

Electronic Supplementary Information

Reactant cum solvent water: generation of transient λ^3 -hypervalent iodane, its reactivity, mechanism and broad application

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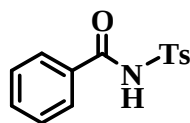
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1. Characterization data of synthesized amides (7)

1.1. *N*-Benzoyl-4-methylbenzenesulfonamide (7a)¹



Yield: 65% (179 mg, 0.65 mmol).

Characteristic: Colourless solid.

Melting point: 144-146 °C.

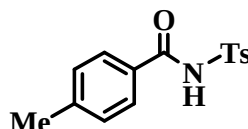
¹H NMR (300 MHz, CDCl₃): δ 2.32 (3H, s), 7.29-7.43 (3H, m), 7.48-7.55 (2H, m), 7.58-7.73 (2H, m), 7.69-7.85 (2H, m), 8.01 (2H, d, *J* = 9.0 Hz).

¹³C NMR (75 MHz, CDCl₃): δ 20.5, 127.2, 127.8, 127.9, 128.0, 128.7, 128.8, 130.2, 131.8, 132.3, 134.5, 136.8, 143.1, 166.8.

FT-IR (KBr, cm⁻¹): 1174, 1326, 1439, 1595, 1732, 3259.

HR-MS (*m/z*) for C₁₄H₁₃NO₃S (M⁺): Calculated 275.0616, found 275.0613.

1.2. 4-Methyl-*N*-(4-methylbenzoyl)benzenesulfonamide (7b)



Yield: 72% (208 mg, 0.72 mmol).

Characteristic: Colourless solid.

Melting point: 137-138 °C.

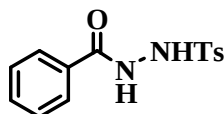
¹H NMR (300 MHz, CDCl₃): δ 2.29 (3H, s), 2.36 (3H, s), 7.24 (2H, d, *J* = 7.8 Hz), 7.33 (2H, d, *J* = 8.1 Hz), 7.74 (2H, d, *J* = 8.1 Hz), 8.04 (2H, d, *J* = 8.1 Hz).

¹³C NMR (75 MHz, CDCl₃): δ 21.5, 29.7, 126.5, 127.2, 127.8, 129.6, 129.7, 134.7, 136.1, 143.1, 172.0.

FT-IR (KBr, cm⁻¹): 1075, 1167, 1254, 1336, 1430, 1595, 1699, 3293.

HR-MS (*m/z*) for C₁₅H₁₅NO₃S (M⁺): Calculated 289.0773, found 289.0778.

1.3. *N*-Benzoyl-4-methylbenzenesulfonohydrazide (7c)



Yield: 60% (174 mg, 0.60 mmol).

Characteristic: Colourless solid.

Melting point: 159-160 °C.

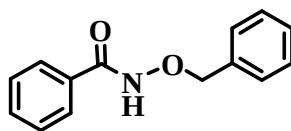
¹H NMR (300 MHz, DMSO-*d*₆): δ 2.40 (3H, s), 7.22-7.40 (4H, m), 7.47-7.59 (3H, m), 7.84-7.92 (3H, m), 11.81 (1H, s).

¹³C NMR (75 MHz, DMSO-*d*₆): δ 26.3, 132.6, 132.9, 133.3, 133.6, 133.9, 134.4, 134.5, 137.2, 137.8, 137.9, 141.2, 169.7.

FT-IR (neat, cm⁻¹): 1170, 1378, 1443, 1595, 1730, 3239.

HR-MS (*m/z*) for C₁₄H₁₄N₂O₃S (M⁺): Calculated 290.0725, found 290.0728.

1.4. *N*-Benzyloxybenzamide (7d)



Yield: 60% (136 mg, 0.60 mmol).

Characteristic: Yellowish solid.

Melting point: 108-110 °C.

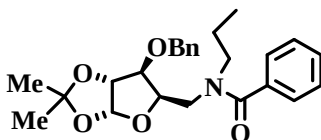
^1H NMR (300 MHz, DMSO- d_6): δ 4.84 (2H, s), 7.25-7.44 (8H, m), 7.66 (2H, d, J = 7.5 Hz), 11.69 (1H, s).

^{13}C NMR (75 MHz, DMSO- d_6): δ 76.5, 126.5, 127.8, 127.9, 128.4, 131.0, 131.8, 135.4, 163.9.

FT-IR (neat, cm^{-1}): 746, 1001, 1144, 1522, 1628, 2968.

HR-MS (m/z) for $\text{C}_{14}\text{H}_{13}\text{NO}_2$ (M^+): Calculated 227.0946, found 227.0945.

1.5. N-(6-Benzyloxy-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-5-ylmethyl)-N-propylbenzamide (7e)



Yield: 65% (276 mg, 0.65 mmol).

Characteristic: Yellow oil.

$[\alpha]_D^{20}$ = -12.4° (c 1.3, CHCl_3).

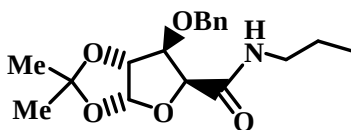
^1H NMR (300 MHz, CDCl_3): δ 1.12-1.19 (6H, m), 1.30-1.35 (5H, m), 2.97-3.04 (2H, m), 3.95-4.06 (1H, m), 4.44-4.55 (4H, m), 5.22 (2H, s), 5.83 (1H, brs), 7.21-7.30 (7H, m), 7.37-7.42 (1H, m), 7.92-7.96 (2H, m).

^{13}C NMR (75 MHz, CDCl_3): δ 11.0, 21.3, 25.9, 26.4, 44.3, 45.1, 66.3, 71.6, 79.1, 81.6, 104.7, 111.3, 126.2, 127.4, 127.6, 127.9, 128.1, 128.2, 129.3, 129.6, 132.5, 132.6, 136.2, 170.1.

FT-IR (neat, cm^{-1}): 1006, 1351, 1408, 1557, 1658, 2621.

HR-MS (m/z) for $\text{C}_{25}\text{H}_{31}\text{NO}_5$ (M^+): Calculated 425.2202, found 425.2200.

1.6. 6-Benzyloxy-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxole-5-carboxylic acid propylamide (7f)



Yield: 75% (251 mg, 0.75 mmol).

Characteristic: Yellow oil.

$[\alpha]_D^{20}$ = -12.3° (c 1.2, CHCl_3).

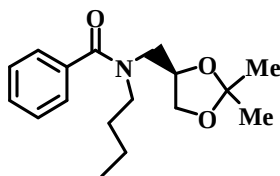
^1H NMR (300 MHz, CDCl_3): δ 1.14 (3H, t, J = 6.0 Hz), 1.24 (3H, s), 1.34-1.36 (2H, m), 1.40 (3H, s), 2.97-3.04 (2H, m), 4.13-4.16 (1H, m), 4.49-4.51 (1H, m), 4.57-4.63 (3H, m), 5.88 (1H, d, J = 3.6 Hz), 7.22-7.30 (5H, m).

^{13}C NMR (75 MHz, CDCl_3): δ 11.0, 21.3, 25.9, 26.4, 44.3, 45.1, 66.3, 71.6, 79.1, 81.6, 104.7, 111.3, 126.5, 127.8, 127.9, 128.4, 136.2, 172.2.

FT-IR (neat, cm^{-1}): 1026, 1076, 1165, 1214, 1373, 1455, 1645, 1739, 1763, 2871, 2931.

HR-MS (m/z) for $\text{C}_{18}\text{H}_{25}\text{NO}_5$ (M^+): Calculated 335.1733, found 335.1737.

1.7. *N*-Butyl-*N*-(2,2-dimethyl-[1,3]dioxolan-4-ylmethyl)benzamide (7g)



Yield: 75% (218 mg, 0.75 mmol).

Characteristic: Yellow oil.

$[\alpha]_D^{20} = -1.5^\circ$ (c 3.4, CHCl₃).

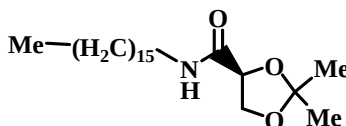
¹H NMR (300 MHz, CDCl₃): δ 0.77-0.83 (3H, m), 1.04 (3H, s), 1.15 (3H, s), 1.23-1.28 (2H, m), 1.42-1.46 (2H, m), 3.22-3.28 (2H, m), 3.50 (1H, brs), 3.93 (2H, brs), 4.50 (2H, d, $J = 2.1$ Hz), 7.18-7.25 (5H, m).

¹³C NMR (75 MHz, CDCl₃): δ 9.1, 14.2, 16.0, 16.1, 23.7, 25.7, 33.8, 58.5, 66.2, 120.9, 121.1, 121.2, 122.4, 122.5, 125.2, 135.7, 161.9.

FT-IR (neat, cm⁻¹): 1271, 1311, 1454, 1542, 1629, 1686, 2829.

HR-MS (m/z) for C₁₇H₂₅NO₃ (M⁺): Calculated 291.1834, found 291.1835.

1.8. 2,2-Dimethyl-[1,3]dioxolane-4-carboxylic acid hexadecylamide (7h)



Yield: 68% (251 mg, 0.68 mmol).

Characteristic: Yellow oil.

$[\alpha]_D^{20} = +8.9^\circ$ (c 1.5, CHCl₃).

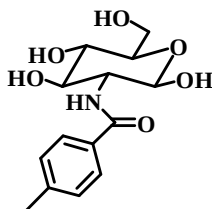
¹H NMR (300 MHz, CDCl₃): δ 0.87 (3H, t, $J = 6.6$ Hz), 1.25-1.31 (32H, m), 1.54-1.61 (2H, m), 3.59 (3H, t, $J = 6.3$ Hz), 3.84-3.91 (2H, m), 3.93-4.03 (1H, m).

¹³C NMR (75 MHz, CDCl₃): δ 14.0, 22.6, 23.8, 24.7, 25.3, 25.5, 25.8, 26.4, 26.9, 27.4, 28.4, 29.3, 29.6, 31.8, 60.4, 66.2, 110.1, 168.3.

FT-IR (neat, cm⁻¹): 1067, 1148, 1217, 1258, 1371, 1456, 1650, 2855, 2990.

HR-MS (m/z) for C₂₂H₄₃NO₃ (M⁺): Calculated 369.3243, found 369.3239.

1.9. 4-Methyl-*N*-(2,4,5-trihydroxy-6-hydroxymethyl-tetrahydro-pyran-3-yl)-benzamide (7i)



Yield: 60% (178 mg, 0.60 mmol).

Characteristic: Yellow oil.

$[\alpha]_D^{20} = +0.1^\circ$ (c 1.6, CHCl₃).

¹H NMR (300 MHz, D₂O): δ 2.38 (3H, s), 3.33-3.40 (1H, m), 3.68-3.97 (5H, m), 4.91 (4H, s), 5.50 (1H, s), 7.24 (2H, d, $J = 7.8$ Hz), 7.65 (2H, d, $J = 8.1$ Hz).

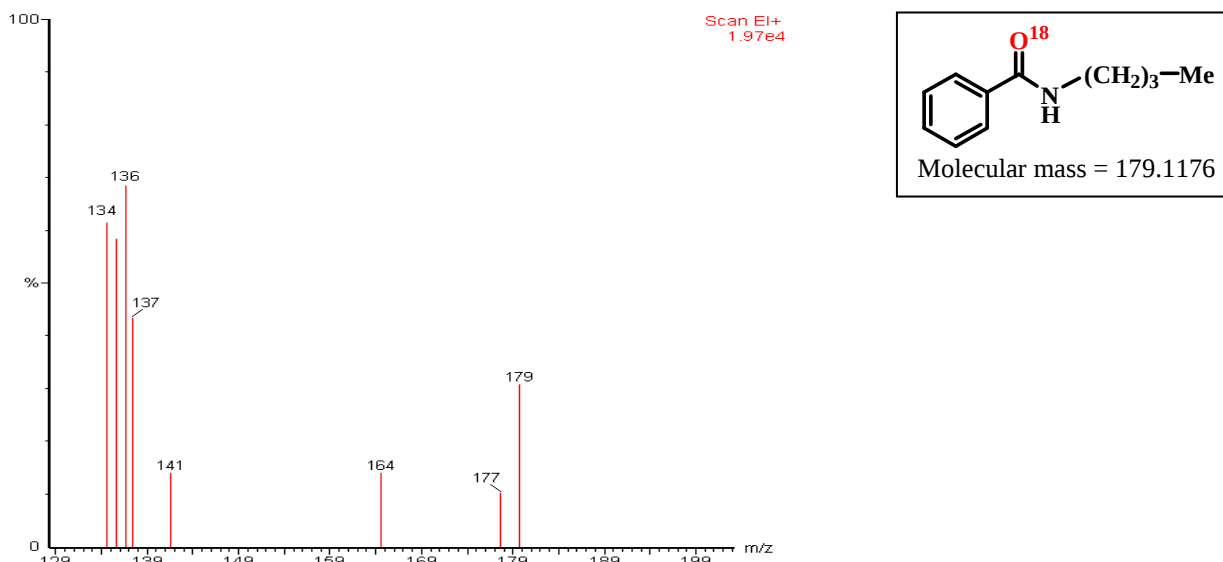
¹³C NMR (75 MHz, D₂O): δ 17.7, 54.2, 65.6, 69.2, 69.7, 71.0, 96.5, 122.5, 123.5, 126.0, 132.7, 165.3.

FT-IR (neat, cm^{-1}): 1048, 1436, 1471, 1634, 1695, 1701, 1712.

HR-MS (m/z) for $\text{C}_{14}\text{H}_{19}\text{NO}_6$ (M^+): Calculated 297.1212, found 297.1217.

2. Isotope labelling experiment

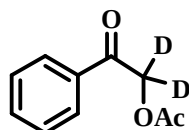
2.1. Incorporation of O^{18} -isotope into amide (9b) from H_2O^{18}



SI Figure 1: EI-MS spectra of 6a

2.2. Incorporation of deuterium into amide (9c) from D_2O

Characterization data of deuterated acetic acid 2-oxo-2-phenyl-ethyl ester (9c)



Yield: 82% (148 mg, 0.82 mmol).

Characteristic: Yellow oil.

^1H NMR (300 MHz, CDCl_3): δ 2.26 (3H, s), 7.48-7.54 (2H, m), 7.61-7.63 (1H, m), 7.92-7.95 (2H, m).

^{13}C NMR (75 MHz, CDCl_3): δ 20.5, 127.7, 128.8, 133.8, 134.3, 170.3, 192.2.

FT-IR (neat, cm^{-1}): 1256, 1275, 1397, 1449, 1493, 1625, 1597, 1690, 2342, 3063.

HR-MS (m/z) for $\text{C}_{10}\text{H}_8\text{D}_2\text{O}_3$ (M^+): Calculated 180.0753, found 180.0752.

3. Investigation of reaction mechanism through NMR study

A) Phenyl acetylene (**1a**) [0 min]

B) $\text{PhI}(\text{OCOMe})_2$ [0 min]

C) Mixture of 1a and $\text{PhI}(\text{OCOMe})_2$ [30 min]

D) After addition of NaHCO_3 to the above mixture formation of a light yellow coloured solid was started.

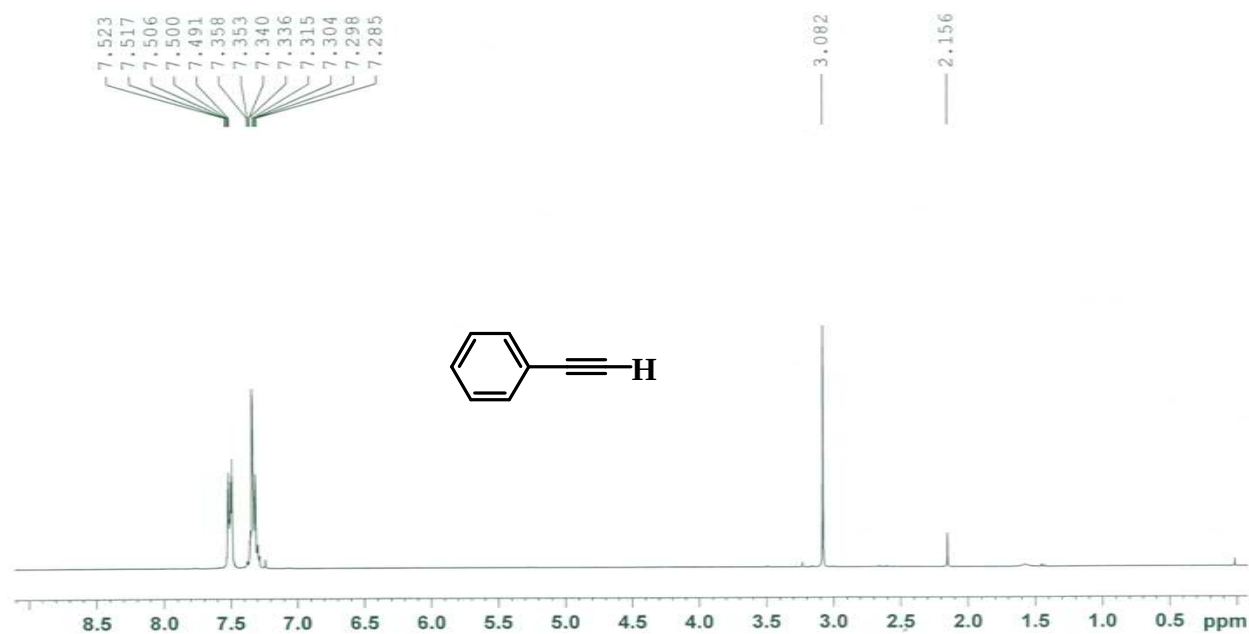
E) Then above mixture was allowed to stir for some times. [1.30 h]

F) n-Butyl amine (**2a**) was added to the above mixture. [1.35 h]

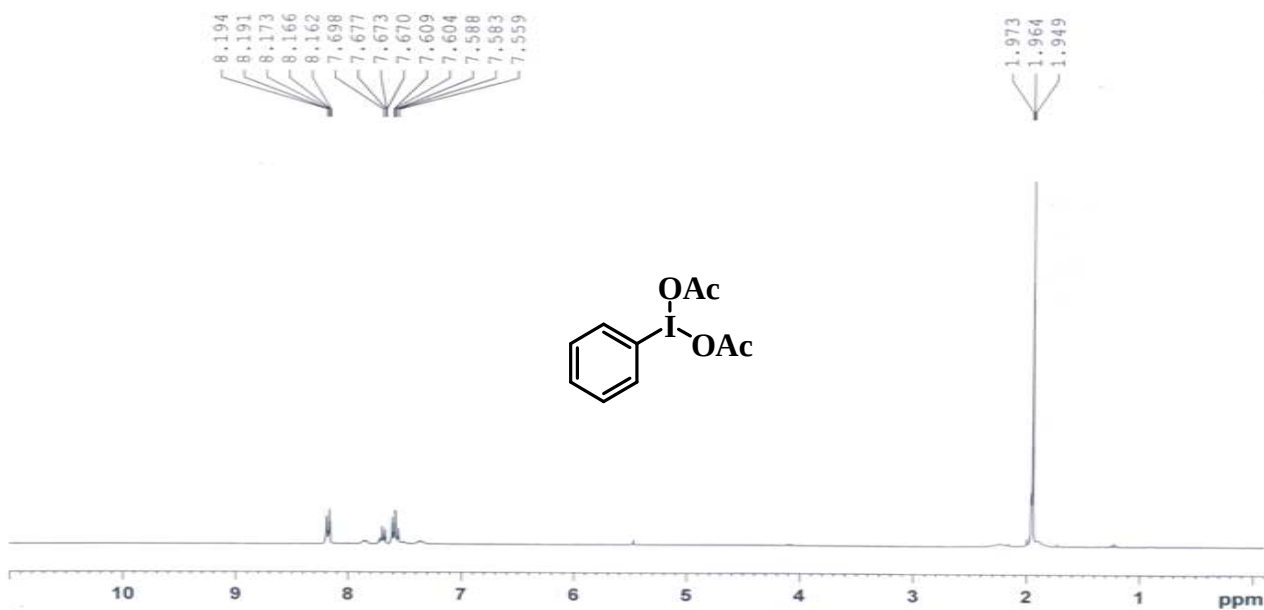
G) Then gradually reaction mixture turned into two layers. [1.45 h]

H) After that yellow organic layer slowly turned into brown. [2.0 h]
All the spectra are given according to the above time sequence.

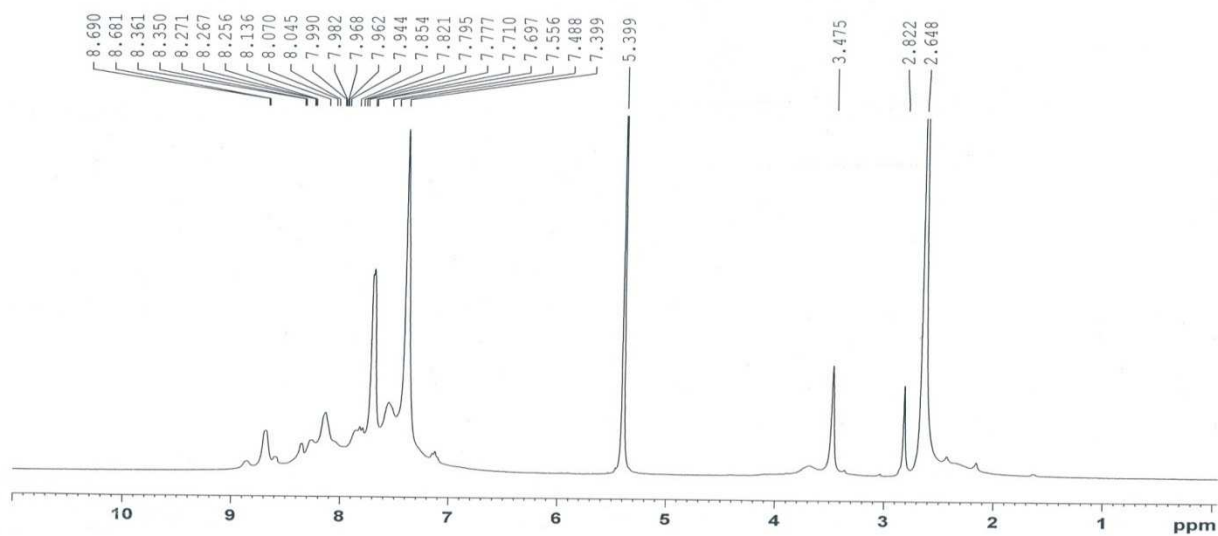
SI Figure 2: ^1H -NMR spectra of A [0 min]



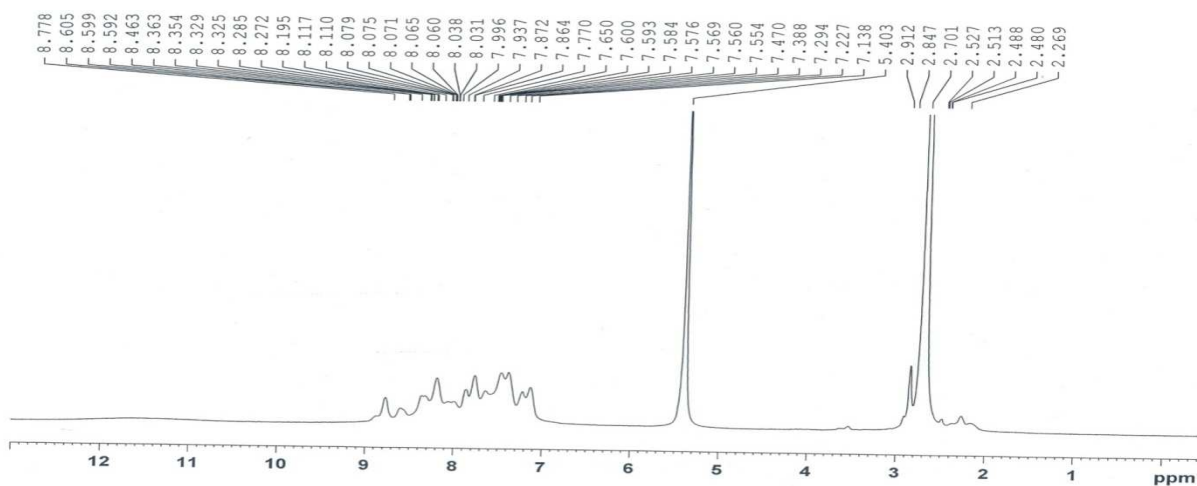
SI Figure 3: ^1H -NMR spectra of B [0 min]



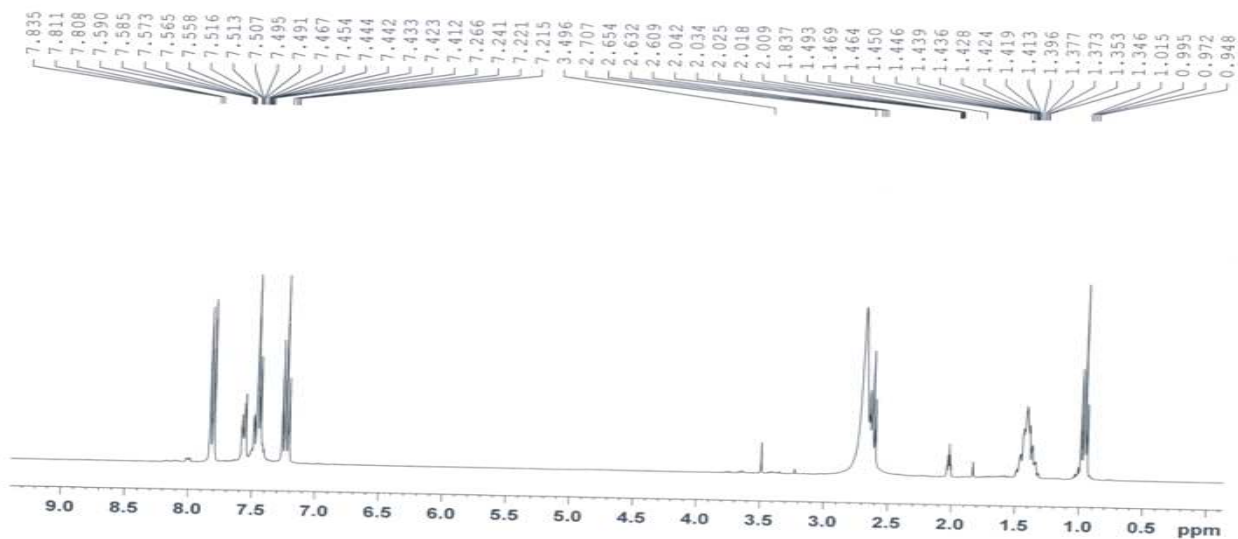
SI Figure 4: ^1H -NMR spectra of C [30 min]



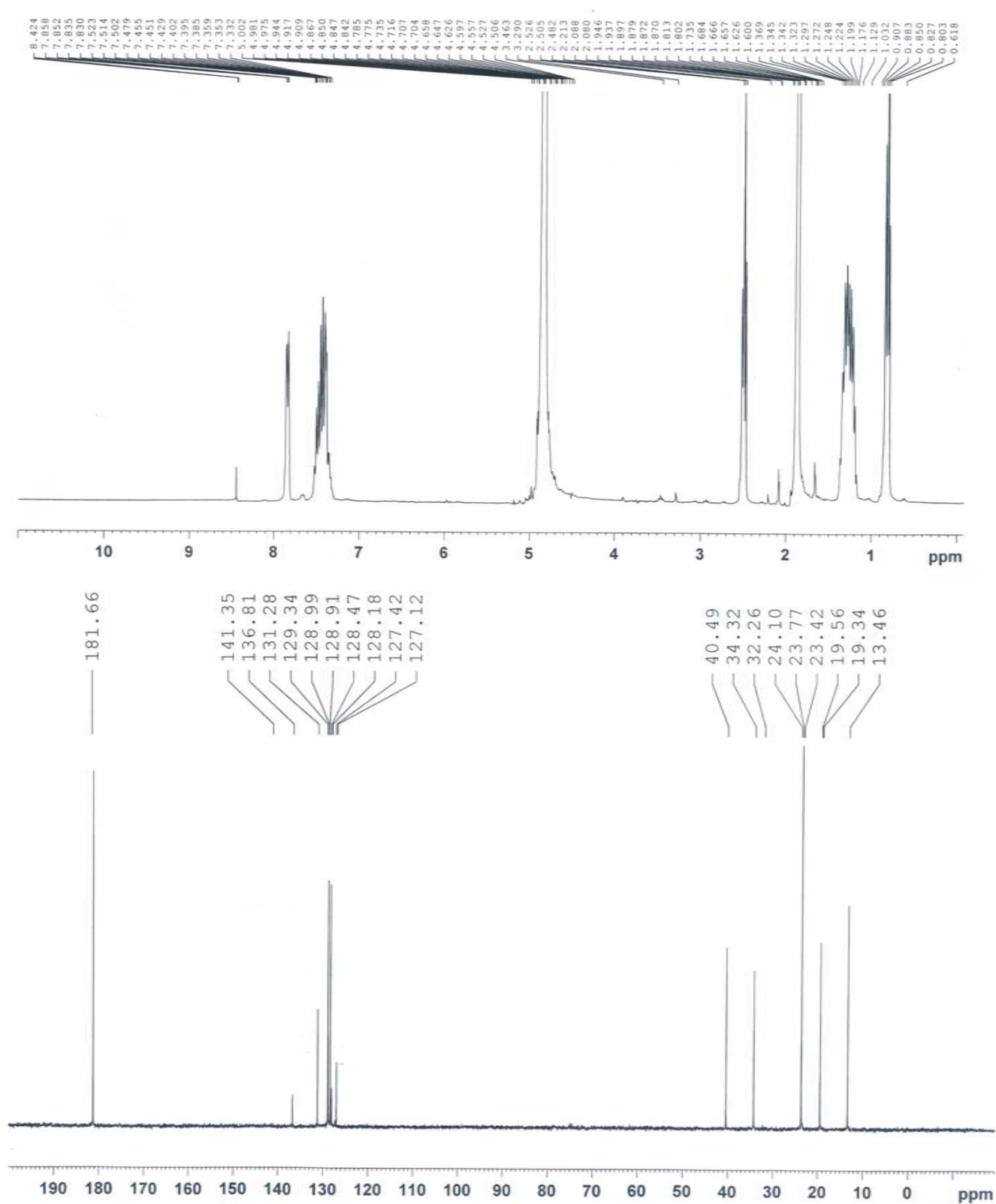
SI Figure 5: ^1H -NMR spectra of E [1.30 h]

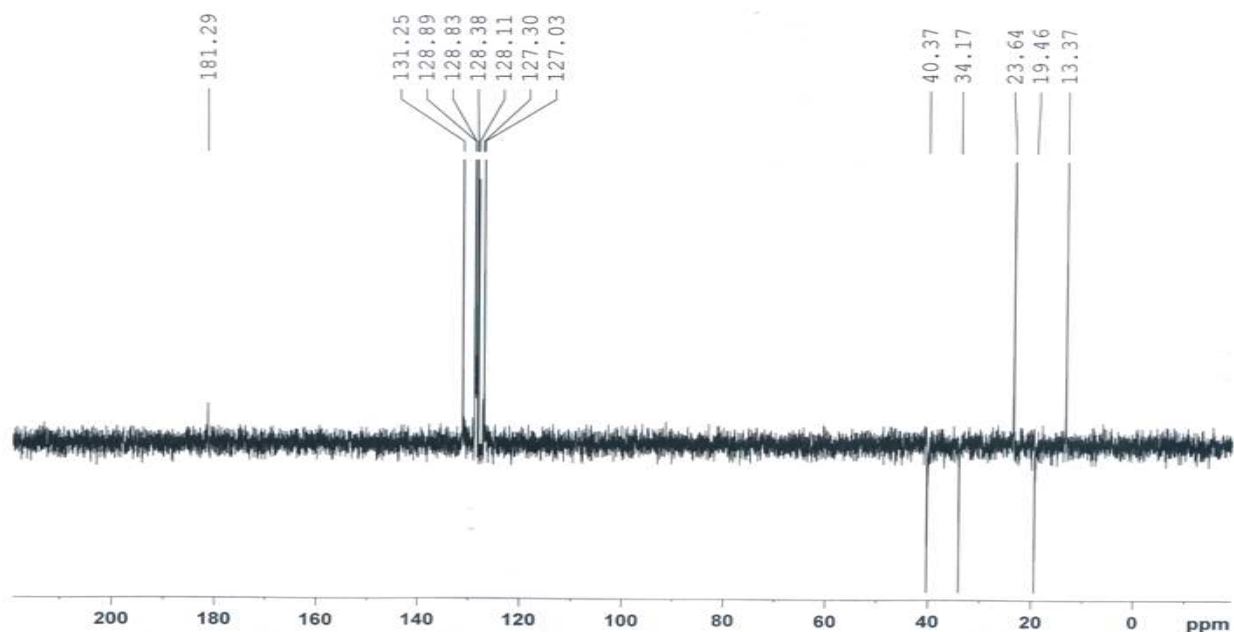


SI Figure 6: ^1H -NMR spectra of F [1:35 h]



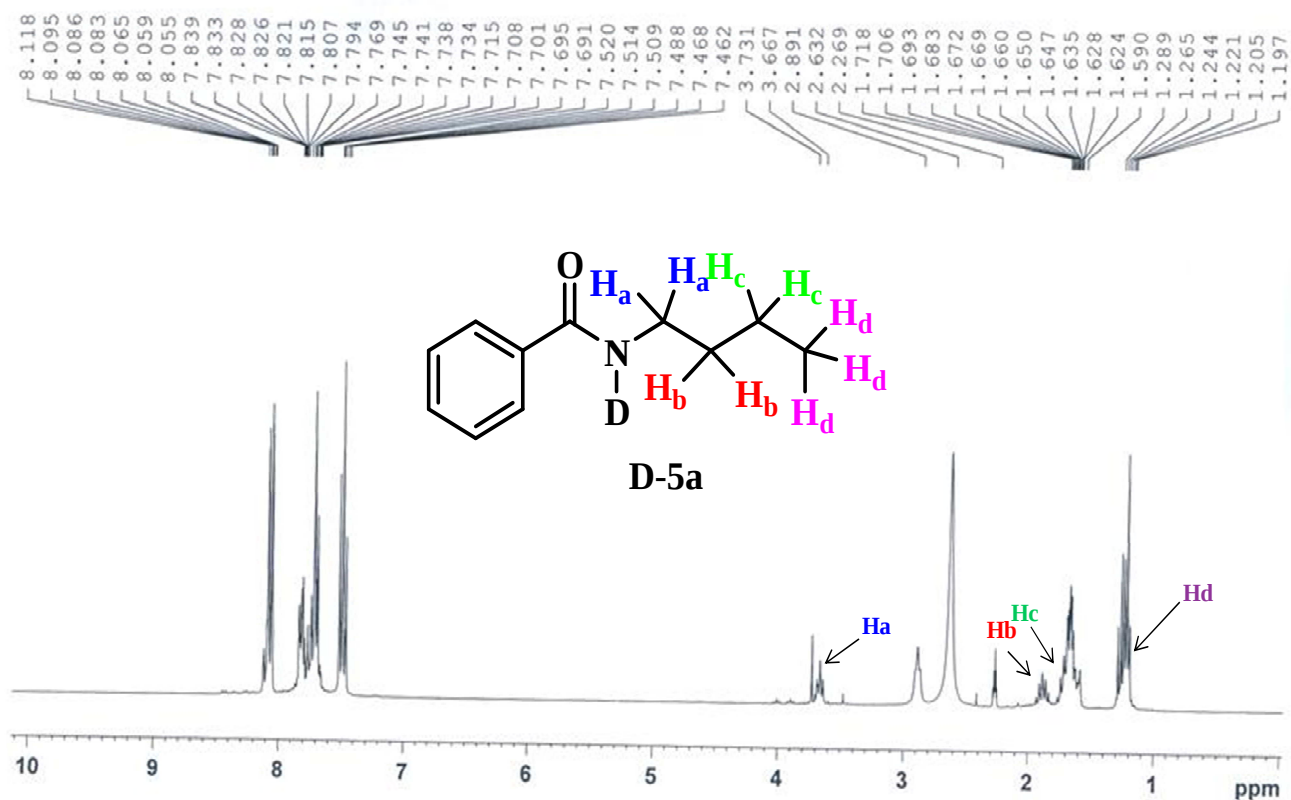
SI Figure 7: ^1H and ^{13}C -NMR and DEPT-135 of the aqueous layer of G [1:45 h]





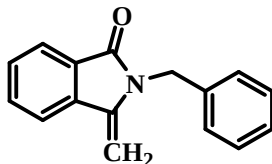
SI Figure 8: ^1H -NMR spectra of H [2.0 h]

It is a mixture of product, PhI and butyl amine : Characteristics proton peaks are assigned below



4. Characterization data of intermediate VIII

- 2-benzyl-3-methylene-2,3-dihydro-isoindol-1-one²



Yield: 50% (120 mg, 0.50 mmol).

Characteristic: Yellow oil.

¹H NMR (300 MHz, CDCl₃): δ 4.81 (1H, d, *J* = 2.4 Hz), 5.02 (2H, s), 5.16 (1H, d, *J* = 2.1 Hz), 7.24-7.36 (5H, m), 7.51-7.58 (2H, m), 7.60-7.73 (1H, m), 7.84-7.91 (1H, m).

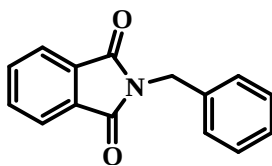
¹³C NMR (75 MHz, CDCl₃): δ 43.1, 89.8, 119.8, 123.3, 127.1, 127.3, 128.5, 129.4, 132.0, 136.4, 136.8, 141.6, 167.2.

FT-IR (neat, cm⁻¹): 1350, 1395, 1456, 1470, 1495, 1638, 1766, 2925.

HR-MS (*m/z*) for C₁₆H₁₃NO (M⁺): Calculated 235.0997, found 235.0996.

5. Characterization data of *N*-substituted phthalimides (8a-f)

5.1. 2-Benzyl-isoindole-1,3-dione (8a)³



Yield: 65% (154 mg, 0.65 mmol).

Characteristic: Colourless solid.

Melting range: 113-115 °C

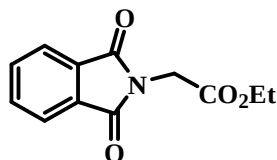
¹H NMR (300 MHz, CDCl₃): δ 4.53 (2H, d, *J* = 5.7 Hz), 7.29-7.38 (5H, m), 7.46 (2H, dd, *J* = 5.4, 3.3 Hz), 7.62 (2H, dd, *J* = 5.4, 3.3 Hz).

¹³C NMR (75 MHz, CDCl₃): δ 43.6, 127.0, 127.6, 128.4, 129.7, 134.9, 138.2, 168.9.

FT-IR (KBr, cm⁻¹): 1233, 1309, 1440, 1545, 1630, 3062.

HR-MS (*m/z*) for C₁₅H₁₂NO₂(M⁺+H): Calculated 238.0868, found 238.0873.

5.2. (1,3-Dioxo-1,3-dihydro-isoindol-2-yl)acetic acid ethyl ester (9b)



Yield: 60% (139 mg, 0.60 mmol).

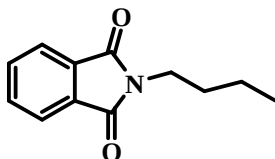
Characteristic: Colourless solid.

Melting range: 112-114 °C

¹H NMR (300 MHz, CDCl₃): δ 1.30 (3H, t, *J* = 7.2 Hz), 4.24 (2H, q, *J* = 7.2 Hz), 4.45 (2H, s), 7.76 (2H, dd, *J* = 5.4, 3.3 Hz), 7.90 (2H, dd, *J* = 5.4, 3.3 Hz).

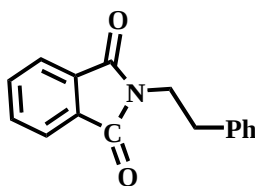
^{13}C NMR (75 MHz, CDCl_3): δ 14.0, 38.9, 61.8, 123.5, 132.0, 134.1, 167.2, 167.4.
FT-IR (KBr, cm^{-1}): 1432, 1544, 1630, 1736, 2928.
HR-MS (m/z) for $\text{C}_{12}\text{H}_{11}\text{NO}_4(\text{M}^+)$: Calculated 233.0688, found 233.0685.

5.3. 2-Butyl-isoindole-1,3-dione (9c)



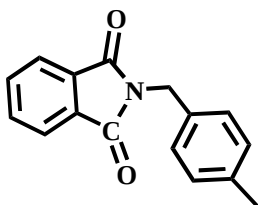
Yield: 60% (122 mg, 0.60 mmol).
Characteristic: Colourless solid.
Melting range: 107-110 °C
 ^1H NMR (300 MHz, CDCl_3): δ 0.89 (3H, t, J = 7.2 Hz), 1.28-1.52 (4H, m), 3.18 (2H, q, J = 6.6 Hz), 7.76 (2H, dd, J = 5.4, 3.3 Hz), 7.90 (2H, dd, J = 5.4, 3.3 Hz).
 ^{13}C NMR (75 MHz, CDCl_3): δ 14.1, 20.0, 31.5, 38.6, 128.1, 129.6, 136.6, 168.5.
FT-IR (KBr, cm^{-1}): 1434, 1541, 1631, 2108, 2926.
HR-MS (m/z) for $\text{C}_{12}\text{H}_{13}\text{NO}_2(\text{M}^+)$: Calculated 203.0946, found 203.0947.

5.4. 2-Phenethylisoindole-1,3-dione (9d)



Yield: 70% (176 mg, 0.70 mmol).
Characteristic: Colourless solid.
Melting point: 128-130 °C.
 ^1H NMR (300 MHz, CDCl_3): δ 2.85-2.92 (2H, m), 3.79-3.85 (2H, m), 7.10-7.19 (5H, m), 7.57-7.63 (2H, m), 7.70-7.74 (2H, m).
 ^{13}C NMR (75 MHz, CDCl_3): δ 34.6, 39.2, 123.2, 126.6, 128.5, 128.8, 132.0, 133.8, 138.0, 168.1.
FT-IR (KBr, cm^{-1}): 1032, 1234, 1400, 1601, 1713, 1760, 2936.
HR-MS (m/z) for $\text{C}_{16}\text{H}_{13}\text{NO}_2(\text{M}^+)$: Calculated 251.0946, found 251.0941.

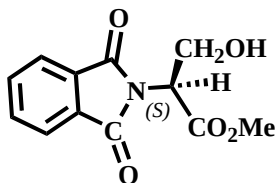
5.5. 2-(4-Methylbenzyl)isoindole-1,3-dione (9e)



Yield: 64% (161 mg, 0.64 mmol).
Characteristic: Colourless solid.
Melting range: 117-119 °C.
 ^1H NMR (300 MHz, CDCl_3): δ 2.23 (3H, s), 4.36 (2H, d, J = 5.7 Hz), 7.03 (2H, d, J = 7.8 Hz), 7.10 (2H, d, J = 7.8 Hz), 7.32-7.36 (2H, m), 7.45-7.49 (2H, m).

^{13}C NMR (75 MHz, CDCl_3): δ 21.0, 43.8, 127.7, 128.3, 129.3, 130.1, 134.4, 134.6, 137.1, 168.8.
FT-IR (KBr, cm^{-1}): 1232, 1312, 1430, 1543, 1630, 2968.
HR-MS (m/z) for $\text{C}_{16}\text{H}_{13}\text{NO}_2$ (M^+): Calculated 251.0946, found 251.0948.

5.6. 2-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-3-hydroxypropionic acid methyl ester (9f)



Yield: 50% (125 mg, 0.50 mmol).

Characteristic: Colourless oil.

$[\alpha]_{\text{D}}^{20} = -17.3^\circ$ (c 1.2, CHCl_3).

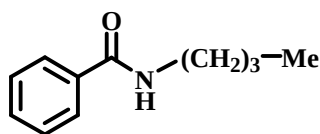
^1H NMR (300 MHz, CDCl_3): δ 3.65 (3H, s), 4.08-4.10 (2H, m), 4.91 (1H, t, $J = 5.4$ Hz), 7.61-7.65 (2H, m), 7.74 (2H, dd, $J = 5.4, 3.0$ Hz).

^{13}C NMR (75 MHz, CDCl_3): δ 52.8, 54.6, 60.8, 123.7, 123.9, 131.6, 134.4, 168.0, 168.4.

FT-IR (neat, cm^{-1}): 1465, 1609, 1715, 1745, 1772, 3479.

HR-MS (m/z) for $\text{C}_{12}\text{H}_{11}\text{NO}_5$ (M^+): Calculated 249.0637, found 249.0642.

6. Characterization data of n-butylbenzamide (9a)



Yield: 85% (150 mg, 0.85 mmol).

Characteristic: Yellow oil.

^1H NMR (300 MHz, CDCl_3): δ 0.97 (3H, t, $J = 6.0$ Hz), 1.36-1.48 (2H, m), 1.56-1.66 (2H, m), 3.43-3.50 (2H, m), 6.15 (1H, brs), 7.40-7.52 (3H, m), 7.75-7.78 (2H, m).

^{13}C NMR (75 MHz, CDCl_3): δ 13.7, 20.1, 31.7, 39.8, 126.8, 128.5, 131.3, 134.8, 167.5.

FT-IR (neat, cm^{-1}): 1308, 1490, 1542, 1578, 1603, 1640, 2872, 2931, 2959.

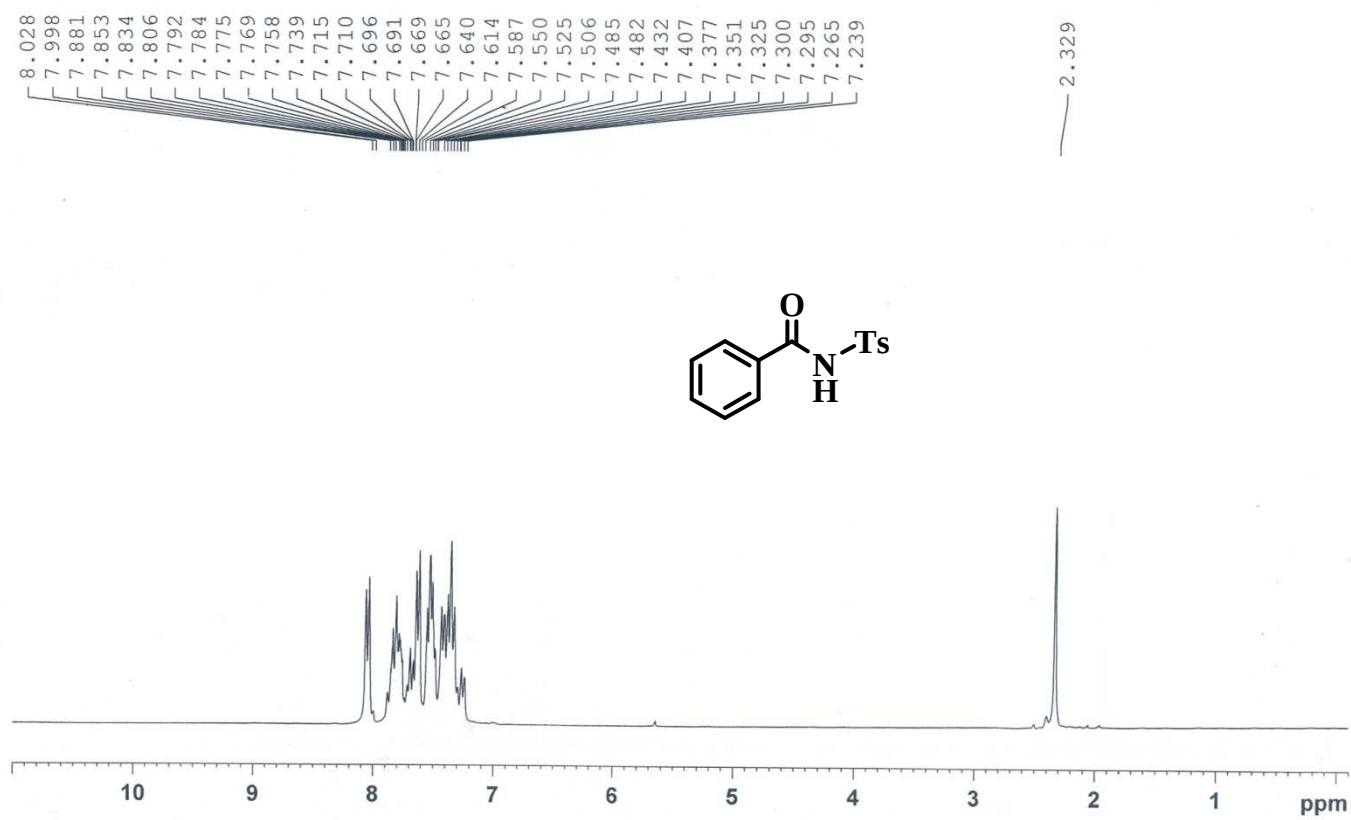
HR-MS (m/z) for $\text{C}_{11}\text{H}_{15}\text{NO}$ (M^+): Calculated 177.1154, found 177.1156.

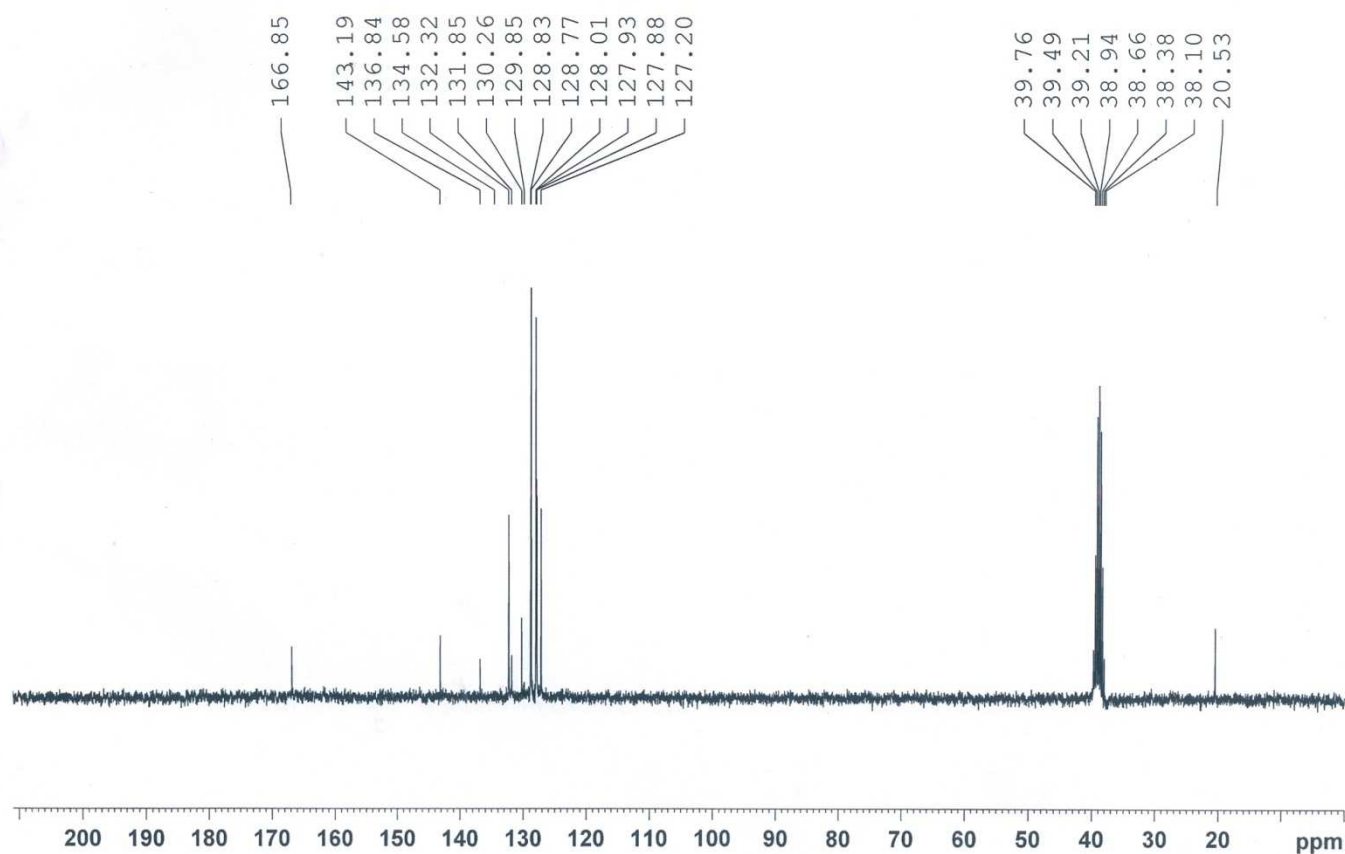
7. Reference

- (1) Wu, Xinghua and Hu, Longqin; *J. Org. Chem.* **2007**, 72, 765-774.
- (2) Howe, R. K. and Shelton, B. R. *J. Org. Chem.* **1990**, 55, 4603-4607.
- (3) V. Pace, P. Hoyos, M. Fernandez, J. V. Sinisterra, and A. R. Alcantara; *Green Chem.* **2010**, 12, 1380-1382.

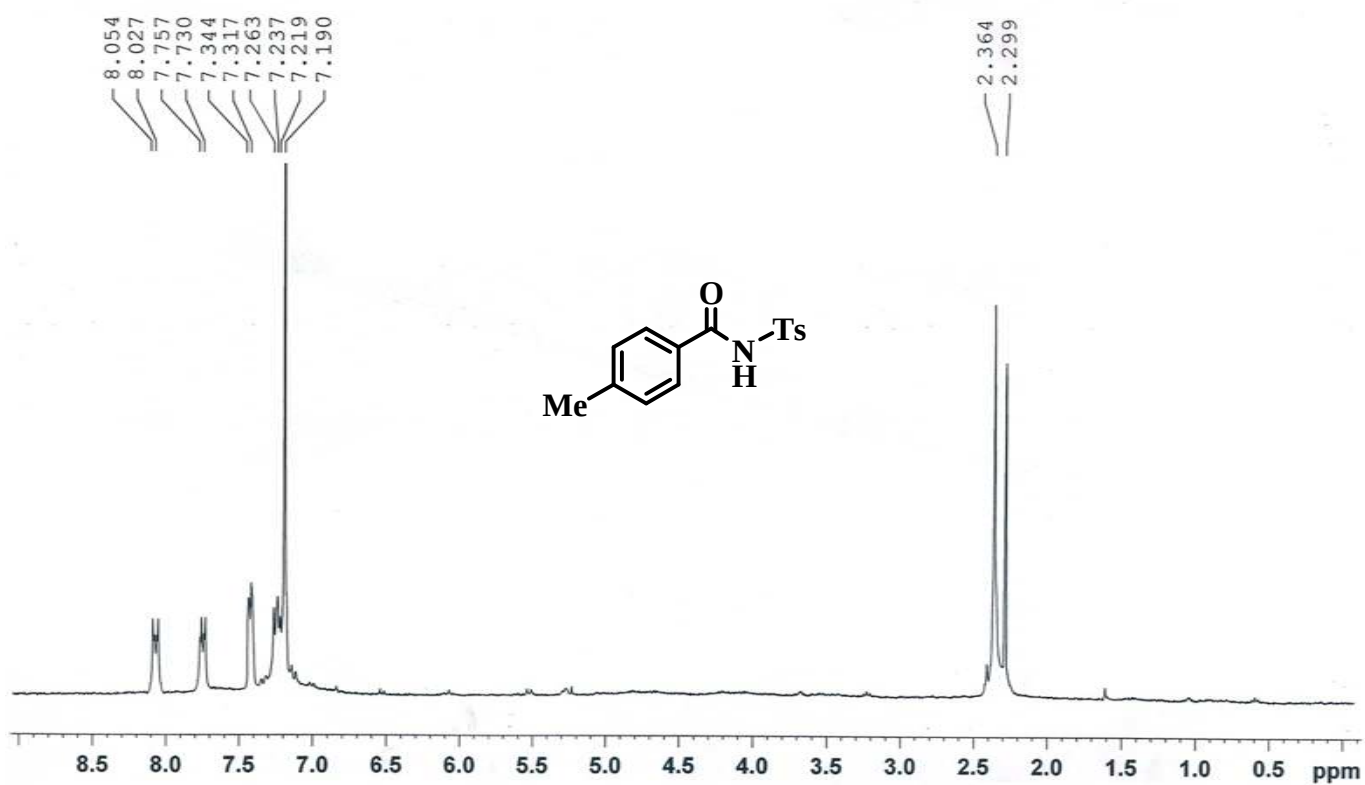
8. ^1H and ^{13}C -NMR spectra of the compounds (7a-i, VIII, 8a-f, 9a, 9c)

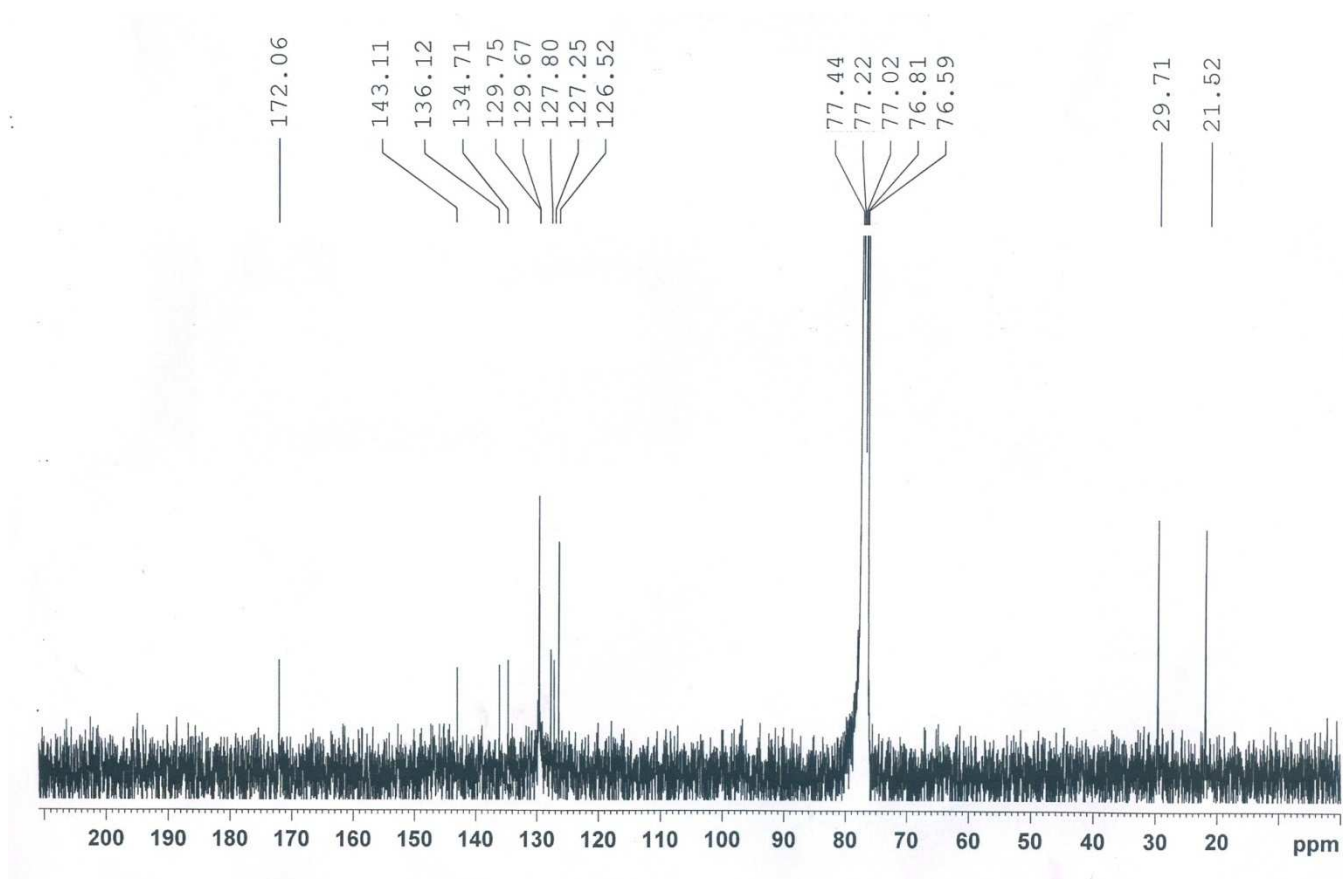
SI Figure 9: ^1H and ^{13}C -NMR spectra of compound 7a



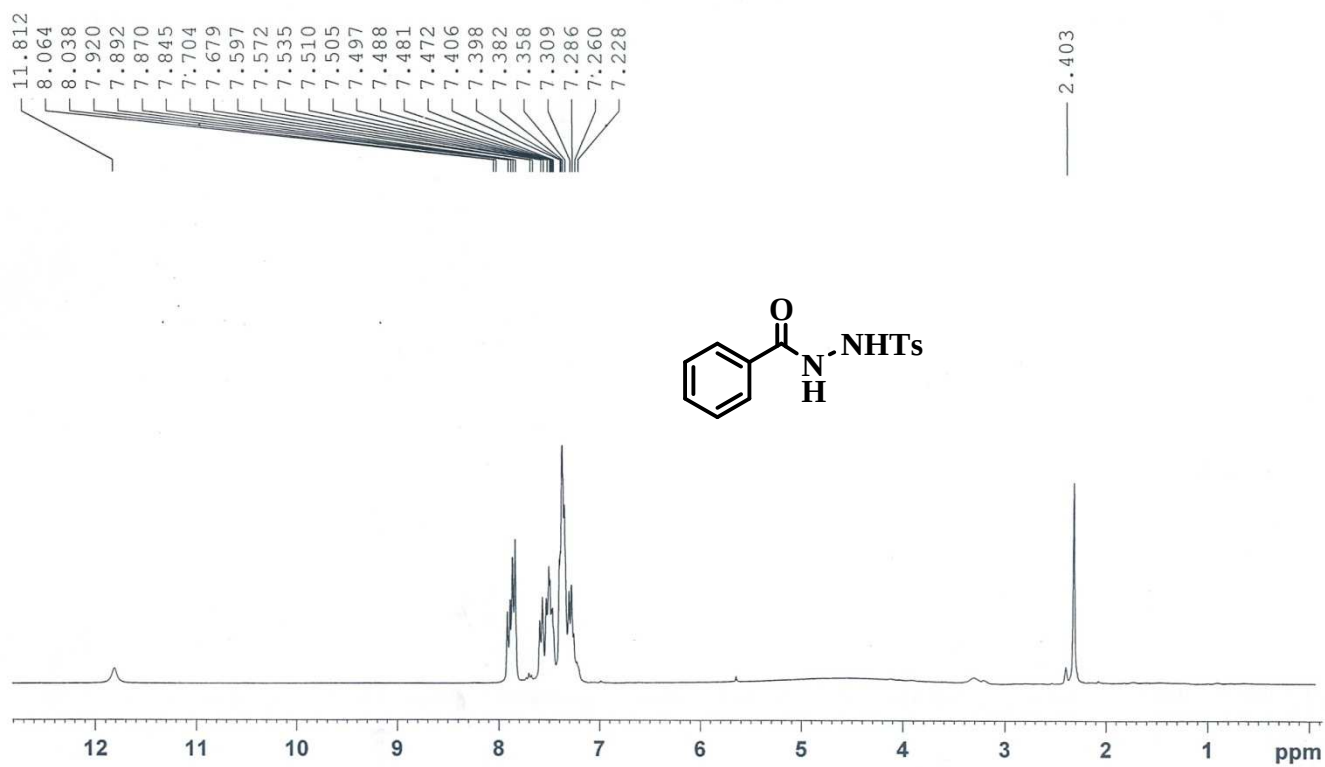


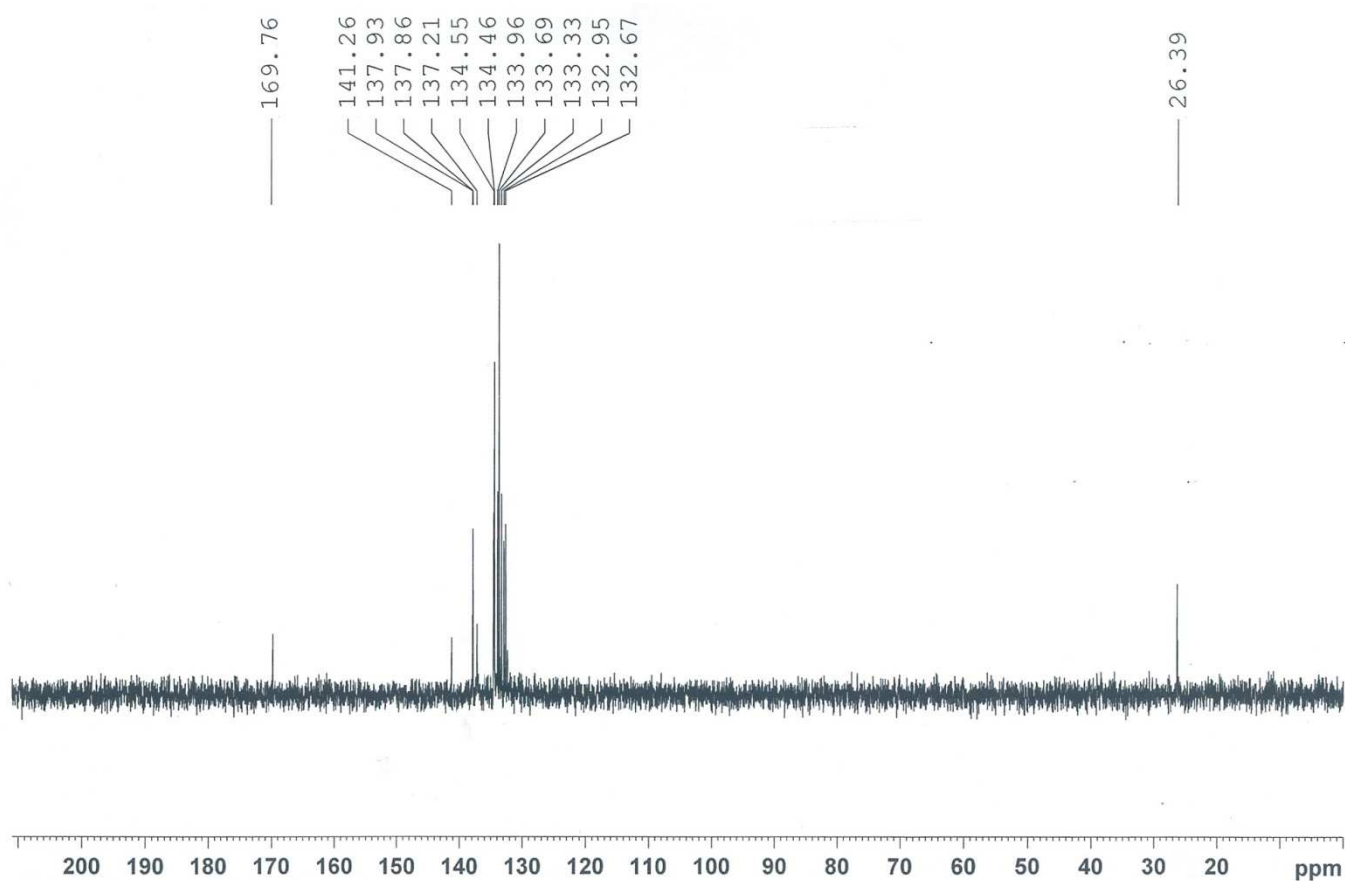
SI Figure 10: ¹H and ¹³C-NMR spectra of compound 7b



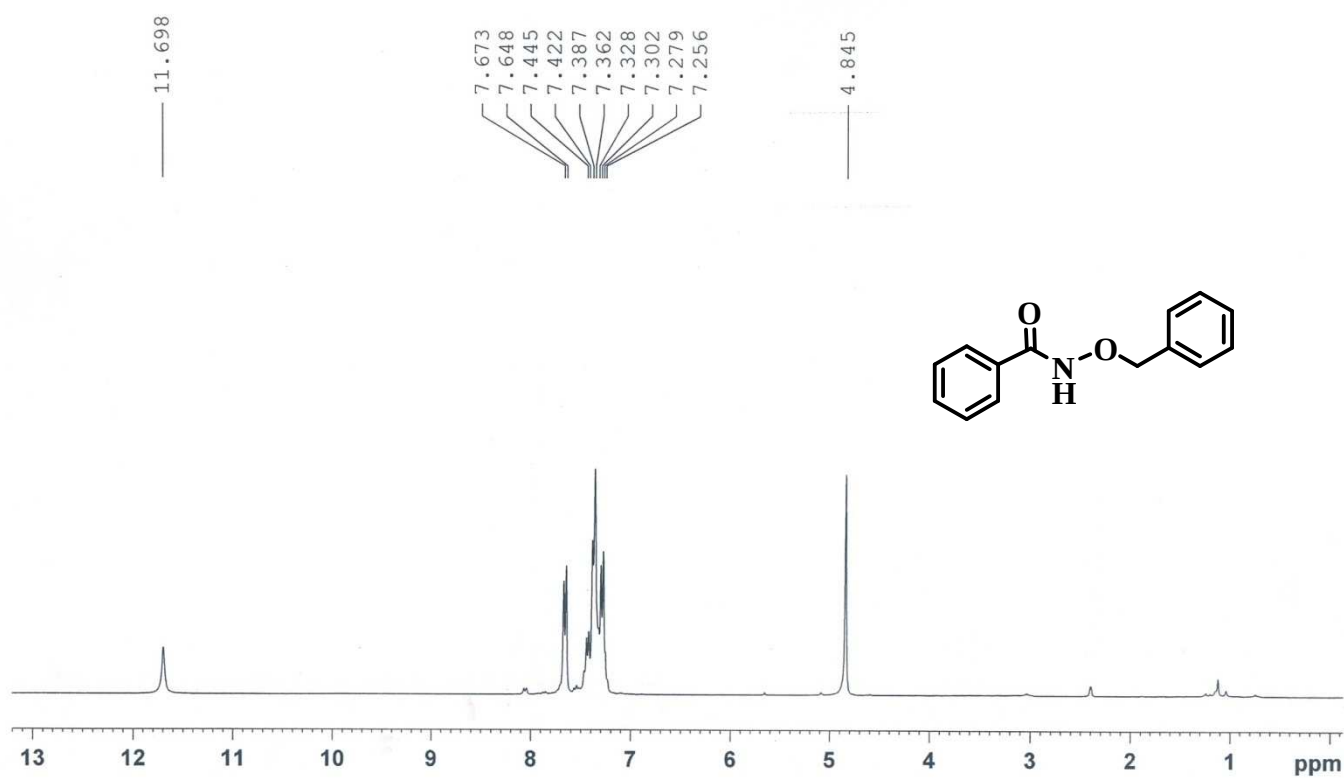


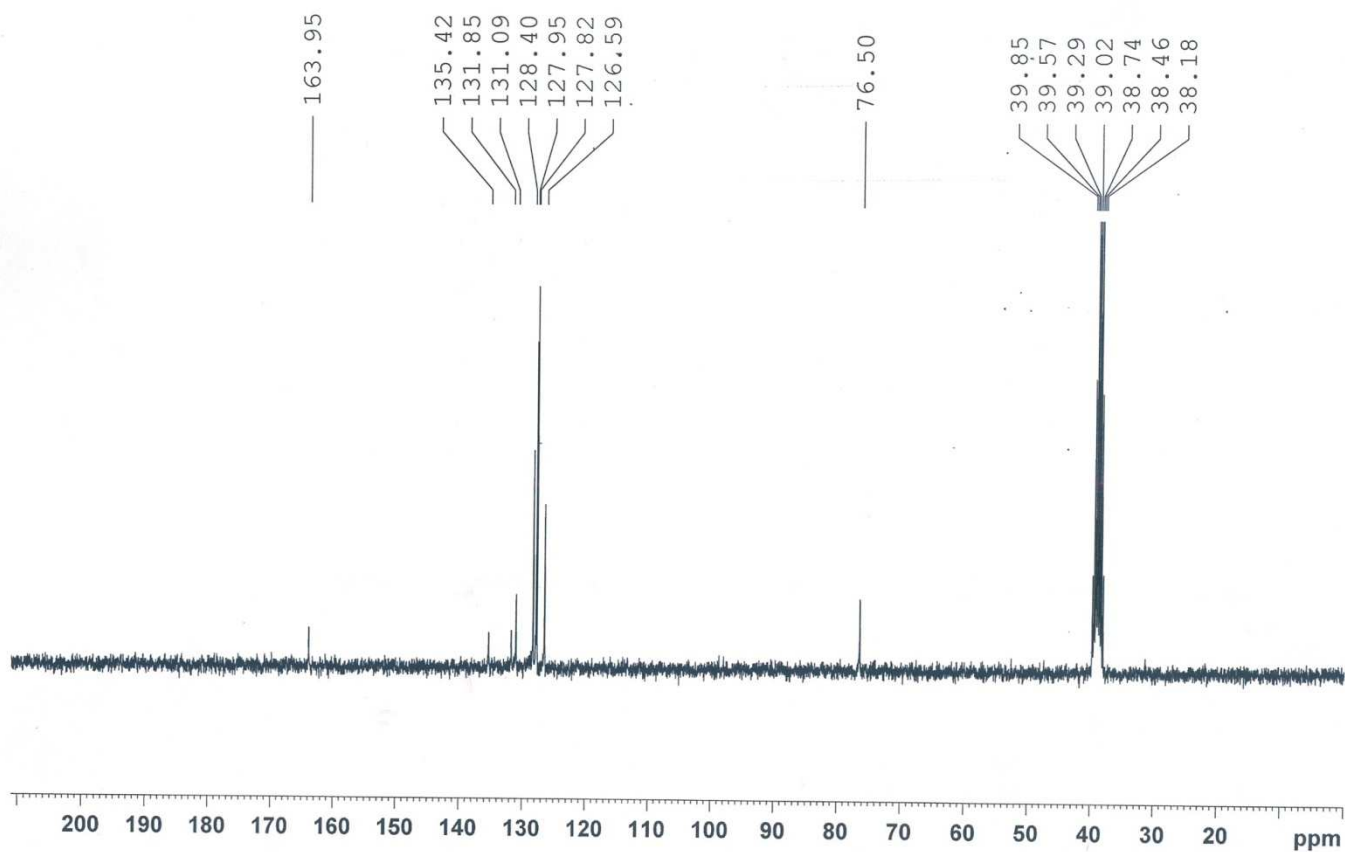
SI Figure 11: ¹H and ¹³C-NMR spectra of compound 7c



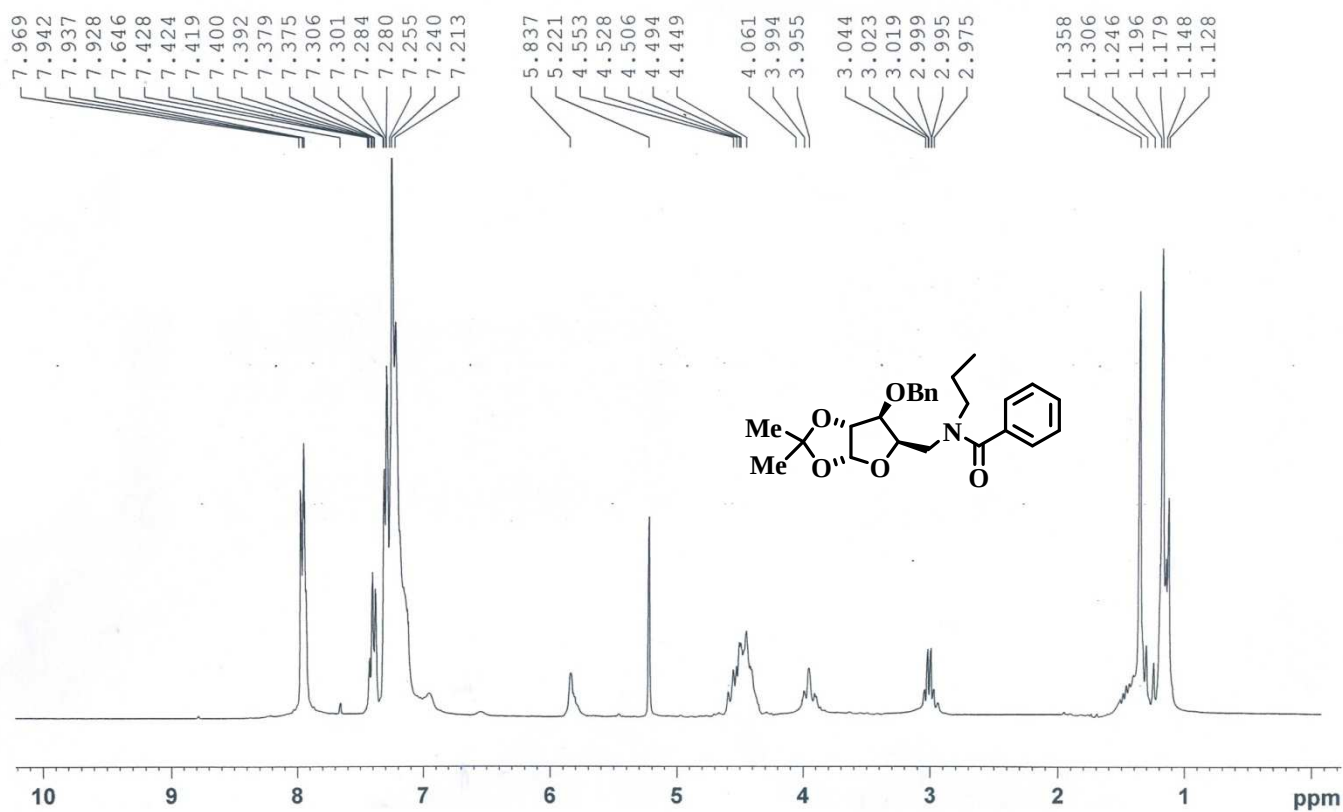


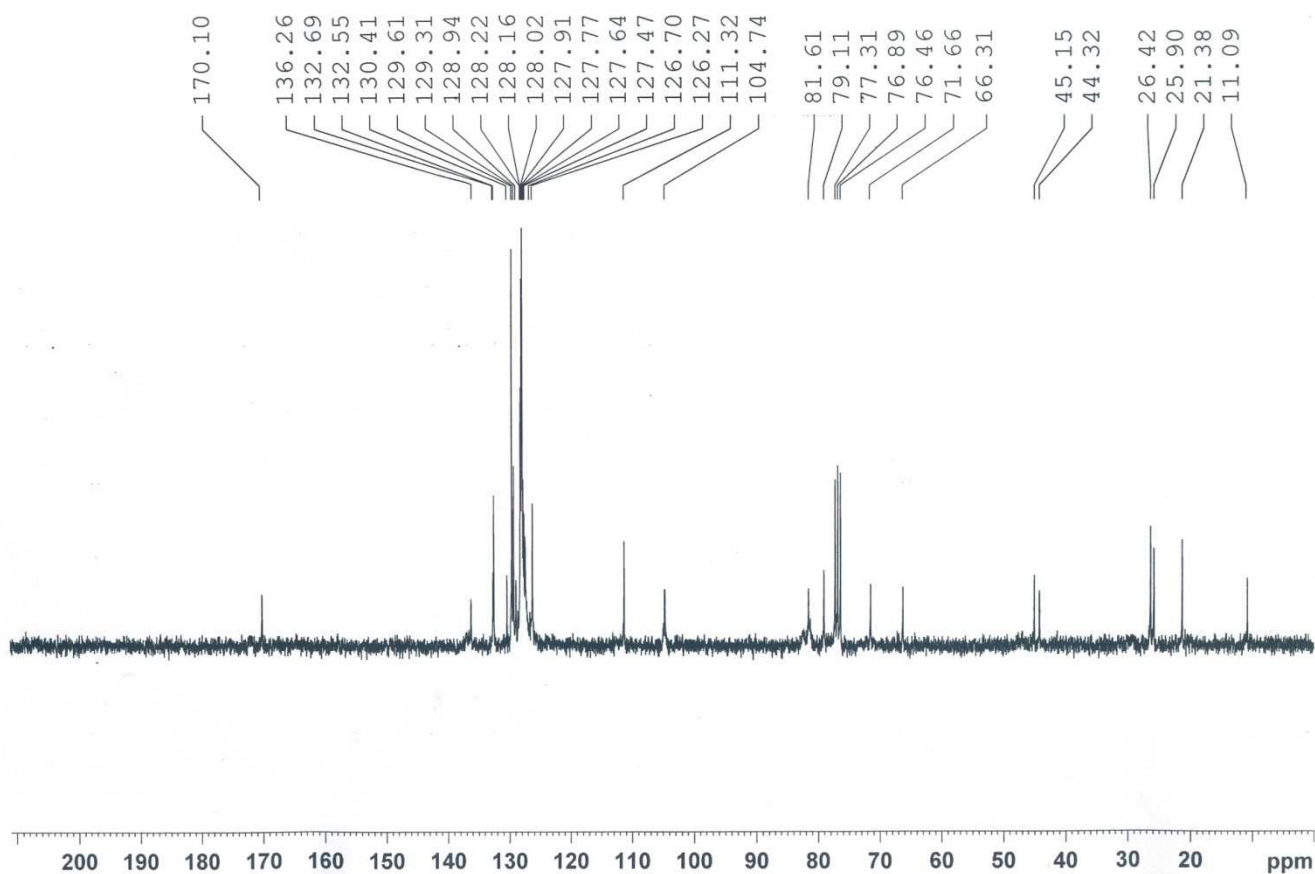
SI Figure 12: ¹H and ¹³C-NMR spectra of compound 7d



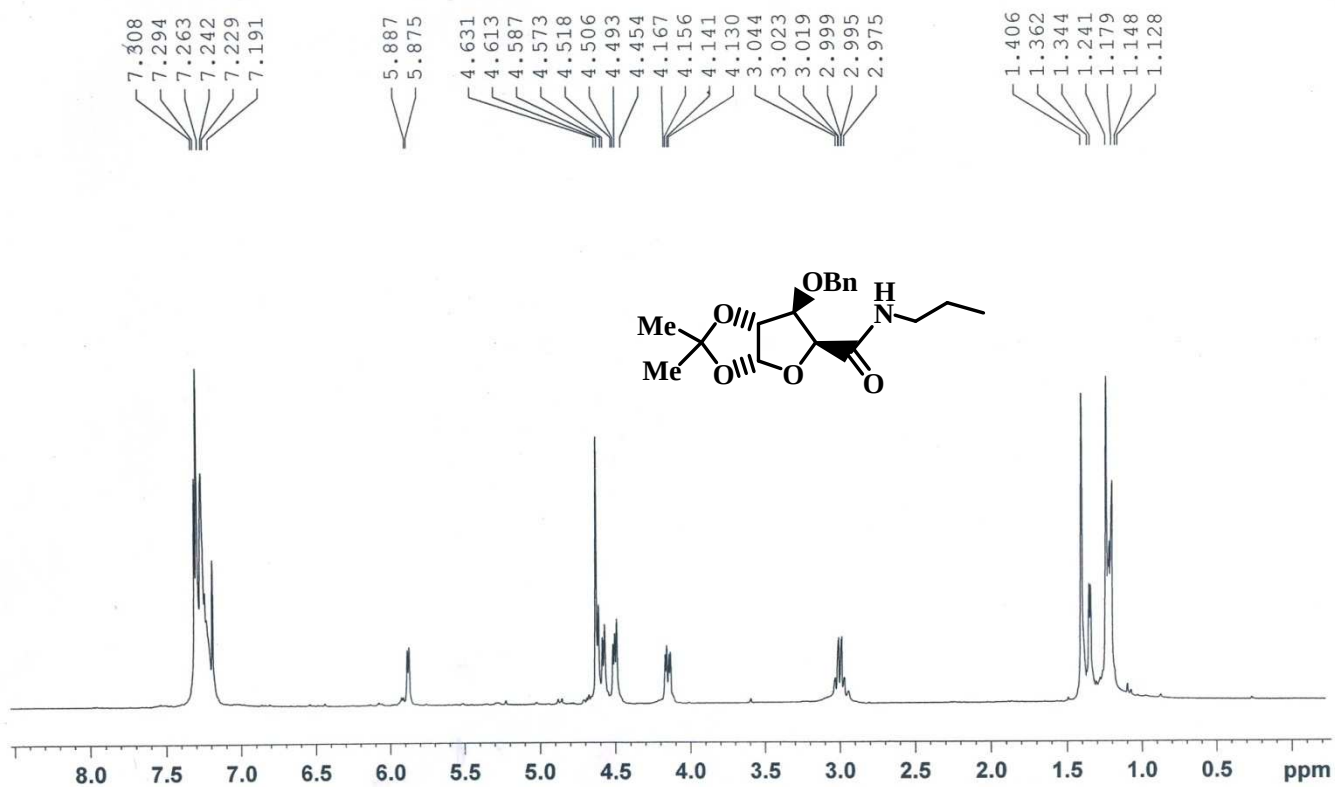


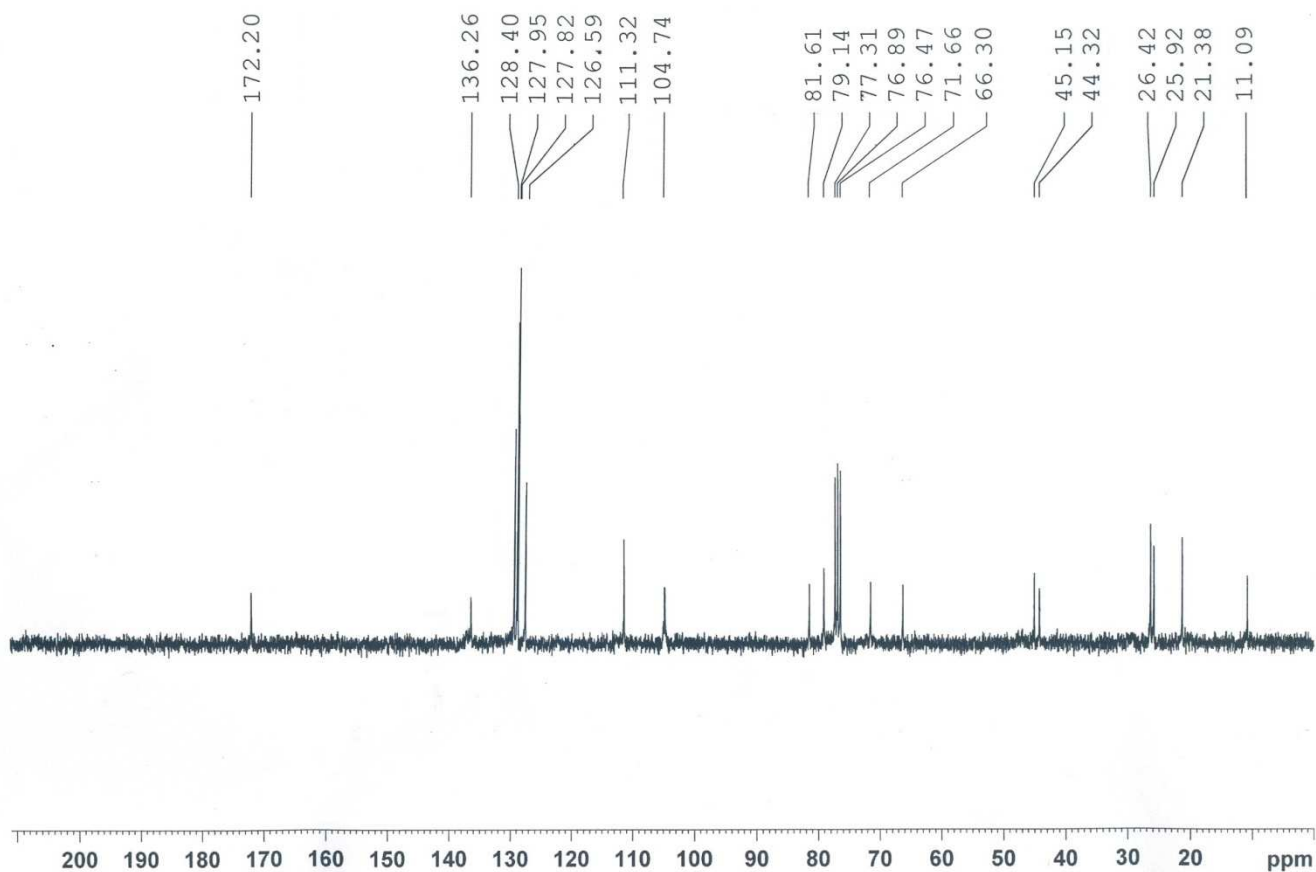
SI Figure 13: ¹H and ¹³C-NMR spectra of compound 7e



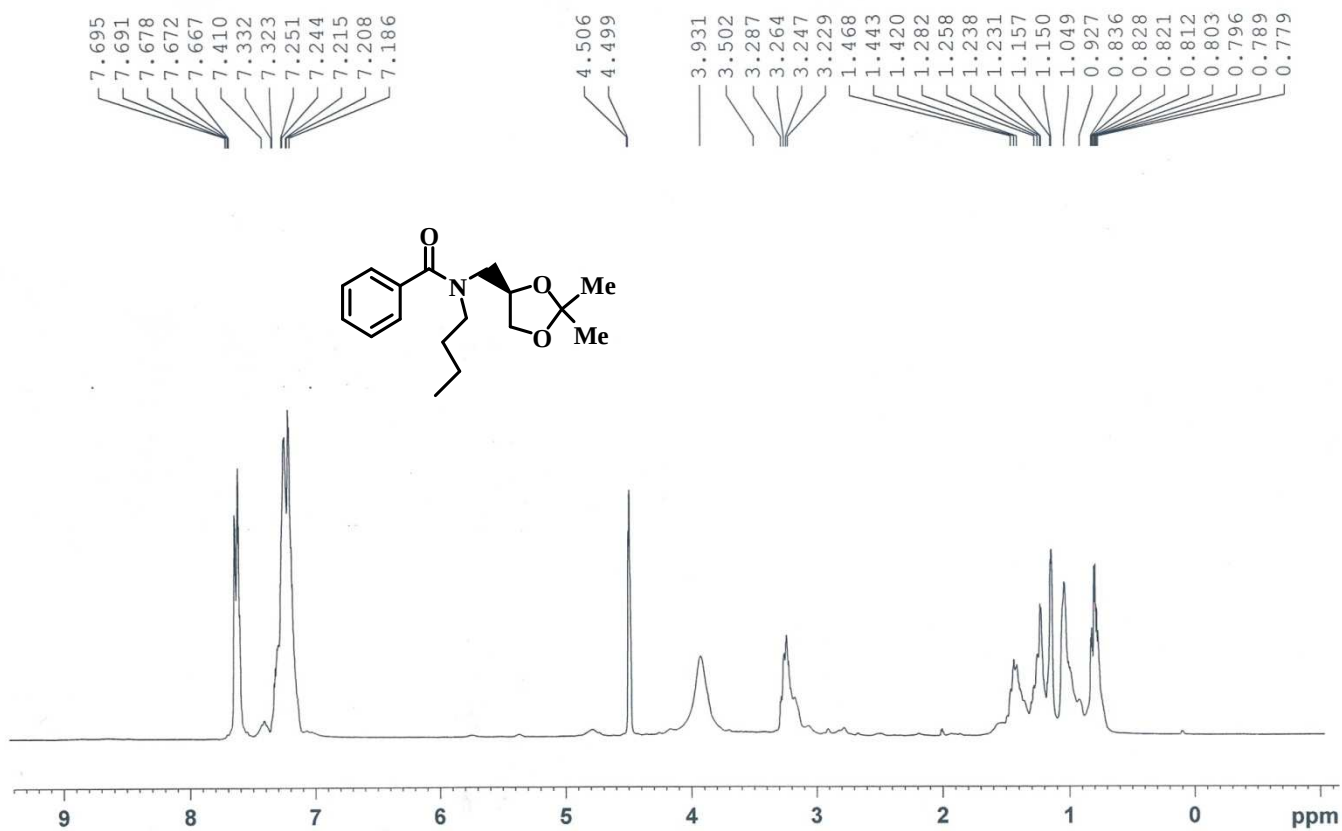


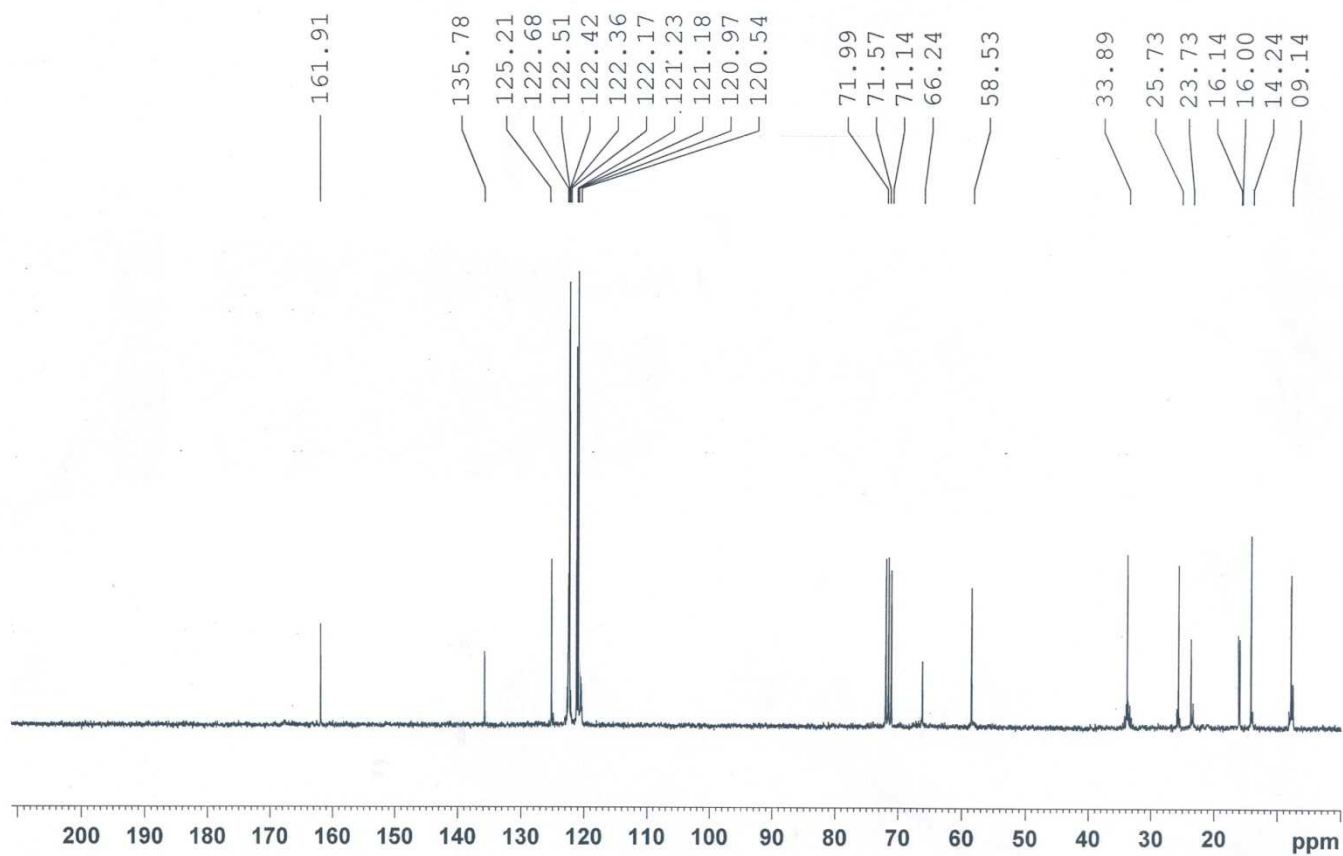
SI Figure 14: ¹H and ¹³C-NMR spectra of compound 7f



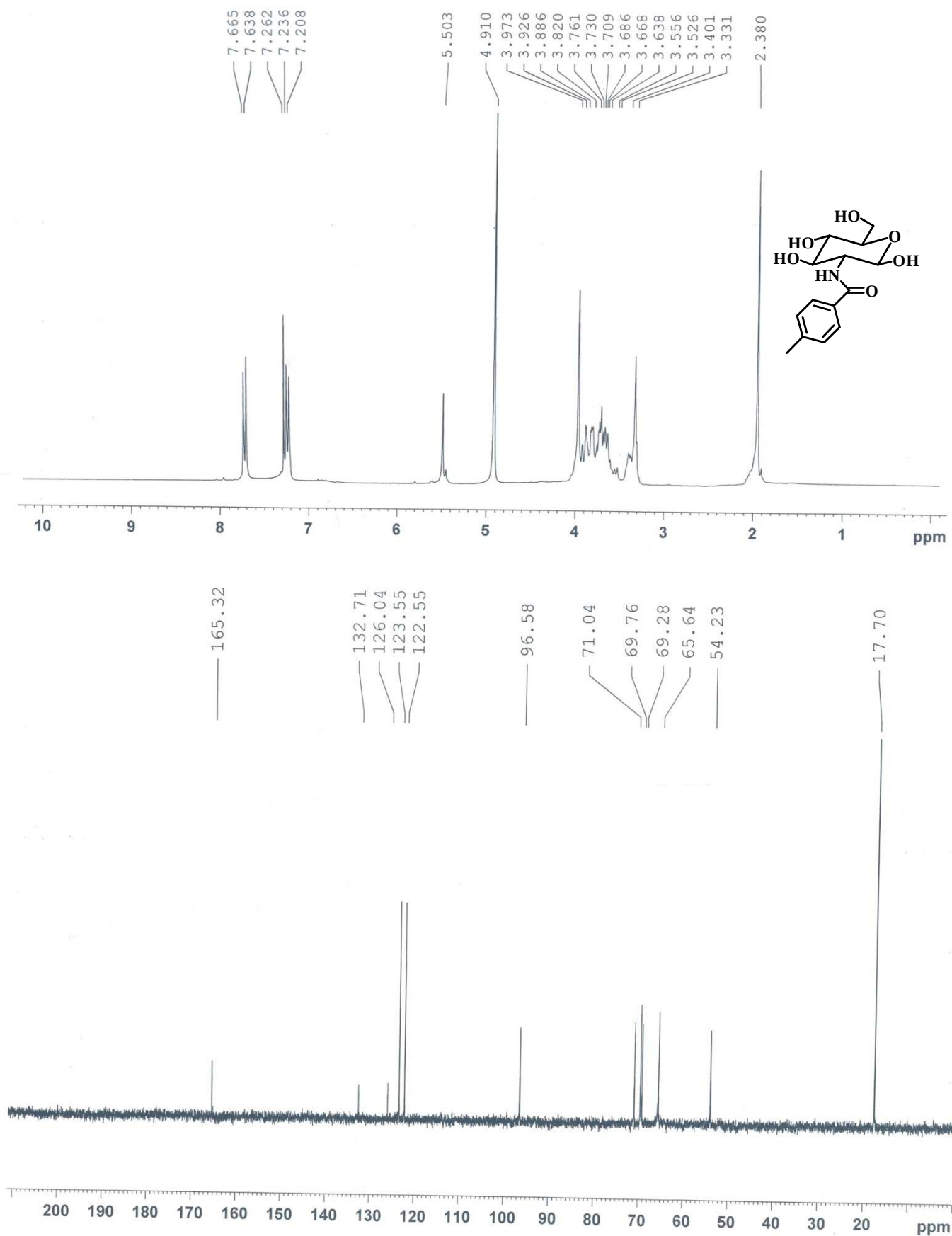


SI Figure 15: ¹H and ¹³C-NMR spectra of compound 7g

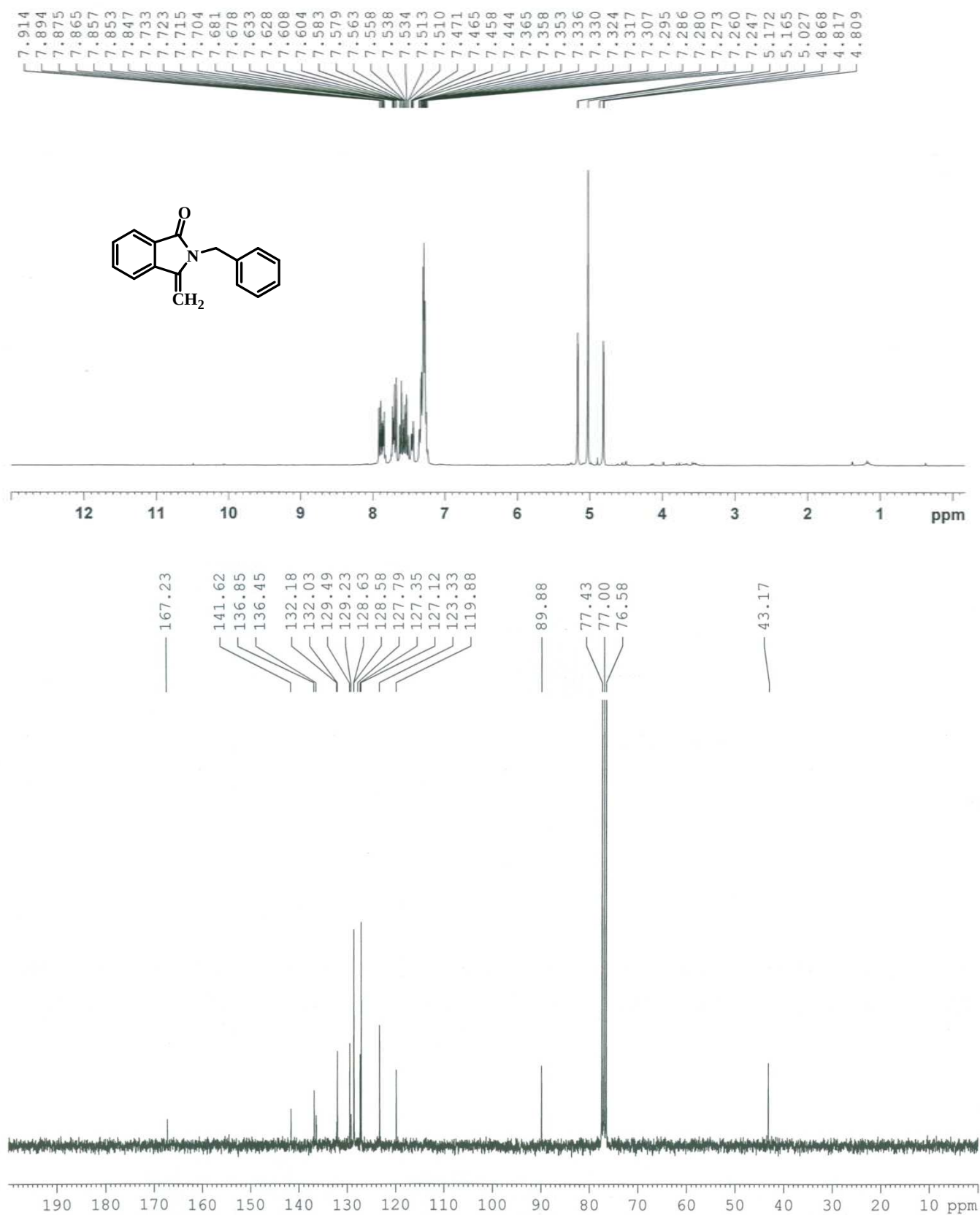




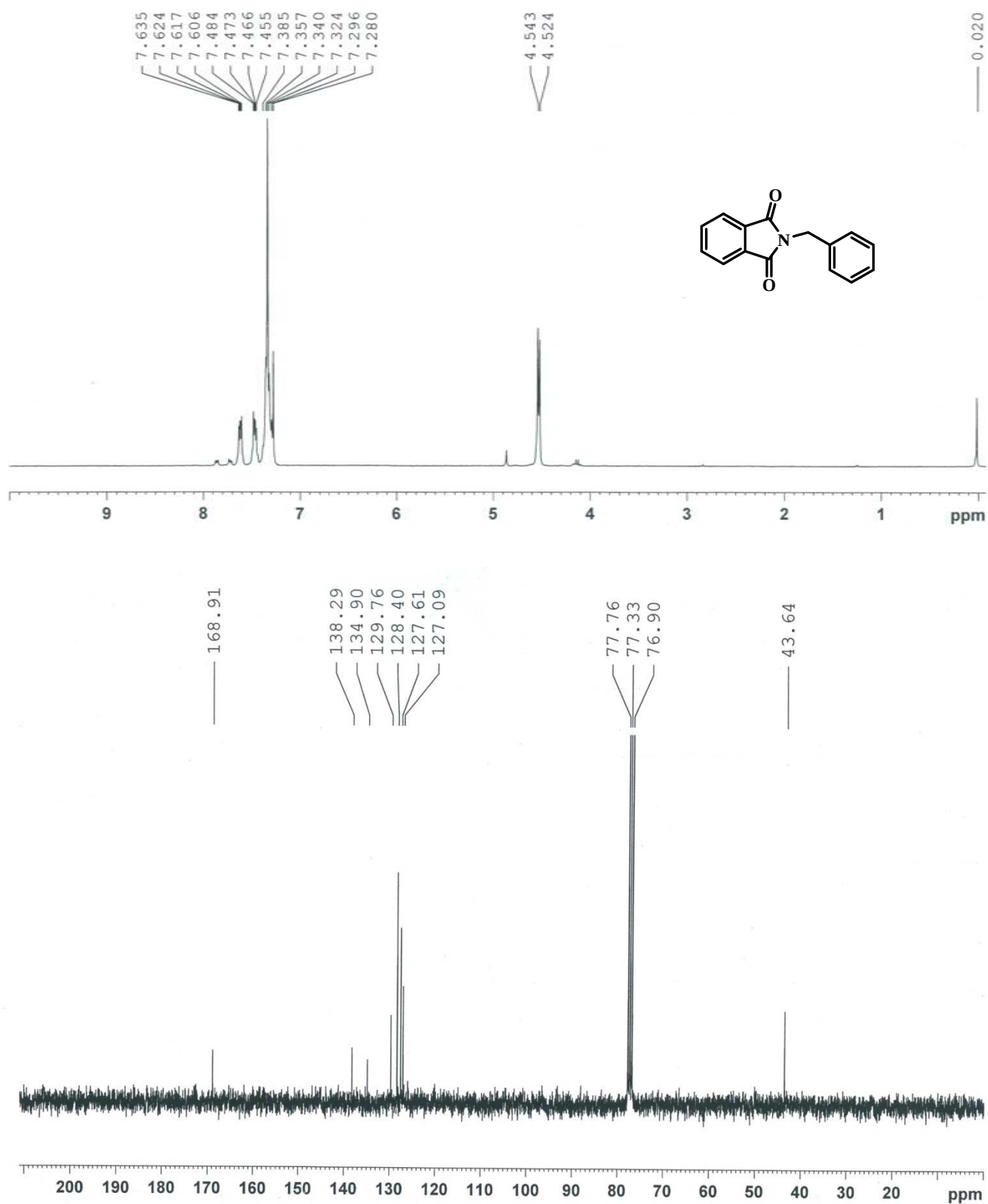
SI Figure 16: ¹H and ¹³C-NMR spectra of compound **7h**



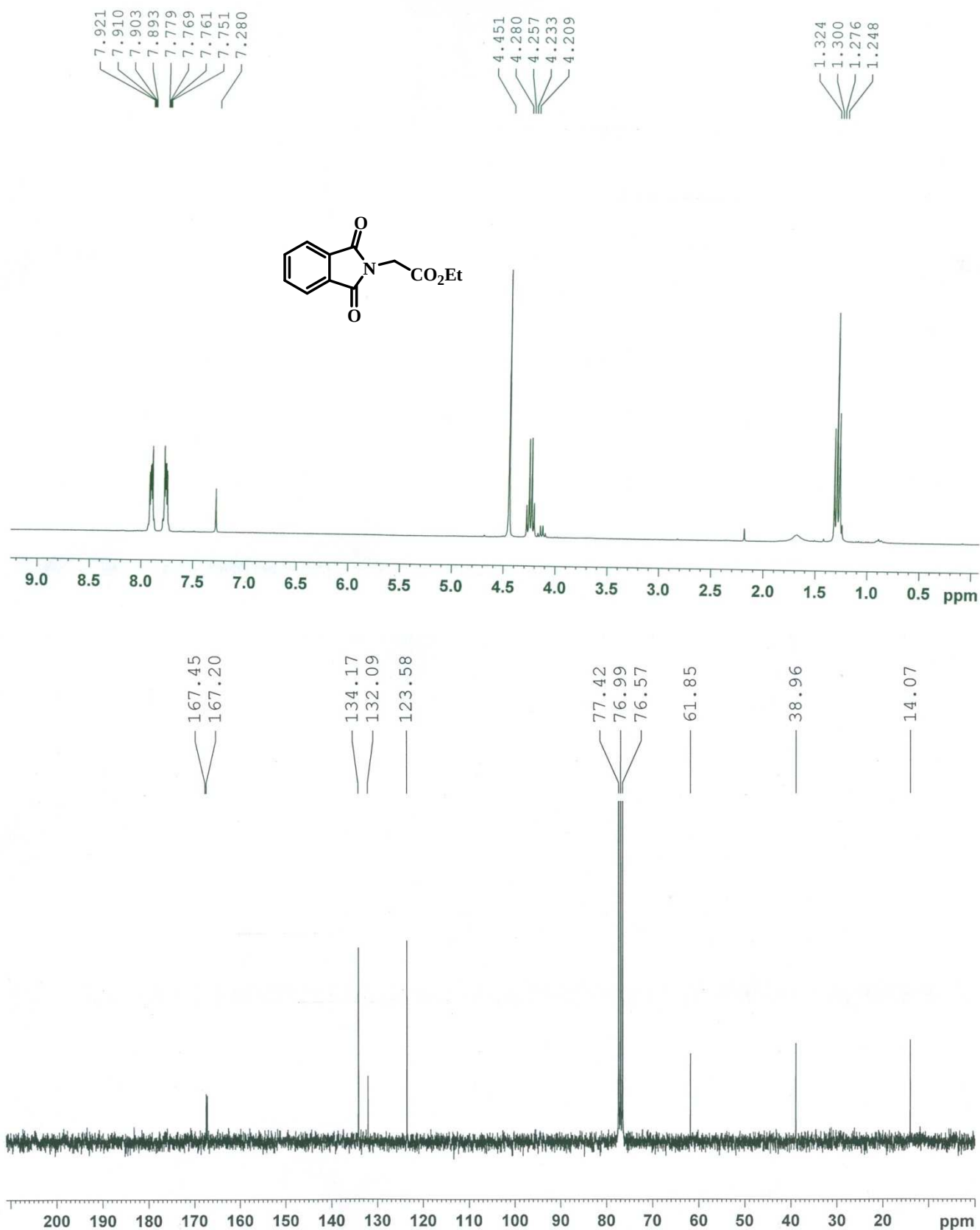
SI Figure 18: ¹H and ¹³C-NMR spectra of compound **VIII**



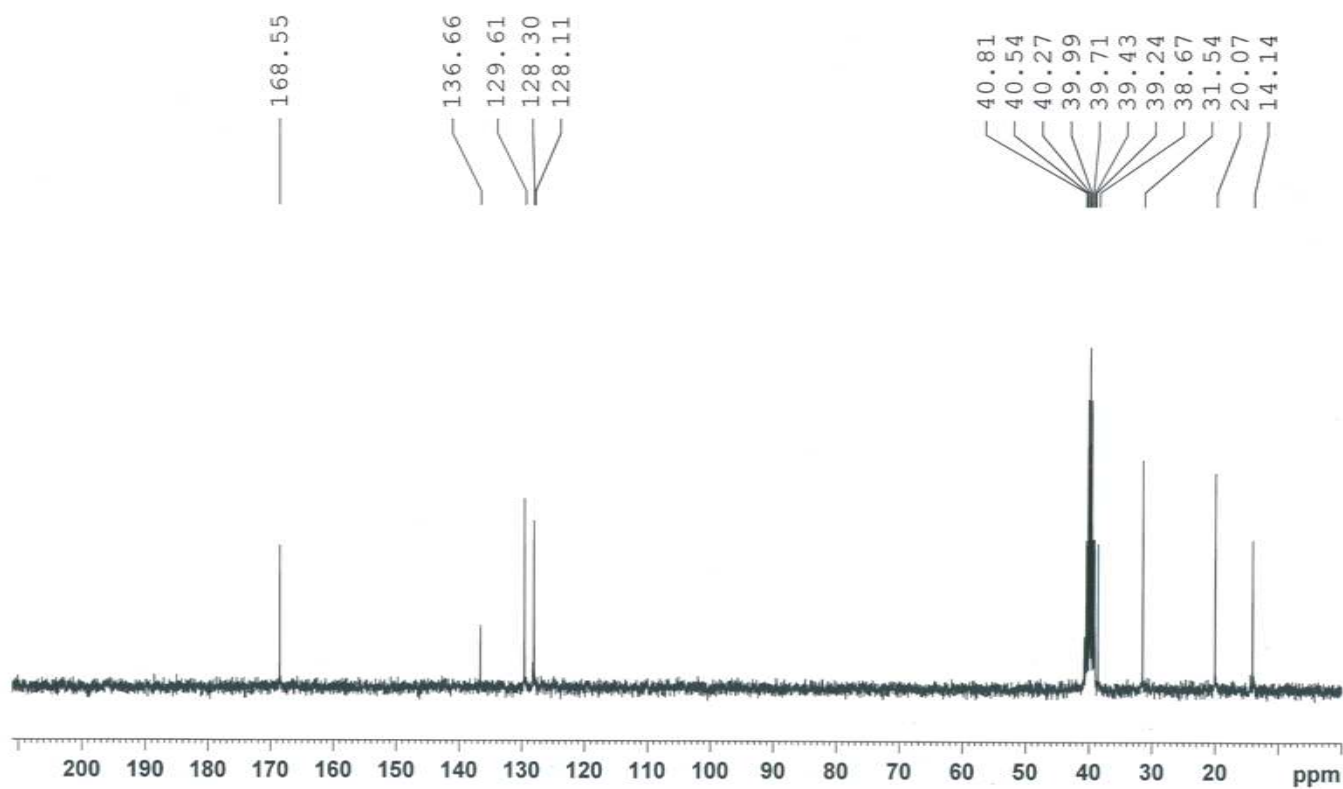
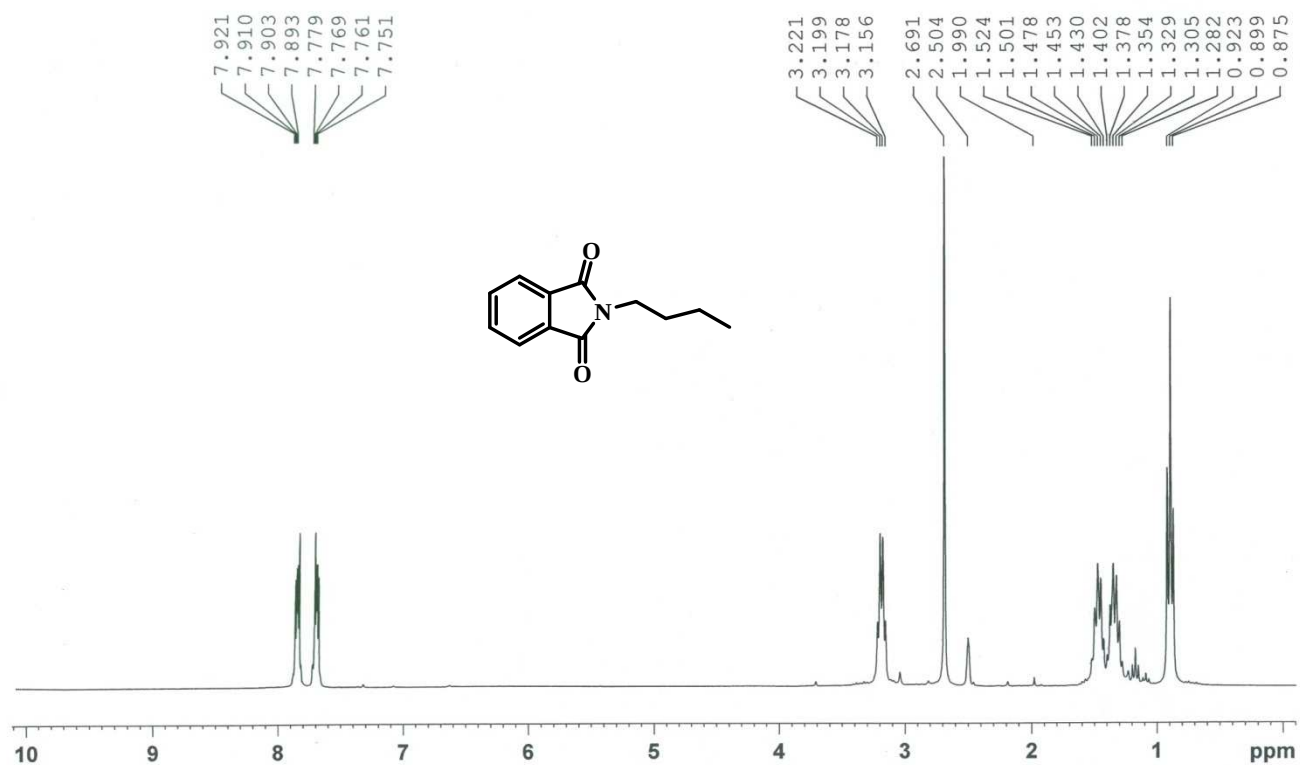
SI Figure 19: ¹H and ¹³C-NMR spectra of compound **8a**



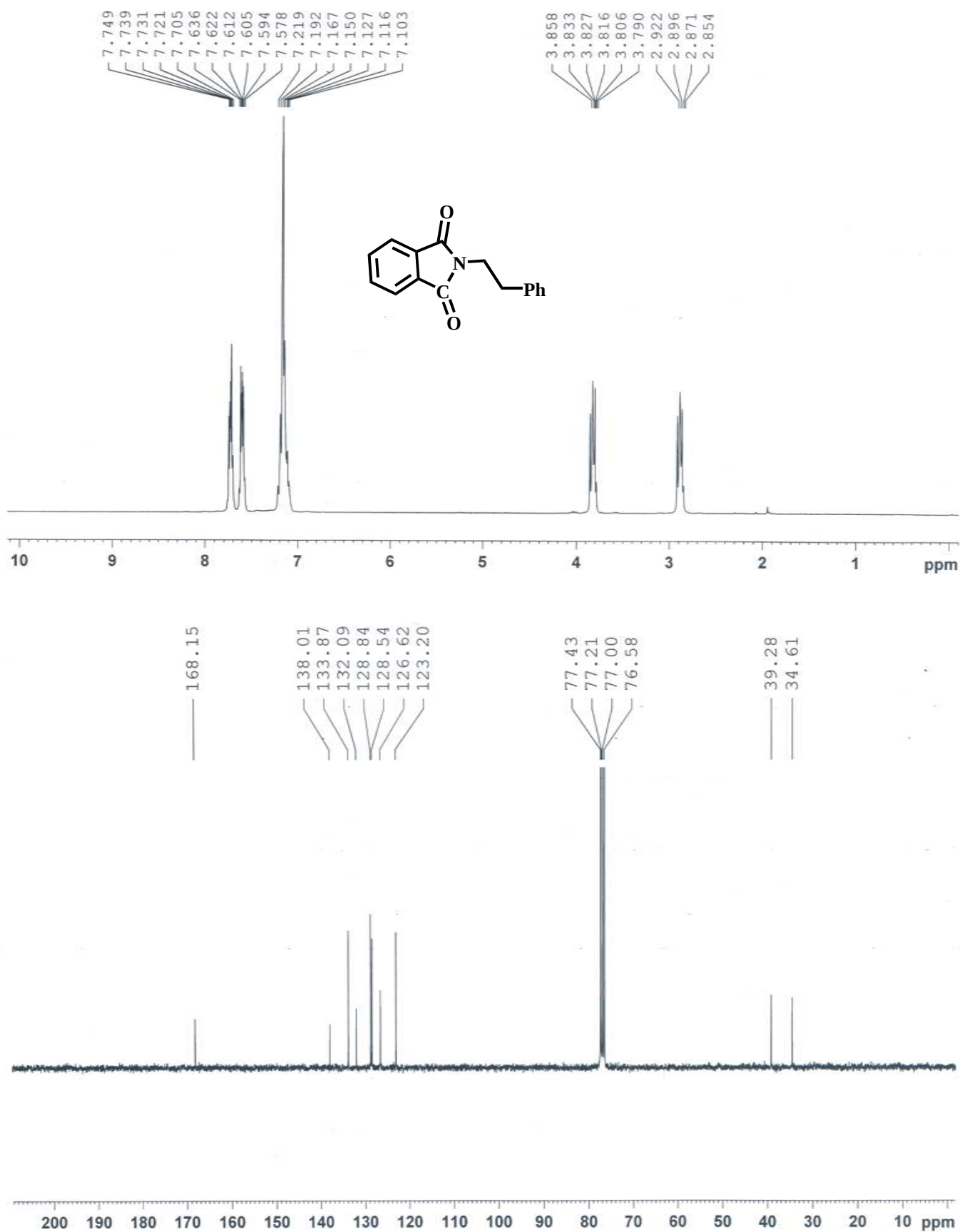
SI Figure 20: ^1H and ^{13}C -NMR spectra of compound **8b**



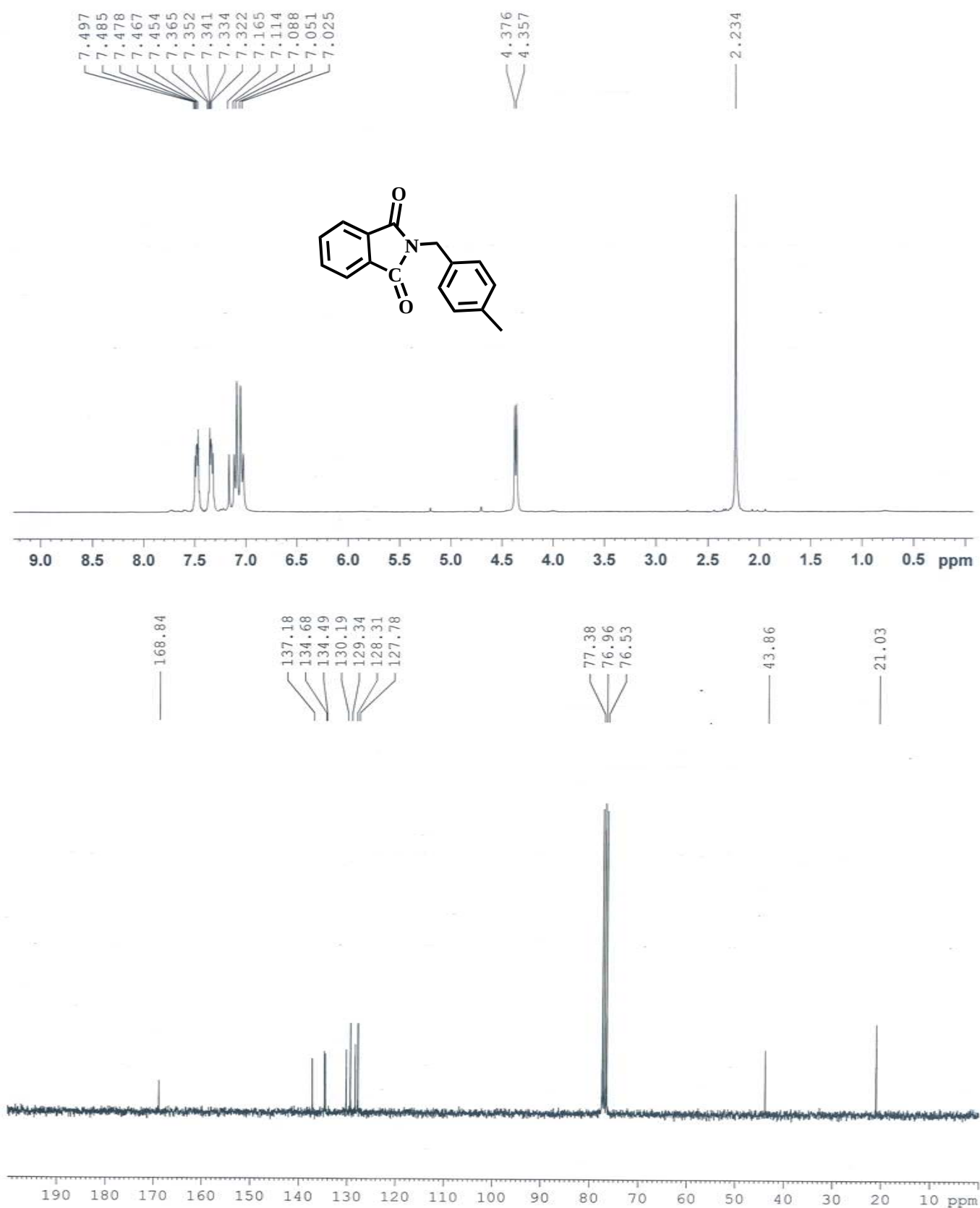
SI Figure 21: ¹H and ¹³C-NMR spectra of compound **8c**



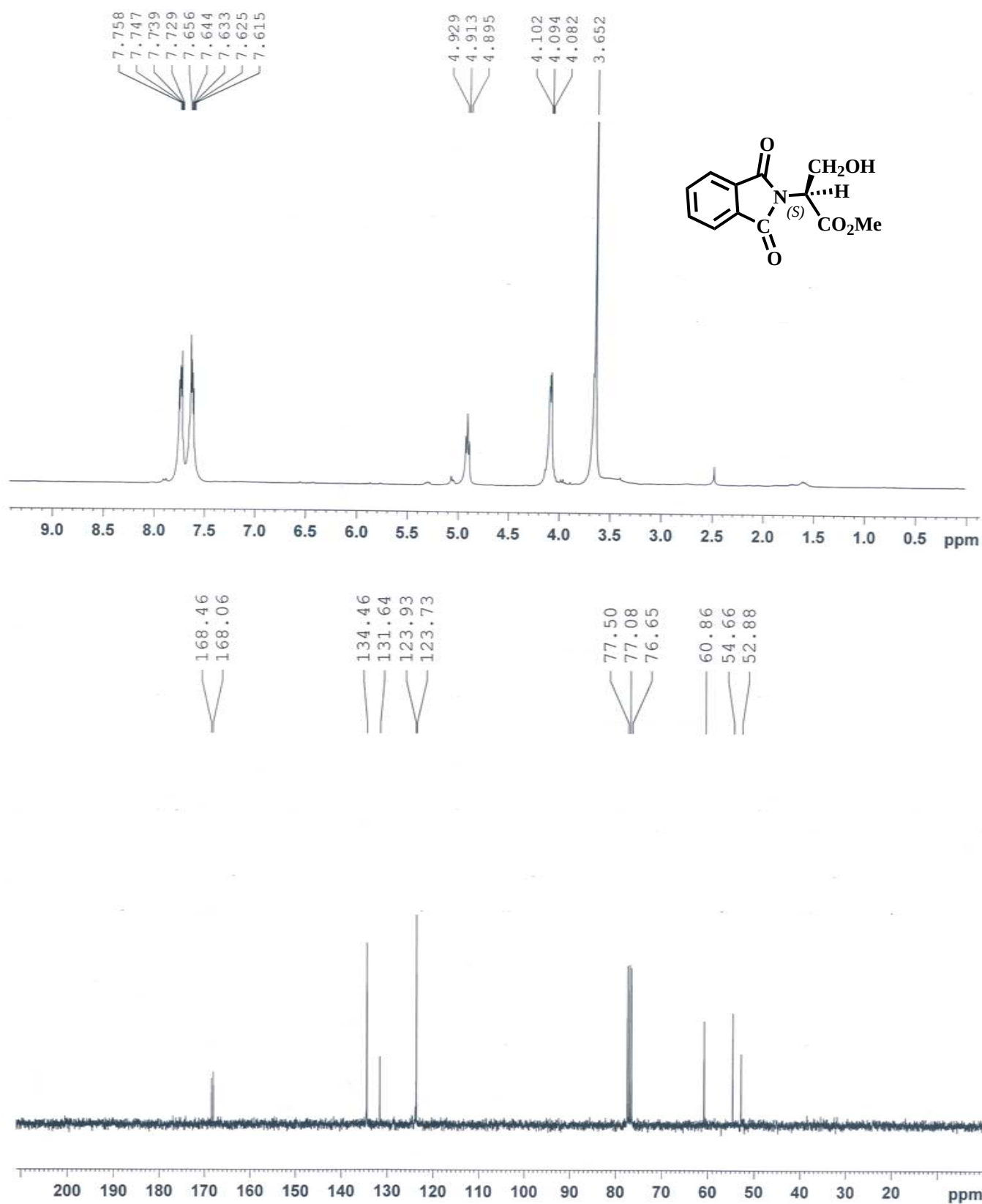
SI Figure 22: ^1H and ^{13}C -NMR spectra of compound **8d**



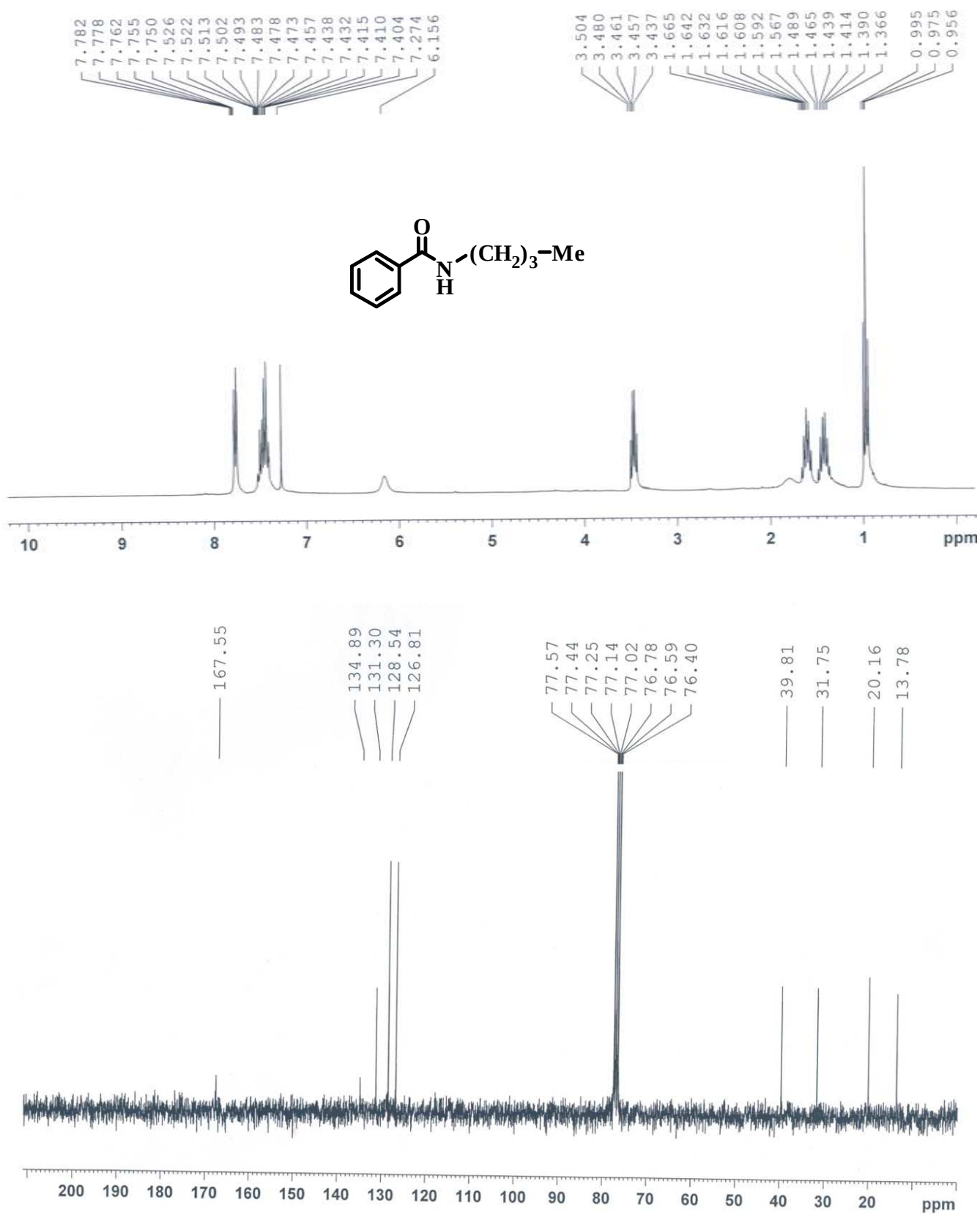
SI Figure 23: ^1H and ^{13}C -NMR spectra of compound **8e**



SI Figure 24: ^1H and ^{13}C -NMR spectra of compound **8f**



SI Figure 25: ^1H and ^{13}C -NMR spectra of compound **9a**



SI Figure 26: ¹H and ¹³C-NMR spectra of compound 9c

