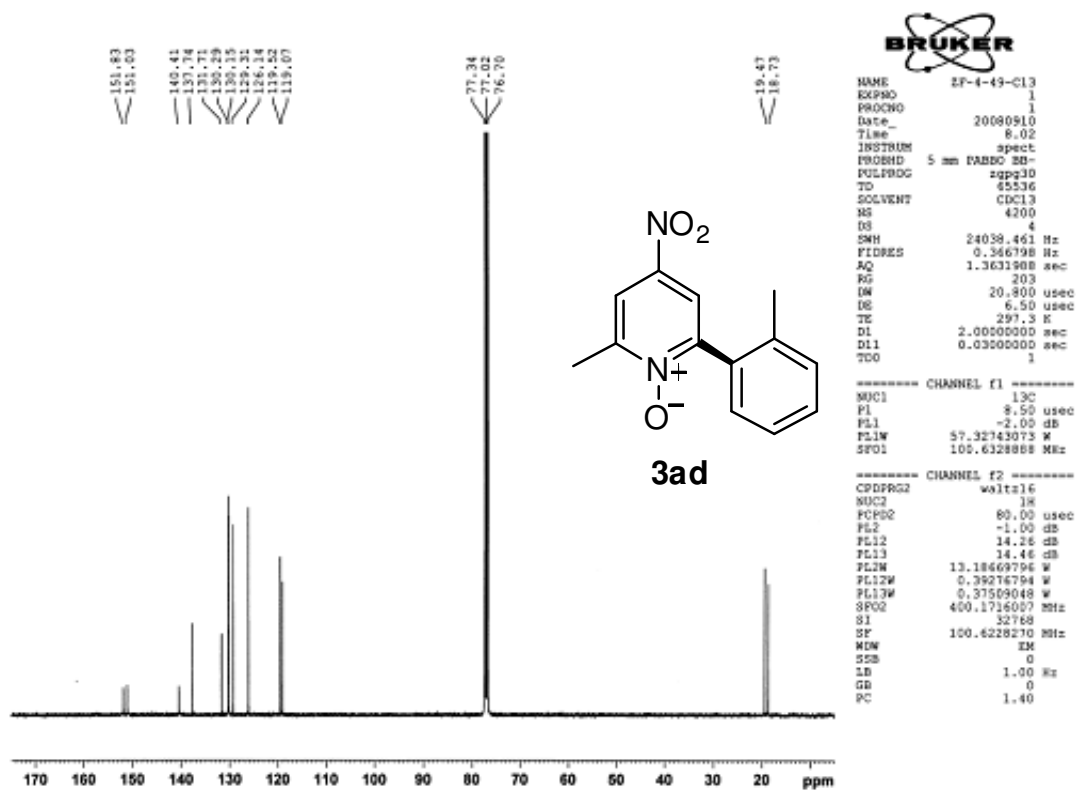
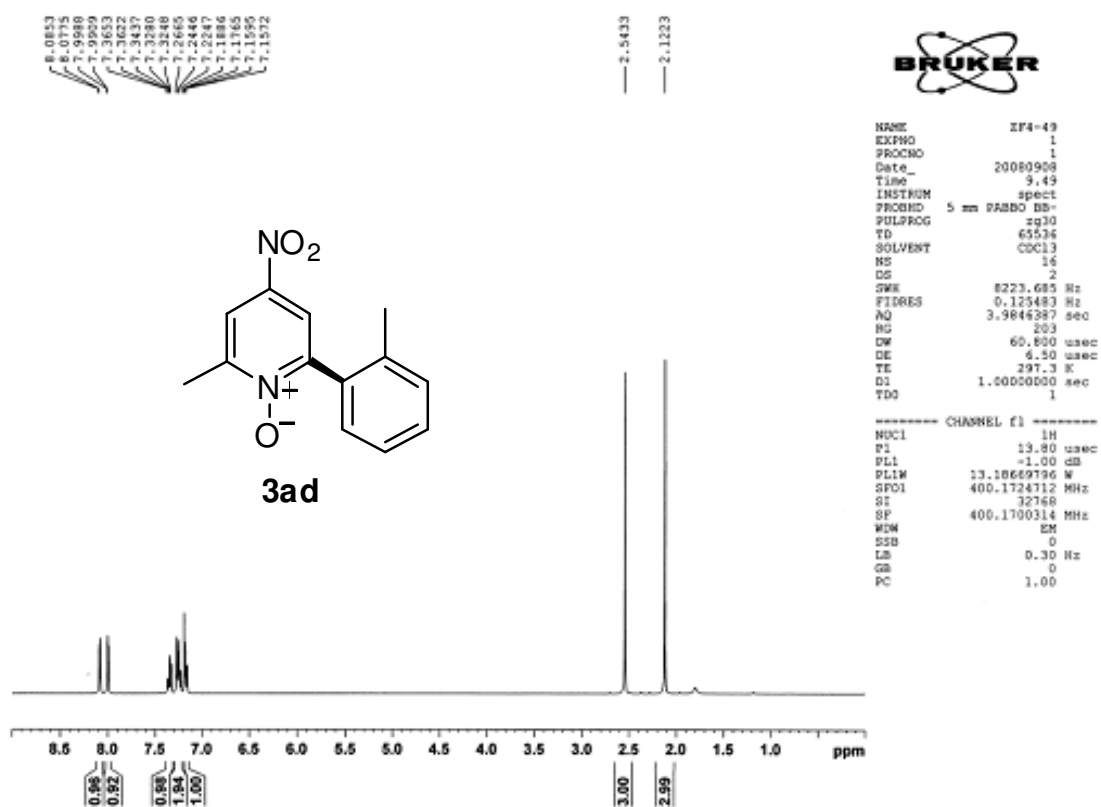
===== CHANNEL f1 =====
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700308 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

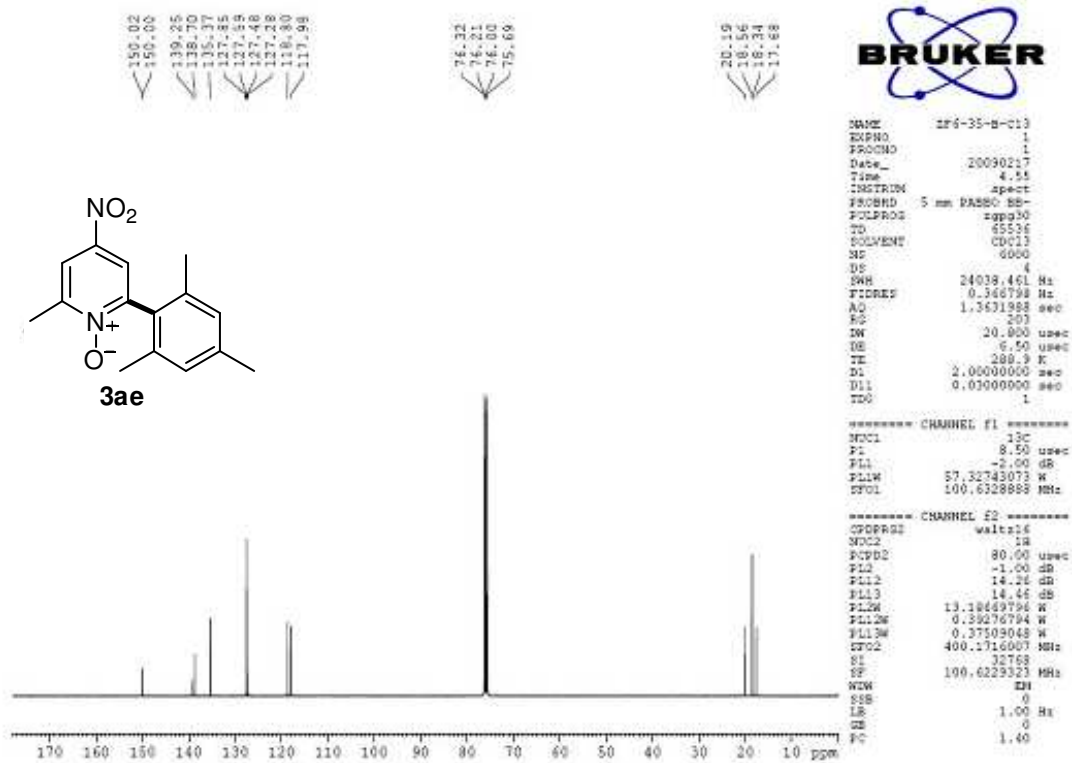
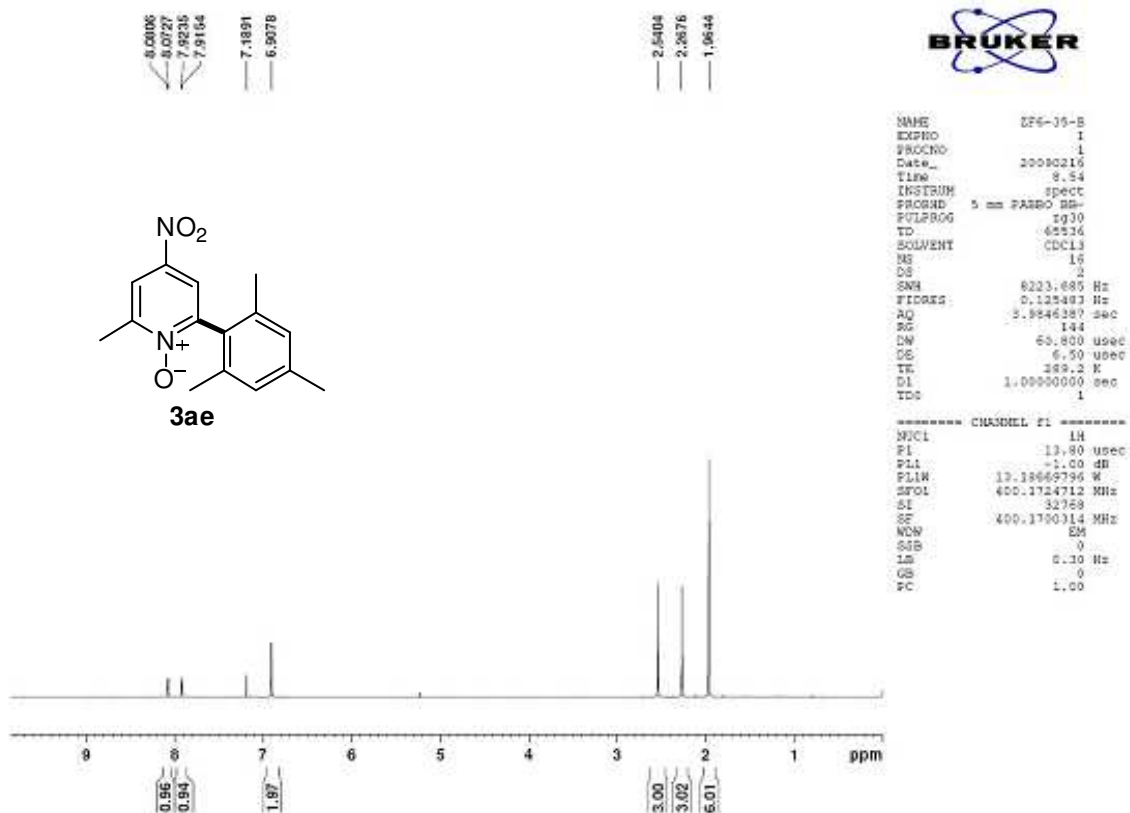


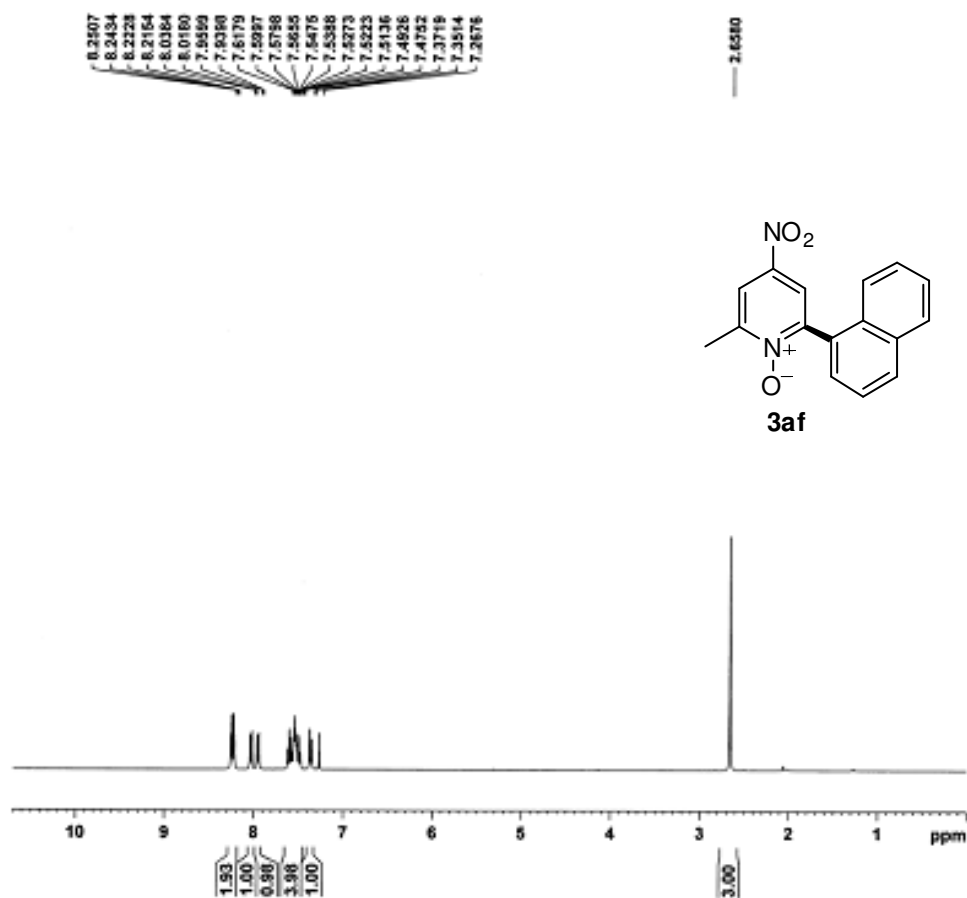
NAME zF-4-48-CL13
EXPNO 1
PROCNO 1
Date_ 20080910
Time 4.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6000
DS 4
SWH 24038.461 Hz
FIDRES 0.368798 Hz
AQ 1.3631888 sec
RG 203
DM 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz

===== CHANNEL f2 =====
CPCOREG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228210 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





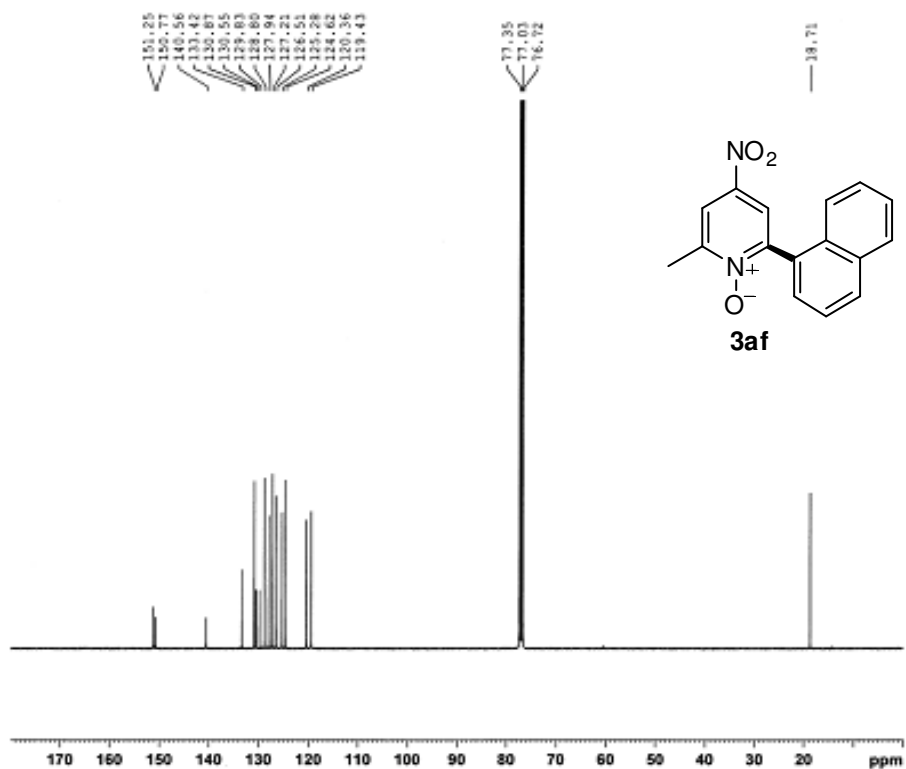


BRUKER

```

NAME      ZF-6-18
EXPNO     1
PROCNO    1
Date_     20081107
Time      8.57
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.485 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         181
DN         60.800 usec
DE         6.50 usec
TE         296.2 K
D1         1.0000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.80 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1700000 MHz
WDW         EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```



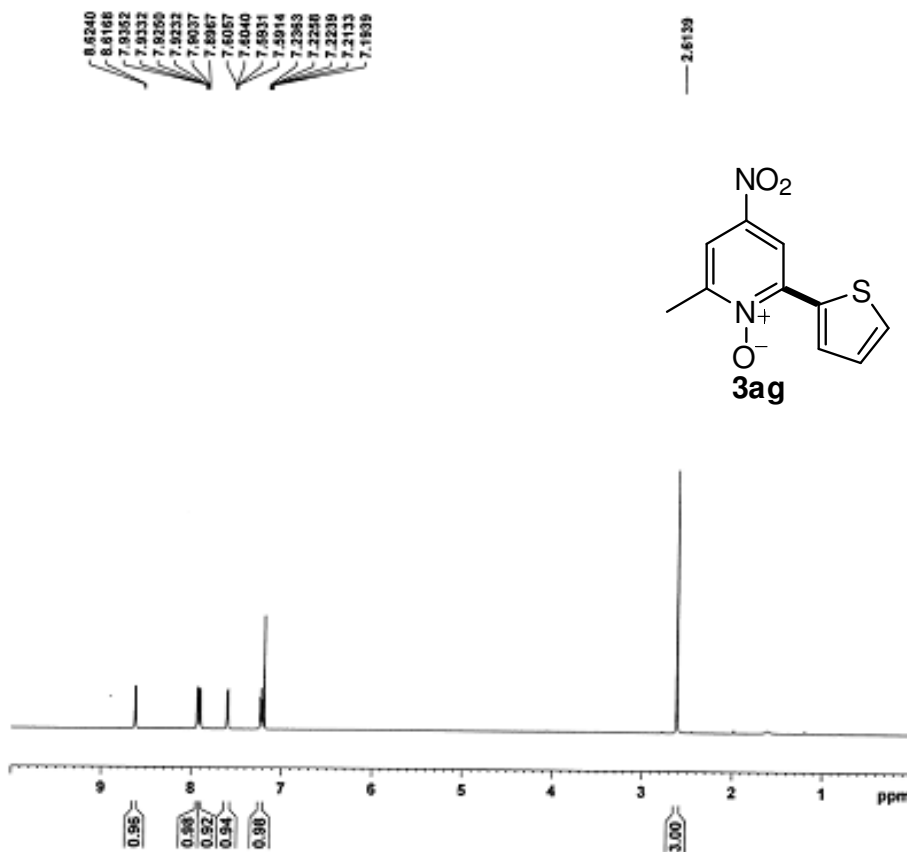
BRUKER

```

NAME      ZF-6-18-CL3
EXPNO     1
PROCNO    1
Date_     20081108
Time      14.18
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         6000
DS         4
SWH        24038.463 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
DN         20.800 usec
DE         8.50 usec
TE         300.3 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         8.50 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1       100.6228889 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -1.00 dB
PL12       14.24 dB
PL13       14.44 dB
PL1W       13.18669796 W
PL12W      0.39276794 W
PL13W      0.37509048 W
SFO2       400.1716007 MHz
SI         32768
SF         100.6228270 MHz
WDW         EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

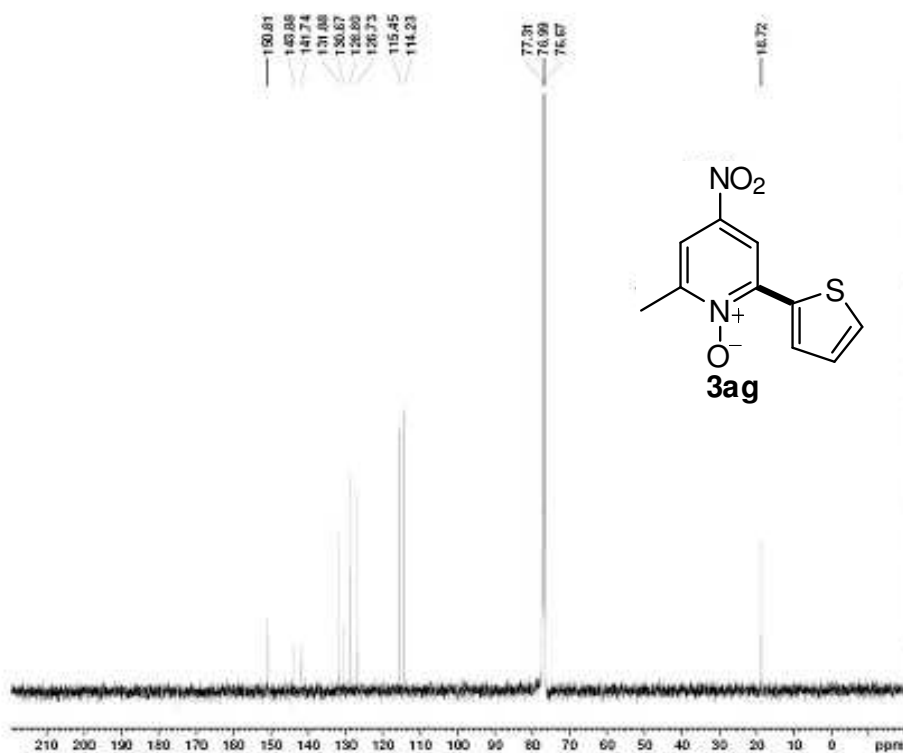


```

NAME      IR6-22
EXPNO     1
PROCNO    1
Date_     20081117
Time      9.19
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125493 Hz
AQ         3.9846387 sec
RG         203
CW         60.800 usec
DE         6.50 usec
TE         291.1 K
D1         1.0000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         13.80 usec
PL1        -1.00 dB
PL1W       13.18669196 W
SFO1       400.1924112 MHz
SI         32768
SF         400.1700296 MHz
WDW         EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```



```

NAME      IR6-22-013
EXPNO     1
PROCNO    1
Date_     20081117
Time      11.02
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
CW         20.600 usec
DE         6.50 usec
TE         291.2 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         9.50 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1       100.6228989 MHz
  
```

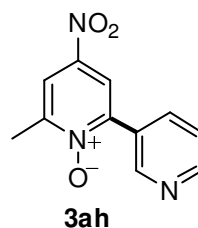
```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -1.00 dB
PL12       14.26 dB
PL13       14.48 dB
PL1W       13.18669196 W
PL12W      0.38276784 W
PL13W      0.37585648 W
SFO2       400.1718607 MHz
SI         32768
SF         100.6228270 MHz
WDW         EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

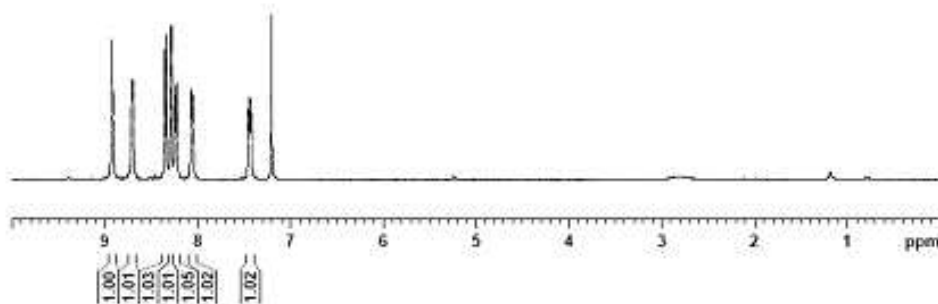
8.8182
8.7046
8.6951
8.3529
8.3500
8.2872
8.2803
8.2591
8.2183
8.0696
8.0624
8.0021
8.0449
7.4607
7.4384
7.4316
7.4191
7.2003



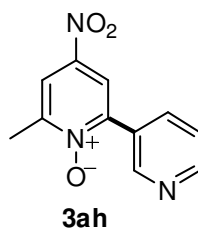
NAME SP6-24-2
EXPNO 1
PROCNO 1
Date_ 20081127
Time 18.16
INSTRUM spect
PROBHD 5 mm TABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8023.495 Hz
FIDRES 0.125493 Hz
AQ 3.9646387 sec
RG 203
BW 80.800 uHz
DE 4.50 uHz
TE 291.9 K
D1 1.00000000 sec
TD0 1



***** CHANNEL f1 *****
NUC1 1H
P1 11.80 uHz
PL1 -1.00 dB
PL1W 13.16669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1708268 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

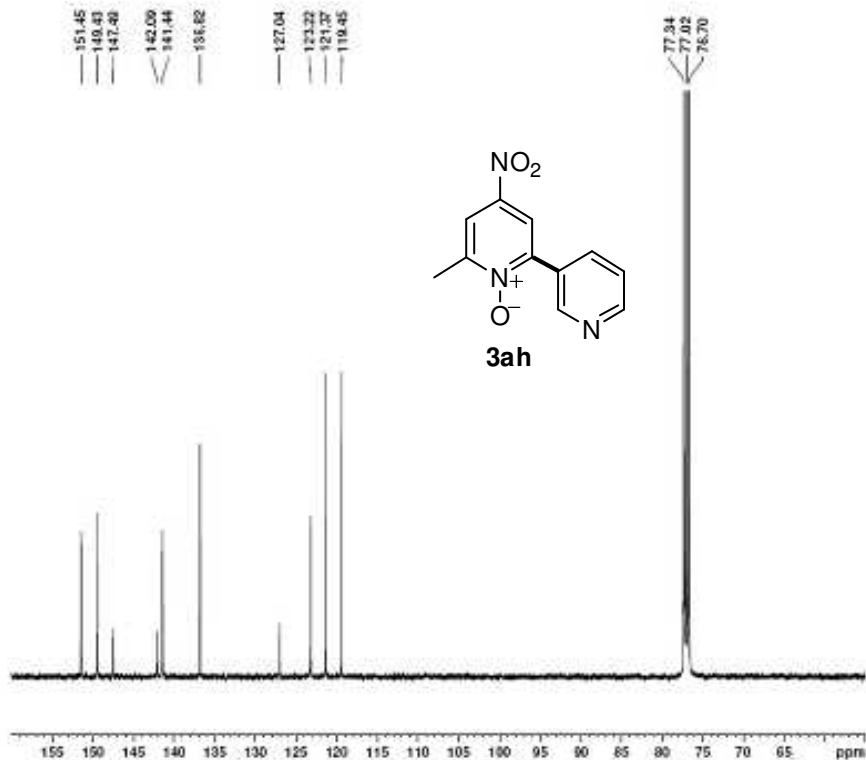


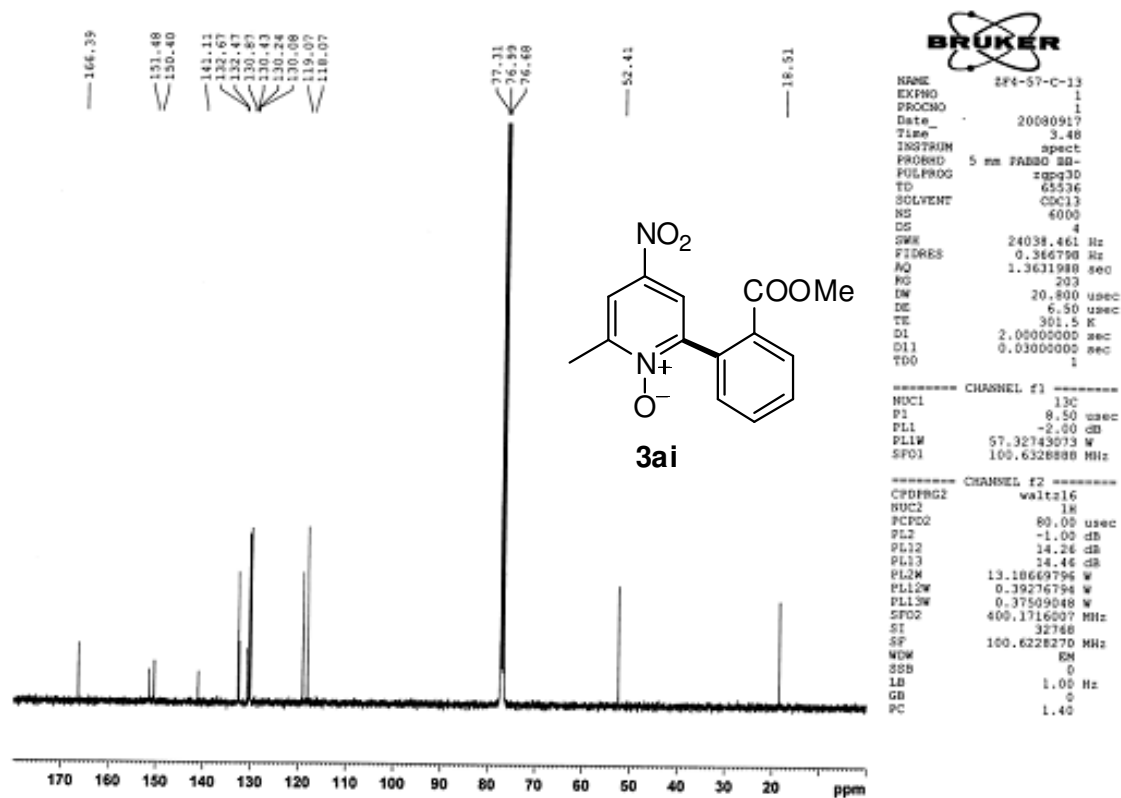
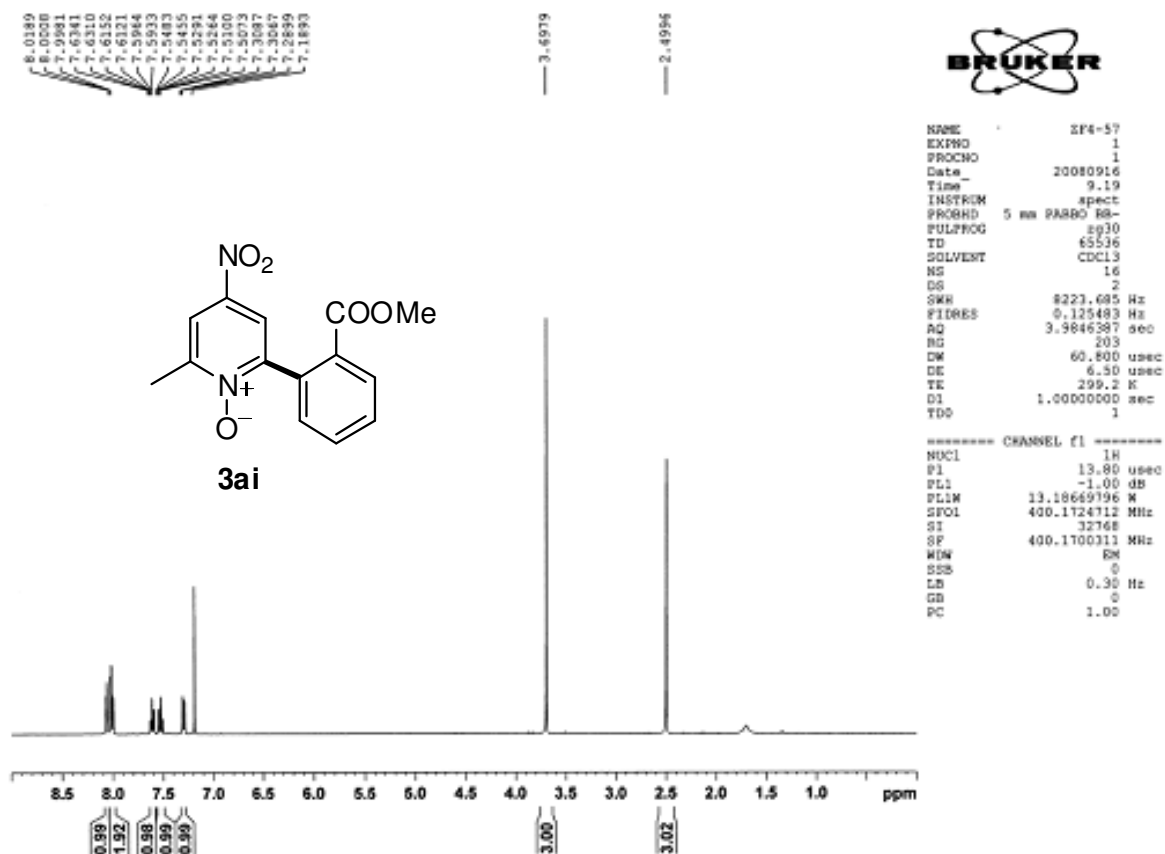
151.45
149.81
147.48
142.09
141.44
136.62
127.04
123.22
121.37
119.45

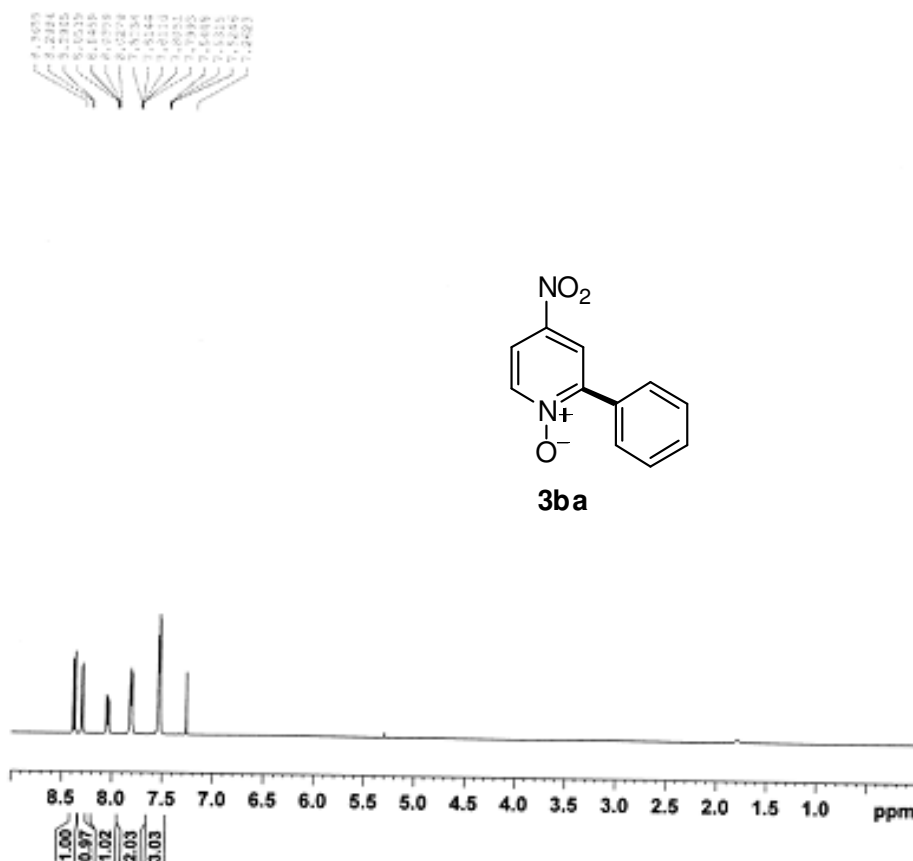


NAME SP-6-20-3-017
EXPNO 1
PROCNO 1
Date_ 20091128
Time 1.14
INSTRUM spect
PROBHD 5 mm TABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 4
SWH 34038.462 Hz
FIDRES 0.366798 Hz
AQ 1.3631908 sec
RG 203
BW 20.800 uHz
DE 6.50 uHz
TE 294.0 K
D1 2.00000000 sec
D11 0.05000000 sec
TD0 1

***** CHANNEL f1 *****
NUC1 13C
P1 8.50 uHz
PL1 -2.00 dB
PL1W 57.32743973 W
SFO1 100.626886 MHz
***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 uHz
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.16669796 W
PL12W 0.39276794 W
PL13W 0.37500448 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6268278 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





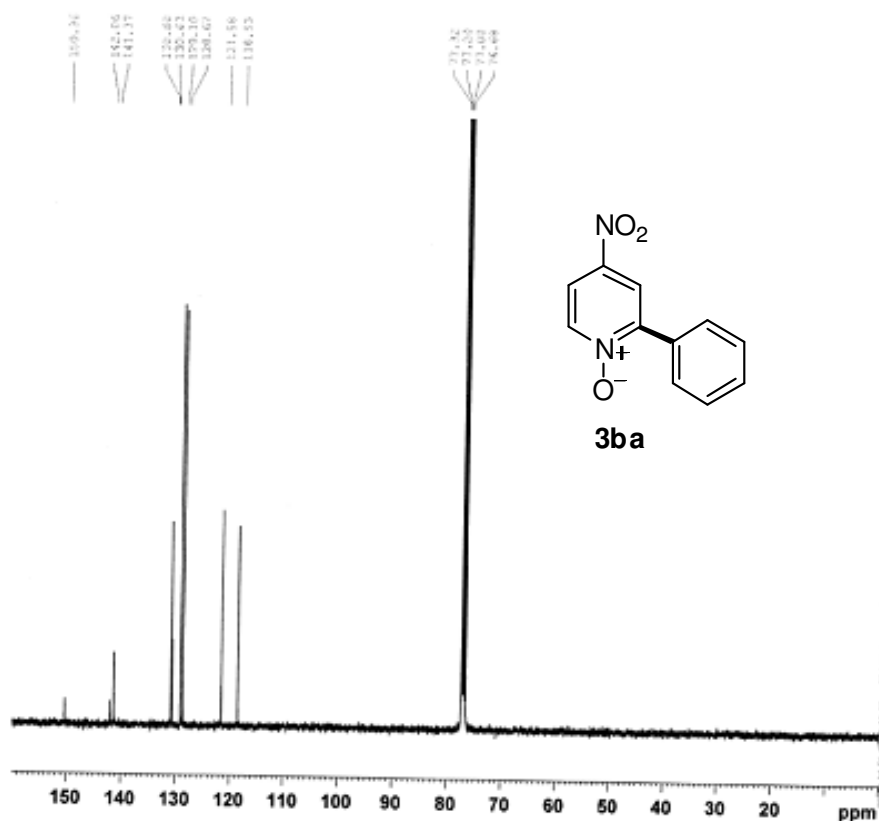


Current Data Parameters
NAME 001
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080409
Time 15.52
INSTRUM spect
PROBHD 5 mm PASPO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.485 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.000 usec
DE 4.50 usec
TE 297.2 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.50 usec
PL1 -1.00 dB
SFO1 400.1724712 MHz

F2 - Processing parameters
SI 32768
SF 400.1705020 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



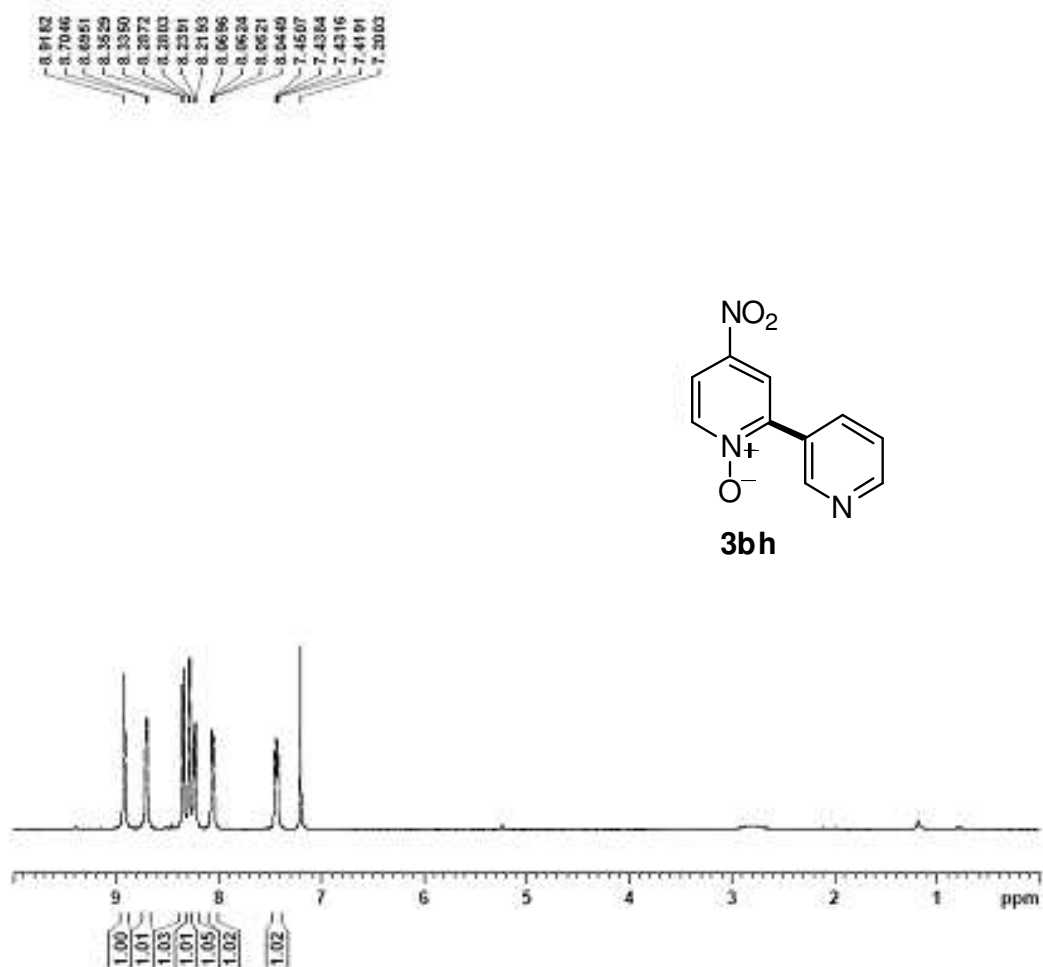
Current Data Parameters
NAME 2FD5092-C13
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080512
Time 3.14
INSTRUM spect
PROBHD 5 mm PASPO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6800
DS 4
SWH 24038.461 Hz
FIDRES 0.364798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999999 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.40 usec
PL1 -2.00 dB
SFO1 100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.46 dB
PL13 14.46 dB
PL2 -1.00 dB
SFO2 400.1716027 MHz

F2 - Processing parameters
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

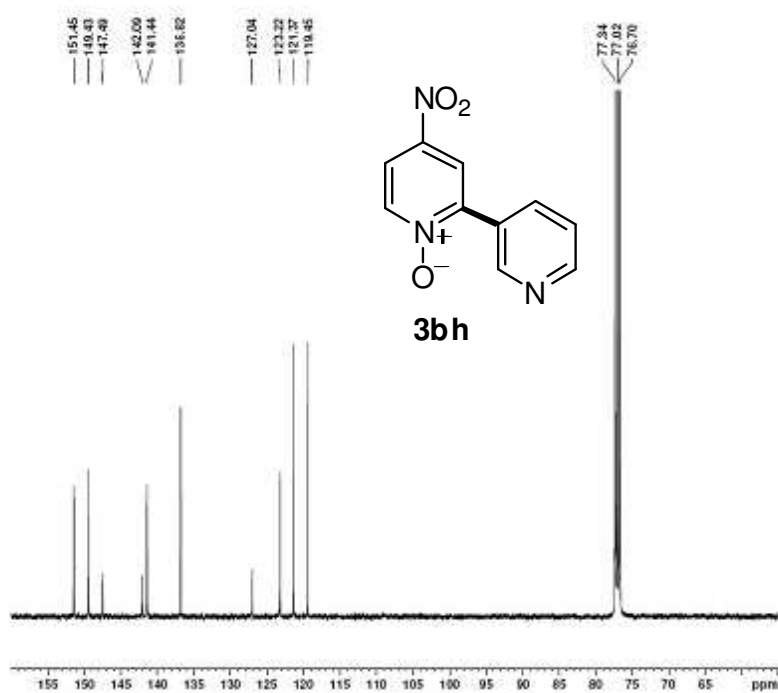


BRUKER

```

NAME      ZFC-26-2
EXPNO     1
PROCNO    1
Date_     20091127
Time      18.16
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8023.495 Hz
FIDRES     0.135483 Hz
AQ         1.9846387 sec
RG         203
EW         60.800 usec
DE         4.50 usec
TE         291.9 K
D1         1.00000000 sec
D11        1
TD0        1

***** CHANNEL f1 *****
NUC1       1H
P1         13.90 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1706260 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```



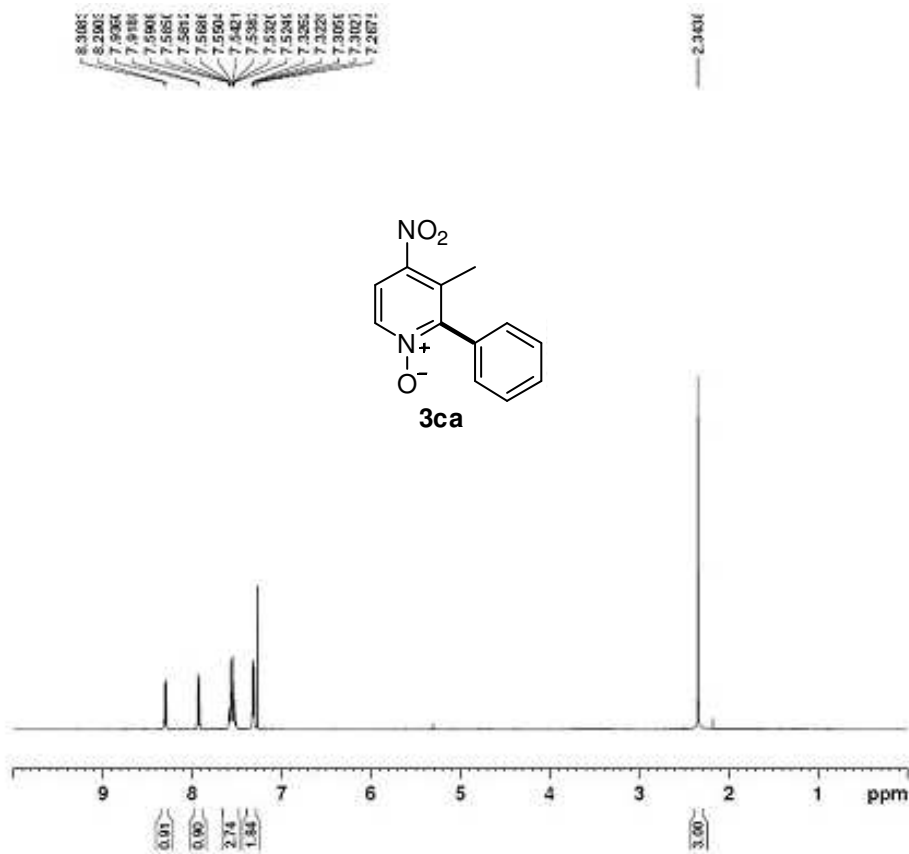
BRUKER

```

NAME      ZFC-26-2-013
EXPNO     1
PROCNO    1
Date_     20091127
Time      17.14
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         8100
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631308 sec
RG         203
EW         30.800 usec
DE         6.50 usec
TE         294.0 K
D1         2.00000100 sec
D11        0.03000100 sec
TD0        1

***** CHANNEL f1 *****
NUC1       13C
P1         8.50 usec
PL1        -2.00 dB
PL1W       57.32163073 W
SFO1       100.6228898 MHz

***** CHANNEL f2 *****
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        -1.00 dB
PL12       14.26 dB
PL13       14.46 dB
PL1W       13.18669796 W
PL12W      0.39276794 W
PL13W      0.37505048 W
SFO2       400.1116207 MHz
SI         32768
SF         100.6228270 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

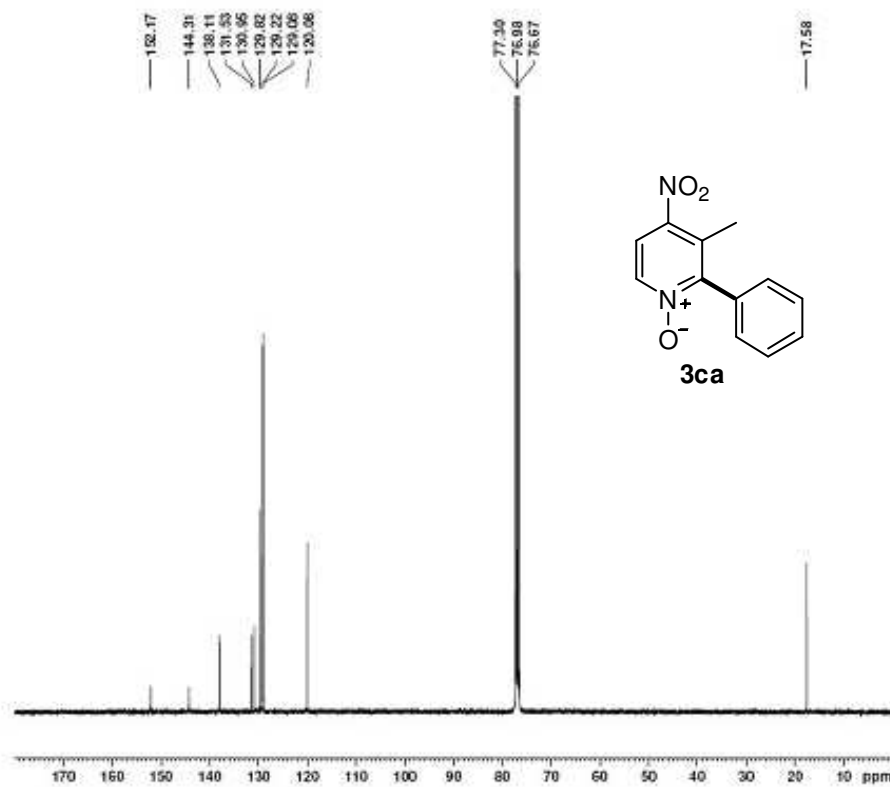


```

NAME      =56-7-b
EXPNO     1
PROCNO    1
Date_     20081006
Time      8.40
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125493 Hz
AQ         3.9846387 sec
RG         203
DW         60.900 usec
DE         6.50 usec
TE         295.3 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         13.80 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1700000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



```

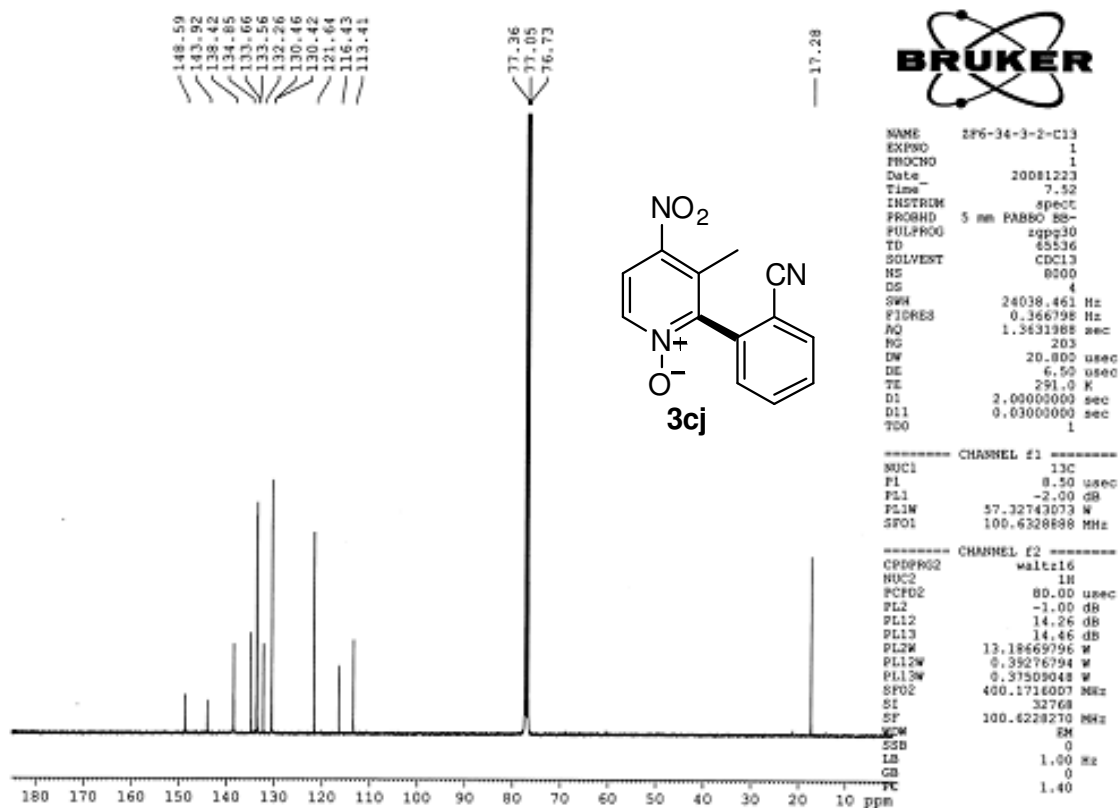
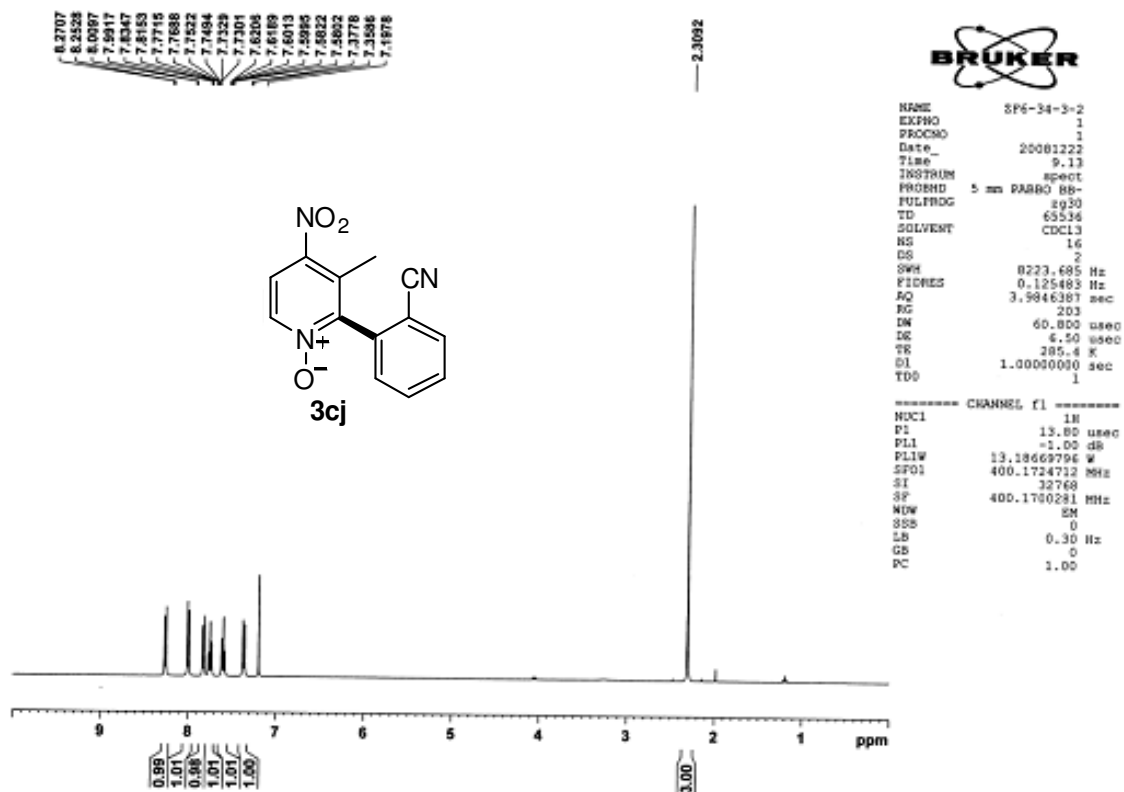
NAME      2FG-T-B-013
EXPNO     1
PROCNO    1
Date_     20081006
Time      22.31
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         10000
DS         4
SWH        24038.461 Hz
FIDRES     0.164798 Hz
AQ         1.1631988 sec
RG         203
DW         20.800 usec
DE         6.50 usec
TE         297.9 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

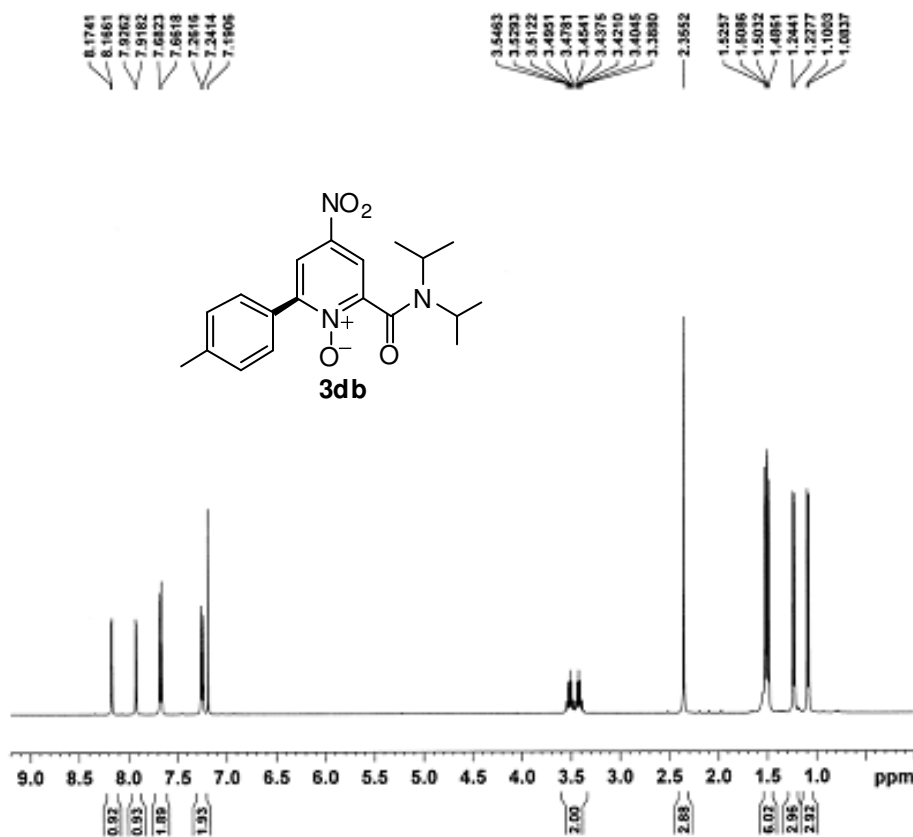
```

===== CHANNEL f1 =====
NUC1       13C
P1         8.50 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1       100.6229888 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -1.00 dB
PL12       14.26 dB
PL13       14.46 dB
PL1W       13.18669796 W
PL12W      0.39216794 W
PL13W      0.37589048 W
SFO2       400.1716007 MHz
SI         32768
SF         100.6229870 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```



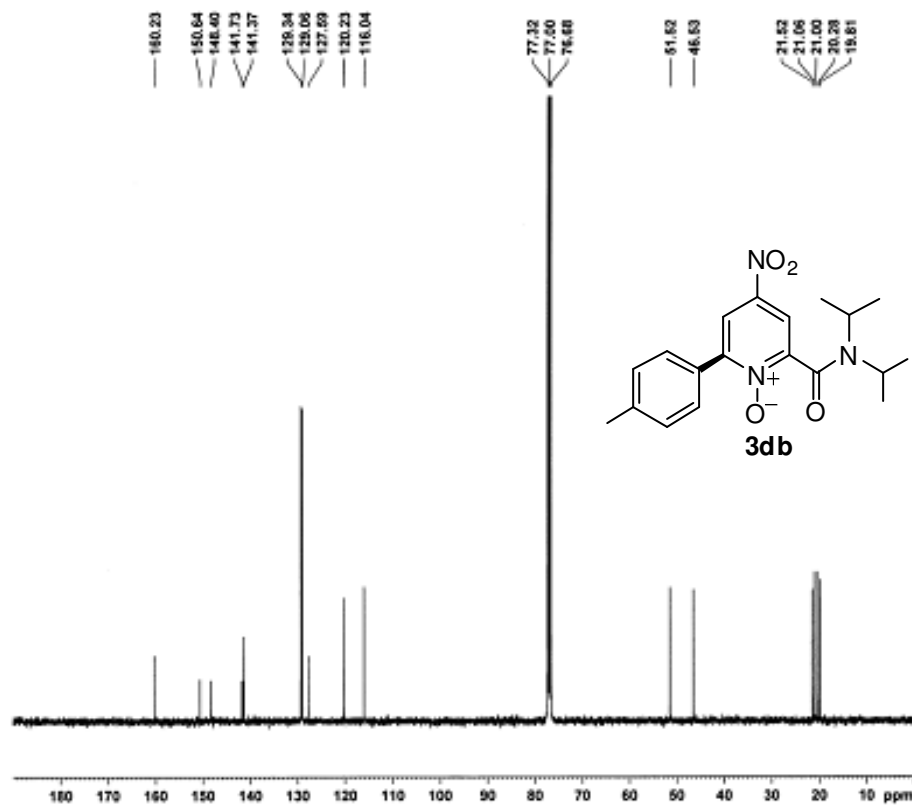


```

NAME      ZFB-20
EXPNO     1
PROCNO    1
Date_     20090413
Time      11.12
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125493 Hz
AQ         3.9846287 sec
RG         203
RW         60.800 usec
DE         6.50 usec
TE         298.7 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.80 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1700308 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

```



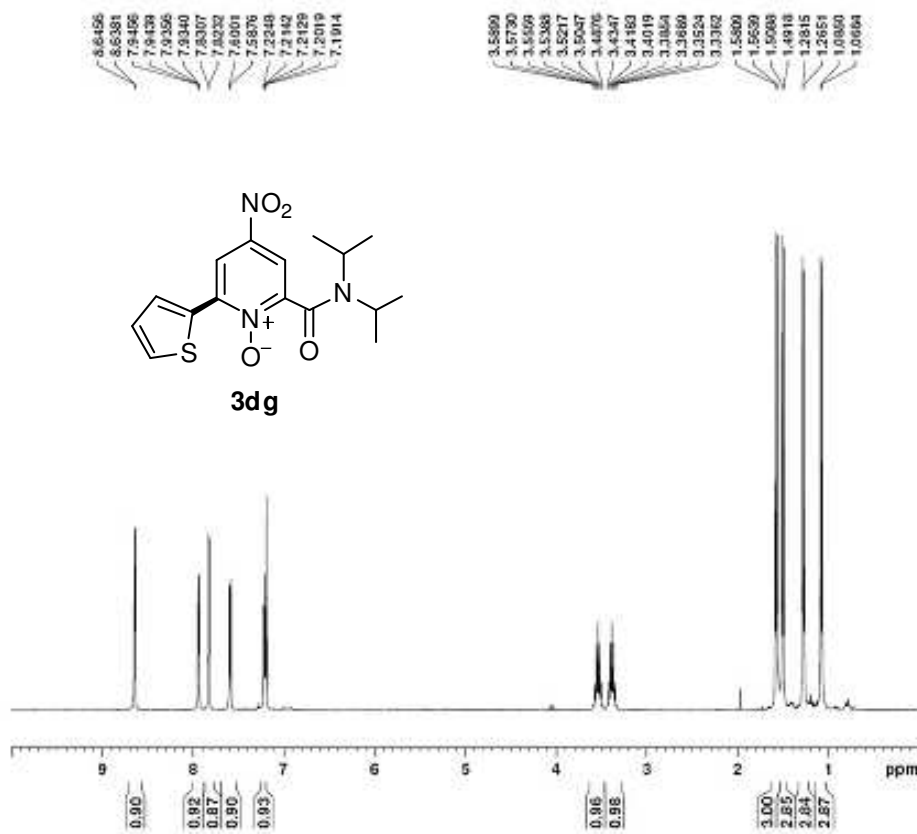
```

NAME      ZFB-20-C13
EXPNO     1
PROCNO    1
Date_     20090416
Time      1.51
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
RG         4000
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
RW         20.800 usec
DE         6.50 usec
TE         299.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         8.50 usec
PL1        -2.00 dB
PL1W       57.32743013 W
SFO1       100.6228270 MHz

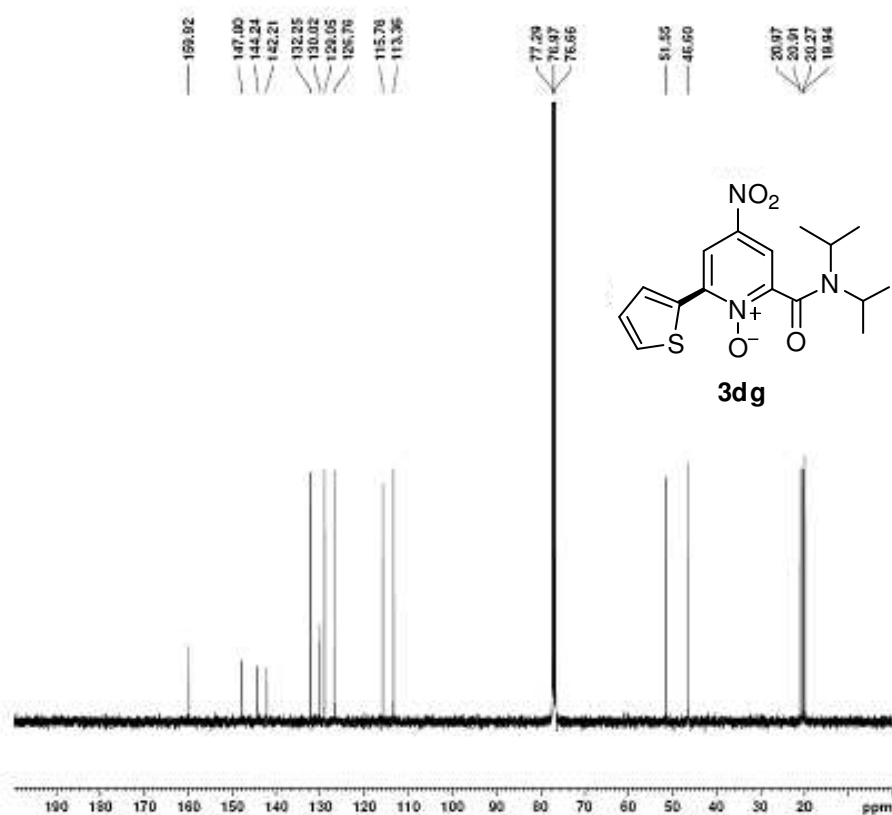
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -1.00 dB
PL12       14.26 dB
PL13       14.46 dB
PL1W       13.18669796 W
PL12W      0.39276794 W
PL13W      0.37509048 W
SFO2       400.1716007 MHz
SI         32768
SF         100.6228270 MHz
WDW        EM
SSB        0
LB         1.50 Hz
GB         0
PC         1.40

```



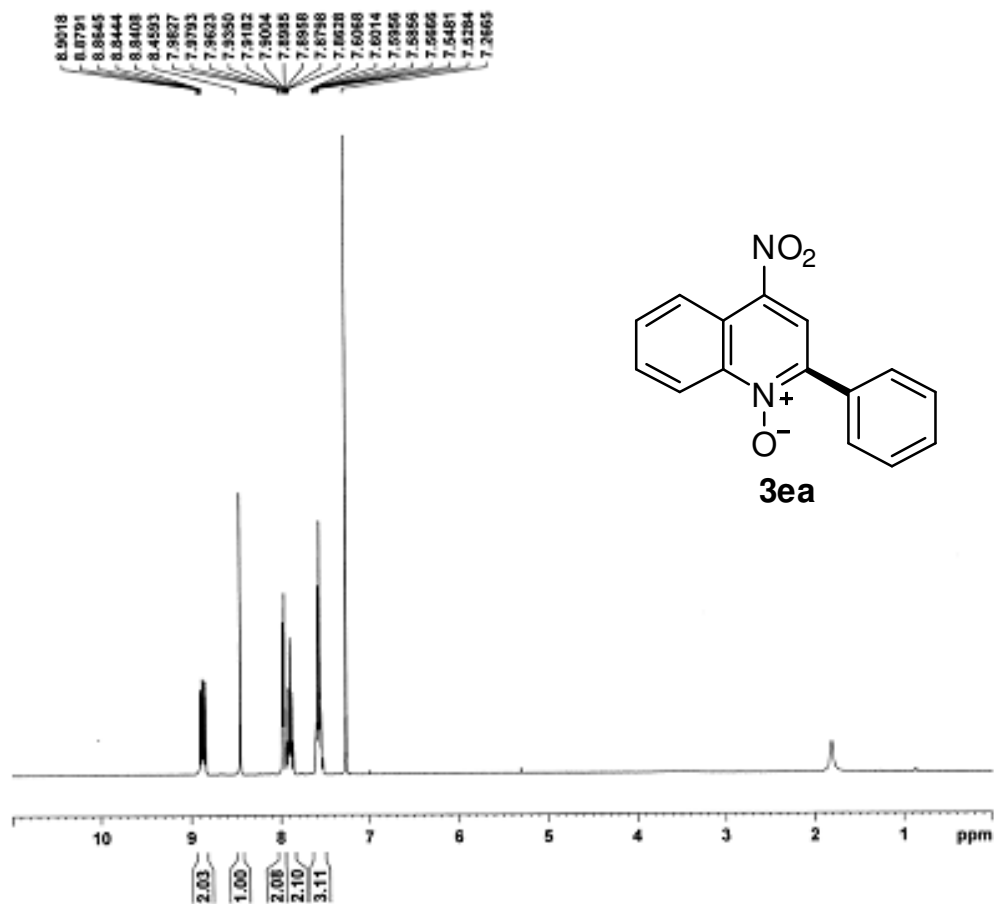
NAME ZF8-43
EXPNO 1
PROCNO 1
Date_ 20090428
Time 10.04
INSTRUM spect
PROBHD 5 mm PABBO B9-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.123483 Hz
AQ 3.9846381 sec
RG 203
DM 68.800 usec
DE 6.50 usec
TE 298.1 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.60 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1134712 MHz
SI 32768
SF 400.1134712 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



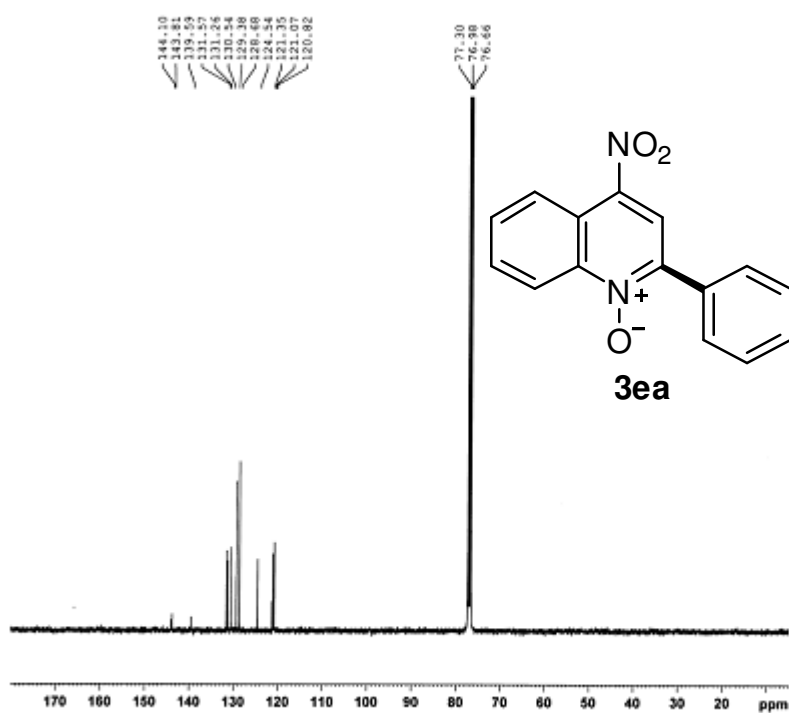
NAME ZF8-43-013
EXPNO 1
PROCNO 1
Date_ 20090503
Time 14.33
INSTRUM spect
PROBHD 5 mm PABBO B9-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 171
DS 4
SWH 24038.461 Hz
FIDRES 0.344798 Hz
AQ 1.3631998 sec
RG 203
DM 20.800 usec
DE 4.50 usec
TE 305.1 K
D1 2.0000000 sec
D11 0.0280000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.628888 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL2W 14.28 dB
PL3 14.46 dB
PL1W 13.18669796 W
PL2W 0.39214794 W
PL3W 0.37533004 W
SFO2 400.1114007 MHz
SI 32768
SF 100.628888 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



NAME IF-6-16-b
EXPNO 1
PROCNO 1
Date_ 20081028
Time 9.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DN 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.0000000 sec
TDO 1

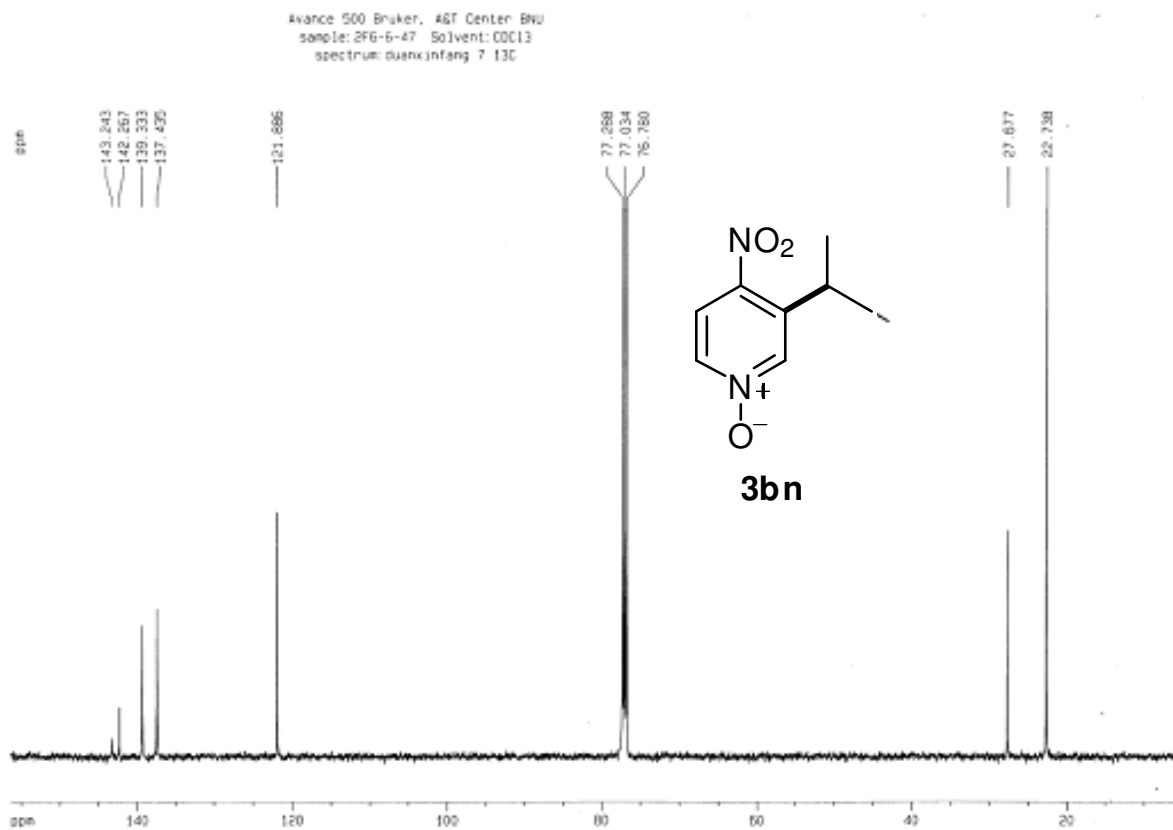
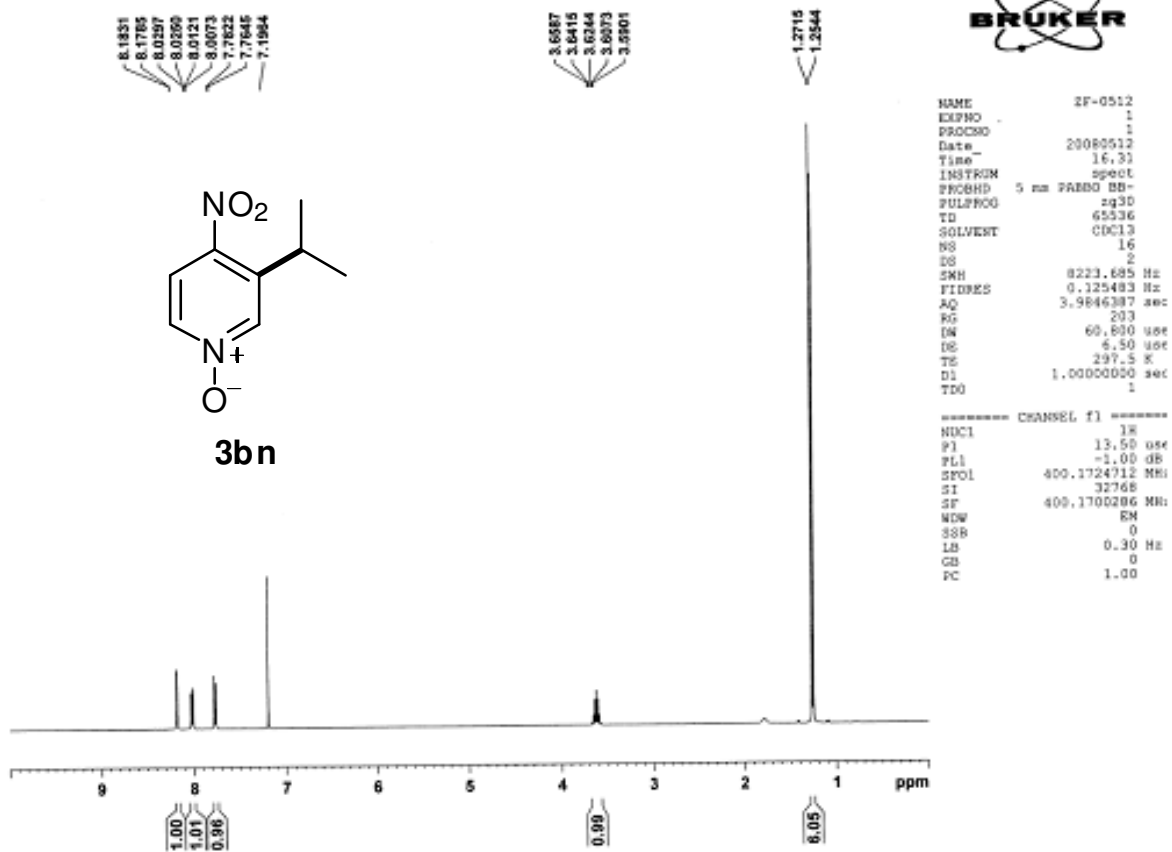
===== CHANNEL f1 =====
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18649796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1720000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

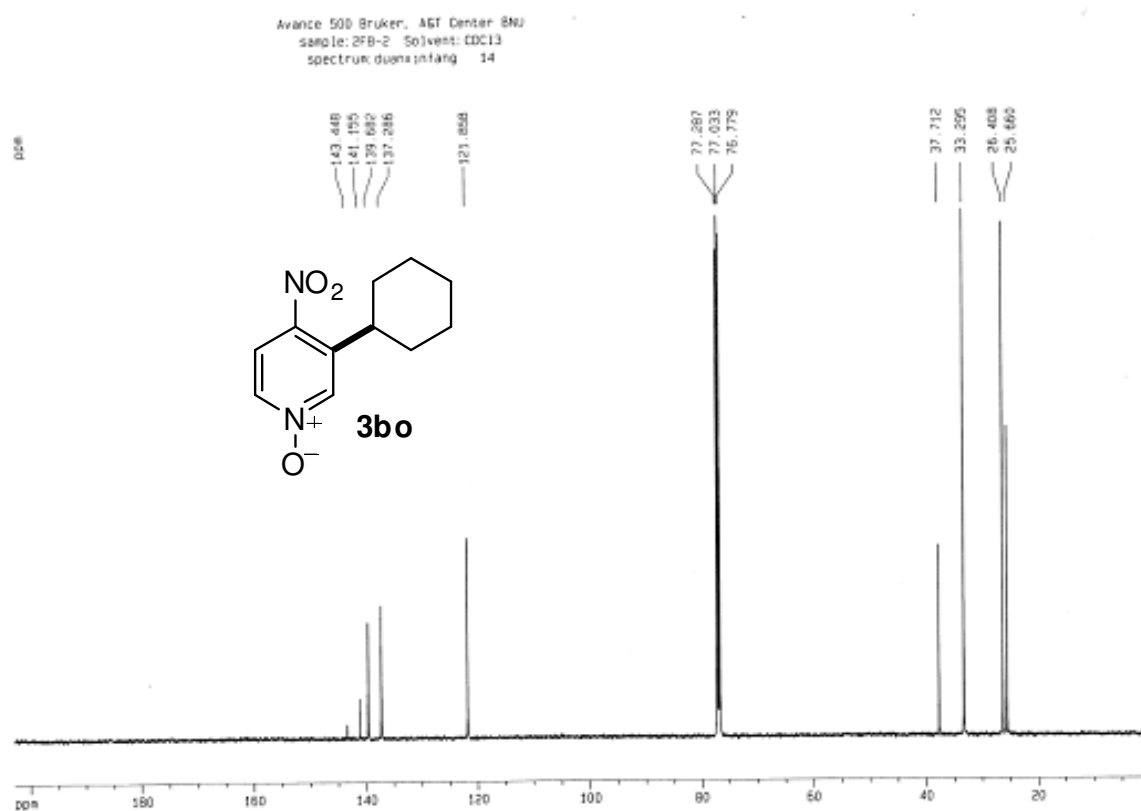
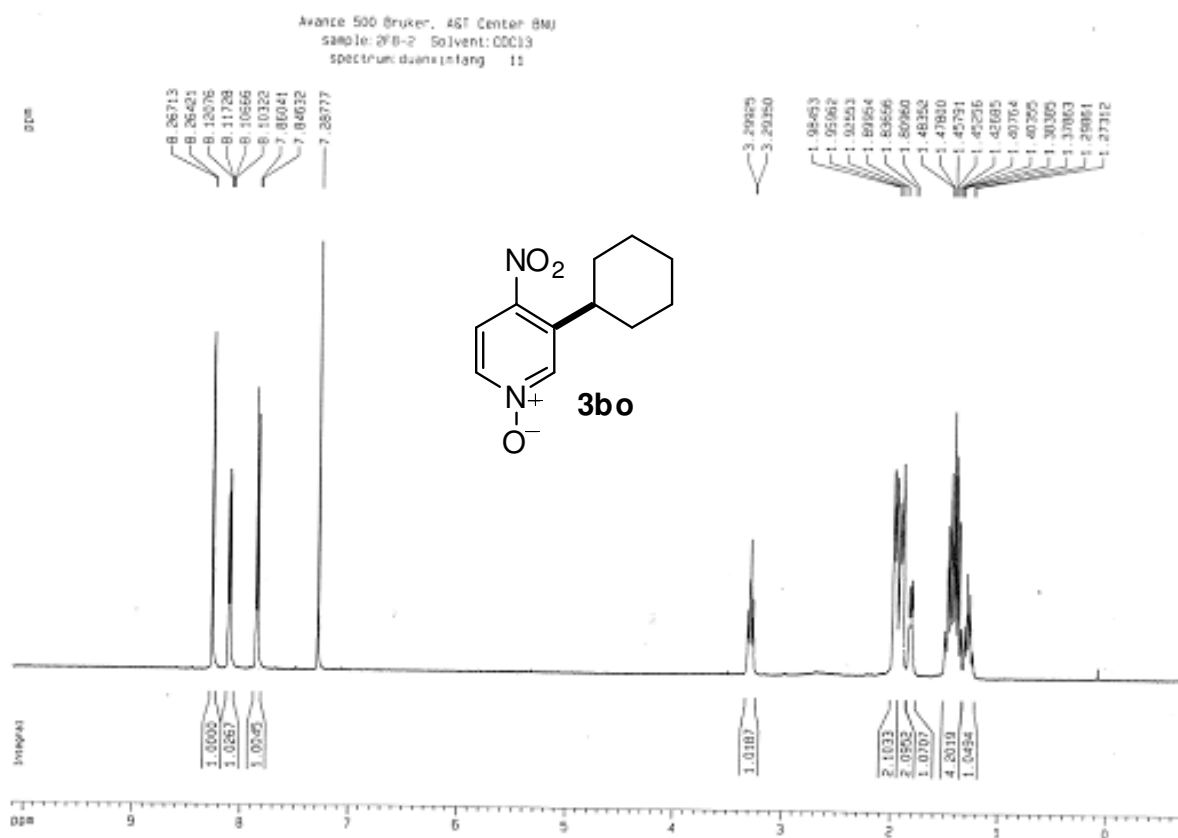


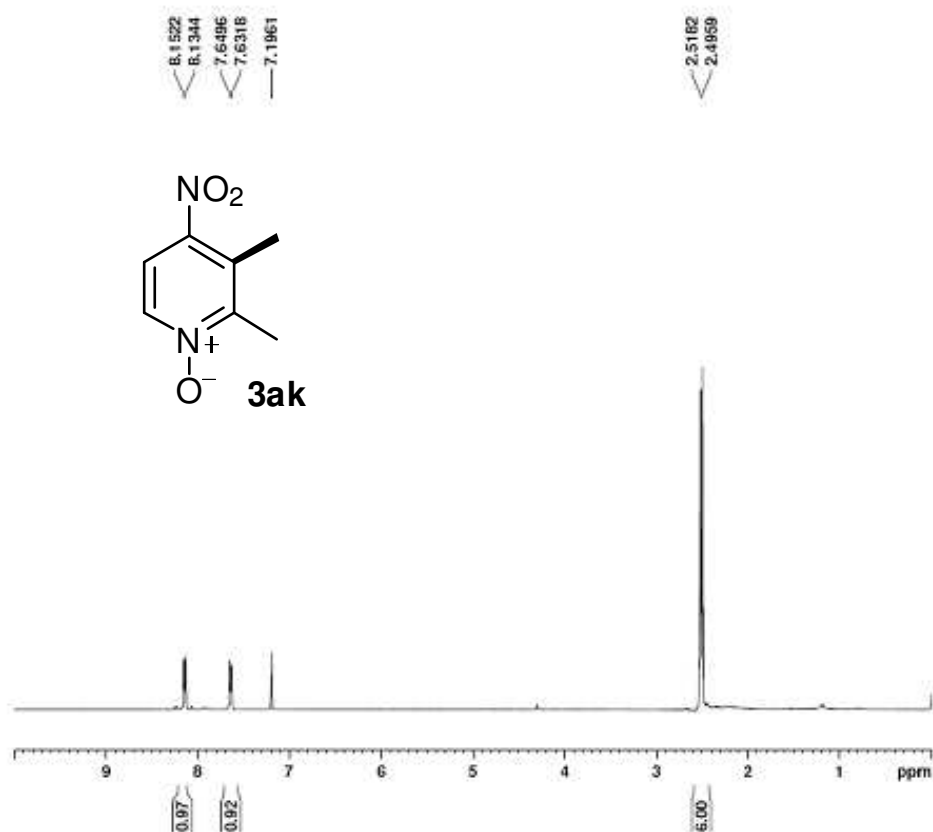
NAME IF6-16-b-Cl3
EXPNO 1
PROCNO 1
Date_ 20081029
Time 17.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DN 20.800 usec
DE 6.50 usec
TE 299.8 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 51.32743073 W
SFO1 100.6328888 MHz

===== CHANNEL f2 =====
CPCORRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL1W 13.18649796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716097 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





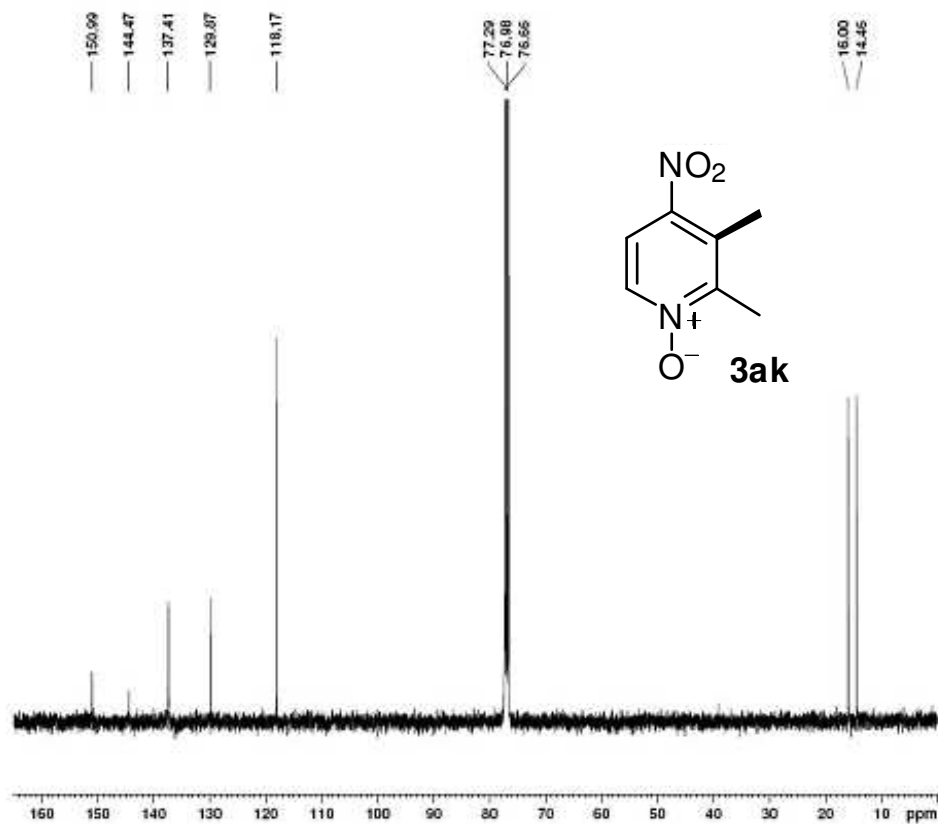


```

NAME      ZF9-9
EXPNO     1
PROCNO    1
Date_     20090625
Time      9.47
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         203
DM         60.800 usec
DE         6.50 usec
TE         298.2 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         14.20 usec
PL1        -1.00 dB
PL1W       13.16669796 W
SFO1      400.1724712 MHz
SI         32768
SF         400.1700284 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB          0
PC          1.00
  
```



```

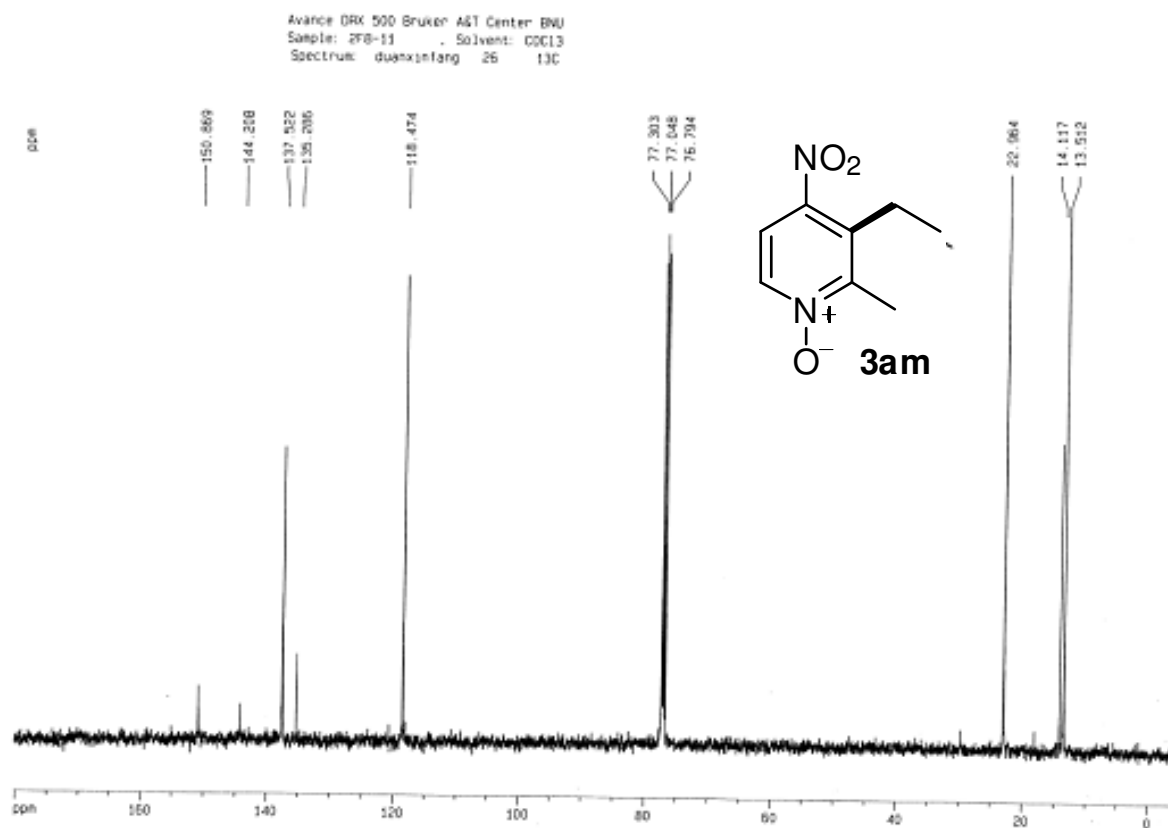
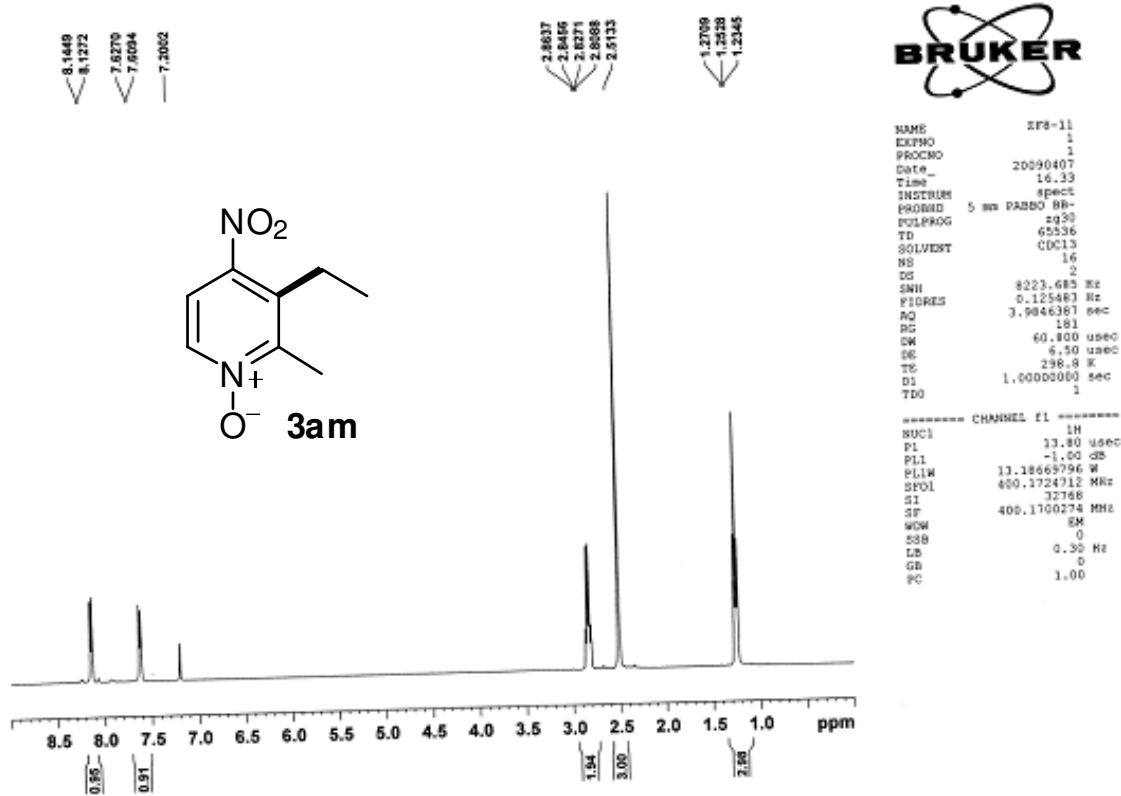
NAME      ZF9-S-C13
EXPNO     1
PROCNO    1
Date_     20090625
Time      16.38
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         2337
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
DM         20.800 usec
DE         6.50 usec
TE         302.4 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         8.50 usec
PL1        -1.00 dB
PL1W       57.32743073 W
SFO1      100.6228888 MHz
  
```

```

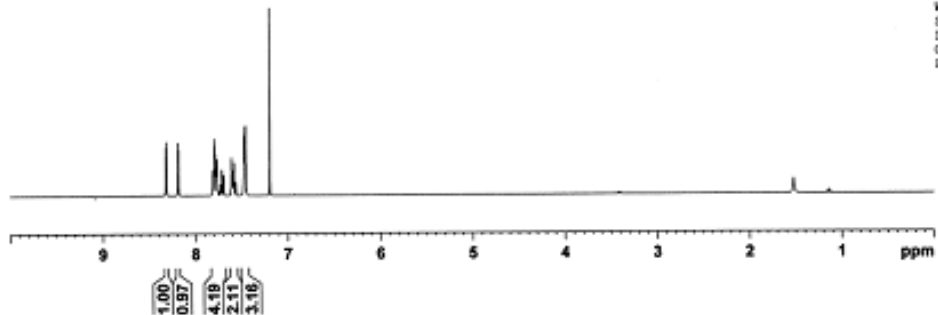
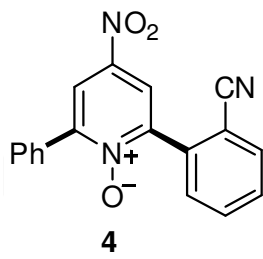
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        -1.00 dB
PL12       14.02 dB
PL13       14.46 dB
PL1W       13.16669796 W
PL12W      0.41508400 W
PL13W      0.37505048 W
SFO2      400.1716007 MHz
SI         32768
SF         100.6228270 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB          0
PC          1.40
  
```



7.8023
7.7925
7.7920
7.7838
7.7800
7.7745
7.7684
7.7620
7.7588
7.7357
7.7326
7.7162
7.7133
7.6871
7.6840
7.6609
7.6005
7.5877
7.5905
7.5890
7.5815
7.5787
7.5623
7.5584
7.4725
7.4632
7.4560
7.1944



NAME 2F6-29-2-2
EXPNO 1
PROCNO 1
Date_ 20081222
Time 9.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
SW 40.800 usec
DE 6.50 usec
TE 285.6 K
D1 1.00000000 sec
TD0 1

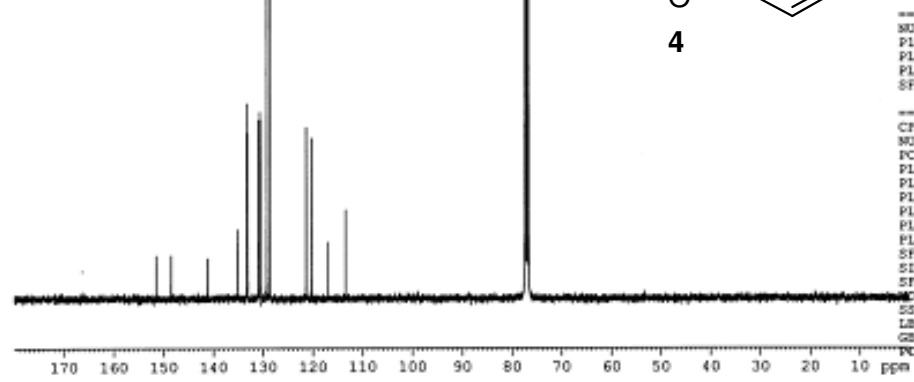
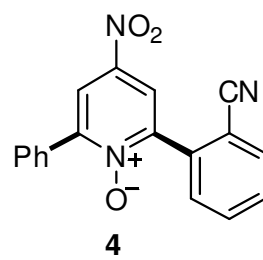


===== CHANNEL f1 =====
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700293 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

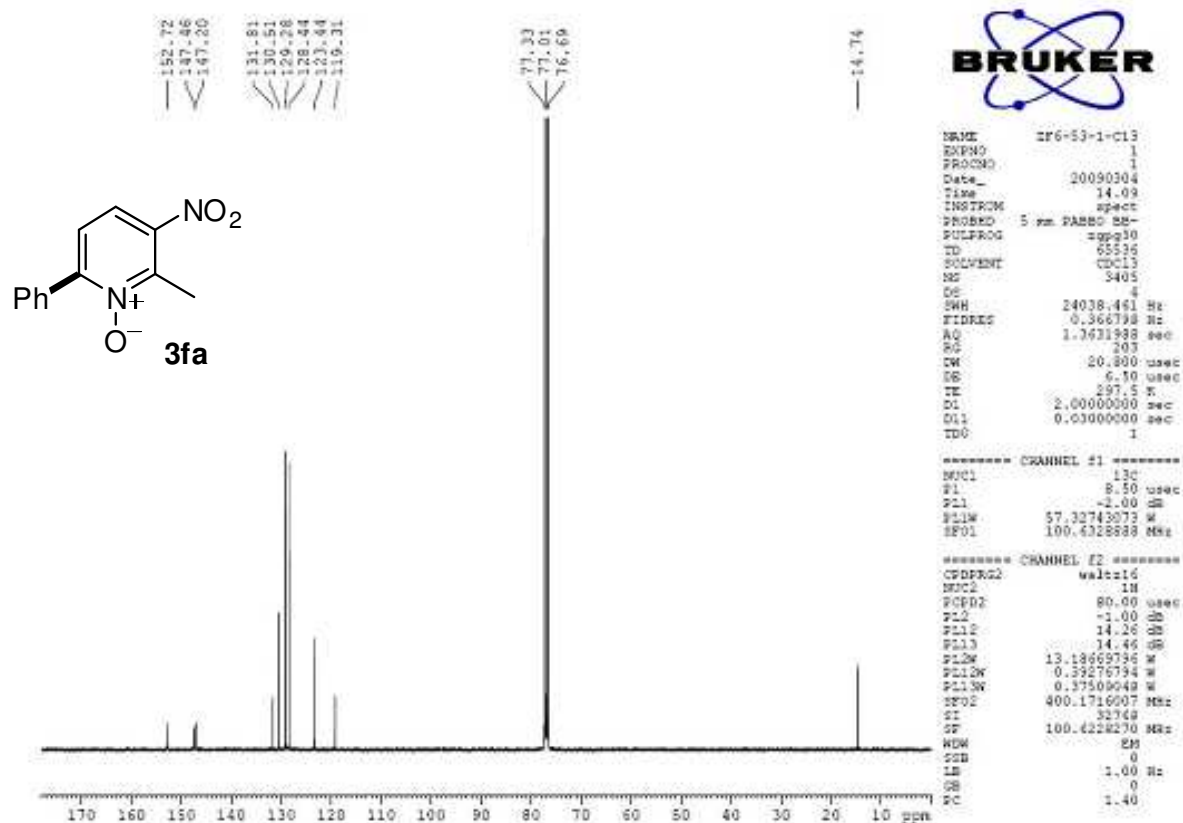
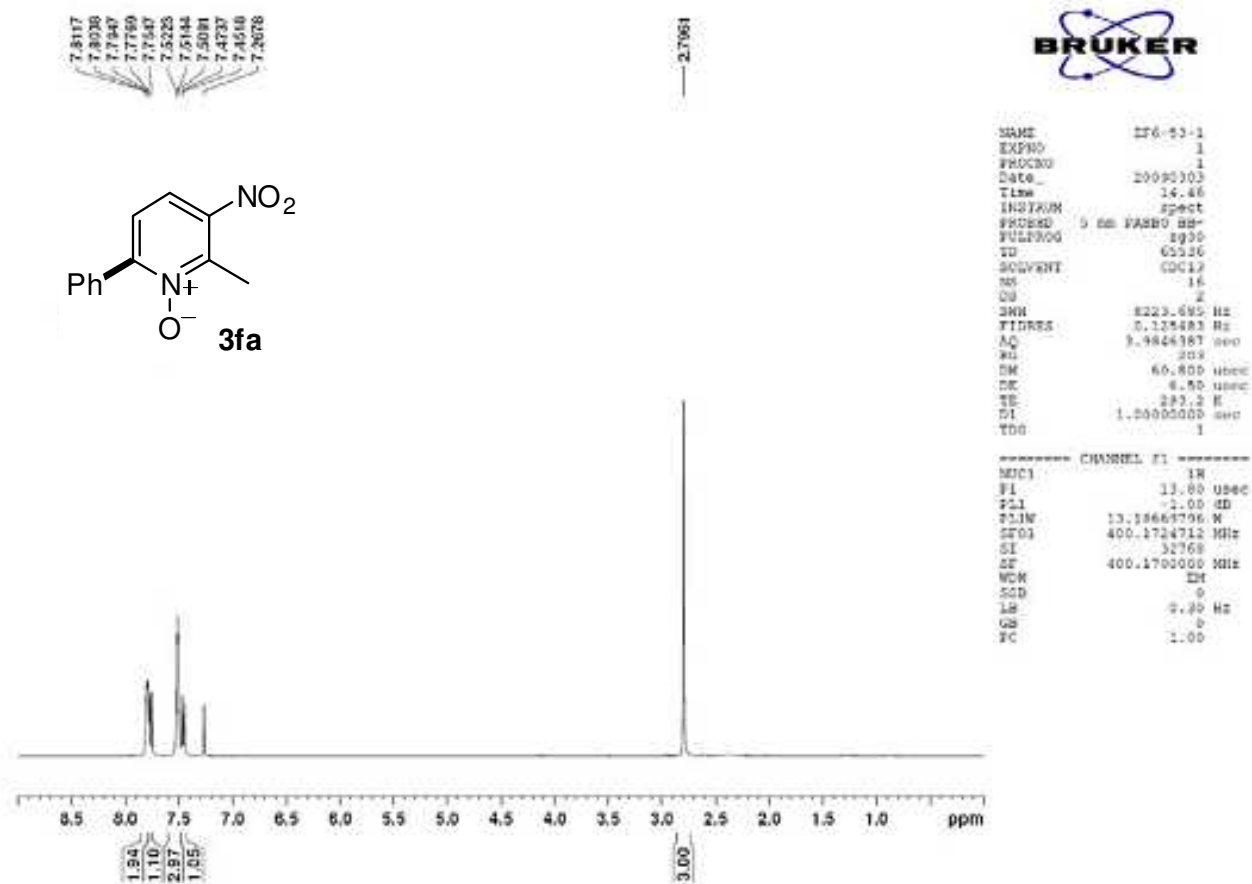
151.43
148.48
141.15
135.19
133.24
133.06
130.87
130.82
130.63
130.52
129.28
128.64
121.33
120.25
117.00
113.39



NAME 2F6-29-2-2-C13
EXPNO 1
PROCNO 1
Date_ 20081222
Time 17.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8000
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
SW 20.800 usec
DE 6.50 usec
TE 290.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1



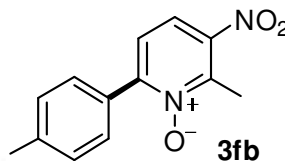
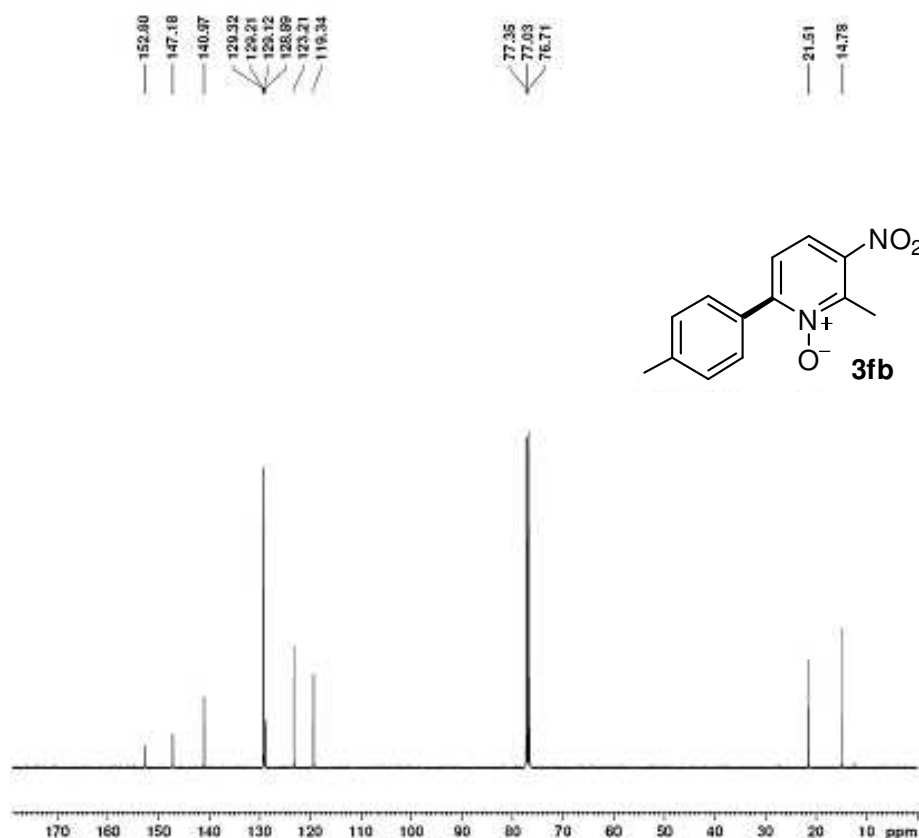
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6328888 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6328270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





NAME	276-18
EXPNO	2
PROCNO	1
DATE_	20090413
TIME	11.02
INSTRUM	spect
PROBHD	5 mm VARIO RD-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
H2	16
D5	2
SWH	8221.695 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	182
PC	65.850 usec
SC	6.55 usec
TE	296.7 K
D1	1.80000000 sec
TD0	

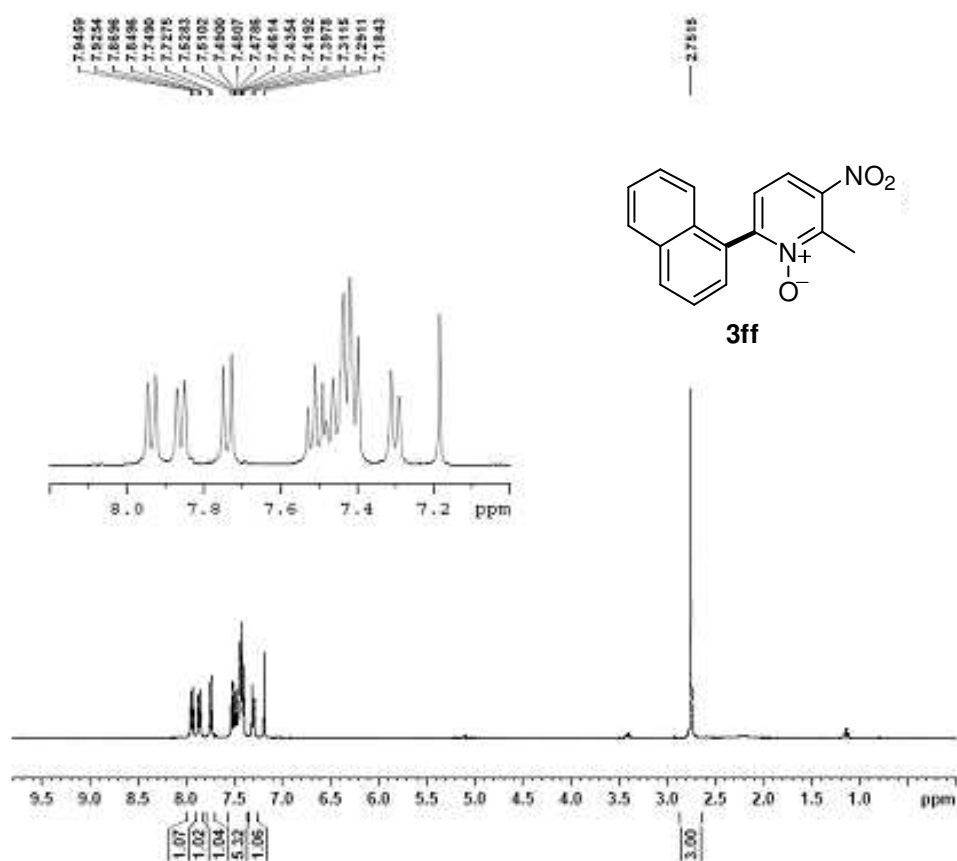
```
***** CHANNEL #1 *****
NUC1      IM
P1         13.85 usec
P2         -1.00 dB
PLIN       13.28649794 W
SF01       400.1724712 MHz
SI         32768
SF         400.1730000 MHz
MCW        dBm
DBB         0
LB         0.70 Hz
GD         0
PC         1.00
```



```
NAME      ZPG-18-113
EXPNO     1
PROCNO    1
Date_     20090416
Time      7.47
INSTRUM    spect
PROCESSED 5 mm PABBO BB-
PULPROG    zgpg30
TD          43536
SOLVENT    CDC113
NS          2136
DS          4
SWH         24038.461 Hz
FIDRES     0.345799 Hz
AQ          1.3631988 sec
RG          203
DW          20.800 nsec
DE          4.50 nsec
TE         300.5 K
D1         2.00000000 nsec
D11        0.03000000 nsec
D20        1
```

```
***** CHANNEL f1 *****
MUC1          100
F1             8.50 used
PL1            -2.00 dB
PL1W          37.32743073 W
SFO1          100.6328888 MHz
```

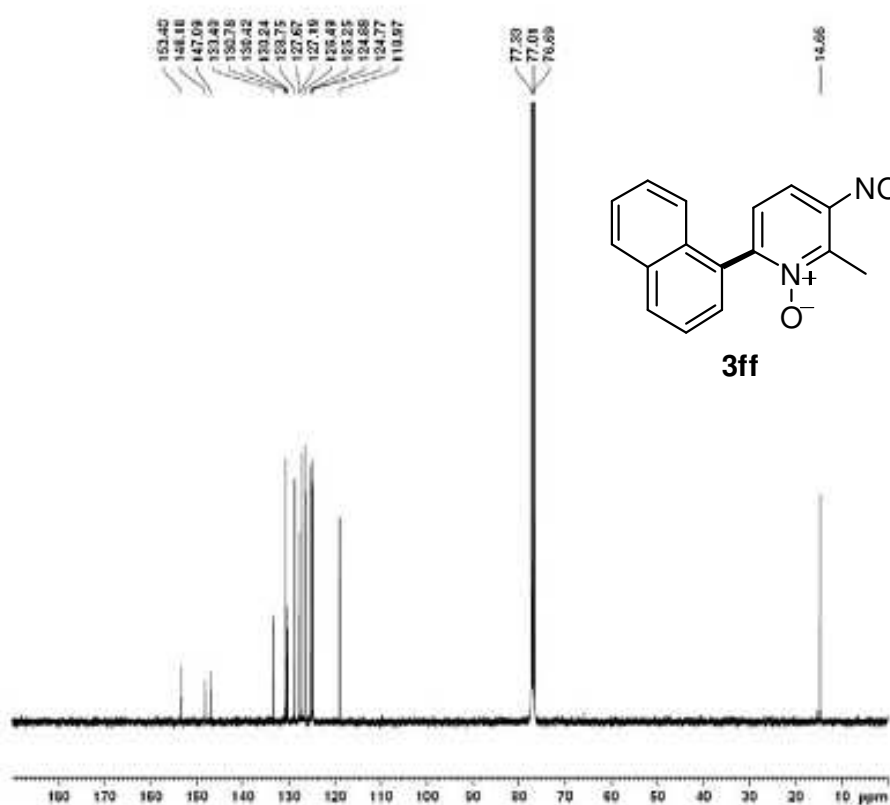
```
***** CHANNEL 12 *****
CFDD962      wait=16
NUG2         1H
PCFD2        80.00 usec
PL2          -1.00 usec
PL12         14.26 usec
PL13         14.86 usec
PL2W         13.18563794 W
PL12W        0.39248794 W
PL13W        0.37503048 W
SF02         400.1715007 MHz
SI           32768
BF           100.6228270 MHz
W2W         MHz
SSB          0
B            1.00 Hz
GB           0
FC           1.40
```



BRUKER

```

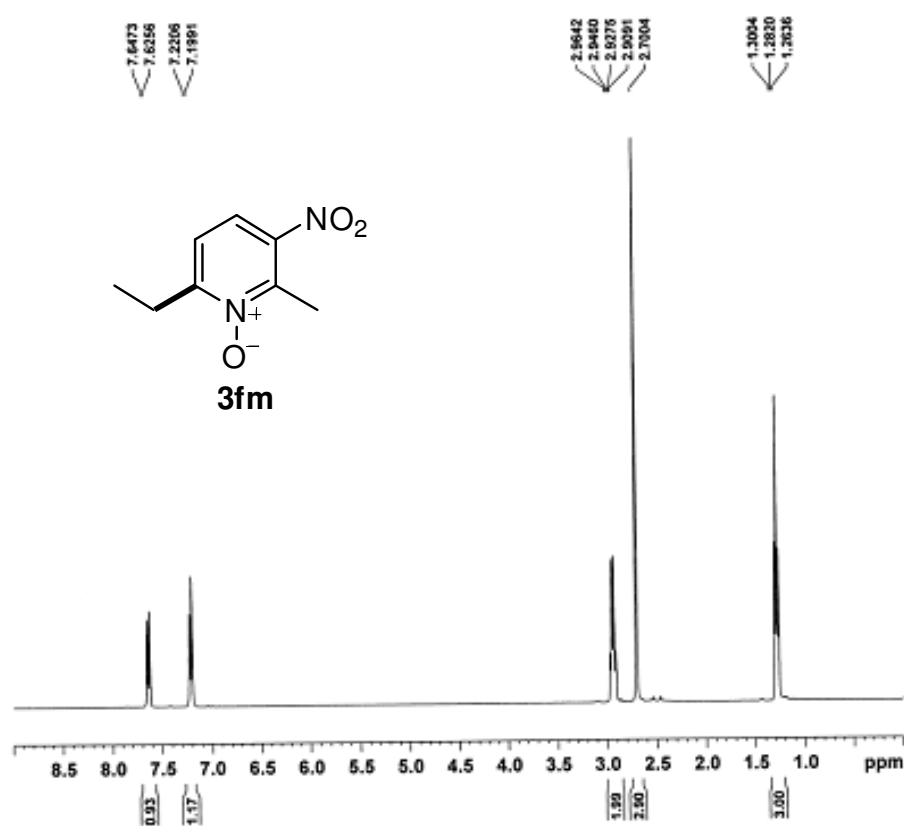
NAME      2F8-23
EXPNO     1
PROCNO    1
Date_     20090415
Time      17.09
INSTRUM    spect
PROBHD     5 mm BBO BB-
PULPROG    zgpg30
TD          65536
SOLVENT    CDCl3
NS          16
DS          2
SWH         8227.686 Hz
FIDRES     0.129483 Hz
AQ          3.9946387 sec
RG          203
DN          40.608 usec
DE          6.58 usec
TE          290.2 K
D1          1.0000000 sec
D11         1
D12         1
===== CHANNEL f1 =====
NUC1        13C
P1          13.00 usec
PL1         -1.00 dB
PL12        13.18469796 MHz
SFO1        400.1724712 MHz
SI          32768
SF          400.1700332 MHz
WDW         EM
SSB          1
LB           0.10 Hz
GB           0
PC           1.00
  
```



BRUKER

```

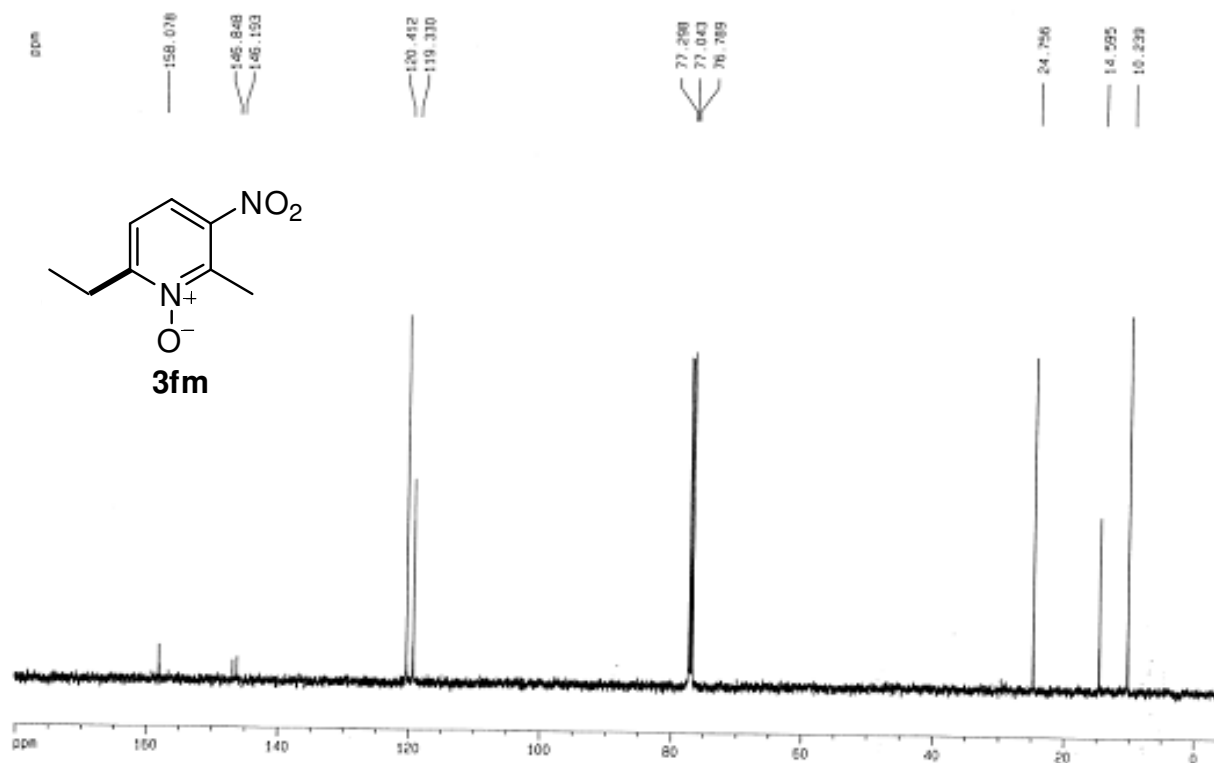
NAME      2F8-23-13
EXPNO     1
PROCNO    1
Date_     20090415
Time      8.57
INSTRUM    spect
PROBHD     5 mm BBO BB-
PULPROG    zgpg30
TD          65536
SOLVENT    CDCl3
NS          4000
DS          4
SWH         24039.461 Hz
FIDRES     0.344798 Hz
AQ          1.3671988 sec
RG          203
DN          30.800 usec
DE          6.50 usec
TE          303.0 K
D1          1.0000000 sec
D11         0.0000000 sec
D12         1
===== CHANNEL #1 =====
NUC1        13C
P1          9.50 usec
PL1         -2.00 dB
PL12        57.33745373 MHz
SFO1        100.6288888 MHz
===== CHANNEL #2 =====
CPDPRG2    waltz16
NUC2        1H
P2          80.00 usec
PL2         -1.00 dB
PL12        14.24 dB
PL13        14.40 dB
PL12N       13.18469796 MHz
PL13N       0.137576794 MHz
PL13W       0.137576794 MHz
SFO2        400.1716007 MHz
SI          32768
SF          100.6228270 MHz
WDW         EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```

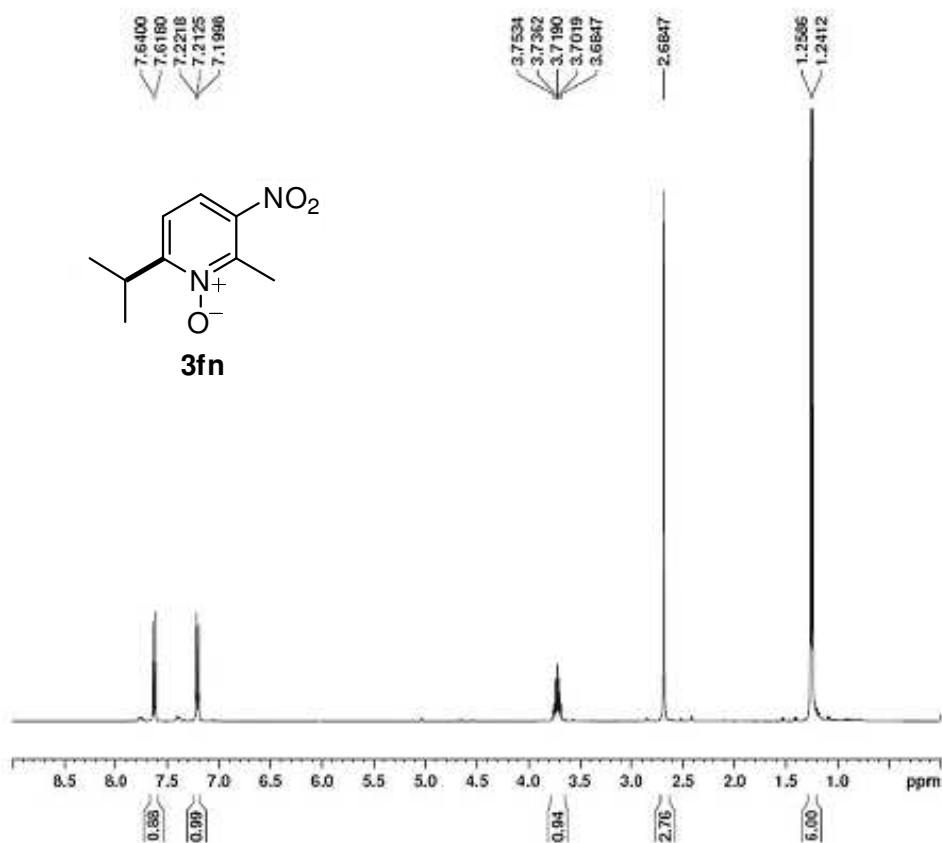


NAME 2FB-9-1
EXPNO 1
PROCNO 1
Date_ 20090401
Time 16.13
INSTRUM spect
PROBHD 5 mm PARRO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DM 60.800 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.184669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700275 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Avance DFX 500 Bruker AET Center BMJ
Sample: 2FB-9-1 Solvent: CDCl3
Spectrum: duanxinrong 24 13C



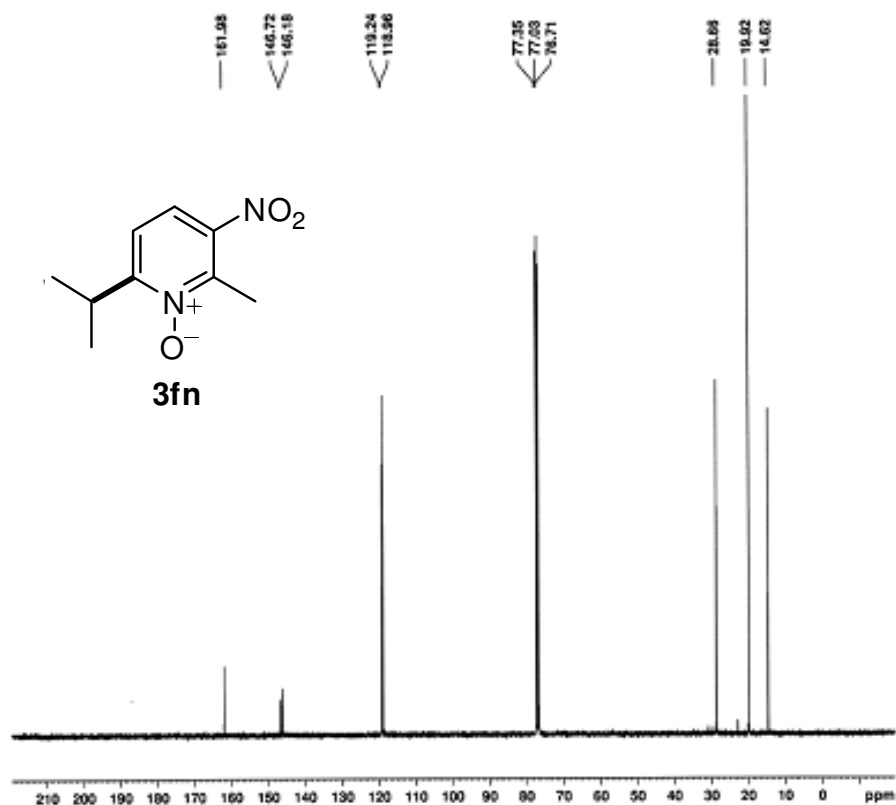


```

NAME      ZFS-28
EXPNO     1
PROCNO    1
Date_     20090413
Time      9.50
INSTRUM    spect
PROBHD     5 mm FAREO BB-
PULPROG    zg30
TD         65536
SOLVENT    CDCl3
NS         16
DS         2
SWH         8223.695 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         101
OW         60.800 usec
DE         6.50 usec
TE         296.4 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SF01       400.1724712 MHz
SI         32768
SF         400.1700221 MHz
WDW         EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```



```

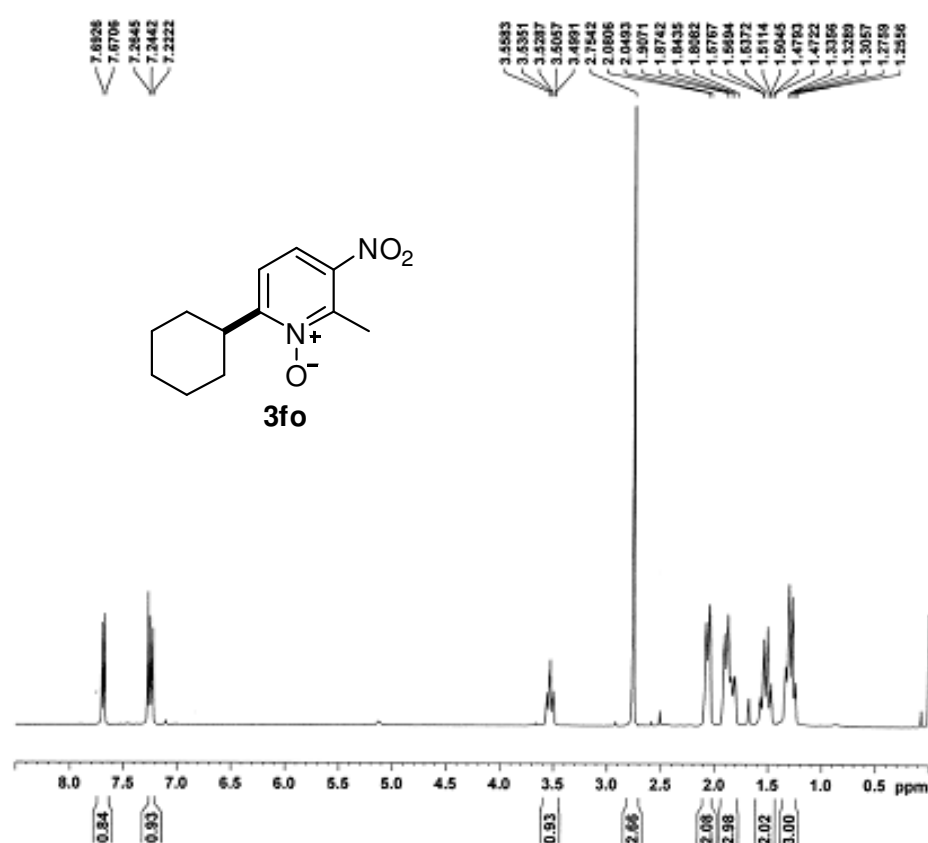
NAME      ZFS-28-C13
EXPNO     1
PROCNO    1
Date_     20090413
Time      9.09
INSTRUM    spect
PROBHD     5 mm FAREO BB-
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS         638
DS         4
SWH         24038.441 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
OW         26.800 usec
DE         6.50 usec
TE         299.5 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         8.50 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SF01       100.628888 MHz
  
```

```

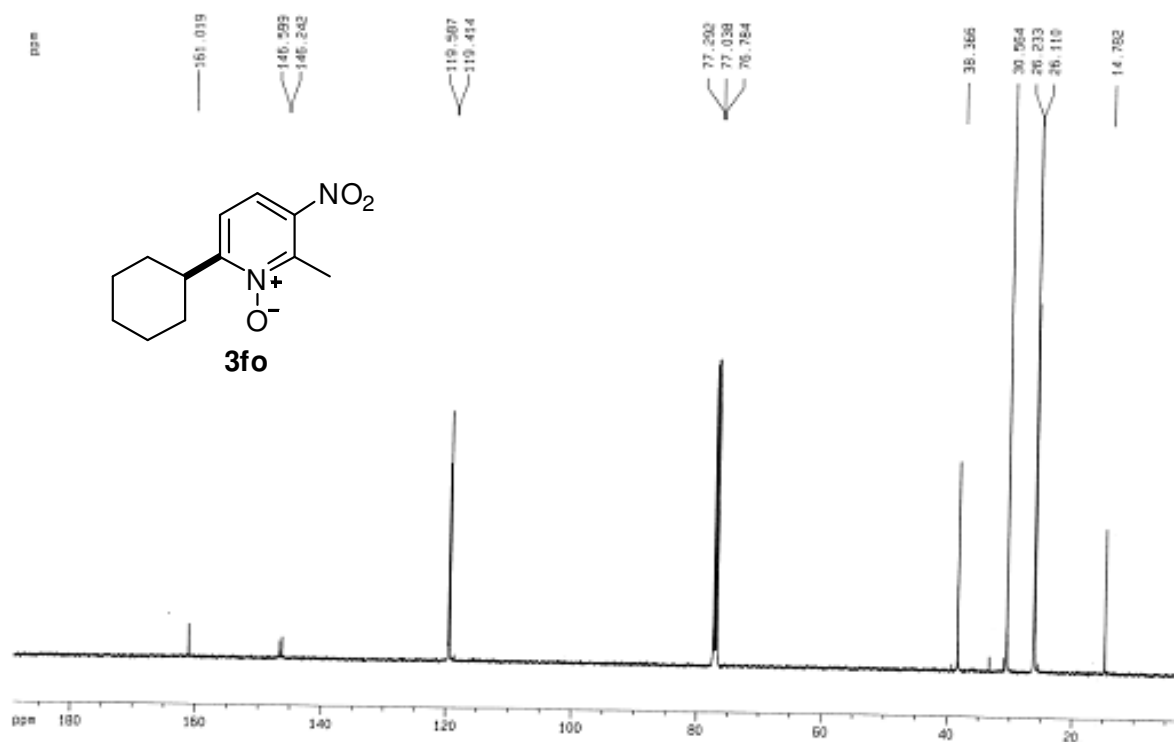
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -1.00 dB
PL12       14.26 dB
PL13       14.46 dB
PL1W       13.18669796 W
PL12W      0.39276794 W
PL13W      0.37509048 W
SF02       400.1716001 MHz
SI         32768
SF         100.6288278 MHz
WDW         RM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

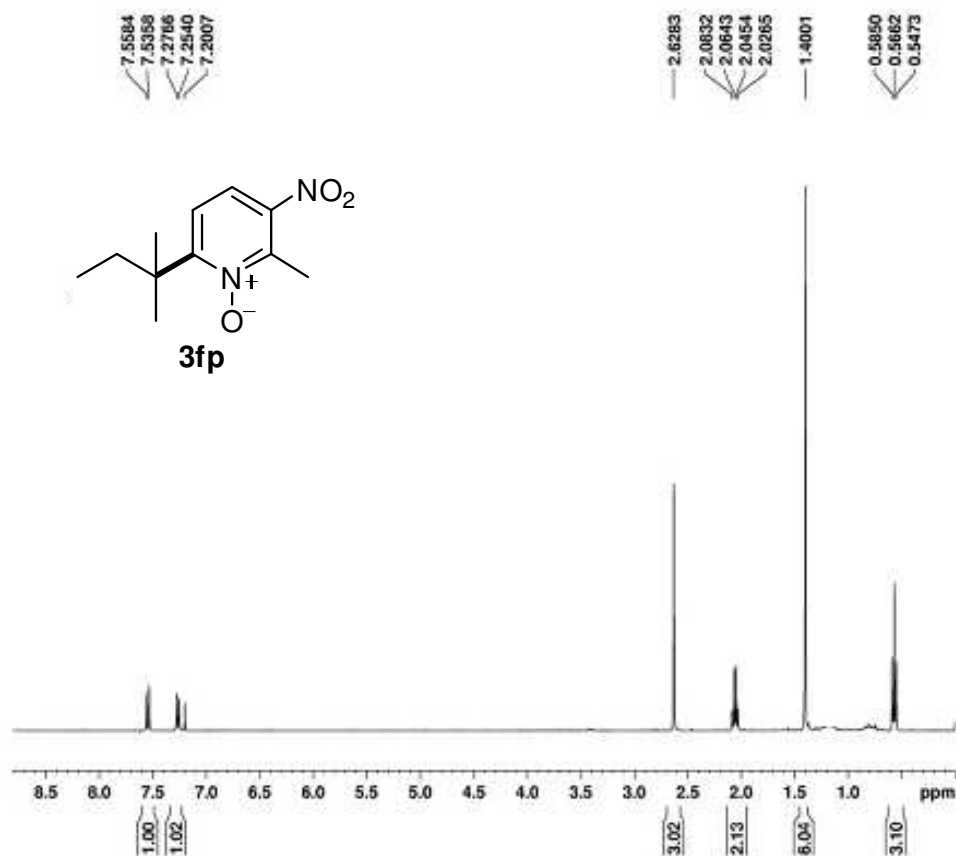


NAME 2FG-65
 EXPNO 1
 PROCNO 1
 Date_ 20090630
 Time 9.18
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9946387 sec
 RG 203
 IN 40.300 usec
 DE 6.50 usec
 TE 300.0 K
 DI 1.00000000 sec
 TDO 1

----- CHANNEL f1 -----
 NUC1 1H
 P1 14.20 usec
 PL1 -1.00 dB
 PL1W 13.18663796 W
 SPOL 400.1724712 MHz
 SI 32768
 SF 400.1700000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

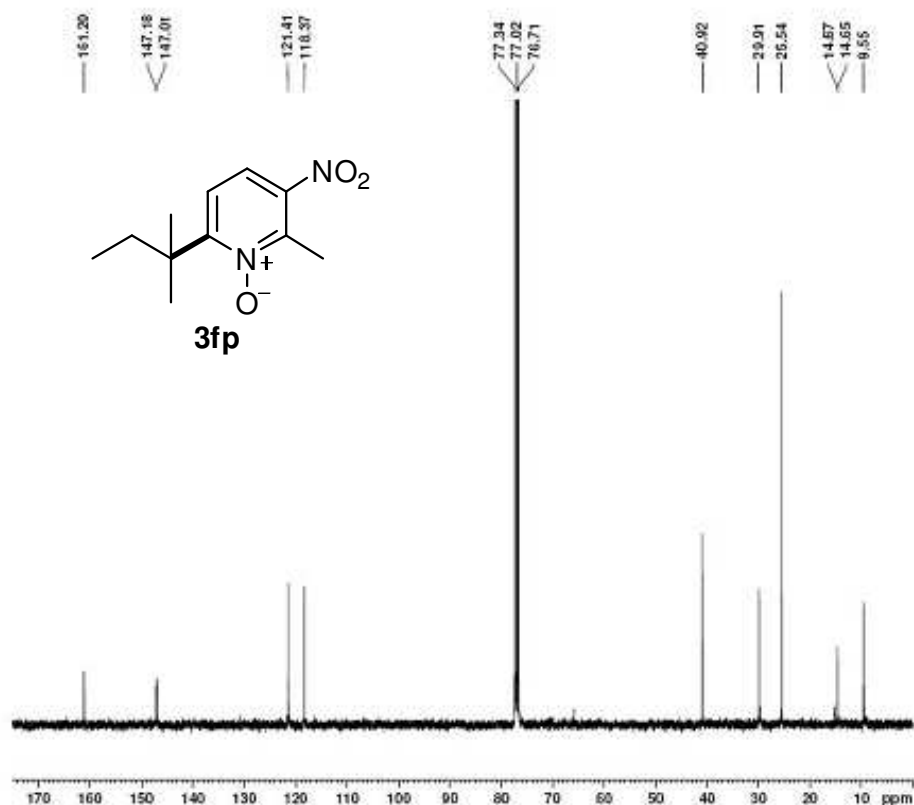
Avance DRX 500 Bruker A&T Center (RM)
 Sample: 2FG-65-1 Solvent: CDCl3
 Spectrum: 13C





NAME: SF10-42
EXPNO: 1
PROCNO: 1
Date_: 20091217
Time: 10.43
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zg30
TD: 65536
SOLVENT: CDCl₃
NS: 16
DS: 2
SWH: 8223.685 Hz
FIDRES: 0.125483 Hz
AQ: 3.9846397 sec
RG: 181
DN: 60.800 usec
DE: 6.50 usec
TE: 292.6 K
D1: 1.00000000 sec
TD0: 1

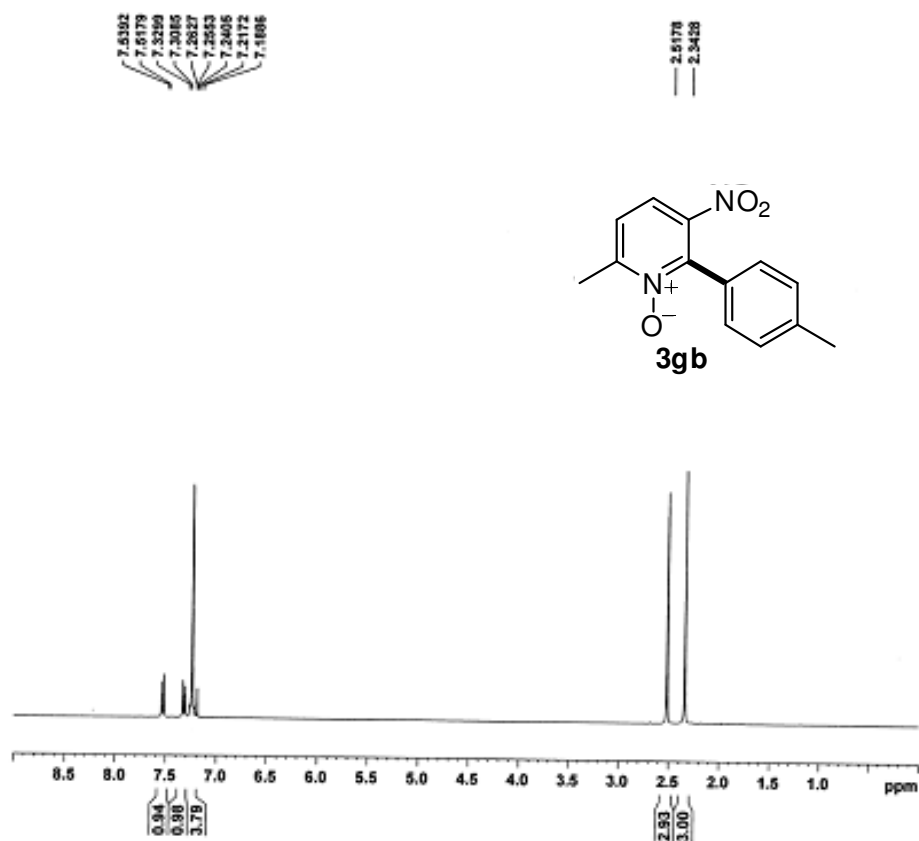
----- CHANNEL f1 -----
NUC1: ¹H
P1: 14.20 usec
PL1: -1.00 dB
PL1W: 13.18669794 W
SFO1: 400.1724712 MHz
SI: 32768
SF: 400.1700267 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00



NAME: 2F10-42-013
EXPNO: 1
PROCNO: 1
Date_: 20091217
Time: 9.07
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl₃
NS: 695
DS: 0
SWH: 24039.40 Hz
FIDRES: 0.366798 Hz
AQ: 1.3631888 sec
RG: 203
DN: 20.900 usec
DE: 6.50 usec
TE: 292.6 K
D1: 2.00000000 sec
D11: 0.03000000 sec
TD0: 1

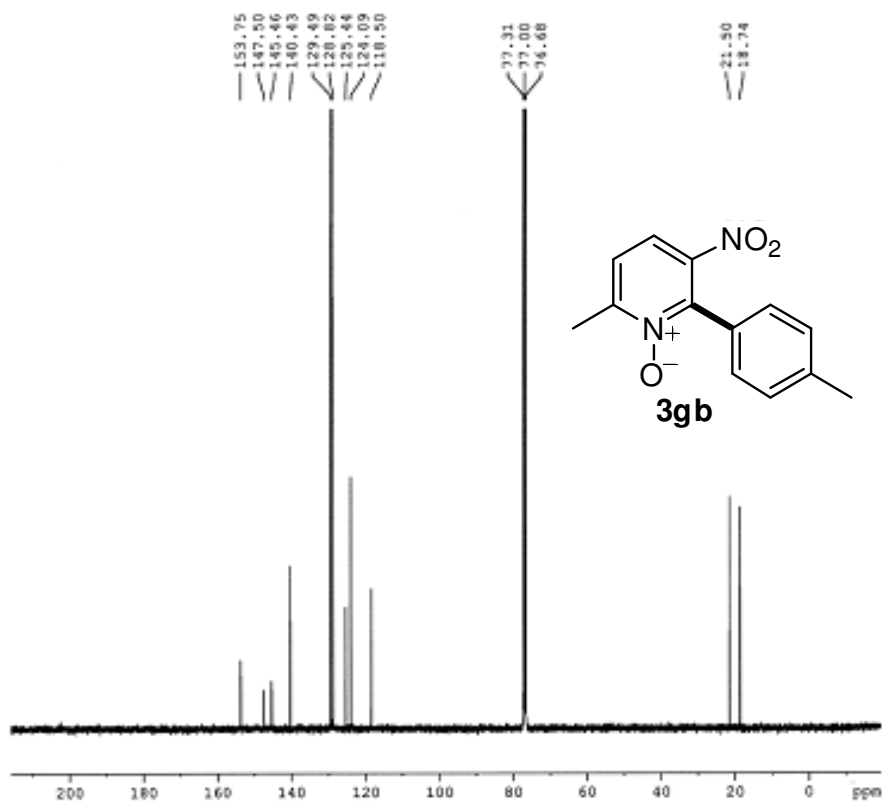
----- CHANNEL f1 -----
NUC1: ¹³C
P1: 8.10 usec
PL1: -2.00 dB
PL1W: 57.32147073 W
SFO1: 100.6328998 MHz

----- CHANNEL f2 -----
CPOBPG2: waltz16
NUC2: ¹H
PCPD2: 80.00 usec
PL2: -1.00 dB
PL12: 14.02 dB
PL13: 14.46 dB
PL1W: 13.18669794 W
PL12W: 6.41689400 W
PL13W: 6.37503048 W
SFO2: 400.176007 MHz
SI: 32768
SF: 100.6228270 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40



NAME SFB-12
EXPNO 1
PROCNO 1
Date_ 20090410
Time 9.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846287 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1

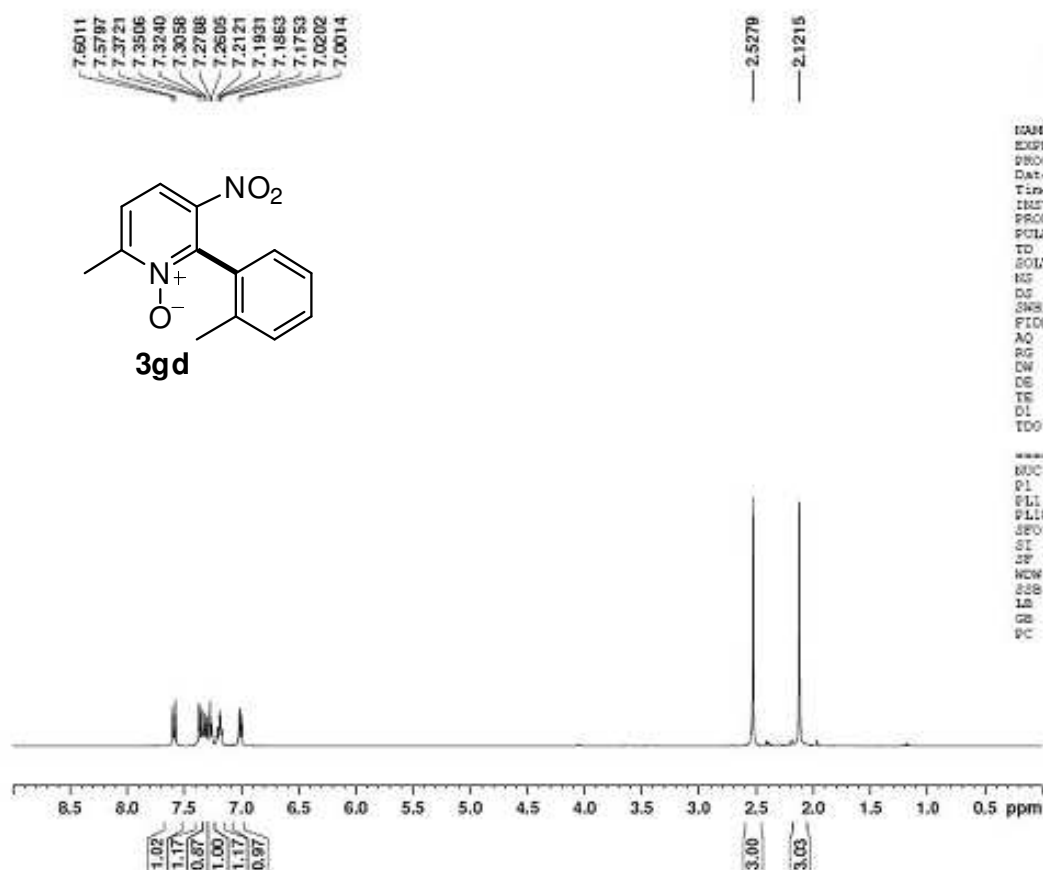
===== CHANNEL f1 =====
NUC1 1H
P1 13.80 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700316 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



NAME SFB-12-Cl3
EXPNO 1
PROCNO 1
Date_ 20090413
Time 11.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2856
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32743873 W
SFO1 100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
MPC2 1W
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 14.46 dB
PL2W 13.18669796 W
PL12W 0.36276794 W
PL13W 0.37509048 W
SFO2 400.1716007 MHz
SI 32768
SF 100.6228270 MHz
WDW RM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

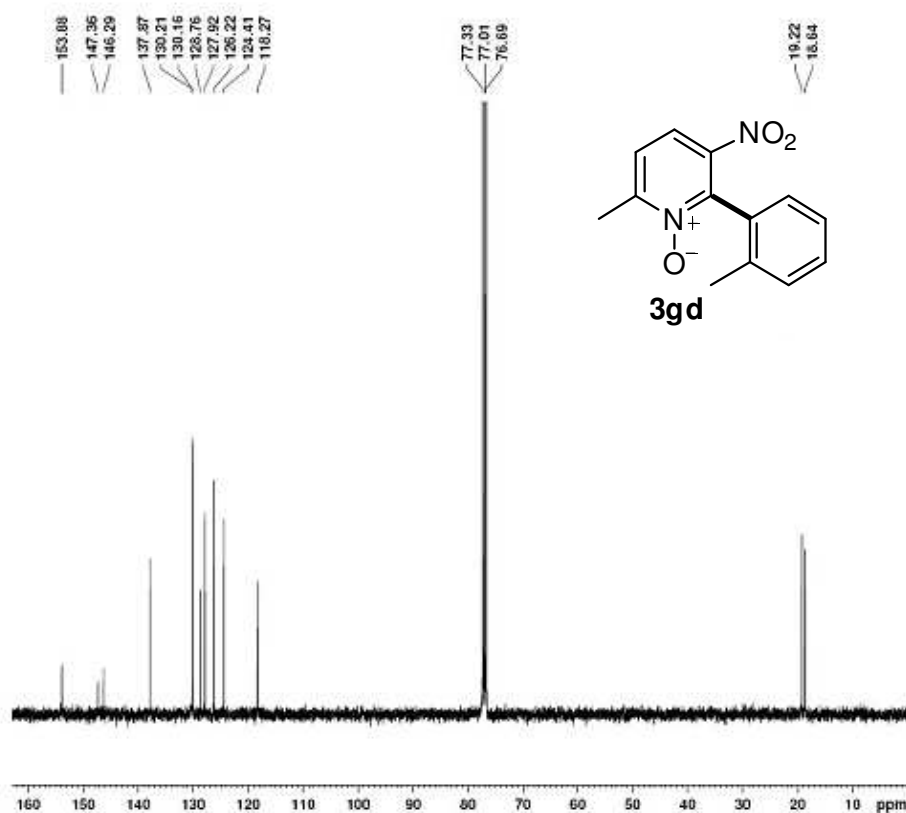


```

NAME      ZF8-46-2
EXPNO     1
PROCNO    1
Date_     20090430
Time      14.32
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125493 Hz
AQ         3.9946387 sec
RG         161
CW         60.900 usec
DE         6.50 usec
TE         300.4 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         13.80 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1700326 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



```

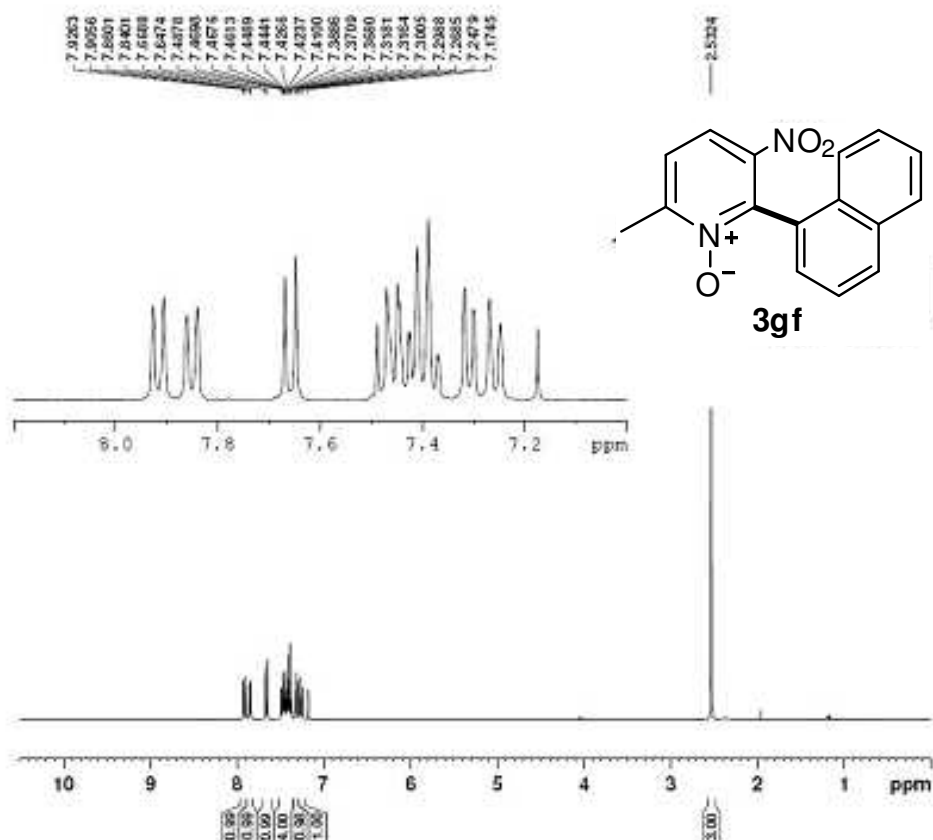
NAME      ZF8-46-2-C13
EXPNO     1
PROCNO    1
Date_     20090501
Time      16.44
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         425
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
CW         20.800 usec
DE         6.50 usec
TE         301.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         8.60 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1       100.6328888 MHz
  
```

```

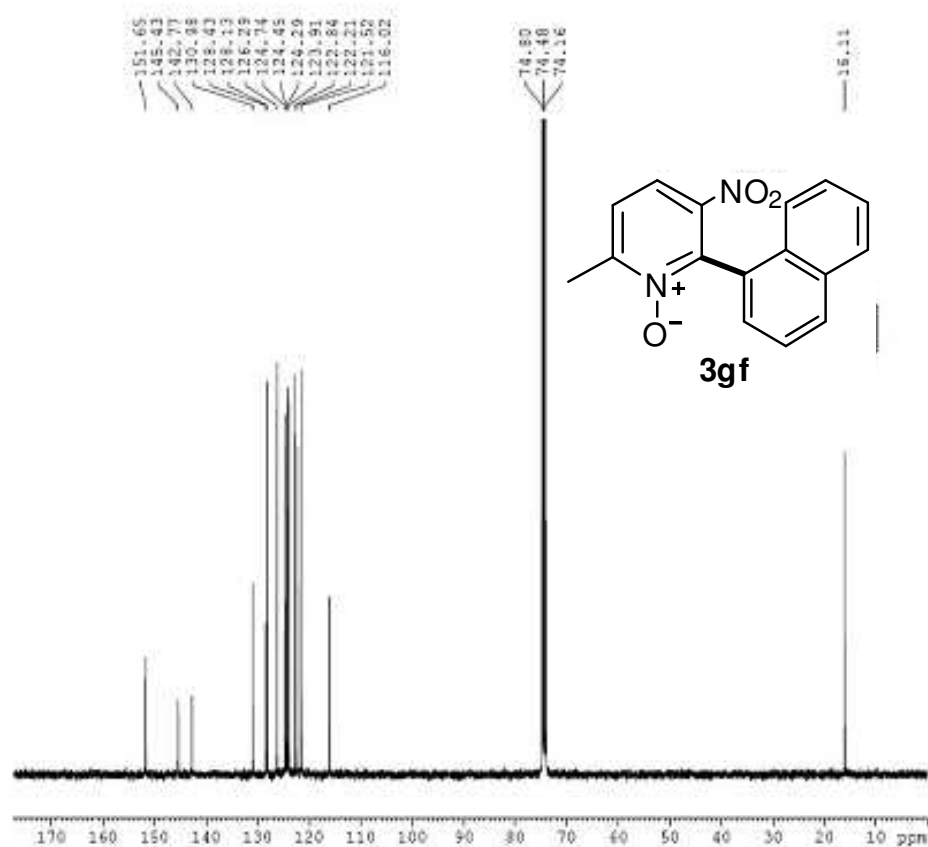
===== CHANNEL f2 =====
CFDPRG2    waltz16
NUC2       1H
PCPD1      80.00 usec
PL2        -1.00 dB
PL12       14.26 dB
PL13       14.46 dB
PL2W       13.18669796 W
PL12W      0.39276794 W
PL13W      0.37509048 W
SFO2       400.1716007 MHz
SI         32768
SF         100.6228270 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```



```

NAME          ZF8-25
EXPNO         1
PROCNO        1
Date_         20090414
Time          9.28
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       DMSO-d6
NS            16
DS            2
SWH           9223.695 Hz
FIDRES        0.125493 Hz
AQ            3.9846381 sec
RG            101
CW            60.500 usec
DE            6.50 usec
TE            297.3 K
D1            1.50000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1           1H
P1            13.80 usec
PL1           -1.00 dB
PL1W          13.18469796 W
SFO1          400.1124713 MHz
SI            32768
SF           400.1100372 MHz
WDW           EM
SSB           0
GB            0.50 Hz
PC            1.00
  
```

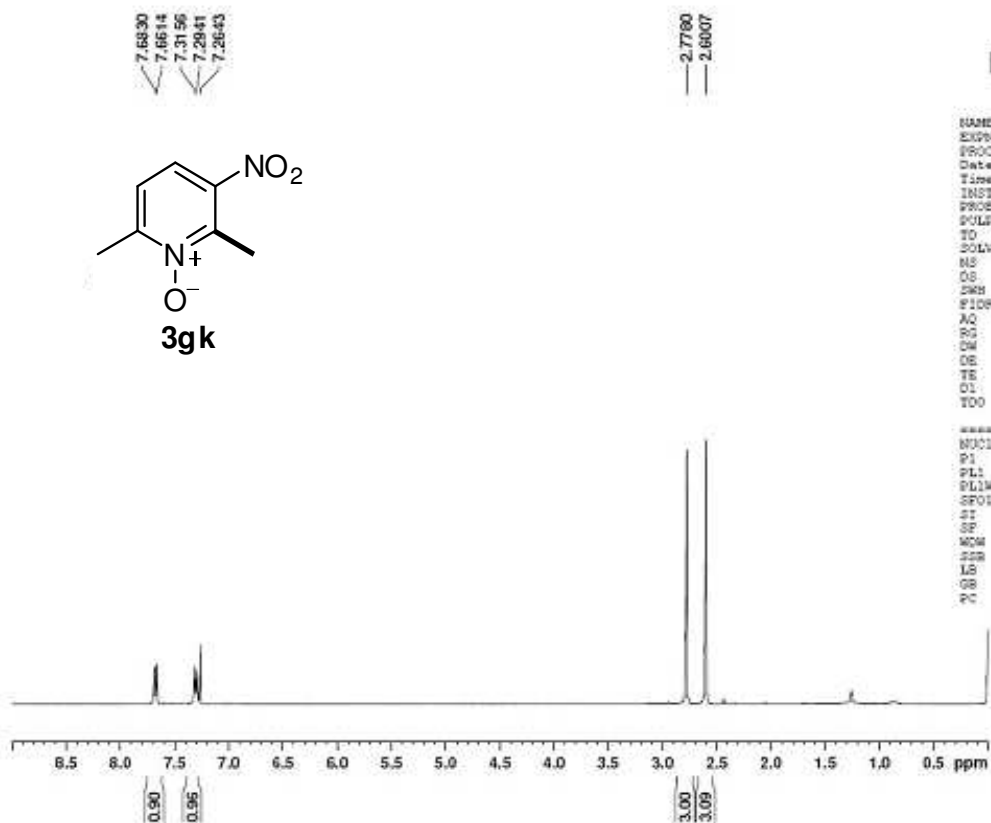


```

NAME          ZF8-25-CL13
EXPNO         1
PROCNO        1
Date_         20090414
Time          11.30
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            3297
DS            4
SWH           24039.441 Hz
FIDRES        0.366739 Hz
AQ            1.3431988 sec
RG            203
CW            20.800 usec
DE            6.50 usec
TE            299.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1           13C
P1            0.50 usec
PL1           -2.00 dB
PL1W          57.32743073 W
SFO1          100.6228888 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         80.00 usec
PL2           -1.00 dB
PL12          14.26 dB
PL13          14.46 dB
PL1W          13.18469796 W
PL12W         0.39276794 W
PL13W         0.37509048 W
SFO2          400.1716007 MHz
SI            32768
SF           100.6228270 MHz
WDW           EM
SSB           0
GB            1.00 Hz
PC            1.40
  
```

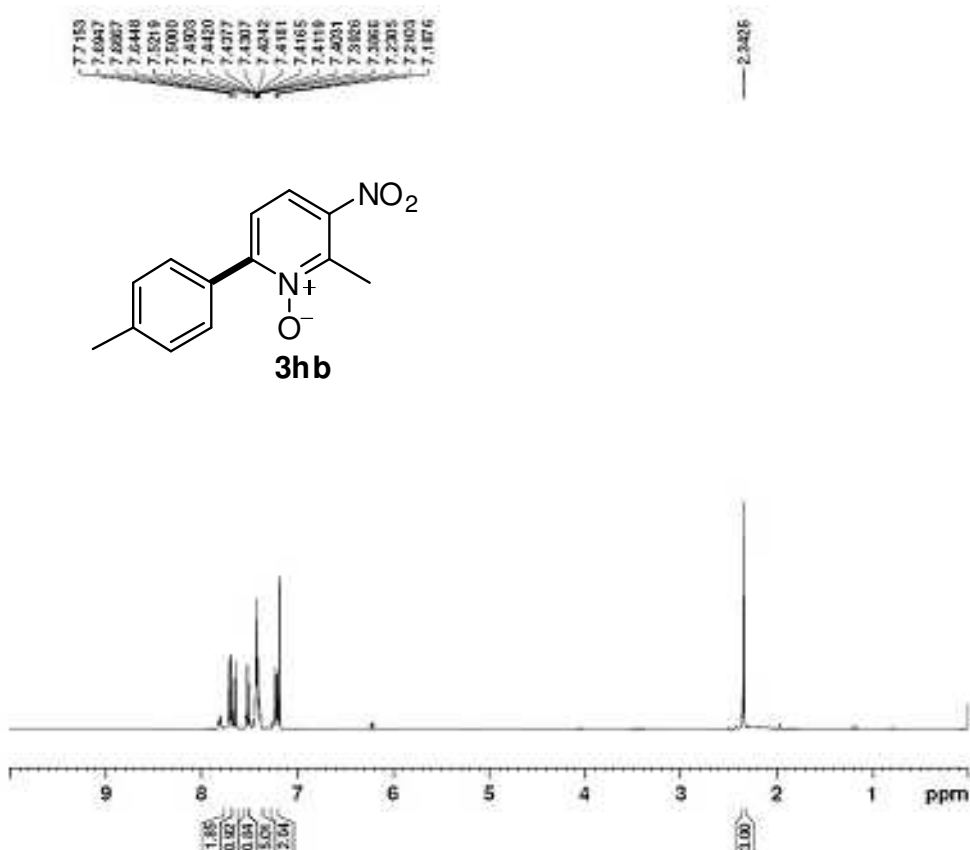



```

NAME      ZF9-15
EXPNO     1
PROCNO    1
Date_     20090701
Time      14.41
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zg30
TD         65536
SOLVENT    CDCl3
NS         16
DS         2
SWH         8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         203
CW         60.800 usec
DE         6.50 usec
TE         302.5 K
D1         1.00000000 sec
TDO        1
  
```

```

***** CHANNEL f1 *****
NUC1       1H
P1         14.20 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1700011 MHz
WDW        EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
  
```

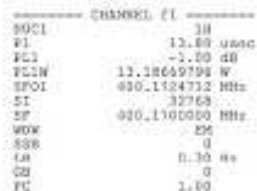
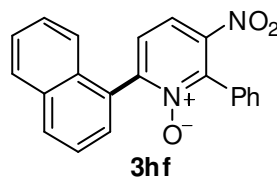
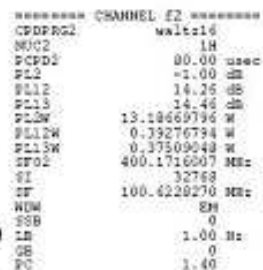
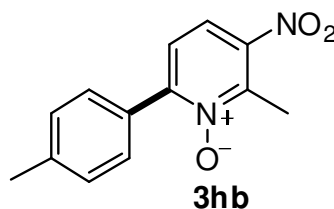


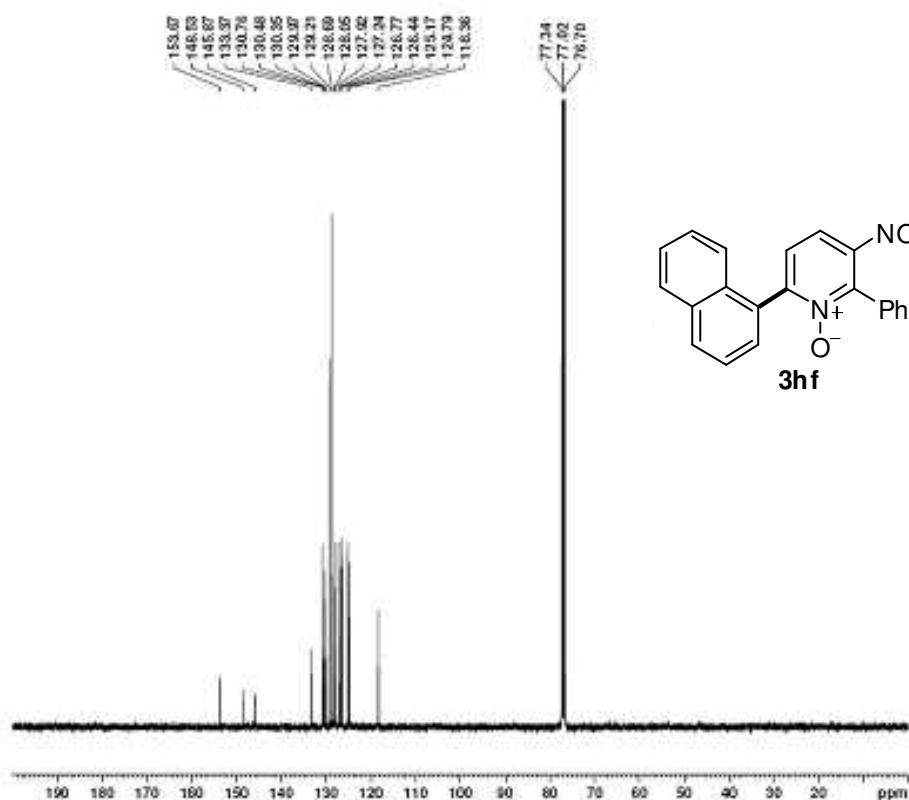
```

NAME      ZF9-19
EXPNO     1
PROCNO    1
Date_     20090413
Time      11.87
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG    zg30
TD         65536
SOLVENT    CDCl3
NS         16
DS         2
SWH         8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         203
CW         60.800 usec
DE         6.50 usec
TE         298.6 K
D1         1.00000000 sec
TDO        1
  
```

```

***** CHANNEL f1 *****
NUC1       1H
P1         13.80 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1700011 MHz
WDW        EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
  
```

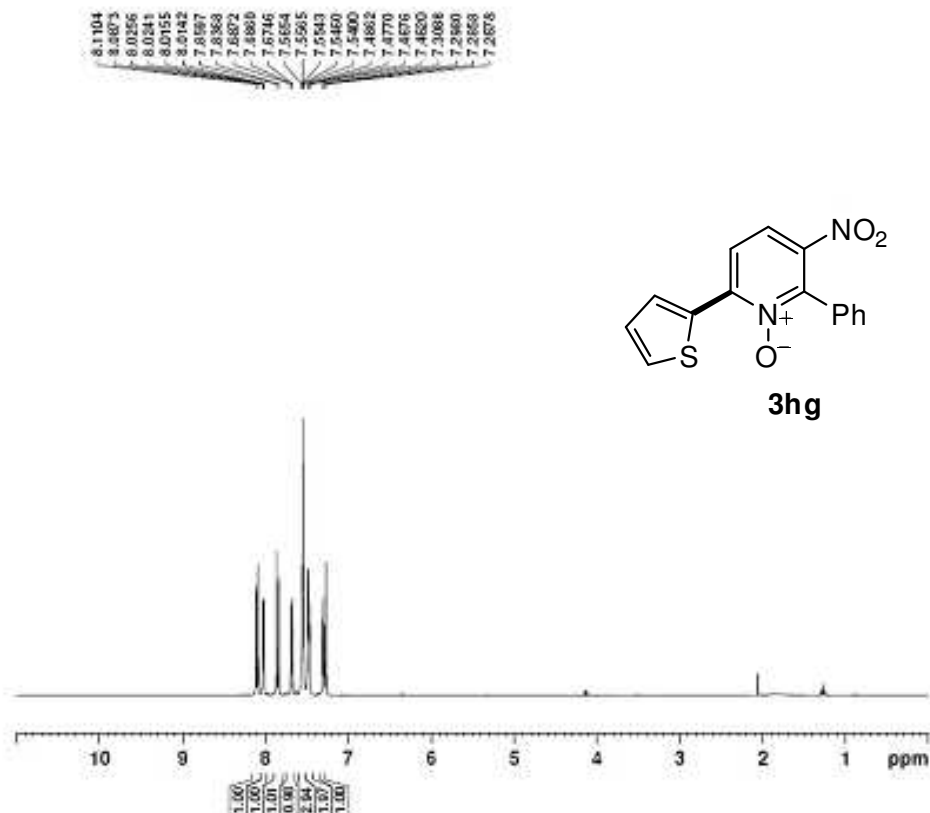




NAME 2F6-24-3-013
 EXPNO 1
 PROCNO 1
 Date_ 20090421
 Time 8.12
 INSTRUM spect
 PROBHD 5 mm FAREO BB-
 PULPROG zgpg30
 TO 65536
 TD 213
 SOLVENT CDCl3
 NS 887
 DS 4
 SWH 24036.461 KHz
 FIDRES 0.366798 KHz
 AQ 1.3671988 sec
 RG 213
 DW 20.800 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

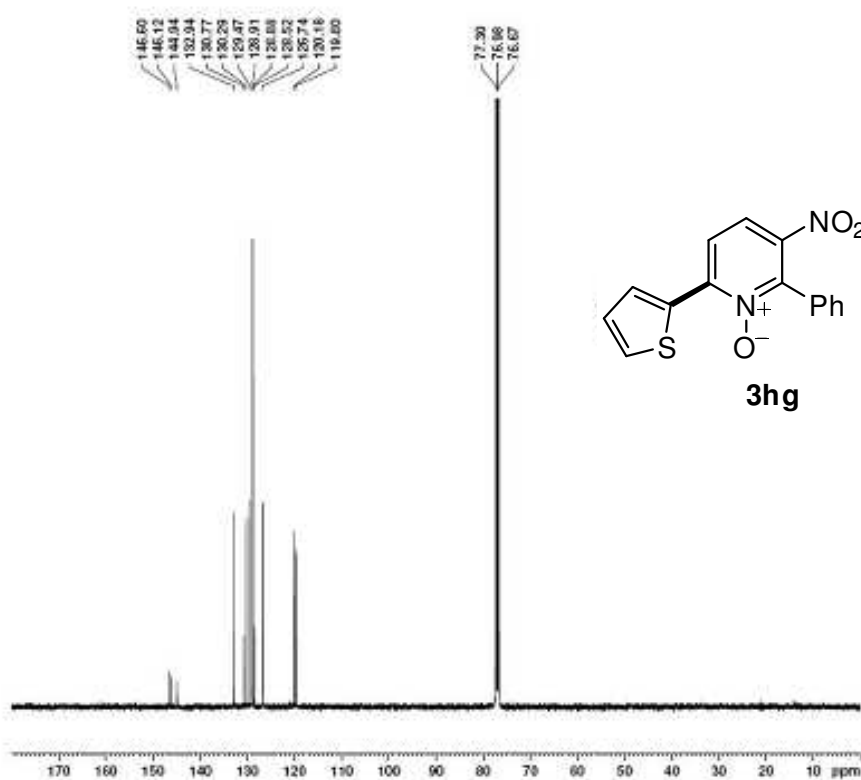
===== CHANNEL f1 =====
 NUCL1 13C
 P1 8.50 usec
 PL1 -2.00 dB
 PL1W 57.32743073 W
 SFO1 100.6228270 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUCL2 1H
 PCPD2 88.60 usec
 PL2 -1.00 dB
 PL12 14.20 dB
 PL13 14.40 dB
 PL1W 13.18669796 W
 PL12W 0.39276794 W
 PL13W 0.37509048 W
 SFO2 400.176007 MHz
 S1 32768
 SF 100.6228270 MHz
 SWH 1M
 SSB 0
 LB 1.00 KHz
 GB 0
 PC 1.40



NAME 2F6-42
 EXPNO 1
 PROCNO 1
 Date_ 20090421
 Time 9.10
 INSTRUM spect
 PROBHD 5 mm FAREO BB-
 PULPROG zgpg30
 TO 65536
 TD 213
 SOLVENT CDCl3
 NS 16
 DS 3
 SWH 8223.485 KHz
 FIDRES 0.125463 KHz
 AQ 3.9846387 sec
 RG 203
 DW 60.800 usec
 DE 6.50 usec
 TE 297.2 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUCL1 1H
 P1 13.60 usec
 PL1 -1.00 dB
 PL1W 13.18669796 W
 SFO1 400.1724713 MHz
 S1 32768
 SF 400.1700000 MHz
 SWH 1M
 SSB 0
 LB 0.30 KHz
 GB 0
 PC 1.00



```

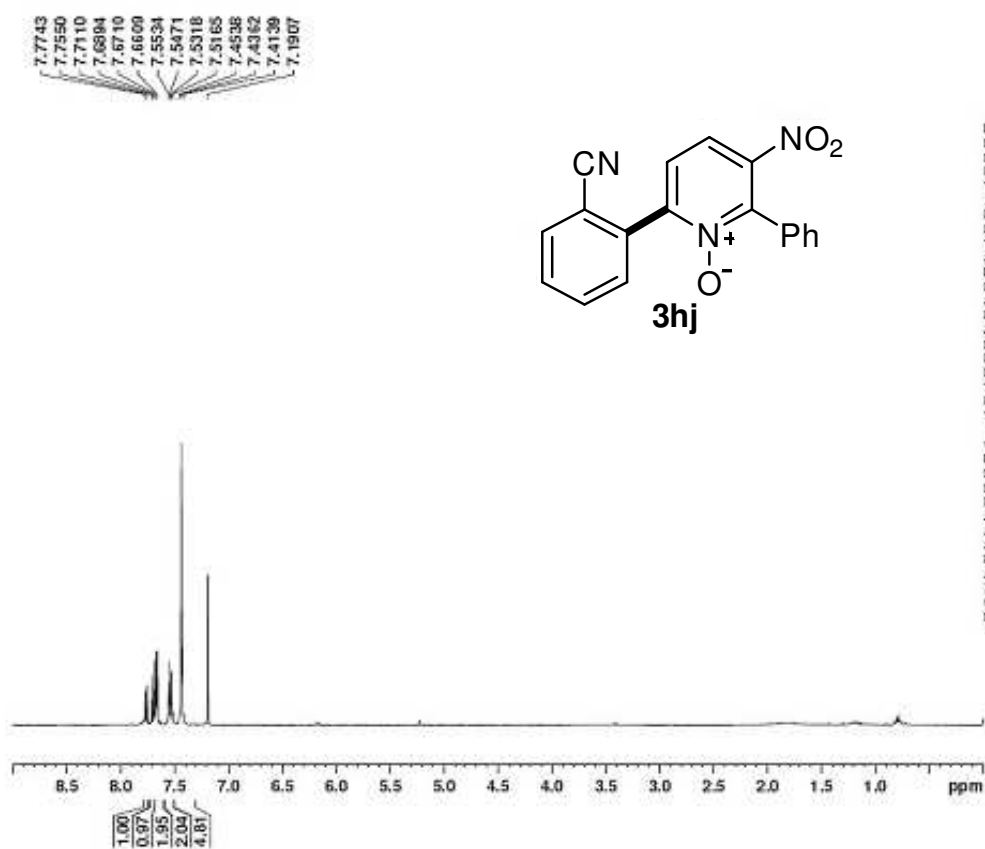
NAME      ZFB-42-013
EXPNO     1
PROCNO    1
Date_     20090513
Time      10.23
INSTRUM   spect
PROBHD    5 mm JABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         3230
DS         4
SWH        24036.461 Hz
FIDRES     0.366738 Hz
AQ          1.7631988 sec
RG          203
CW          20.820 usec
DE          6.50 usec
TE          303.2 K
D1          2.00000000 sec
D12         0.03000000 sec
TD0         1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1          8.50 usec
PL1         -1.00 dB
PL1W        13.18669796 W
PL1W        0.35276734 W
PL1W        0.37500048 W
SFO1        400.1756007 MHz
SI          32768
SF          100.6218210 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       60.00 usec
PL2          -1.00 dB
PL2W        16.26 dB
PL2W        16.44 dB
PL2W        13.18669796 W
PL2W        0.35276734 W
PL2W        0.37500048 W
SFO2        400.1756007 MHz
SI          32768
SF          100.6218210 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

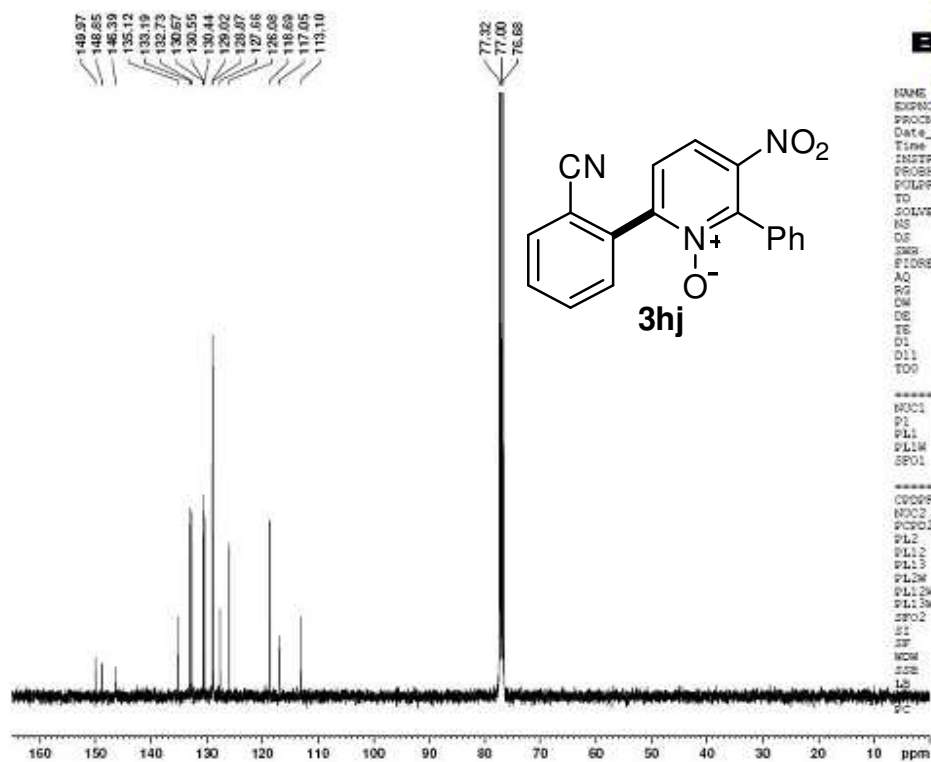


```

NAME      ZFB-34-2
EXPNO     1
PROCNO    1
Date_     20090424
Time      10.50
INSTRUM   spect
PROBHD    5 mm JABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         32
DS         2
SWH        8023.695 Hz
FIDRES     0.125403 Hz
AQ          3.9846367 sec
RG          203
CW          60.800 usec
DE          6.50 usec
TE          295.2 K
D1          1.00000000 sec
TD0         1
  
```

```

===== CHANNEL f1 =====
NUC1       1H
P1          13.00 usec
PL1         -1.00 dB
PL1W        13.18669796 W
SFO1        400.1724712 MHz
SI          32768
SF          400.1700309 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
  
```

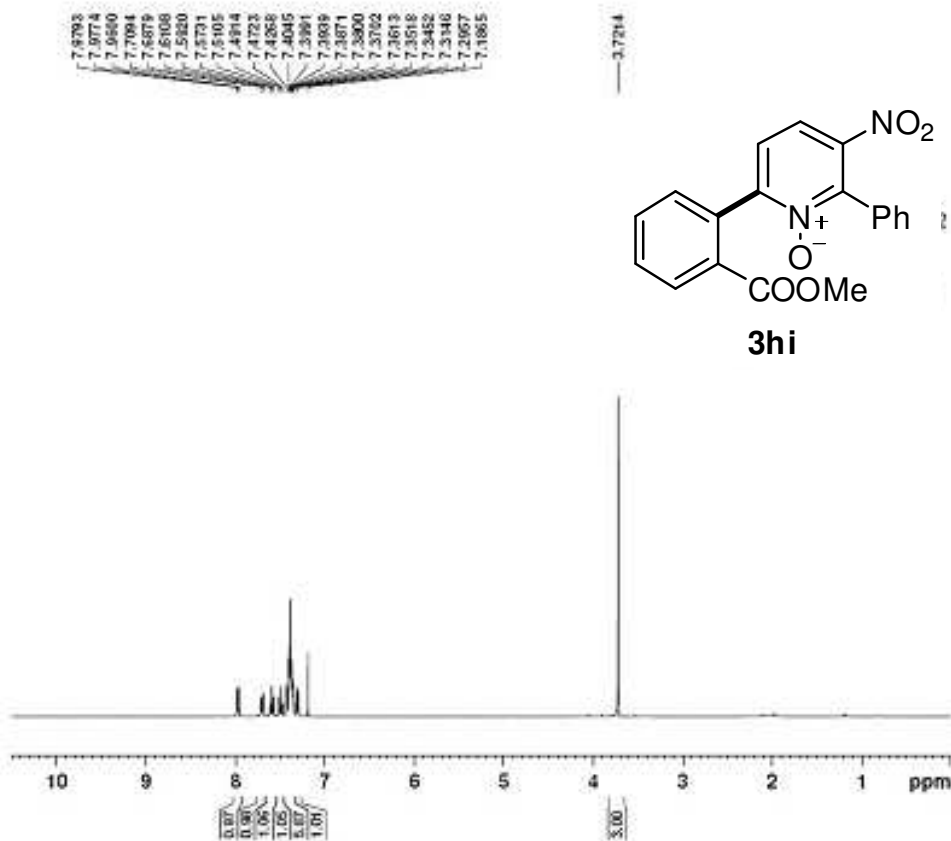


BRUKER

NAME: zfr-34-2-013
EXPNO: 1
PROCNO: 1
Date_: 20090427
Time: 6.50
INSTRUM: spect
PROBHD: 5 mm DABBO BB-
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 3200
DS: 4
SWH: 24039.461 Hz
FIDRES: 0.366738 Hz
AQ: 1.0361988 sec
RG: 203
DN: 20.800 usec
DE: 6.50 usec
TE: 298.2 K
D1: 2.00000000 sec
D11: 0.03000000 sec
TD0: 1

===== CHANNEL f1 =====
NUC1: 13C
P1: 8.50 usec
PL1: -2.00 dB
PL1W: 57.32743073 W
SFO1: 100.6329888 MHz

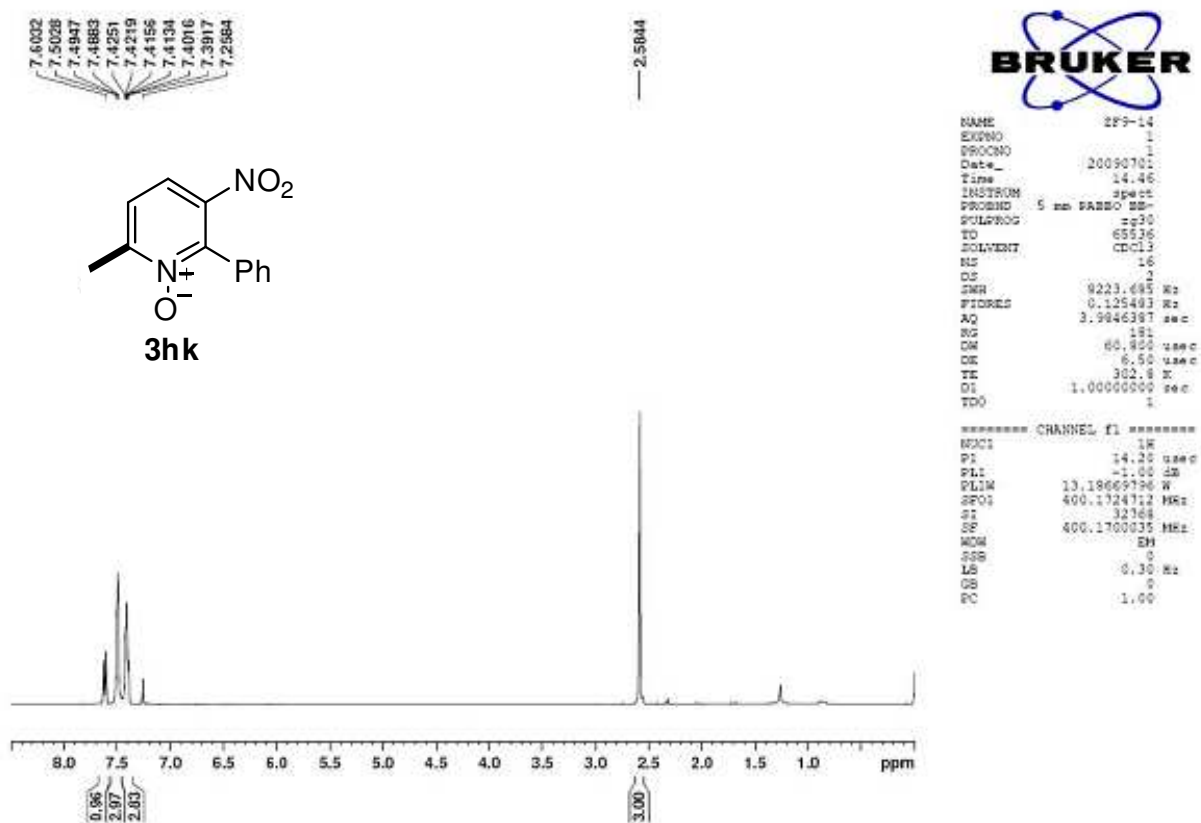
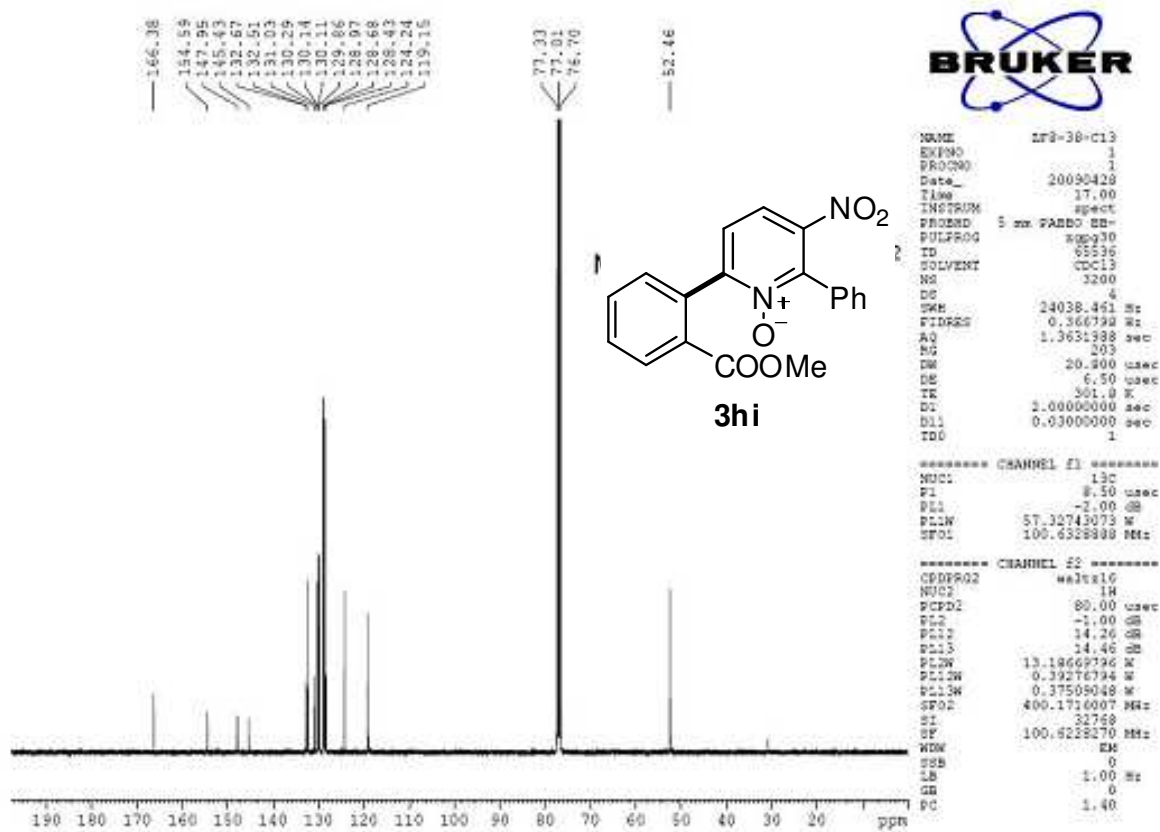
===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: -1.00 dB
PL12: 14.26 dB
PL13: 14.46 dB
PL1W: 13.18669796 W
PL1W: 0.39276754 W
PL1W: 0.37509048 W
SFO2: 400.1716007 MHz
S1: 32768
SF: 100.6229270 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

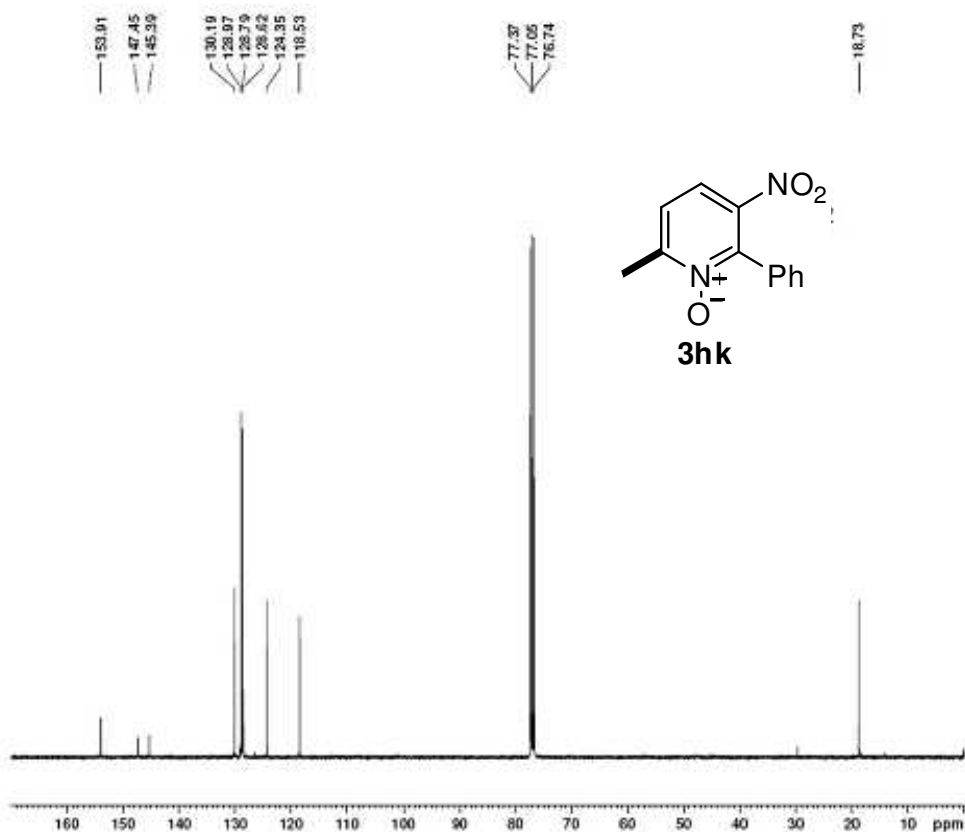


BRUKER

NAME: zfr-36
EXPNO: 1
PROCNO: 1
Date_: 20090422
Time: 17.19
INSTRUM: spect
PROBHD: 5 mm DABBO BB-
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 16
DS: 2
SWH: 8229.695 Hz
FIDRES: 0.125493 Hz
AQ: 3.9846197 sec
RG: 203
DN: 60.800 usec
DE: 6.50 usec
TE: 298.2 K
D1: 1.00000000 sec
TD0: 1

===== CHANNEL f1 =====
NUC1: 1H
P1: 13.00 usec
PL1: -1.00 dB
PL1W: 13.18669796 W
SFO1: 400.1724112 MHz
S1: 32768
SF: 400.1700325 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00





BRUKER

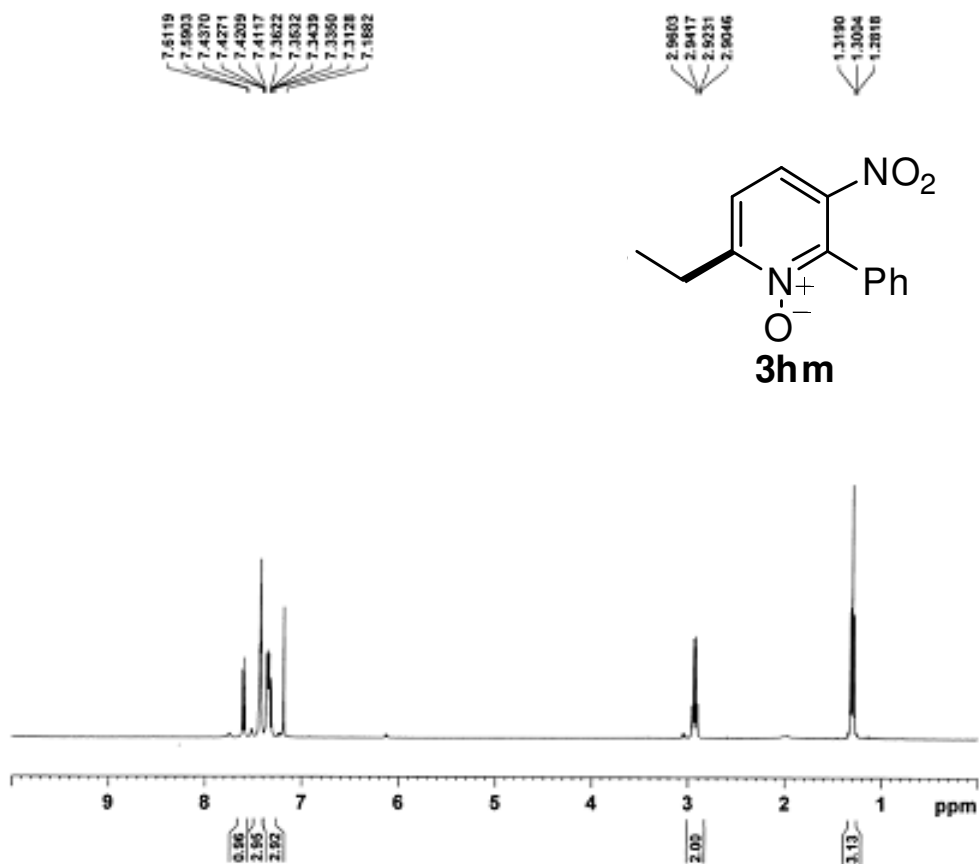
```

NAME      ZF9-14-013
EXPNO     1
PROCNO    1
Date_     20090701
Time      16.19
INSTRUM   spect
PROBHD    5 mm PARBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1835
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.2631988 sec
RG         203
OW         20.800 usec
DE         6.50 usec
TE         304.4 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1

===== CHANNEL F1 =====
NUC1       13C
P1         8.50 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1       100.6328898 MHz

===== CHANNEL F2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -1.00 dB
PL12       14.02 dB
PL13       14.46 dB
PL2W       13.18669796 W
PL12W      0.41508400 W
PL13W      0.37509048 W
SFO2       400.1516007 MHz
SI         32768
SF         100.6228219 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```



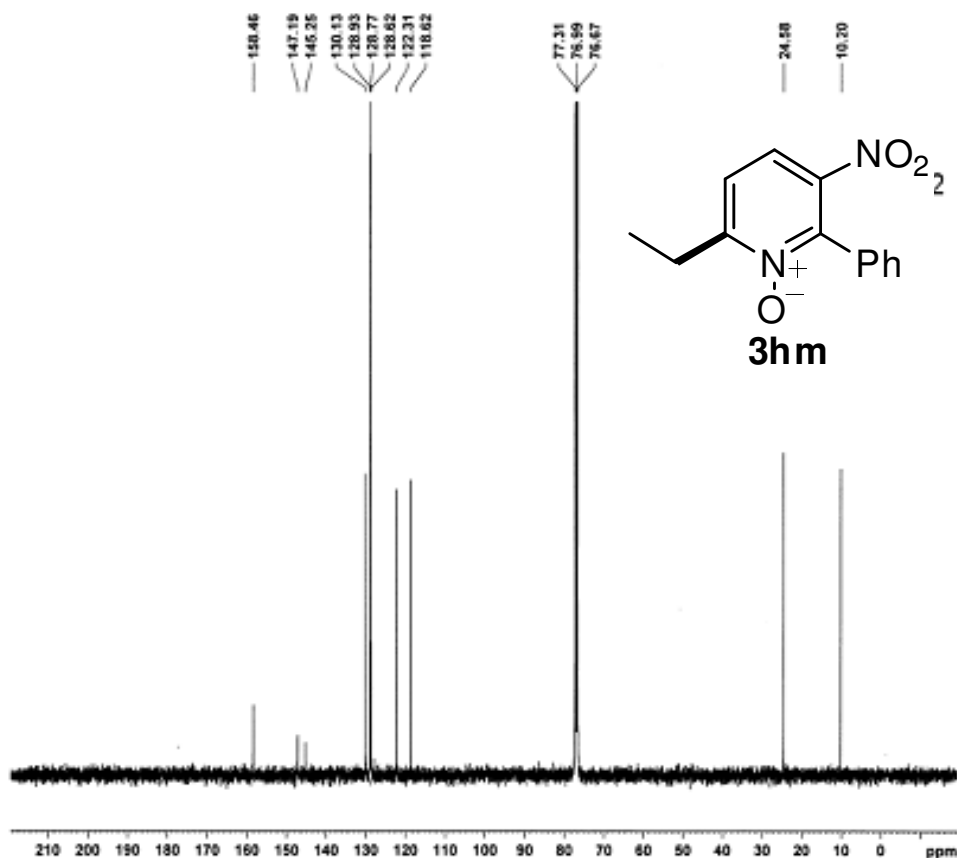
BRUKER

```

NAME      ZF9-47
EXPNO     1
PROCNO    1
Date_     20090504
Time      9.13
INSTRUM   spect
PROBHD    5 mm PARBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         203
OW         60.800 usec
DE         6.50 usec
TE         300.2 K
D1         1.6000000 sec
D11        0.0300000 sec
TD0        1

===== CHANNEL F1 =====
NUC1       1H
P1         13.80 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1708317 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

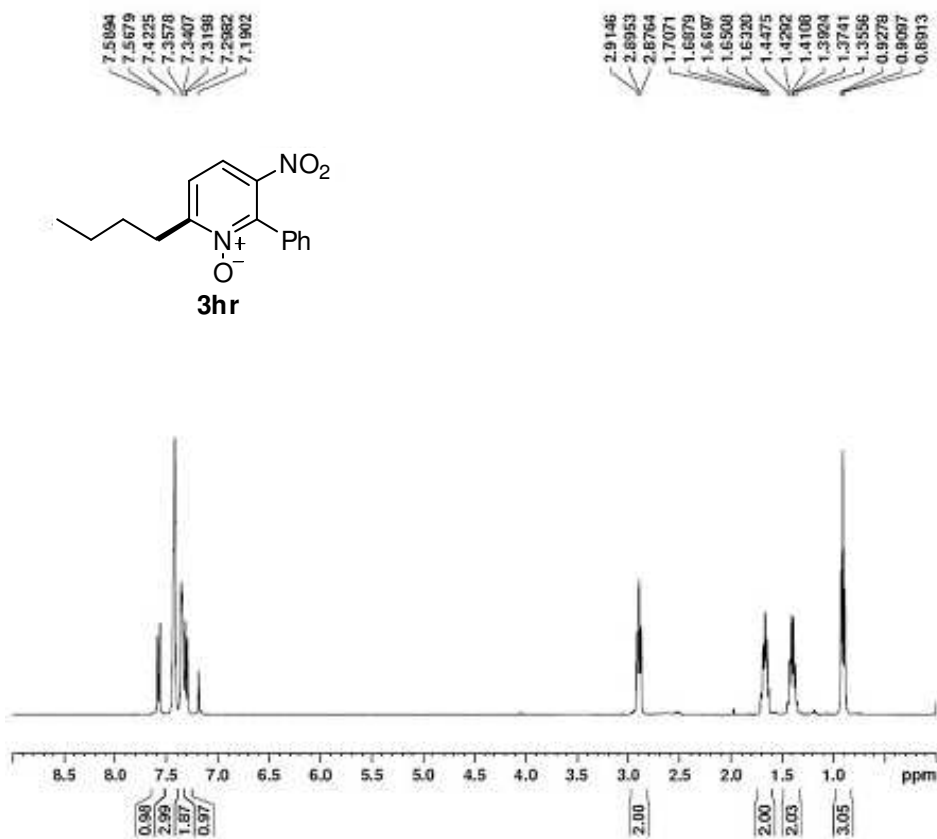
```



NAME ZFG-47-C13
EXPNO 1
PROCNO 1
Date_ 20090504
Time_ 1.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3200
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.680 usec
DE 6.50 usec
TE 302.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

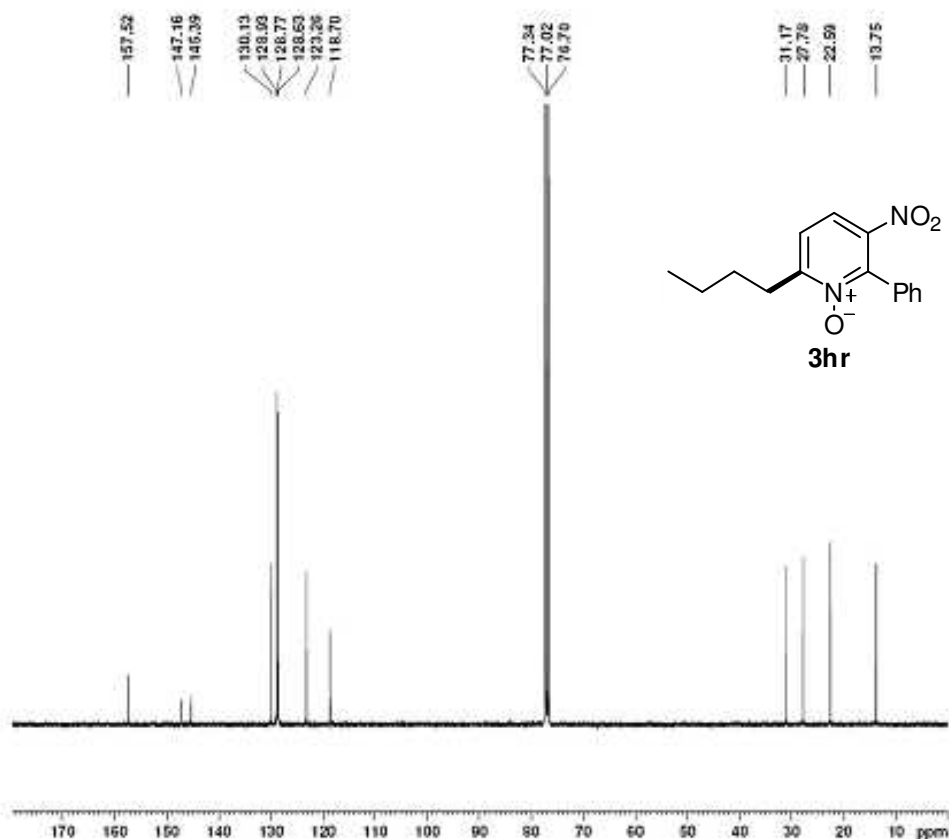
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 57.32143073 W
SFO1 100.6328888 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.26 dB
PL13 16.46 dB
PL1W 13.18669796 W
PL12W 0.39276794 W
PL13W 0.37509248 W
SFO2 400.1716807 MHz
SI 32768
SF 100.6228270 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



NAME ZFG-6-1
EXPNO 1
PROCNO 1
Date_ 20090330
Time_ 9.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
PL1W 13.18669796 W
SFO1 400.1724712 MHz
SI 32768
SF 400.1700311 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



```

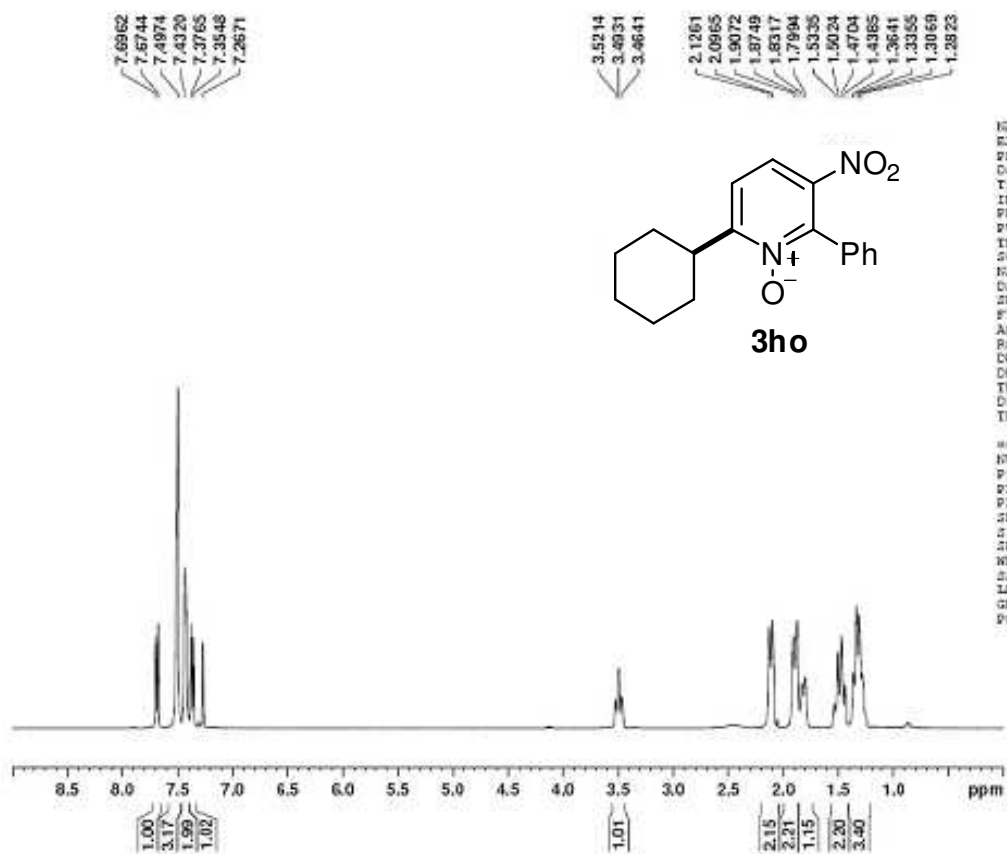
NAME      ZF6-6-1-013
EXPNO     1
PROCNO    1
Date_     20090331
Time      11.13
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1325
DS         4
SWH        24036.461 Hz
FIDRES     0.366798 Hz
AQ          1.3631988 sec
RG          203
CW          20.800 usec
DE          6.50 usec
TE          296.5 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1          8.50 usec
PL1         -2.00 dB
PL1W       57.32743073 W
SFO1       100.6328888 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2          1.00 dB
PL12        14.26 dB
PL13        34.46 dB
PL1W        13.18669796 W
PL12W       0.398276794 W
PL13W       0.37509048 W
SFO2        400.1714007 MHz
SI          32768
SF          100.6228270 MHz
WDW         EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```

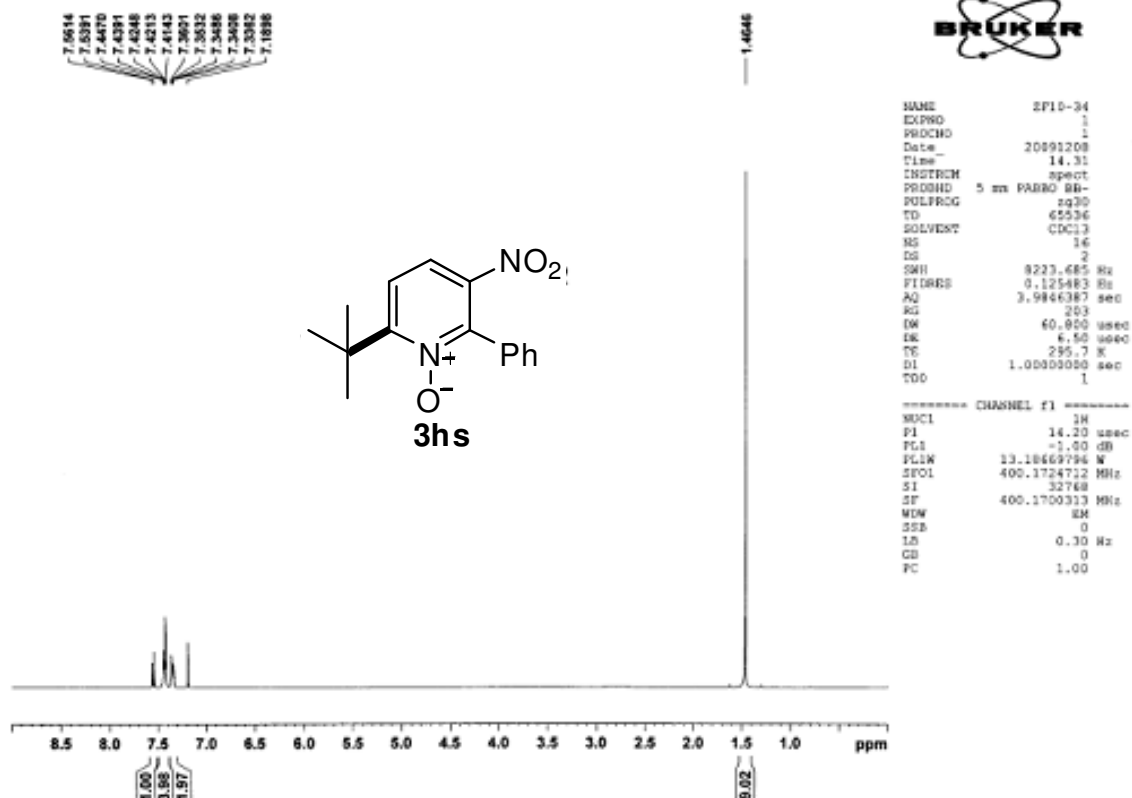
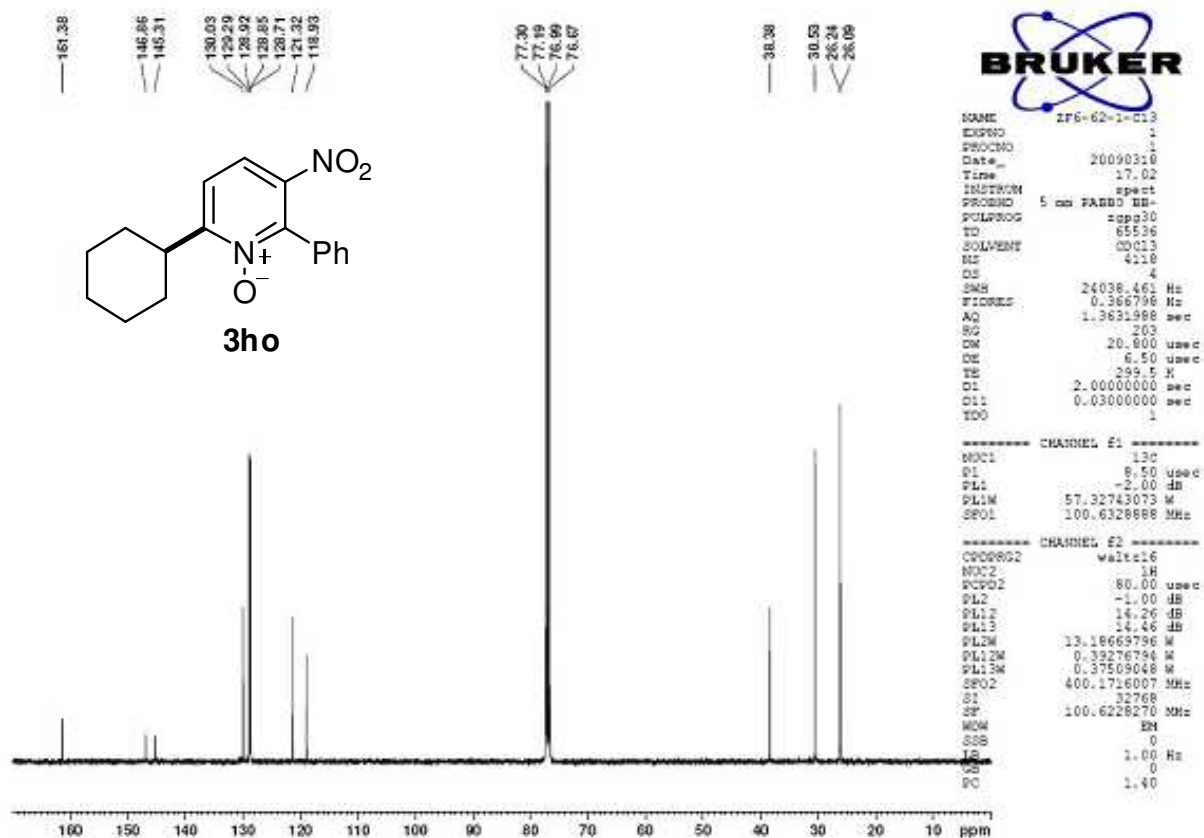


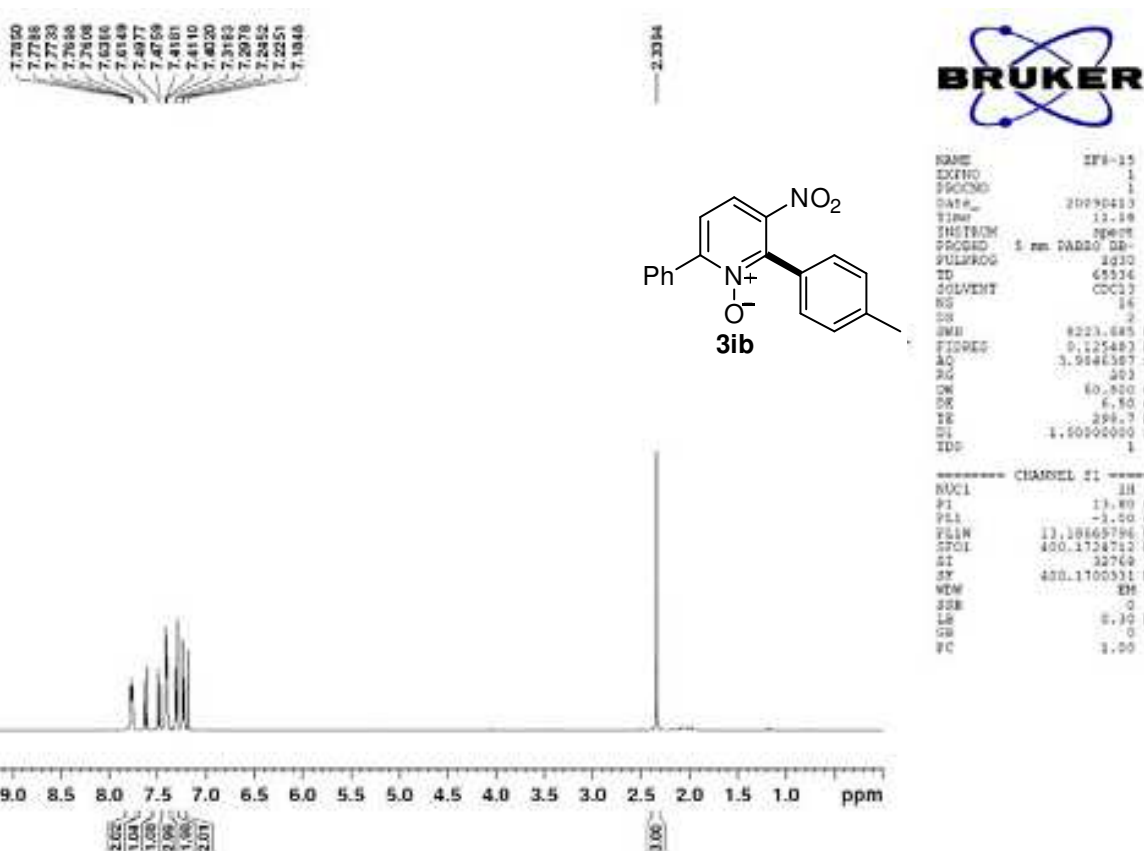
```

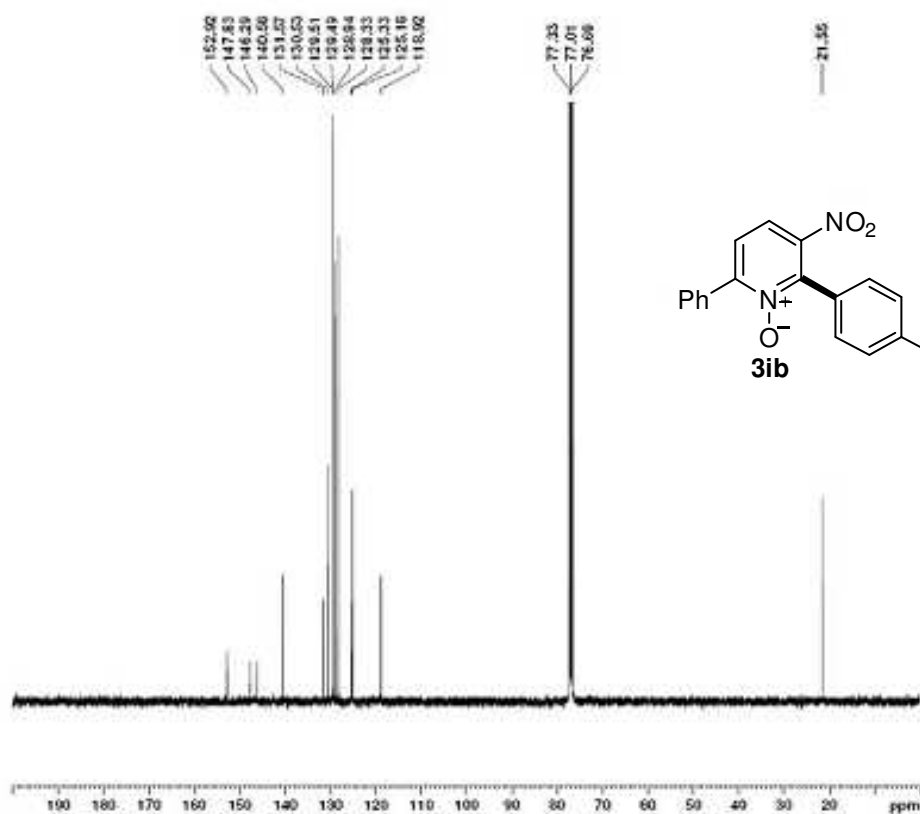
NAME      ZF6-62-1-2
EXPNO     1
PROCNO    1
Date_     20090317
Time      9.16
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.682 Hz
FIDRES     0.125483 Hz
AQ          3.9846387 sec
RG          181
CW          60.800 usec
DE          6.50 usec
TE          294.3 K
D1          1.20000000 sec
TD0         1
  
```

```

===== CHANNEL f1 =====
NUC1       1H
P1          13.80 usec
PL1          1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI          32768
SF          400.1700000 MHz
WDW         EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```



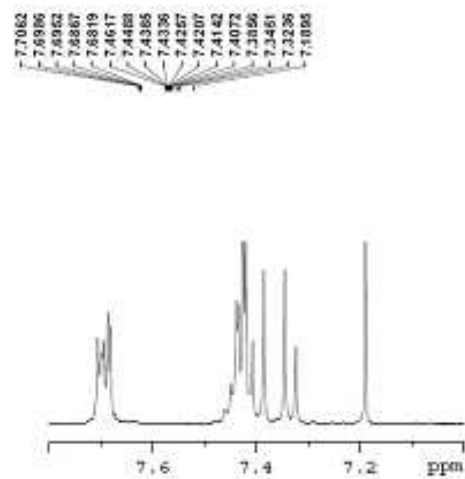




NAME: 2F8-15-013
 EXPNO: 1
 PROCNO: 1
 Date_: 20090414
 Time: 7.49
 INSTRUM: spect
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 2460
 DS: 4
 SWH: 24038.461 Hz
 FIDRES: 0.369198 Hz
 AQ: 1.3631888 sec
 RG: 203
 DW: 20.800 usec
 DE: 6.50 usec
 TE: 300.7 K
 D1: 2.00000000 sec
 D11: 0.00000000 sec
 TDO: 1

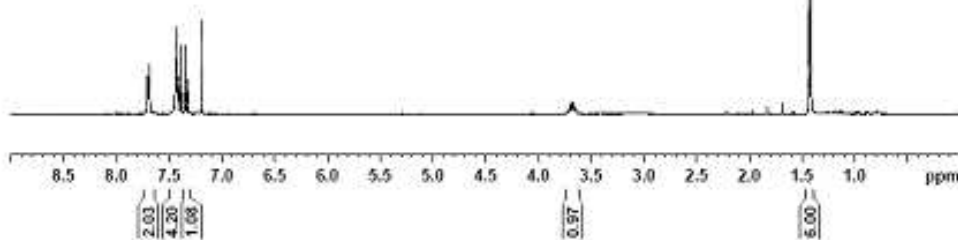
===== CHANNEL f1 =====
 NUC1: 13C
 P1: 8.50 usec
 PL1: -2.00 dB
 PL1W: 57.32743073 W
 SF01: 100.6260800 MHz

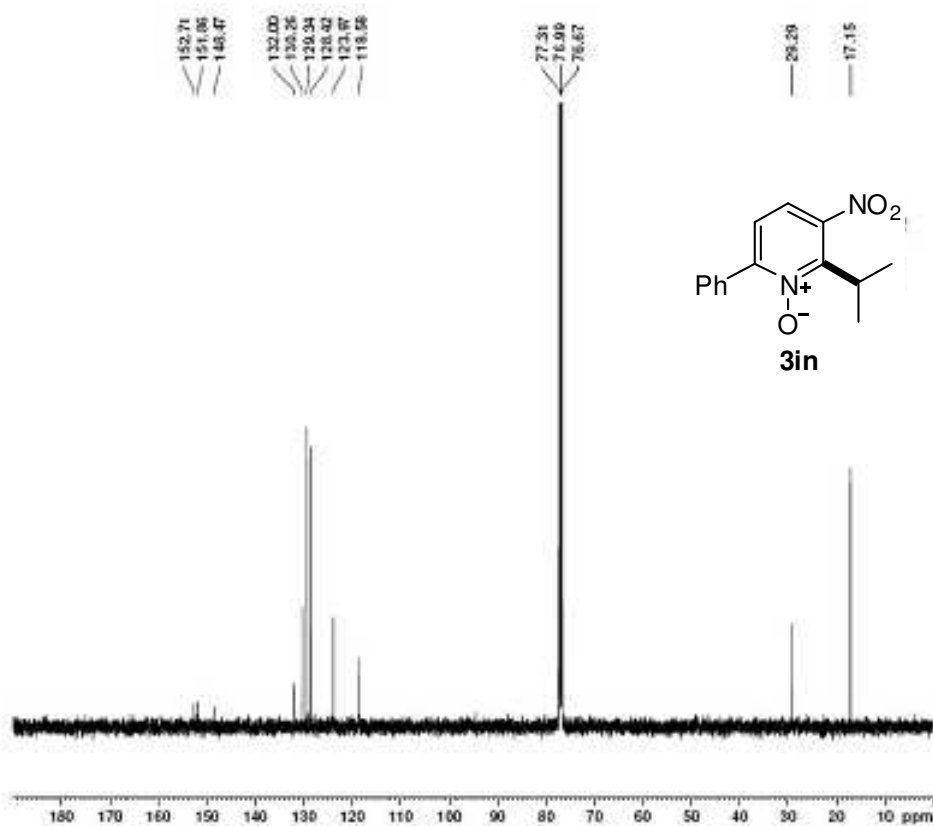
===== CHANNEL f2 =====
 CPDPRG2: waltz16
 NUC2: 1H
 PCDP2: 80.00 usec
 PL2: -1.00 dB
 PL12: 14.20 dB
 PL13: 14.46 dB
 PL1W: 13.18669796 W
 PL1W2: 0.39216194 W
 PL1W3: 0.37509248 W
 SF02: 400.1476007 MHz
 S1: 32768
 S2: 100.6260270 MHz
 SWH: 8M
 SSB: 0
 LB: 1.90 Hz
 GB: 0
 PC: 1.40



NAME: 2F8-29
 EXPNO: 1
 PROCNO: 1
 Date_: 20090415
 Time: 17.13
 INSTRUM: spect
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 16
 DS: 2
 SWH: 8223.696 Hz
 FIDRES: 0.125403 Hz
 AQ: 3.9846387 sec
 RG: 203
 DW: 60.800 usec
 DE: 6.50 usec
 TE: 300.7 K
 D1: 1.00000000 sec
 TDO: 1

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 13.00 usec
 PL1: -1.00 dB
 PL1W: 13.18669796 W
 SF01: 400.1476112 MHz
 S1: 32768
 S2: 400.1700312 MHz
 SWH: 8M
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00





```

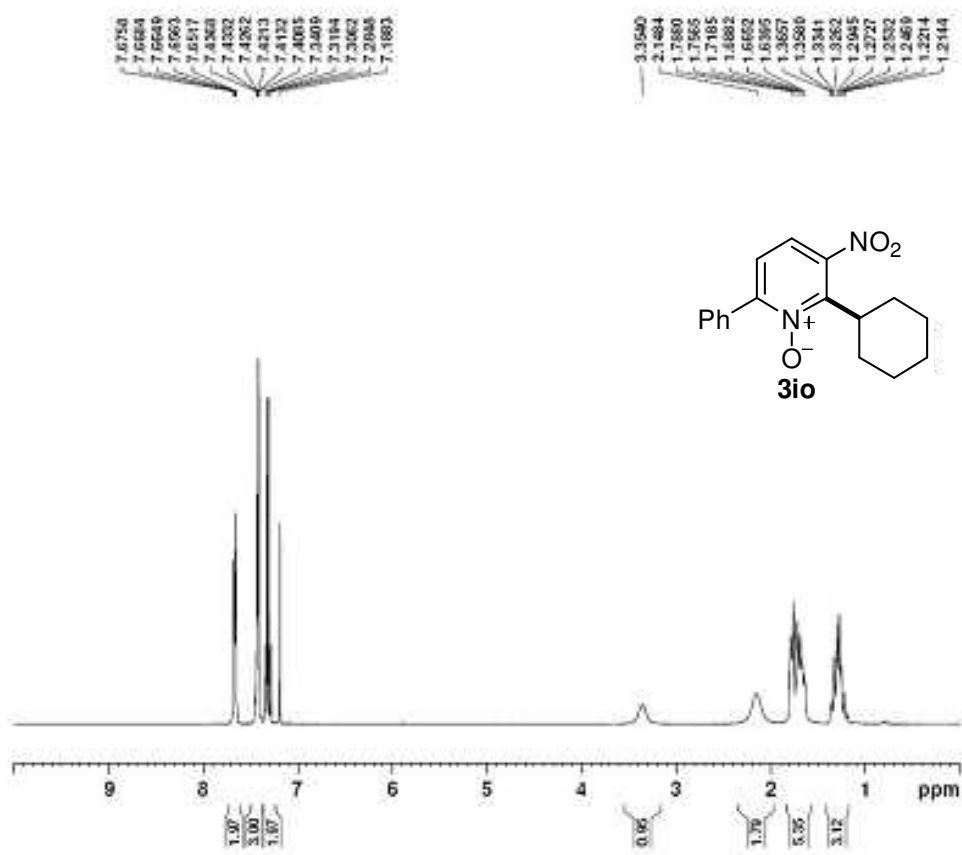
NAME      ZFS-29-013
EXPNO     1
PROCNO    1
Date_     20090410
Time      8.24
INSTRUM   spect
PROBHD    5 mm PABBO 80-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         638
DS         4
SWH        24039.461 Hz
FIDRES     0.366798 Hz
AQ         1.3632368 sec
RG         203
CW         20.903 usec
DE         6.50 usec
TE         298.6 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       13C
P1         13.00 usec
PL1        -1.00 dB
PL1W       57.32743073 W
SFO1       100.6283698 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       90.00 usec
PL2         -1.00 dB
PL12        14.24 dB
PL13        14.46 dB
PL1W        13.18669796 W
PL12W       0.39276794 W
PL13W       0.37509048 W
SFO2       400.1716007 MHz
S1         32768
SF         400.1700317 MHz
WDW         RM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

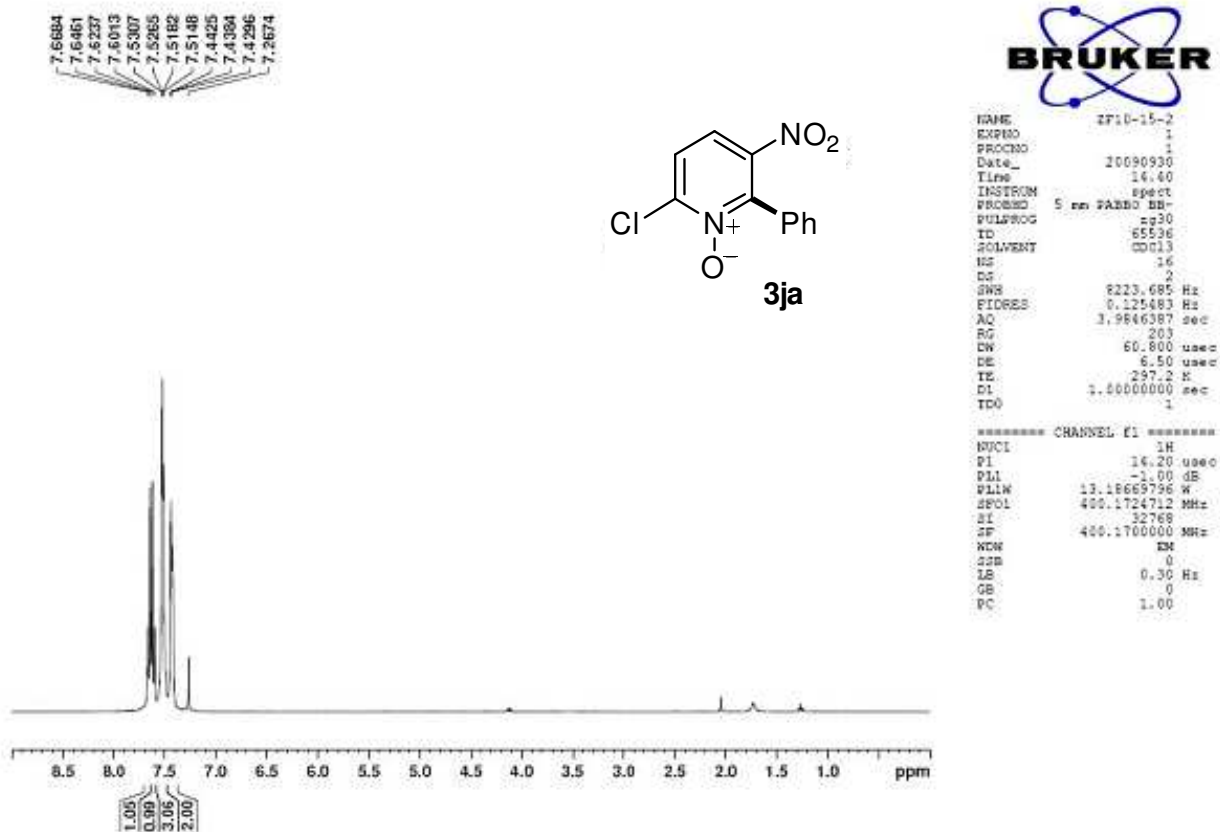
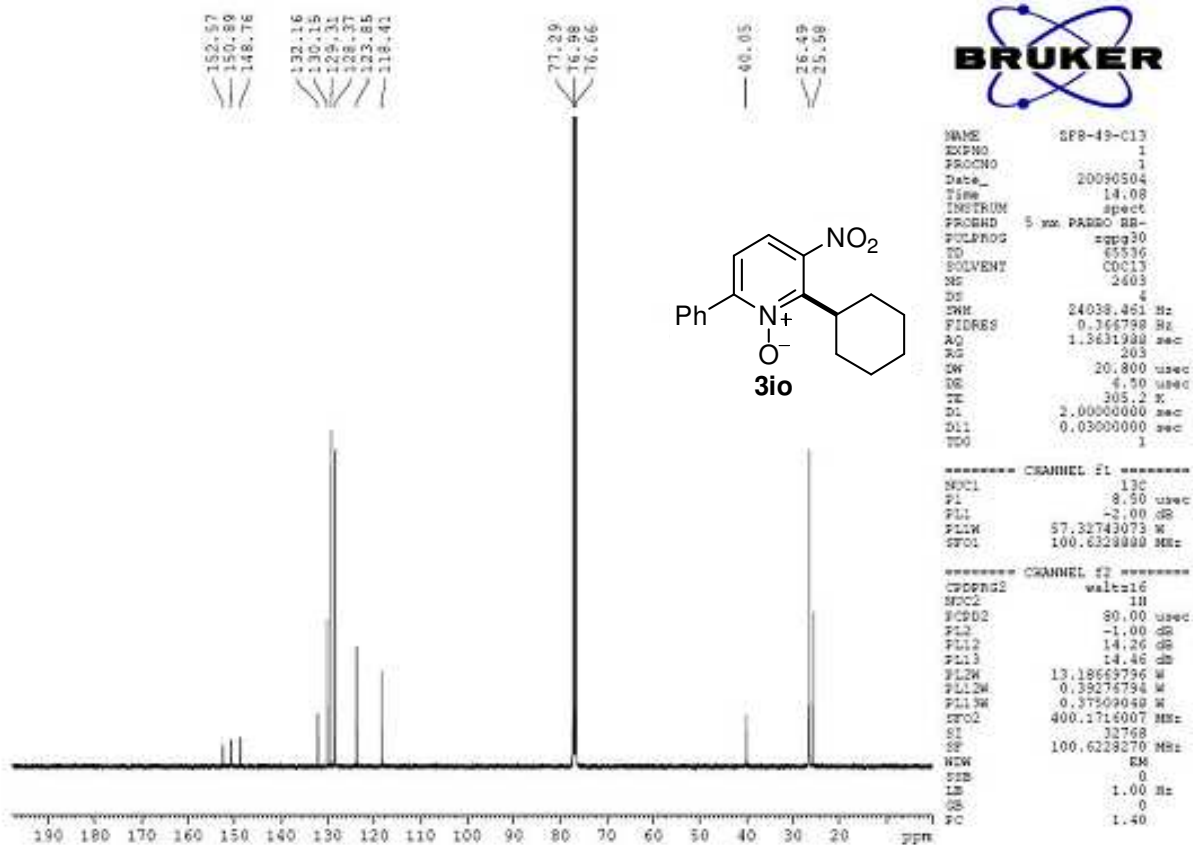


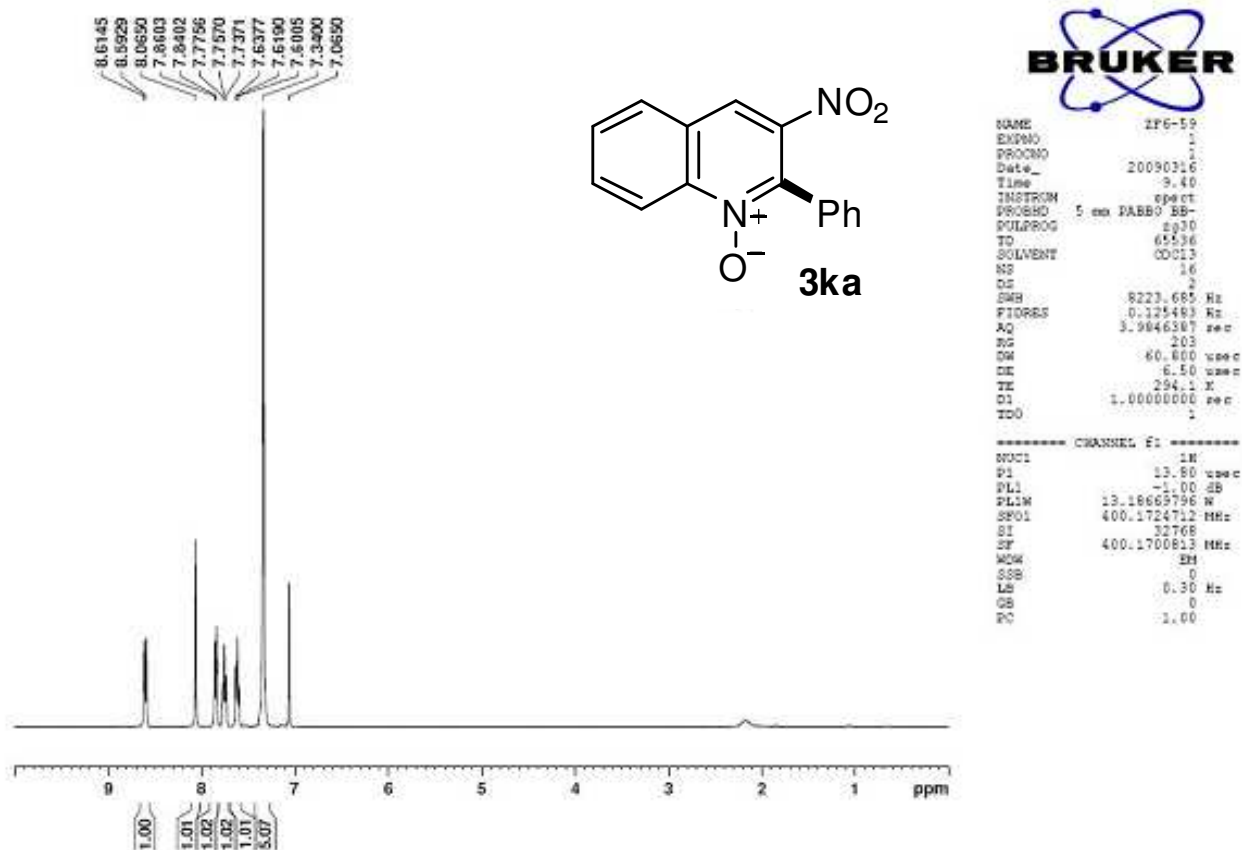
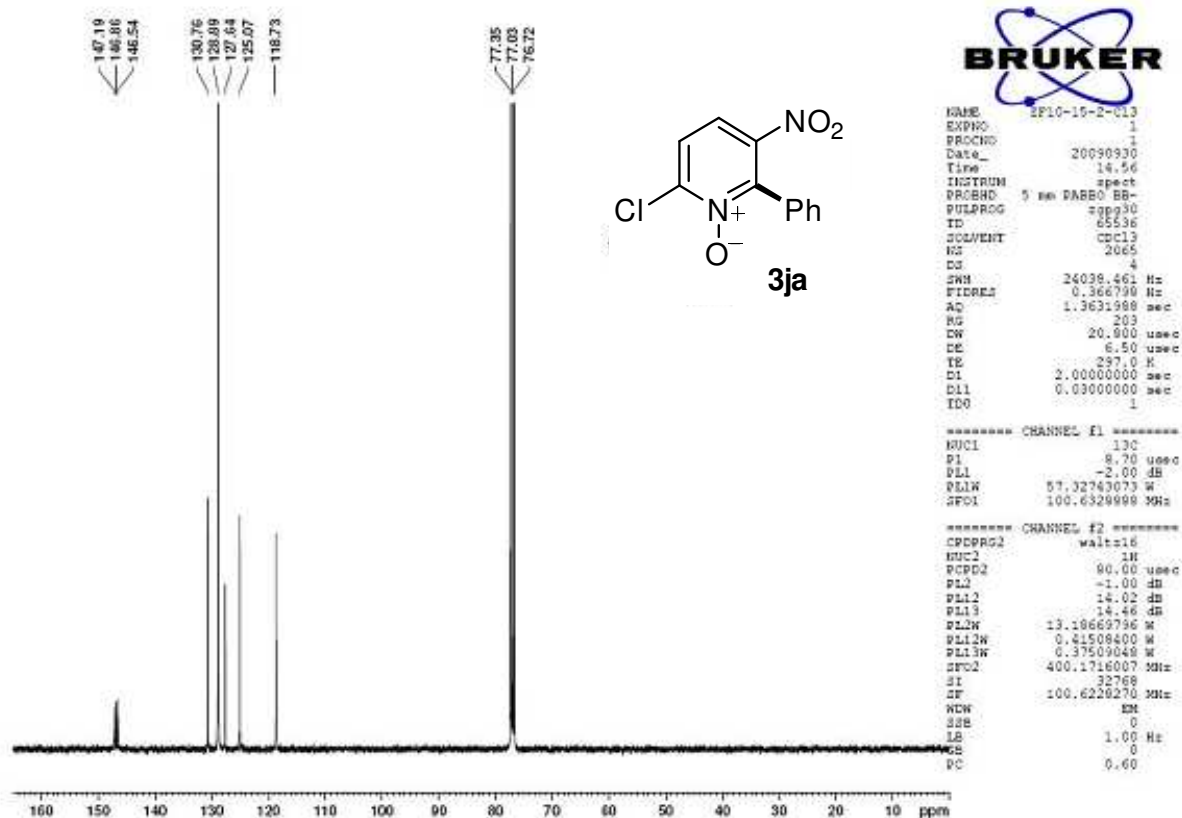
```

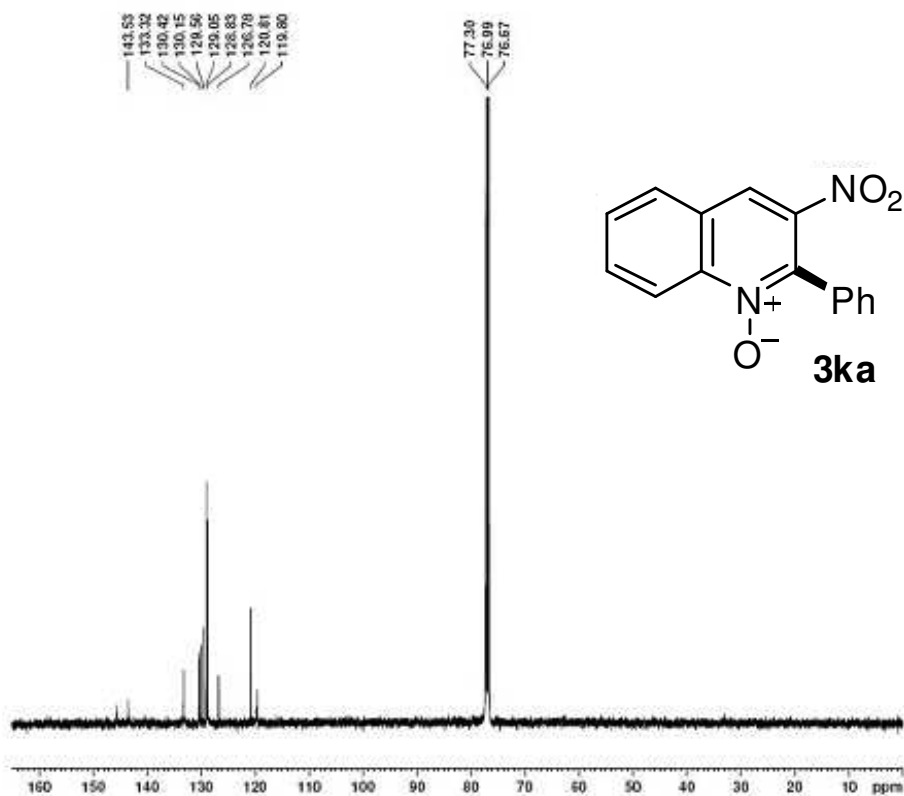
NAME      ZFS-49
EXPNO     1
PROCNO    1
Date_     20090504
Time      9.22
INSTRUM   spect
PROBHD    5 mm PABBO 80-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         203
CW         60.800 usec
DE         6.50 usec
TE         300.1 K
D1         1.0000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         13.00 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1734712 MHz
S1         32768
SF         400.1700317 MHz
WDW         RM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
  
```







```

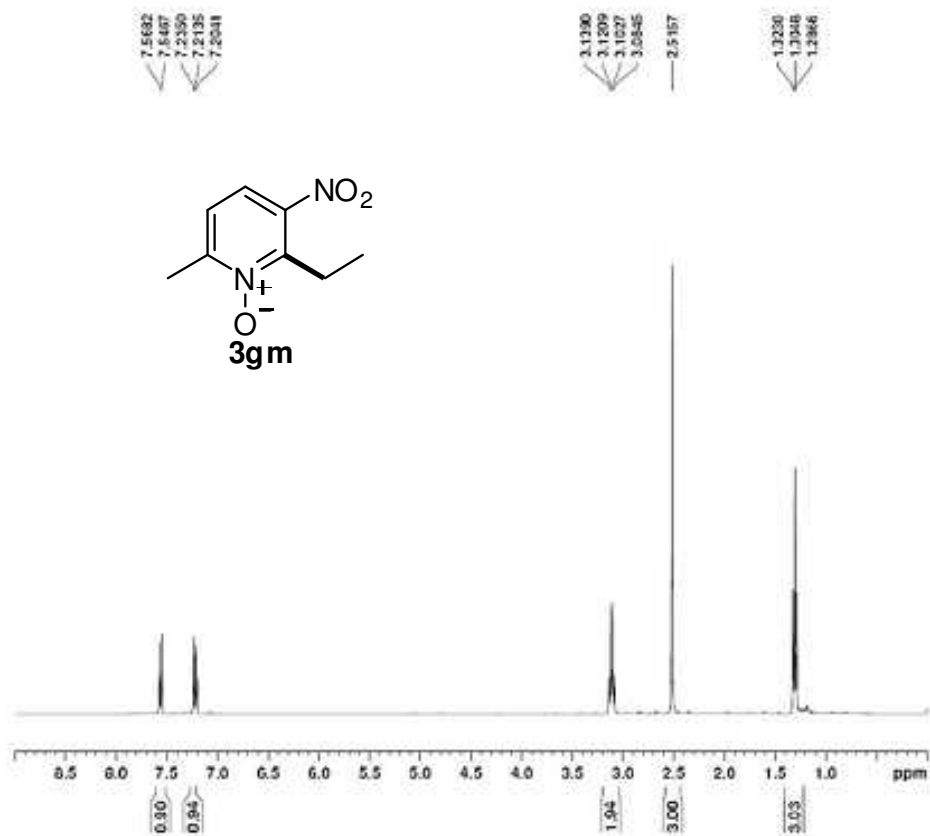
NAME      ZF6-59-013
EXPNO     1
PROCNO    1
Date_     20090316
Time      16.14
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         3501
DS         4
SWH        24030.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
SW         20.800 usec
DE         6.50 usec
TE         298.0 K
D1         3.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

----- CHANNEL f1 -----
NUC1       13C
P1         9.50 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1       100.6328889 MHz
  
```

```

----- CHANNEL f2 -----
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -1.00 dB
PL12        14.26 dB
PL13        14.46 dB
PL1W       13.18669796 W
PL12W      0.33276794 W
PL13W      0.37509049 W
SFO2       400.1716007 MHz
SI         32769
SF         100.6228270 MHz
WDW         EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

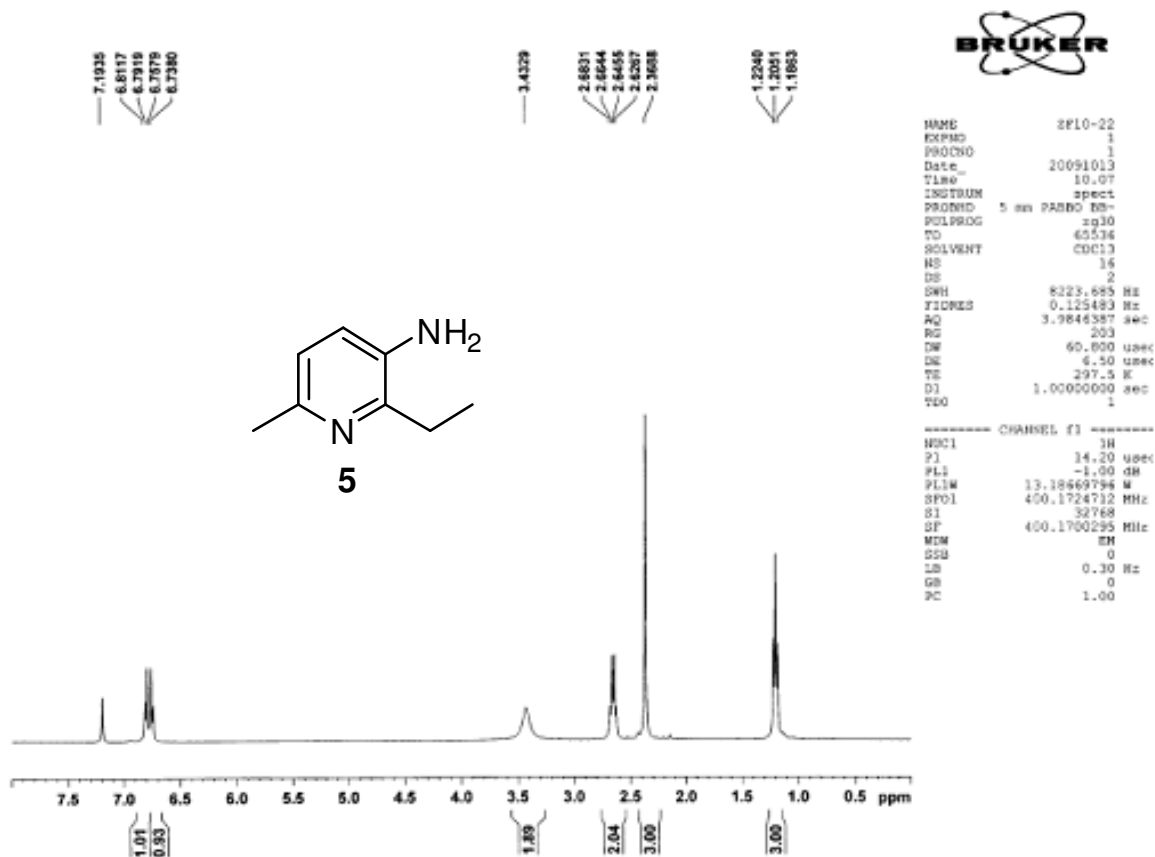
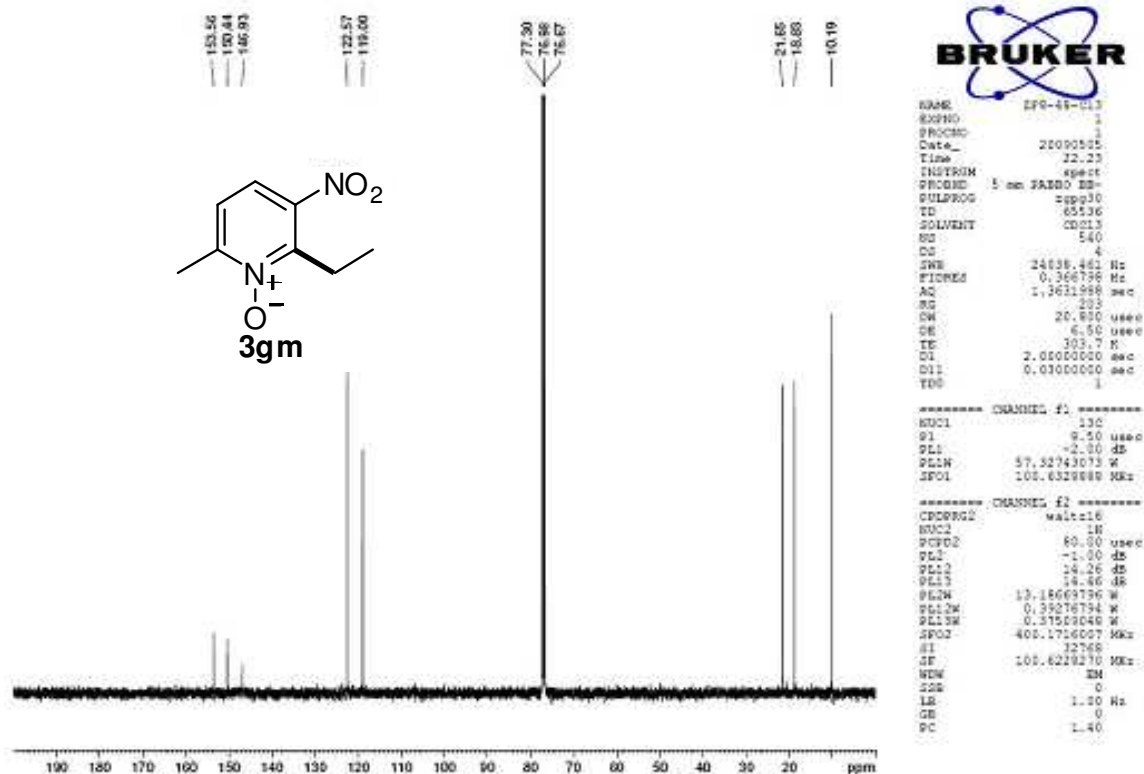


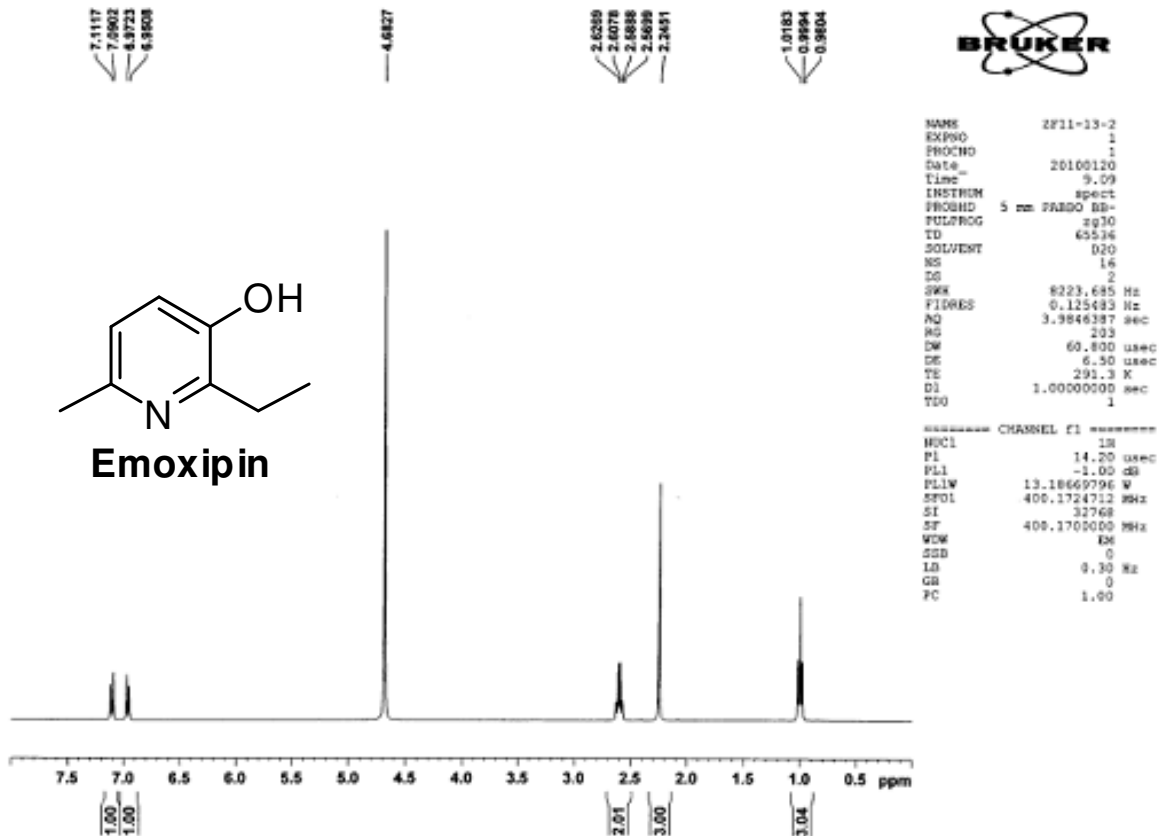
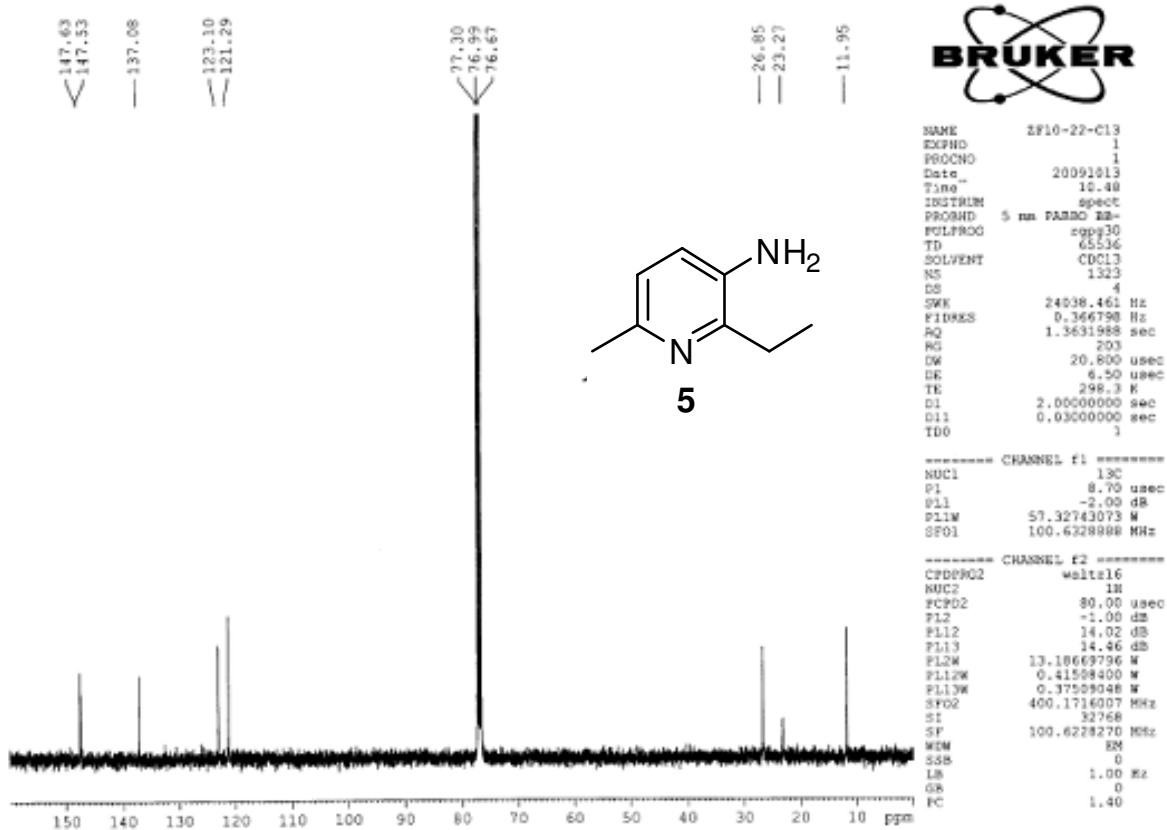
```

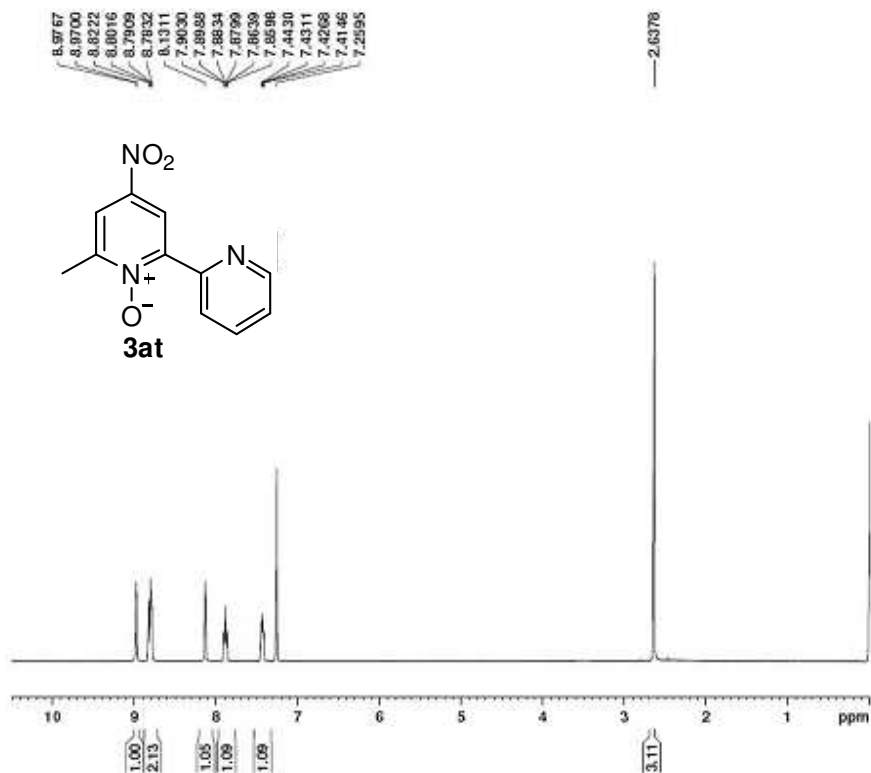
NAME      ZF6-48
EXPNO     1
PROCNO    1
Date_     20090504
Time      16.37
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         0
SWH        8323.685 Hz
FIDRES     0.155483 Hz
AQ         3.9846187 sec
RG         161
SW         40.800 usec
DE         6.50 usec
TE         295.5 K
D1         1.00000000 sec
TD0        1
  
```

```

----- CHANNEL f1 -----
NUC1       1H
P1         13.80 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32769
SF         400.1700257 MHz
WDW         EM
SSB         0
LB         2.30 Hz
GB         0
PC         1.00
  
```



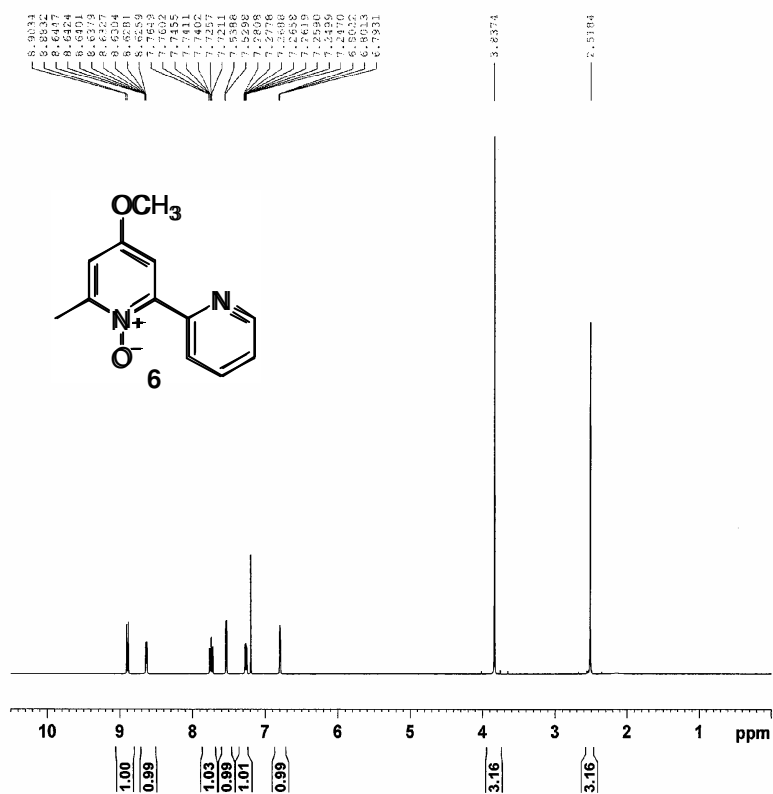




```

NAME      ZF3-17
EXPNO     1
PROCNO    1
Date_     20090717
Time      9.15
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         203
DW         60.800 usec
DE         6.50 usec
TE         303.6 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.20 usec
PL1        -1.00 dB
PL1W       13.18669796 W
SFO1       400.1724712 MHz
SI         32768
SF         400.1700032 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



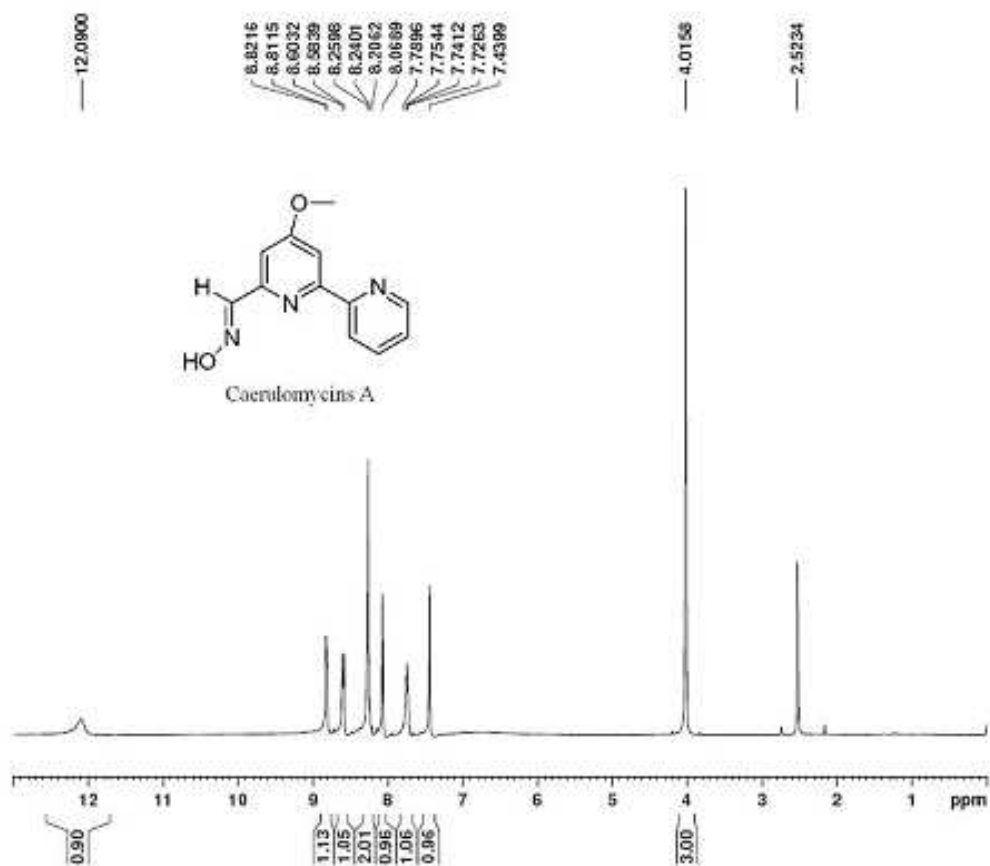
```

Current Data Parameters
NAME      mqzTR-OCH3
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20080516
Time      16.03
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         203
DW         60.800 usec
DE         6.50 usec
TE         301.2 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         13.50 usec
PL1        -1.00 dB
SFO1       400.1724712 MHz

F2 - Processing parameters
SI         32768
SF         400.1700286 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

NAME 2F11-28-2
EXPNO 1
PROCNO 1
Date_ 20100407
Time 9.57
INSTRUM spect
PROBHD 5 mm DABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9946387 sec
RG 181
DM 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.20 usec
PL1 -1.00 dB
PL1W 13.16669796 W
SFO1 400.1724112 MHz
SI 32768
SF 400.1699851 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00