

**Manganese Catalyzed
Cross-Coupling Reactions of Nitrogen Nucleophiles
with Aryl Halides in Water**

Supporting Information

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General Methods

Chemicals and solvents were either purchased from commercial suppliers or purified by standard techniques. Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Flash chromatography was performed using Merck silica gel 60 with AR grade solvents. Proton nuclear magnetic resonance spectra (^1H NMR) were recorded on a Bruker Avance DPX 400 spectrophotometer (CDCl_3 as solvent). Chemical shifts for ^1H NMR spectra are reported as δ in units of parts per million (ppm) downfield from SiMe_4 (δ 0.0) and relative to the signal of chloroform-*d* (δ 7.2600, singlet). Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from SiMe_4 (δ 0.0) and relative to the signal of chloroform-*d* (δ 77.03, triplet).

All reagents were purchased from commercial supplier and were used without any purification.

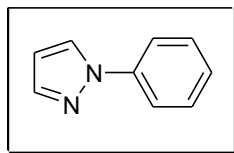
$\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$, (Sigma Aldrich, $\geq 99\%$ purity) was purchased from Sigma Aldrich. The N-arylated products are all known compounds that exhibited spectroscopic data identical to those reported in the literature¹⁻⁷ except for **6(b)** and **6(c)**.

General procedure for N-arylation of nitrogen nucleophiles

The N-nucleophile (1.47 mmol), $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ (Sigma-Aldrich, $\geq 99\%$ purity, 0.147 mmol), $\text{K}_3\text{PO}_4 \cdot \text{H}_2\text{O}$ (2.94 mmol), the aryl halide (2.21 mmol), *trans*-1,2-diaminocyclohexane **L3** (0.294 mmol) and water (0.75 mL) were added to a reaction vial and a screw cap was fitted to it. The reaction mixture was stirred under air in a closed system at 130°C for 24 h, then the heterogeneous mixture was cooled to RT and diluted with dichloromethane. The resulting solution was directly filtered through a pad of Celite. The combined organic extracts were dried with anhydrous MgSO_4 and the solvent was removed under reduced pressure. The crude product was purified by silica-gel column chromatography to afford the N-arylated product. The identity and purity of known products was confirmed by ^1H and ^{13}C NMR spectroscopic analysis.

¹H and ¹³C NMR data of N-arylated products

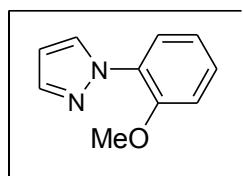
1-Phenyl-1H-pyrazole (3a)^{1,2,3}. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and iodobenzene (0.25 mL, 2.21 mmol) provided 165 mg (78% yield) of the coupling product as a yellowish oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



¹H-NMR (400 MHz, CDCl₃) δ 7.93 (dd, J = 2.3, 0.5 Hz, 1H), 7.69-7.73 (m, 3H), 7.44-7.48 (m, 2H), 7.26-7.31 (m, 1H), 6.47 (dd, J = 2.5, 1.8 Hz, 1H).

¹³C-NMR (100 MHz, CDCl₃) δ 141.1, 140.2, 129.2, 126.9, 126.3, 118.9, 107.4.

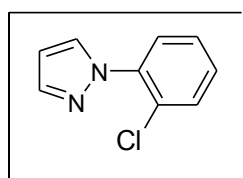
1-(2-Methoxyphenyl)-1H-pyrazole (3b)^{1,2,3}. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 2-iodoanisole (0.29 mL, 2.21 mmol) provided 38 mg (16% yield) of the coupling product as a yellowish oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



¹H-NMR (400 MHz, CDCl₃) δ 8.03 (d, J = 2.3 Hz, 1H), 7.71-7.73 (m, 2H), 7.28-7.32 (m, 1H), 7.04-7.08 (m, 2H), 6.42-6.43 (m, 1H), 3.84 (s, 3H).

¹³C-NMR (100 MHz, CDCl₃) δ 150.9, 140.0, 131.7, 129.4, 127.8, 125.1, 120.9, 112.6, 106.3, 55.8.

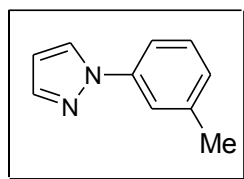
1-(2-Chlorophenyl)-1H-pyrazole (3c)^{1,2,3}. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 1-chloro-2-iodobenzene (0.27 mL, 2.21 mmol) provided 25 mg (10% yield) of the coupling product as a yellowish oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



^1H -NMR (300 MHz, CDCl_3) δ 7.88 (dd, J = 2.3, 0.5 Hz, 1H), 7.75 (d, J = 1.2 Hz, 1H), 7.57-7.60 (m, 1H), 7.51-7.53 (m, 1H), 7.31-7.40 (m, 2H), 6.47-6.48 (m, 1H).

^{13}C -NMR (100 MHz, CDCl_3) δ 140.9, 138.4, 131.3, 130.8, 128.9, 128.4, 127.8, 127.5, 106.7.

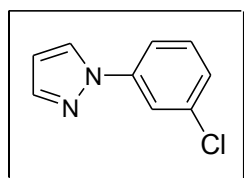
1-(3-Methylphenyl)-1H-pyrazole (3d)^{1,2,3}. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 3-iodotoluene (0.28 mL, 2.21 mmol) provided 175 mg (75% yield) of the coupling product as a yellowish oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



^1H -NMR (400 MHz, CDCl_3) δ 7.91 (d, J = 2.3 Hz, 1H), 7.72 (d, J = 1.2 Hz, 1H), 7.56 (s, 1H), 7.46-7.48 (m, 1H), 7.32 (t, J = 7.7 Hz, 1H), 7.11 (d, J = 7.5 Hz, 1H), 6.46 (t, J = 2.1 Hz, 1H), 2.43 (s, 3H).

^{13}C -NMR (100 MHz, CDCl_3) δ 140.7, 140.0, 139.3, 129.1, 127.6, 126.7, 119.9, 116.2, 107.3, 21.7.

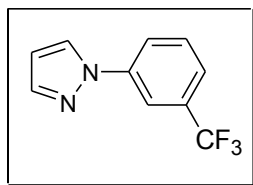
1-(3-Chlorophenyl)-1H-pyrazole (3e)^{1,2,3}. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 1-chloro-3-iodotoluene (0.27 mL, 2.21 mmol) provided 209 mg (80% yield) of the coupling product as a yellowish oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



^1H -NMR (400 MHz, CDCl_3) δ 7.91 (dd, J = 2.4, 0.5 Hz, 1H), 7.75 (d, J = 1.9 Hz, 1H), 7.73 (d, J = 1.5 Hz, 1H), 7.57-7.60 (m, 1H), 7.38 (t, J = 8.0 Hz, 1H), 7.25-7.27 (m, 1H), 6.48-6.49 (m, 1H).

^{13}C -NMR (100 MHz, CDCl_3) δ 141.7, 139.9, 135.3, 130.6, 126.7, 126.2, 119.0, 116.8, 108.2.

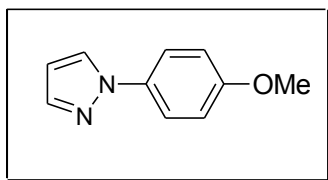
1-(3-Trifluoromethylphenyl)-1H-pyrazole (3f)⁴. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 1-trifluoro-3-iodobenzene (0.33 mL, 2.21 mmol) provided 230 mg (74% yield) of the coupling product as a colourless oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.98 (s, 1H), 7.91 (d, $J = 2.4$ Hz, 1H), 7.81-7.82 (m, 1H), 7.72 (s, 1H), 7.47-7.52 (m, 2H), 6.45-6.46 (m, 1H).

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 141.7, 140.5, 130.0, 126.7, 125.1, 122.8, 122.7, 121.8, 116.0, 108.3.

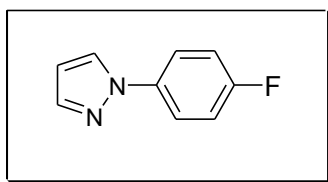
1-(4-Methoxyphenyl)-1H-pyrazole (3g)^{1,2,3}. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 4-iodoanisole (510 mg, 2.21 mmol) provided 172 mg (67% yield) of the coupling product as a yellowish oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.82 (dd, $J = 2.2, 0.5$ Hz, 1H), 7.68-7.69 (m, 1H), 7.57-7.60 (m, 2H), 6.92-6.96 (m, 2H), 6.43-6.44 (m, 1H, H-8), 3.84 (s, 3H).

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 158.2, 140.5, 134.2, 126.9, 120.9, 114.7, 107.2, 55.7.

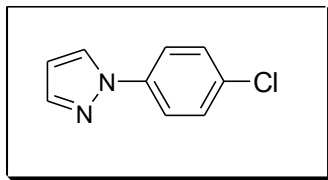
1-(4-Fluorophenyl)-1H-pyrazole (3h)^{1,2,3}. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 1-fluoro-4-iodobenzene (0.25 ml, 2.21 mmol) provided 174 mg (73% yield) of the coupling product as a yellowish oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.85-7.86 (m, 1H), 7.71 (d, $J = 1.0$ Hz, 1H), 7.63- 7.67 (m, 2H), 7.12-7.18 (m, 2H), 6.46 (dd, $J = 2.5, 1.9$ Hz, 1H).

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 160.3 (d, $J = 243.6$ Hz), 141.2, 136.6, 126.7, 121.0 (d, $J = 8.0$ Hz), 116.4 (d, $J = 22.4$ Hz), 107.9.

1-(4-Chlorophenyl)-1H-pyrazole (3i)^{1,2,3}. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 1-chloro-4-iodobenzene (525 mg, 2.21 mmol) provided 226 mg (86% yield) of the coupling product as a yellowish oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



¹H-NMR (400 MHz, CDCl₃) δ 7.89-7.90 (m, 1H), 7.72 (d, J = 1.6 Hz, 1H), 7.62- 7.65 (m, 2H), 7.40-7.43 (m, 2H), 6.47-6.48 (m, 1H).

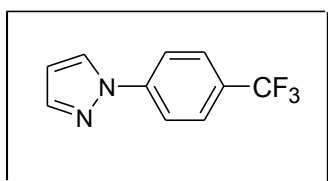
¹³C-NMR (100 MHz, CDCl₃) δ 141.4, 138.4, 132.0, 129.6, 126.8, 120.5, 107.8.

1-(4-Bromophenyl)-1H-pyrazole (3j)³. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 1-bromo-4-iodobenzene (625 mg, 2.21 mmol) provided 312 mg (75% yield) of the coupling product as a yellowish oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.

¹H-NMR (400 MHz, CDCl₃) δ 7.90-7.91 (m, 1H), 7.72-7.73 (m, 1H), 7.56- 7.61 (m, 4H), 6.47-6.48 (m, 1H).

¹³C-NMR (100 MHz, CDCl₃) δ 141.8, 139.6, 132.8, 127.0, 121.0, 120.0, 108.4.

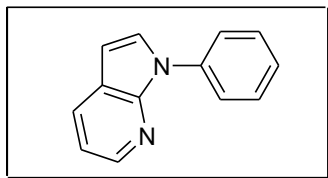
1-(4-Trifluoromethylphenyl)-1H-pyrazole (3k)¹. Following the general procedure using 1H-pyrazole (100 mg, 1.47 mmol) and 1-trifluoro-4-iodobenzene (0.33 mL, 2.21 mmol) provided 216 mg (70% yield) of the coupling product as a white solid after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



¹H-NMR (400 MHz, CDCl₃) δ 7.99 (d, J = 2.5 Hz, 1H), 7.82 (d, J = 8.7, 2H), 7.77 (s, 1H), 7.72 (d, J = 8.7, 2H), 6.52-6.53 (m, J = 2.2, 1H).

^{13}C -NMR (100 MHz, CDCl_3) δ 142.6.0, 141.9, 126.8, 126.7, 125.3, 122.6, 118.8, 108.5.

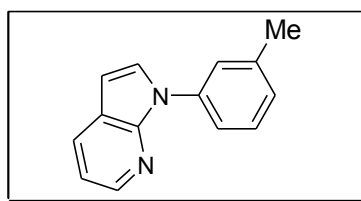
1-Phenyl-7-aza-1*H*-indole (4a)^{1,2,3}. Following the general procedure using 7-azaindole (173 mg, 1.47 mmol) and iodobenzene (0.25 mL, 2.21 mmol) provided 249 mg (87% yield) of the coupling product as a yellow oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



^1H -NMR (400 MHz, CDCl_3) δ 8.38 (dd, J = 4.6, 1.2 Hz, 1H), 7.98 (dd, J = 7.8, 1.4 Hz, 1H), 7.76-7.78 (m, 2H), 7.51-7.55 (m, 3H), 7.34 (t, J = 7.4 Hz, 1H), 7.15 (dd, J = 7.8, 4.7 Hz, 1H), 6.63 (d, J = 3.6 Hz, 1H).

^{13}C -NMR (100 MHz, CDCl_3) δ 147.6, 143.6, 138.6, 129.4, 129.3, 127.9, 126.4, 124.1, 121.5, 116.7, 101.9.

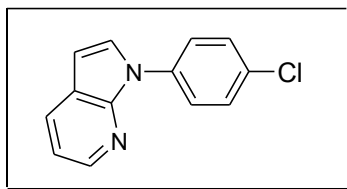
1-(3-Methylphenyl)-7-aza-1*H*-indole (4b)^{2,3}. Following the general procedure using 7-azaindole (173 mg, 1.47 mmol) and 3-iodotoluene (0.28 mL, 2.21 mmol) provided 280 mg (91% yield) of the coupling product as a yellow oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



^1H -NMR (400 MHz, CDCl_3) δ 8.37 (dd, J = 4.6, 1.3 Hz, 1H), 7.97 (dd, J = 7.8, 1.4 Hz, 1H), 7.54-7.55 (m, 2H), 7.49 (d, J = 3.6 Hz, 1H), 7.43 (t, J = 7.7 Hz, 1H), 7.11-7.17 (m, 2H), 6.61 (d, J = 3.6 Hz, 1H).

^{13}C -NMR (100 MHz, CDCl_3) δ 147.4, 143.5, 139.2, 138.3, 129.1, 129.0, 128.0, 127.2, 124.7, 121.5, 121.2, 116.5, 101.3, 21.5.

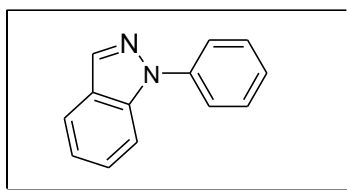
1-(4-Chlorophenyl)-7-aza-1*H*-indole (4c)⁵. Following the general procedure using 7-azaindole (173 mg, 1.47 mmol) and 1-chloro-4-iodobenzene (527 mg, 2.21 mmol) provided 259 mg (77% yield) of the coupling product as a yellowish solid after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 8.37 (d, J = 4.6 Hz, 1H), 7.97-7.99 (m, 1H), 7.74 (d, J = 8.7 Hz, 2H), 7.48-7.50 (m, 3H), 7.13-7.17 (m, 1H), 6.64-6.65 (d, J = 3.7 Hz, 1H).

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 147.4, 143.8, 137.0, 131.7, 129.5, 129.3, 127.5, 125.0, 121.6, 117.0, 102.2.

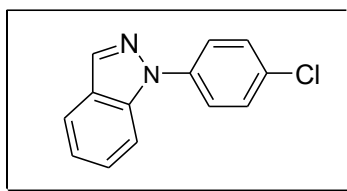
1-Phenyl-1H-indazole (5a)⁶. Following the general procedure using 1H-indazole (100 mg, 0.847 mmol) and iodobenzene (0.14 mL, 1.27 mmol) provided 147 mg (90% yield) of the coupling product as a yellowish solid after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 8.24 (s, 1H), 7.83 (t, J = 9.1 Hz, 2H), 7.77 (d, J = 7.8 Hz, 2H), 7.57 (t, J = 7.9 Hz, 2H), 7.46 (t, J = 7.7 Hz, 1H), 7.39 (t, J = 7.4 Hz, 1H), 7.27 (t, J = 7.6 Hz, 1H).

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 140.2, 138.8, 135.4, 129.5, 127.1, 126.6, 125.4, 122.8, 121.5, 121.3, 110.4.

1-(4-Chlorophenyl)-1H-indazole (5b)⁷. Following the general procedure using 1H-indazole (100 mg, 0.847 mmol) and 1-chloro-4-iodobenzene (303 mg, 1.27 mmol) provided 160 mg (82% yield) of the coupling product as a yellowish solid after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 8.18 (s, 1H), 7.77 (d, J = 8.1 Hz, 1H), 7.64-7.68 (m, 3H), 7.45-7.47 (d, J = 8.7, 2H), 7.41 (t, J = 7.8 Hz, 1H), 7.21 (t, J = 7.4 Hz, 1H).

^{13}C -NMR (100 MHz, CDCl_3) δ 138.7, 138.6, 135.7, 131.9, 125.5, 127.3, 125.4, 123.6, 121.7, 121.4, 110.1.

3-methyl-1-phenyl-1H-pyrazole (6a)³. Following the general procedure using 3-methyl-1H-pyrazole (180 mg, 2.16 mmol) and iodobenzene (0.365 mL, 3.24 mmol) provided 182 mg (78% yield) of the coupling product as a yellowish oil after purification by flash chromatography (95:5 hexane/ethyl acetate) of the crude brown oil.

^1H -NMR (400 MHz, CDCl_3) δ 7.80-7.81 (m, 1H), 7.66-7.68 (m, 2H), 7.41-7.45 (m, 2H), 7.24-7.25 (m, 1H), 6.25-6.26 (m, 1H), 2.41 (s, 3H).

^{13}C -NMR (100 MHz, CDCl_3) δ 150.5, 140.2, 129.4, 127.3, 125.9, 118.8, 107.5, 13.7.

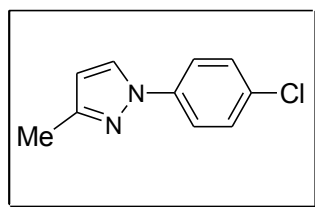
3-Methyl-1-Phenyl-1H-pyrazole (6b). Following the general procedure using 3-methyl-1H-pyrazole (0.12 mL, 1.47 mmol) and iodobenzene (0.25 mL, 2.21 mmol) provided 190 mg (75% yield) of the coupling product as a yellow solid after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



^1H -NMR (400 MHz, CDCl_3) δ 7.75 (s, 1H), 7.50 (s, 1H), 7.38 (d, J = 8.1 Hz 1H), 7.27 (t, J = 7.8 Hz, 1H), 7.02 (d, J = 7.5 Hz, 1H), 6.19-6.20 (m, 1H), 2.37 (s, 6H).

^{13}C -NMR (100 MHz, CDCl_3) δ 150.2, 140.1, 138.7, 129.0, 127.3, 126.6, 119.5, 115.7, 107.3, 21.3, 13.7.

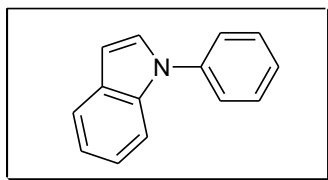
3-Methyl-1-(4-Chlorophenyl)-1H-pyrazole (6c). Following the general procedure using 3-methyl-1H-pyrazole (0.12 mL, 1.47 mmol) and 1-chloro-4-iodobenzene (527mg, 2.21 mmol) provided 203 mg (72% yield) of the coupling product as a yellowish solid after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.75 (d, J = 2.3 Hz, 1H), 7.56-7.59 (m, 2H), 7.36-7.39 (m, 2H), 6.23-6.24 (m, 1H), 2.36 (s, 3H).

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 150.9, 138.8, 131.2, 129.5, 127.3, 119.9, 107.9, 13.7.

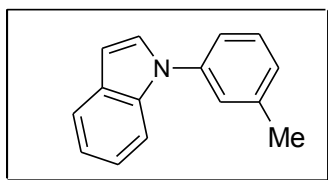
1-Phenyl-1H-indole (7a)^{1,2,3}. Following the general procedure using 1H-indole (172 mg, 1.47 mmol) and iodobenzene (0.25 mL, 2.21 mmol) provided 71 mg (25% yield) of the coupling product as a yellow oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.68 (d, J = 7.5 Hz 1H), 7.57 (d, J = 8.0 Hz 1H), 7.50-7.51 (m, 4H), 7.32-7.37 (m, 2H), 7.15-7.24 (m, 2H), 6.68 (d, J = 3.1 Hz, 1H).

$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 139.8, 136.0, 129.8, 129.7, 128.0, 126.6, 124.3, 122.4, 121.3, 120.5, 110.8, 103.8.

1-(3-Methylphenyl)-1H-indole (7b)^{2,3}. Following the general procedure using 1H-indole (172 mg, 1.47 mmol) and 3-iodotoluene (0.28 mL, 2.21 mmol) provided 58 mg (20% yield) of the coupling product as a yellow oil after purification by flash chromatography (98:2 hexane/ethyl acetate) of the crude oil.



$^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.71 (d, J = 7.7 Hz 1H), 7.59 (d, J = 8.1 Hz 1H), 7.40-7.42 (m, 1H), 7.32-7.36 (m, 3H), 7.17-7.24 (m, 3H), 6.68 (d, J = 3.1 Hz, 1H).

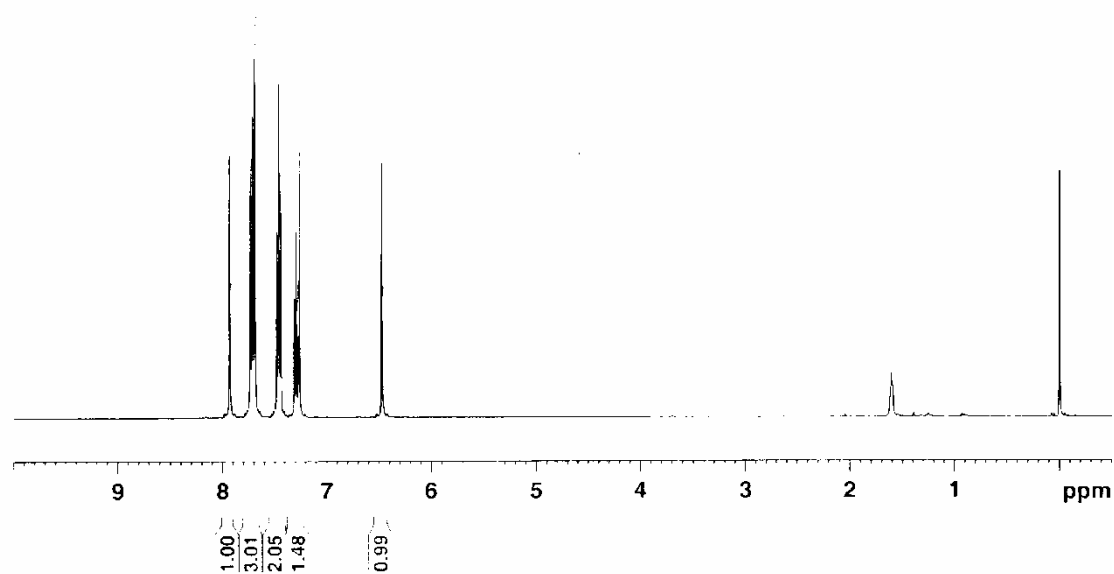
$^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 139.7, 139.6, 135.8, 129.4, 129.3, 128.0, 127.2, 125.0, 122.2, 121.4, 121.1, 120.2, 110.6, 103.3, 21.4.

References

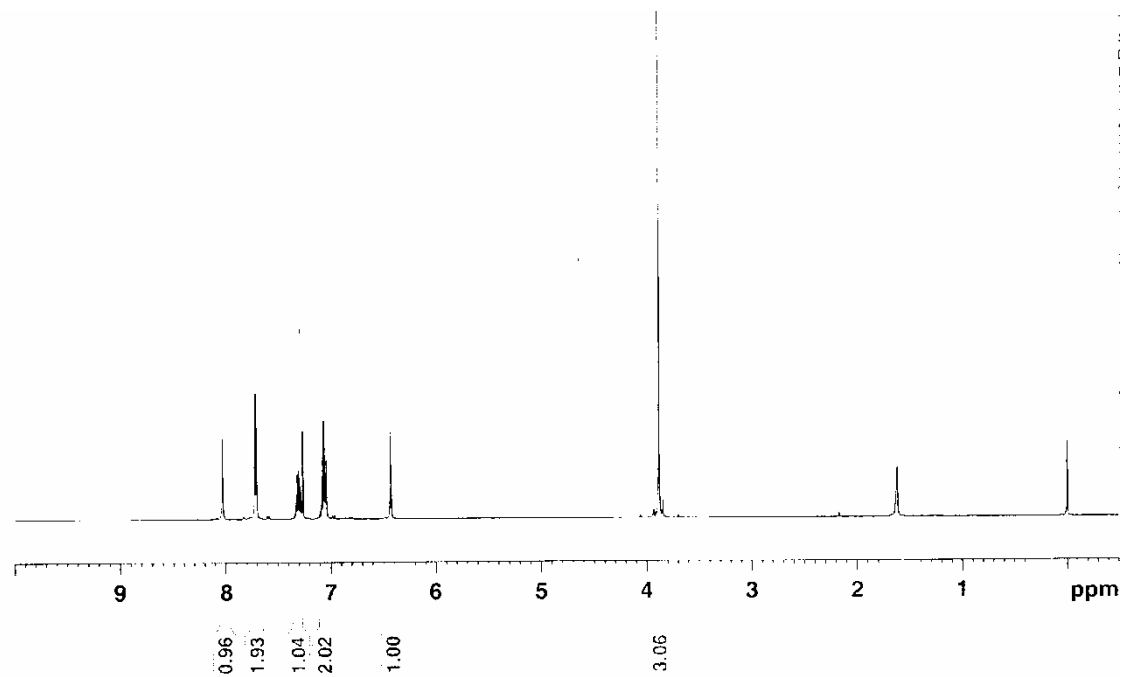
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¹H NMR spectroscopic data for N-arylated compounds

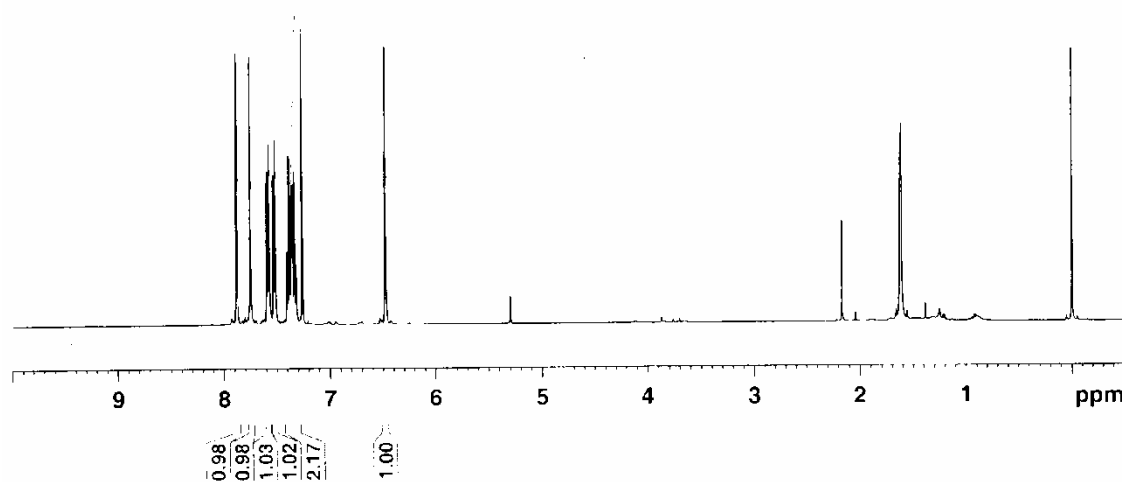
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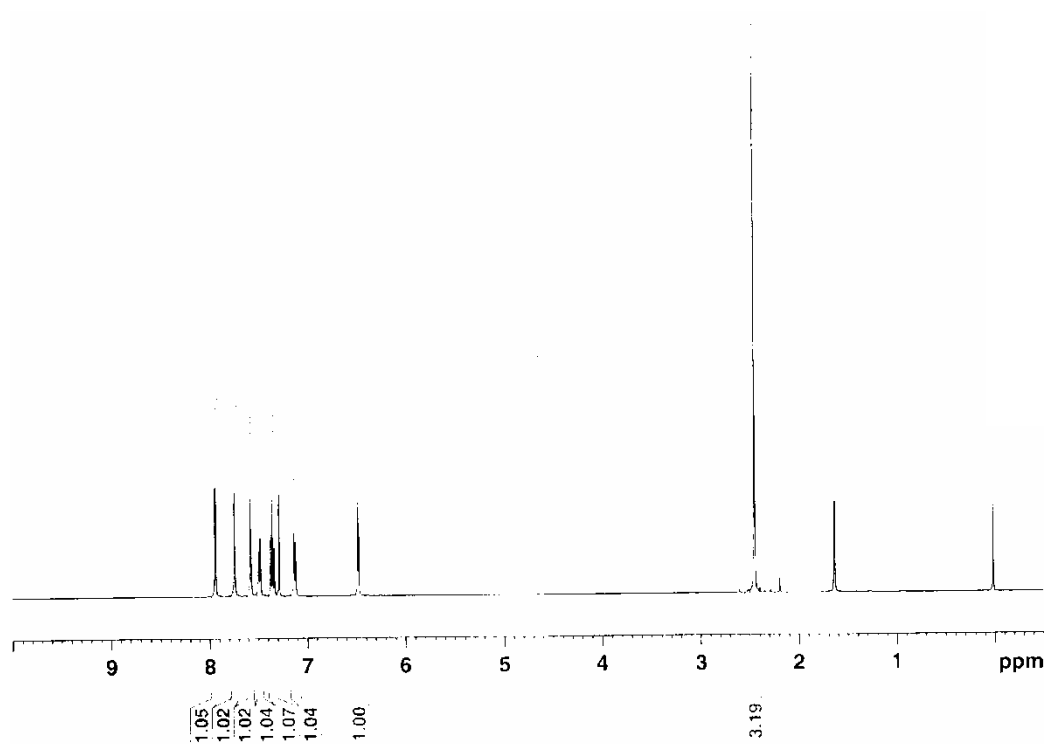
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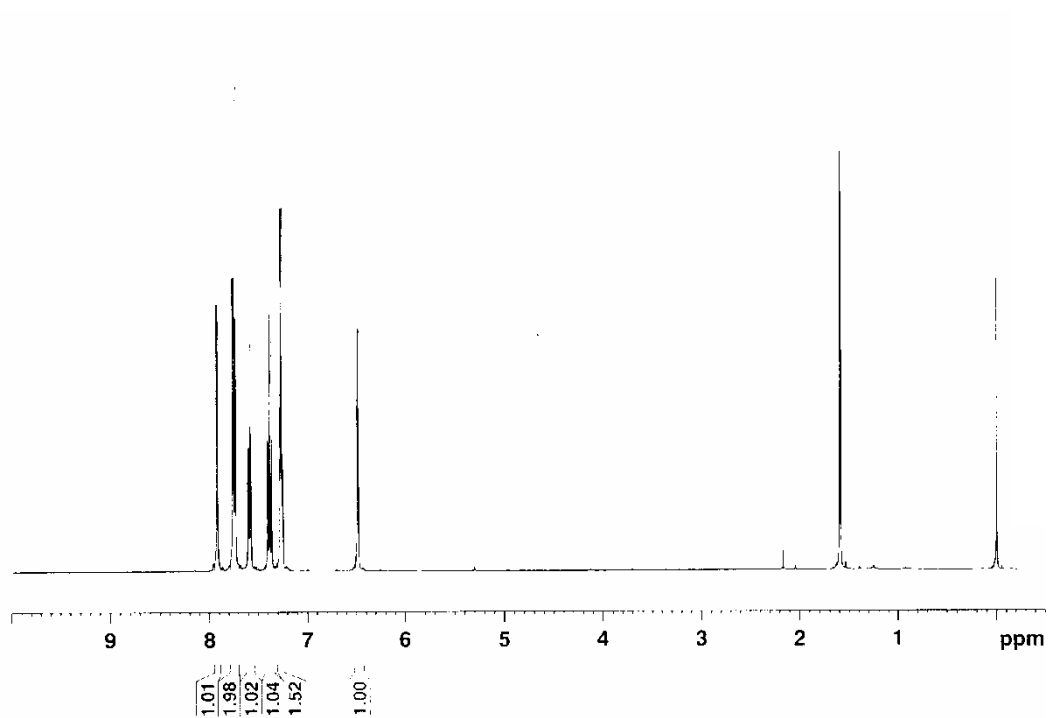
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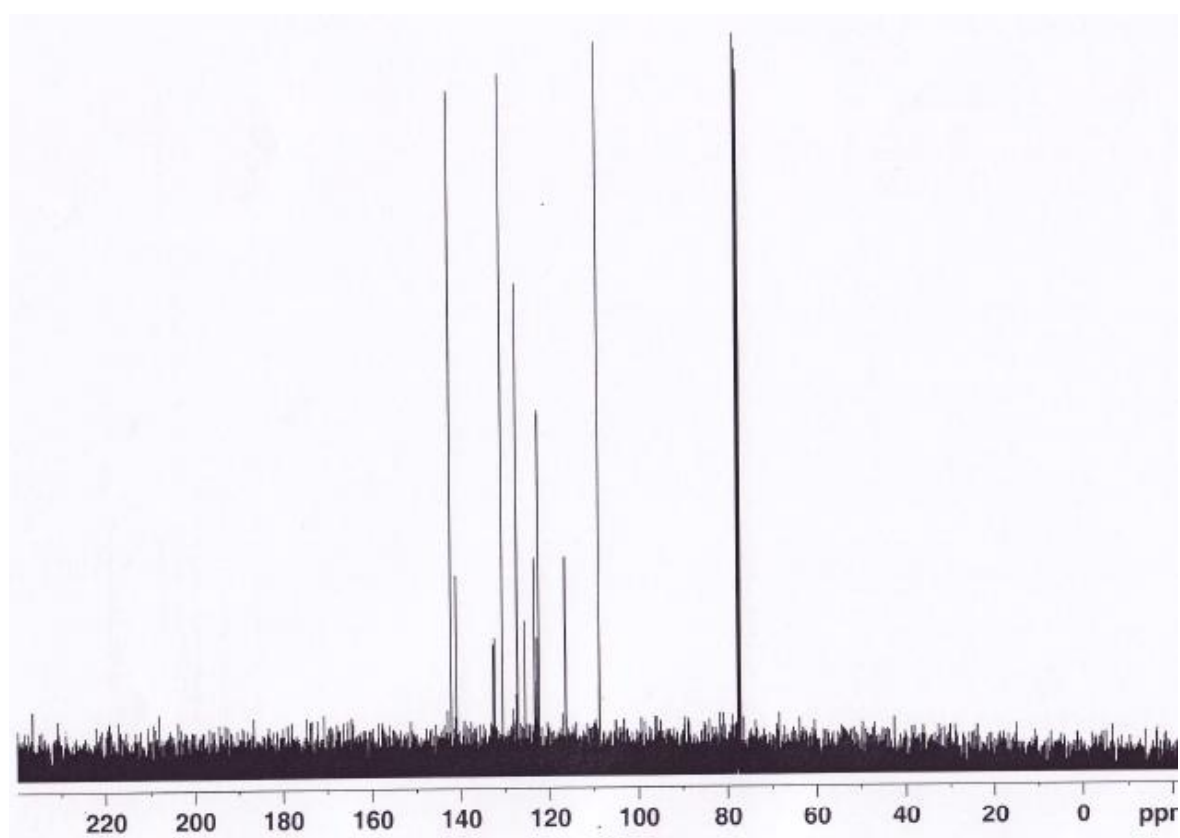
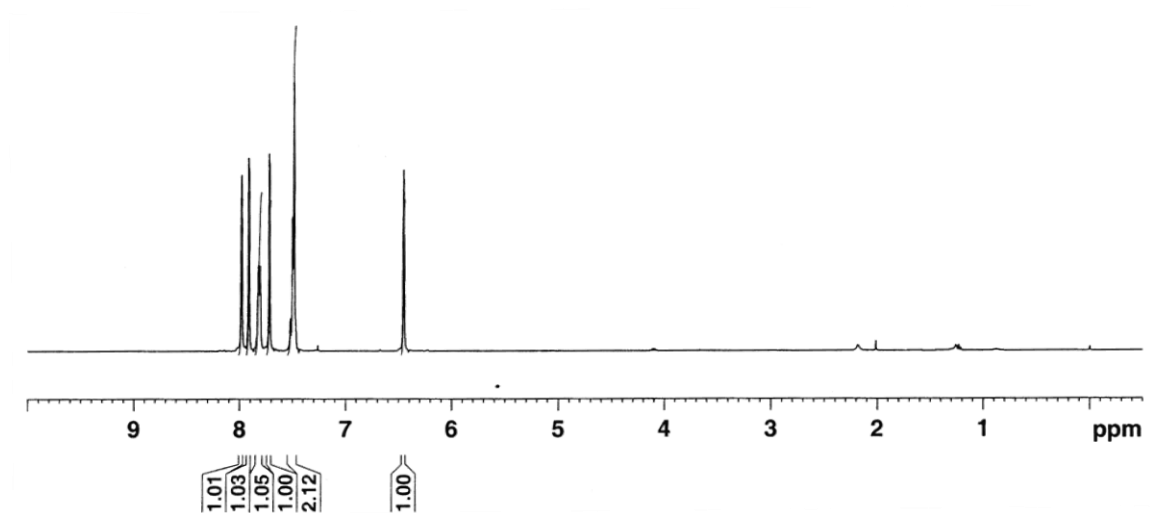
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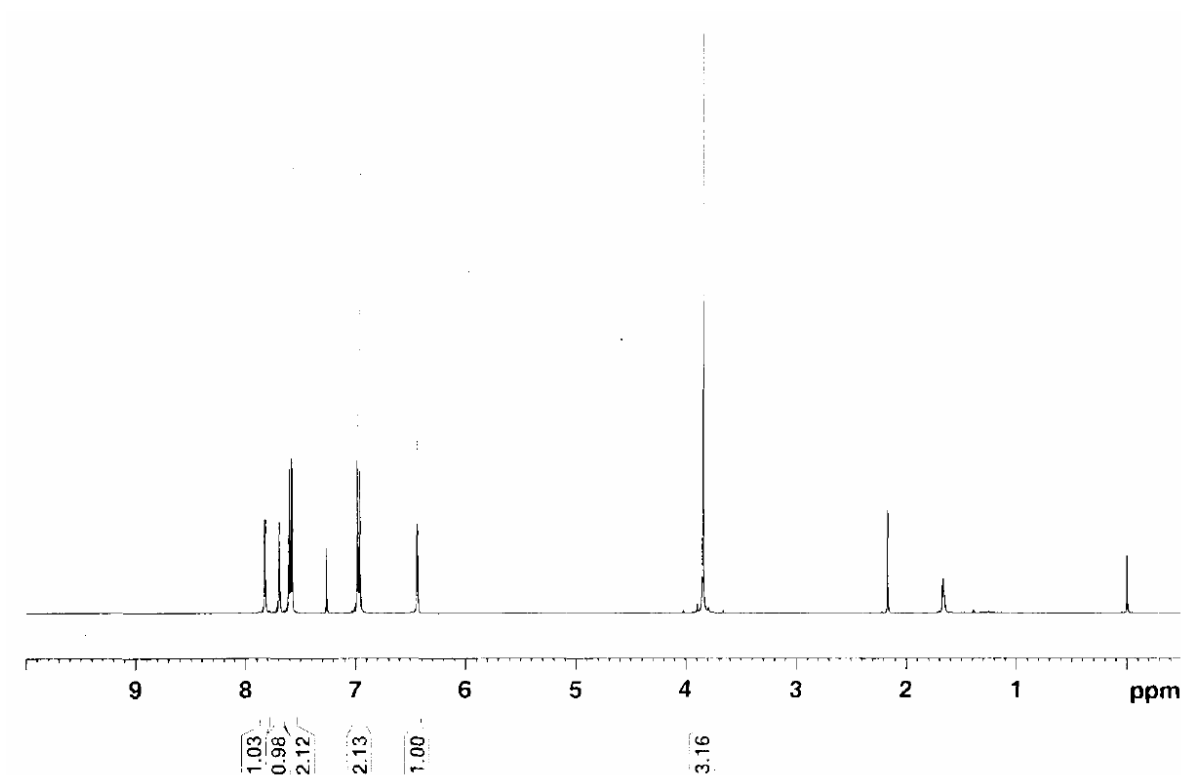
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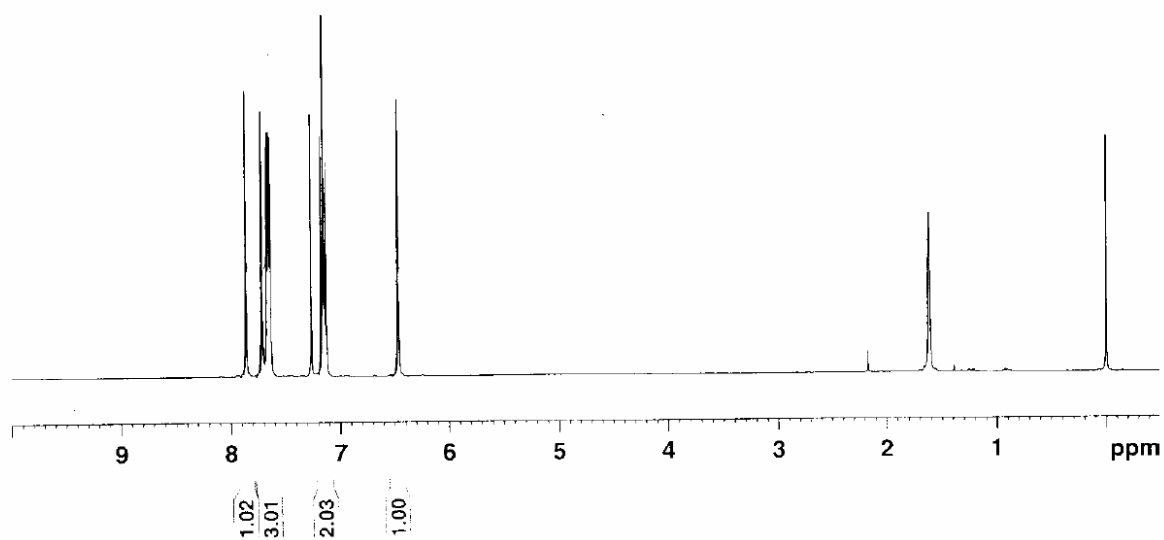
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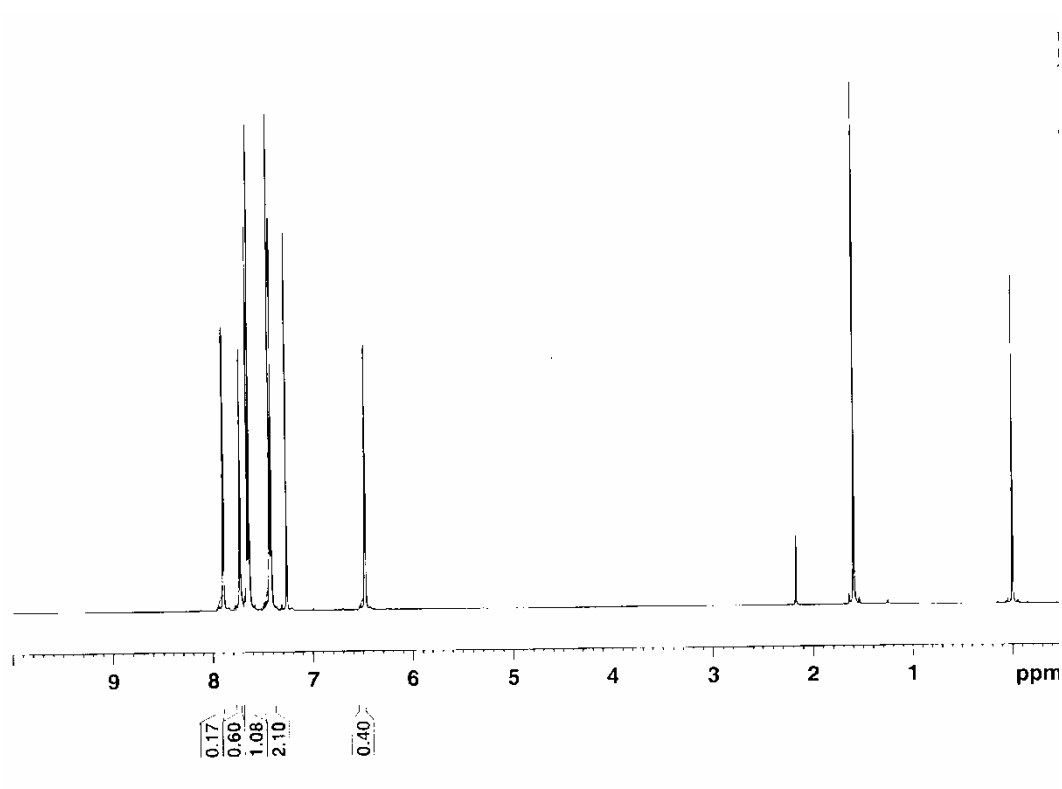
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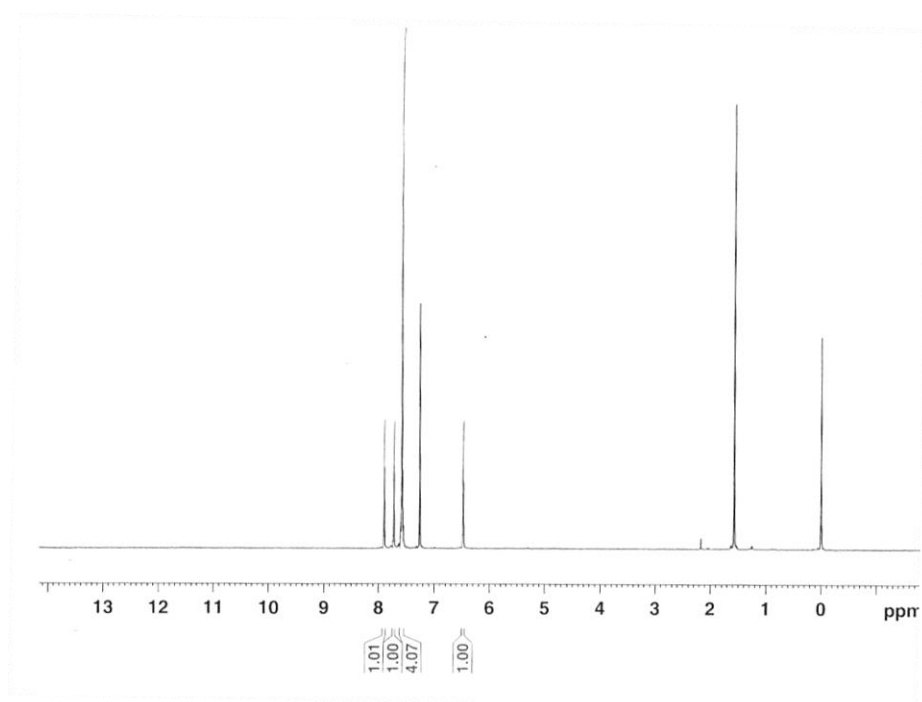
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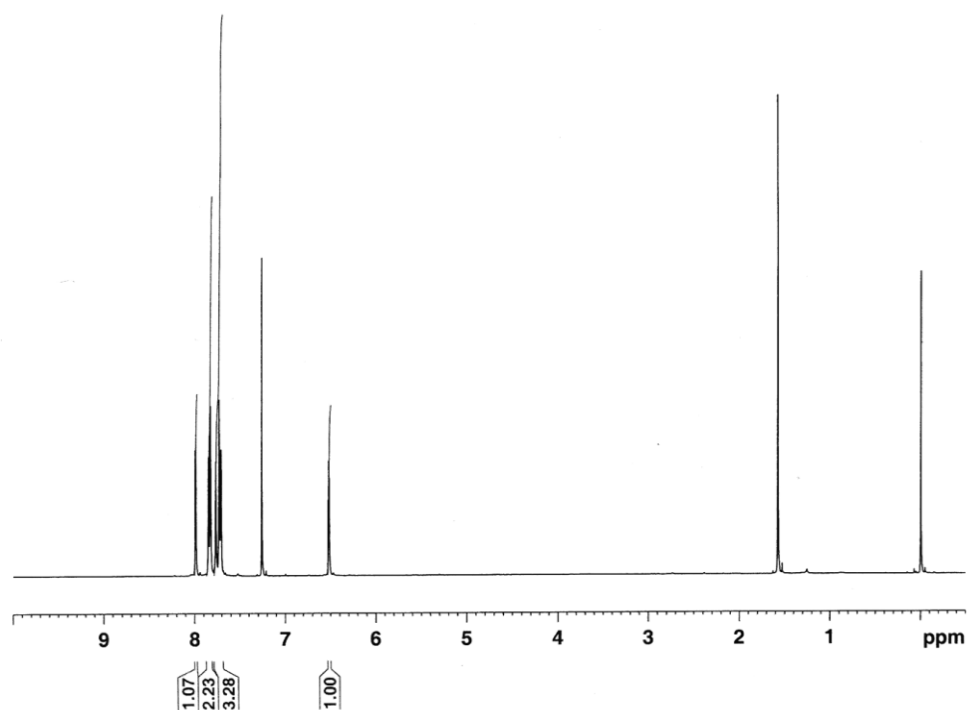
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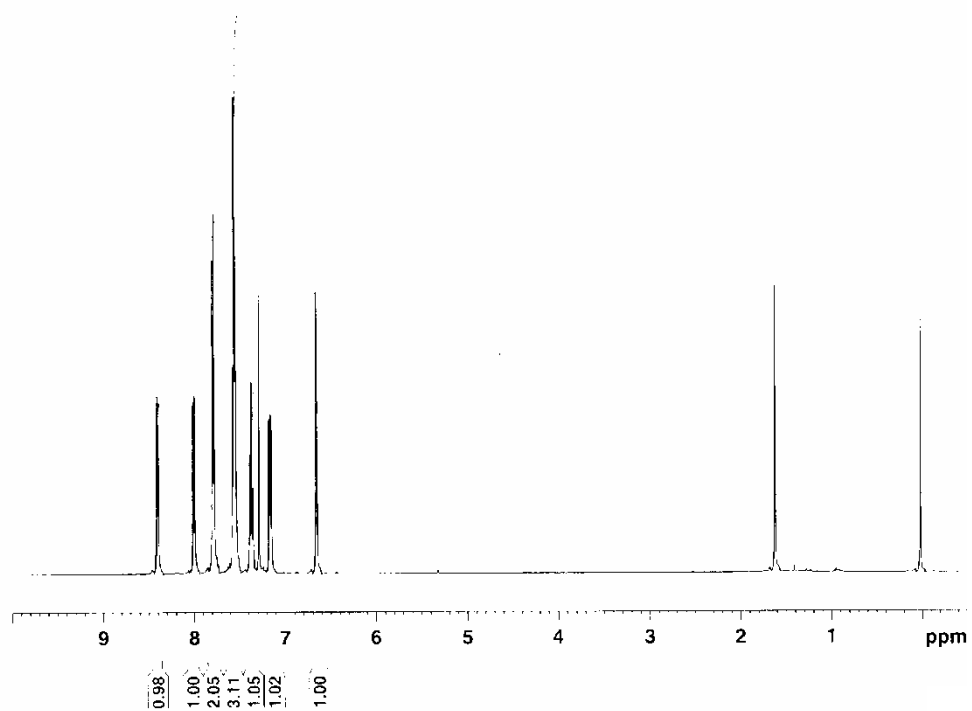
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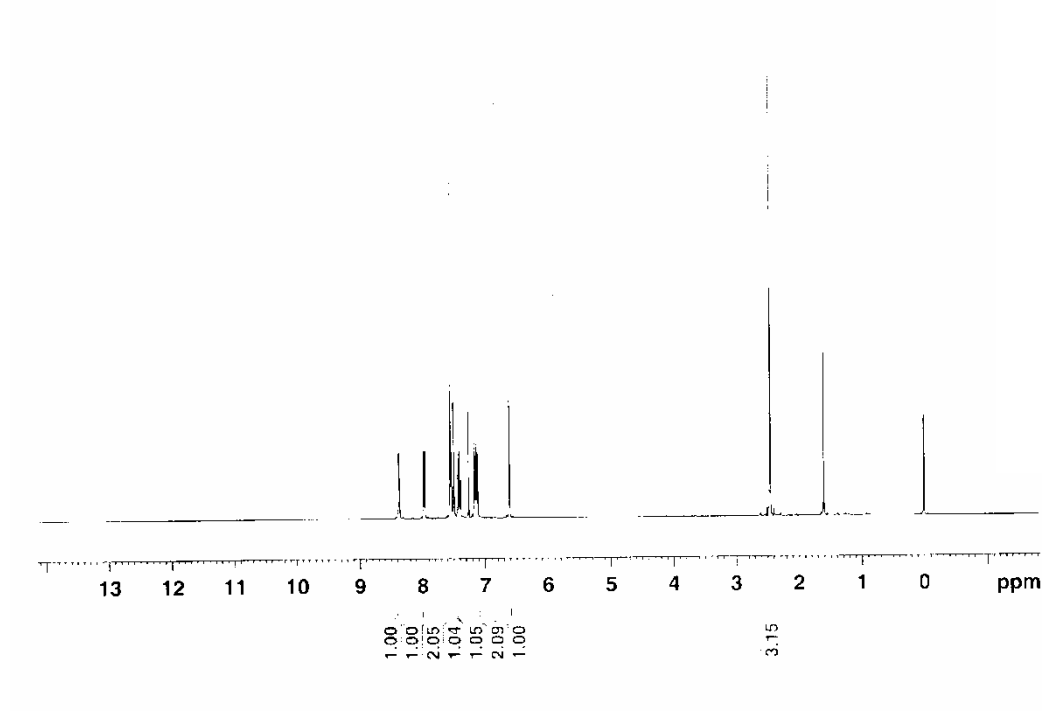
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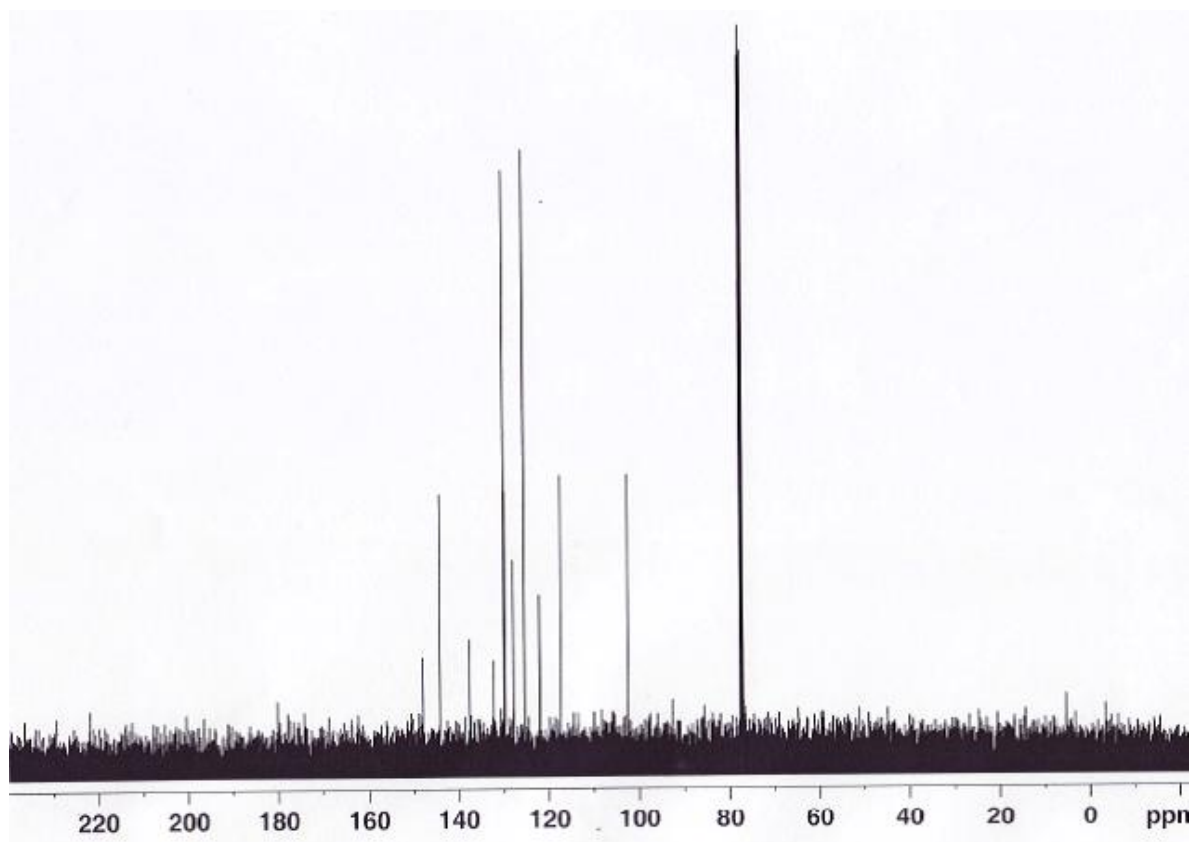
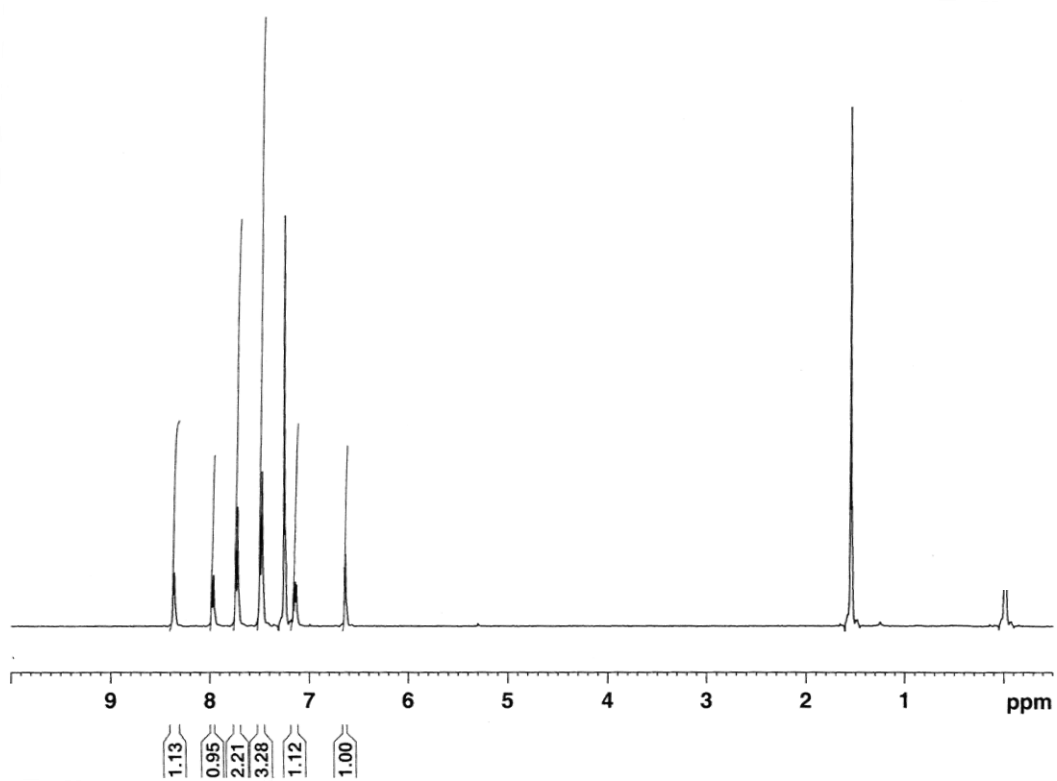
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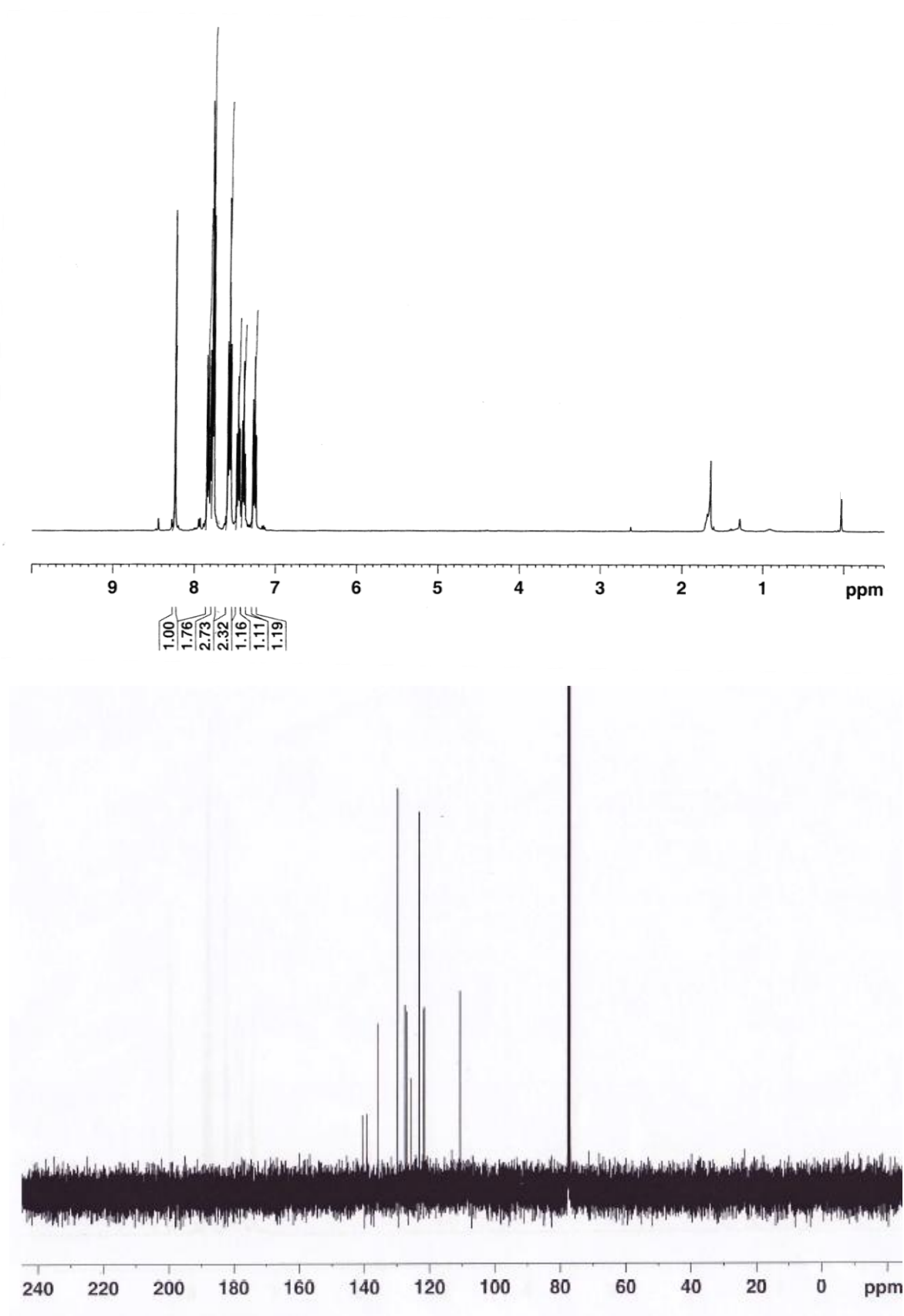
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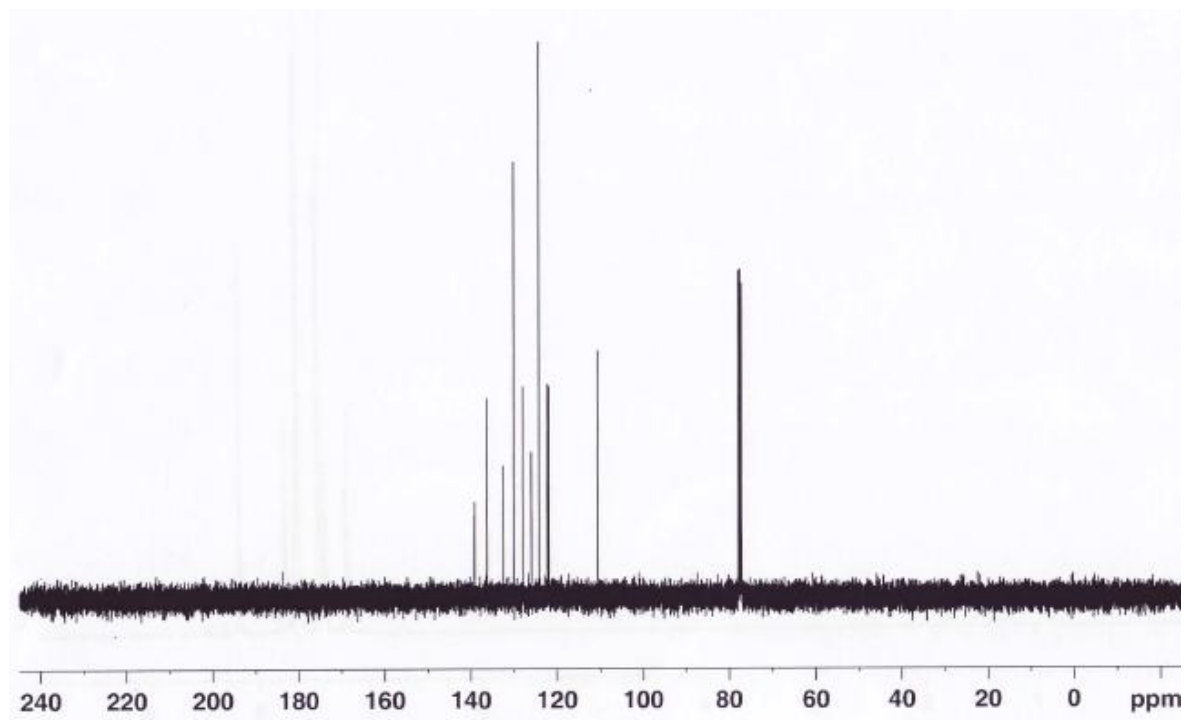
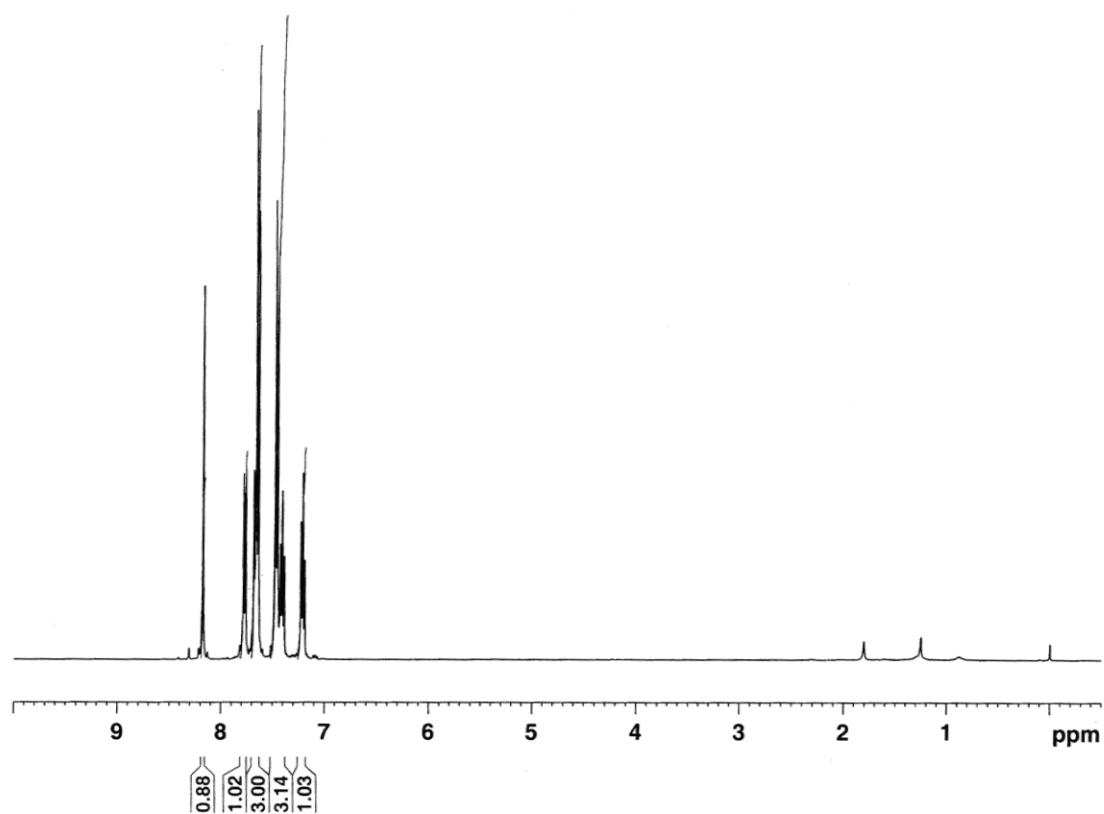
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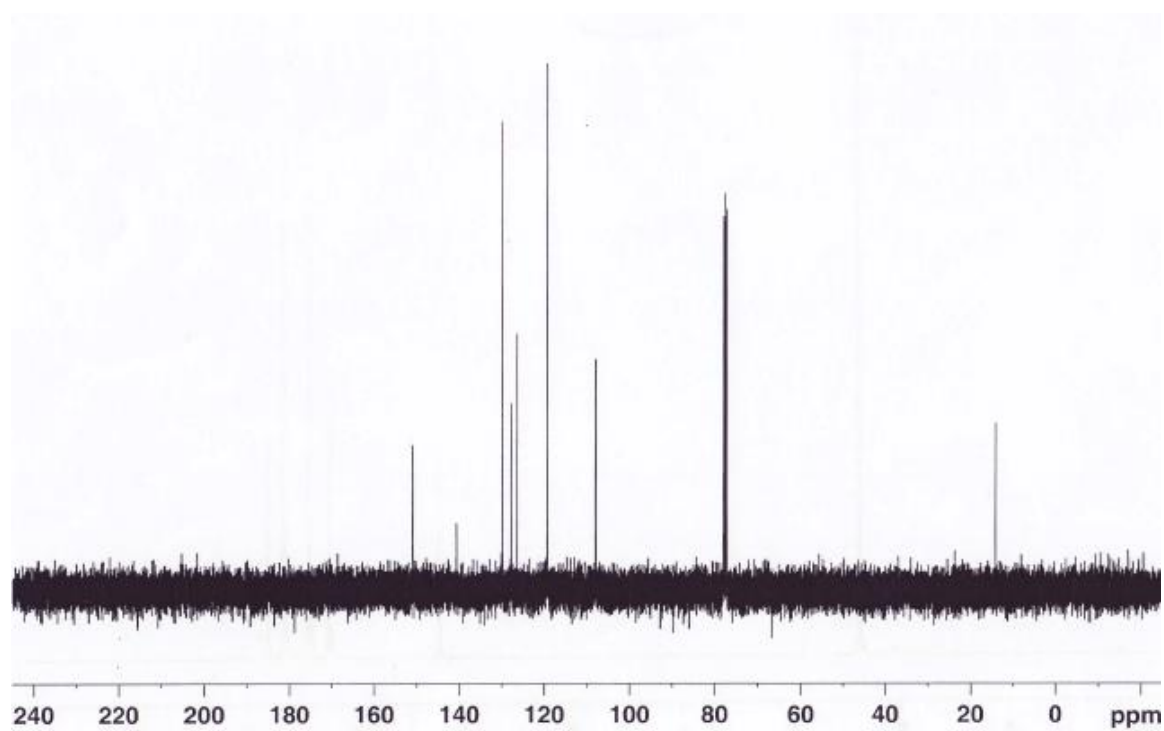
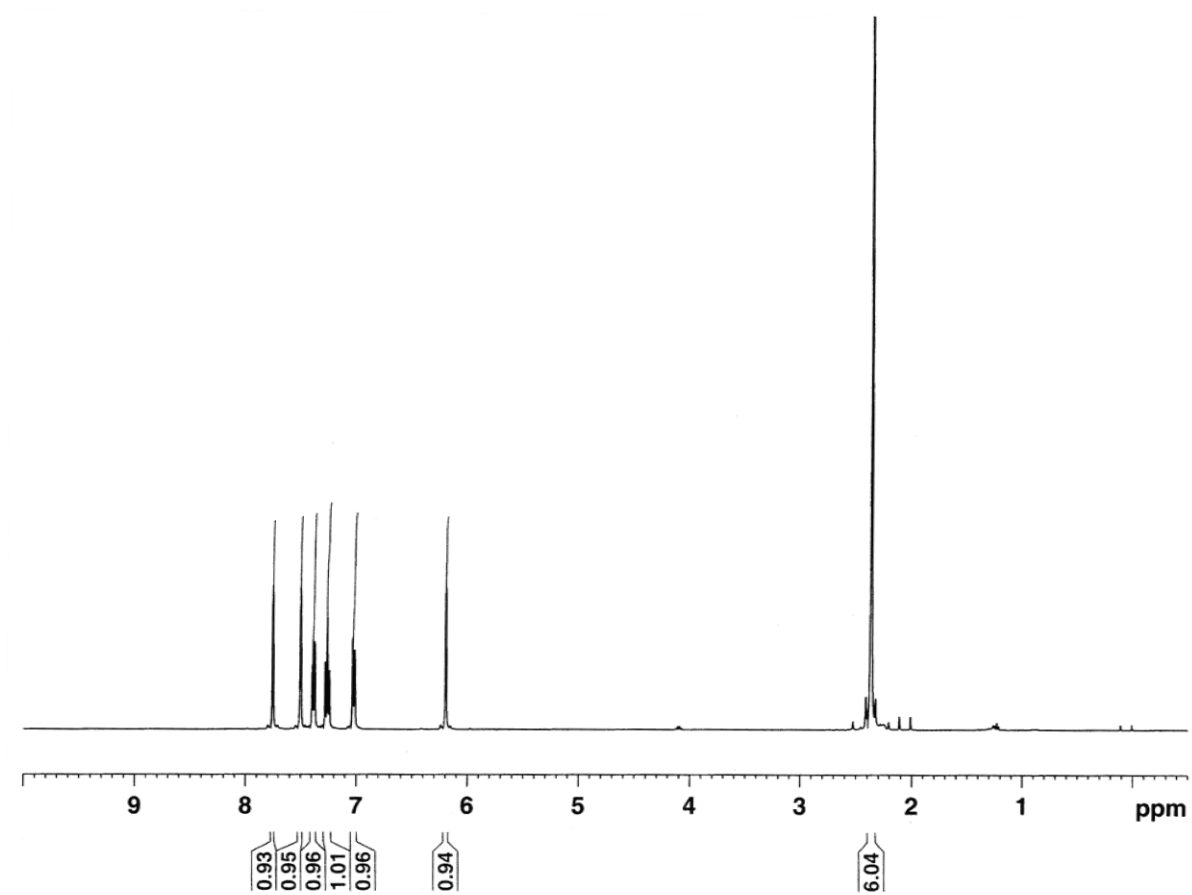
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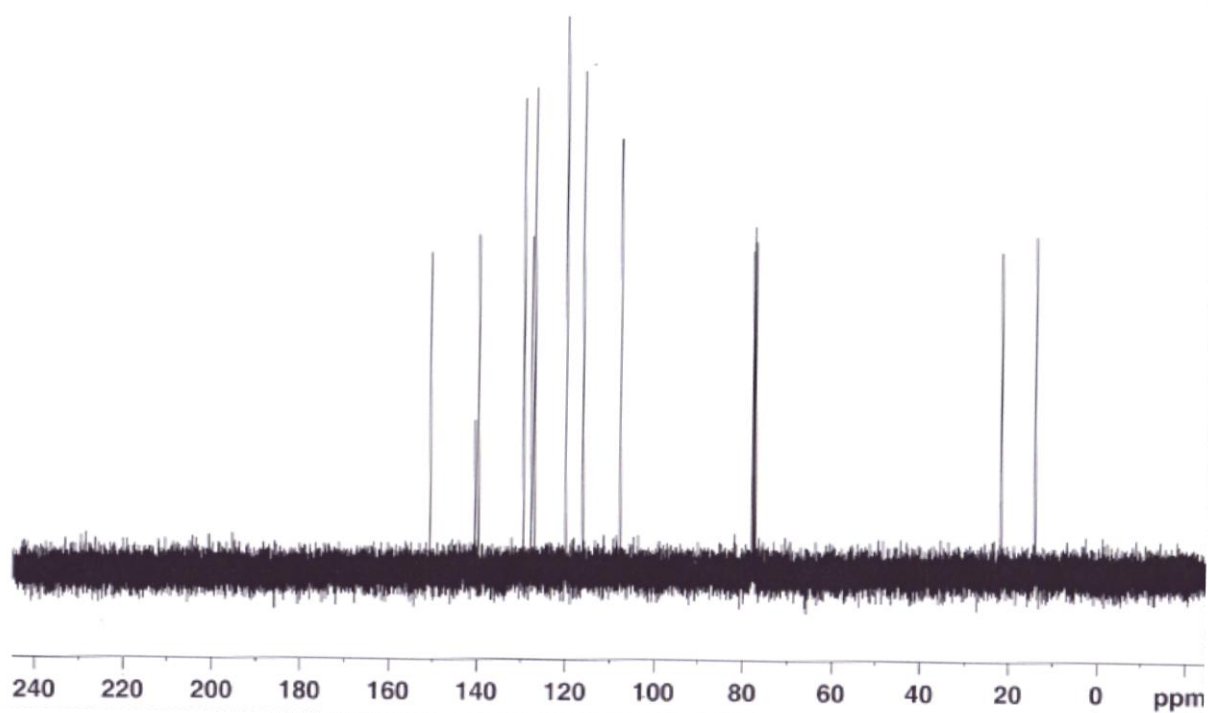
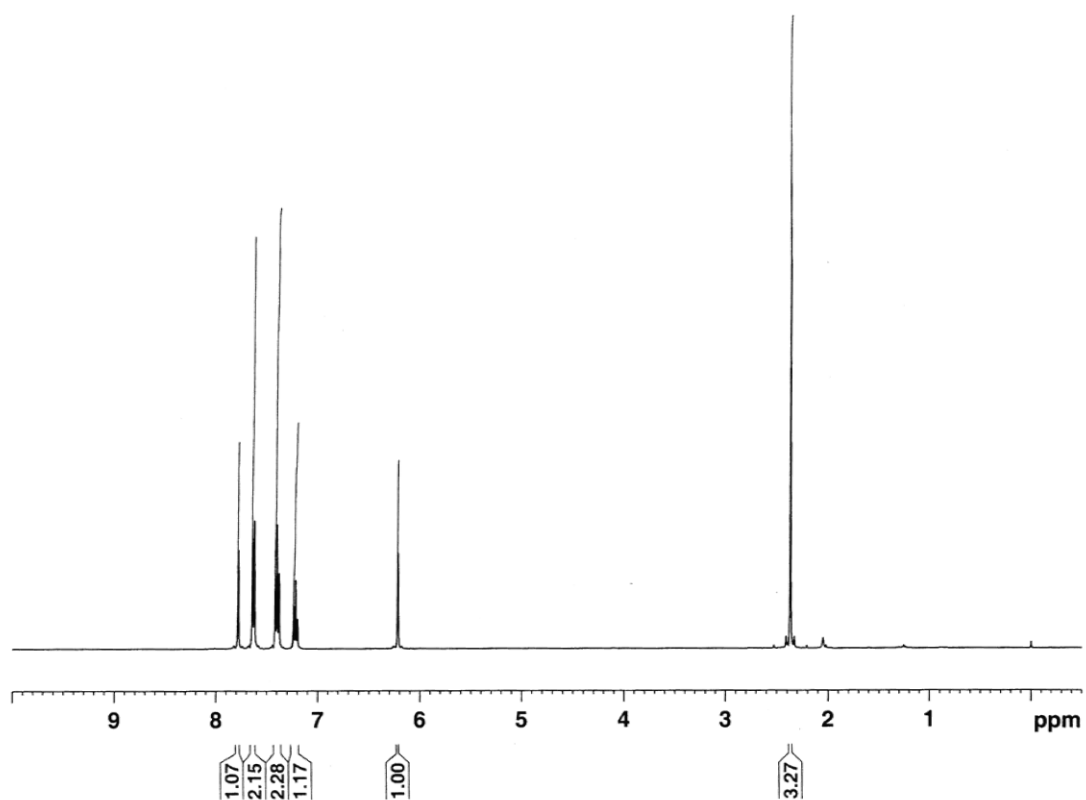
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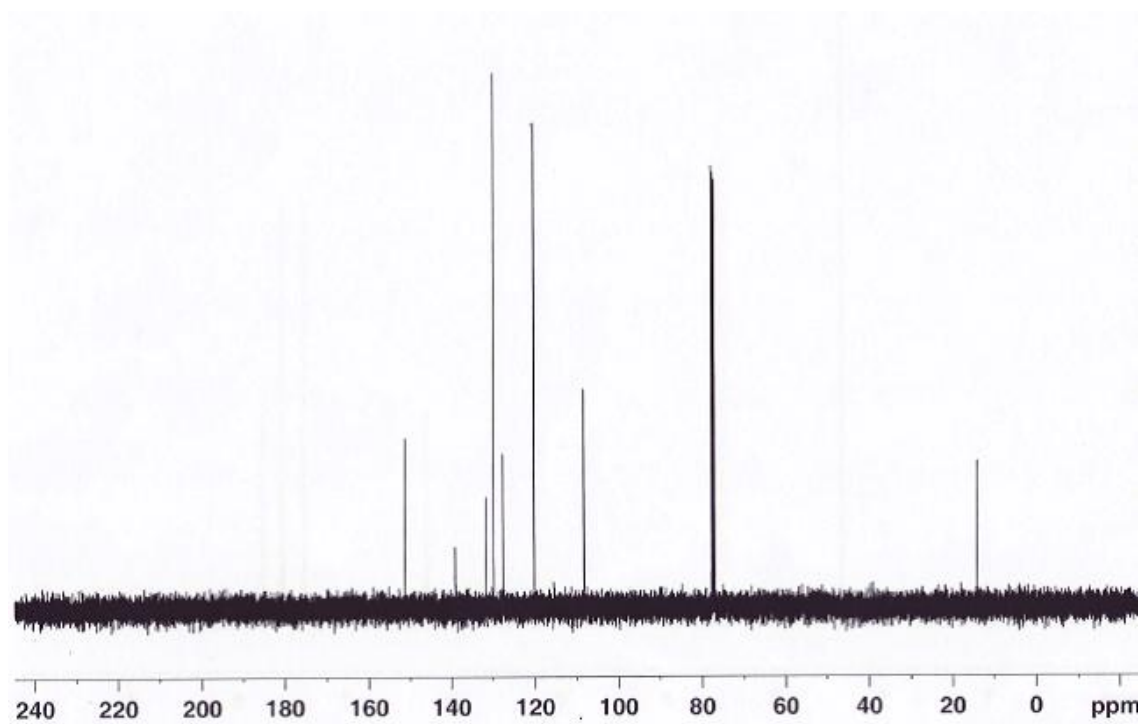
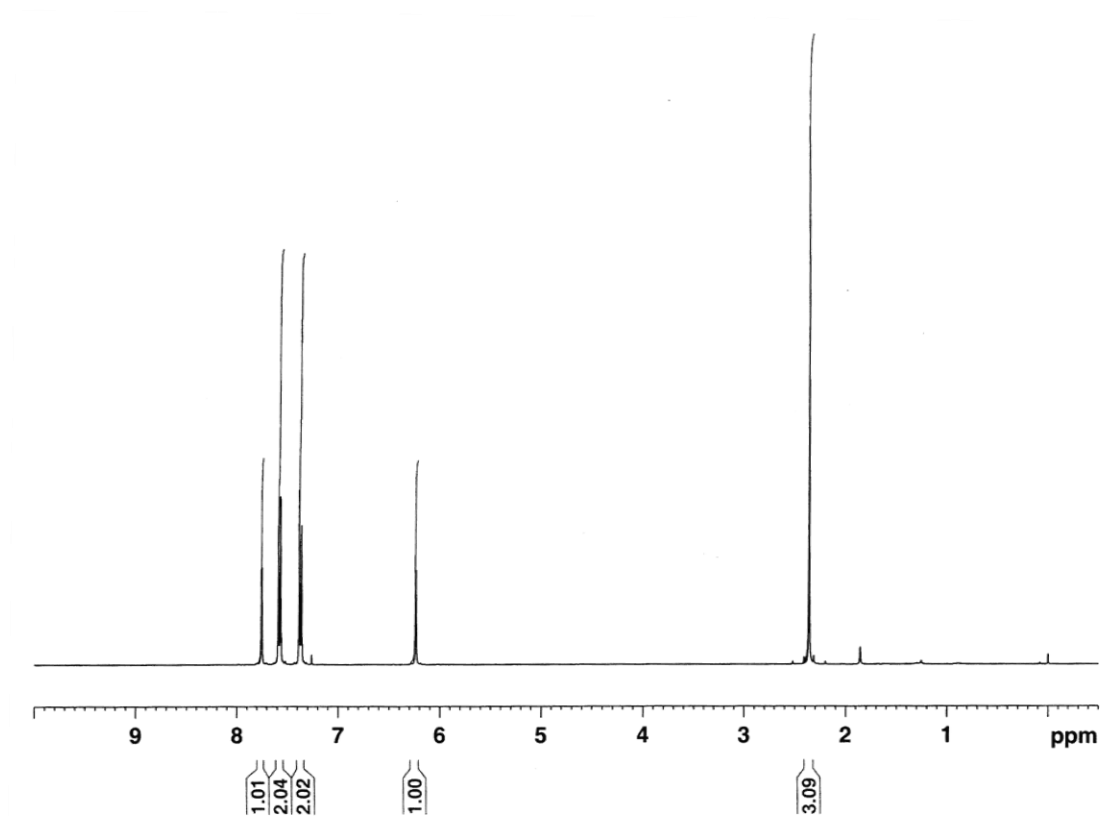
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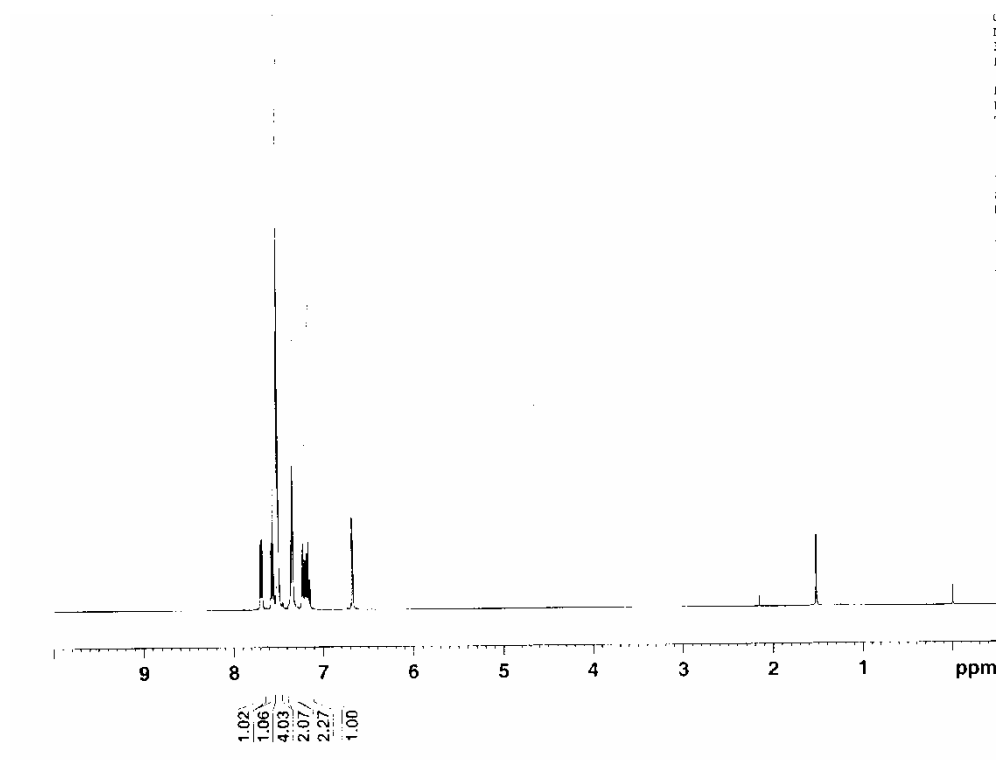
3-Methyl-1-(3-MethylPhenyl)-1H-pyrazole (6b)



3-Methyl-1-(4-ChloroPhenyl)-1H-pyrazole (6c)



1-Phenyl-1*H*-indole (7a)



1-(3-Methylphenyl)-1*H*-indole (7b)

