

Supporting information for

**H₂O₂ in WERSA: An efficient Green Protocol for *Ips*-
hydroxylation of Aryl/Heteroarylboronic Acid**

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1. General Information

Chemicals and reagents were purchased from commercial suppliers and used without further purification. Reactions were monitored by thin layer chromatography (TLC) on silica gel plates (60 F254), visualizing with ultraviolet light. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra was determined in CDCl_3 solution by using 400 MHz spectrometer taking tetramethylsilane (TMS, $\delta = 0.00$) as internal standard and expressed in ppm. Spin multiplicities are given as s (singlet), d (doublet), t (triplet) and m (multiplet) as well as b (broad). Infrared spectra were recorded on a FT- IR spectrometer. Melting points were determined using melting point apparatus and are uncorrected.

2. Synthesis and Characterization data of the products:

General procedure for the ipso-hydroxylation of arylboronic acid:

In a 50 mL round-bottomed flask, a mixture of arylboronic acid (1 mmol), H_2O_2 (30% aq, 0.2 mL) and WERSA (2 mL) were added and stirred at room temperature in aerobic condition. The reaction was monitored by TLC. After completion of the reaction the reaction mixture was diluted with 20 mL of water and extracted with 20 mL of diethylether and the combined organic layer was washed with brine and dried over by Na_2SO_4 and evaporated in a rotary evaporator under reduced pressure. The products were confirmed by $^1\text{H-NMR}$, $^{13}\text{C-NMR}$ and FT-IR spectroscopy without any further purification by column chromatography.

Characterization of the products:

Phenol (Entry 1, Table 2): Brown liquid (Yield =98%), $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 7.18-7.30 (m, 2H), 6.94-6.78 (m, 3H), 5.30 (s, br, 1H) ppm; $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ 155.4, 129.5, 120.6, 115.2 ppm.

3-Methoxyphenol (Entry 2, Table 2): Colorless oil (Yield =92%), $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 7.13 (t, 1H), 6.50-6.41 (m, 3H), 5.34 (s, 1H), 3.77 (s, 3H) ppm; $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ 161.2, 155.6, 131.5, 105.2, 104.5, 102.2, 55.3 ppm.

4-Hydroxyacetophenone (Entry 6, Table 2): Powder (Yield =95%), $^1\text{H-NMR}$ (400 MHz,

$CDCl_3$): δ 7.92(d, 2H), 6.93 (d, 2H), 5.55 (s, br, 1H), 2.56 (s, 3H) ppm; ^{13}C -NMR (100 MHz, $CDCl_3$): δ 197.5, 161.2, 130.9, 129.5, 115.3, 26.1 ppm. m.p. 110 °C

4-Methoxyphenol (Entry 5, Table 2): White gum (Yield =94%), 1H -NMR (400 MHz, $CDCl_3$): δ 6.84-6.74 (m, 4H), 4.62 (s, br, 1H), 3.81 (s, 3H) ppm; ^{13}C -NMR (100 MHz, $CDCl_3$): δ 155.5, 152.5, 116.1, 115.5, 55.5 ppm. m.p. 56 °C

4-Chlorophenol (Entry 8, Table 2): White gum (Yield=90%), 1H -NMR (400 MHz, $CDCl_3$): δ 7.19 (d, 2H), 6.77 (d, 2H), 4.87 (s, br, 1H) ppm; ^{13}C -NMR (100 MHz, $CDCl_3$): δ 154.2, 129.4, 125.5, 116.6 ppm.

3-Methylphenol (Entry 3, Table 2): Colourless gum (Yield =92%), 1H -NMR (400 MHz, $CDCl_3$): δ 7.13 (t, 1H), 6.75-6.62 (m, 3H), 4.98 (s, br, 1H), 2.25 (s, 3H) ppm; ^{13}C -NMR (100 MHz, $CDCl_3$): δ 154.3, 139.2, 129.5, 122.6, 115.3, 112.4, 20.9 ppm.

4-Nitrophenol (Entry 7, Table 2): Yellow solid (Yield =95%) 1H -NMR (400 MHz, $CDCl_3$): δ 8.21 (d, 2H), 6.98 (d, 2H), 6.57 (s, br, 1H) ppm; ^{13}C -NMR (100 MHz, $CDCl_3$): δ 161.7, 141.3, 126.3, 115.8 ppm. m.p. 112 °C

4-Methylphenol (Entry 4, Table 2): Colourless gum (Yield =94%), 1H NMR (400 MHz, $CDCl_3$): δ 7.03 (d, 2H), 6.75 (d, 2H), 4.17 (s, br, 1H), 2.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.7, 132.2, 130.3, 116.3, 21.4 ppm.

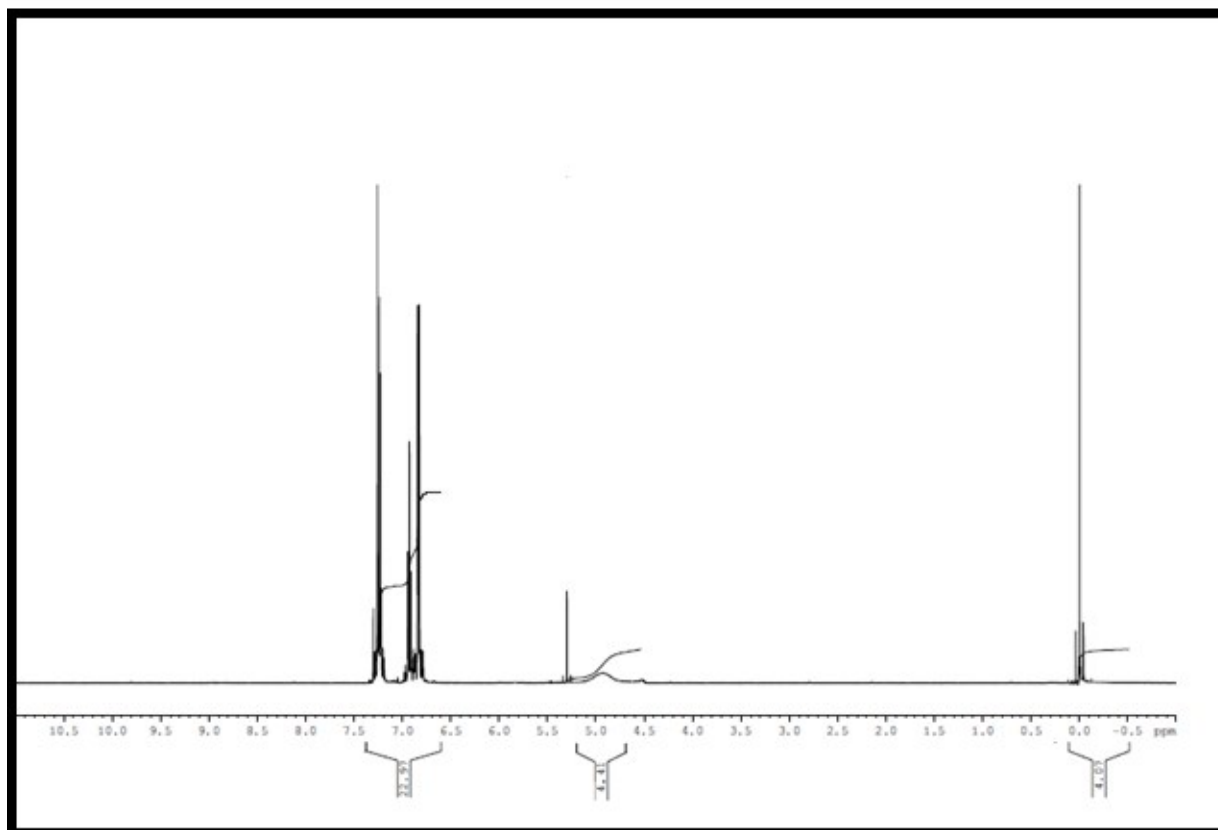
3-Nitrophenol (Entry 10, Table 2): Yellow Solid (Yield =97%), 1H -NMR (400 MHz, $CDCl_3$): δ 7.81 (d, 1H), 7.70 (s, 1H), 7.41 (d, 1H), 7.18 (d, 1H) 5.59 (s, br, 1H) ppm; ^{13}C -NMR (100 MHz, $CDCl_3$): δ 158.2, 149.6, 135.2, 121.2, 115.6, 110.2 ppm. m.p 98 °C

2-methylphenol (Entry 9, Table 2) Brown liquid (yield=92%), 1H -NMR (400 MHz, $CDCl_3$): δ 7.13-7.06 (m, 2H), 6.84 (t, 1H), 6.77 (d, 1H), 6.12 (s, br, 1H), 2.35 (s, 3H) ppm; ^{13}C -NMR (100 MHz, $CDCl_3$): δ 154.5, 130.2, 127.3, 124.9, 121.3, 115.7, 14.9 ppm.

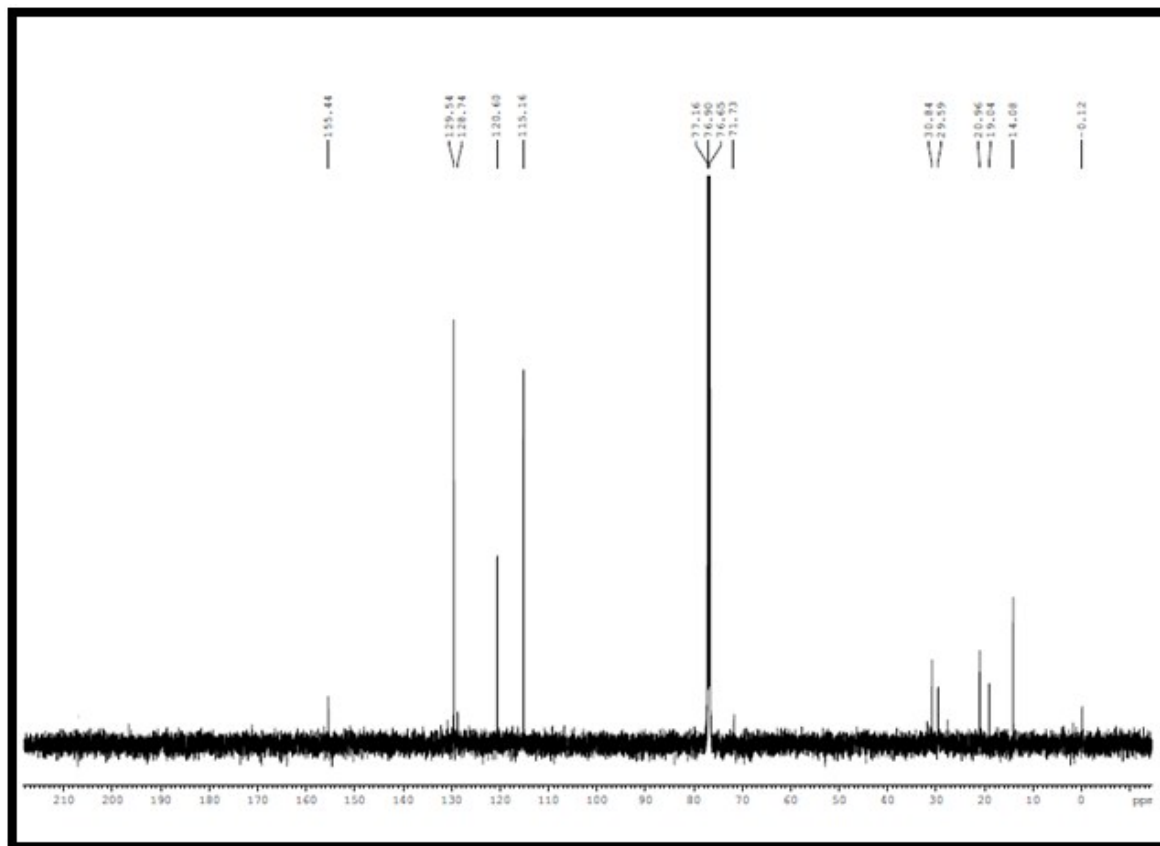
Thiophen-2-ol (Entry 11, Table 2): Colorless oil (yield=90%), ^1H NMR (400 MHz, CDCl_3) 7.73 (t, 1H), 7.55-7.53 (m, 2H), 5.46 (s, br, 1H) ppm; ^{13}C -NMR (100 MHz, CDCl_3): 165.2, 131.6, 128.2, 111.2 ppm.

Furan-2-ol (Entry 12, Table 2): Colorless oil (yield=91%), ^1H NMR (400 MHz, CDCl_3) 7.72 (t, 1H), 7.12-6.67 (m, 2H), 5.57 (s, br, 1H) ppm; ^{13}C -NMR (100 MHz, CDCl_3): 145.3, 141.2, 117.3, 116.2 ppm.

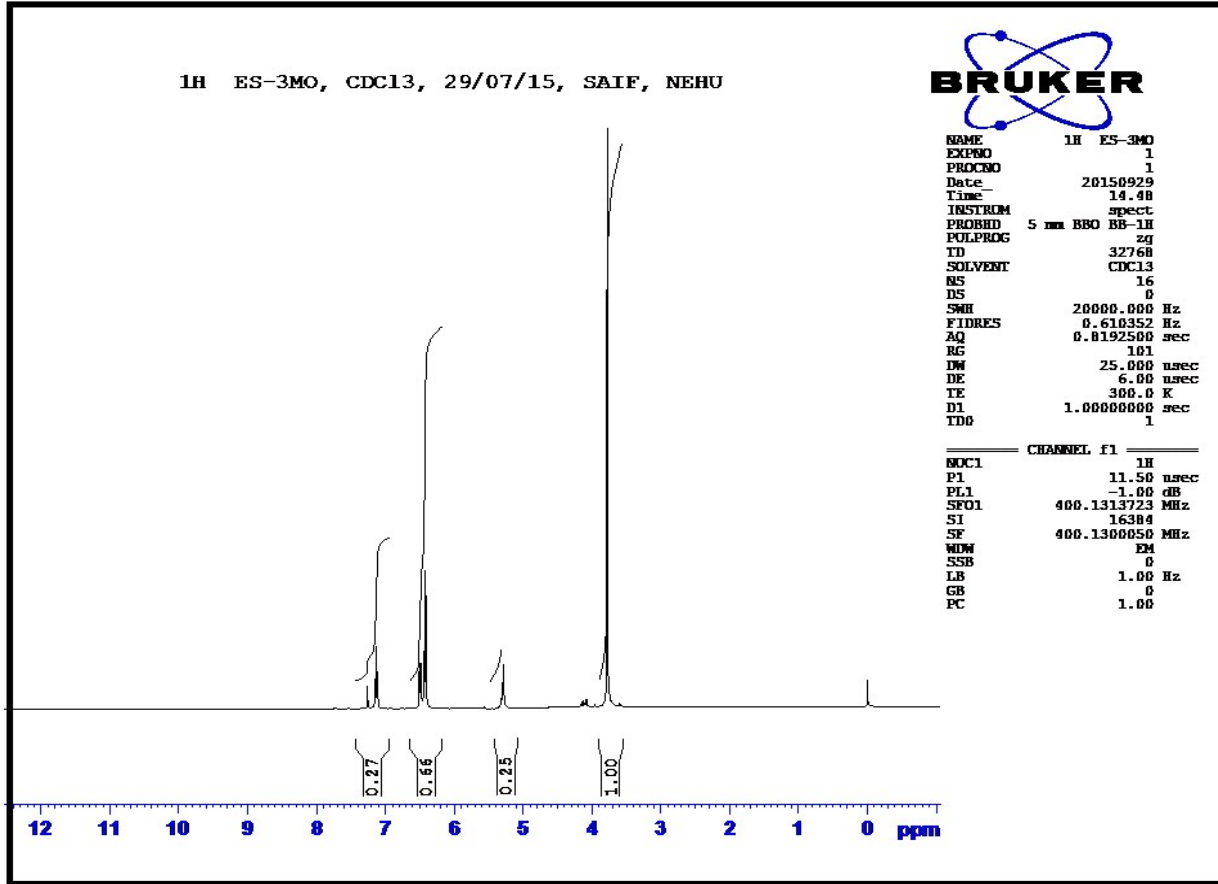
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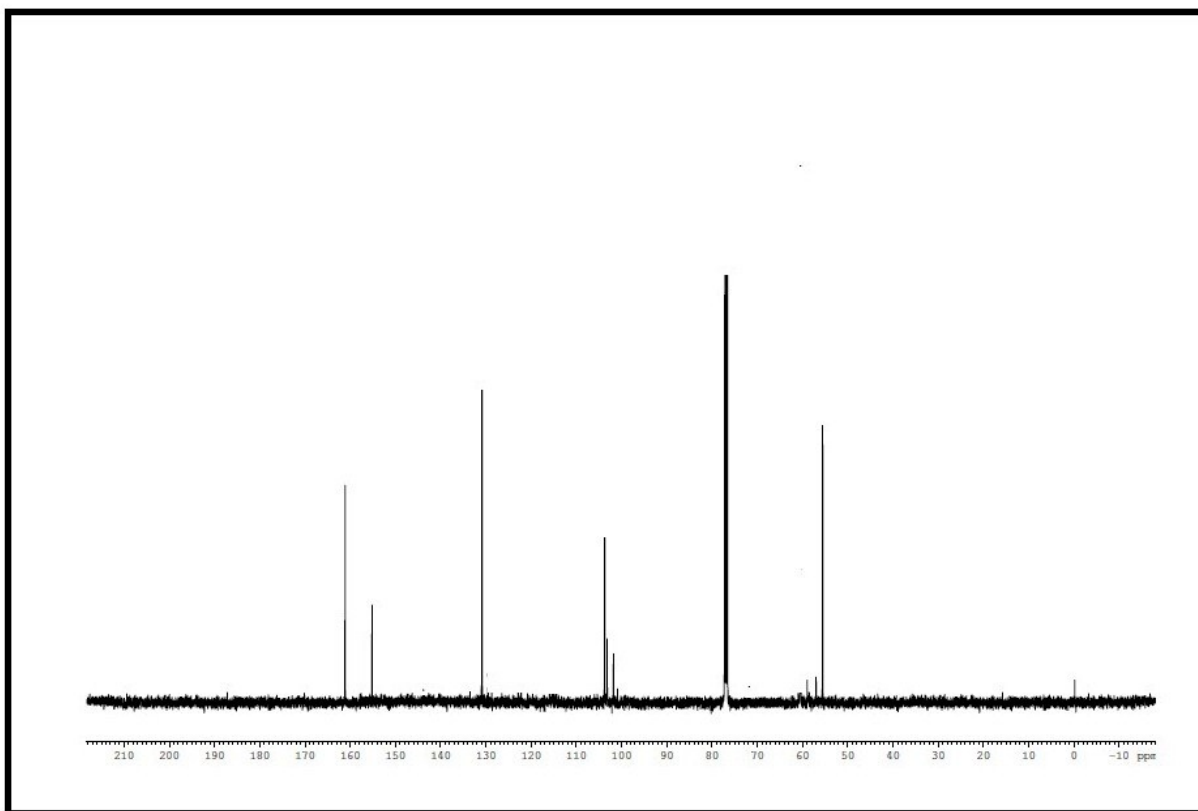
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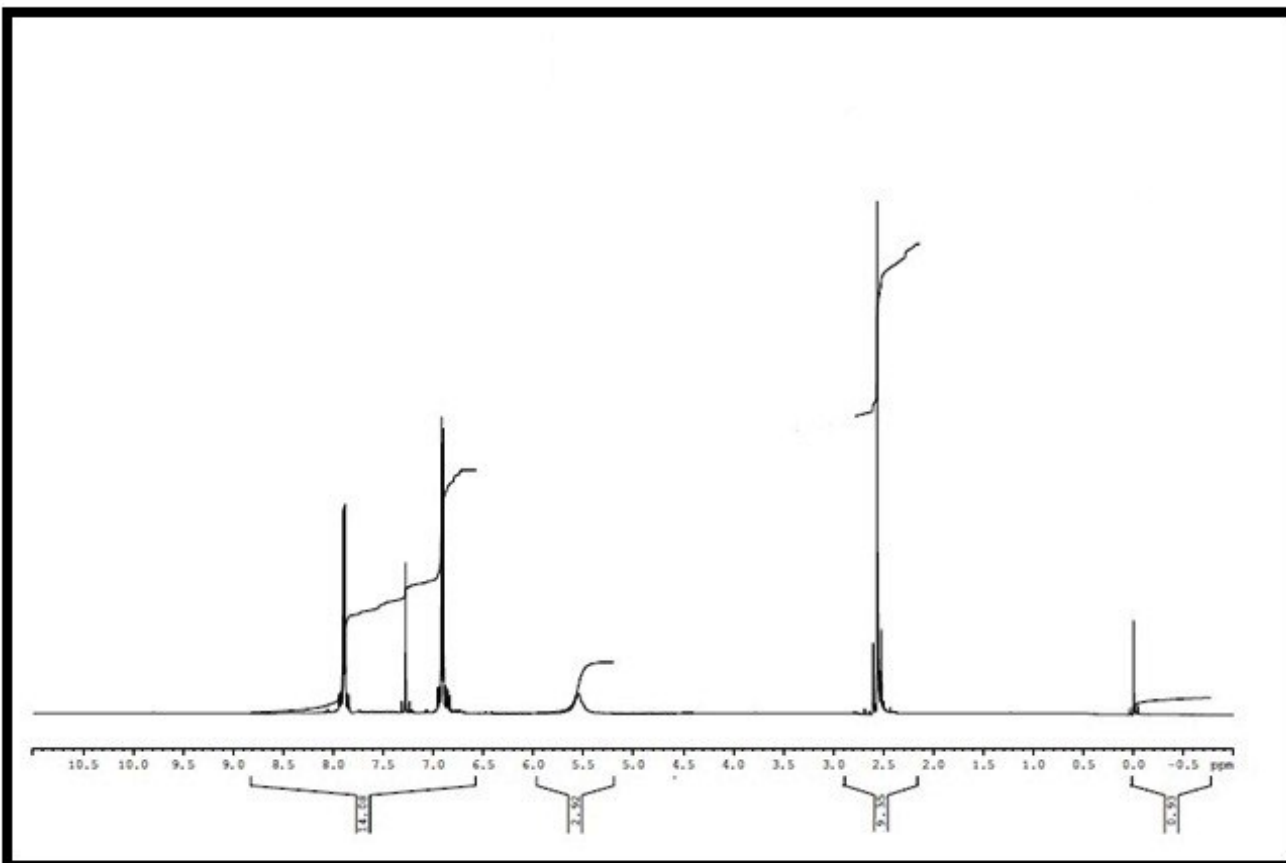
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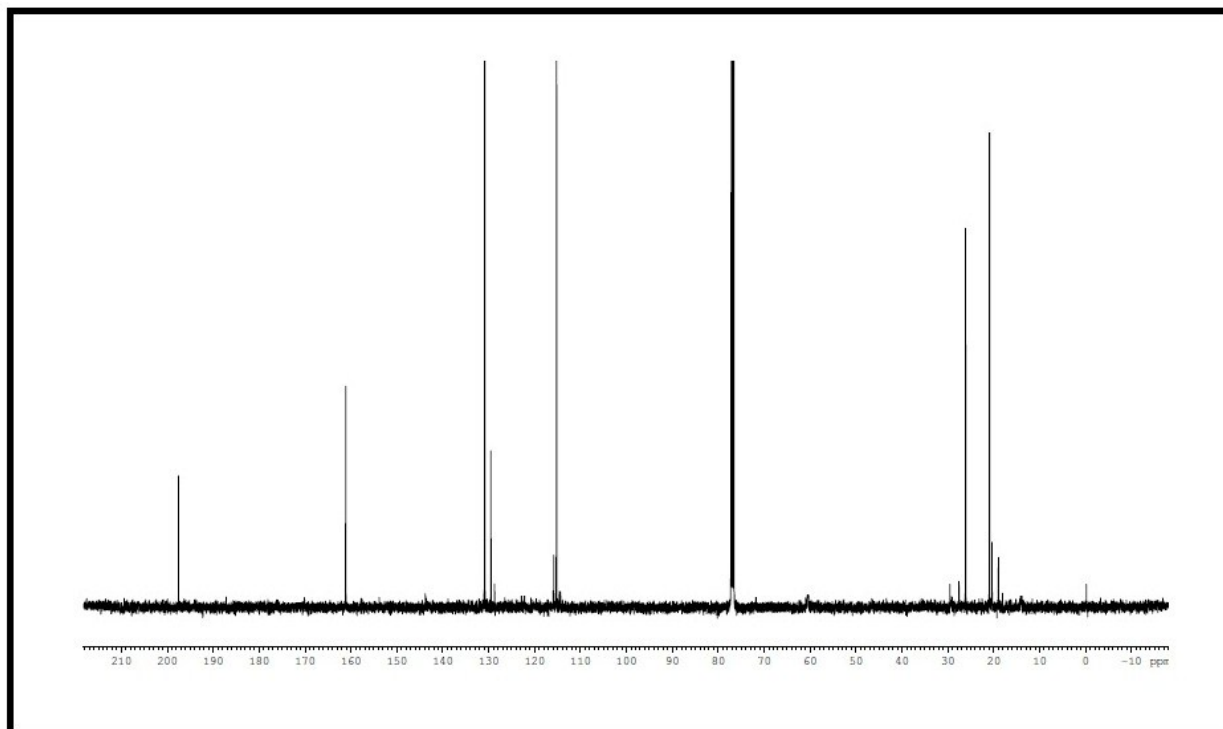
^{13}C -NMR spectrum of 3-Methoxyphenol:



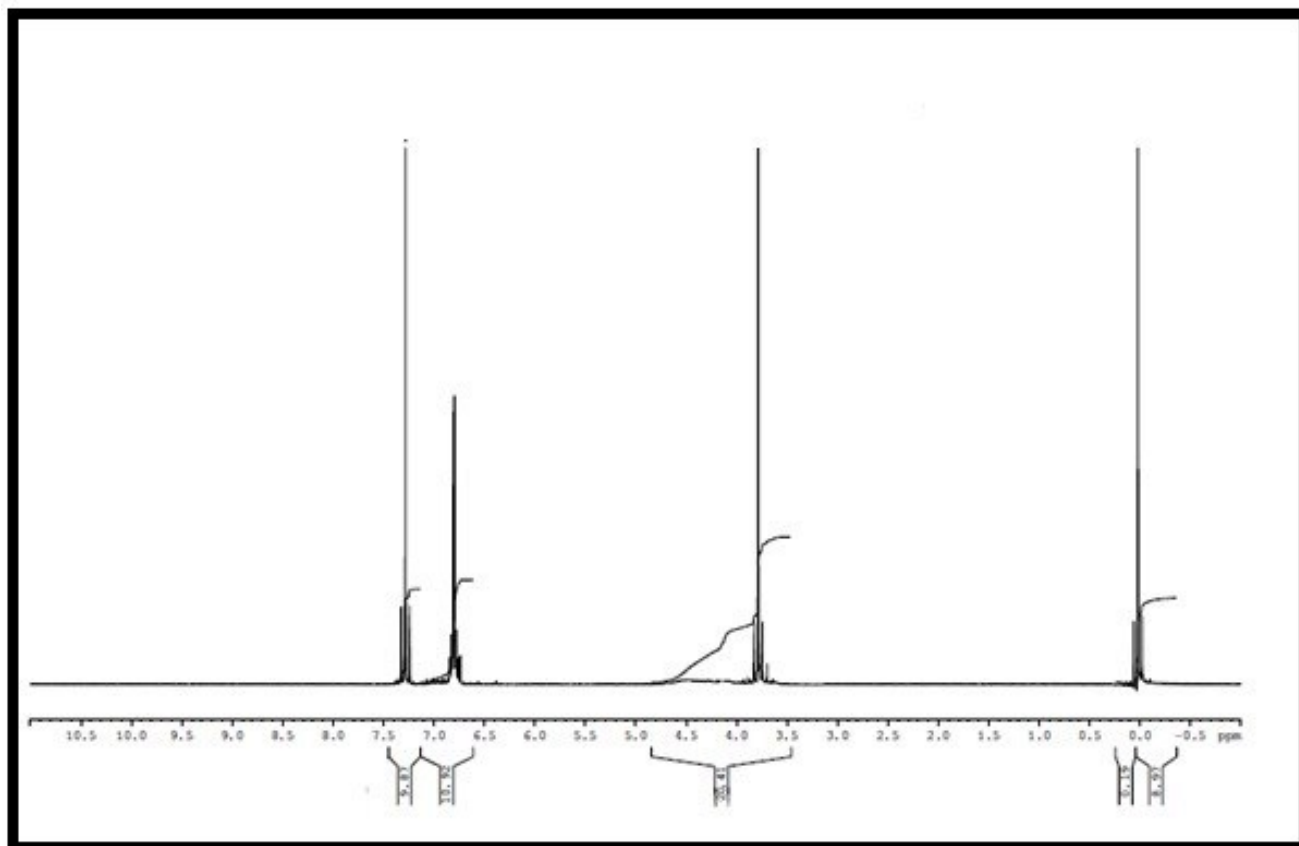
¹H-NMR spectrum of 4-Hydroxyacetophenone:



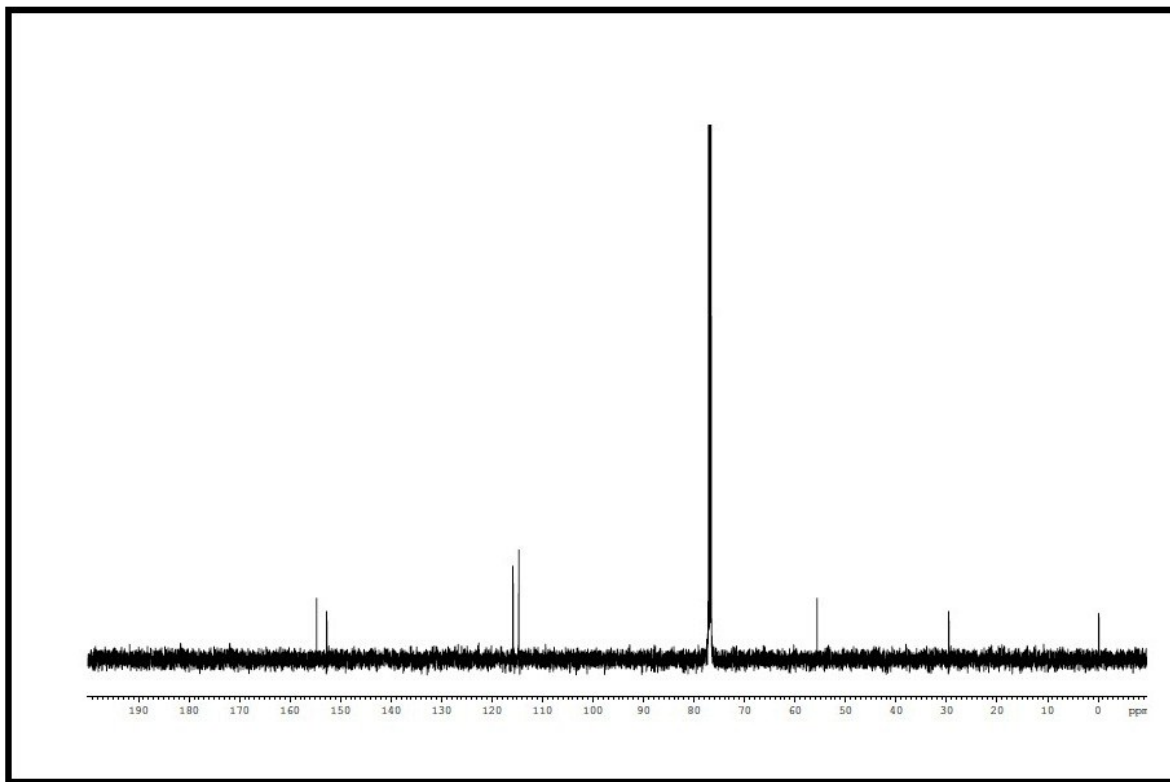
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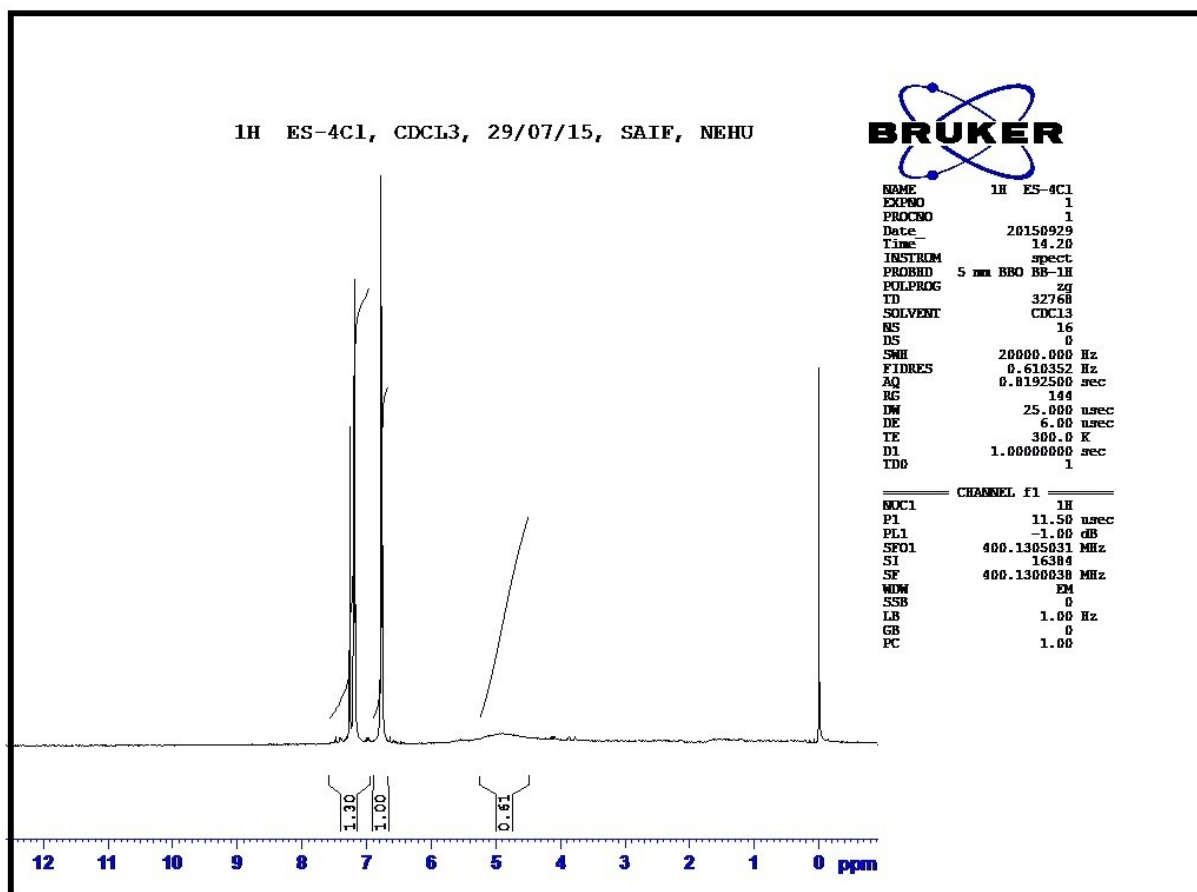
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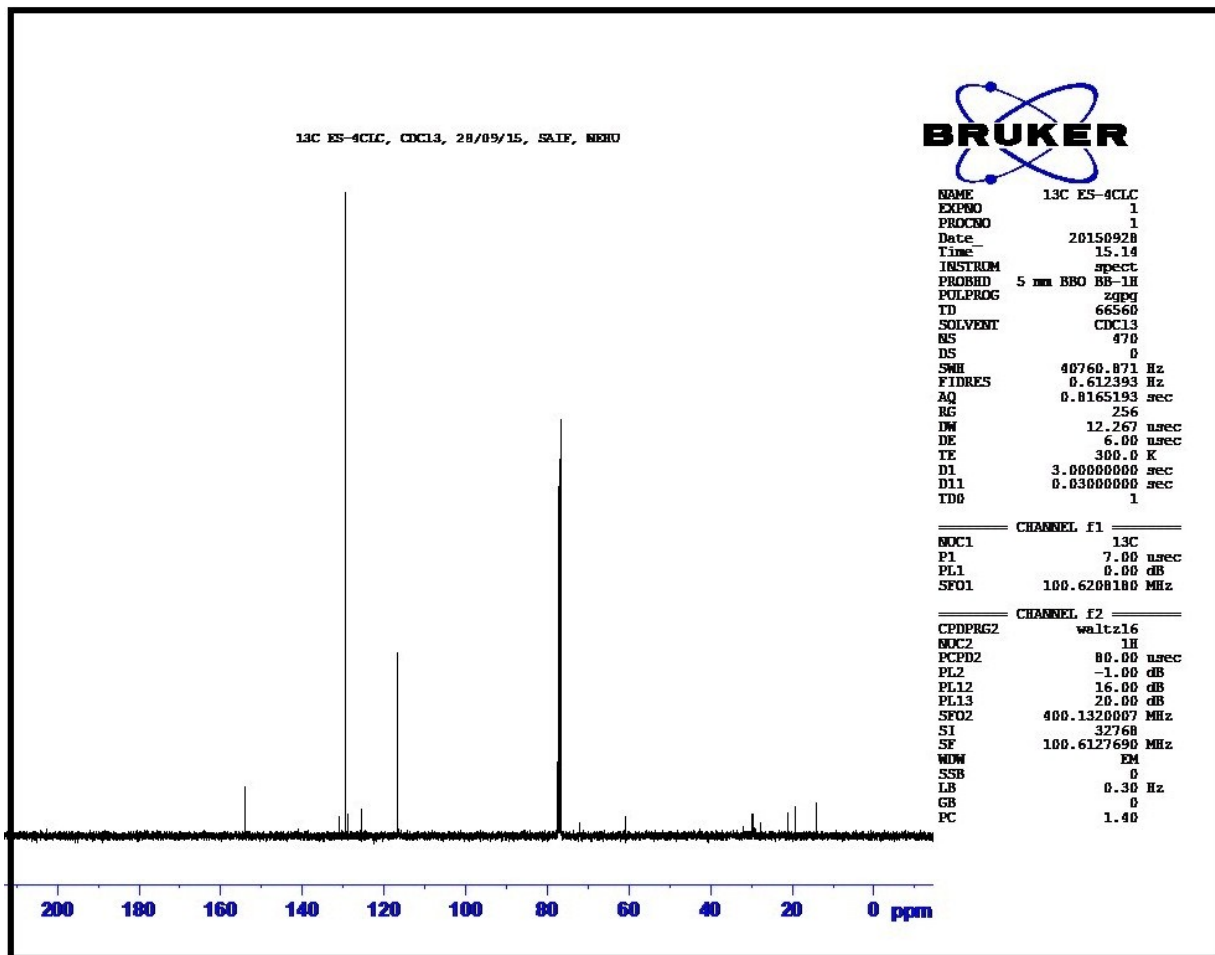
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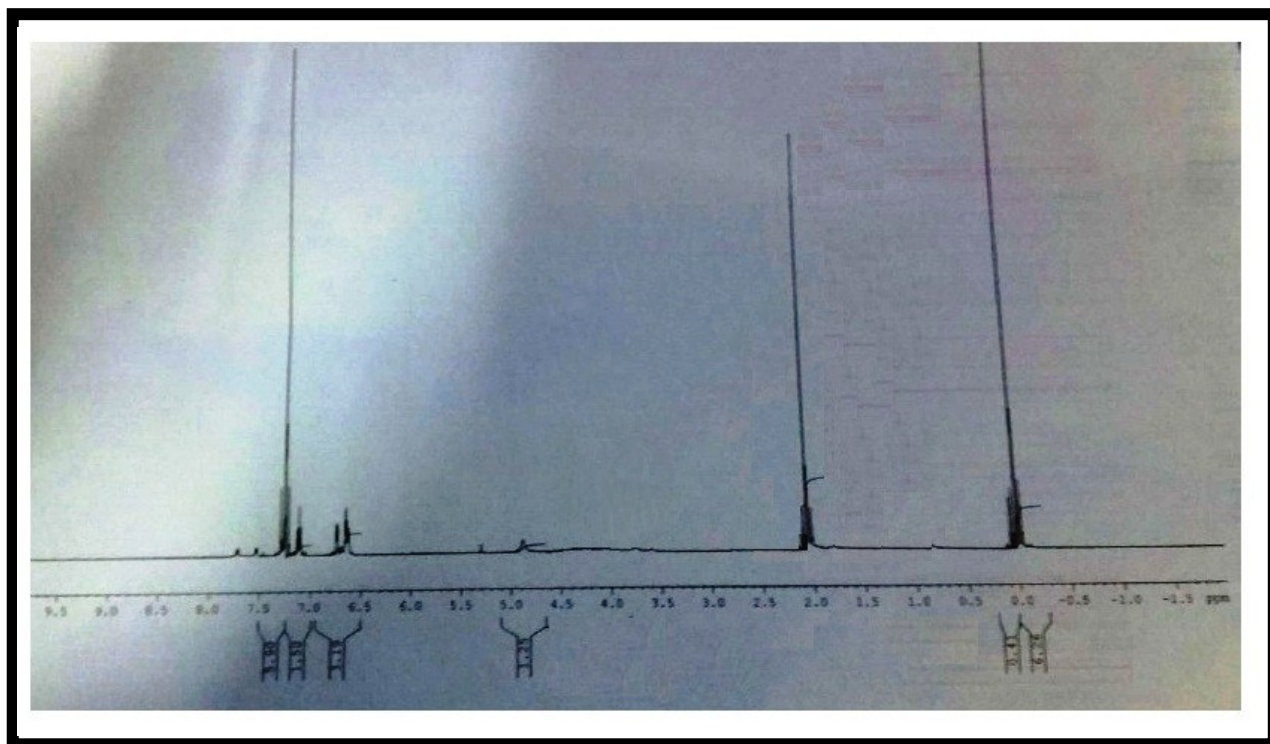
¹H-NMR spectrum of 4-Chlorophenol:



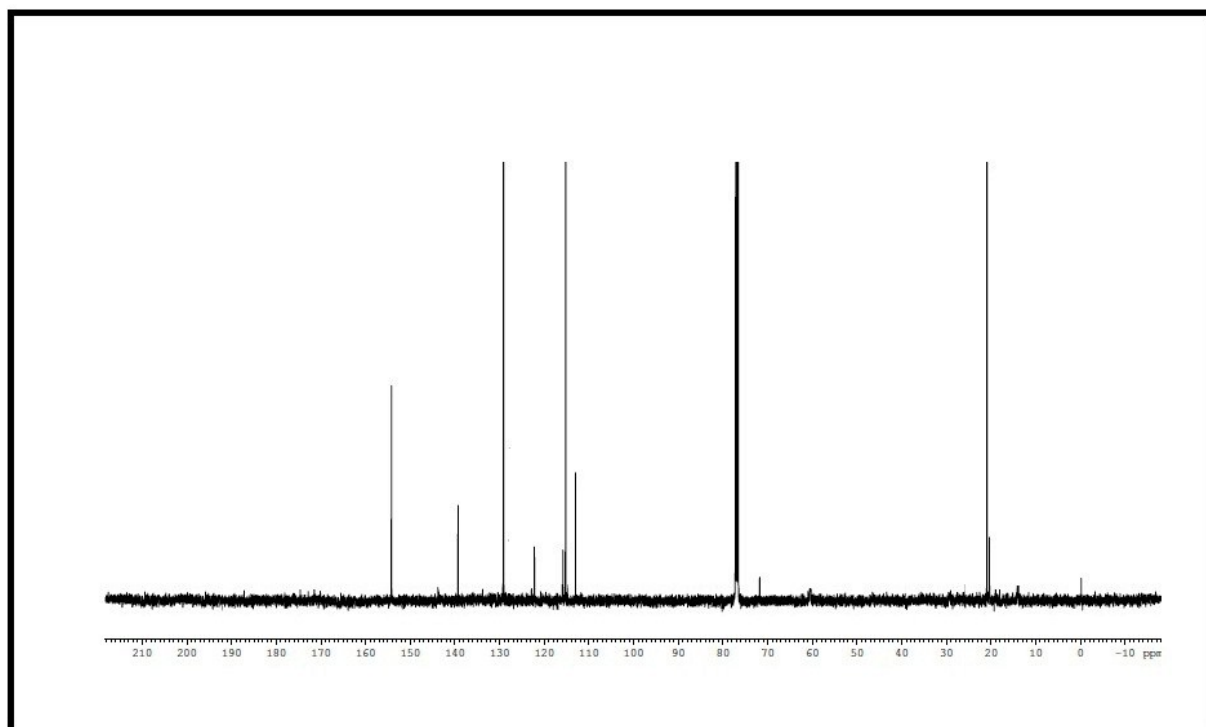
¹³C-NMR of 4-Chlorophenol:



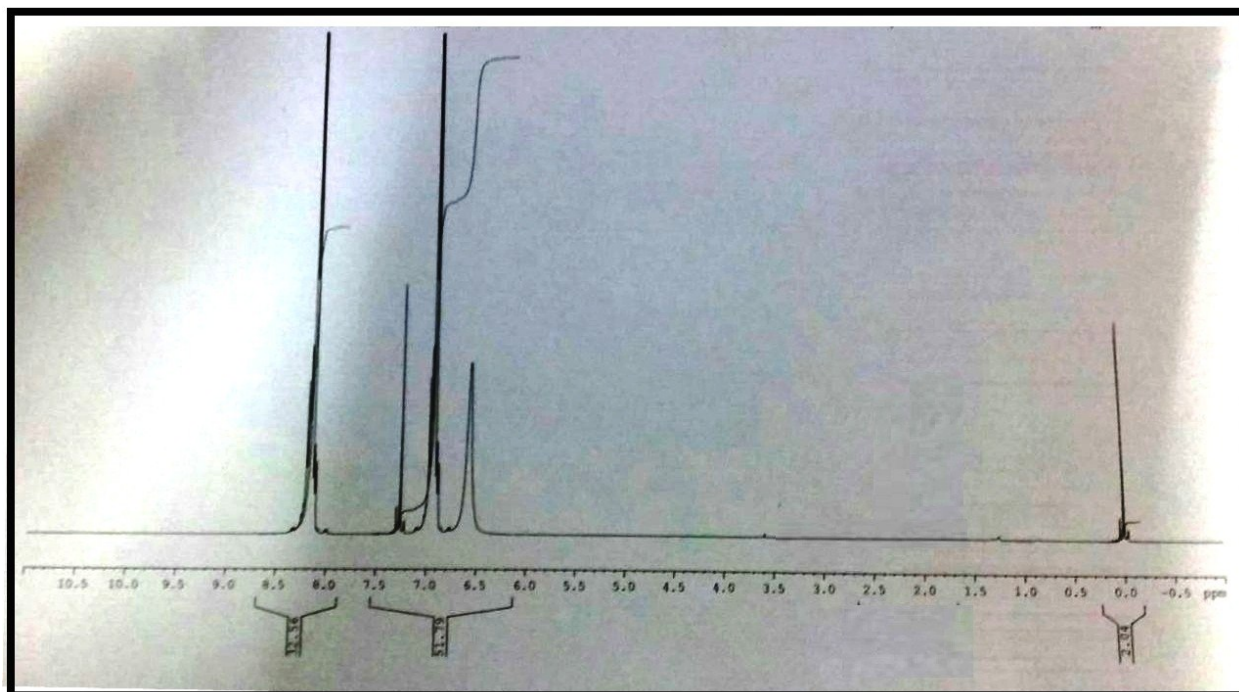
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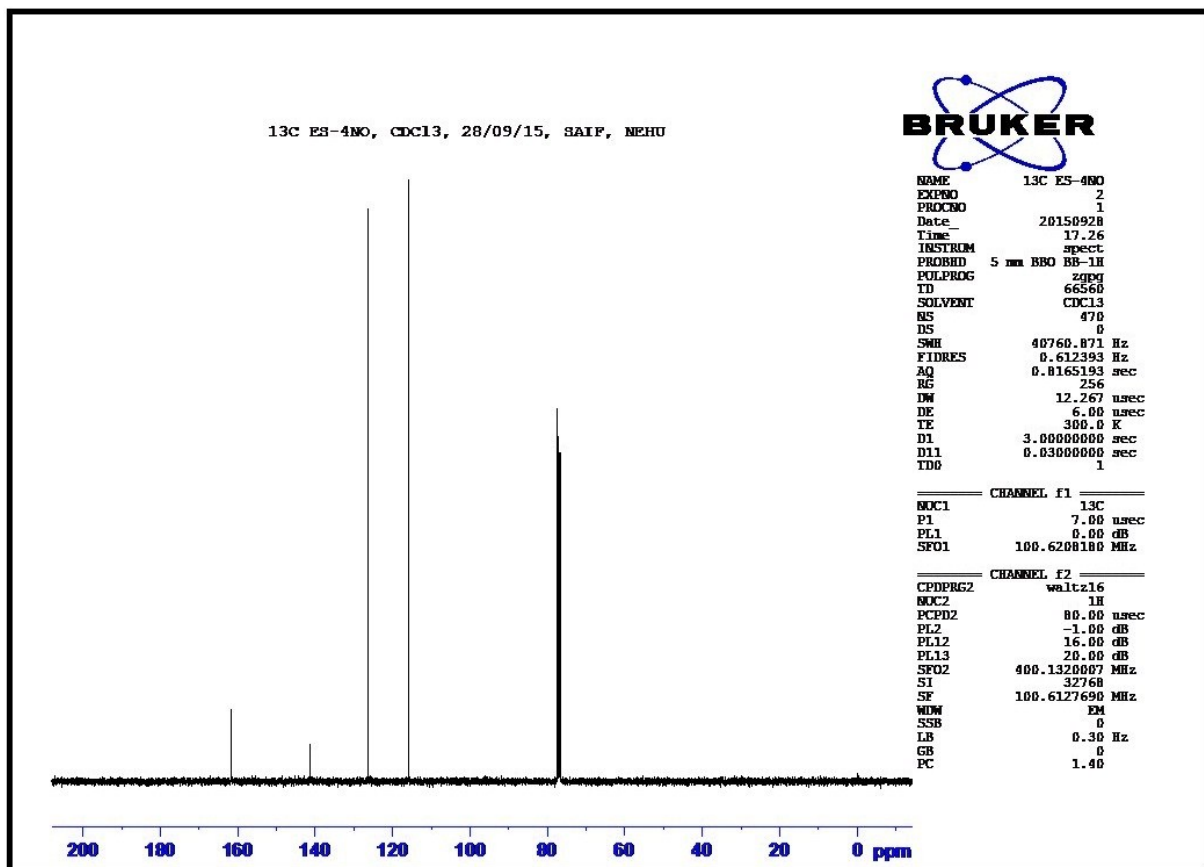
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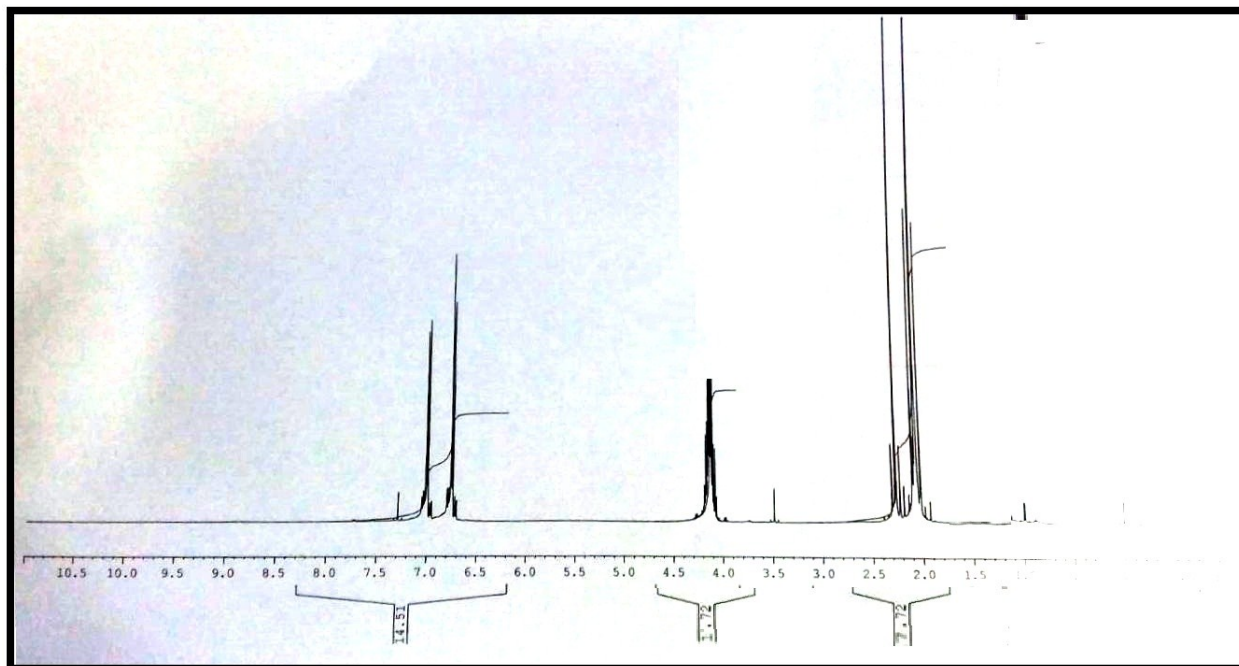
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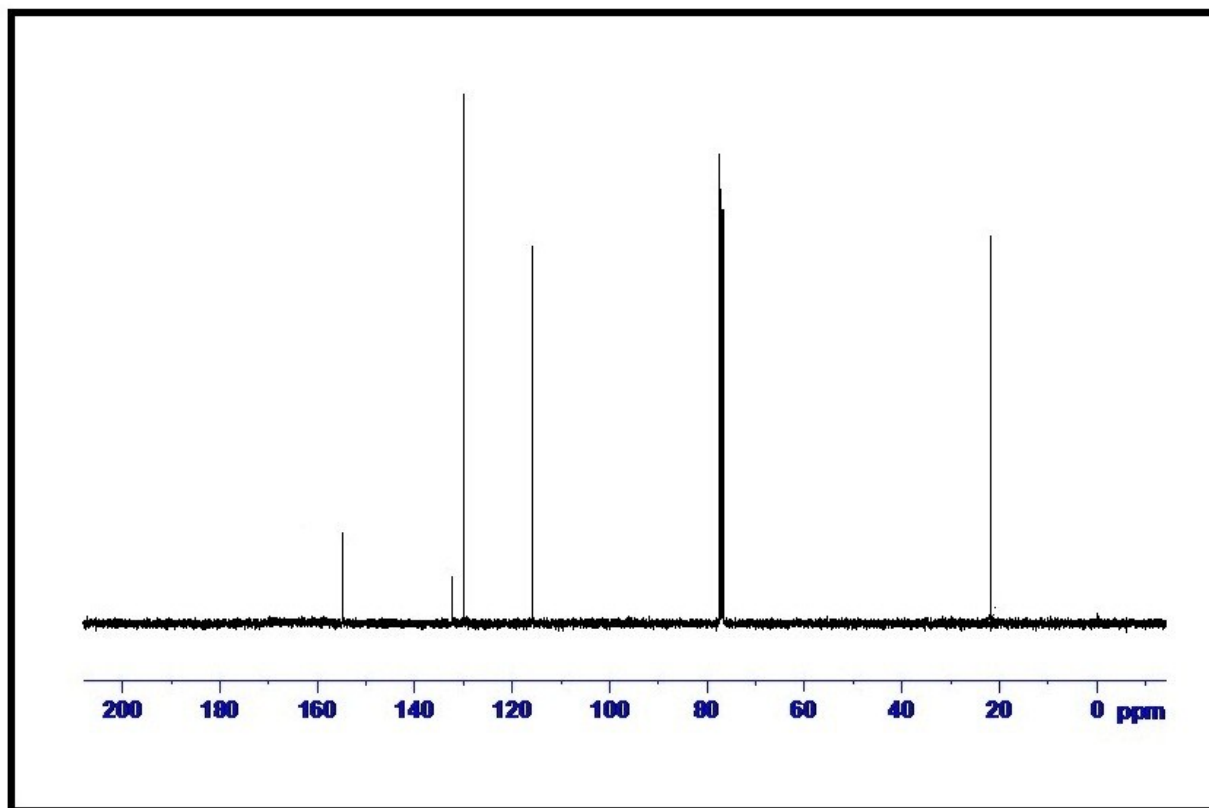
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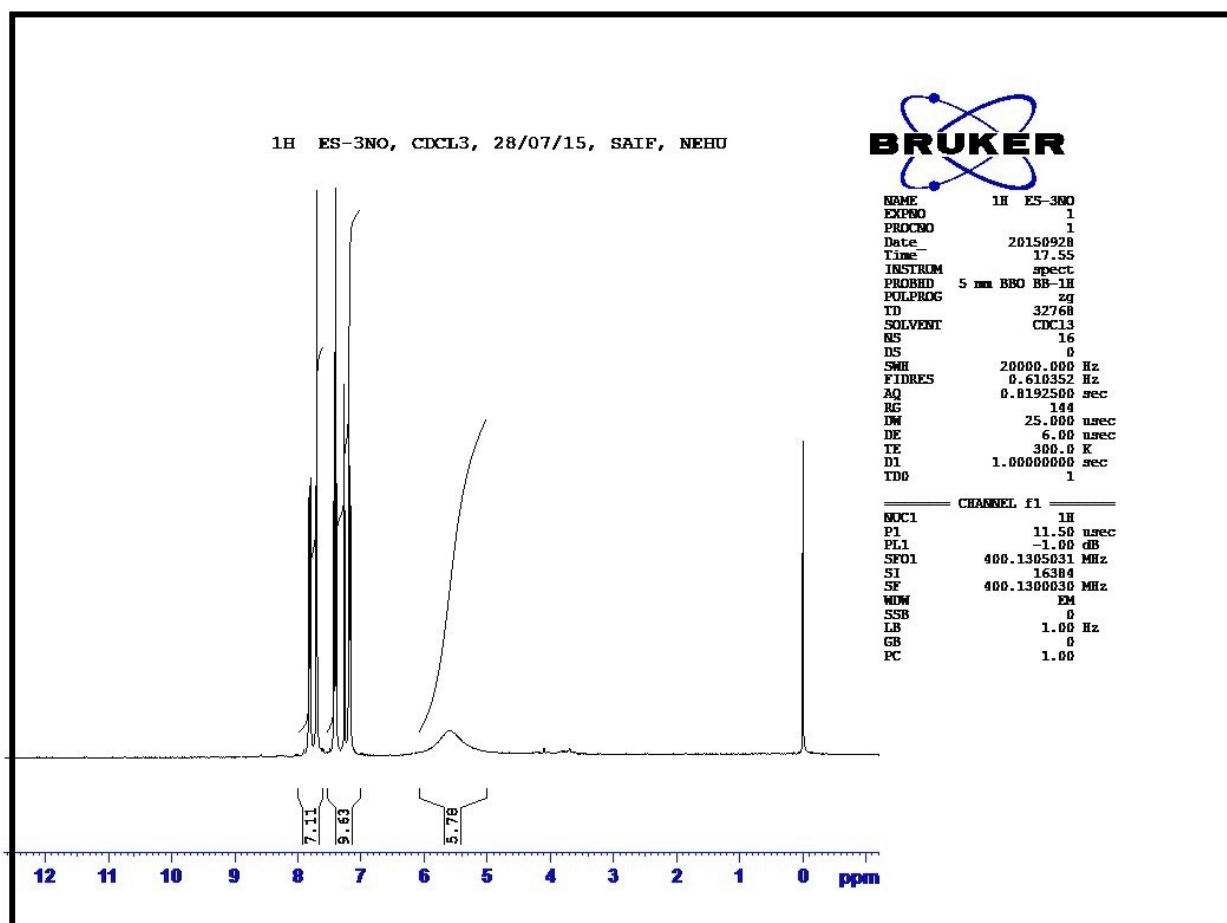
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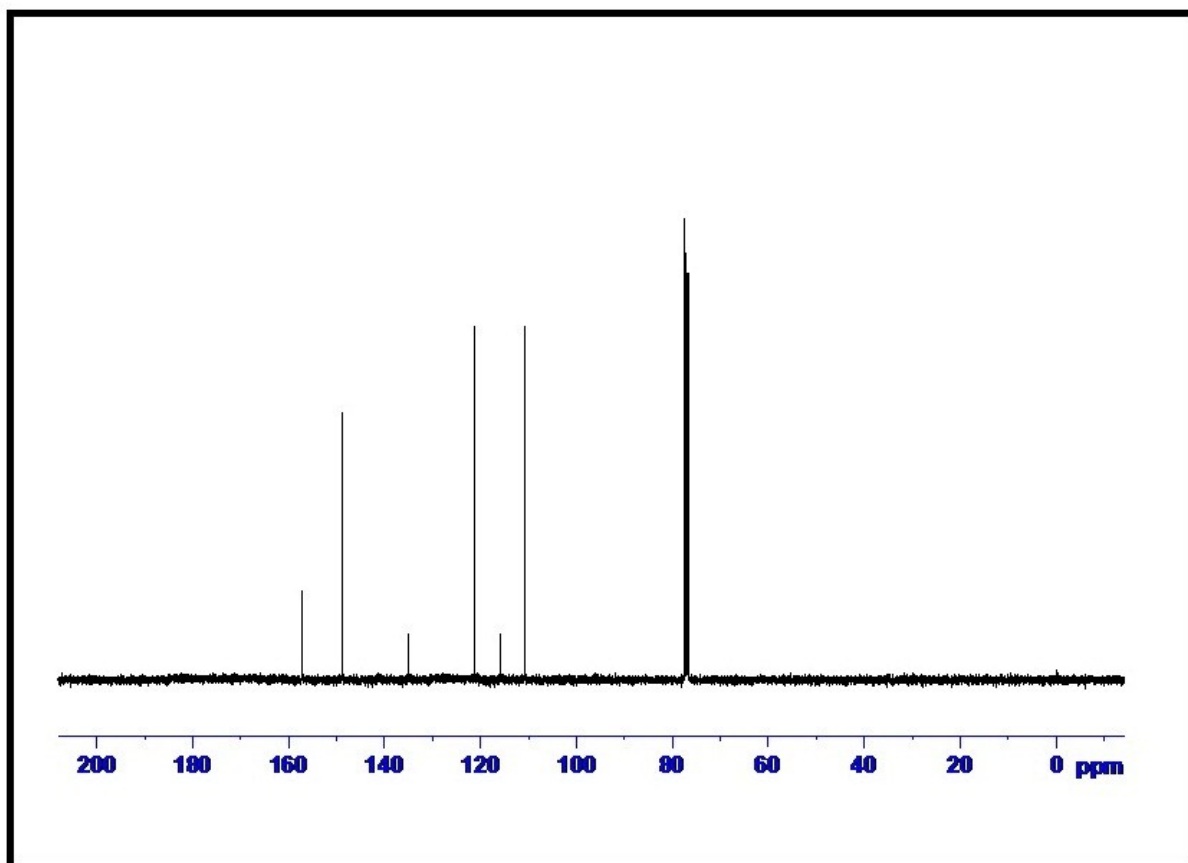
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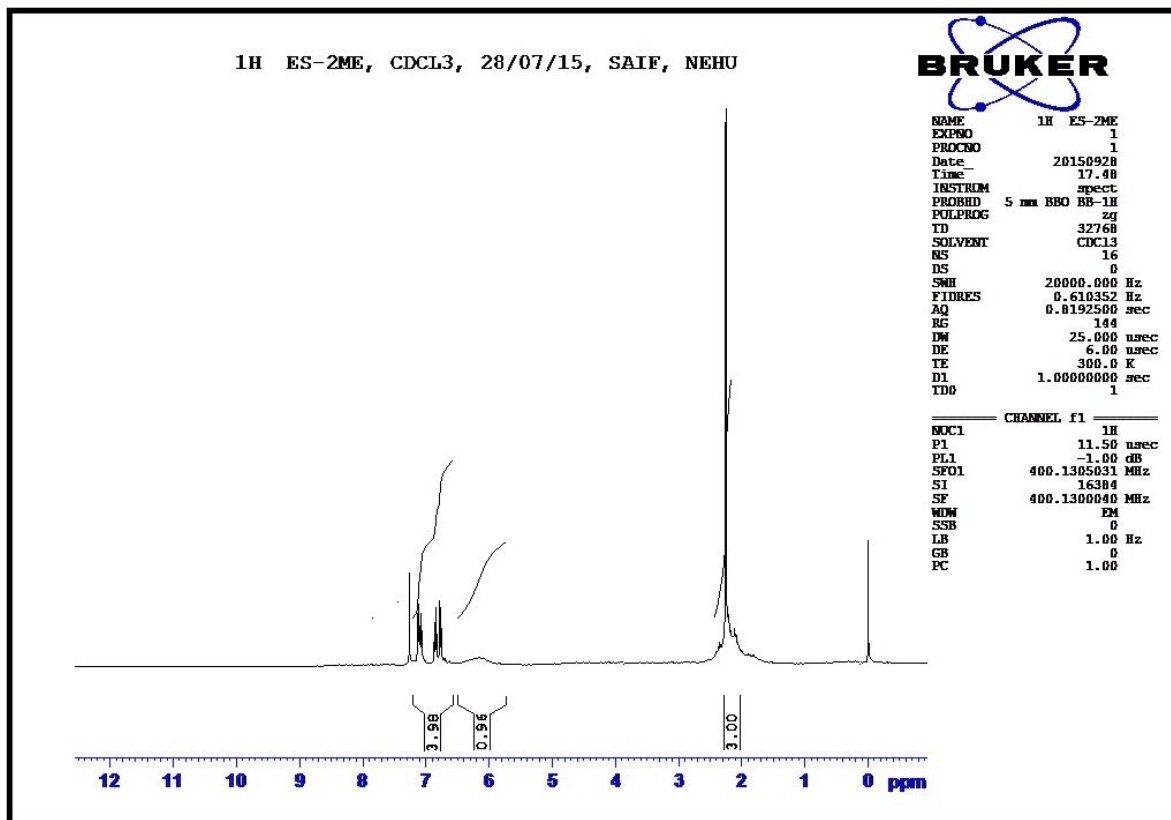
¹H-NMR of 3-Nitrophenol:



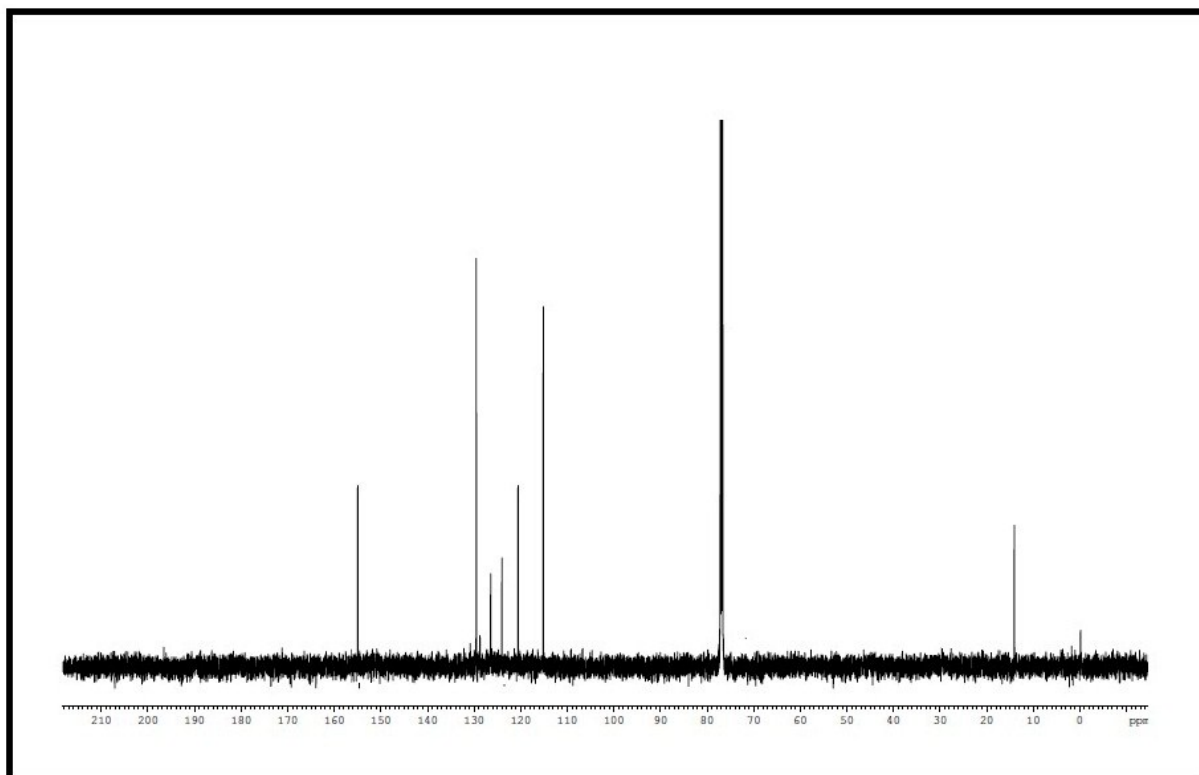
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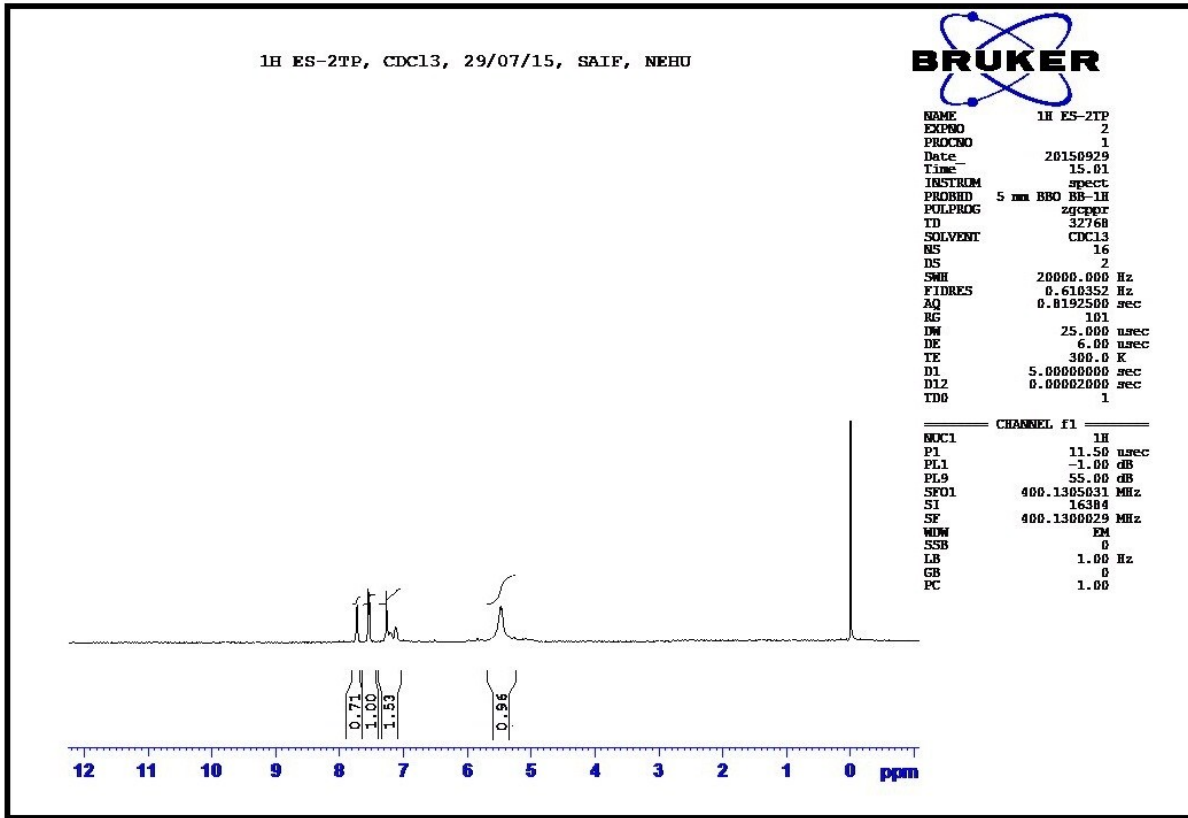
¹H-NMR spectrum of 2-Methylphenol:



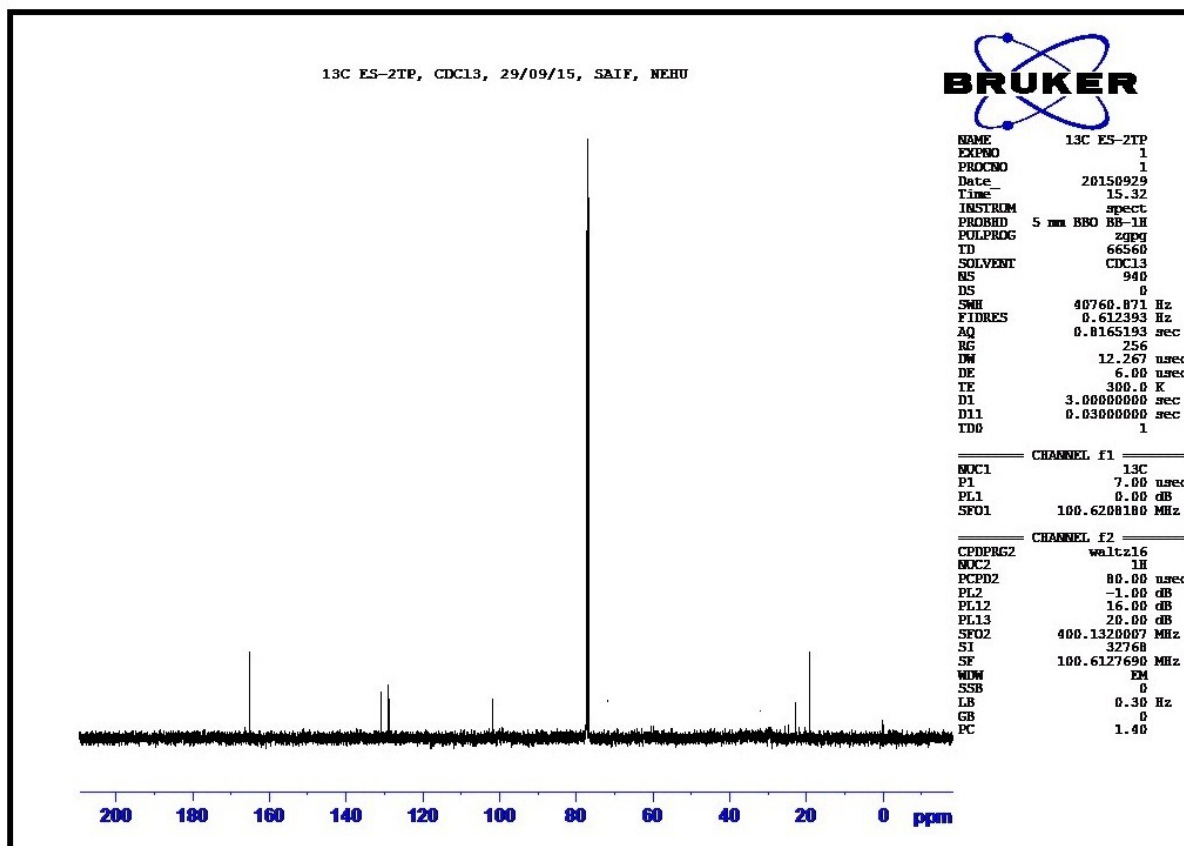
^{13}C -NMR spectrum of 2-Methylphenol:



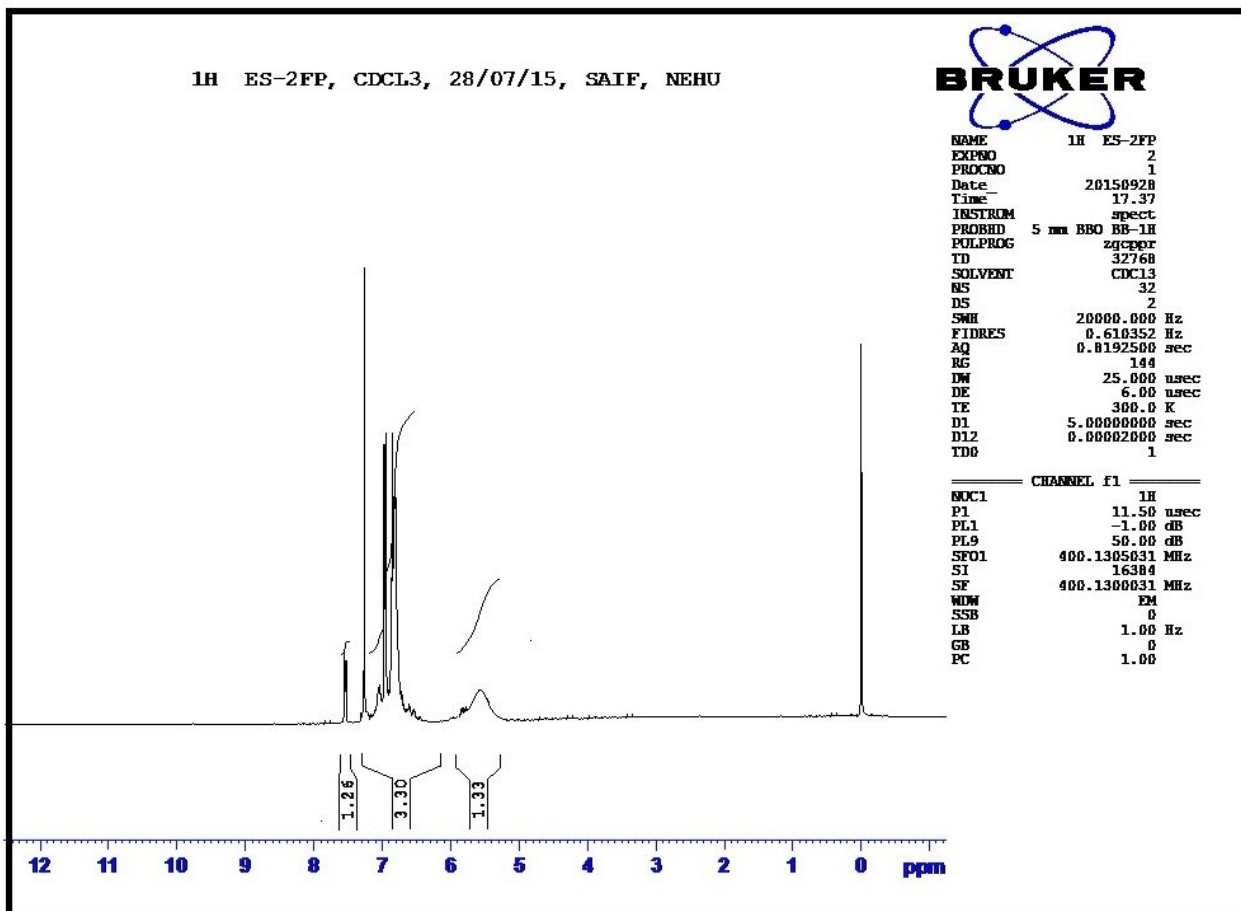
¹H-NMR spectrum of Thiophen-2-ol:



¹³C-NMR spectrum of Thiophen-2-ol:



¹H-NMR spectrum of Furan-2-ol:



¹³C-NMR spectrum of Furan-2-ol:

