

Nickel Catalyzed One Pot Synthesis of Biaryls under Air at Room Temperature

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Supporting data

Characterization data

All the compounds gave satisfactory spectroscopic values which are given below and are analogue to spectroscopic data reported in the literature. Elemental analyses are given for each compound.

4,4'-Dimethoxybiphenyl (2a): Yield: (0.5913 g, 92%) white solid; mp: 178-180 °C, Lit.¹ = 179 °C; ¹H NMR δ_H (500 MHz, CDCl₃): 3.84 (s, 6H), 6.96 (d, J = 9.0 Hz, 4H), 7.48 (d, J = 8.5 Hz, 4H); ¹³C NMR δ_C (125 MHz, CDCl₃): 55.5, 114.4, 127.9, 133.6, 158.9; **Elemental analysis** calcd for C₁₄H₁₄O₂ (%): C, 78.48; H, 6.59; O, 14.93. found: C, 78.23; H, 6.44; O, 14.78.

2,2'-Dimethoxybiphenyl (2b): Yield: (0.5078 g, 79%); white solid; mp: 154.5-156 °C, Lit.¹ = 156 °C; ¹H NMR (500 MHz, CDCl₃) δ: 3.77 (s, 6H), 6.99 (m, 4H), 7.28 (d, J = 7.0 Hz, 2H), 7.32 (t, J = 7.0 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 55.8, 111.3, 120.5, 127.9, 128.8, 131.6, 157.2; **Elemental analysis** calcd for C₁₄H₁₄O₂ (%): C, 78.48; H, 6.59; O, 14.93. found: C, 78.35; H, 6.28; O, 14.84.

4,4'-Dimethylbiphenyl (2c): Yield: (0.7834 g, 86%); white solid; mp: 124-125 °C, Lit.² = 125 °C; ¹H NMR (500 MHz, CDCl₃) δ: 2.38 (s, 6H), 7.24 (d, J = 8.0 Hz, 4H), 7.48 (d, J = 8.0 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ: 21.3, 127.0, 129.7, 136.8, 138.5; **Elemental analysis** calcd for C₁₄H₁₄ (%): C, 92.26; H, 7.74. found: C, 92.18; H, 7.64.

2,2'-Dimethylbiphenyl (2d): Yield: (0.6832 g, 75%); yellow liquid; bp: 259 °C, Lit.³ = 257 °C; ¹H NMR (400 MHz, CDCl₃) δ: 2.05 (s, 6H), 7.11 (d, J = 8.8 Hz, 2H), 7.22-7.25 (m, 6H); ¹³C NMR (100 MHz, CDCl₃): δ = 18.8, 124.5, 126.1, 128.2, 128.7, 134.8, 140.6; **Elemental analysis** calcd for C₁₄H₁₄ (%): C, 92.26; H, 7.74. found: C, 92.29; H, 7.37.

Biphenyl (2e): Yield: (0.1.279 g, 83%); white solid; mp: 67-69 °C, Lit.¹ = 68 °C; ¹H NMR (500 MHz, CDCl₃) δ: 7.34 (t, J = 7.3 Hz, 2H), 7.44 (t, J = 7.5 Hz, 4H), 7.60 (d, J = 7.5 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ: 127.3, 127.4, 128.7, 141.4; **Elemental analysis** calcd for C₁₂H₁₀ (%): C, 93.46; H, 6.54. found: C, 93.28; H, 6.48.

4,4'-Dichlorobiphenyl (2f): Yield: (0.8477 g, 76%); white solid; mp: 150 °C, Lit.⁴ = 149 °C; ¹H NMR (400 MHz, CDCl₃) δ: 7.39 (d, J = 7.6 Hz, 4H), 7.46 (d, J = 8 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃) δ: 127.2, 128.0, 132.7, 137.4; **Elemental analysis** calcd for C₁₂H₈Cl₂ (%): C, 64.60; H, 3.61. found: C, 64.12; H, 3.38.

2,2'-Dinitrobiphenyl (2g): Yield: (0.3175 g, 65%); pale brown solid; mp: 120-122 °C, Lit.¹ = 120 °C; ¹H NMR (500 MHz, CDCl₃) δ: 7.31 (d, J = 7.5 Hz, 2H), 7.59 (t, J=7.25 Hz, 2H), 7.68 (t, J = 7 Hz, 2H), 8.23 (d, J = 8 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 124.8, 129.2, 131.0, 133.6, 134.2, 147.2; **Elemental analysis** calcd for C₁₂H₈N₂O₄(%): C, 59.02; H, 3.30; N, 11.47; O, 26.21 found: C, 58.96; H, 3.12; N, 11.34; O, 26.15.

4,4'-Dicyanobiphenyl (2h): Yield: (0.2491 g, 61%); white solid; mp: 234 °C, Lit.¹ = 233-234 °C; ¹H-NMR (500 MHz, CDCl₃): 7.68-7.74 (m, 4 H), 7.75-7.82 (m, 4 H); ¹³C-NMR (125 MHz, CDCl₃): 112.4, 118.4, 127.9, 132.8, 143.5; **Elemental Analysis** calcd For C₁₄H₈N₂ (%): C, 82.33; H, 3.95; N, 13.72. found: C, 82.15; H, 4.13; N, 13.45.

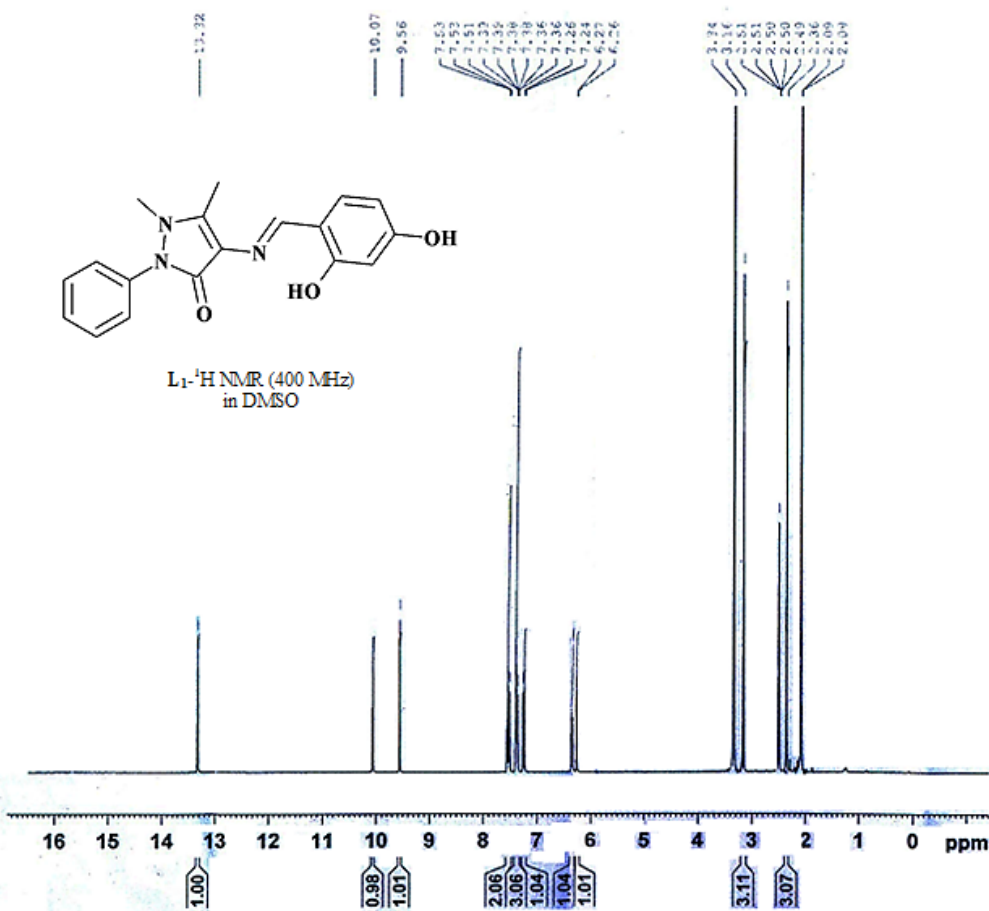
1,1'-Binaphthyl (2i): Yield: (0.6917 g, 68%); white solid; mp: 157 °C, Lit.⁵ = 155-156°C; ¹H NMR (300 MHz, CDCl₃) δ : 7.26-7.62 (m, 10H), 7.96 (d, *J* = 8.1, 4H); ¹³C NMR (75 MHz, CDCl₃) δ: 125.5, 125.9, 126.1, 126.7, 127.9, 128.0, 128.3, 133.0, 133.6, 138.6; **Elemental analysis** calcd for C₂₀H₁₄ (%): C, 94.45; H, 5.55. found: C, 94.18; H, 5.44.

4,4'-Bipyridine (2j): Yield: (0.2280 g, 73%); mp: 112 °C, Lit.¹ = 110 °C; ¹H NMR (500 MHz, CDCl₃) δ: 7.55 (dd, *J* = 4.5 Hz, 4H), 8.75 (dd, *J* = 4.5 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ: 121.4, 145.4, 150.6; **Elemental analysis** calcd for C₁₀H₈N₂ (%): C, 76.90; H, 5.16; N, 17.94 found: C, 76.78; H, 5.10; N, 17.88.

The ¹H NMR, TG analysis of the ligand **L** and complex **NiL** are given in S4 – S7. The ¹H NMR and ¹³C NMR spectra of the biaryl compounds are given in the S8 - S19.

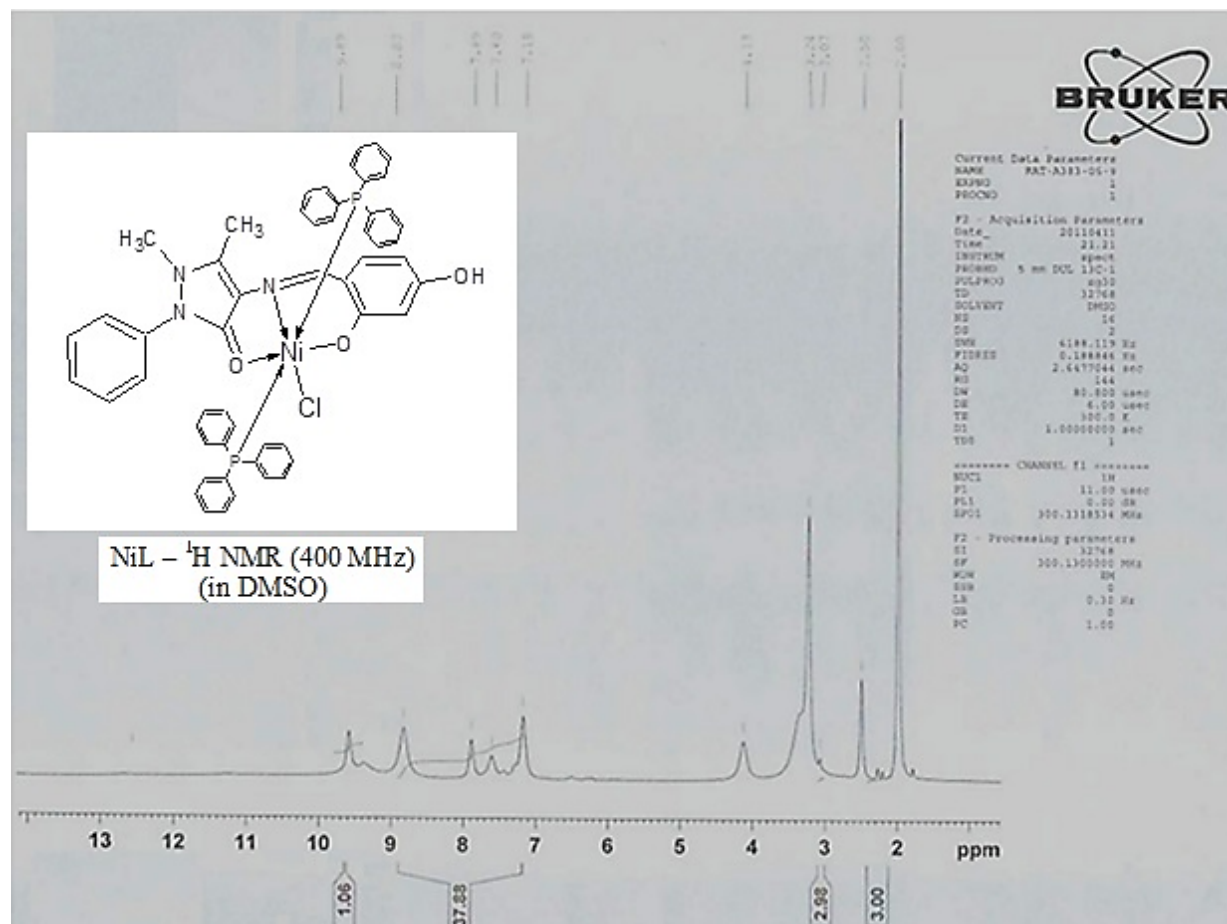
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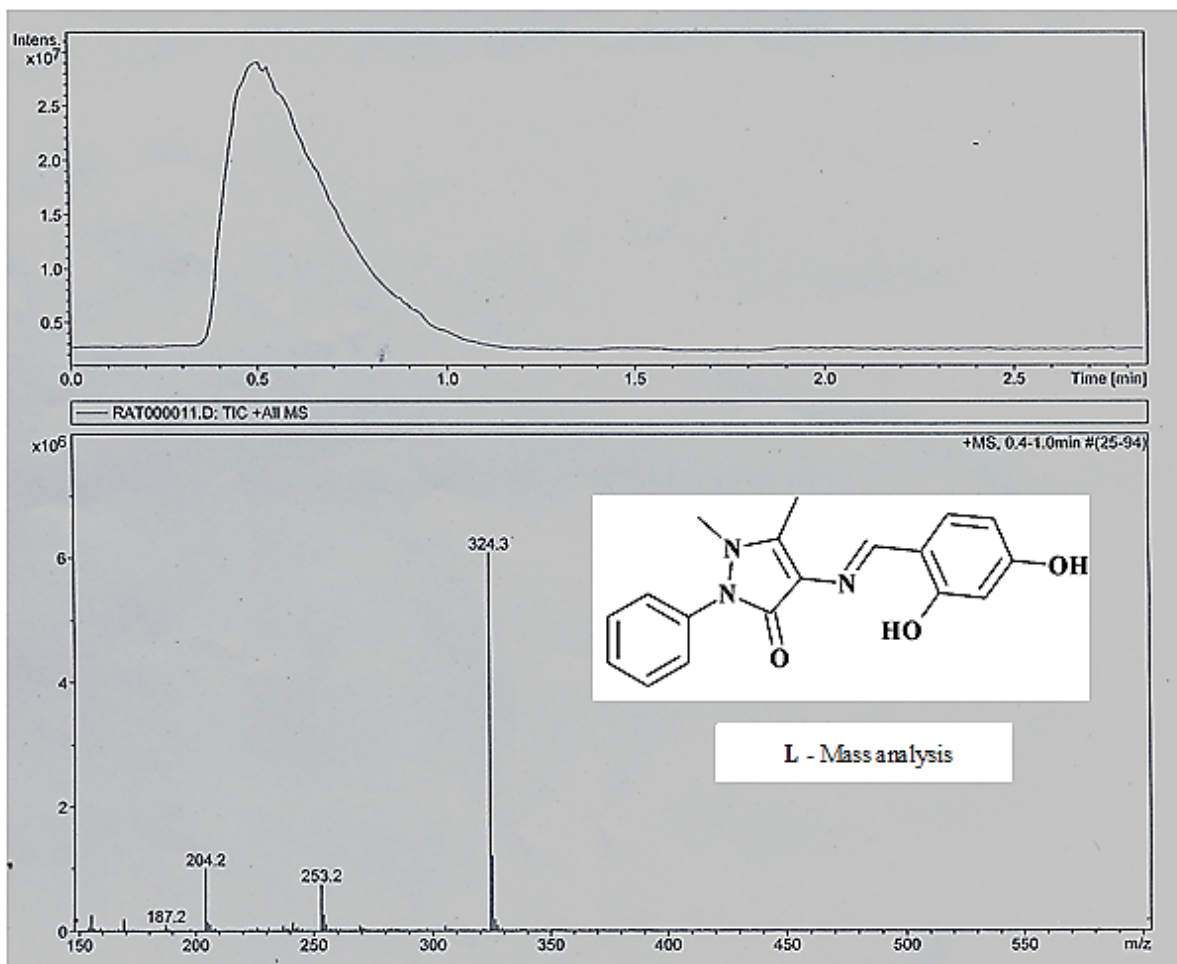
- 1 G. Cahiez, A. Moyeux, J. Buendia, C. Duplais, J. Am. Chem. Soc. 129 (2007) 13788-13789.
- 2 X. Xu, D. Cheng, W. Pei, J. Org. Chem. 71 (2006) 6637-6639.
- 3 S. Punna, D.D. Diaz, M.G. Finn, Synlett. 13 (2004) 2351-2354.
- 4 Y. Yamamoto, R. Suzuki, K. Hattori, H. Nishiyama, Synlett. 7 (2006) 1027-1030.
- 5 T. Hayashi, K. Hayashizaki, T. Kiyoi, Y. Ito, J. Am. Chem. Soc. 110 (1988) 8153-8156.

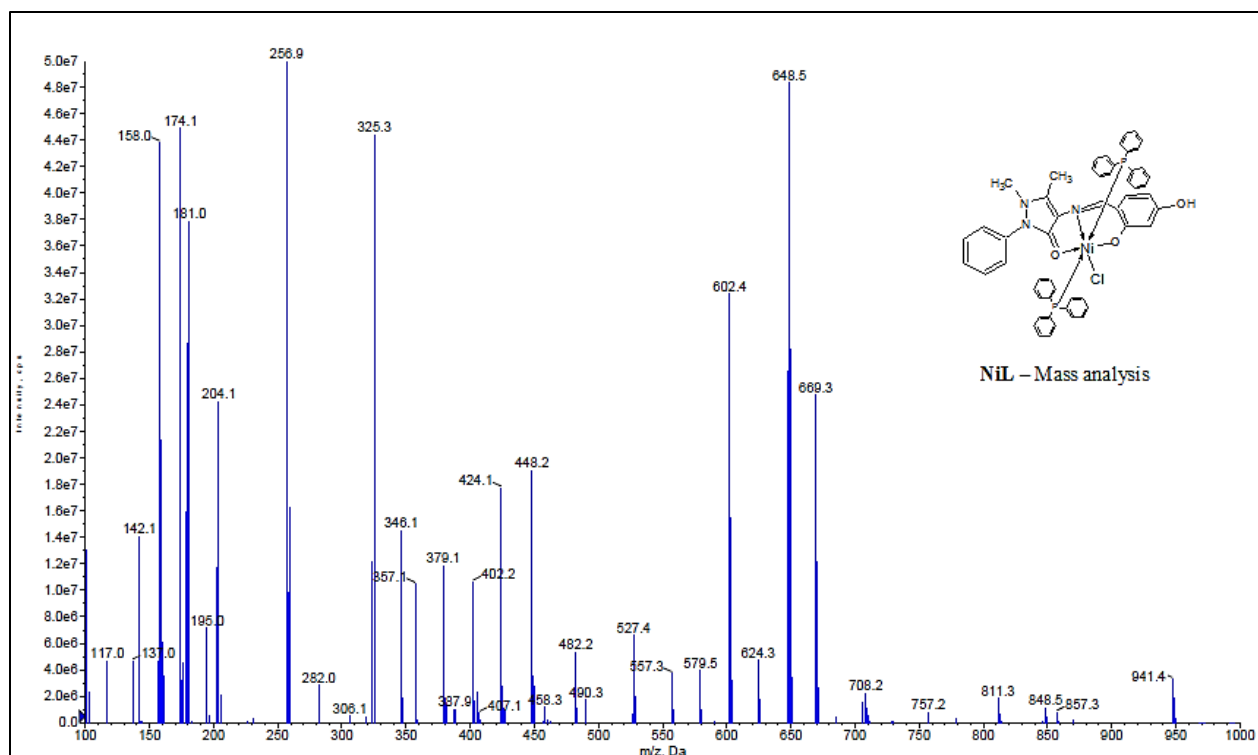


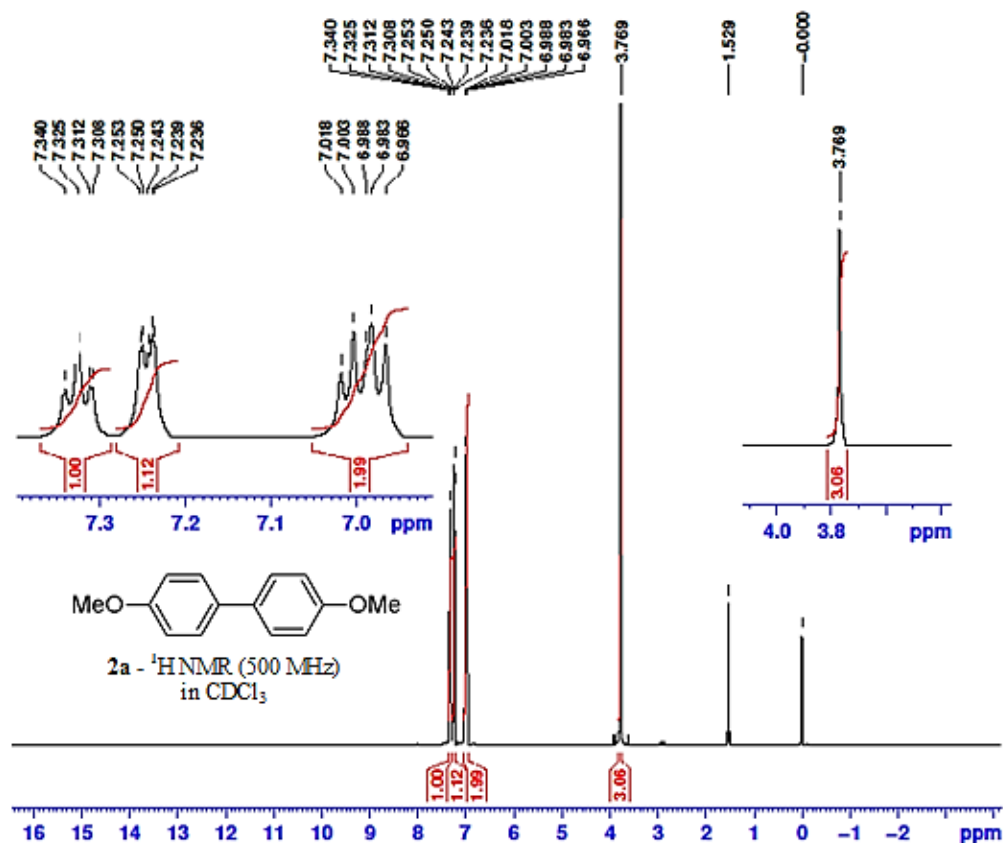
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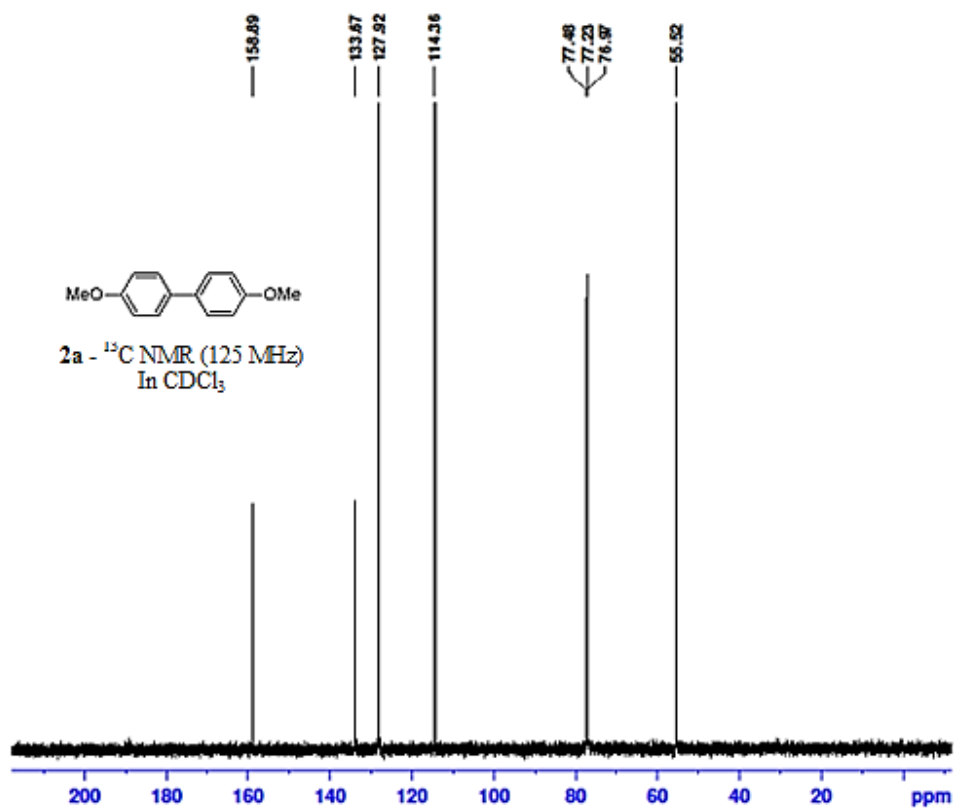


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FIDRES 0.157632 Hz
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RG 75.89
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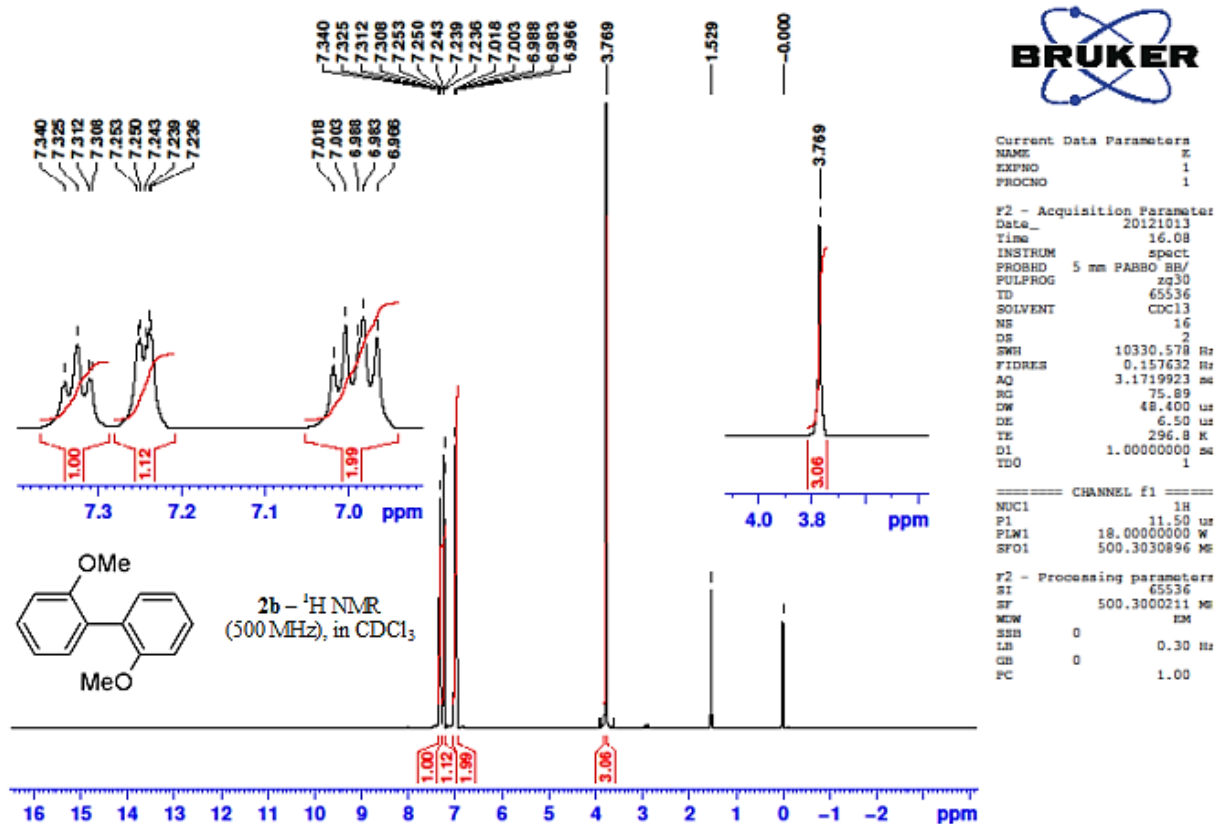
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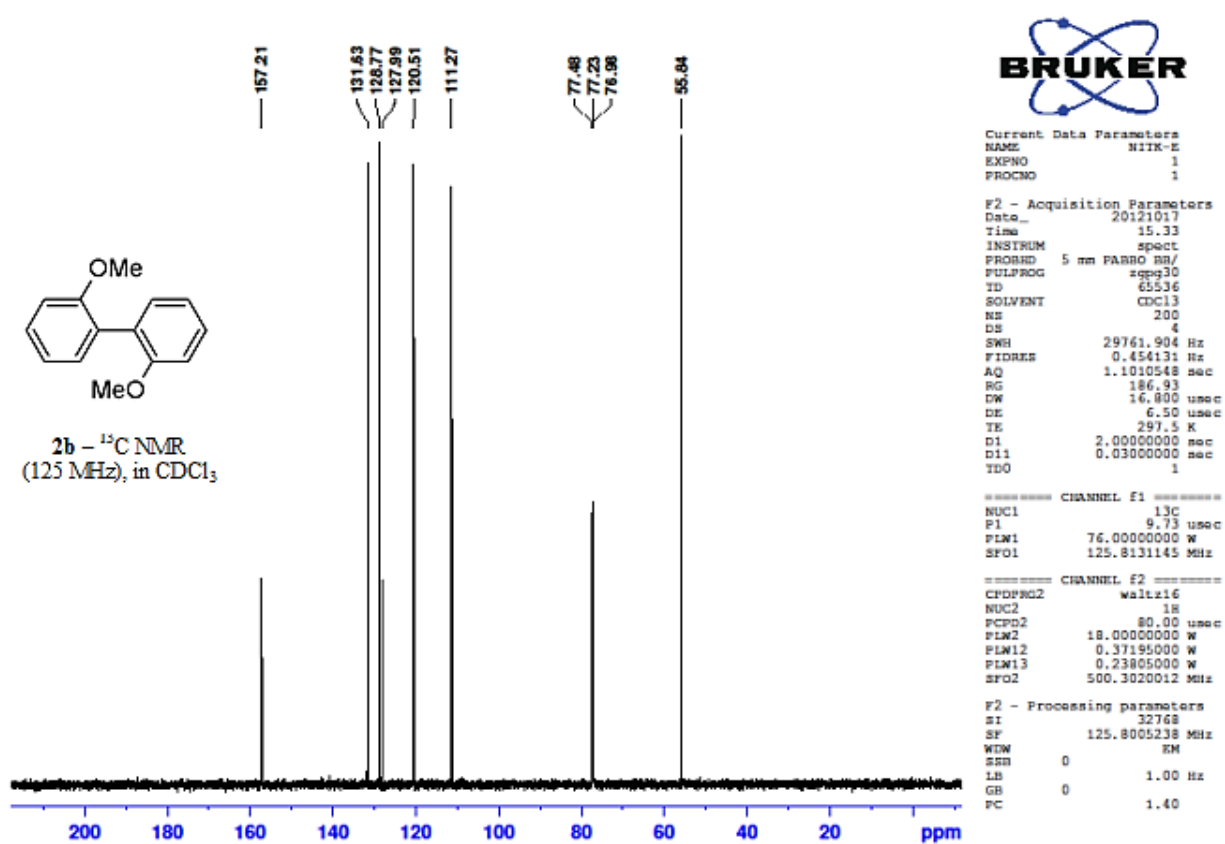
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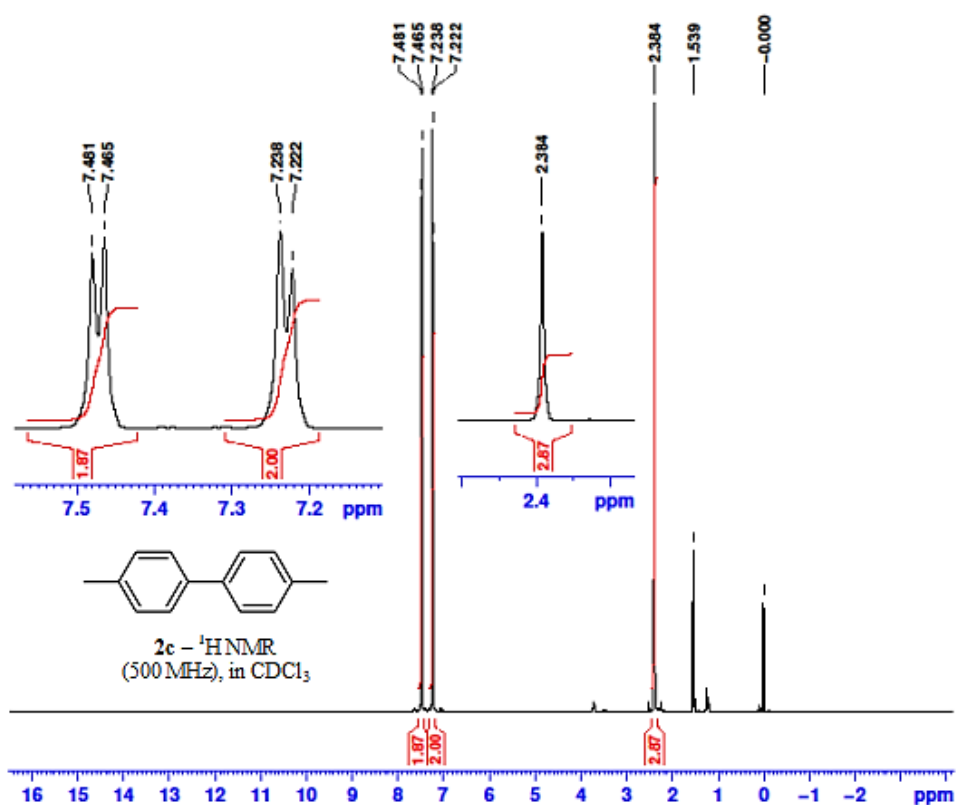
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PCPD2      80.00 usec
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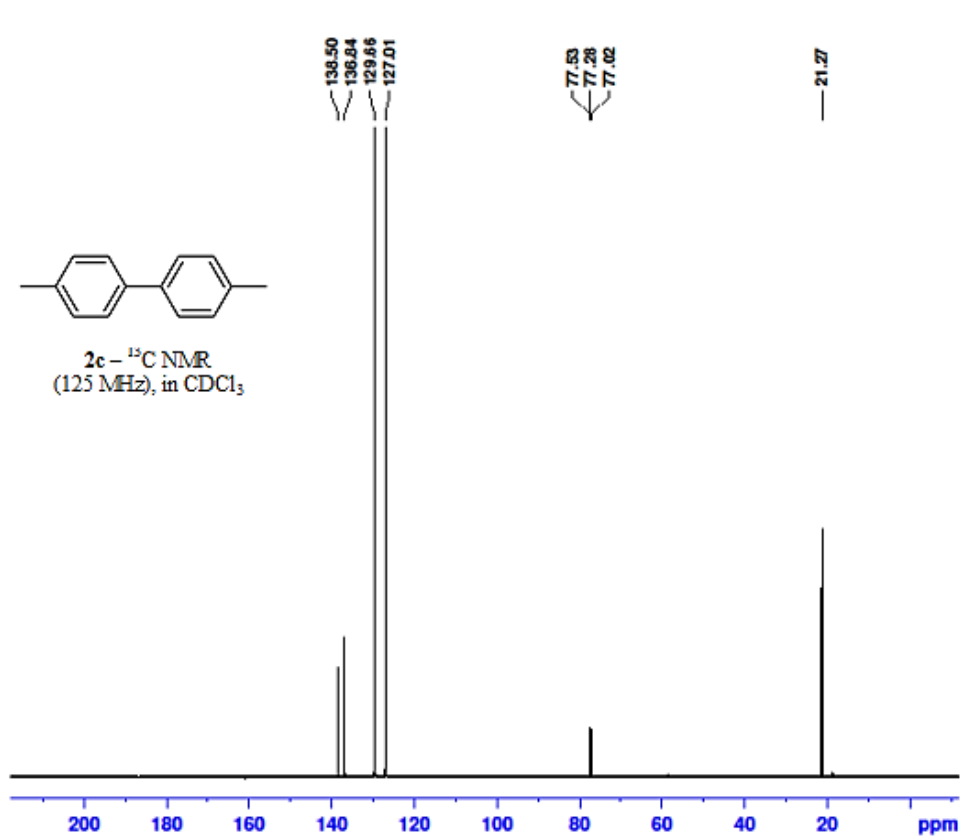


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TE 296.9 K
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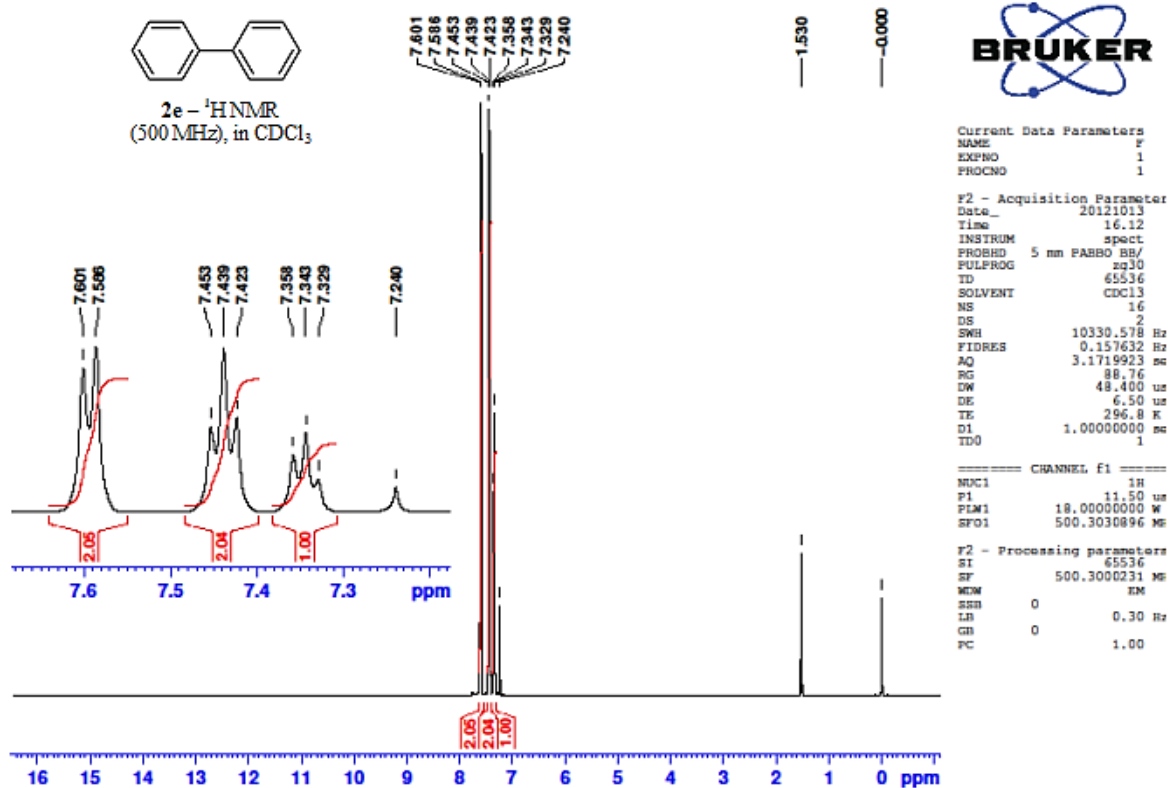
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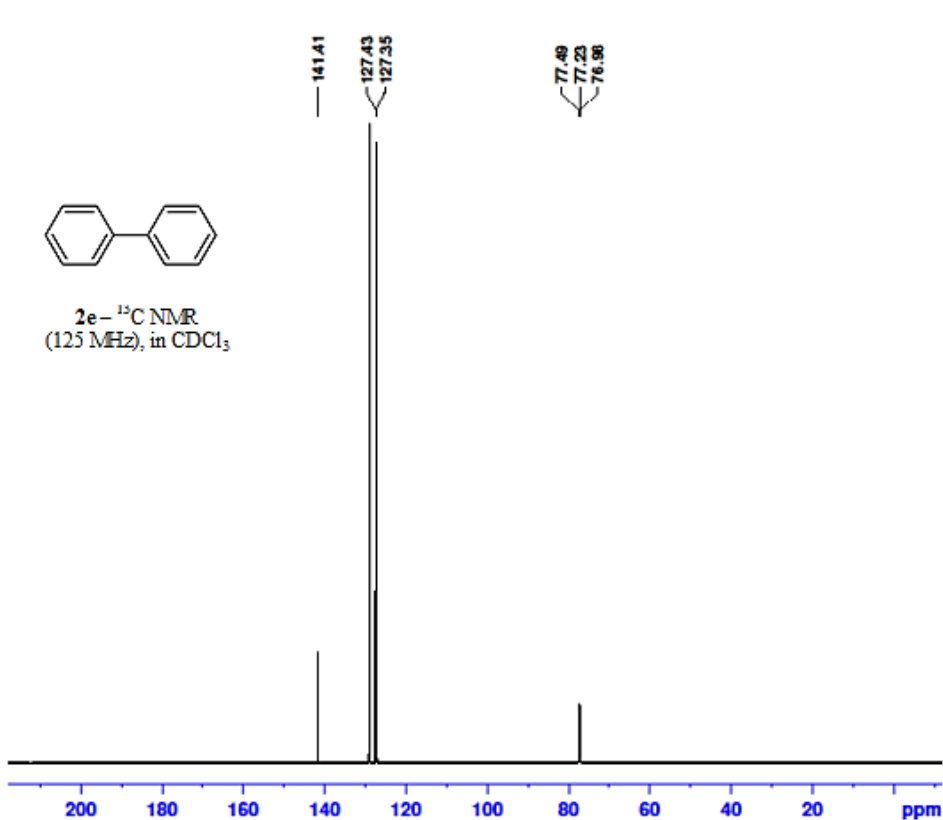
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FIDRES   0.454131 Hz
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DE       6.50 usec
TE       297.9 K
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D11      0.0300000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     13C
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PLW1     76.0000000 W
SFO1     125.8131145 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PLW2     18.0000000 W
PLW12    0.37195000 W
PLW13    0.23805000 W
SFO2     500.3020012 MHz

F2 - Processing parameters
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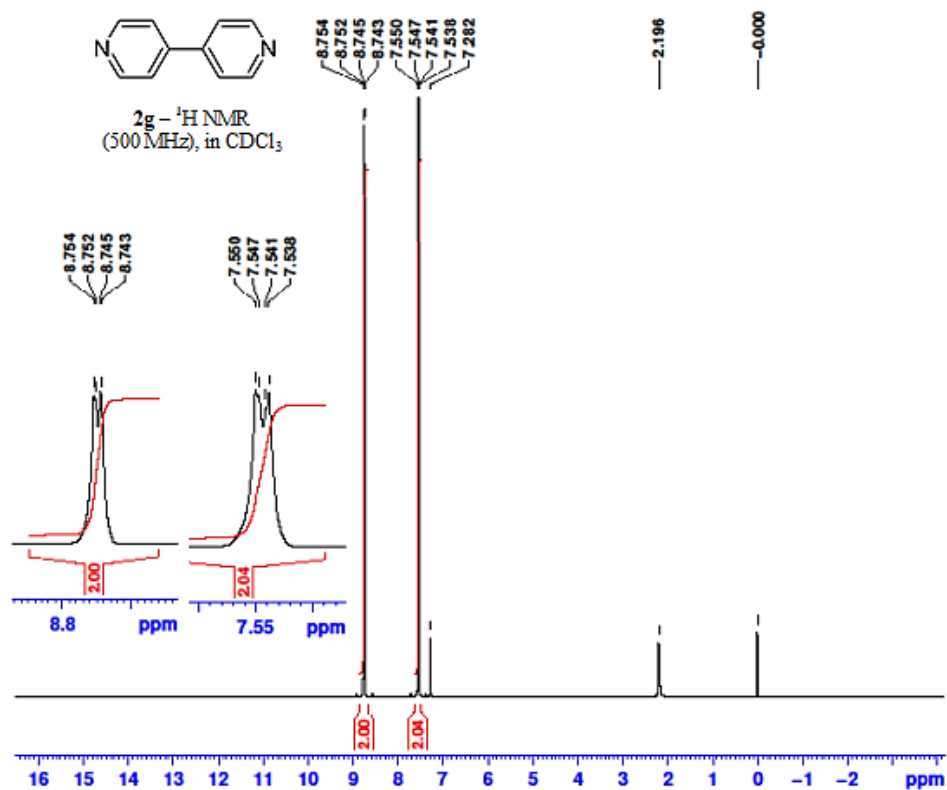
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RG: 186.93
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TE: 298.0 K
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D11: 0.0300000 sec
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==== CHANNEL f2 =====
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NUC2: 1H
PCPD2: 80.00 usec
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PLW12: 0.37195000 W
PLW13: 0.23805000 W
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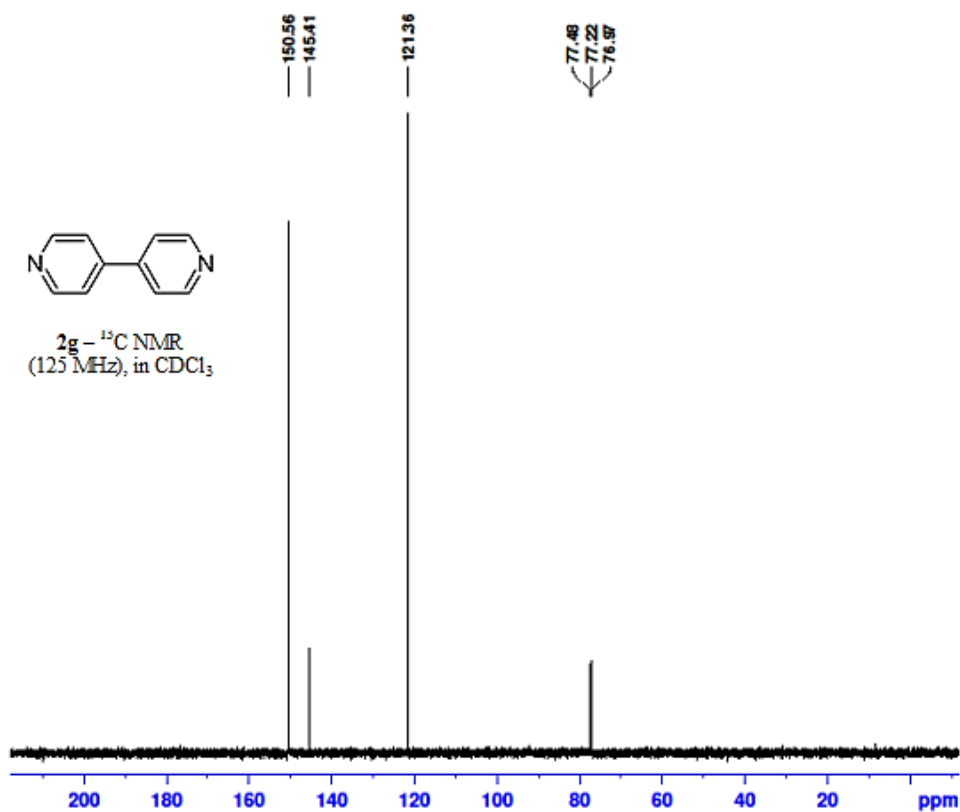


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AQ 3.1719923 sec
RG 75.89
DW 48.400 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TDO 1

===== CHANNEL F1 =====
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PLW1 18.00000000 W
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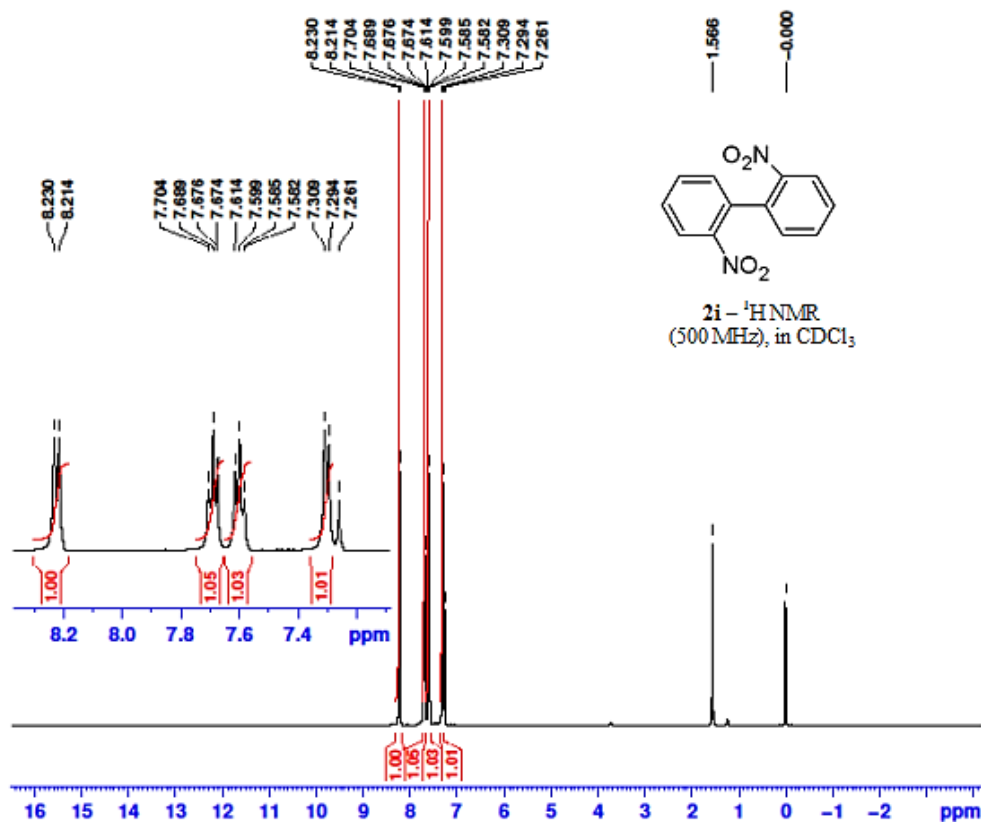
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==== CHANNEL f2 =====
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PLW13 0.23805000 W
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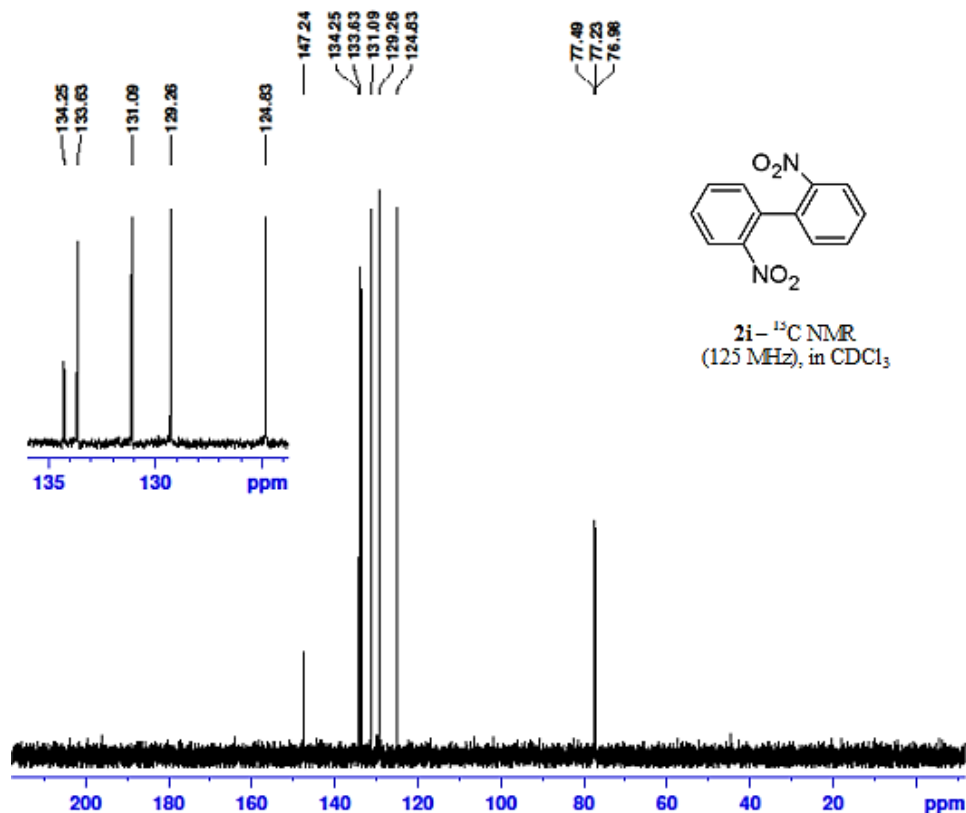


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TE 296.9 K
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TD0 1

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Current Data Parameters
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DE 6.50 usec
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D11 0.0300000 sec
TD0 1

==== CHANNEL F1 =====
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SFO1 125.8131145 MHz

==== CHANNEL F2 =====
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NUC2 ^1H
PCPD2 80.00 usec
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PLW13 0.2380500 W
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