Electronic Supplementary Information

Water - the best solvent for DMAP mediated dual cyclization towards metal-free first synthesis of fully substituted phthalimide

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1. Materials and methods

All reagents were purchased from commercial suppliers and used without further purification. Commercially supplied ethyl acetate and petroleum ether were distilled before use. Petroleum ether used in our experiments was in the boiling range of 60-80 °C. Column chromatography was performed on silica gel (100-200 mesh). All the compounds are new and reported melting points are uncorrected. ^{1}H NMR and ^{13}C NMR spectra (Bruker Advance 300) were recorded at ambient temperature using 300 MHz spectrometers (300 MHz for ^{1}H and 75 MHz for ^{13}C). Chemical shift is reported in ppm from internal reference tetramethylsilane and coupling constant in Hz. Proton multiplicities are represented as s (singlet), d (doublet), t (triplet), q (quartet), and m (multiplet). Infrared spectra were recorded on FT-IR spectrometer (Perkin Elmer Spectrum 100) in thin film. HR-MS data were acquired by electron spray ionization technique on a Q-tof-micro quadriple mass spectrophotometer (Waters XEVO G2-S QTof). UV data were recorded in Perkin Elmer Lambda 750. X-RAY crystallographic data was taken in Bruker-Apex-II-CCD Difractometer. DLS (Dynamic Light Scattering) study was done in Zeta sizer Nano ZS90.

2. Synthesis of *N*-aryl phthalimides

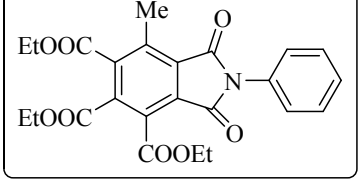
2.a. General procedure of *N*-aryl phthalimides (3a-r)

In a 25 mL round bottom flask 1.0 mmol of acetyl acetanilide (**1**) and 2.2 mmol of dialkyl acetylene dicarboxylate (**2**) were taken in presence of 50 mg (0.15 mmol) of CTAB in 4 mL of H_2O. Then 1.0 mmol of 4-dimethylaminopyridine (DMAP) was added to it and the reaction mixture was stirred for the stipulated period of time at room temperature. The progress of the reaction was monitored by TLC. To the post-reaction mixture 15 mL of water and 20 mL of DCM were added and taken in a separating funnel. The organic layer was separated and dried over anhydrous Na_2SO_4. It was evaporated under reduced pressure and the crude product was purified by column chromatography with silica gel using n-hexane-ethyl acetate as eluent to yield the pure *N*-aryl phthalimides (**3**). The washing water was collected together and removed in a lyophilizer at ambient temperature. The solid residue was dissolved in minimum volume of ethanol. Pure DMAP was crystallized out. M.p. 109-111 ºC.

2.b. Characterization data of compounds (3a-r)

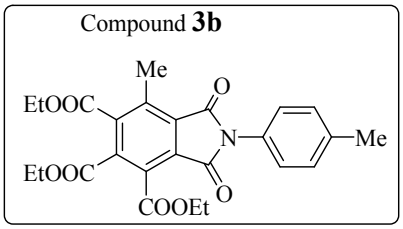
7-Methyl-1,3-dioxo-2-phenyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (3a)

Compound **3a.**



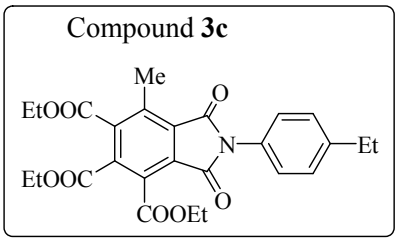
Colourless solid; m.p. 168-170 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 (d, J = 7.2 Hz, 2H), 7.41-7.38 (m, 3H), 4.48-4.43 (m, 4H), 4.32 (q, J = 6.9 Hz, 2H), 2.79 (s, 3H), 1.42-1.34 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.2, 165.8, 164.7, 164.0, 163.9, 142.0, 137.6, 132.4, 131.0, 130.9, 129.8, 129.1, 129.0, 128.7 128.5, 126.5, 126.4, 62.8, 62.6, 62.3, 14.5, 13.9, 13.8, 13.8; FT-IR (KBr,cm^{-1}); 3481, 2984, 2924, 1780, 1719, 1613, 1596,1496, 1461, 1445; HR-MS (*m/z)* for $C_{24}H_{23}NO_8$: Calculated 453.1424, found 453.1426.

7-Methyl-1,3-dioxo-2-phenyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (3b)



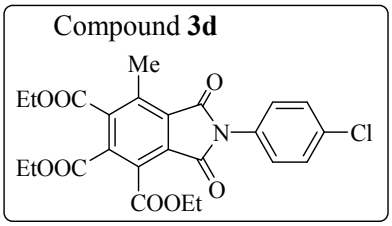
Pale yellow solid; m.p. 196-198 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25-7.18 (m, 4H), 4.43-4.36 (m, 4H), 4.33 (q, J = 7.2 Hz, 2H), 2.70 (s, 3H), 2.34 (s, 3H), 1.37-1.27 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.3, 166.0, 164.8, 164.1, 163.9, 142.0, 138.6, 137.6, 132.3, 131.0, 129.9, 129.8, 129.7, 129.1, 128.3, 126.4, 126.3, 62.8, 62.6, 62.3, 21.2, 14.5, 14.0, 13.8, 13.8; FT-IR (KBr, cm^{-1}); 3446, 2982, 2927, 1732, 1604, 1556, 1516, 1466, 1446; HR-MS (*m/z)* for $C_{25}H_{25}NO_8$: Calculated 467.1580, found 467.1581.

2(4-Ethylphenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (3c)



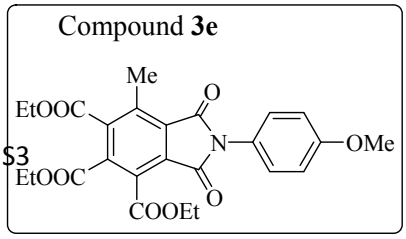
Pale yellow solid; m.p. 124-126 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24-7.16 (m, 4H), 4.40-4.23 (m, 6H), 2.67 (s, 3H), 2.63 (q, J = 7.5 Hz, 2H), 1.31-1.24 (m, 9H), 1.18 (t, J = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.2, 165.9, 164.7, 164.1, 163.8, 144.8, 141.9, 137.5, 130.9, 129.6, 129.0, 128.6, 128.3, 126.4, 62.8, 62.5, 62.3, 28.5, 15.3, 14.4, 13.9, 13.8, 13.7; FT-IR (KBr, cm^{-1}); 3435, 2973, 2936, 2873, 1777, 1735, 1720, 1636, 1616, 1515, 1468, 1445; HR-MS (*m/z)* for $C_{26}H_{27}NO_8$: Calculated 481.1737, found 481.1734.

2-(4-Chlorophenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (3d)



Pale yellow solid; m.p. 178-180 ˚C; ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.50-7.48 (m, 2H), 7.37-7.34 (m, 2H), 4.29-4.18 (m, 6H), 2.56 (s, 3H), 1.22-1.06 (m, 9H); ^{13}C NMR (75 MHz, DMSO-d_6 + $CDCl_3$) δ 164.9, 163.5, 163.1, 139.4, 136.0, 132.4, 131.4, 131.2, 129.7, 128.7, 128.5, 128.4, 127.4, 62.2, 61.7, 61.5, 13.6, 13.1,13.1, 12.9; FT-IR (KBr, cm^{-1}); 3442, 2984, 2935, 2902, 1781, 1737, 1722, 1614, 1589, 1492, 1476; HR-MS (*m/z)* for $C_{24}H_{22}ClNO_8$: Calculated 487.1034, found 487.1039 (One of the major peaks).

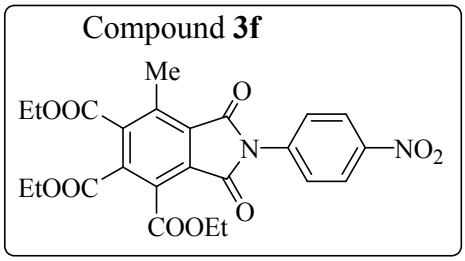
2-(4-Methoxyphenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (3e)



Yellow solid; m.p. 146-148 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.18 (d, J = 8.7 Hz, 2H), 6.88 (d, J = 9.0 Hz, 2H), 4.37-4.20 (m, 6H), 3.70 (s, 3H), 2.63 (s, 3H), 1.31-1.21 (m, 9H);

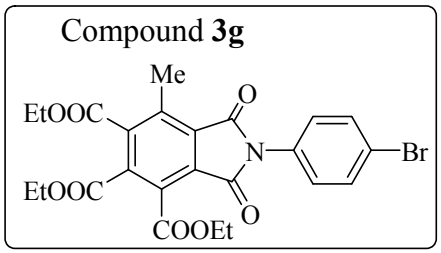
^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 165.9, 164.6, 164.1, 163.8, 159.4, 141.7, 137.3, 132.1, 130.8, 129.4, 129.0, 127.7, 123.4, 114.3, 62.6, 62.5, 62.4, 55.3, 14.3, 13.8, 13.7, 13.6; FT-IR (KBr, cm^{-1}) 3443, 2981, 2940, 2843, 1778, 1738, 1721, 1615, 1558, 1515, 1467; HR-MS (*m/z)* for $C_{25}H_{25}NO_9$: Calculated 483.1529, found 483.1525.

7-Methyl-2-(4-nitrophenyl)-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (3f)



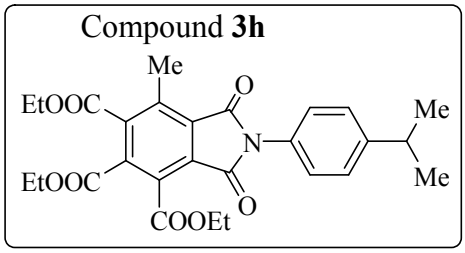
Pale yellow solid; m.p. 193-194˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.41 (d, J = 9 Hz, 2H), 7.78 (d, J = 9 Hz, 2H), 4.53-4.38 (m, 6H), 2.82 (s, 3H), 1.48-1.34 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.9, 164.9, 164.2, 163.6, 163.0, 151.6, 146.6, 142.5, 142.3, 138.1, 136.6, 130.3, 130.0, 128.5, 126.5, 124.5, 124.3, 62.9, 62.6, 62.4, 14.5, 13.9, 13.8, 13.7; FT-IR (KBr, cm^{-1}) 3415, 2985, 2937, 1780, 1735, 1637, 1611, 1595, 1528, 1509; HR-MS (*m/z)* for $C_{24}H_{22}N_2O_{10}$: Calculated 498.1274, found 498.1272.

2-(4-Bromophenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (3g)



Colourless solid; m.p. 168-170 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.64 (d, 2H, J = 8.4 Hz), 7.33 (d, 2H, J = 8.7 Hz), 4.51-4.34 (m, 6H), 2.77 (s, 3H), 1.42-1.34 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 165.5, 164.6, 163.8, 163.7, 142.2, 137.8, 132.5, 132.8, 130.7, 130.0, 129.9, 128.8, 127.9, 122.3, 62.9, 62.6, 62.4, 14.5, 13.9, 13.8, 13.7; FT-IR (KBr, cm^{-1}) 3440, 2986, 2939, 2904, 1780, 1738, 1721, 1613, 1588, 1492; HR-MS (*m/z)* for $C_{24}H_{22}BrNO_8$: Calculated 531.0529, found 531.0530 (One of the major peaks).

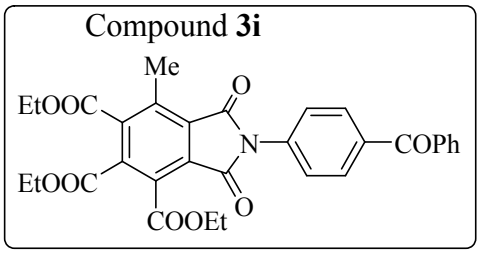
2-(4-Isopropylphenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (3h)



Pale yellow solid; m.p. 142-143 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26-7.16 (m, 4H), 4.40-4.23 (m, 6H), 2.92-2.83 (m, 1H), 2.67 (s, 3H), 1.31-1.23 (m, 9H), 1.17 (d, J = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 165.8, 164.6, 164.0, 163.5, 149.2, 141.8, 137.4, 132.0, 130.8, 129.5, 128.9, 128.3, 127.1, 126.2, 62.6, 62.4, 62.1, 33.7, 23.6, 14.3, 13.8, 13.7, 13.6; FT-IR

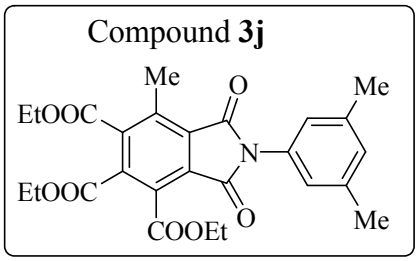
(KBr, cm^{-1}) 3451, 2986, 2963, 1778, 1741, 1723, 1637, 1615, 1519, 1504, 1458; HR-MS (*m/z)* for $C_{27}H_{29}NO_8$ 495.1893, found 495.1897.

2-(4-Benzoylphenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6- tricarboxylic acid triethyl ester (3i)



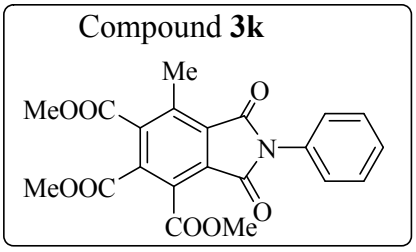
Pale yellow solid; m.p. 203-204 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 (d, J = 8.4 Hz, 2H), 7.88 (d, J = 7.8 Hz, 2H), 7.66 (d, J = 7.8 Hz, 3H), 7.57-7.52 (m, 2H), 4.54-4.39 (m, 6H), 2.84 (s, 3H), 1.48-1.30 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.9, 166.1, 165.4, 164.3, 163.8, 163.1, 151.7, 142.6, 137.9, 137.1, 134.6, 132.6, 130.8, 130.7, 130.0, 128.4, 126.9, 126.0, 62.8, 62.7, 62.4, 14.6, 14.0, 13.8, 13.8; FT-IR (KBr, cm^{-1}) 3445, 2981, 2935, 1720, 1655, 1603, 1576, 1511, 1467, 1448; HR-MS (*m/z)* for $C_{31}H_{27}NO_9$: Calculated 557.1686, found 557.1690.

2-(3,5-Dimethylphenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (3j)



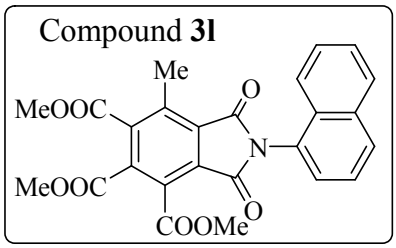
Pale yellow solid; m.p. 170-171 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.04 (s, 1H), 6.97 (s, 2H), 4.41-4.35 (m, 6H), 2.76 (s, 3H), 2.35 (s, 6H), 1.41-1.33 (m, 9H) ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.3, 166.1, 164.8, 164.2, 163.9, 142.0, 139.0, 137.5, 131.0, 130.7, 130.5. 129.7, 124.4, 62.8, 62.6, 62.3, 21.2, 14.5, 14.0, 13.8, 13.8; FT-IR (KBr, cm^{-1}) 3434, 2980, 2923, 1737, 1725, 1721, 1642, 1617, 1476, 1444; HR-MS (*m/z)* for $C_{26}H_{27}NO_8$: Calculated 481.1737, found 481.1734.

7-Methyl-1,3-dioxo-2-phenyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester(3k)



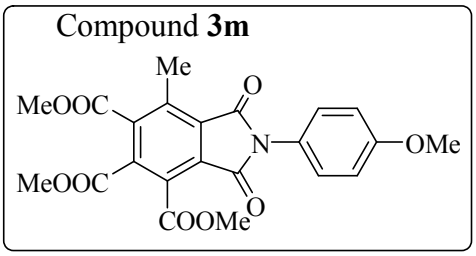
Light brown solid; m.p. 189-190 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, J = 7.5 Hz, 2H), 7.45 (t, J = 9.0 Hz, 3H), 4.01 (s, 3H), 3.98 (s, 3H), 3.92 (s, 3H), 2.78 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 165.7, 165.2, 164.1, 164.0, 141.8, 137.9, 132.0, 131.1, 130.9, 129.5, 129.2, 129.1, 128.8, 128.5, 126.5, 53.4, 53.3, 53.0, 14.5; FT-IR (KBr, cm^{-1}) 3417, 2953, 2924, 2853, 1783, 1741, 1638, 1618, 1597, 1492; HR-MS (*m/z)* for $C_{21}H_{17}NO_8$: Calculated 411.0954, found 411.0956.

7-Methyl-2-napthalen-1-yl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (3l)



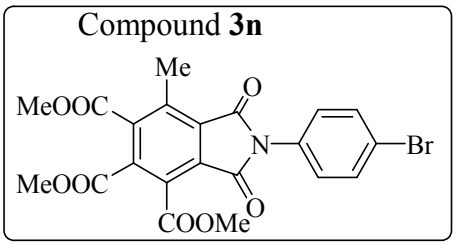
Light brown solid; m.p. 208-210 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00-7.92 (m, 2H), 7.59-7.51 (m, 4H), 7.45-7.42 (m, 1H), 3.99-3.97 (m, 6H), 3.93-3.92 (m, 3H), 2.80 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 166.1, 165.1, 164.3, 164.2, 141.9, 138.1, 134.3, 132.1, 131.3, 130.2, 129.9, 129.6, 129.5, 128.5, 127.3, 126.8, 126.6, 125.2, 122.0, 53.5, 53.3, 53.1, 14.6; FT-IR (KBr, cm^{-1}) 3415, 2924, 2853, 1745, 1723, 1638, 1616, 1597, 1511; HR-MS (*m/z)* for $C_{25}H_{19}NO_8$: Calculated 461.1111, found 461.1110.

2-(4-Methoxyphenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (3m)



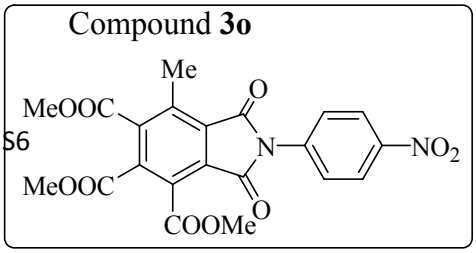
Brown solid; m.p. 169-170 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 (d, J = 4.5 Hz, 2H), 6.97 (d, 2H, J = 9.0 Hz), 3.95 (s, 3H), 3.92 (s, 3H), 3.86 (s, 3H), 3.79 (s, 3H), 2.71 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 166.0, 165.2, 164.2, 164.2, 159.5, 141.8, 137.9, 131.9, 131.2, 129.4, 129.3, 127.8, 123.4, 114.5, 55.5, 53.5, 53.4, 53.3, 14.5; FT-IR (KBr, cm^{-1}) 3467, 3414, 2953, 2924, 2852, 1740, 1720, 1637, 1617, 1514; HR-MS (*m/z)* for $C_{22}H_{19}NO_9$: Calculated 441.1060, found 441.1069.

2-(4-Bromophenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (3n)



Brown solid; m.p. 196-197 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 (d, J = 8.7 Hz, 2H), 7.30 (d, J = 8.7 Hz, 2H), 3.97 (s, 3H), 3.94 (s, 3H), 3.89 (s, 3H), 2.73 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.5, 165.3, 165.0, 164.1, 163.6, 141.9, 138.0, 132.3, 132.1, 130.9, 129.9, 129.5, 129.1, 127.8, 127.6, 122.3, 53.5, 53.3, 53.1, 14.6; FT-IR (KBr, cm^{-1}) 3452, 3416, 2953, 2923, 2852, 1783, 1736, 1723, 1638, 1615; HR-MS (*m/z)* for $C_{21}H_{16}BrNO_8$: Calculated 489.0059, found 489.0061 (One of the major peaks).

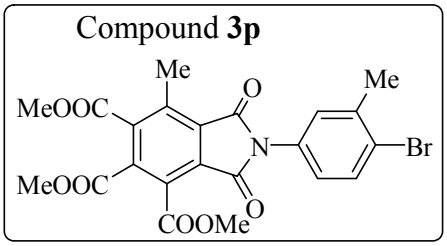
7-Methyl-2-(4-nitrophenyl)-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (3o)



Light brown solid; m.p. 186-188 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.37 (d, J = 9.0 Hz, 2H), 7.72 (d, J = 9.0 Hz, 2H), 3.97 (s, 3H), 3.83 (s, 3H), 3.97 (s, 3H),

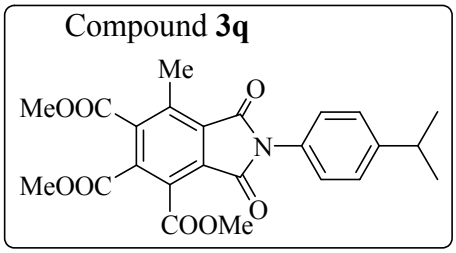
2.77 (s, 3H), ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.4, 164.9, 164.0, 163.2, 146.8, 142.3, 138.4, 136.6, 132.6, 130.6, 129.9, 128.8, 126.5, 124.4, 53.6, 53.4, 53.2, 14.7; FT-IR (KBr, cm^{-1}) 3412, 2961, 2924, 2853, 1741, 1639, 1614, 1598, 1527, 1500; HR-MS (*m/z)* for $C_{21}H_{16}N_2O_{10}$: Calculated 456.0805, found 456.0806.

2-(4-Bromo-3-methylphenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (3p)



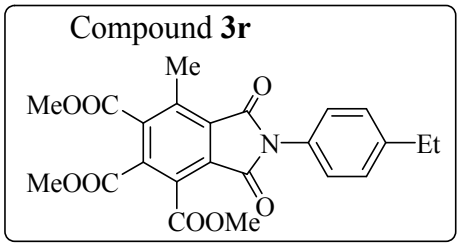
Light brown solid; m.p. 162-163 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (d, J = 8.4 Hz, 1H), 7.28 (s, 1H), 7.11-7.08 (m, 1H), 4.00 (s, 3H), 3.97 (s, 3H), 3.91 (s, 3H), 2.75 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.6, 164.5, 164.1, 163.1, 162.8, 140.9, 138.2, 137.0, 132.0, 130.0, 129.0, 128.5, 128.1, 127.5, 124.3, 124.0, 52.5, 52.4, 52.1, 22.0, 13.6; FT-IR (KBr, cm^{-1}) 3419, 2952, 2923, 2852, 1779, 1734, 1722, 1638, 1617, 1447; HR-MS (*m/z)* for $C_{22}H_{18}BrNO_8$: Calculated 503.0216, found 503.0219 (One of the major peaks).

2-(4-Isopropylphenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (3q)



White solid; m.p. 168-169 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27-7.16 (m, 4H), 3.90 (s, 3H), 3.87 (s, 3H), 3.81 (s, 3H), 2.90-2.81 (m, 1H), 2.66 (s, 3H), 1.18 (d, J = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7. 165.8, 165.2, 164.2, 149.5, 141.8, 137.8, 131.1, 129.4, 129.3, 128.3, 127.2, 126.3, 53.5, 53.3, 53.1, 33.9, 23.8, 14.5; FT-IR (KBr, cm^{-1}) 3458, 2959, 2927, 2872, 1740, 1717, 1635, 1516, 1440; HR-MS (*m/z)* for $C_{24}H_{23}NO_8$: 453.1424, found 453.1422.

2-(4-Ethylphenyl)-7-methyl-1,3-dioxo-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (3r)



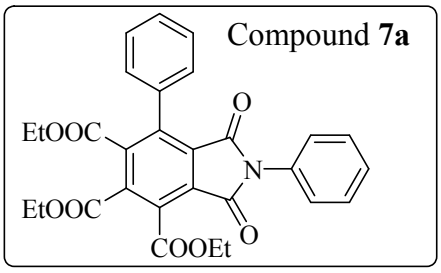
White solid; m.p. 165-166 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.26 (m, 4H), 3.98 (s, 3H), 3.95 (s, 3H), 3.90 (s, 3H), 2.75 (s, 3H), 2.72 (q, J = 7.8 Hz, 2H), 1.27 (t, J = 7.8 Hz, 3H), ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 165.8, 165.2, 164.2, 144.9, 141.8, 137.8, 131.2, 129.4, 129.3, 128.6, 128.3, 126.4, 53.4, 53.3, 53.1, 28.5, 15.3, 14.6; FT-IR (KBr, cm^{-1}) 3418, 2955, 2922, 2851, 1740, 1720, 1637, 1618, 1515, 1438; HR-MS (*m/z)* for $C_{23}H_{21}NO_8$: Calculated 439.1267, found 439.1261.

2.c. General procedure of *N*-aryl biarylimides (7a-g)

In a 25 mL round bottom flask 1.0 mmol of benzoyl acetanilide (**6**) and 2.2 mmol of dialkyl acetylene dicarboxylate (**2**) were taken in presence of 50 mg (0.15 mmol) of CTAB in 4 mL of H_2O. Then 1.0 mmol of 4-dimethylaminopyridine (DMAP) was added to it and the reaction mixture was stirred for the stipulated period of time at room temperature. The progress of the reaction was monitored by TLC. To the post-reaction mixture 15 mL of water and 20 mL of DCM were added and teken in a separating funnel. The organic layer was separated and dried over anhydrous Na_2SO_4. Solvent was evaporated under reduced pressure and the crude reaction mixture was purified by column chromatography using silica gel and n-hexane-ethyl acetate as eluent to yield the pure product unsymmetrical biarylimides **7**.

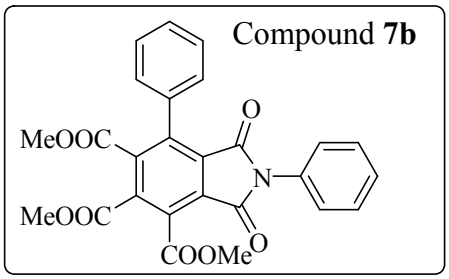
2.d. Characterization data of compound (7a-g)

1,3-Dioxo-2,7-diphenyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (7a)



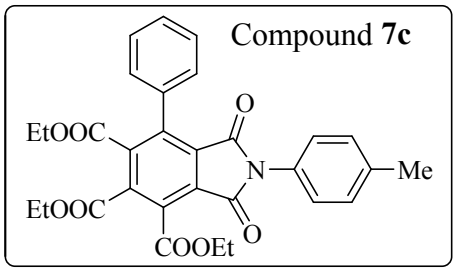
Light brown solid; m.p. 156-158 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.32 (m, 10H), 4.56 (q, J = 6.9 Hz, 2H), 4.42 (q, J = 7.2 Hz, 2H), 4.05 (q, J = 7.2Hz, 2H), 1.45-1.30 (m, 6H), 0.96 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7, 164.6, 164.3, 163.8, 163.7, 141.9, 140.0, 132.6, 131.0, 130.8, 130.5, 129.1, 129.1, 129.0, 128.8, 128.4, 127.9, 126.4, 63.0, 62.7, 62.0, 13.8, 13.7, 13.4; FT-IR (KBr, cm^{-1}) 3548, 3418, 2990, 2903, 1785, 1734, 1723, 1638, 1615, 1495; HR-MS (*m/z)* for $C_{29}H_{25}NO_8$: Calculated 515.1580, found 515.1577.

1,3-Dioxo-2,7-diphenyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (7b)



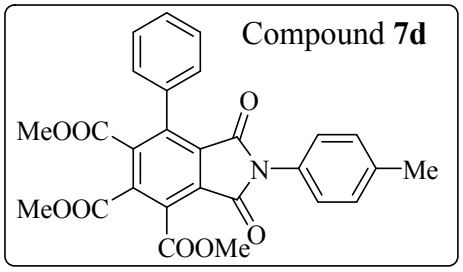
Light brown solid; m.p. 210-212 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.35 (m, 4H), 7.30-7.27 (m, 6H), 3.97 (s, 3H), 3.85 (s, 3H), 3.49 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 165.1, 164.1, 164.0, 163.8, 141.9, 140.2, 132.4, 132.1, 130.7, 129.4, 129.2, 129.0, 128.8, 128.5, 127.9, 126.4, 53.5, 53.4, 52.7; FT-IR (KBr, cm^{-1}) 3443, 2949, 2924, 2854, 1784, 1732, 1613, 1596, 1496; HR-MS (*m/z)* for $C_{26}H_{19}NO_8$: Calculated 473.1111, found 473.1113.

1,3-Dioxo-7-phenyl-2-p-tolyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (7c)



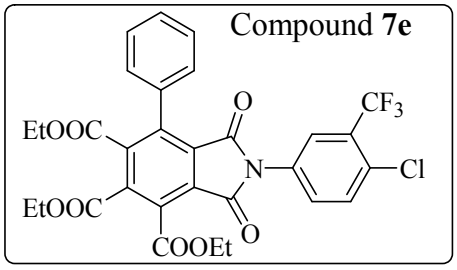
Pale yellow solid; m.p. 138-139 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 (d, J = 4.8 Hz, 3H), 7.27-7.23 (m, 2H), 7.19-7.11 (m , 4H), 4.48 (q, J = 7.2 Hz, 2H), 4.35 (q, J = 7.2 Hz, 2H), 3.98 (q, J = 7.2 Hz, 2H), 2.29 (s, 3H), 1.38-1.27 (m, 6H), 0.89 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7, 164.6, 164.3, 163.9, 163.7, 141.8, 139.9, 138.4, 132.6, 130.5, 129.5, 129.2, 128.9, 128.8, 128.1, 127.8, 126.1, 62.8, 62.6, 61.9, 21.0, 13.8, 13.6, 13.3; FT-IR (KBr, cm^{-1}) 3439, 2992, 2962, 2926, 1784, 1738, 1725, 1638, 1516, 1465, 1447; HR-MS (*m/z)* for $C_{30}H_{27}NO_8$: Calculated 529.1737, found 529.1734.

1,3-Dioxo-7-phenyl-2-p-tolyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (7d)



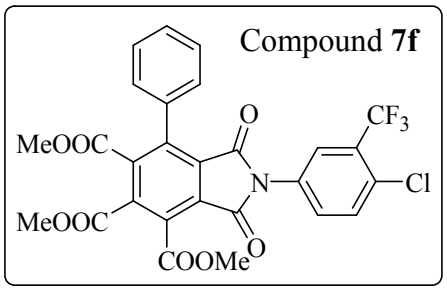
Pale yellow; m.p. 228-230 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.37 (m, 3H), 7.28-7.26 (m, 2H), 7.19-7.17 (m, 4H), 4.02 (s, 3H), 3.86 (s, 3H), 3.50 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 165.2, 164.2, 164.0, 163.9, 141.8, 140.1, 138.5, 134.3, 132.4, 130.7, 130.6, 129.6, 129.5, 129.1, 128.7, 128.1, 127.9, 126.1, 55.5, 53.4, 52.7, 21.1; FT-IR (KBr, cm^{-1}) 3449, 3004, 2950, 1784, 1744, 1734, 1615, 1516, 1448, 1441; HR-MS (*m/z)* for $C_{27}H_{21}NO_8$: Calculated 487.1267, found 487.1263.

2-(4-Chloro-3-trifluoromethylphenyl)-1,3-dioxo-7-phenyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid triethyl ester (7e)



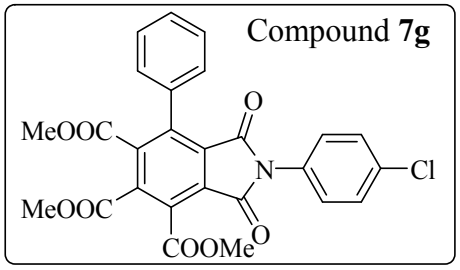
White solid; m.p. 166-168 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (s, 1H), 7.54-7.44 (m, 2H), 7.40-7.35 (m, 3H), 7.27-7.19 (m, 2H), 4.49 (q, J = 7.2 Hz, 2H), 4.36 (q, J = 7.2 Hz, 2H), 3.99 (q, J = 7.2 Hz, 2H), 1.39-1.27 (m, 6H), 0.90 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.5, 164.4, 163.6, 163.3, 142.3, 140.4, 132.4, 132.1, 131.3, 130.4, 130.1, 129.8, 129.3, 128.8, 128.0, 125.4, 63.1, 62.9, 62.2, 13.8, 13.7, 13.4; FT-IR (KBr, cm^{-1}) 3450, 2987, 2942, 2906, 1732, 1723, 1613, 1583, 1485; HR-MS (*m/z)* for $C_{30}H_{23}ClF_3NO_8$: Calculated 617.1064, found 617.1066 (One of the major peaks).

2-(4-Chloro-3-trifluoromethylphenyl)-1,3-dioxo-7-phenyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (7f)



Pale yellow solid; m.p. 126-128 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (s, 1H), 7.51 (s, 2H), 7.41 (d, J = 6.6 Hz, 3H), 7.27-7.19 (m, 2H), 3.98 (s, 3H), 3.86 (s, 3H), 3.51 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.8, 164.7, 163.7, 163.4, 163.1, 142.1, 140.5, 131.9, 130.8, 130.2, 129.5, 129.3, 128.9, 128.5, 127.9, 125.3, 125.2, 53.5, 53.4, 52.6; FT-IR (KBr, cm^{-1}) 3467, 3411, 2955, 1788, 1733, 1637, 1614, 1485, 1448; HR-MS (*m/z)* for $C_{27}H_{17}ClF_3NO_8$: Calculated 575.0595, found 575.0591 (One of the major peaks).

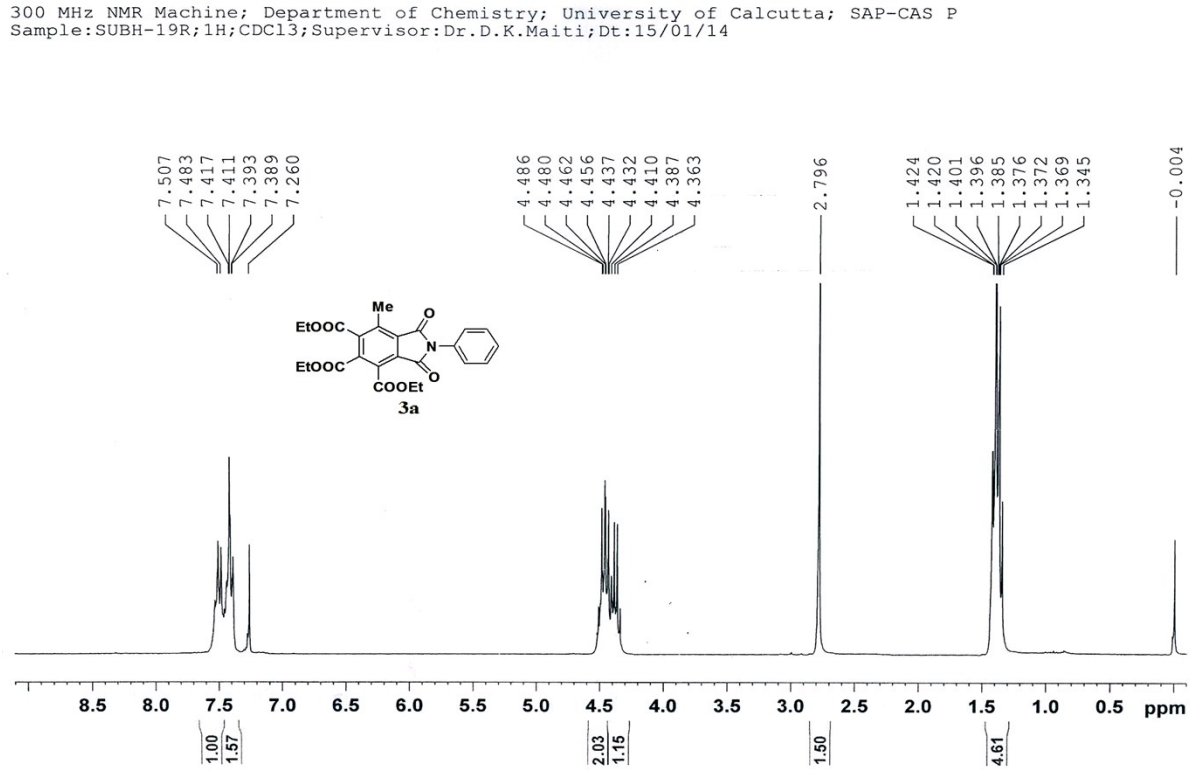
2-(4-Chlorophenyl)-1,3-dioxo-7-phenyl-2,3-dihydro-1*H*-isoindole-4,5,6-tricarboxylic acid trimethyl ester (7g)

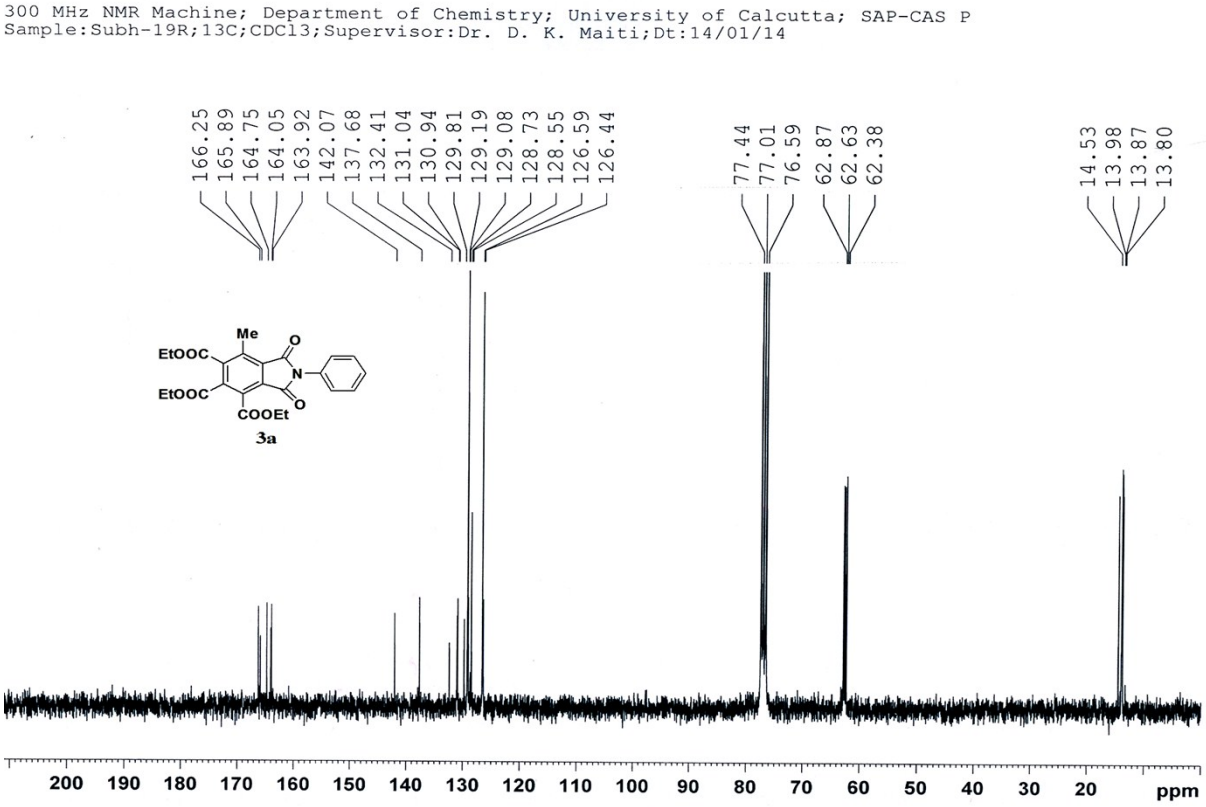


Orange solid; m.p. 184-185 ˚C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.34 (m, 6H), 7.28-7.25 (m, 2H), 7.20 (s, 1H), 3.98 (s, 3H), 3.86 (s, 3H), 3.50 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.9, 164.9, 163.8, 163.7, 163.4, 141.9, 140.2, 134.1, 132.1, 130.7, 130.4, 129.3, 129.2, 129.1, 128.6, 128.3, 128.2, 127.9, 127.8, 127.4, 127.3, 53.4, 53.3, 52.6; FT-IR (KBr, cm^{-1}) 3445, 3003, 2951, 2851, 1732, 1639, 1617, 1597, 1495, 1448; HR-MS (*m/z)* for $C_{26}H_{17}ClNO_8$: Calculated 507.0721, found 507.0724 (One of the major peaks).

3) ^{1}H & ^{13}C NMR of compounds 3a-r & 7a-g

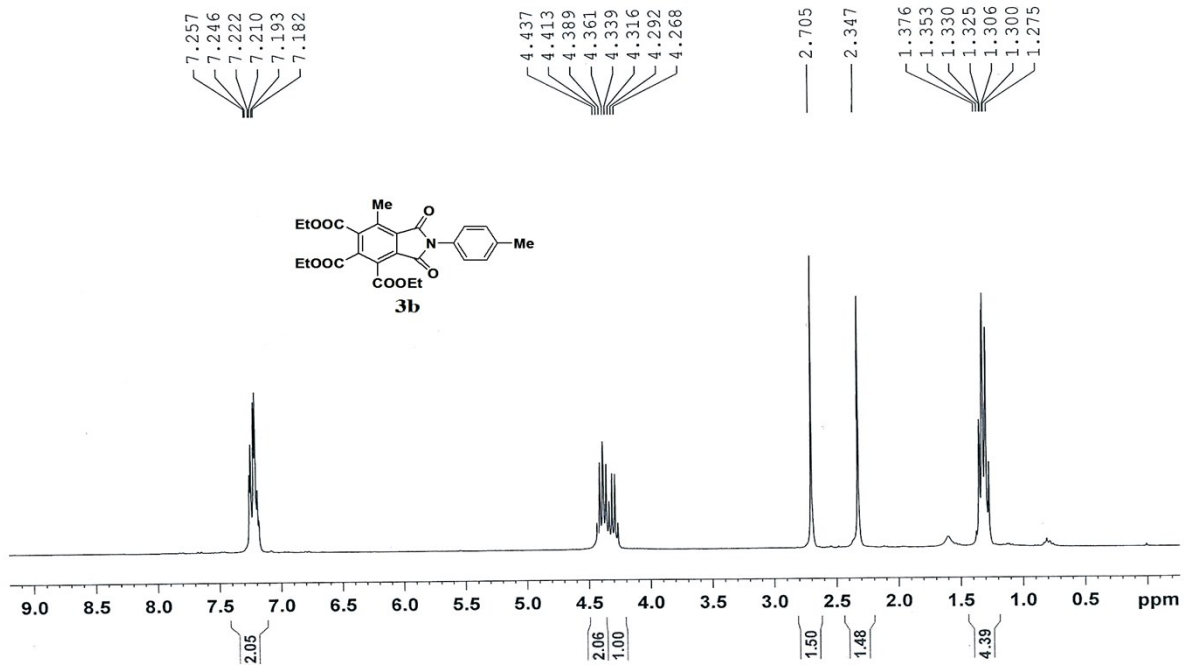
^{1}H & ^{13}C NMR of **3a**



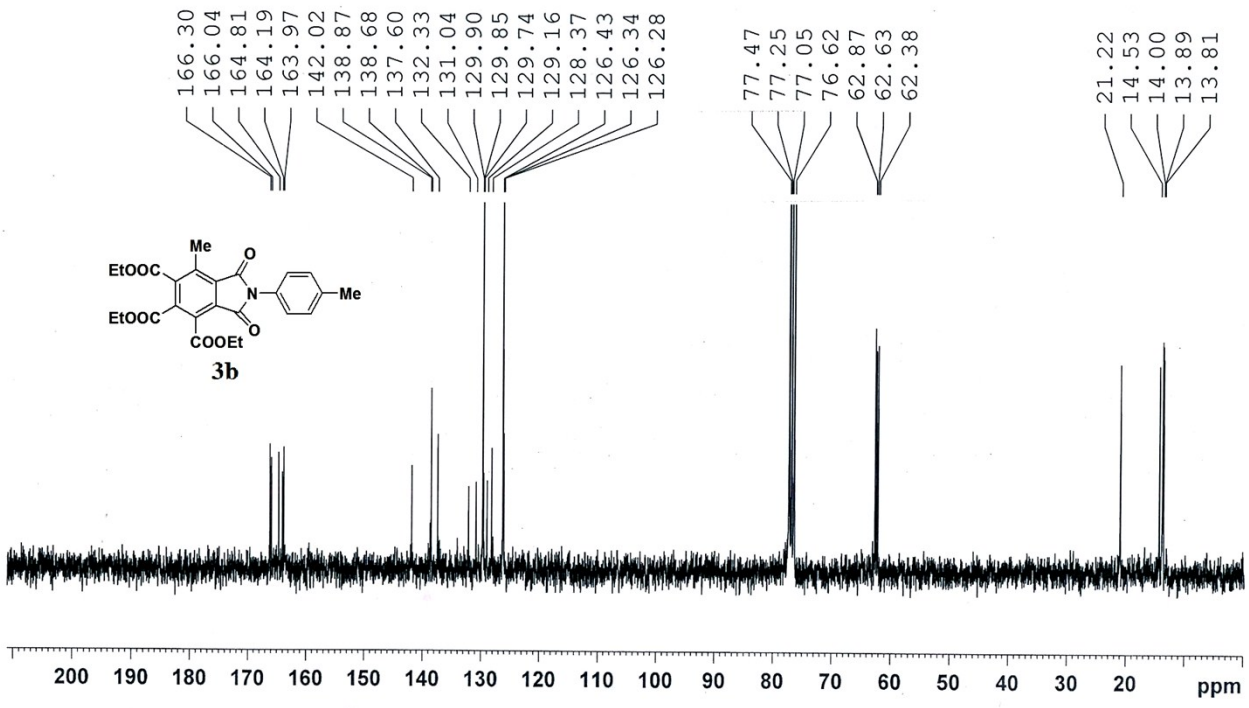


^{1}H & ^{13}C NMR of **3b**

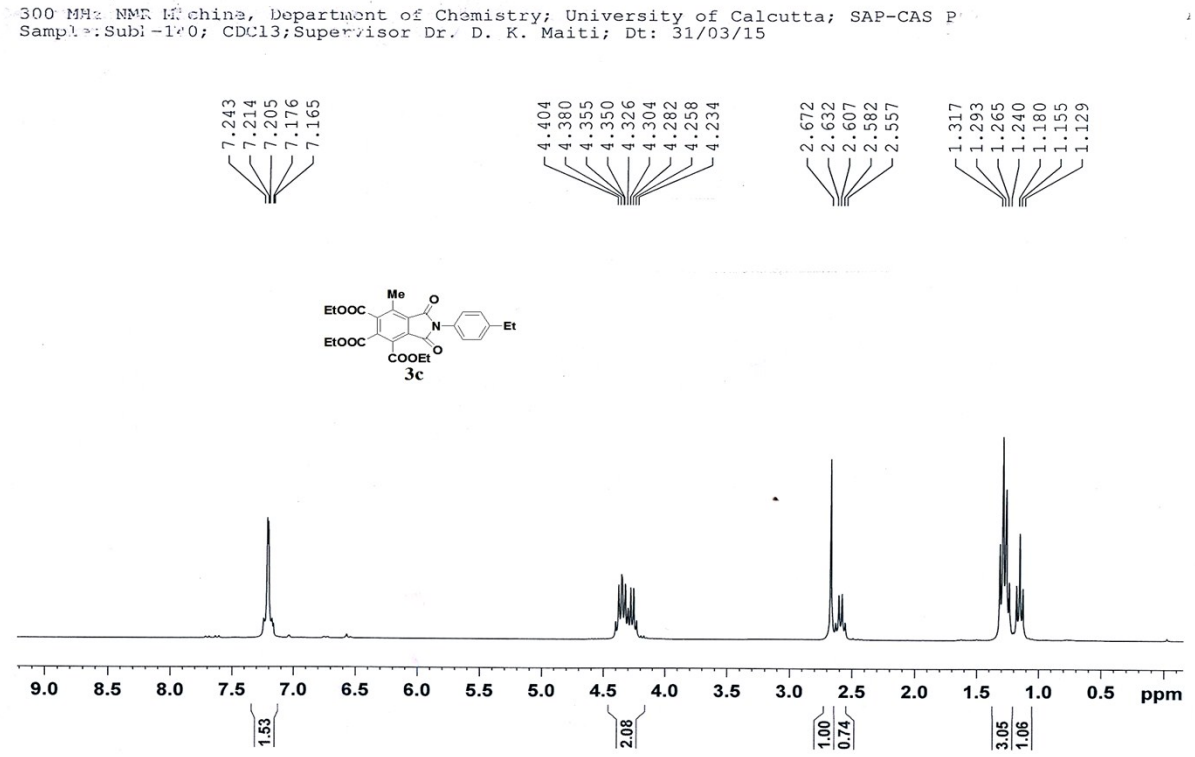
300 MHz NMR Machine; Department of Chemistry; University of Calcutta; SAP-CAS P
Sample:Subh-141R; PMR, CDCl3;Supervisor:Dr. D. K. Maiti; Dt: 27/05/15

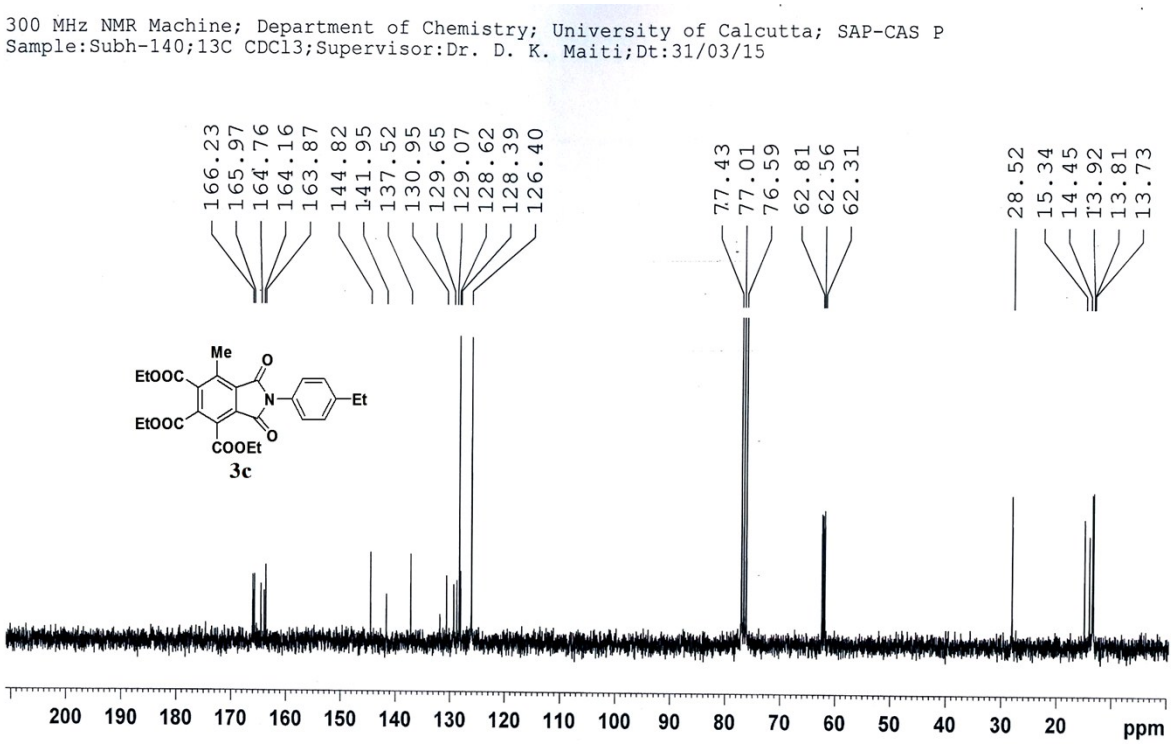


300 MHz NMR Machine; Department of Chemistry; University of Calcutta; SAP-CAS P
Sample:Subh-141R;13C; CDCl3;Supervisor:Dr. D. K. Maiti;Dt:28/05/15

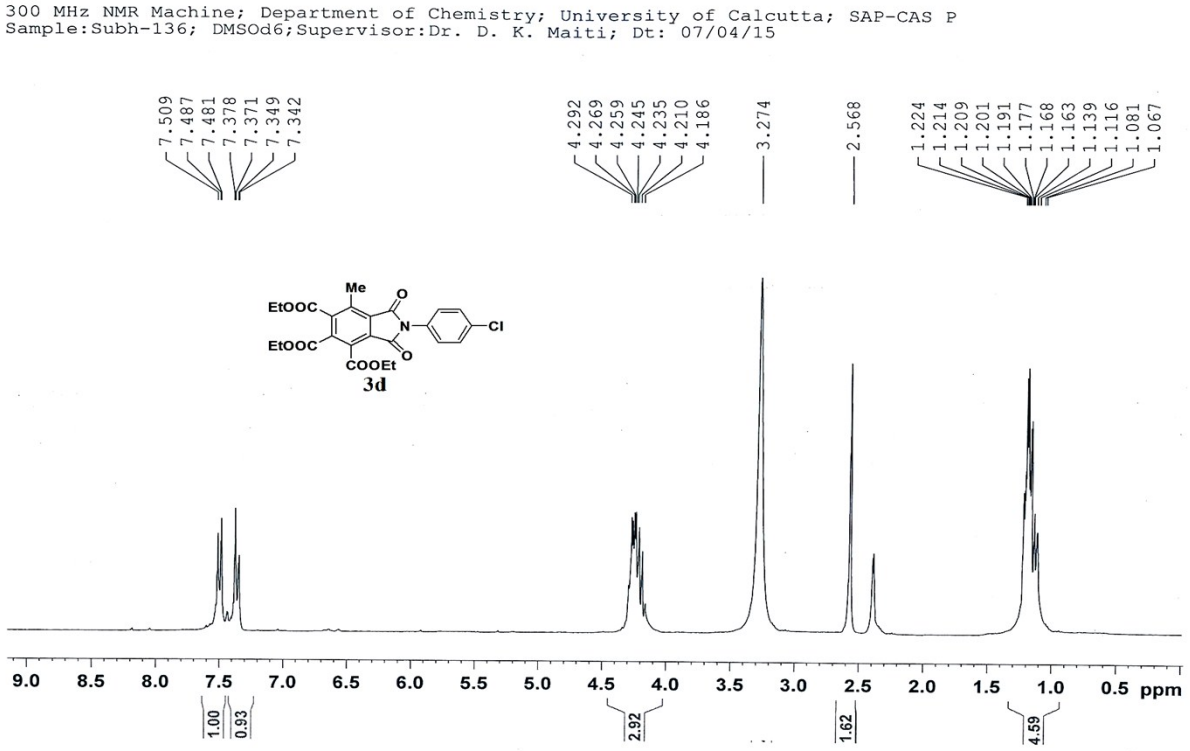


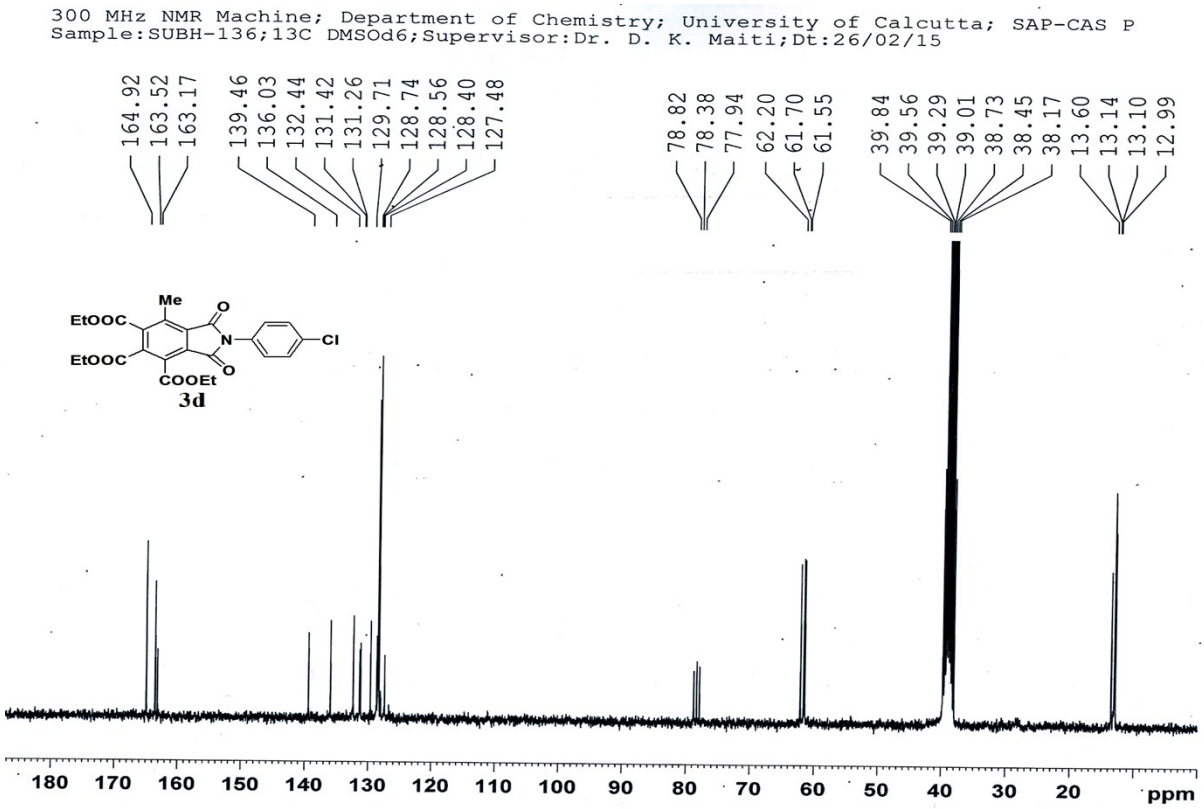
^{1}H & ^{13}C NMR of **3c**



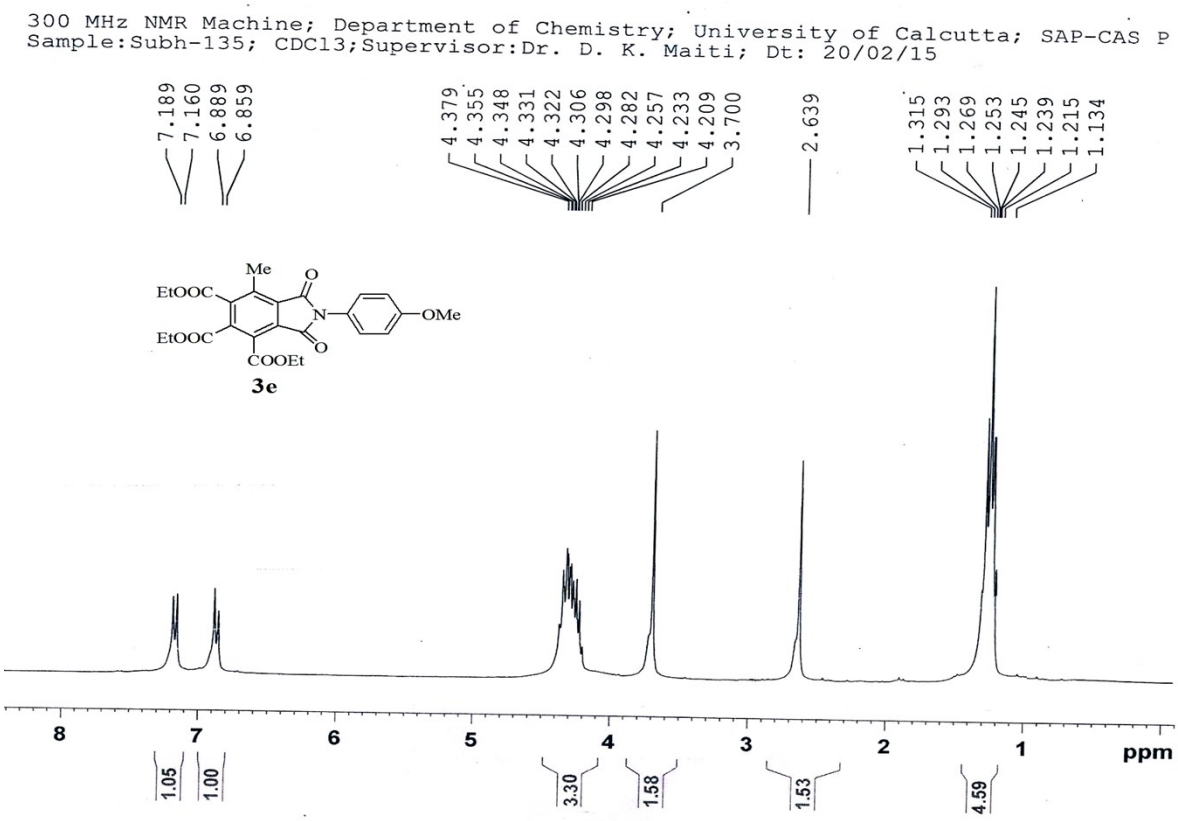


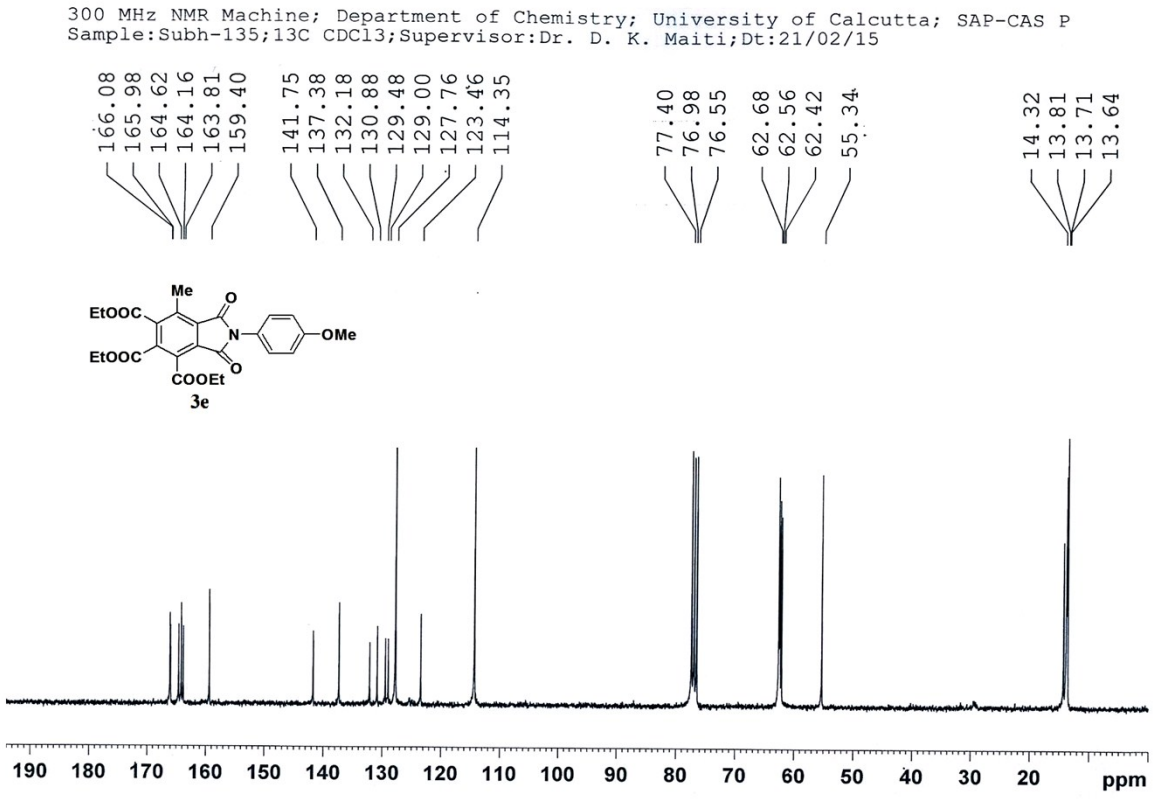
^{1}H & ^{13}C NMR of **3d**



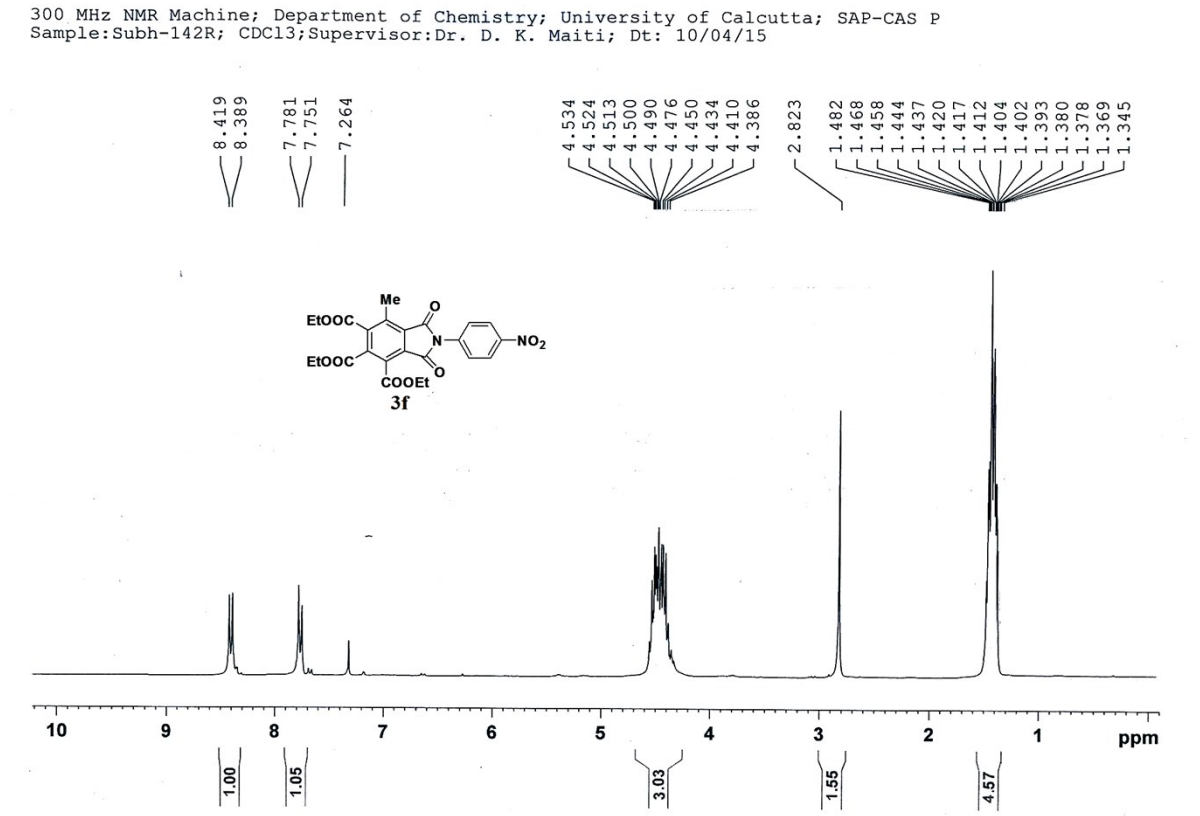


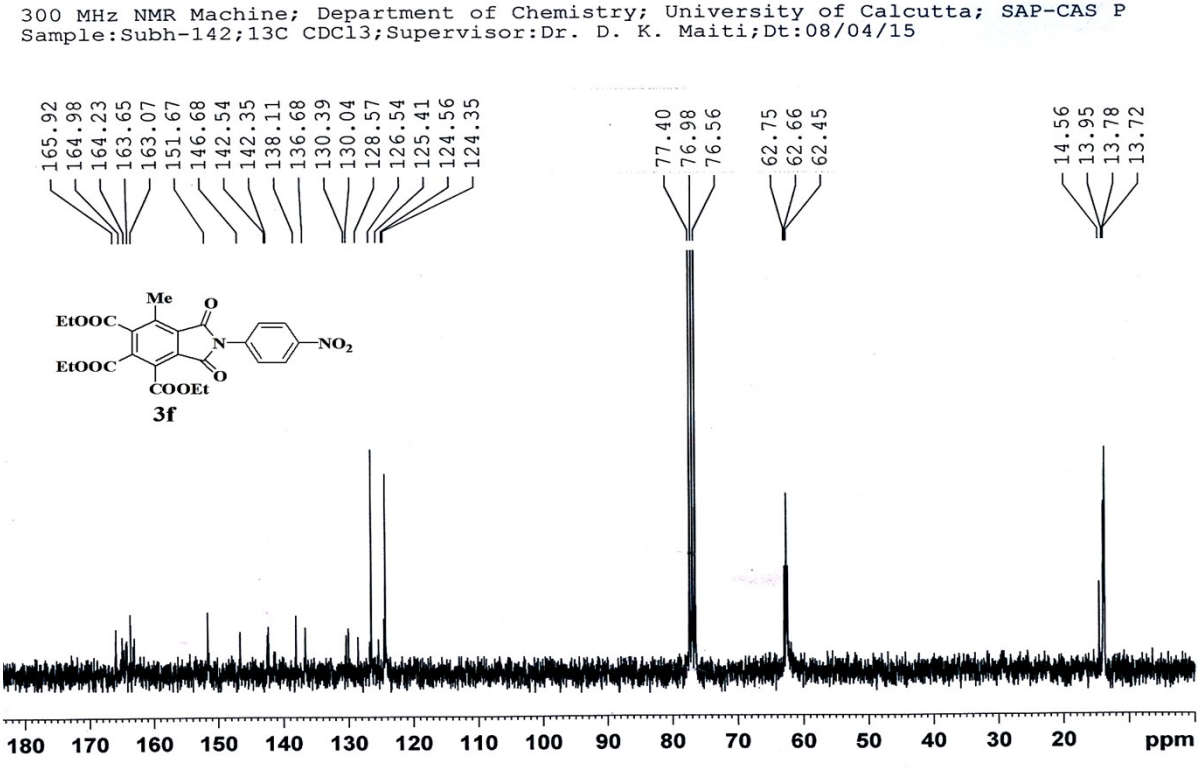
^{1}H & ^{13}C NMR of **3e**



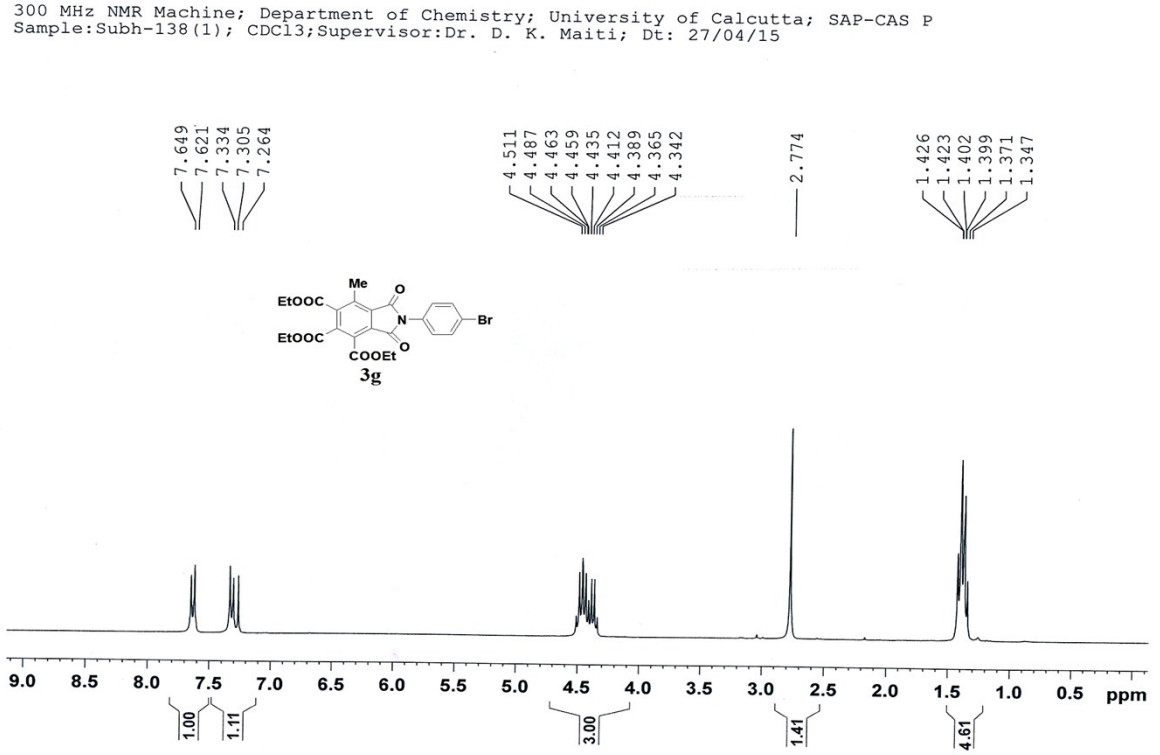


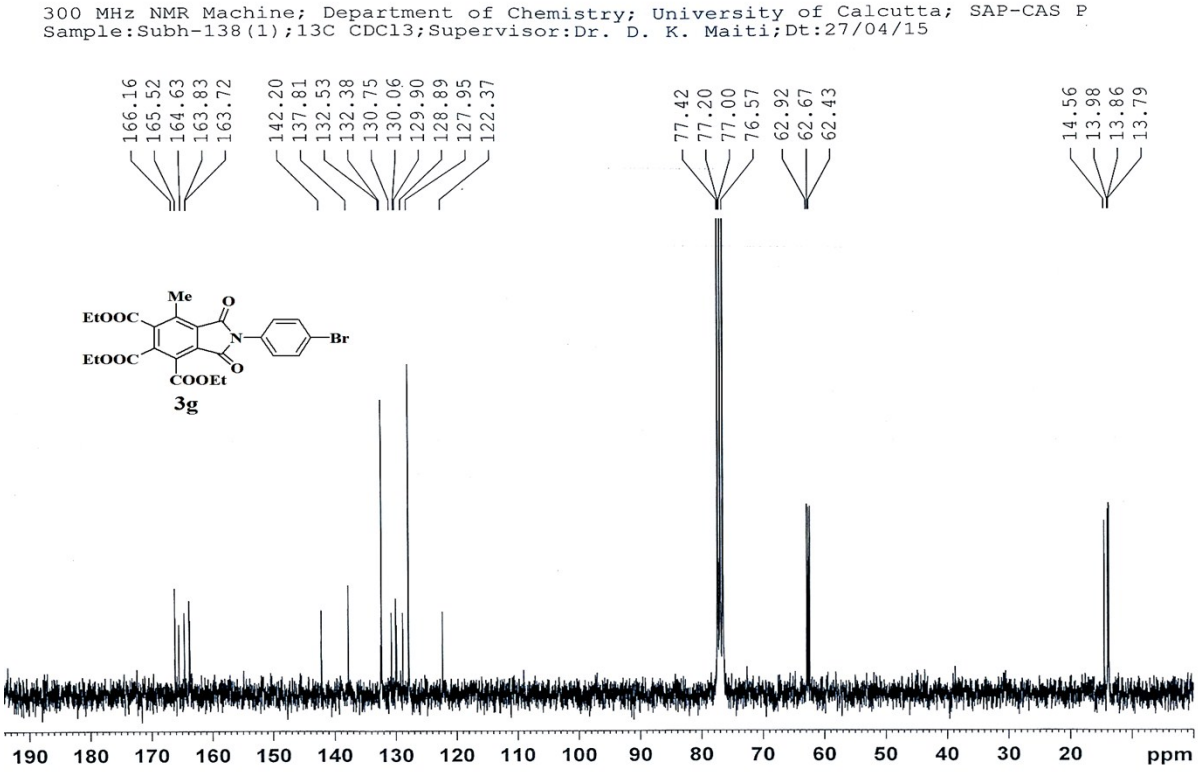
^{1}H & ^{13}C NMR of **3f**



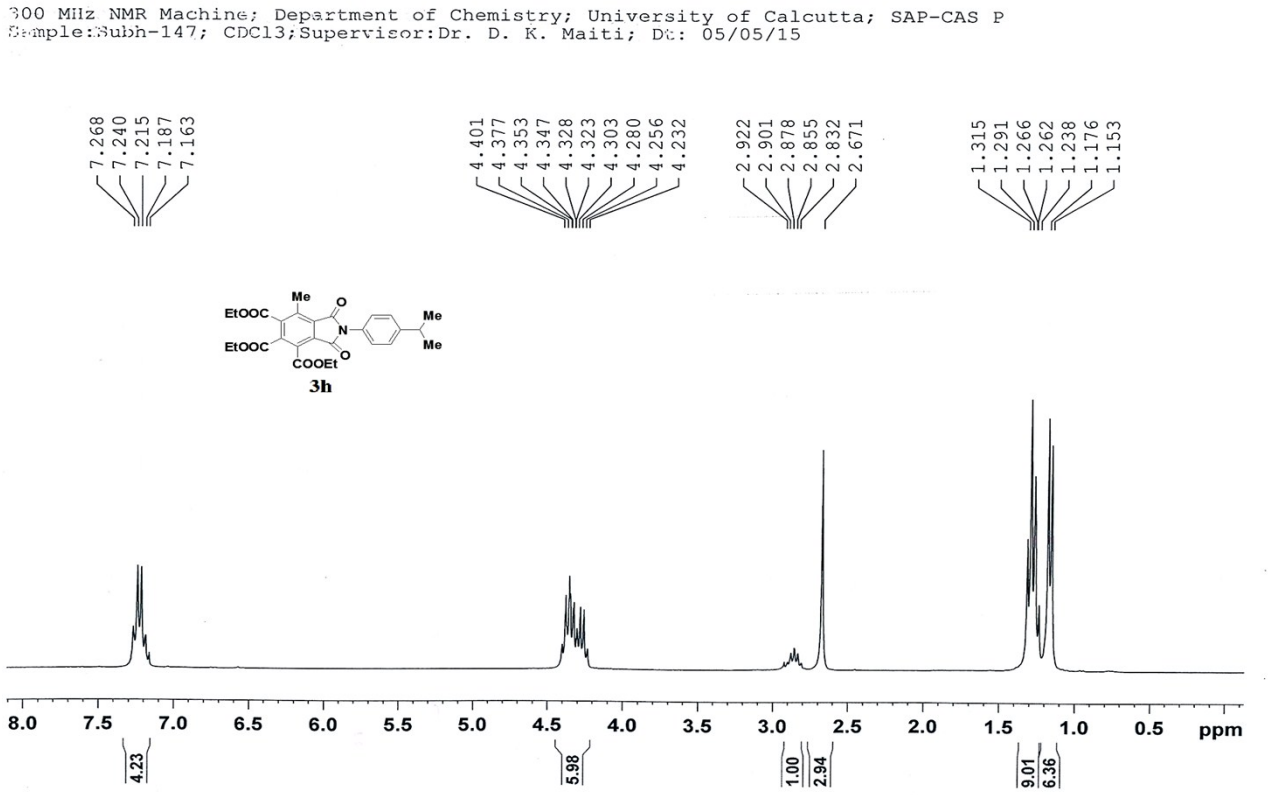


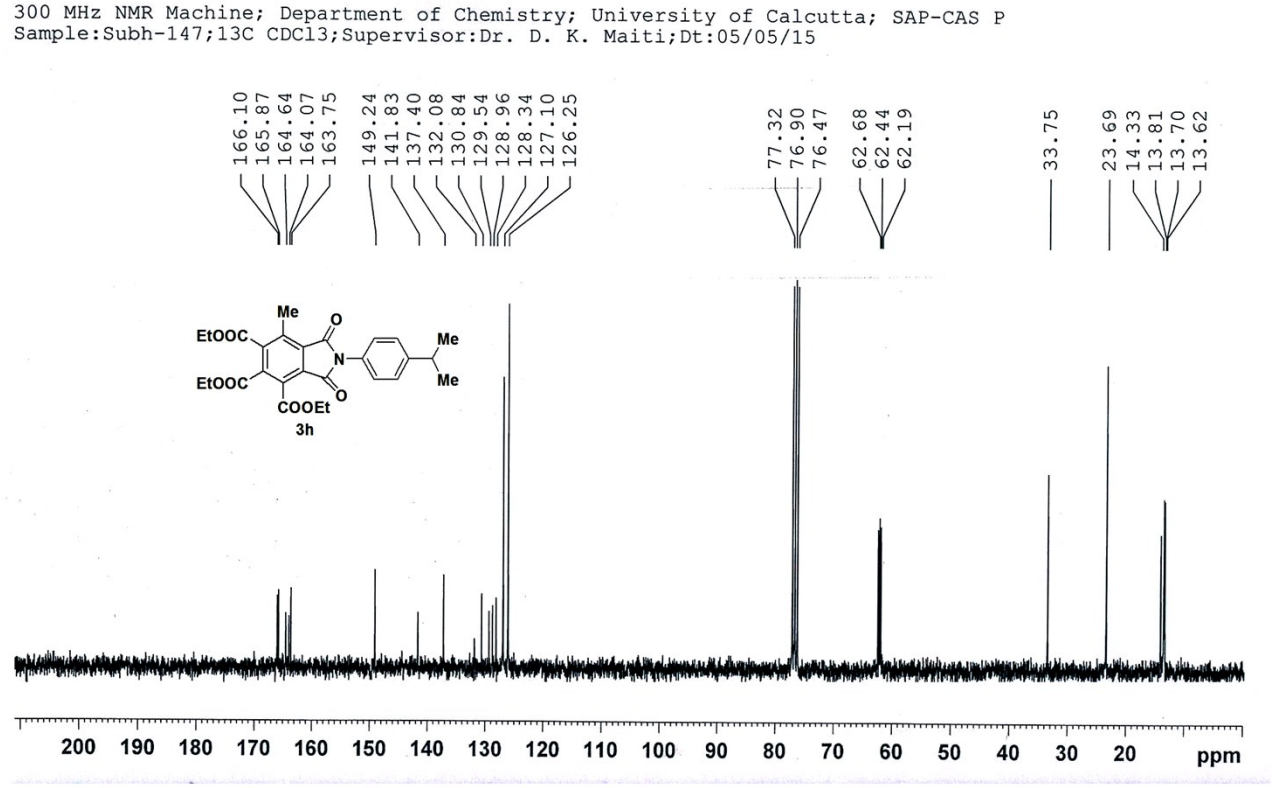
^{1}H & ^{13}C NMR of **3g**



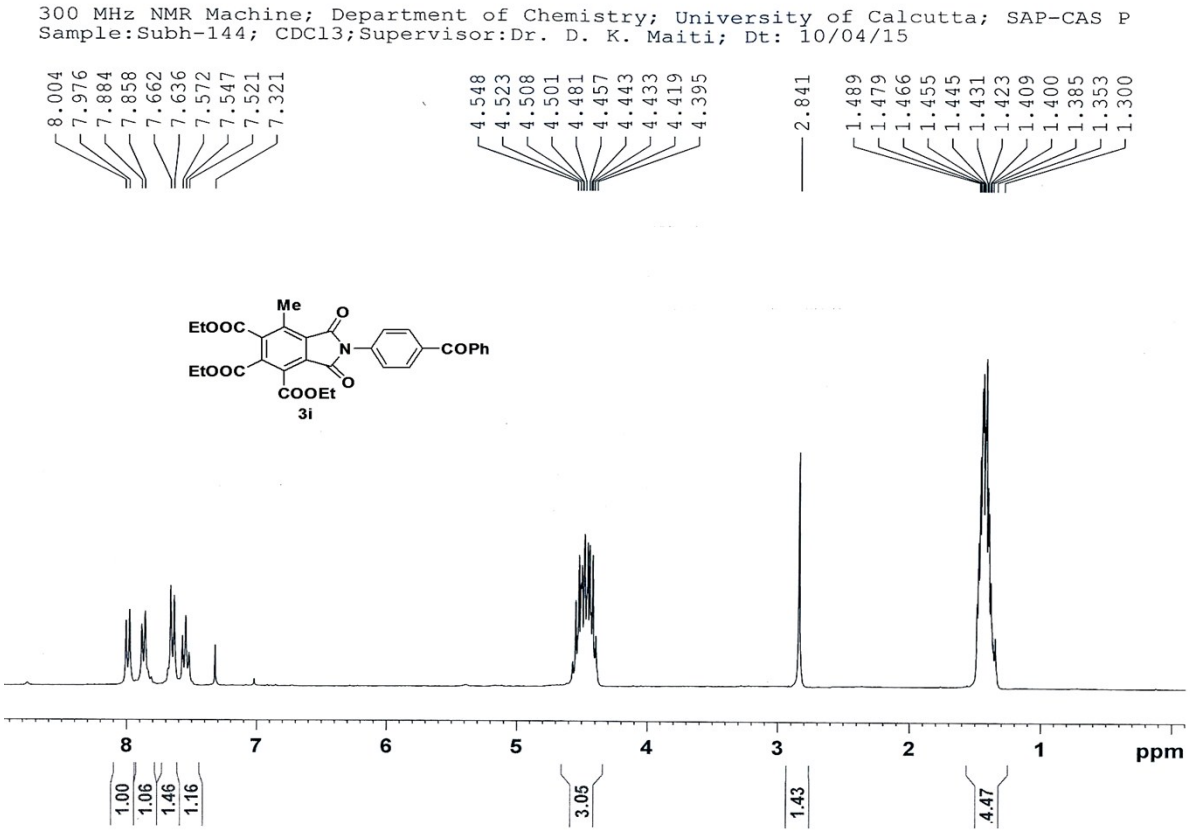


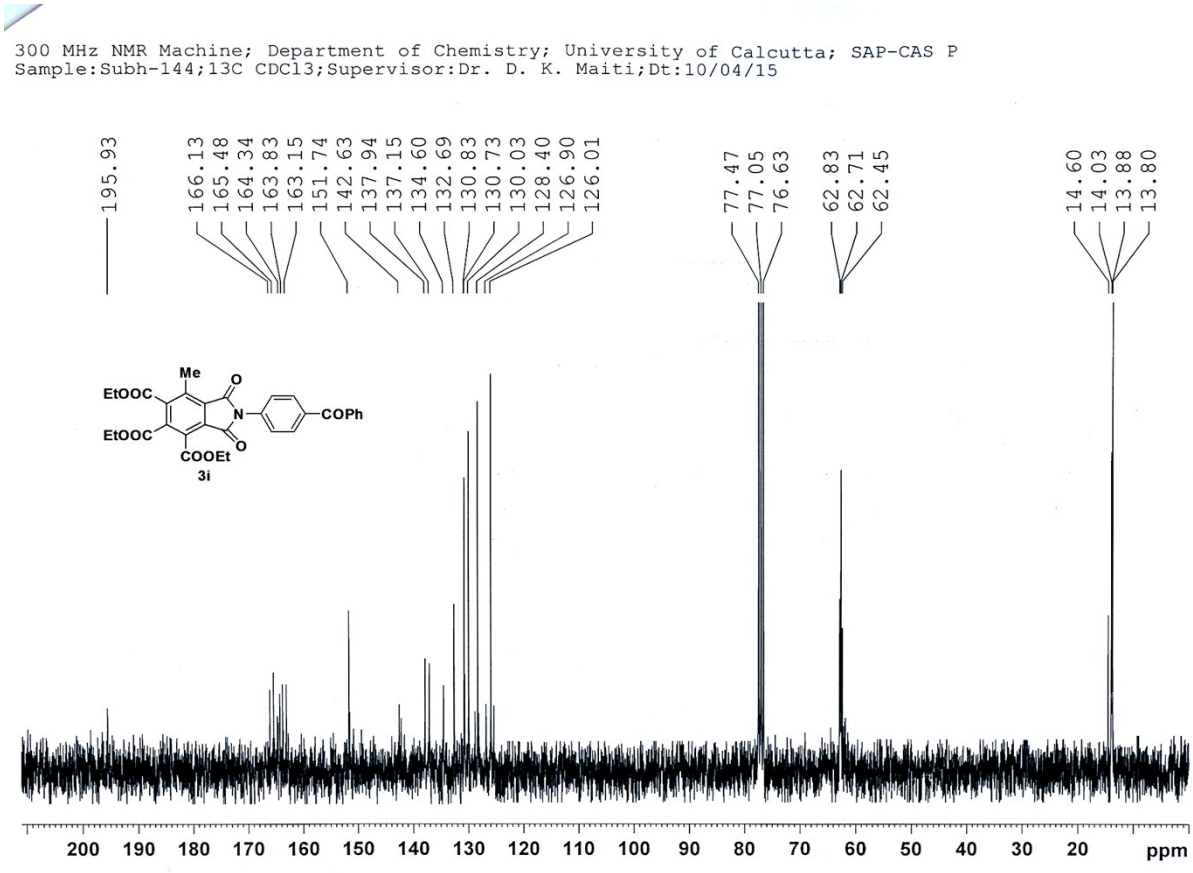
^{1}H & ^{13}C NMR of **3h**



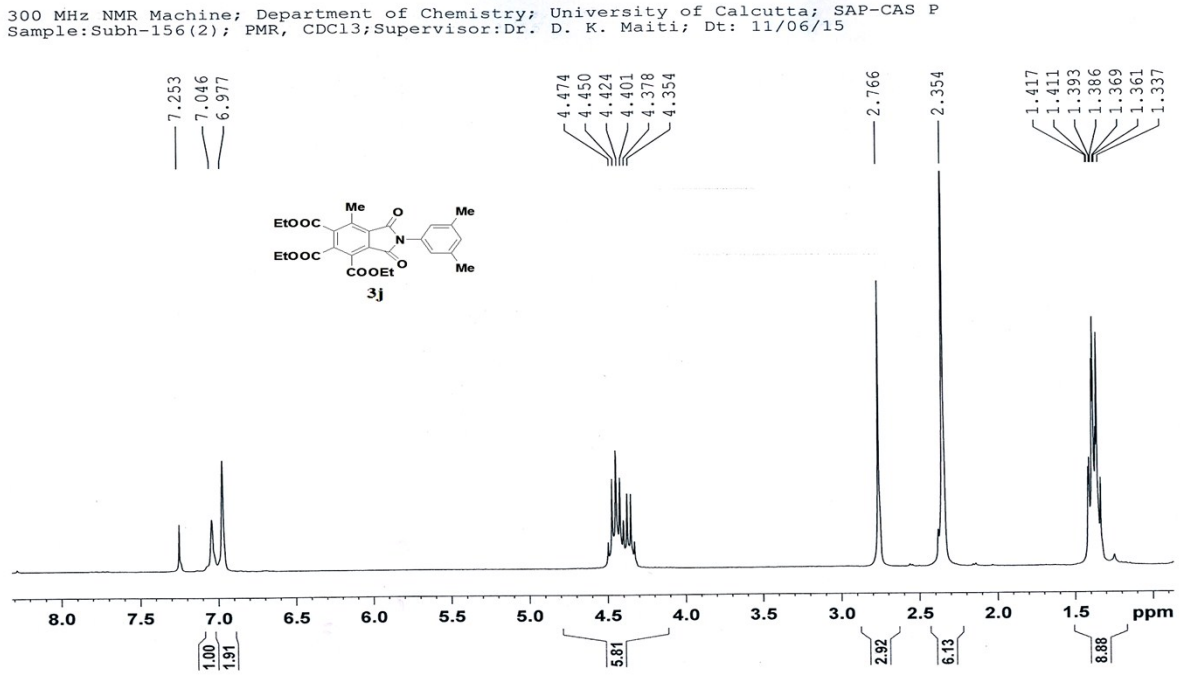


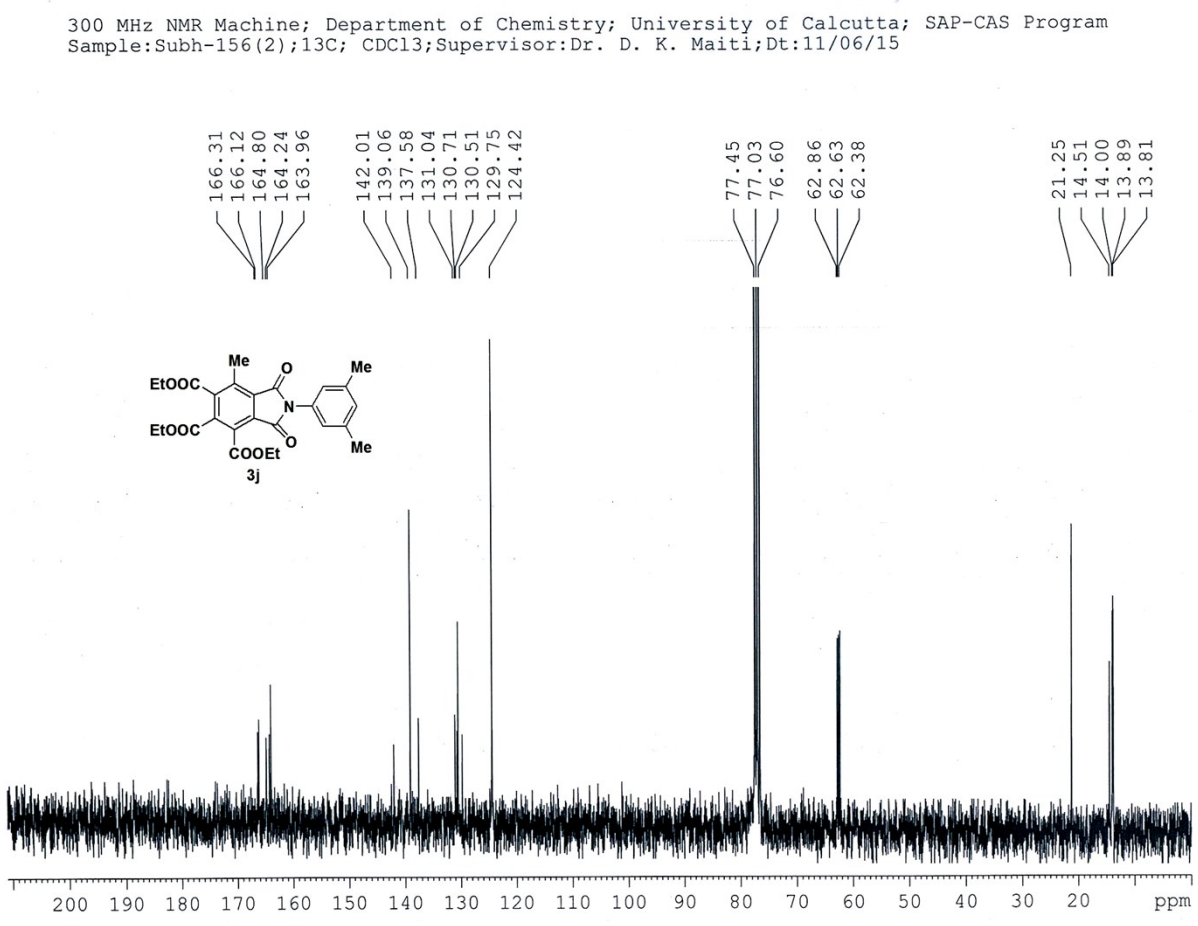
^{1}H & ^{13}C NMR of **3i**



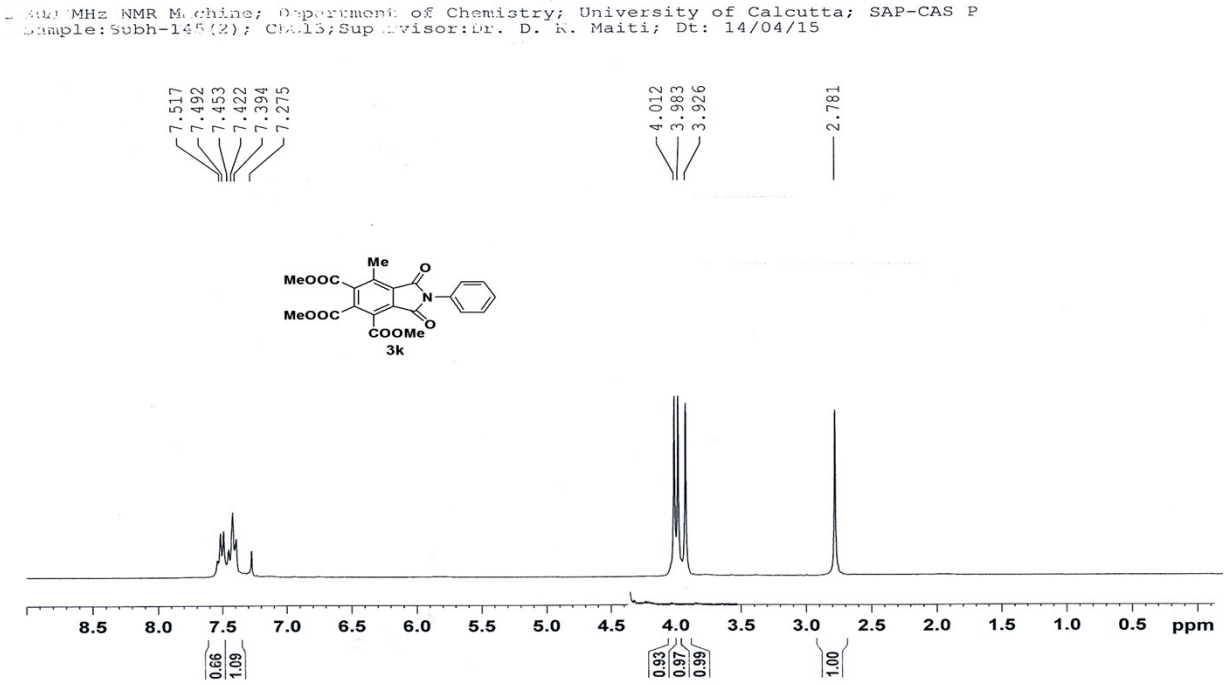


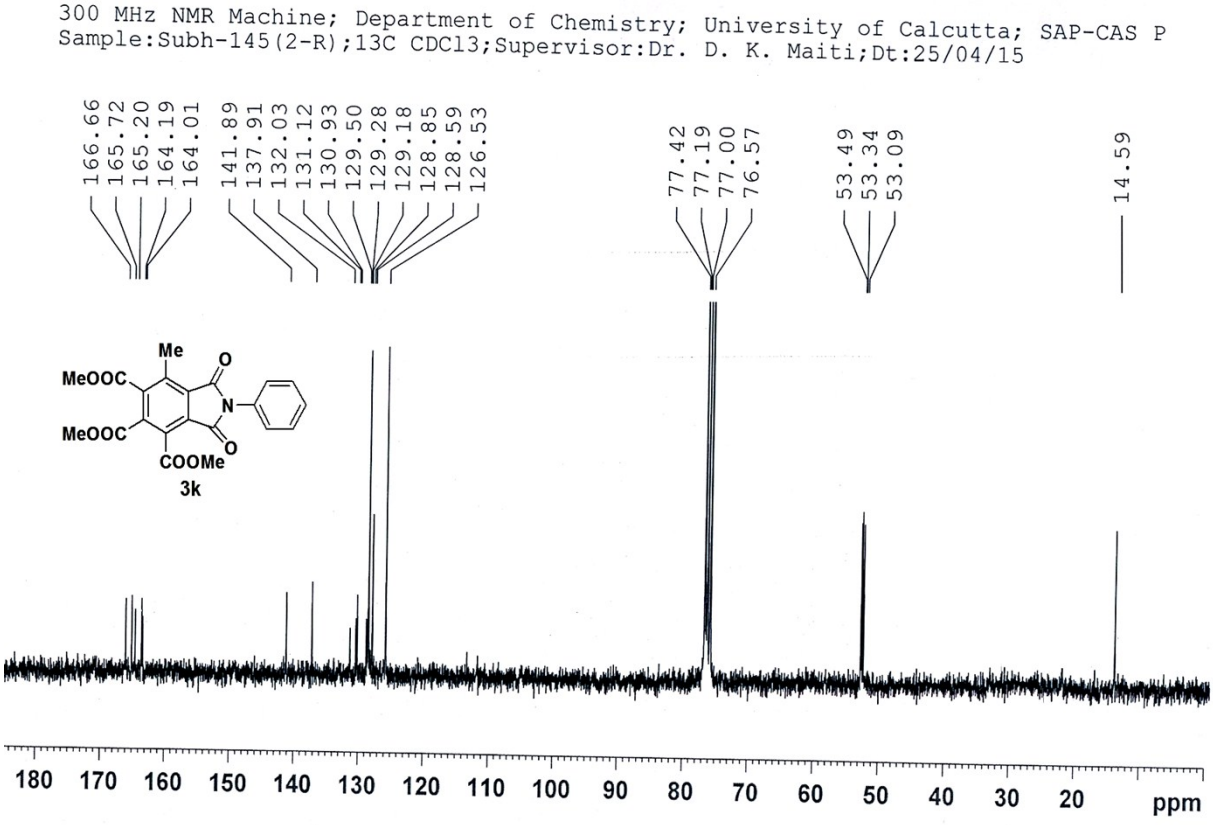
^{1}H & ^{13}C NMR of **3j**



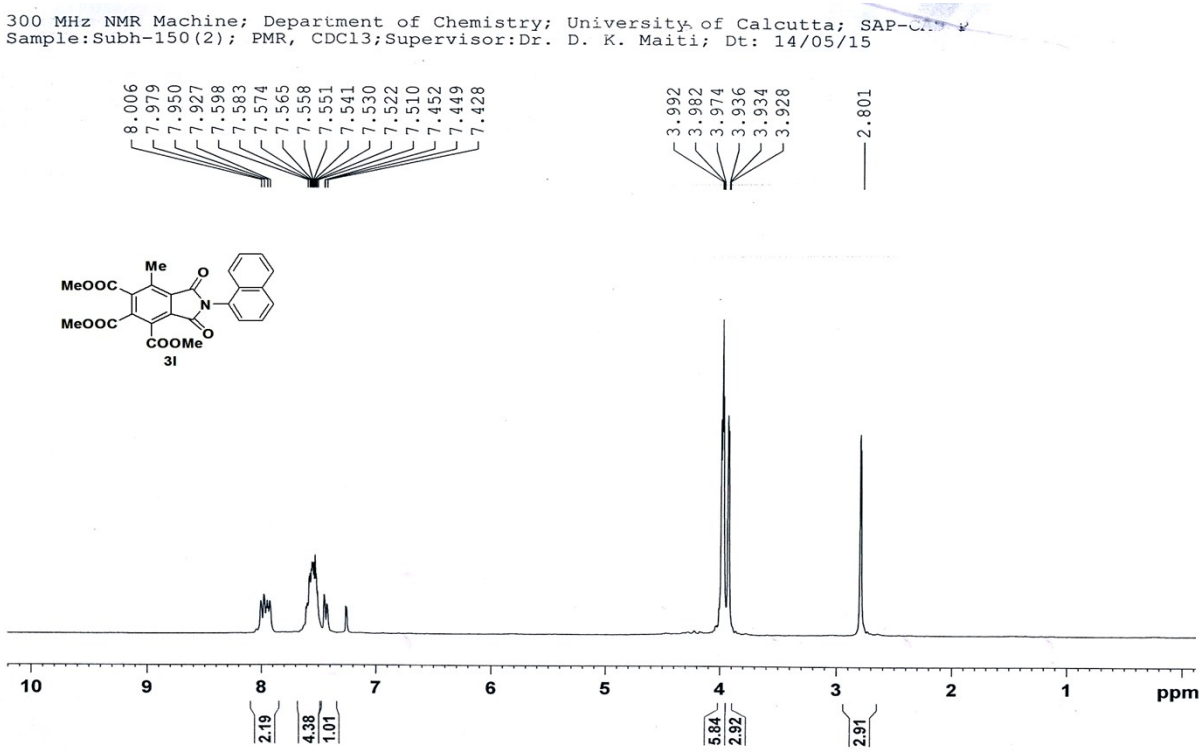


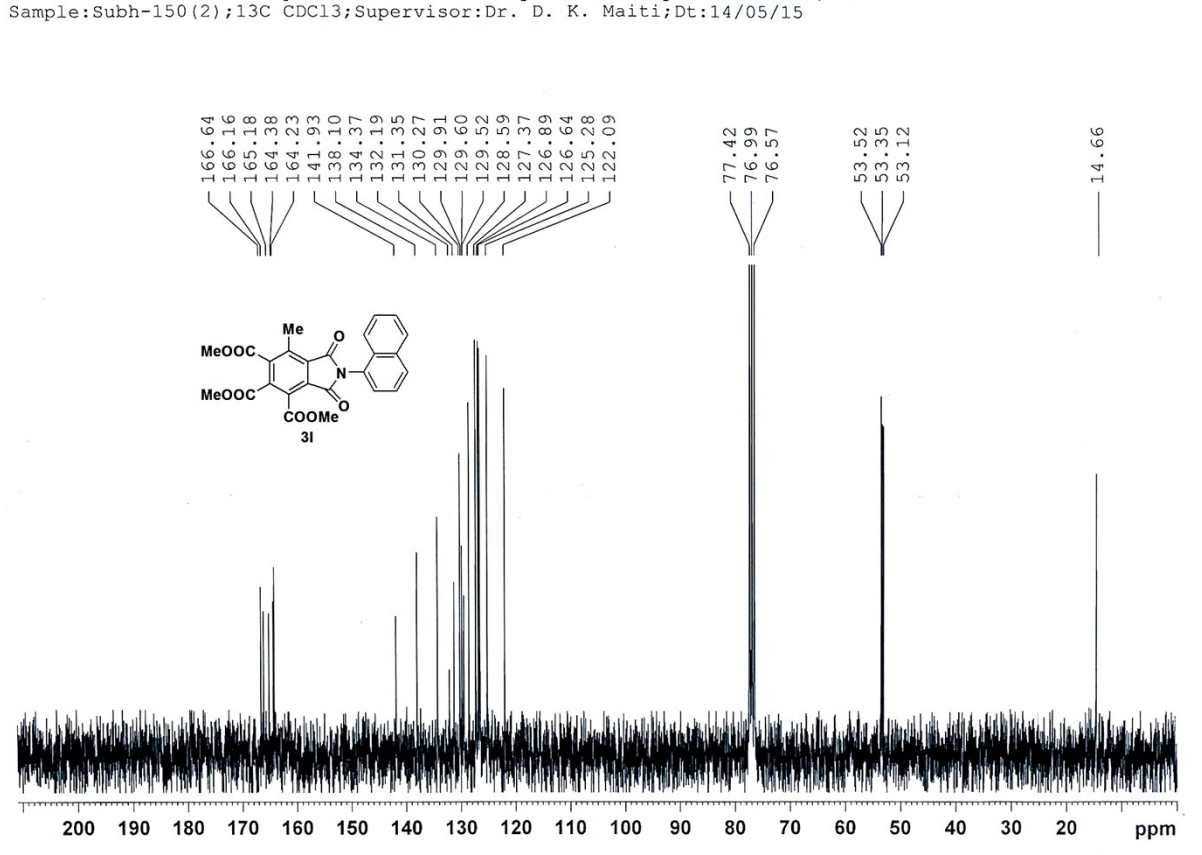
^{1}H & ^{13}C NMR of **3k**



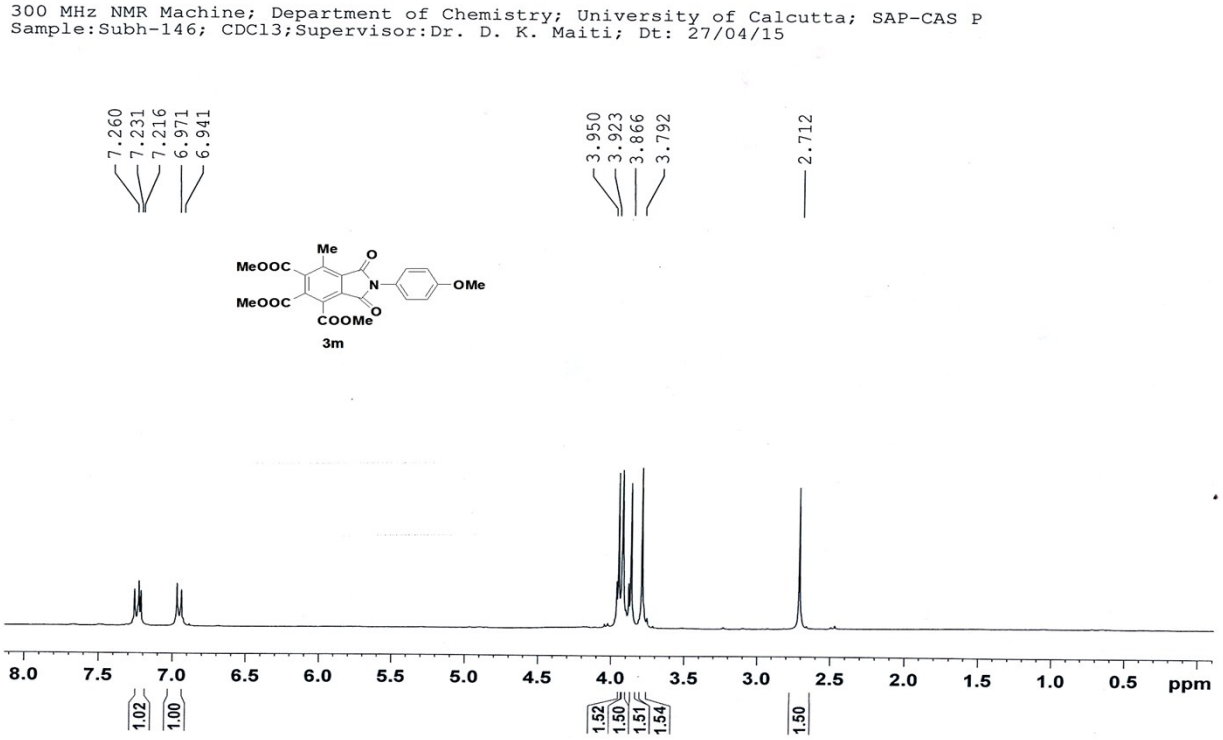


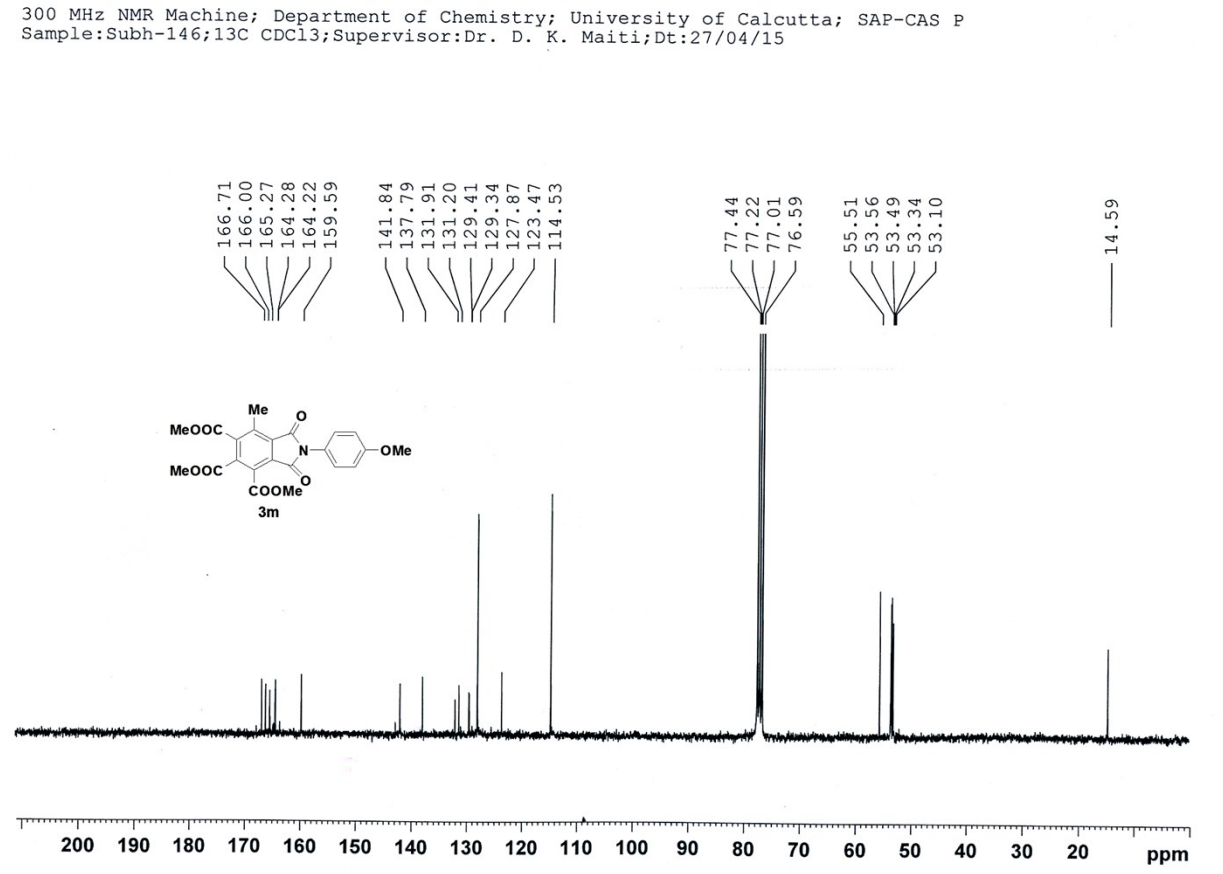
^{1}H & ^{13}C NMR of **3l**



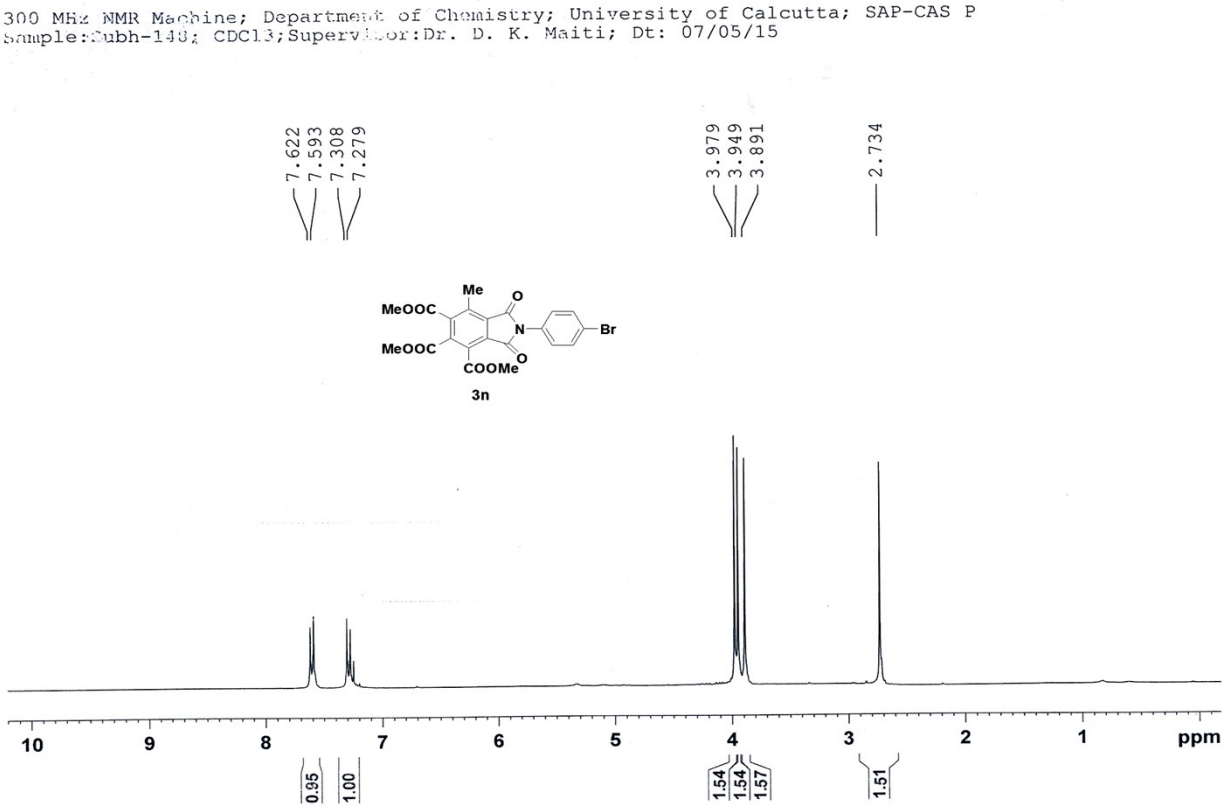


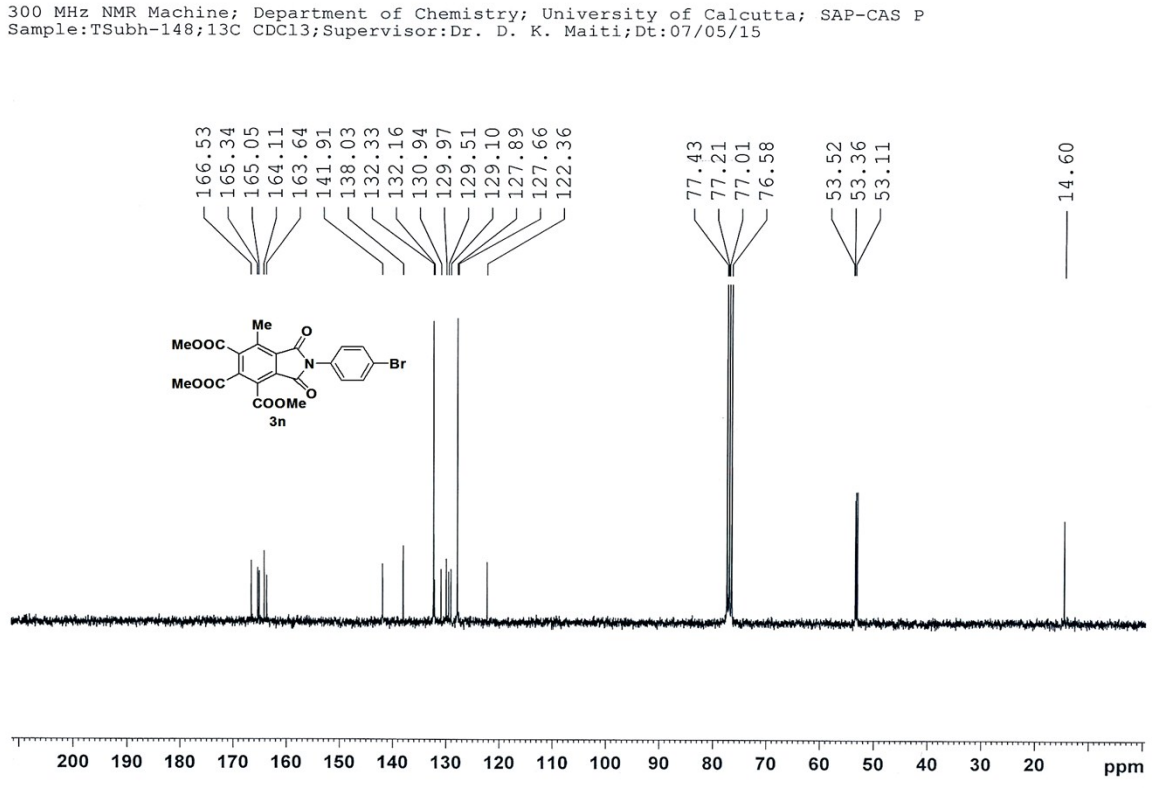
^{1}H & ^{13}C NMR of **3m**



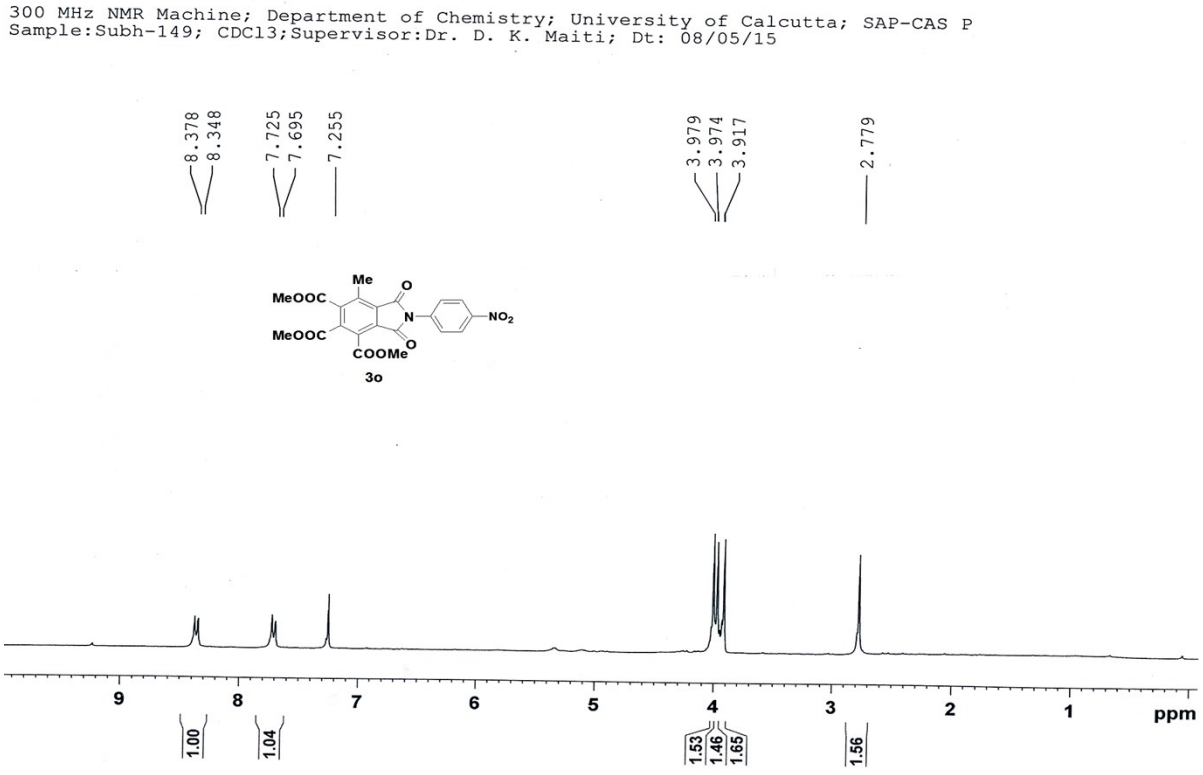


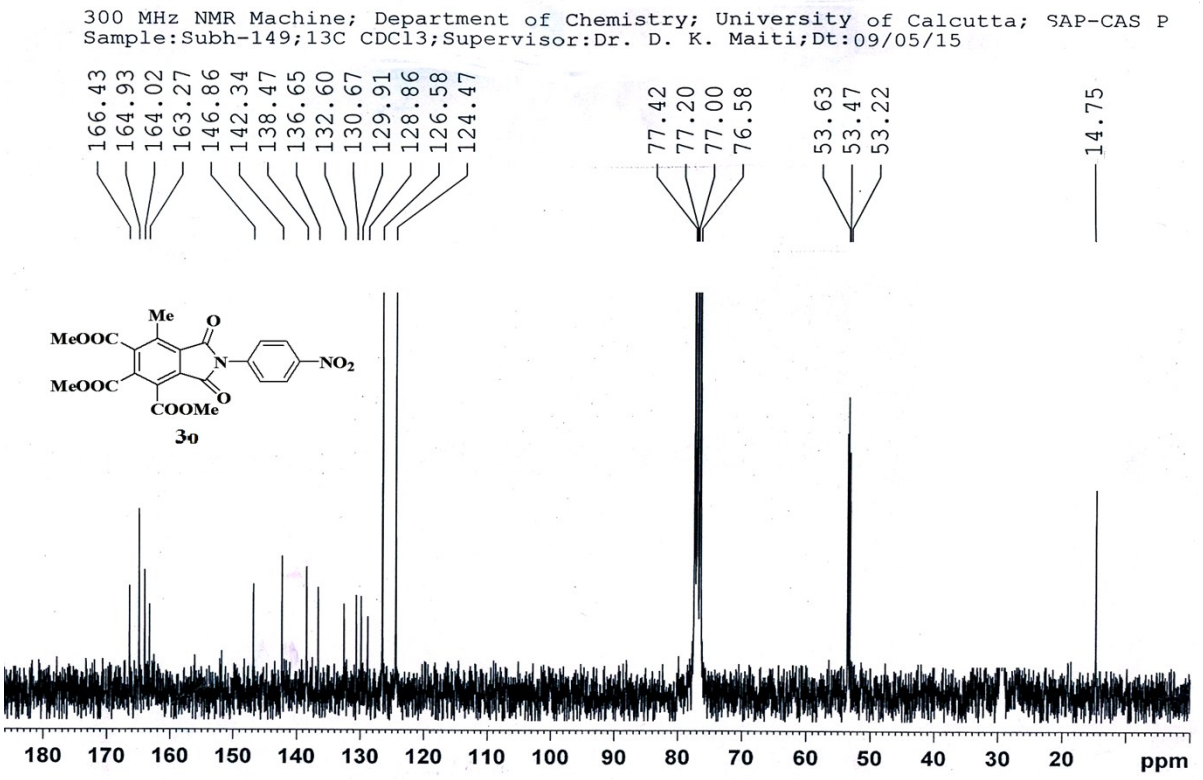
^{1}H & ^{13}C NMR of **3n**



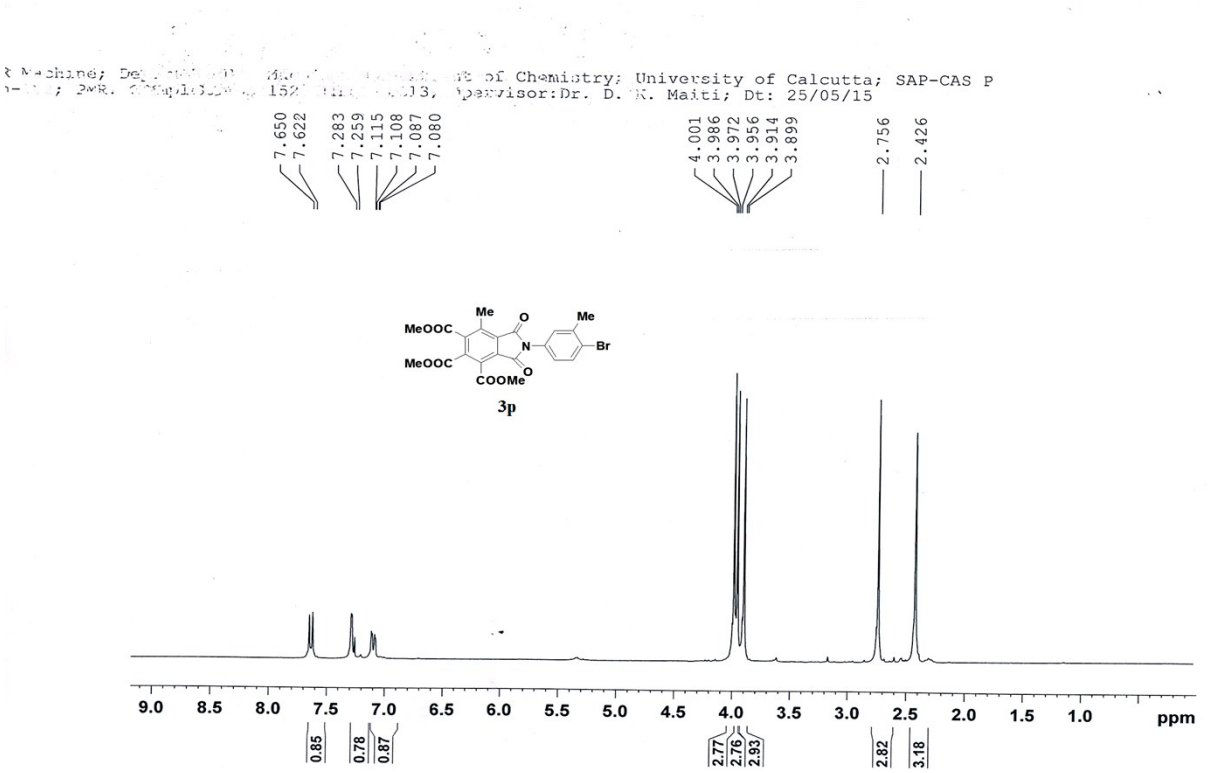


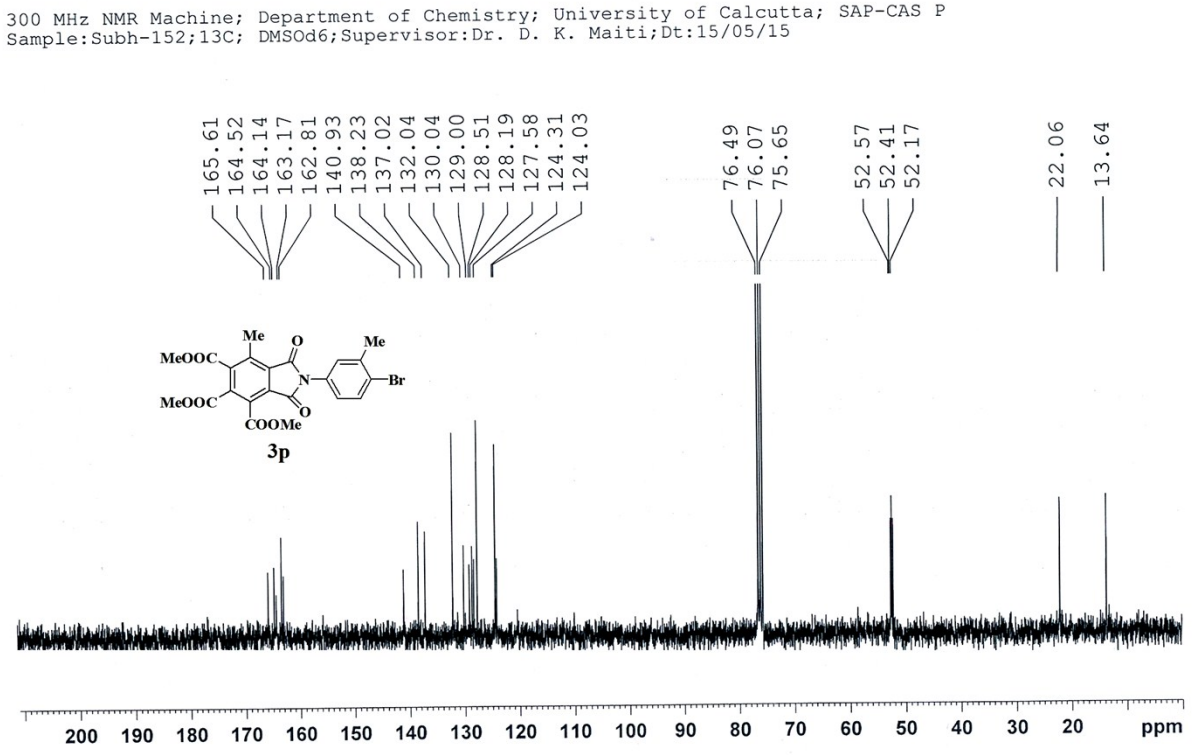
^{1}H & ^{13}C NMR of **3o**



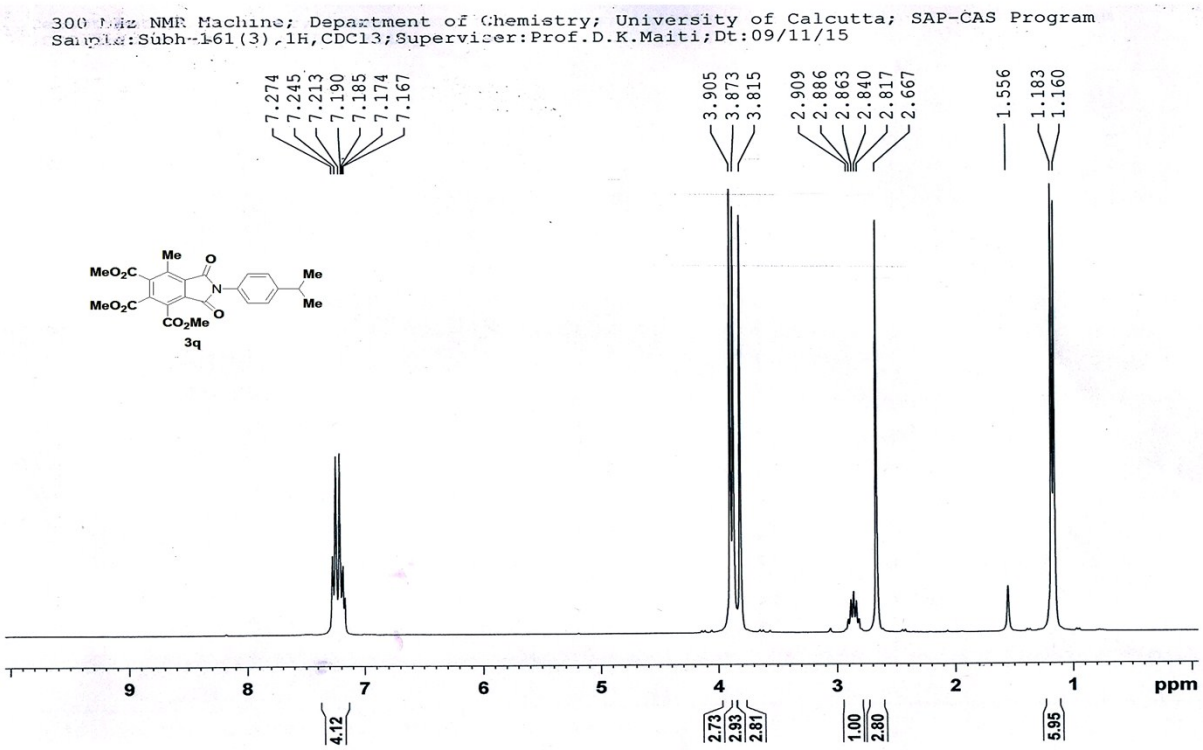


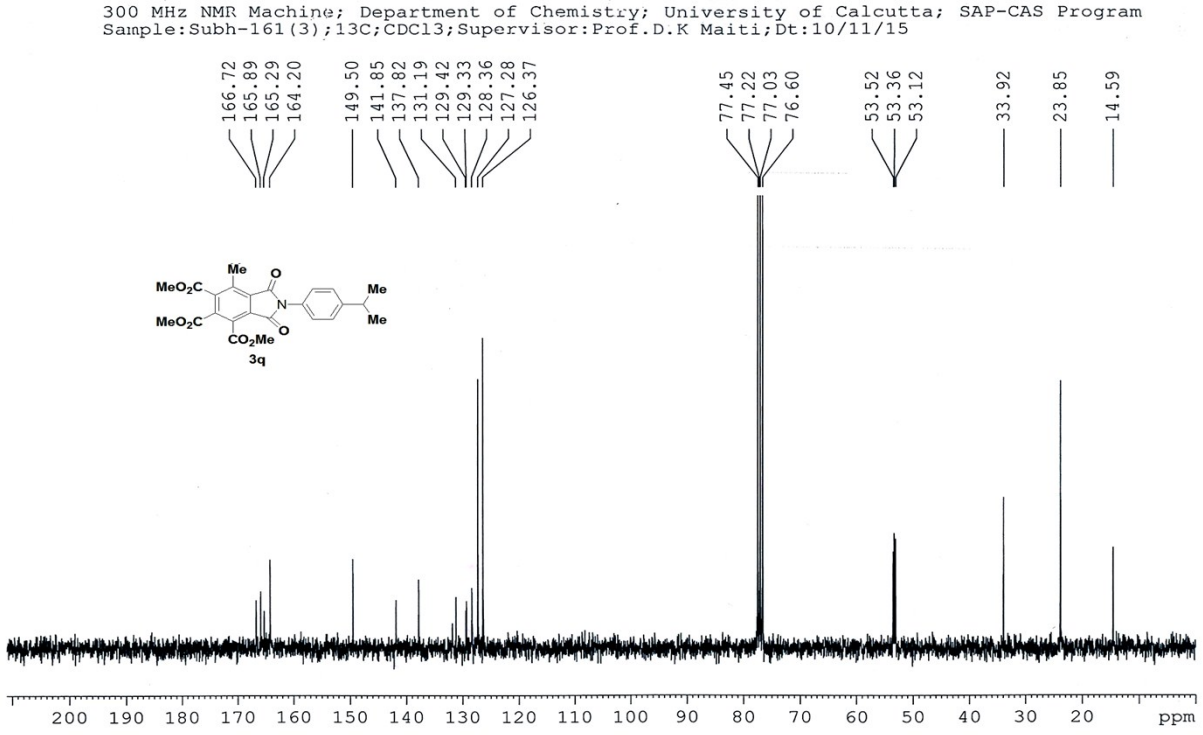
^{1}H & ^{13}C NMR of **3p**



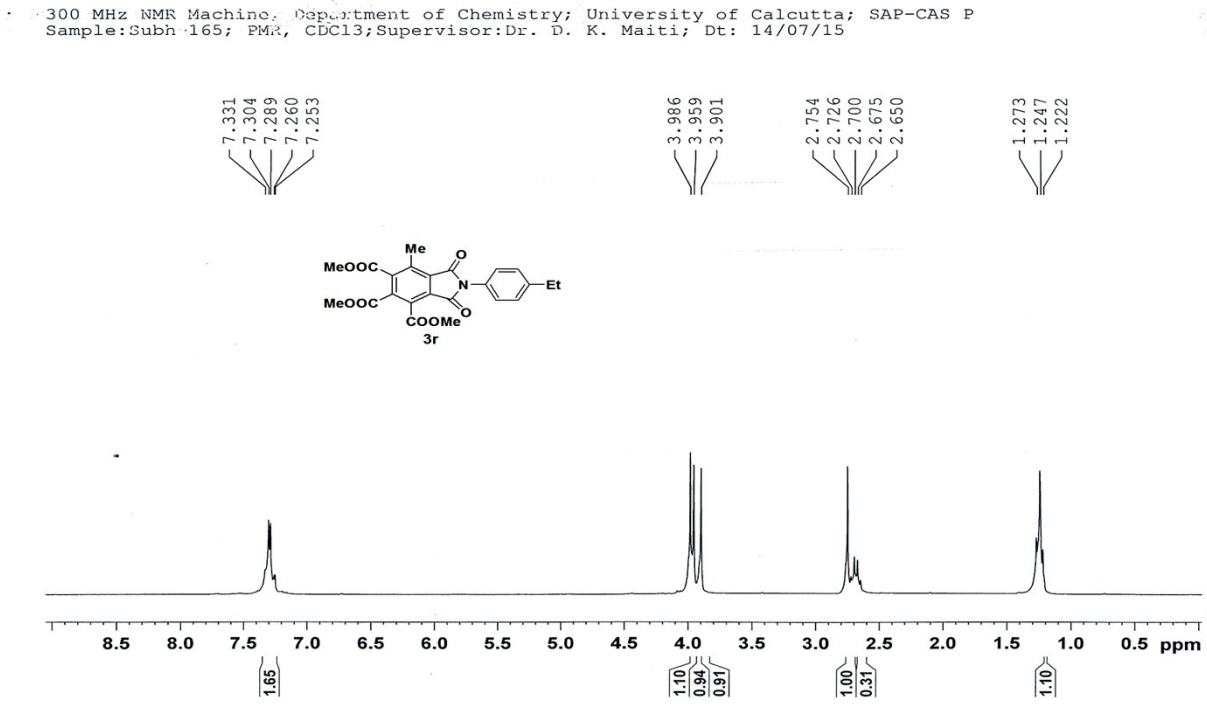


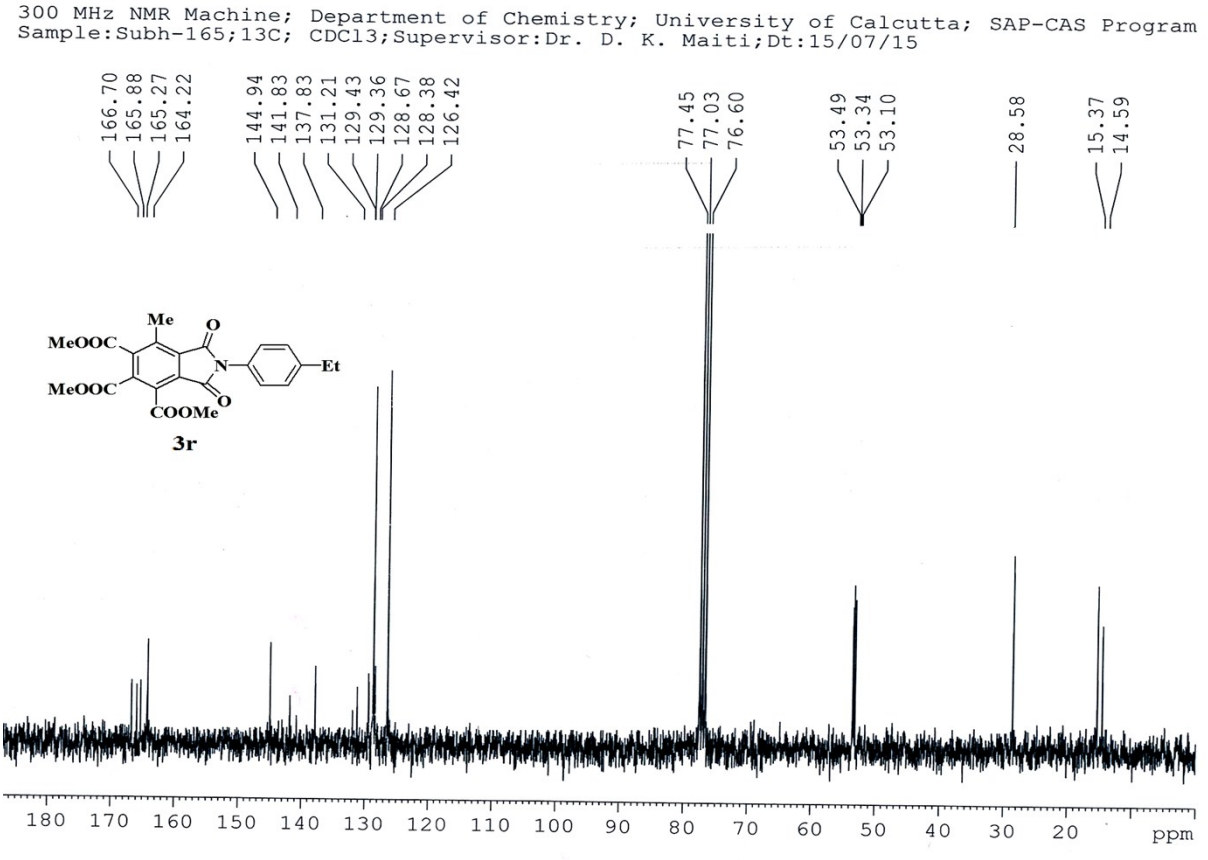
^{1}H & ^{13}C NMR of **3q**



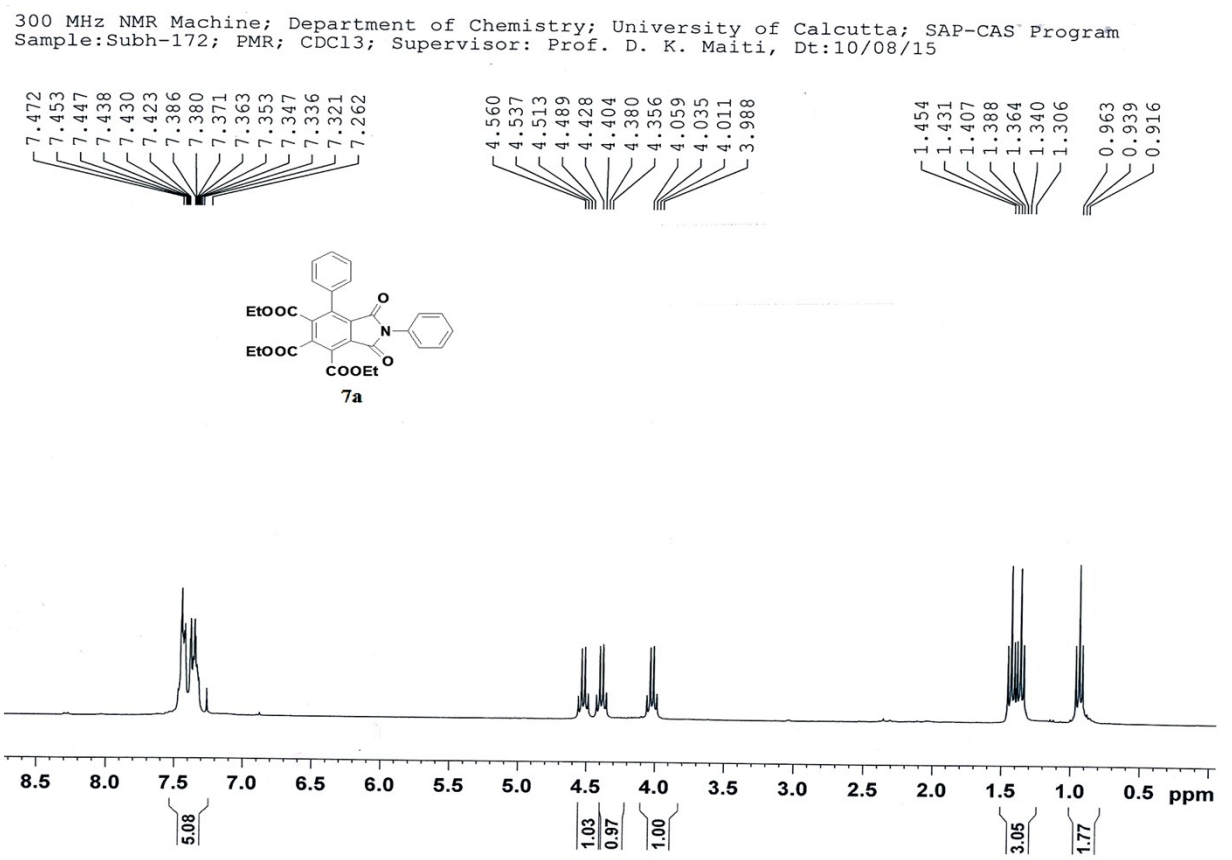


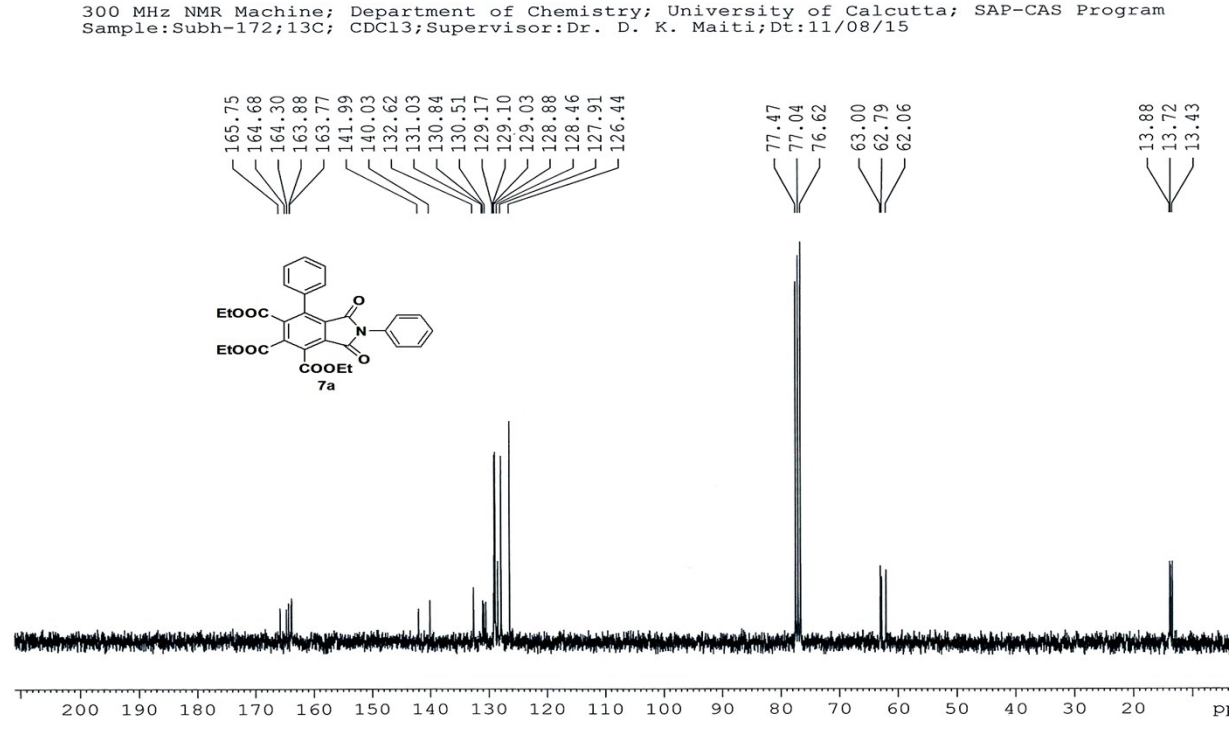
^{1}H & ^{13}C NMR of **3r**



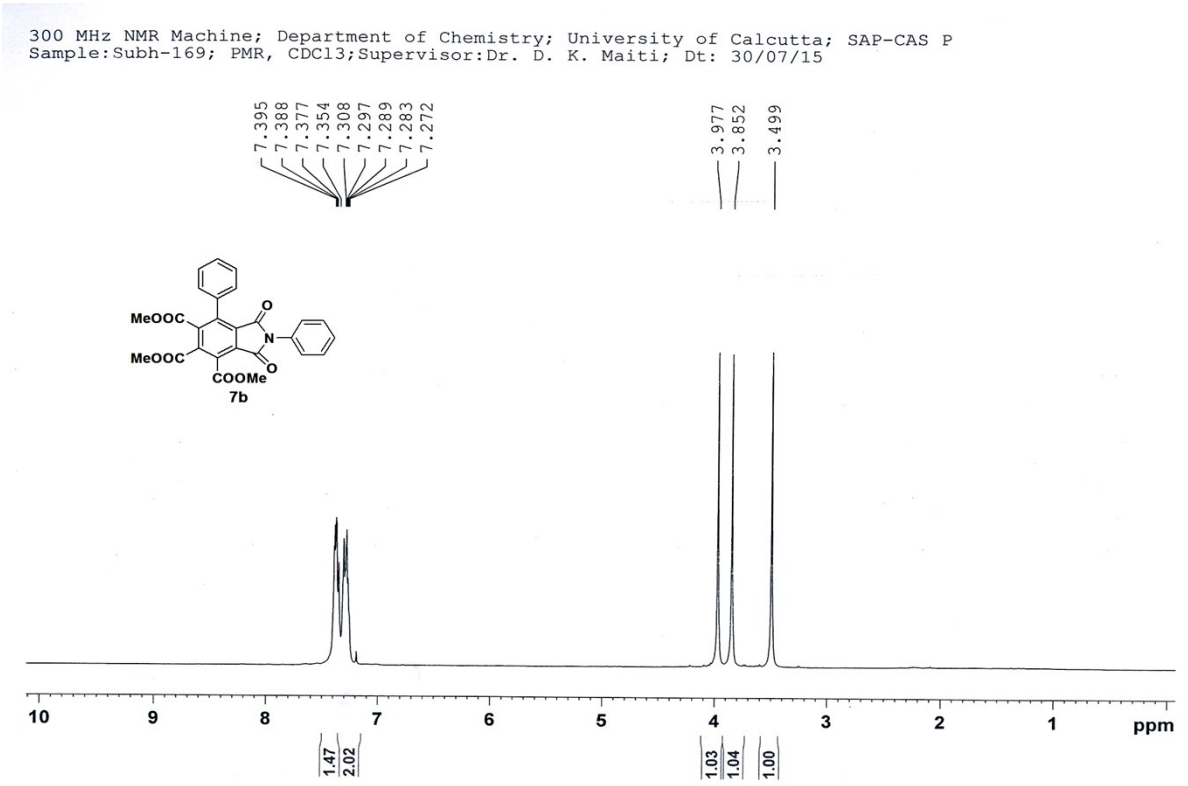


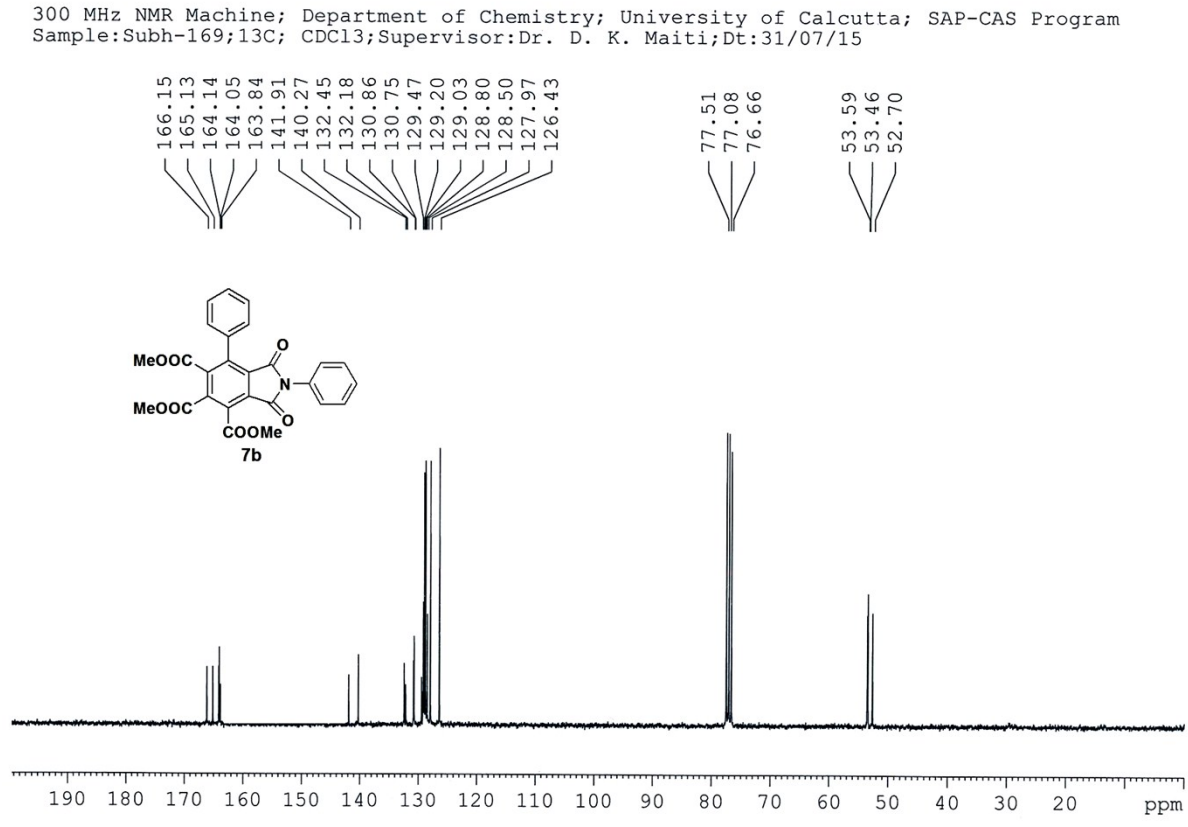
^{1}H & ^{13}C NMR of **7a**



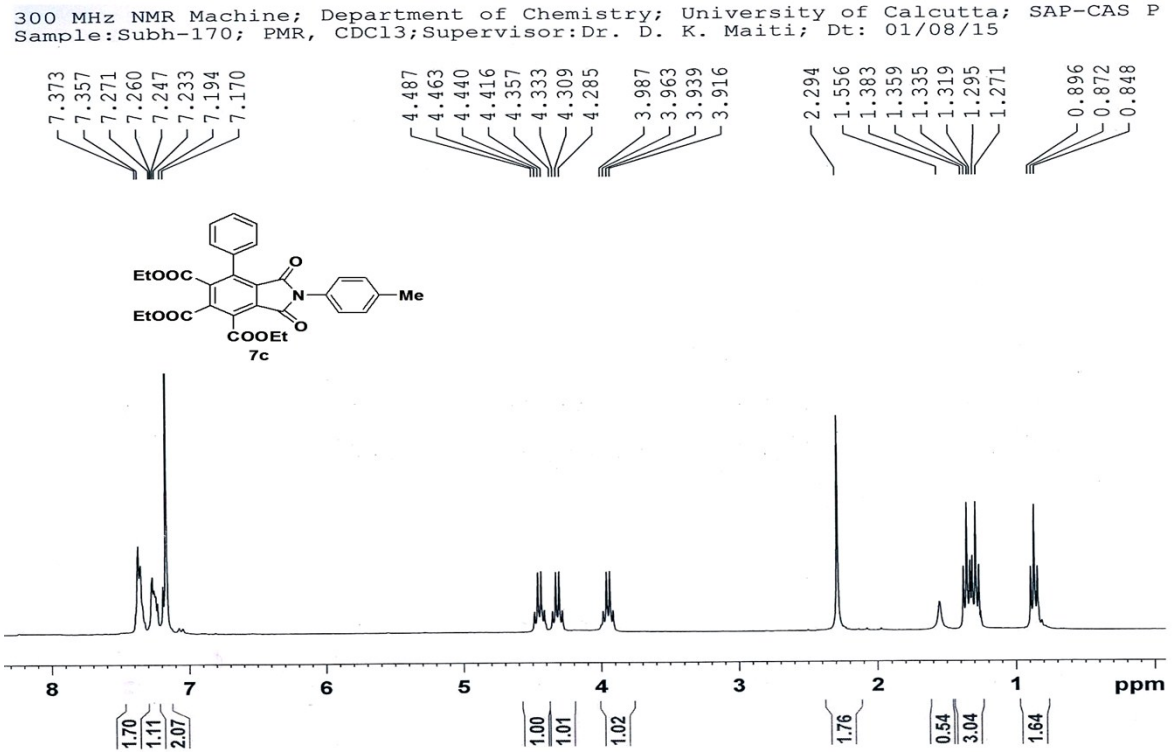


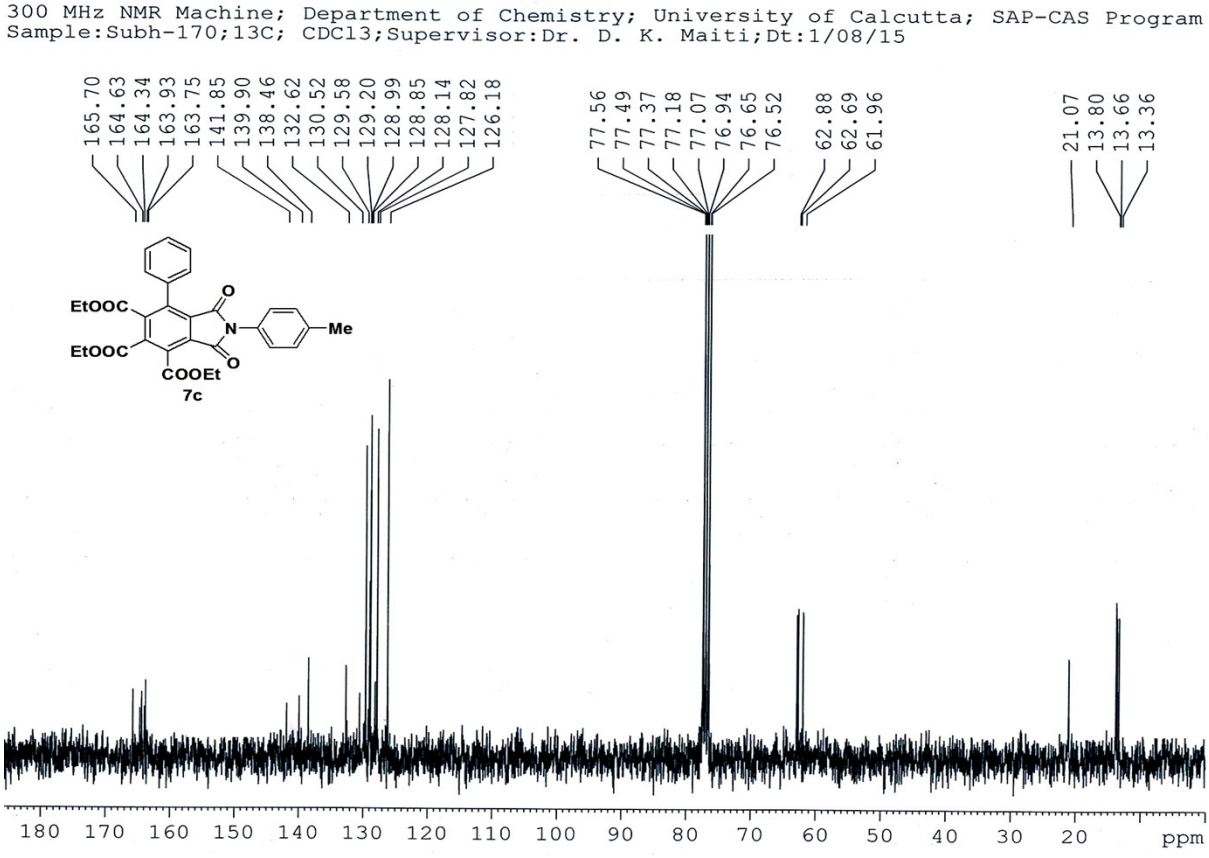
^{1}H & ^{13}C NMR of **7b**



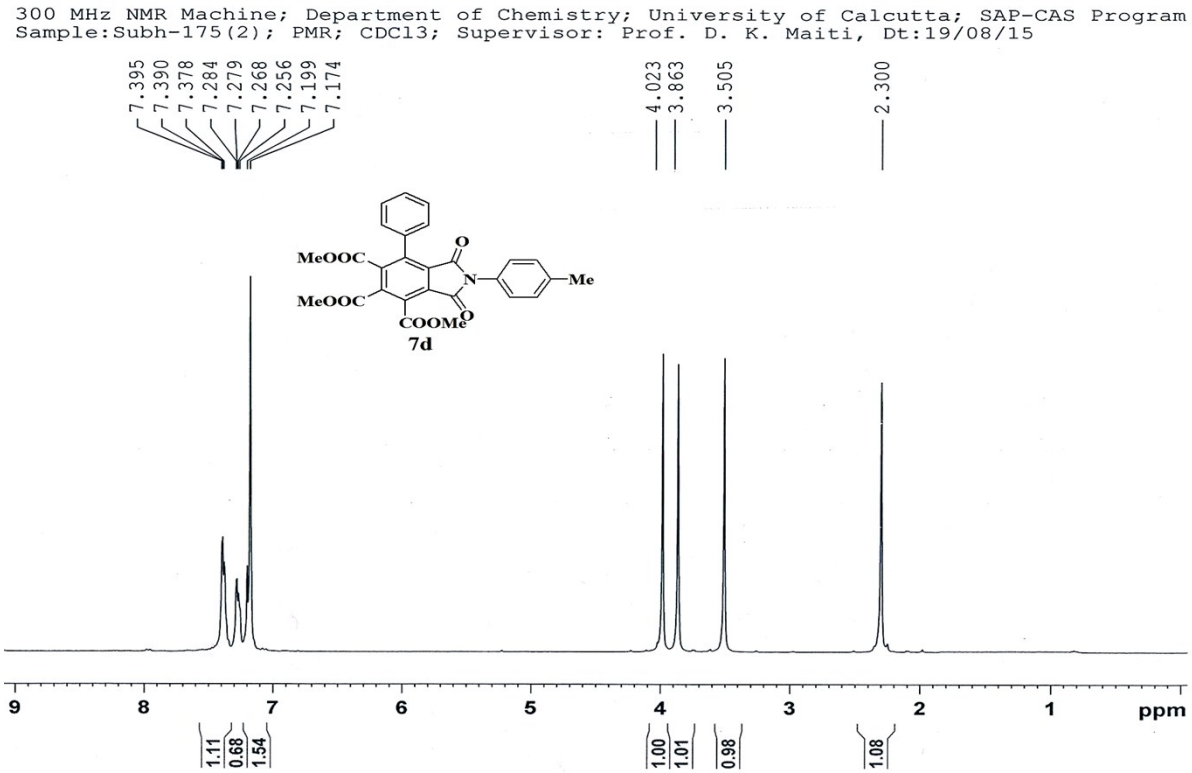


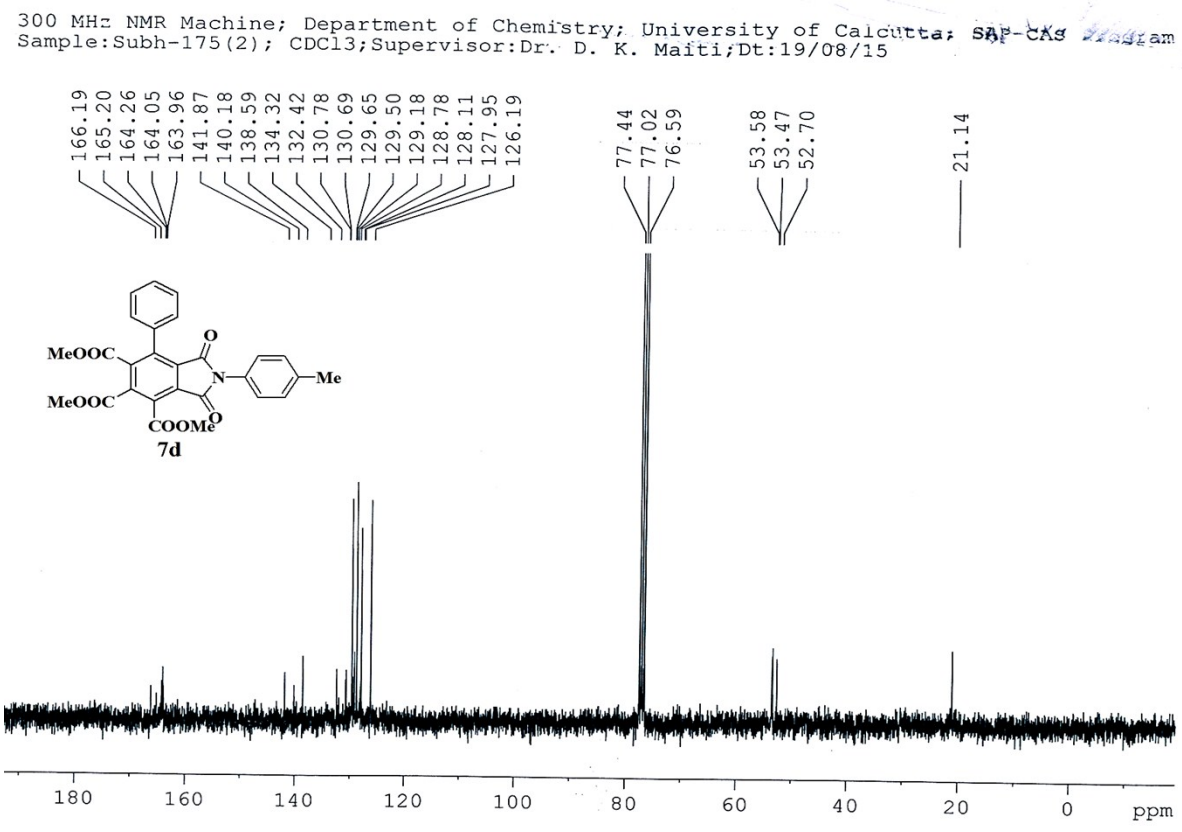
^{1}H & ^{13}C NMR of **7c**





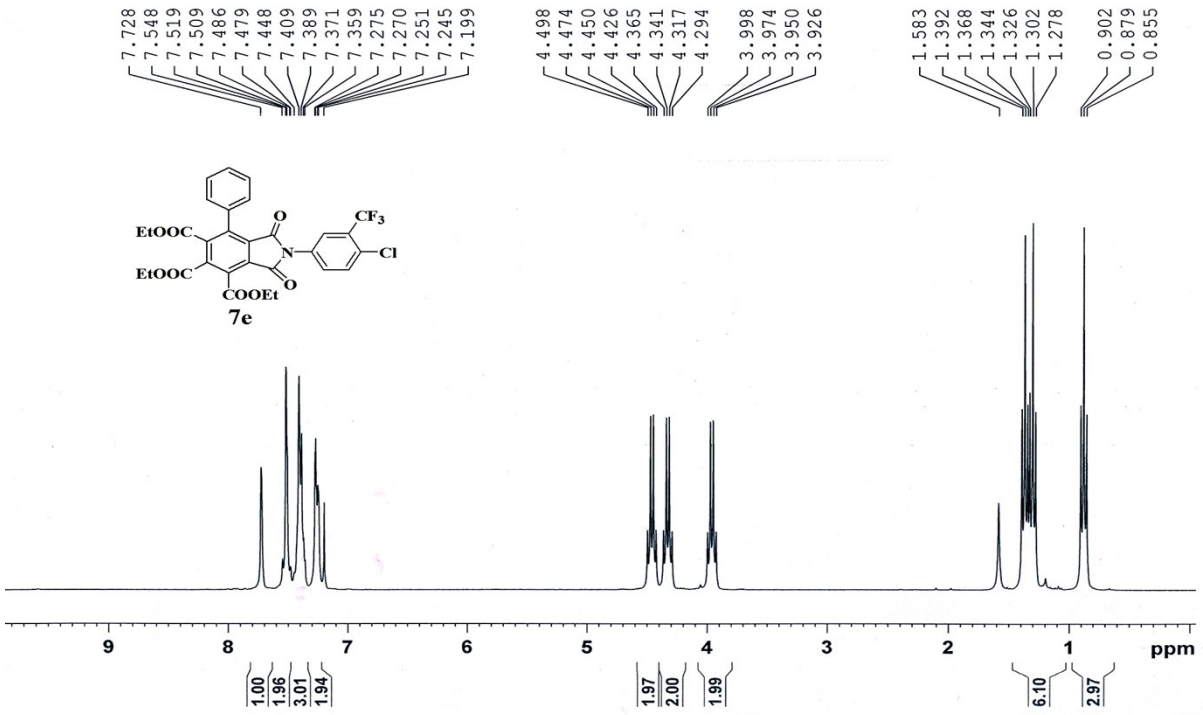
^{1}H & ^{13}C NMR of **7d**



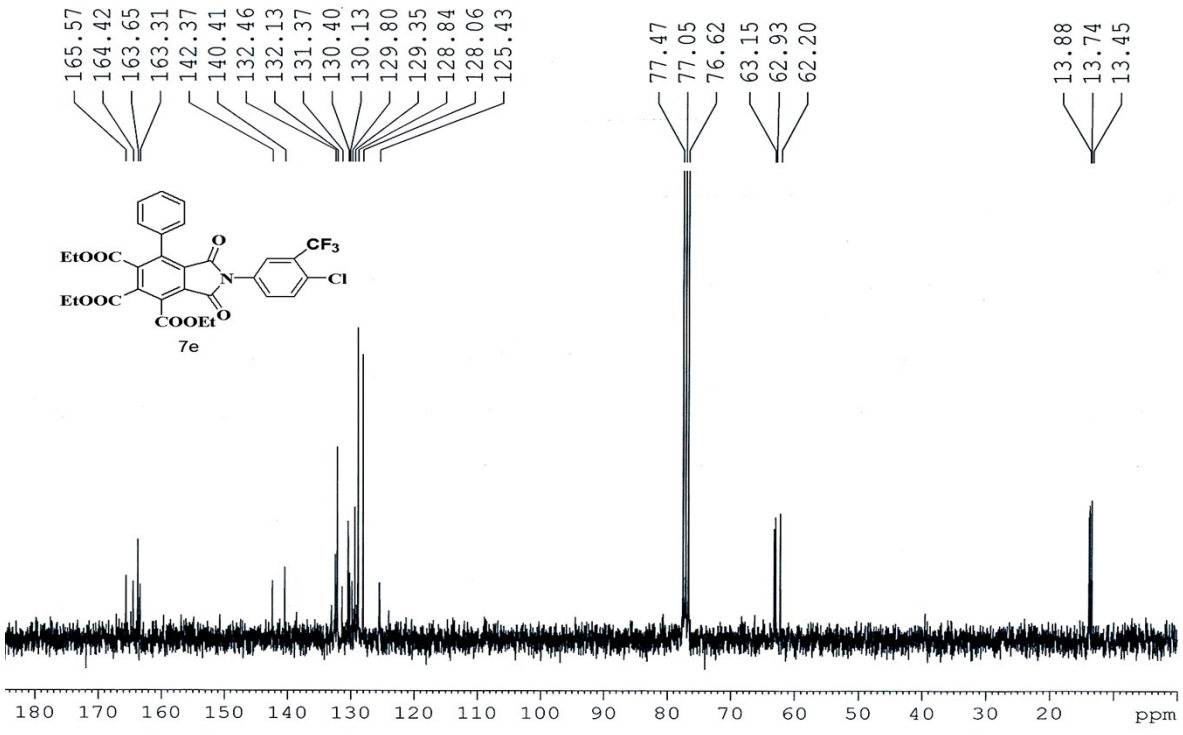


^{1}H & ^{13}C NMR of **7e**

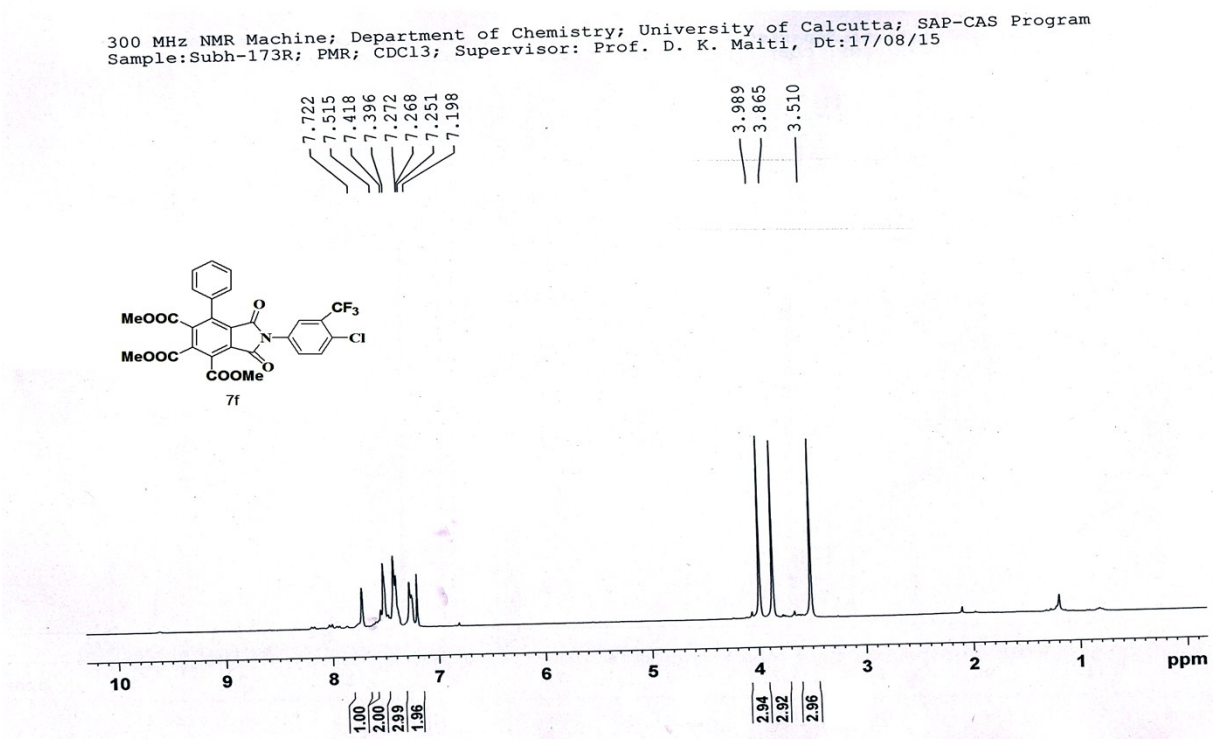
300 MHz NMR Machine; Department of Chemistry; University of Calcutta; SAP-CAS Program
Sample:Subh-174; PMR; CDCl3; Supervisor: Prof. D. K. Maiti, Dt:12/08/15

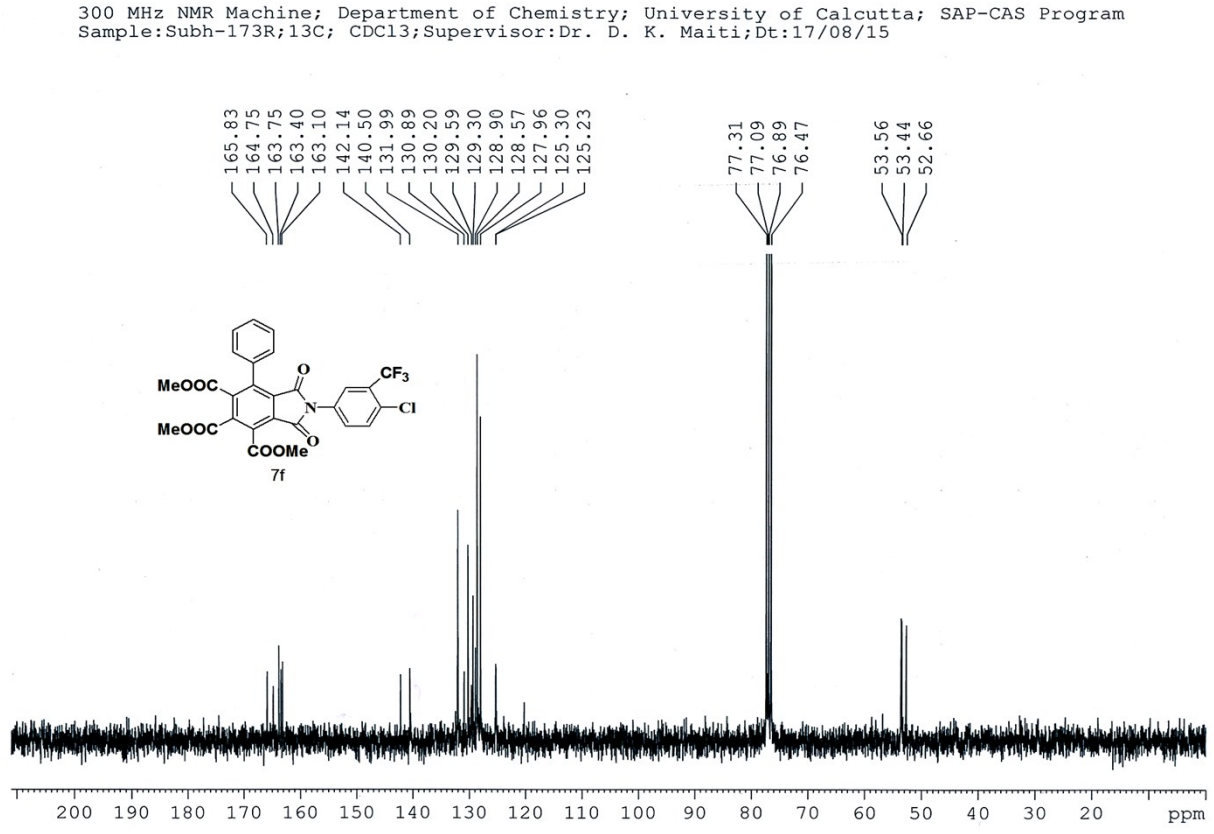


300 MHz NMR Machine; Department of Chemistry; University of Calcutta; SAP-CAS Program
Sample:Subh-174;13C; CDCl3;Supervisor:Dr. D. K. Maiti;Dt:14/08/15

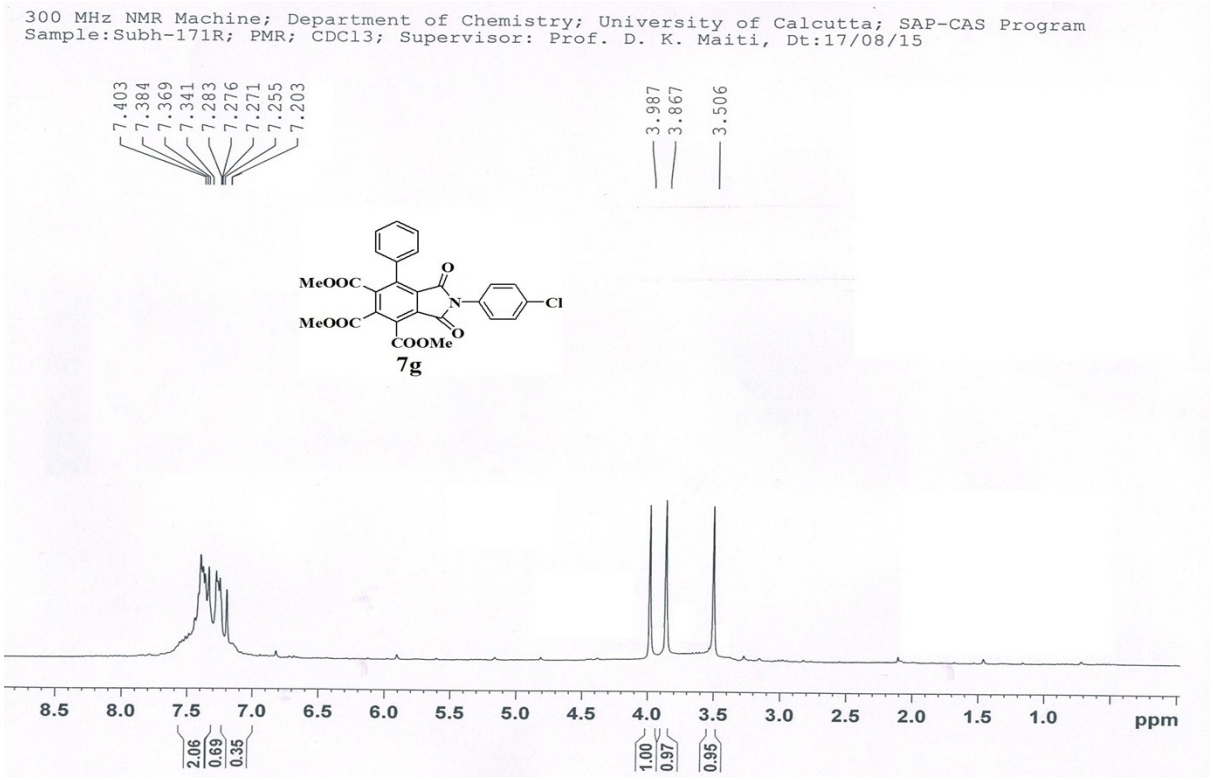


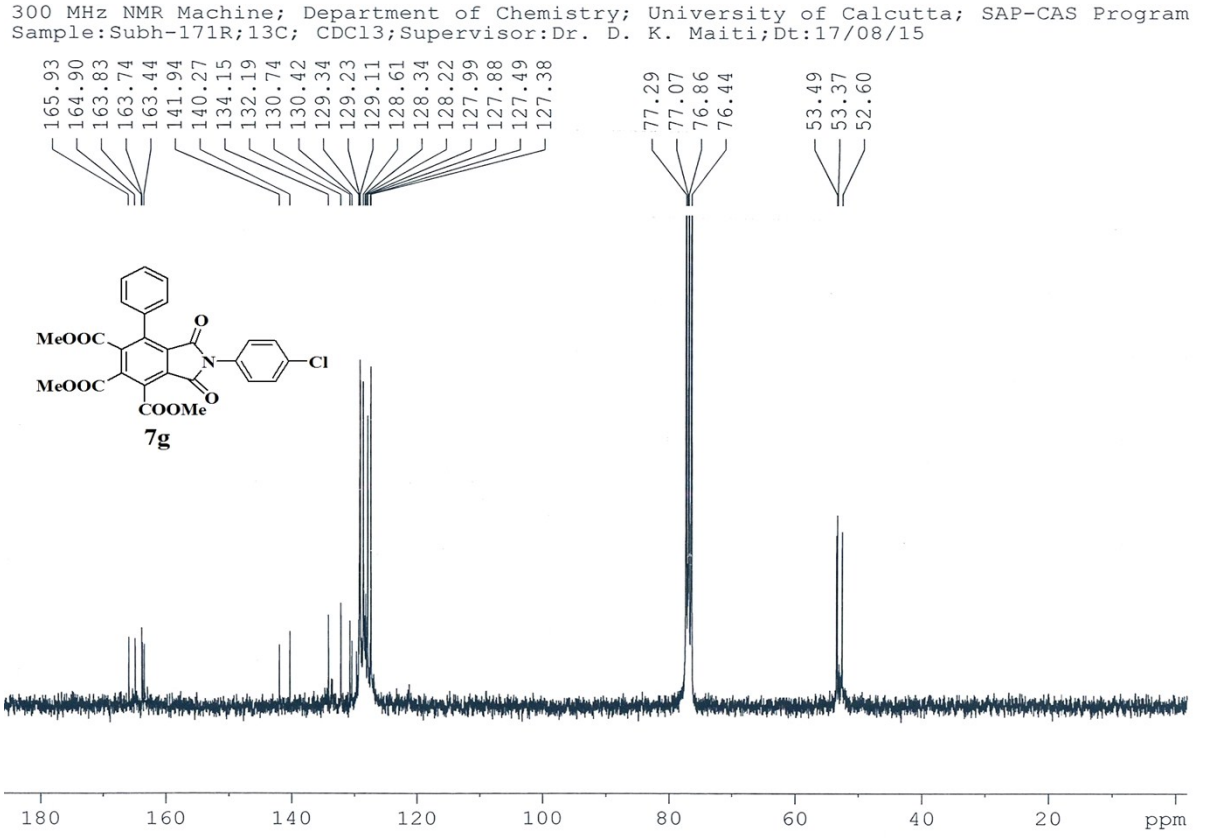
^{1}H & ^{13}C NMR of **7f**



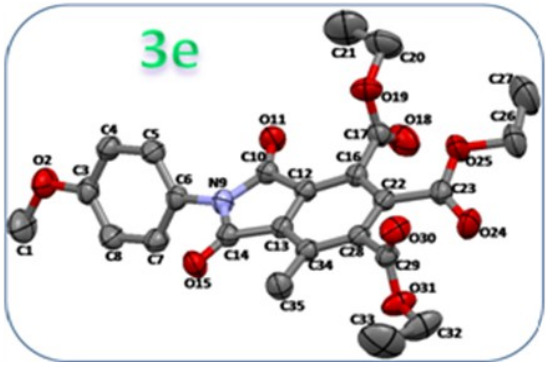


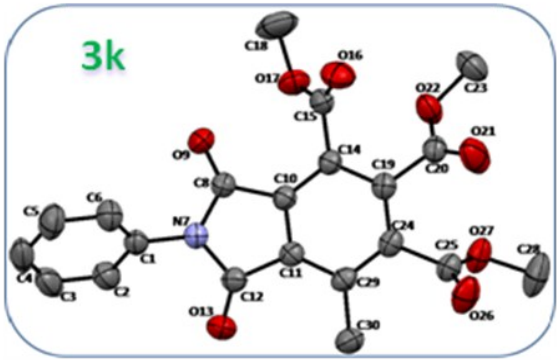
^{1}H & ^{13}C NMR of **7g**





4. Crystallographic data of compound 3e & 3k





Crystal parameters	3e	3k
CCDC No. Empirical formula	1436308 C_{25} H_{25} N O_9	1436272 C_{21} H_{17} N O_8
Formula weight	483.46	411.36
Crystal size/mm	0.92x 0.41 x 0.07	0.32x 0.16 x 0.05
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	*P-1*
a /Å	23.7419(9)	8.133(3)
b/Å	8.0217(3)	11.671(4)
c /Å	31.6881(9)	11.708(3)
$\alpha/^0$	90.00	63.061(14)
$\beta/^0$	127.152(2)	84.432(14)
$\gamma/^0$	90.00	75.914(14)
Volume/Å^3	4810.1(3)	960.9(5)
Z	8	2
D_{calc} /gcm^{-3}	1.335	1.422
F(000)	2032	428
μ MoKα /mm^{-1}	0.102	0.111
Temperature/K	298(2)	298(2)
R_{int}	0.0367	0.0248
Range of h, k, l	-26/28,-9/9, -37/36	-9/9, -13/13, -13/13
θ min/max/°	1.08/25.00	1.95/25.00
Reflections collected/unique/observed[I>2σ(I)]	59544 / 8471/ 6526	8531 / 3361/ 2729
Data/restraints/ parameters	8471/0/641	3361/0/275
Goodness of fit on F^2	1.024	1.026
Final R indices [I>2σ(I)]	R_1 = 0.0506, wR_2 = 0.1259	R_1 = 0.0506, 0.0405, wR_2 = 0.1108
R indices (all data)	R_1 = 0.0671, wR_2 = 0.1408	R_1 = 0.0671, 0.0498, wR_2 = 0.1200

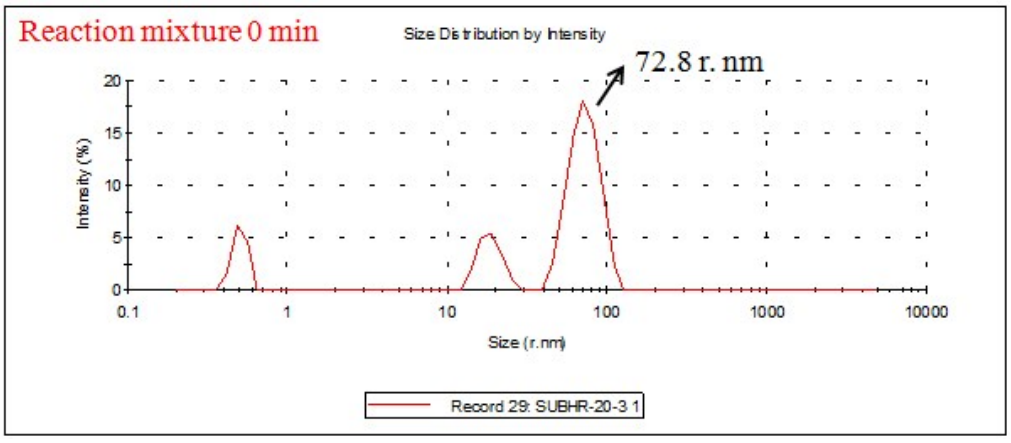
5. DLS study of the reaction in water medium

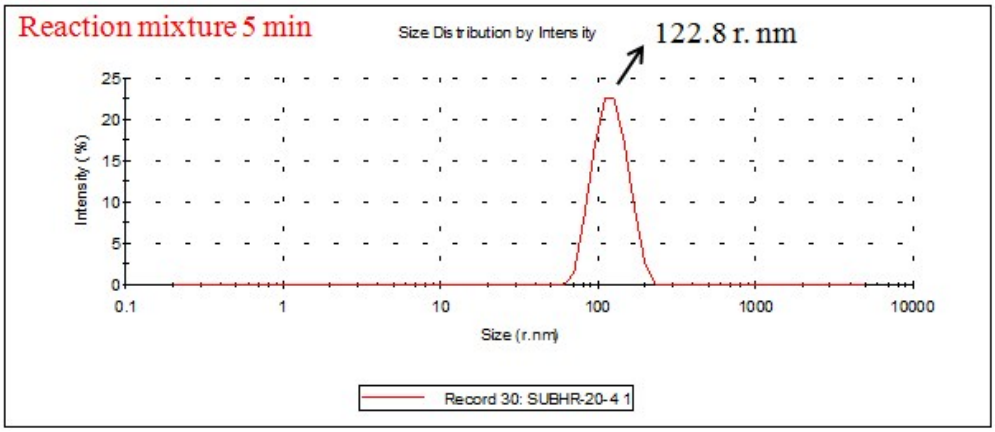
Above the CMC concentration, suitable surfactants can generate an organized media in water. Inside of the surfactant-assembled spherical micelle is sufficiently lipophilic in nature and makes both organic substrates and reagents soluble. It was confirmed by DLS (Dynamic Light Scattering) experiment of the solutions contain surfactant and solvent as well as the reaction mixture and the details result is given in the Table S1.

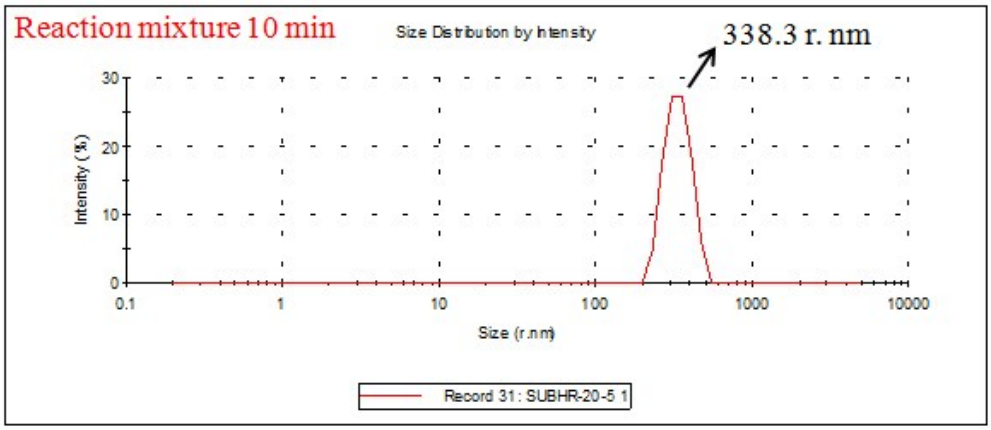
Table S1. DLS data of the reaction mixture in presence of DMAP

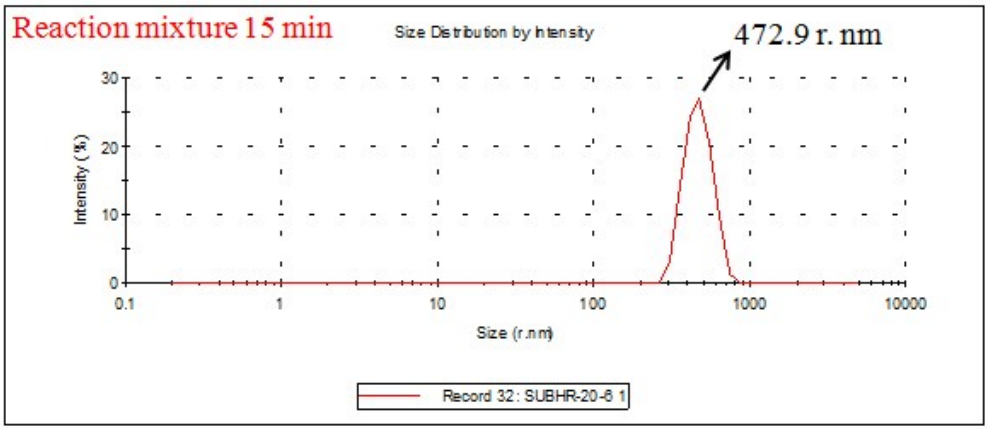
Composition	Time (min)	Size (r.nm)	Width (r.nm)
Reaction mixture	0	72.8	15.2
Reaction mixture	5	122.8	28.3
Reaction mixture	10	338.3	62.9
Reaction mixture	15	472.9	92.9
Reaction mixture	20	599.8	117.4
Reaction mixture	25	700.0	171.2
Reaction mixture	30	945.0	449.3

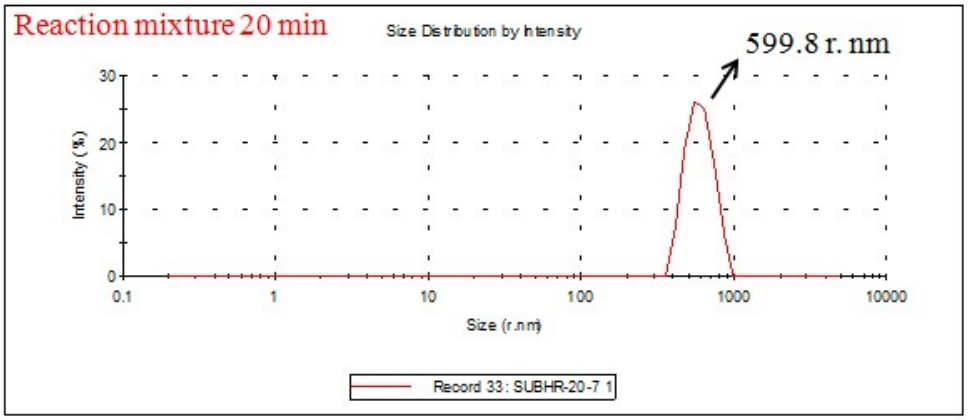
DLS data with DMAP

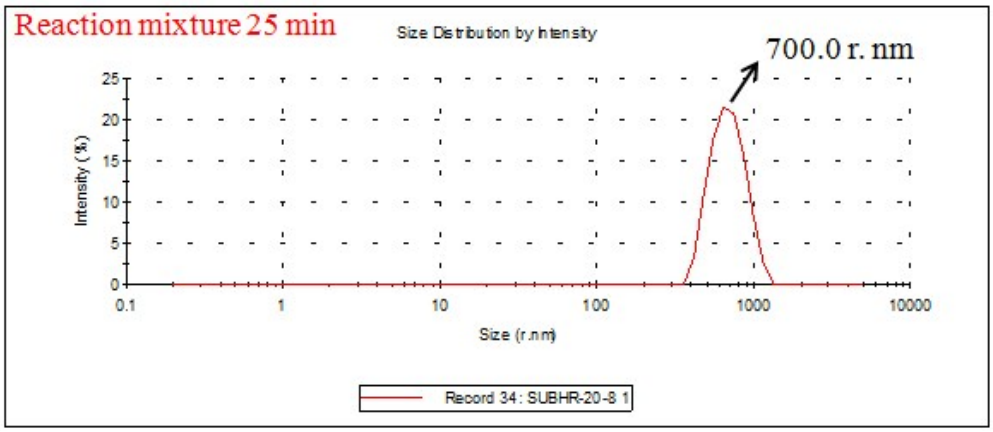



Reaction mixture 5 min
Size Distribution by Intensity
122.8 r. nm
Intensity (%)
Size (r.nm)
Record 30: SUBHR-20-4 1


Reaction mixture 10 min
Size Distribution by Intensity
338.3 r. nm
Intensity (%)
Size (r.nm)
Record 31: SUBHR-20-5 1


Reaction mixture 15 min
Size Distribution by Intensity
472.9 r. nm
Intensity (%)
Size (r.nm)
Record 32: SUBHR-20-6 1


Reaction mixture 20 min
Size Distribution by Intensity
599.8 r. nm
Intensity (%)
Size (r.nm)
Record 33: SUBHR-20-7 1



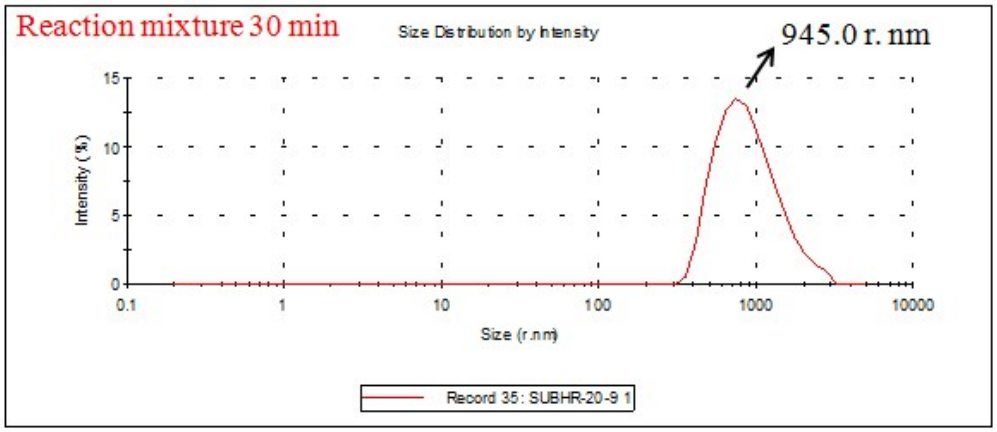
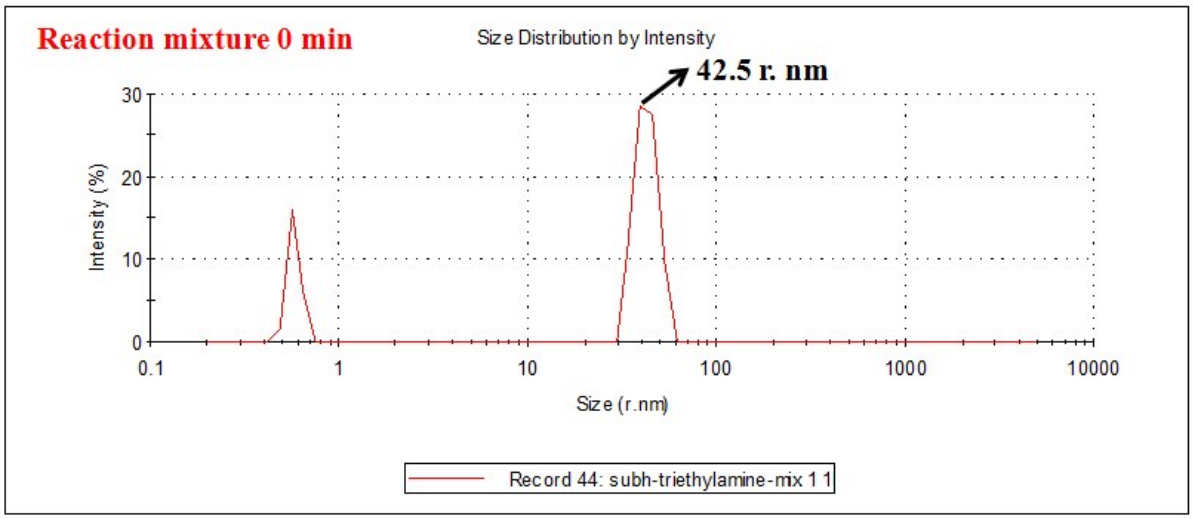
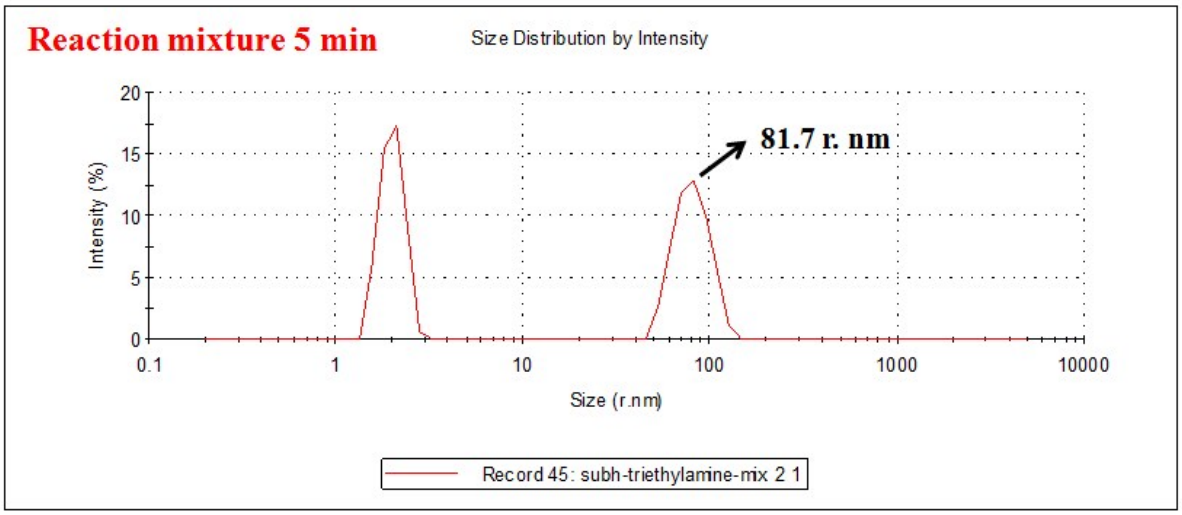
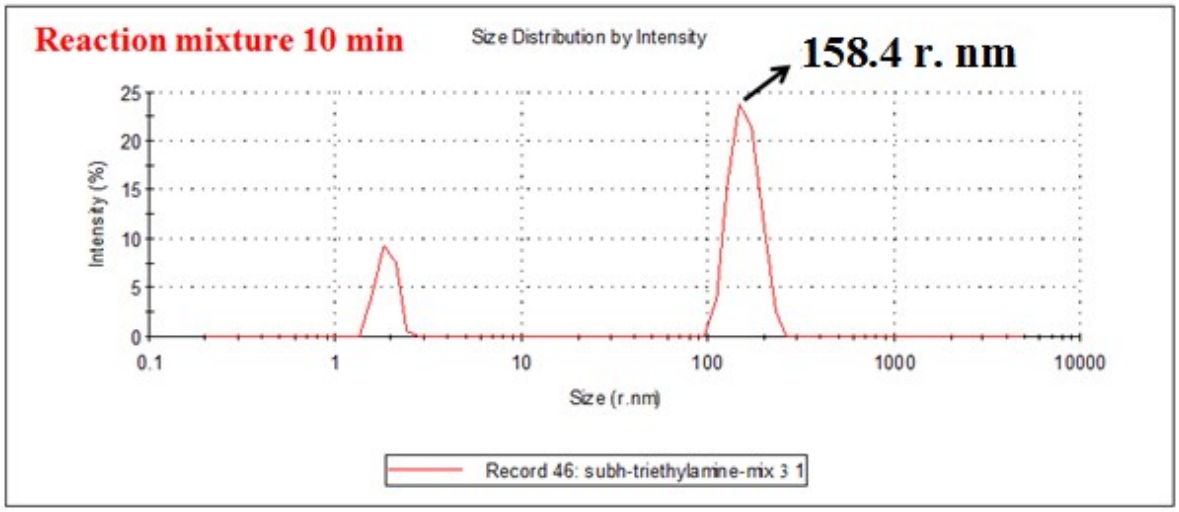


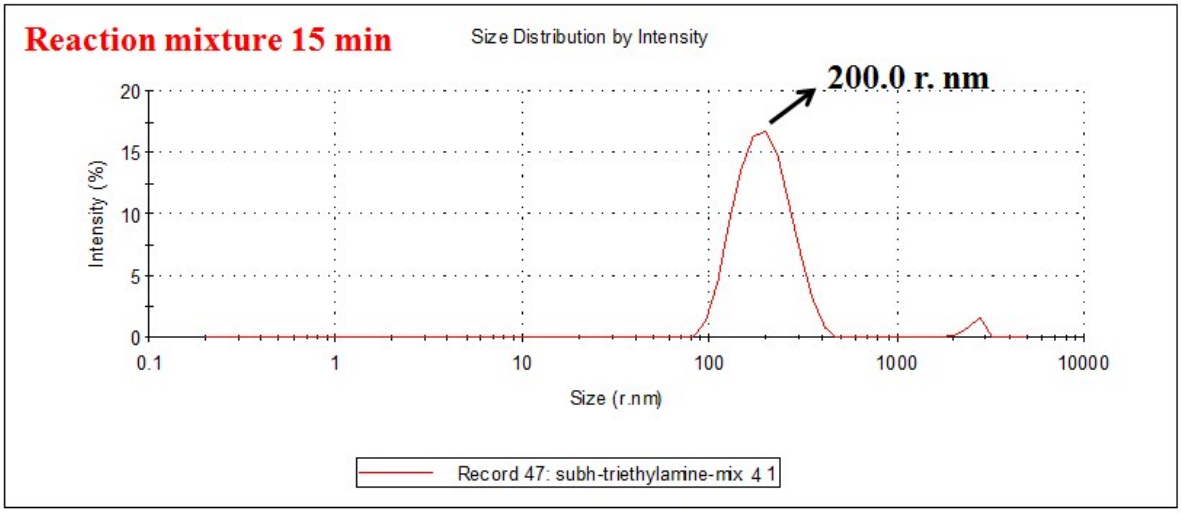
Table S2. DLS data of the reaction mixture in presence of NEt_3

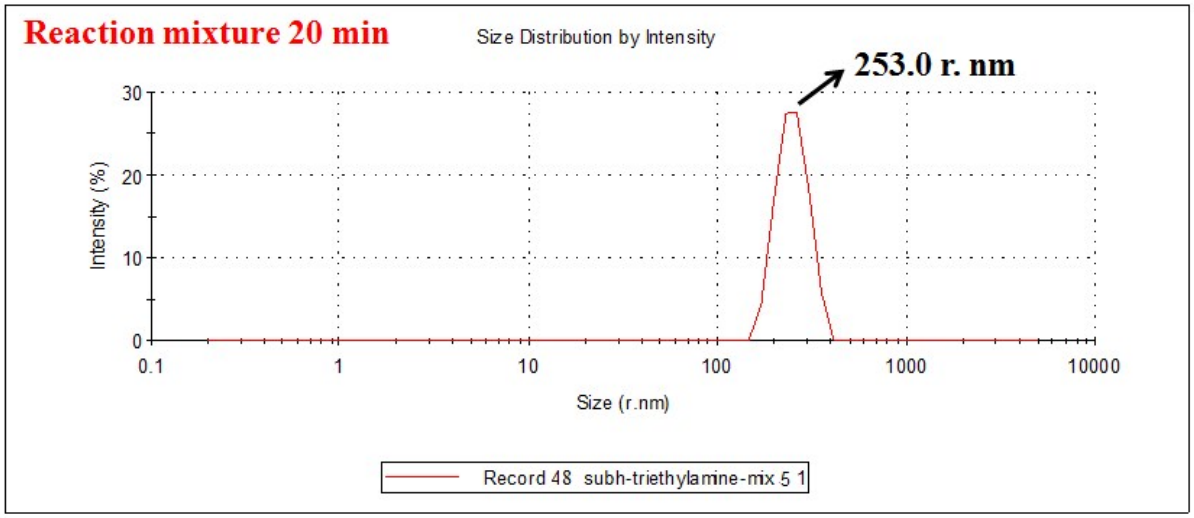
Composition	Time (min)	Size (r.nm)	Width (r.nm)
Reaction mixture	0	42.5	5.5
Reaction mixture	5	81.7	17.7
Reaction mixture	10	158.4	27.7
Reaction mixture	15	200.0	33.2
Reaction mixture	20	253.0	46.3
Reaction mixture	25	337.8	54.2
Reaction mixture	30	309.4	91.4
Reaction mixture	35	157.8	50.6

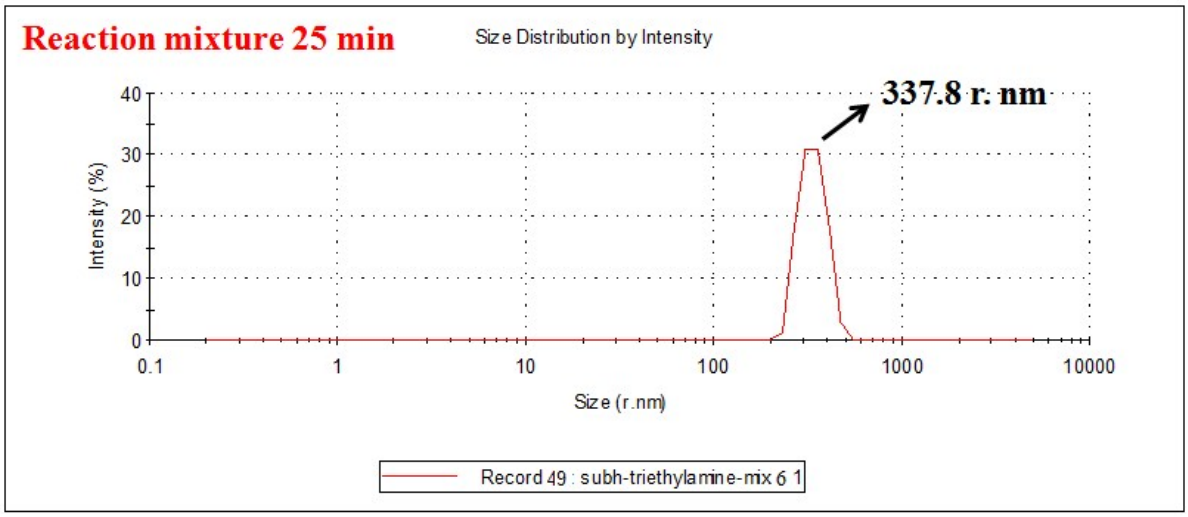

Reaction mixture 0 min
Size Distribution by Intensity
42.5 r. nm
Intensity (%)
Size (r.nm)
Record 44: subh-triethylamine-mix 1 1

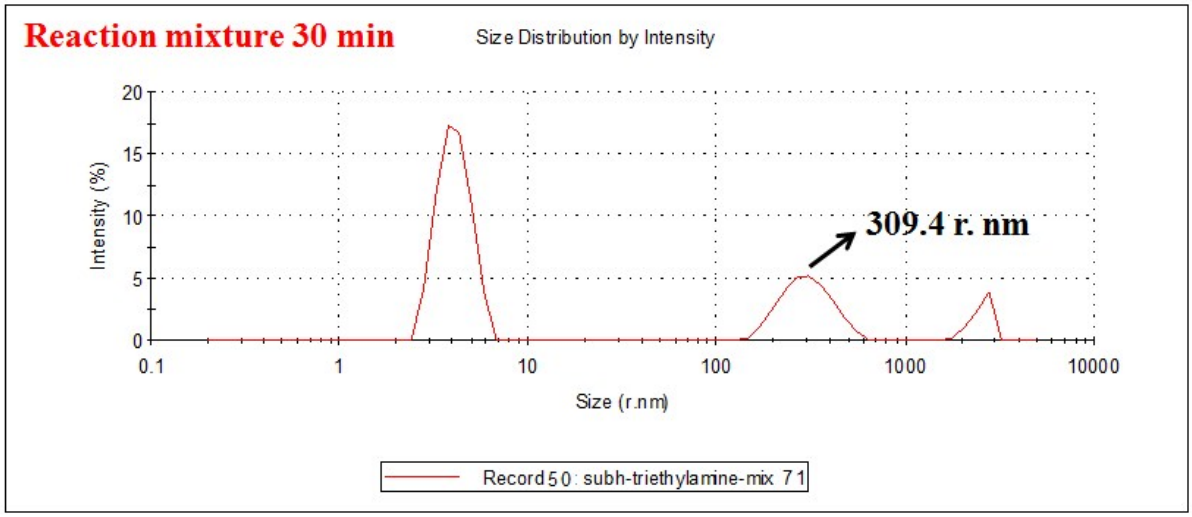

Reaction mixture 5 min
Size Distribution by Intensity
81.7 r. nm
Intensity (%)
Size (r.nm)
Record 45: subh-triethylamine-mix 2 1


Reaction mixture 10 min
Size Distribution by Intensity
158.4 r. nm
Intensity (%)
Size (r.nm)
Record 46: subh-triethylamine-mix 3 1


Reaction mixture 15 min
Size Distribution by Intensity
200.0 r. nm
Intensity (%)
Size (r.nm)
Record 47: subh-triethylamine-mix 4 1


Reaction mixture 20 min
Size Distribution by Intensity
253.0 r. nm
Intensity (%)
Size (r.nm)
Record 48 subh-triethylamine-mix 5 1


Reaction mixture 25 min
Size Distribution by Intensity
337.8 r. nm
Intensity (%)
Size (r.nm)
Record 49 : subh-triethylamine-mix 6 1


Reaction mixture 30 min
Size Distribution by Intensity
309.4 r. nm
Intensity (%)
Size (r.nm)
Record 50 : subh-triethylamine-mix 7 1

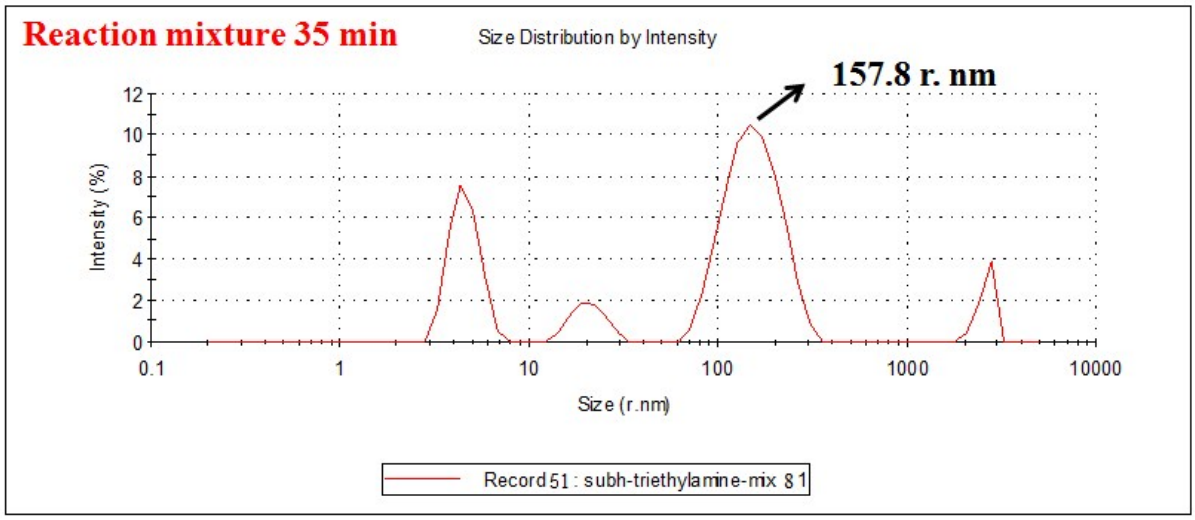
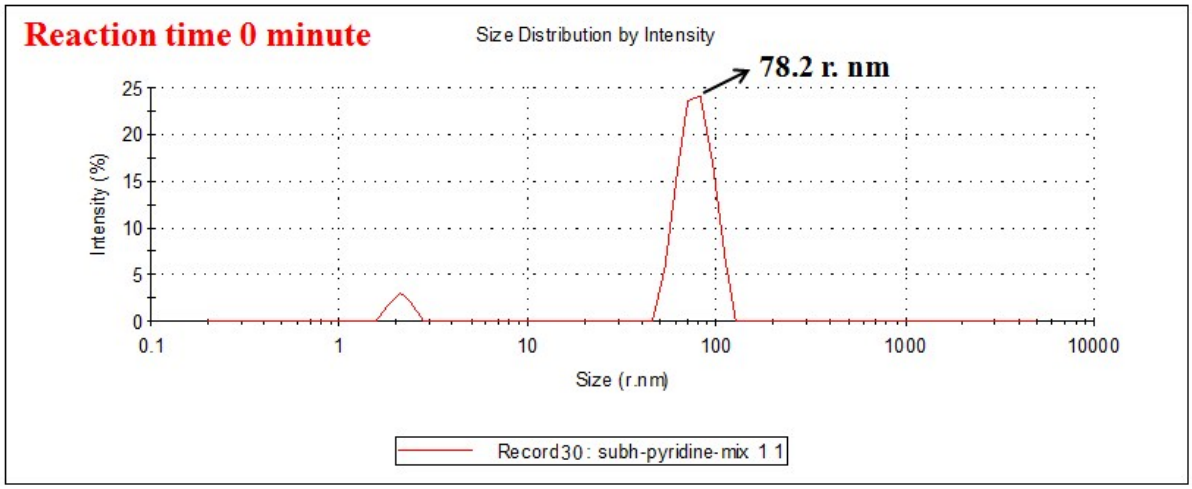
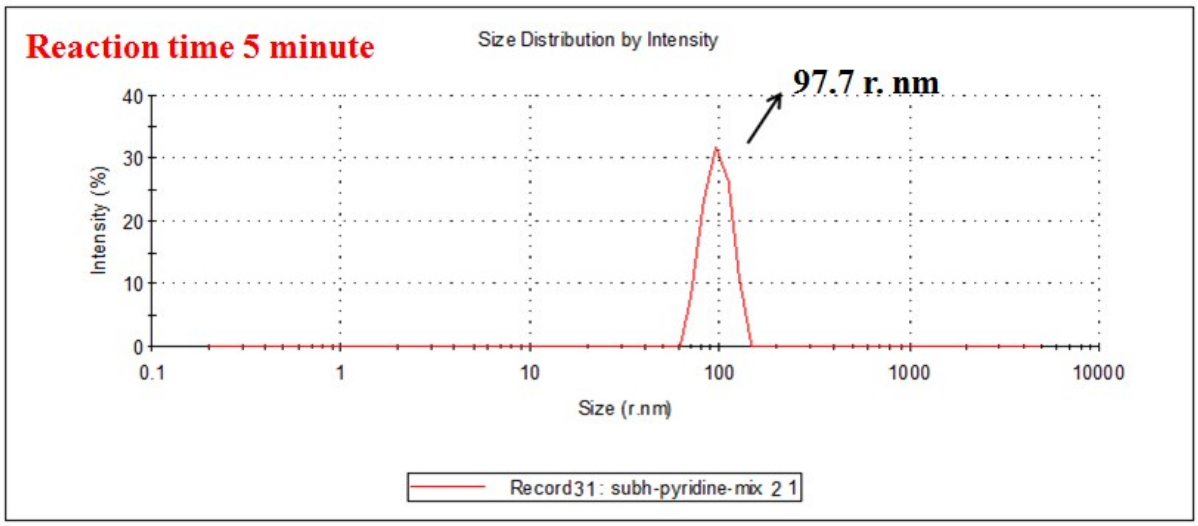
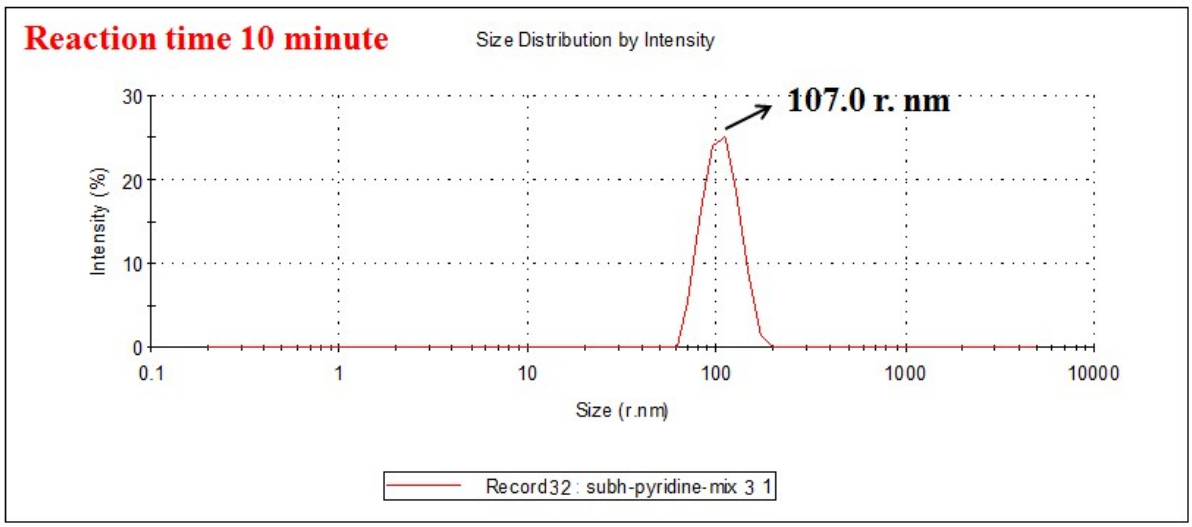

Reaction mixture 35 min
Size Distribution by Intensity
157.8 r. nm
Intensity (%)
Size (r.nm)
Record 51 : subh-triethylamine-mix 8 1

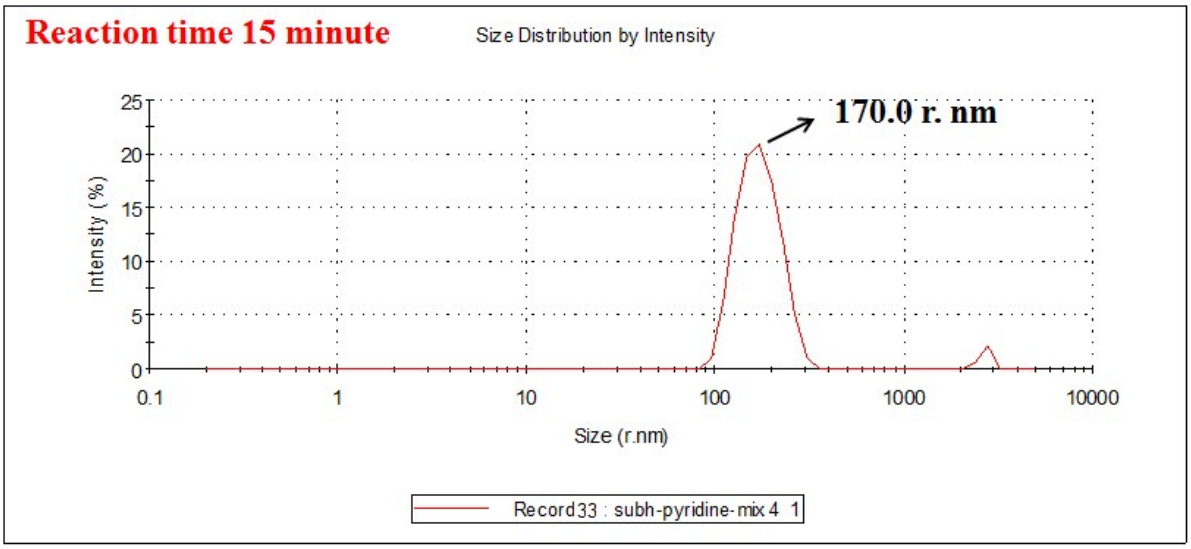
Table S3. DLS data of the reaction mixture in presence of pyridine

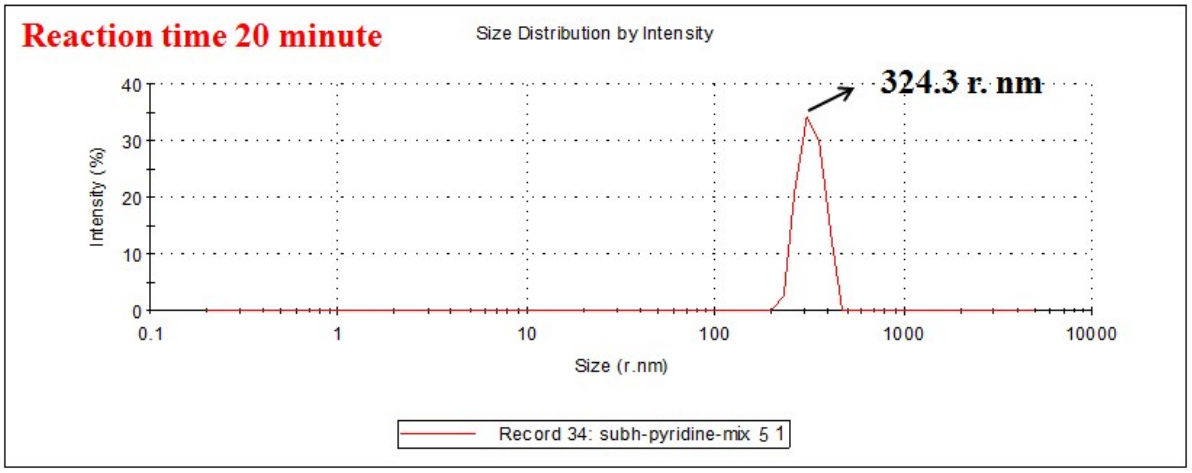
Composition	Time (min)	Size (r.nm)	Width (r.nm)
Reaction mixture	0	78.2	15.3
Reaction mixture	5	97.8	15.9
Reaction mixture	10	107.0	22.3
Reaction mixture	15	170.0	43.5
Reaction mixture	20	324.3	45.1
Reaction mixture	25	344.0	48.2
Reaction mixture	30	362.6	48.7
Reaction mixture	35	405.2	63.0
Reaction mixture	40	439.9	70.8
Reaction mixture	45	491.6	77.4
Reaction mixture	50	624.8	126.2
Reaction mixture	55	678.5	146.2
Reaction mixture	60	806.4	194.6

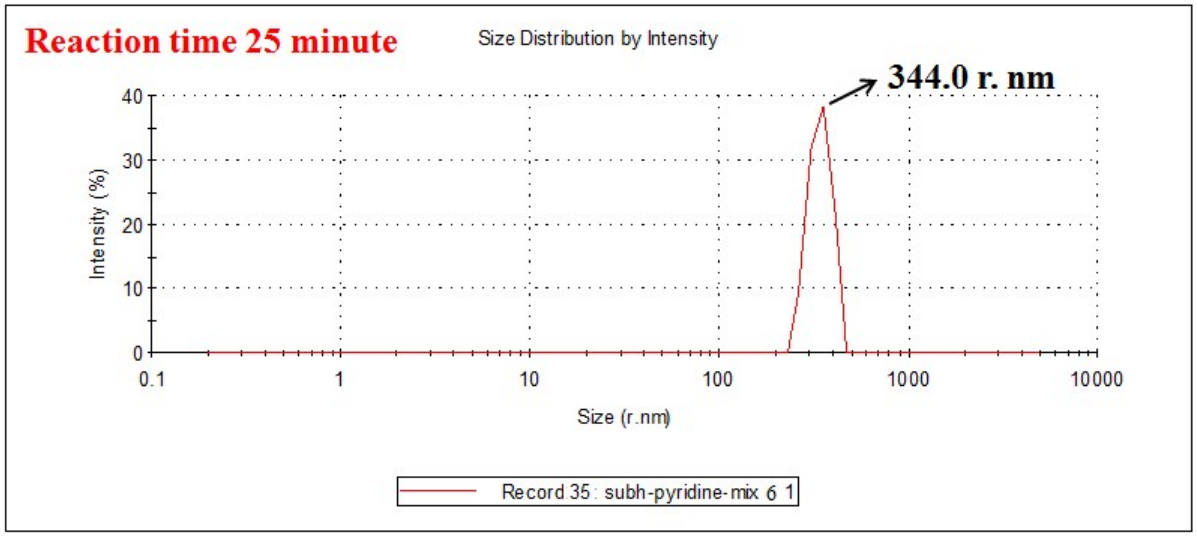


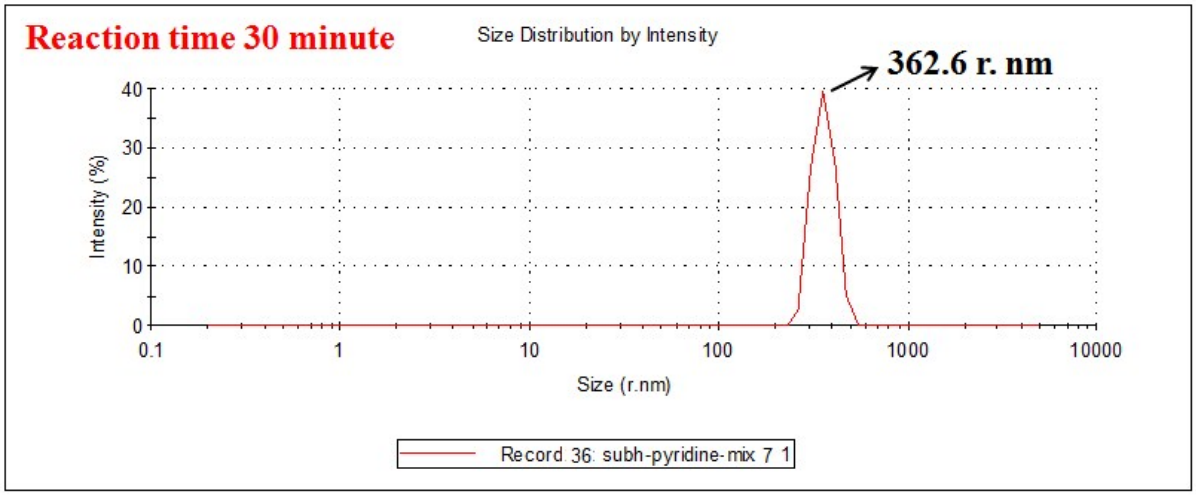

Reaction time 5 minute
Size Distribution by Intensity
97.7 r. nm
Intensity (%)
Size (r.nm)
Record31 : subh-pyridine-mix 2 1

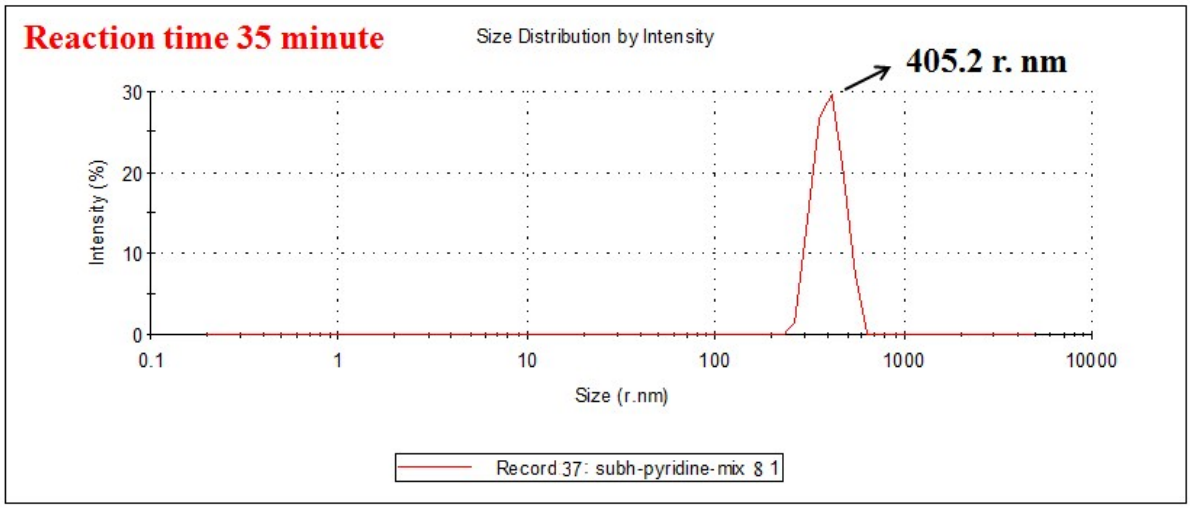

Reaction time 10 minute
Size Distribution by Intensity
107.0 r. nm
Intensity (%)
Size (r.nm)
Record32 : subh-pyridine-mix 3 1

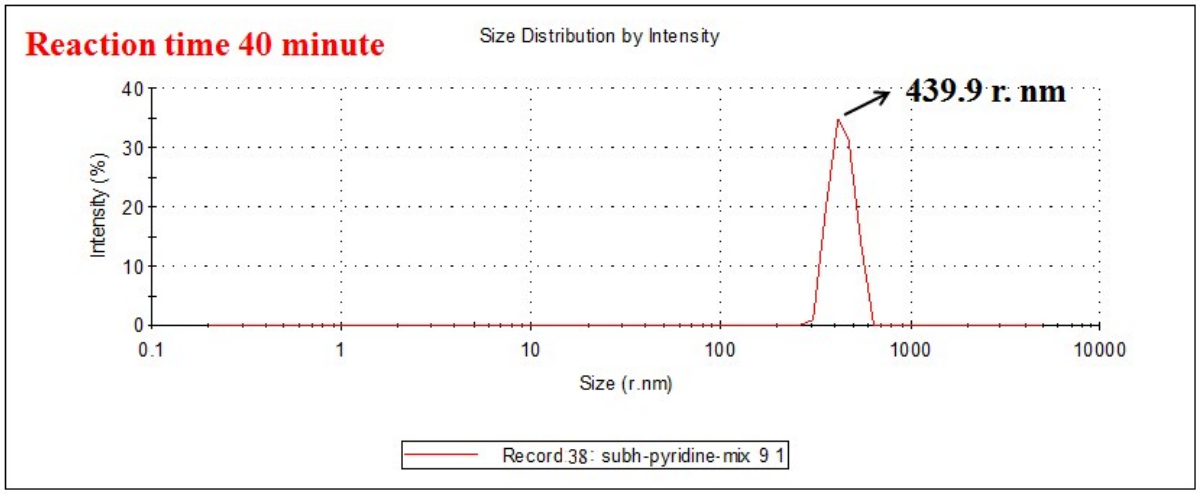

Reaction time 15 minute
Size Distribution by Intensity
170.0 r. nm
Intensity (%)
Size (r.nm)
Record33 : subh-pyridine-mix 4 1

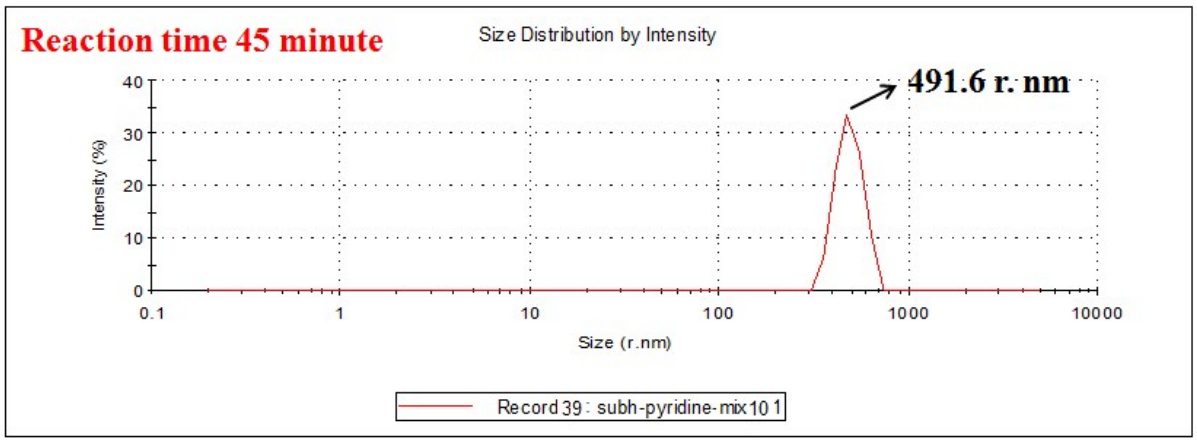

Reaction time 20 minute
Size Distribution by Intensity
324.3 r. nm
Intensity (%)
Size (r.nm)
Record 34: subh-pyridine-mix 5 1

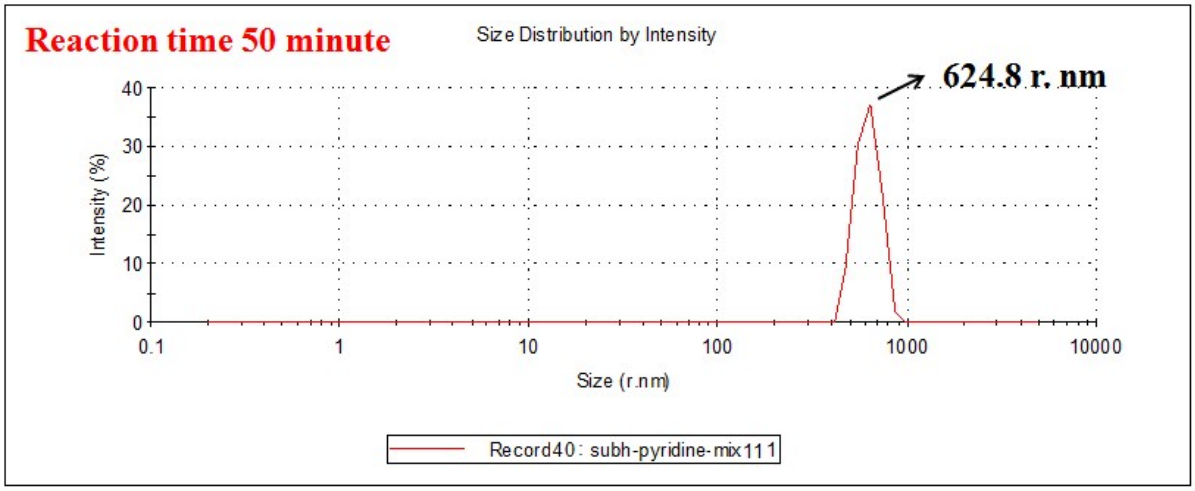

Reaction time 25 minute
Size Distribution by Intensity
344.0 r. nm
Intensity (%)
Size (r.nm)
Record 35: subh-pyridine-mix 6 1

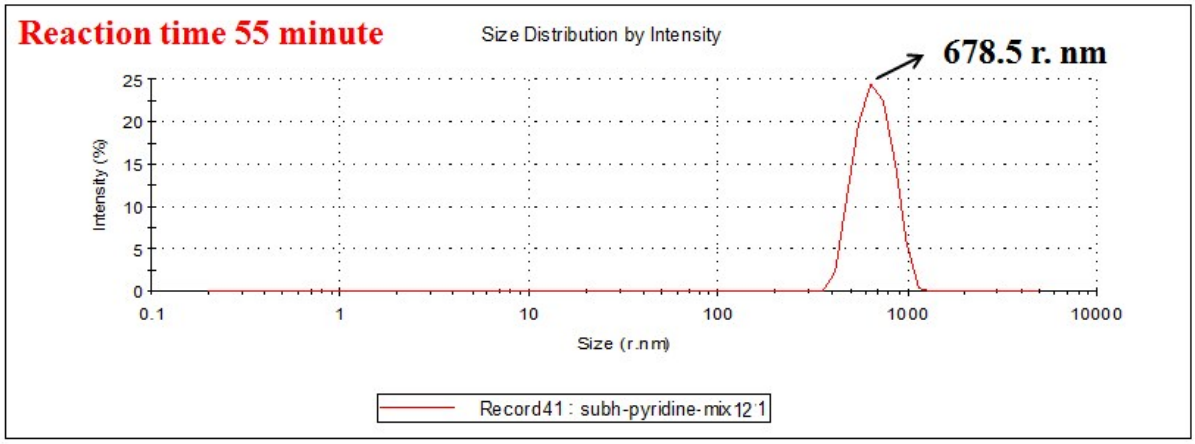

Reaction time 30 minute
Size Distribution by Intensity
362.6 r. nm
Intensity (%)
Size (r.nm)
Record 36: subh-pyridine-mix 7 1

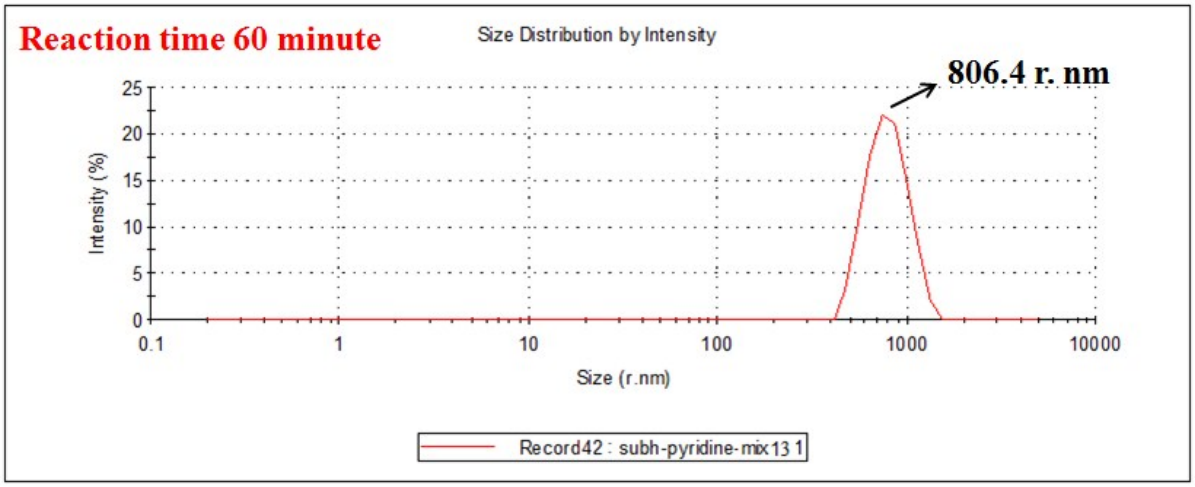

Reaction time 35 minute
Size Distribution by Intensity
405.2 r. nm
Intensity (%)
Size (r.nm)
Record 37: subh-pyridine-mix 8 1


Reaction time 40 minute
Size Distribution by Intensity
439.9 r. nm
Intensity (%)
Size (r.nm)
Record 38: subh-pyridine-mix 9 1


Reaction time 45 minute
Size Distribution by Intensity
491.6 r. nm
Intensity (%)
Size (r.nm)
Record 39: subh-pyridine-mix10 1


Reaction time 50 minute
Size Distribution by Intensity
624.8 r. nm
Intensity (%)
Size (r.nm)
Record40 : subh-pyridine-mix11 1


Reaction time 55 minute
Size Distribution by Intensity
678.5 r. nm
Intensity (%)
Size (r.nm)
Record41 : subh-pyridine-mix12 1


Reaction time 60 minute
Size Distribution by Intensity
806.4 r. nm
Intensity (%)
Size (r.nm)
Record42 : subh-pyridine-mix13 1