SUPPORTING INFORMATION

An elegant approach for the synthesis of multisubstituted imidazole via $FeCl_3/SiO_2$ catalysed activation of acetals: A photo physical study of imidazole-carbazole hybrid

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Experimental Section

General

NMR spectra were measured on a Bruker Ascend 400 spectro-photometer. ^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature using 400 MHz spectrometers (400 MHz for ^{1}H and 100 MHz for ^{13}C). Chemical shifts were reported in parts per million from the tetramethyl silane internal reference, and coupling constants were reported in Hertz. Proton multiplicities were represented as s (singlet), d (doublet), dd (double doublet), t (triplet), q (quartet), and m (multiplet). FTIR spectra were recorded on Bruker Alpha II FTIR spectrometer on Neat or KBr pellets. Mass spectra (HRMS) were obtained from Orbitrap Exploris 120 (Thermo Scientific) using 70 eV in positive ion mode. The single-crystal X-ray diffraction (XRD) data were collected on a Bruker D8 Venture system with a microfocus optics using Cu Kα radiation. The data were analysed and processed with Bruker Apex III software suite 61 incorporated with multiple tools such as cell_now and RLATT for the determination of the unit cell, SAINT-plus for data reduction, and SADABS for absorption correction. The structure solutions were performed with SHELXT and the full-matrix least-squares refinements were performed with SHELXL suite of programs incorporated in Olex 2.6. The steady state absorption spectra were collected on a Shimadzu® UV-1900 Absoprtion spectrophotometer. The steady state emission spectra were collected in a Shimadzu RF-6000 emission spectrophotometer. Emission lifetime experiments were performed using a HORIBA® Jobin-Yvon TCSPC setup using a nano-LED light source of 296 nm (IRF~0.6 ns). Emission lifetime data were deconvoluted and fitted using the DAS-6 software by HORIBA which allows individual and batch fits.

Materials

All reagents were purchased either from Sigma Aldrich chemical Co., USA, Across chemical company or SRL India and was used as received unless otherwise specified. Commercially supplied petroleum ether (60−80°C) and ethyl acetate was distilled before use. Column chromatography was performed on silica gel (60−120 mesh, 0.12−0.25 mm). Analytical thin-layer chromatography (TLC) was performed on 0.25 mm extra-hard silica gel plates with a UV254 fluorescent indicator. Silica-supported ferric chloride reagent was prepared as per procedure described by us using silica-gel 230-400 mesh.

Synthesis

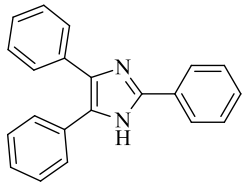
Preparation of silica supported Ferric Chloride

To the slurry of silica gel (230-400 mesh, 40.0g) in acetone (80.0 mL), anhydrous ferric chloride (5.0 g, 30.83 mmol) was added with vigorous stirring for 1hr. The excess acetone was removed under reduced pressure and then the mixture was dried under vacuum for 24 hrs to obtain a free flowing solid. The catalyst was stored in a brown color bottle at 4^{o}C for longer shelf life. This supporting reagent was then characterized by FESEM and EDAX.

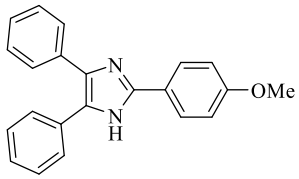
General procedure for the synthesis of tri and tetra-substituted imidazoles:

A small glass vial was charged with catalyst ($FeCl_3/SiO_2$) (20 mg, 2 mol% of $FeCl_3$), benzil (1.0 mmol), acetal (1.1 mmol), ammonium acetate (5.0 mmol) [for trisubstituted imidazoles] or ammonium acetate (2.5 mmol) and amine (2.5 mmol) [for tetra-substituted imidazoles] and then the mixture was heated at 100^{o}C until the full consumption of benzil (TLC). After completion of reaction, the reaction mixture was diluted with EtOAc (5 mL), filtered and washed the catalyst with ethyl acetate. The combined filtrate was evaporated under vacuum. The desired product was isolated either by crystallization or by column chromatography using ethyl acetate-hexane (1:3 to 3:1).

Physical and spectral data of tri-substituted imidazole 3a-g

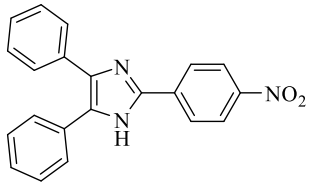


2,4,5-triphenyl-1H-imidazole (**3a**):[1] Yield: 93%, white solid, m. p. 264 - 266 ^{o}C, lit. m. p. 269 ^{o}C; IR (KBr) ν_{max} 3043, 1591, 1490, 1130 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.70 (s, 1H), 8.09 (d, *J* = 7.6 Hz, 2H), 7.56 (d, *J* = 7.6 Hz, 2H), 7.52 - 7.44 (m, 6H), 7.38 (t, *J* = 7.2 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.23 (t, *J* = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.9, 137.6, 135.6, 131.6, 130.8, 129.2, 129.1, 128.9, 128.7, 128.6, 128.2, 127.5, 127.0, 125.6.

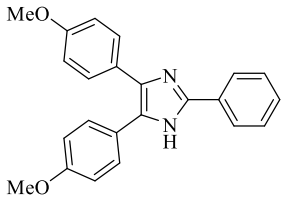


2-(4-methoxyphenyl)-4,5-diphenyl-1H-imidazole (**3b**):[2] Yield: 90%, white solid, m. p. 230 - 232 ^{o}C, lit. m. p. 232 ^{o}C ; IR (KBr) ν_{max} 2953, 1613, 1492, 1239 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.52 (s, 1H), 8.02 (d, *J* = 8.8 Hz, 2H), 7.55 (d, *J* =7.6 Hz, 2H), 7.50 (d, *J* = 7.2

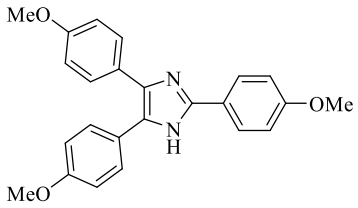
Hz, 2H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.30 (t, *J* = 8.0 Hz, 2H), 7.23 (t, *J* = 7.2Hz, 1H), 7.05 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 159.9, 146.1, 137.2, 135.8, 131.7, 129.1, 128.8, 128.7, 128.6, 128.1, 128.0, 127.7, 127.5, 127.2, 126.9, 123.6, 114.6, 113.8, 55.7



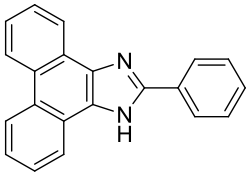
2-(4-nitrophenyl)-4,5-diphenyl-1H-imidazole (**3c**):[2] Yield: 92%, yellow solid, m. p. 230 - 232 ^{o}C, lit. m. p. 232-234 ^{o}C; IR (KBr) ν_{max} 3072, 1589, 1513, 1332, 848 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 13.15 (s, 1H), 8.34 (m, 4H), 7.55 (m, 4H), 7.49 –7.42 (m, 3H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 147.0, 143.9, 138.9, 136.6, 135.1, 131.0, 130.5, 129.2, 129.0, 128.7, 128.6, 127.6, 127.4, 126.2, 124.7.



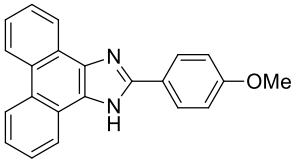
4,5-bis(4-methoxyphenyl)-2-phenyl-1H-imidazole (**3d**): Yield: 90%, white solid, m. p. 170 - 172 ^{o}C; IR (KBr) ν_{max} 2944, 2830, 1501, 1241, 1174, 830, 690 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 6.8 Hz, 2H), 7.42 – 7.35 (m, 7H), 6.84 (d, *J* = 8.0 Hz, 4H), 3.81 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 145.6, 132.3, 130.0, 129.1, 128.7, 128.5, 125.4, 125.3, 113.9, 55.2; HRMS calcd for ($C_{23}H_{20}N_2O_2$ +H^+) 357.1603, found: 357.1591 (M+H^+).



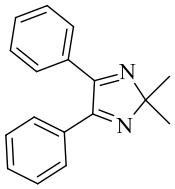
2,4,5-tris(4-methoxyphenyl)-1H-imidazole (**3e**): Yield: 87%, white solid, m. p. 162-164 ^{o}C; IR (KBr) ν_{max} 2943, 2832, 1610, 1500, 1239, 1173, 1024, 826 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 2H), 7.43 (d, *J* = 7.2 Hz, 4H), 6.91 (d, *J* = 8.0 Hz, 2H), 6.85 (d, *J* = 8.0Hz , 4H), 3.83 (s, 3H), 3.82 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.0, 158.8, 145.6, 129.0, 126.8, 125.6, 122.9, 114.2, 113.9, 55.3, 55.2; HRMS calcd for ($C_{24}H_{22}N_2O_3$ +H^+) 387.1708, found: 387.1697 (M+H^+).



2-phenyl-1H-phenanthro [9,10-d]imidazole (**3f**):[3] Yield: 87%, white solid, m. p. 302-304 ^{o}C, lit. m. p. 311-313 ^{o}C; IR (KBr) ν_{max} 3058, 1459 1137, 924, 755, 701 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 13.48 (s, 1H), 8.89 – 8.83 (m, 2H), 8.62 – 8.57 (m, 2H), 8.34 - 8.30 (s, 2H), 7.79 – 7.72 (m, 2H), 7.65 – 7.59 (m, 4H), 7.51 (t, *J* = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 149.6, 137.5, 130.9, 129.7, 129.4, 128.2, 128.0, 127.6, 127.5, 127.5, 126.6, 125.8, 125.6, 124.6, 124.2, 122.9, 122.5, 122.4

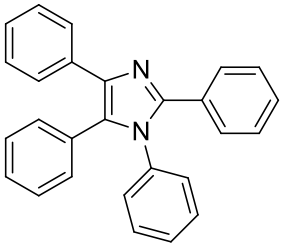


2-(4-methoxyphenyl)-1H-phenanthro [9,10-d]imidazole (**3g**):[4] Yield: 88%, white solid, m. p. 252 - 254 ^{o}C, lit. m. p. 255-256 ^{o}C; IR (KBr) ν_{max} 1605, 1470, 1243, 1172, 1030, 826 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d$_6$) δ 13.3 (s, 1H), 8.84 (d, *J* = 7.6 Hz, 2H), 8.57 (s, 2H), 8.27 (d, *J* = 7.6 Hz, 2H), 7.73 (t, *J* = 7.2 Hz, 2H), 7.62 (t, *J* = 7.6 Hz, 2H), 7.18 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 160.7, 149.8, 128.2, 127.9, 127.5, 125.5, 124.4, 123.5, 122.3, 114.8, 55.8.

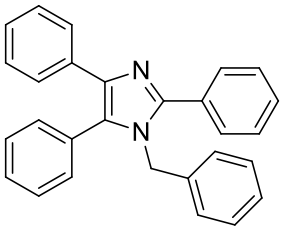


2,2-dimethyl-4,5-diphenyl-2H-imidazole (**3h**): Yield: 85%, white solid, m. p. 58 - 60 ^{o}C; IR (KBr) ν_{max} 3053, 2981, 2929, 1610, 1492, 1445, 1215, 801cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 -7.52 (m, 4H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.37 (t, *J* = 7.6 Hz, 4H), 1.68 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 132.7, 130.2, 128.9, 128.3, 101.6, 24.2; HRMS calcd for ($C_{17}H_{16}N_2$ +H$^+$) 249.1392, found: 249.1434 (M+H$^+$).

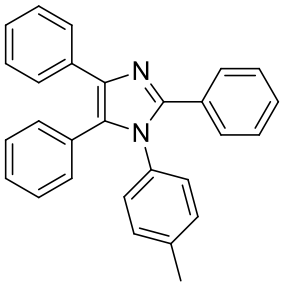
Physical and Spectral data of 5a-g



1,2,4,5-tetraphenyl-1H-imidazole (**5a**):[2] Yield: 91%, white solid, m. p. 216-218 ^{o}C, lit. m. p. 220 ^{o}C; IR (KBr) ν_{max} 3742, 3603, 3052, 1486, 1136, 918, 764, 686 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64- 7.62 (m, 2H), 7.47 (d, *J* = 1.6 Hz, 1H), 7.45 (d. *J* = 2.4 Hz, 1H) , 7.33 – 7.20 (m, 12H), 7.17 (d, *J* =1.6 Hz, 1H), 7.14 (d, *J* = 2.0Hz, 1H), 7.07 (d, *J* = 1.2 Hz, 1H), 7.05 (d, *J* = 2.0 Hz, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 138.2, 137.0, 134.4, 131.1, 130.8, 130.6, 130.4, 129.0, 128.9, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.4, 126.5

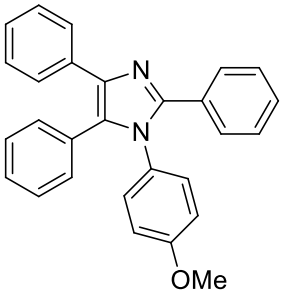


1-benzyl-2,4,5-triphenyl-1H-imidazole (**5b**):[5] Yield: 93%, white solid, m. p. 162-164 ^{o}C, lit. m. p. 170 ^{o}C; IR (KBr) ν_{max} 3057, 1597, 1443, 1080, 919, 690 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 - 7.68 (m, 2H), 7.63 – 7.61 (m, 2H), 7.44 – 7.42 (m, 3H), 7.39 – 7.32 (m, 3H), 7.26 – 7.16 (m, 8H), 6.85 – 6.83 (m, 2H), 5.14 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.0, 138.0, 137.5, 134.4, 131.0, 130.9, 130.0, 129.0, 128.8, 128.7, 128.5, 128.5, 128.0, 127.3, 126.7, 126.3, 125.9, 48.2.

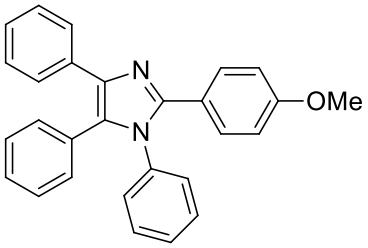


2,4,5-triphenyl-1-(p-tolyl)-1H-imidazole (**5c**):[6] Yield: 89%, white solid, m. p. 280-182 ^{o}C, lit. m. p. 284-285 ^{o}C ; IR (KBr) ν_{max} 3027, 1596, 1393, 1137, 1079, 830, 689 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.2 Hz, 2H), 7.49 - 7.47 (m, 2H), 7.29 – 7.19 (m, 9H), 7.18 – 7.15 (m, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 6.94 (d, *J* = 8.0 Hz, 2H), 2.34 (s, 3H); ^{13}C NMR

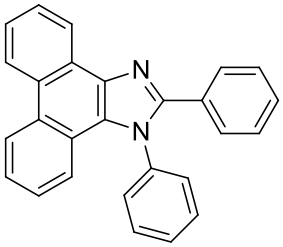
(100 MHz, $CDCl_3$) δ 147.0, 138.2, 138.1, 134.5, 134.5, 131.2, 130.9, 130.8, 130.7, 129.7, 129.0, 128.3, 128.2, 128.2, 128.1, 128.1, 127.9, 127.4, 126.6, 21.2



1-(4-methoxyphenyl)-2,4,5-triphenyl-1H-imidazole (**5d**):[7] Yield: 85%, white solid, m. p. 180 – 182 ^oC, lit. m.p. 185-186 ^oC; IR (KBr) ν_{max} 1598, 1290, 1240, 1025, 835, 770, 691 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.6 Hz, 2H), 7.48 (t, *J* =4.4 Hz, 2H), 7.27 – 7.21 (m, 9H), 7.17 - 7.16 (m, 2H), 6.99 (d, *J* = 8.4 Hz, 2H), 6.78 (d, *J* = 8.4 Hz, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 147.1, 138.1, 134.5, 131.2, 131.1, 130.8, 130.6, 129.9, 129.4, 128.9, 128.3, 128.2, 128.2, 128.1, 127.9, 127.4, 126.6, 114.2, 55.4.

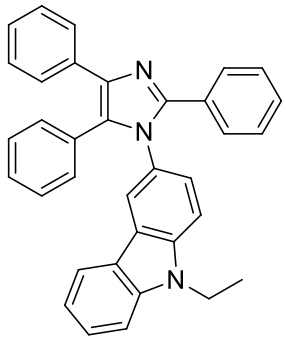


2-(4-methoxyphenyl)-1,4,5-triphenyl-1H-imidazole (**5e**):[8] Yield: 82%, white solid, m. p. 180 – 182 ^oC, lit. m. p. 180-182 ^oC; IR (KBr) ν_{max} 2197, 1951, 1482, 1182, 918, 765, 691 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.62-7.63 (m, 2H), 7.38 (d, *J* = 8.8 Hz, 2H), 7.30 – 7.19 (m, 10H), 7.15(d, *J* = 1.6 Hz, 1H), 7.13 (d, *J* = 2.0Hz, 1H), 7.07(d, *J* = 1.6 Hz 1H), 7.05 (d, *J* = 2.4Hz, 1H), 6.79 (d, *J* = 8.8 Hz, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 146.9, 138.0, 137.3, 134.5, 131.1, 130.8, 130.5, 130.3, 129.1, 128.5, 128.3, 128.2, 128.1, 127.9, 127.4, 126.5, 123.1, 113.6, 55.2.



1,2-diphenyl-1H-phenanthro[9,10-d]imidazole (**5f**): Yield: 86%, white solid, m. p. 192 – 194 ^oC; IR (KBr) ν_{max} 1693, 1459, 1385, 1136, 919, 692 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.92 (d, *J* = 8.0 Hz, 1H), 8.80 – 8.72 (m, 2H), 7.79 – 7.54 (m, 10H), 7.33 – 7.20 (m, 5H);

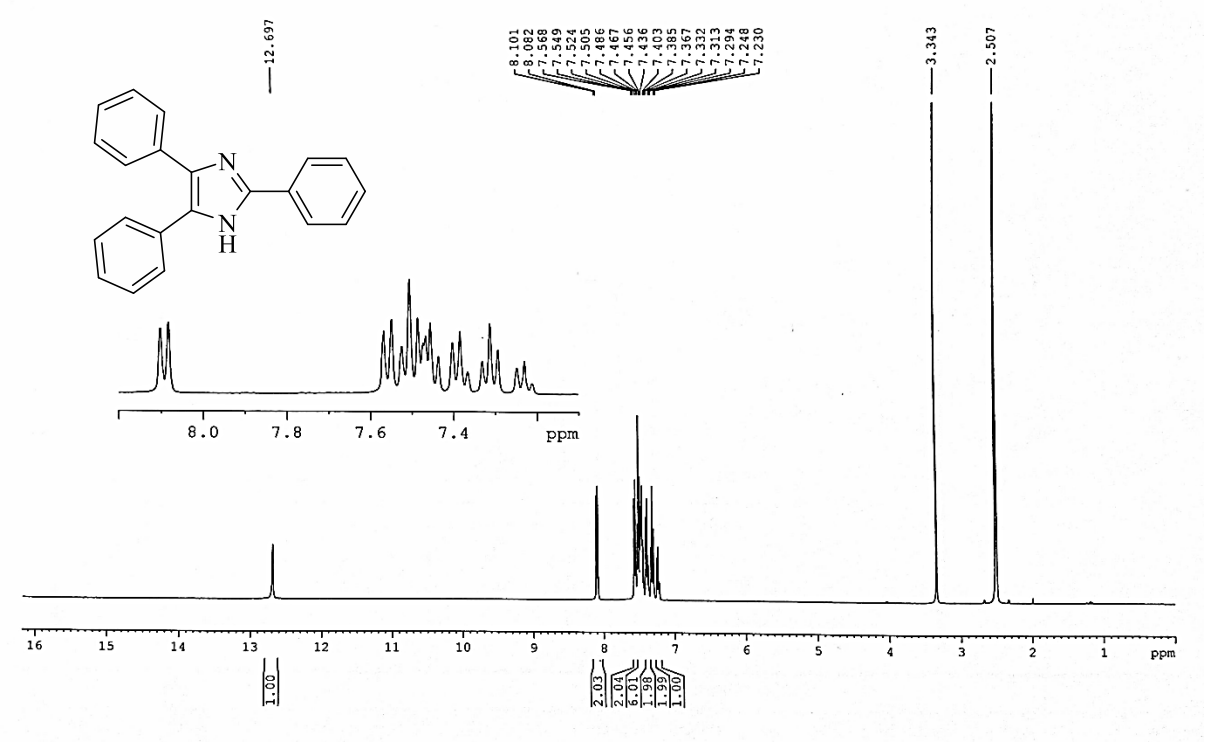
^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 138.7, 137.4, 130.5, 130.1, 129.8, 129.5, 129.3, 129.1, 128.8, 128.3, 128.2, 128.1, 127.3, 127.2, 126.3, 125.6, 124.9, 124.1, 123.1, 123.1, 122.8, 120.9; HRMS calcd for ($C_{27}H_{18}N_2$ +H^+) 371.1548, found: 371.1538 (M+H^+).



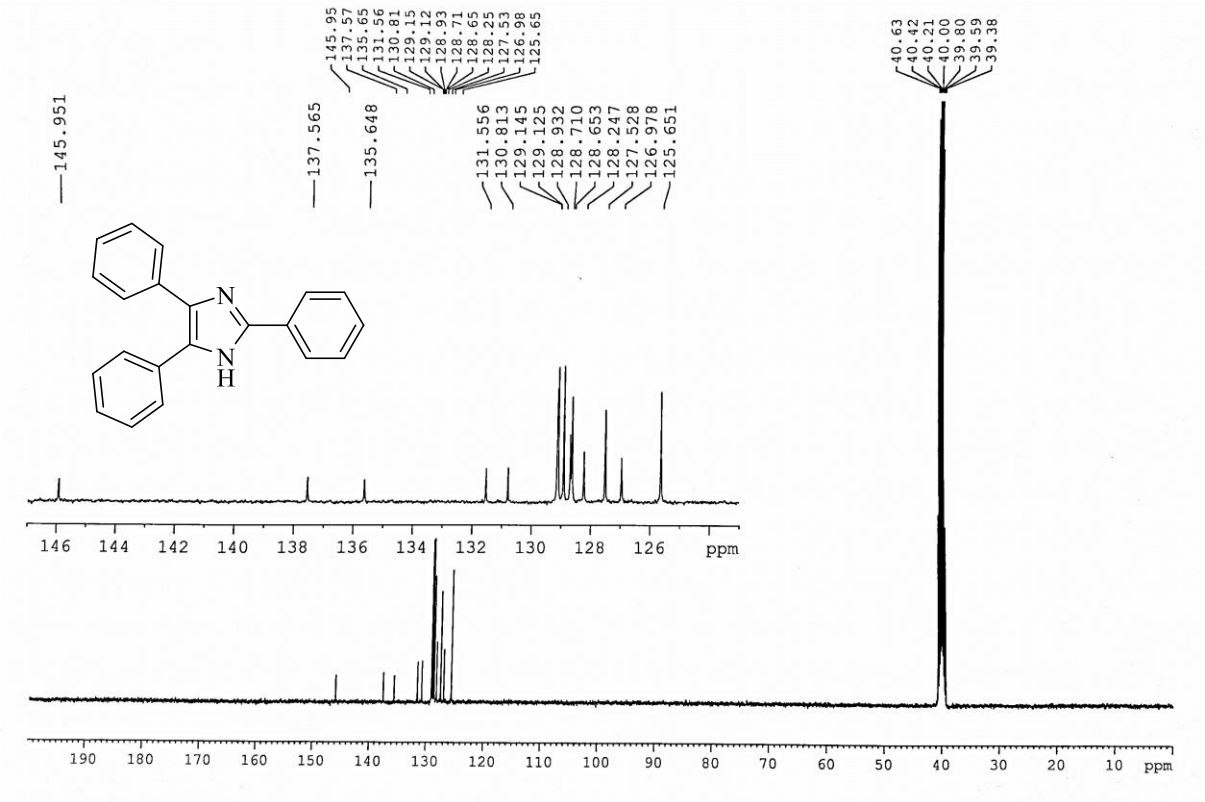
9-ethyl-3-(2,4,5-triphenyl-1H-imidazol-1-yl)-9H-carbazole (**5g**): Yield: 82%, white solid, m. p. 146 – 148 ^{o}C ; IR (KBr) ν_{max} 3051, 2972, 1593, 1476, 1223, 1136, 1080, 923, 688 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 1H), 7.83 (s, 1H), 7.67 (d, *J* = 8.0 Hz, 2H), 7.53 – 7.50 (m, 3H), 7.44 (d, *J* = 8.4 Hz, 1H), 7.32 – 7.18 (m, 14H), 4.35 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 140.5, 139.1, 138.0, 134.7, 131.6, 131.1, 130.9, 130.8, 128.8 128.6, 128.2, 128.1, 128.0, 127.7, 127.4, 126.5, 126.4, 125.9, 122.9, 122.4, 120.7, 120.4, 119.2, 108.8, 108.5, 37.7, 13.8; HRMS calcd for ($C_{35}H_{27}N_3$ +H^+) 490.2283, found: 490.2273 (M+H^+).

^{1}H and ^{13}C NMR Spectra of 3a-h

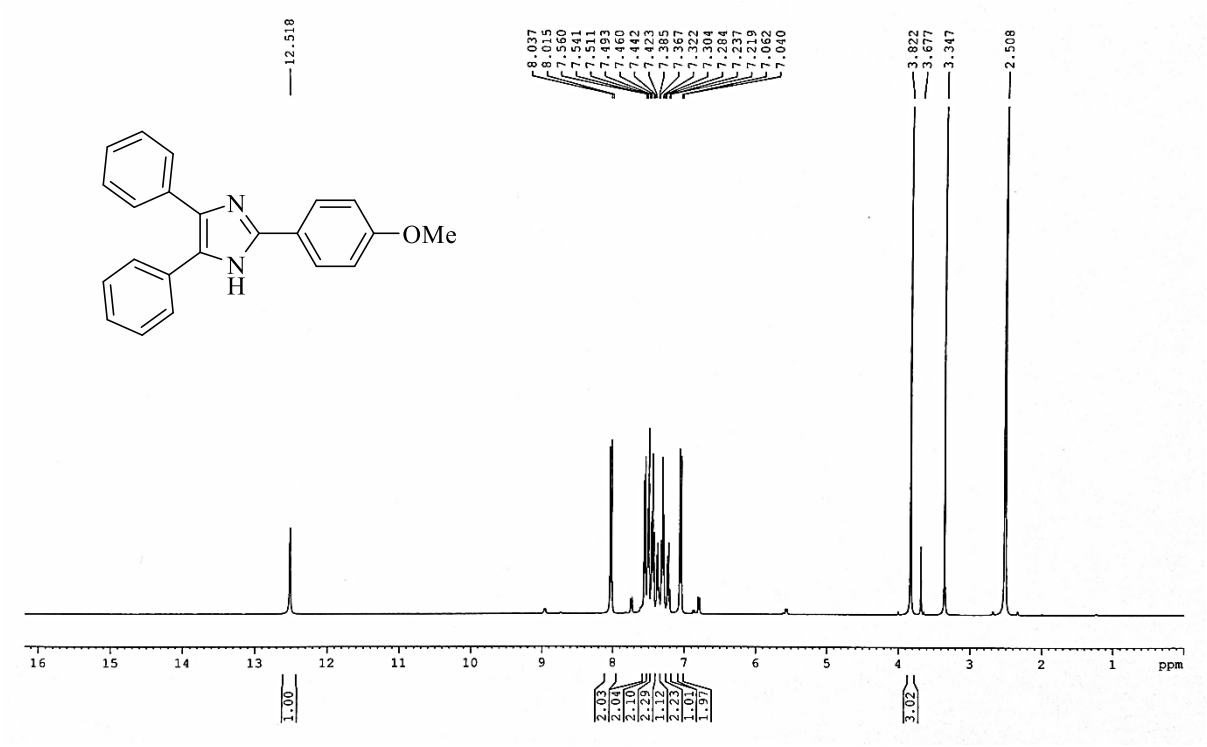
^{1}H NMR (400 MHz, DMSO-d_6) of (**3a**)



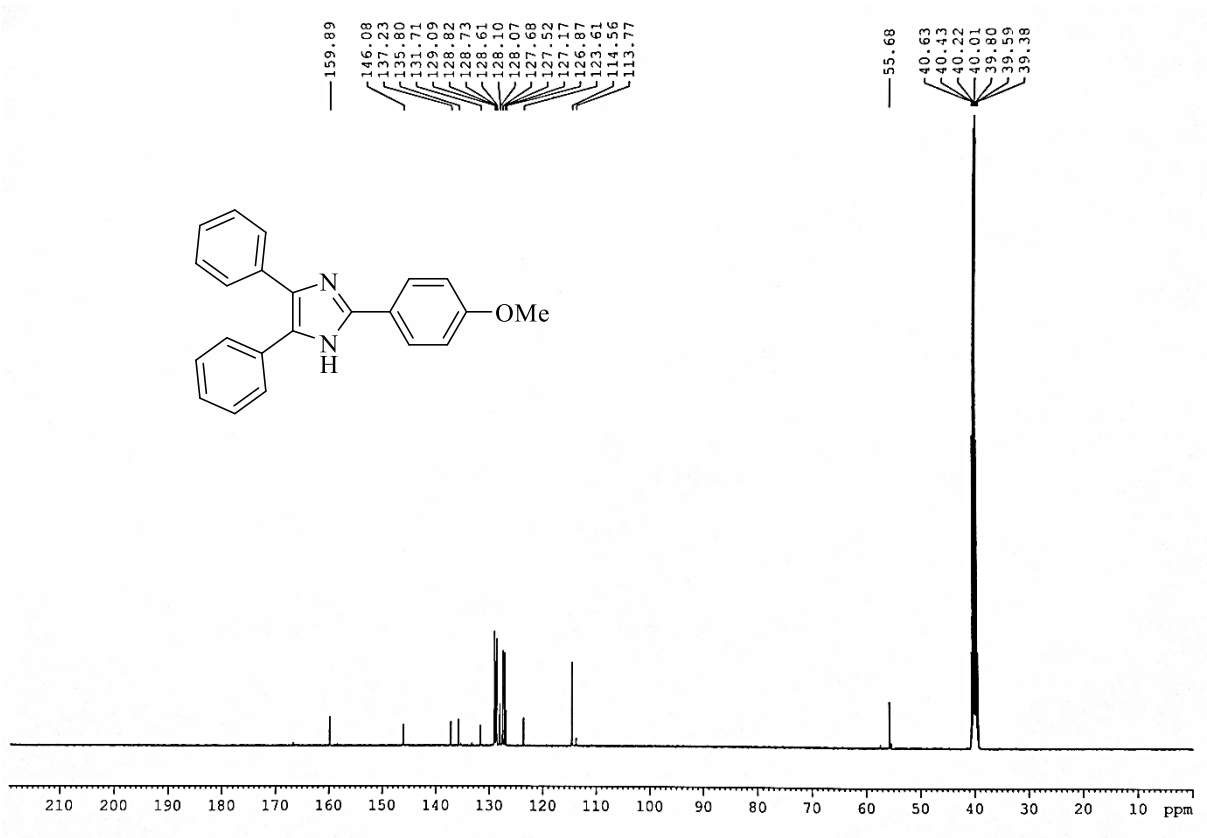
^{13}C NMR (100 MHz, DMSO-d_6) of (**3a**)



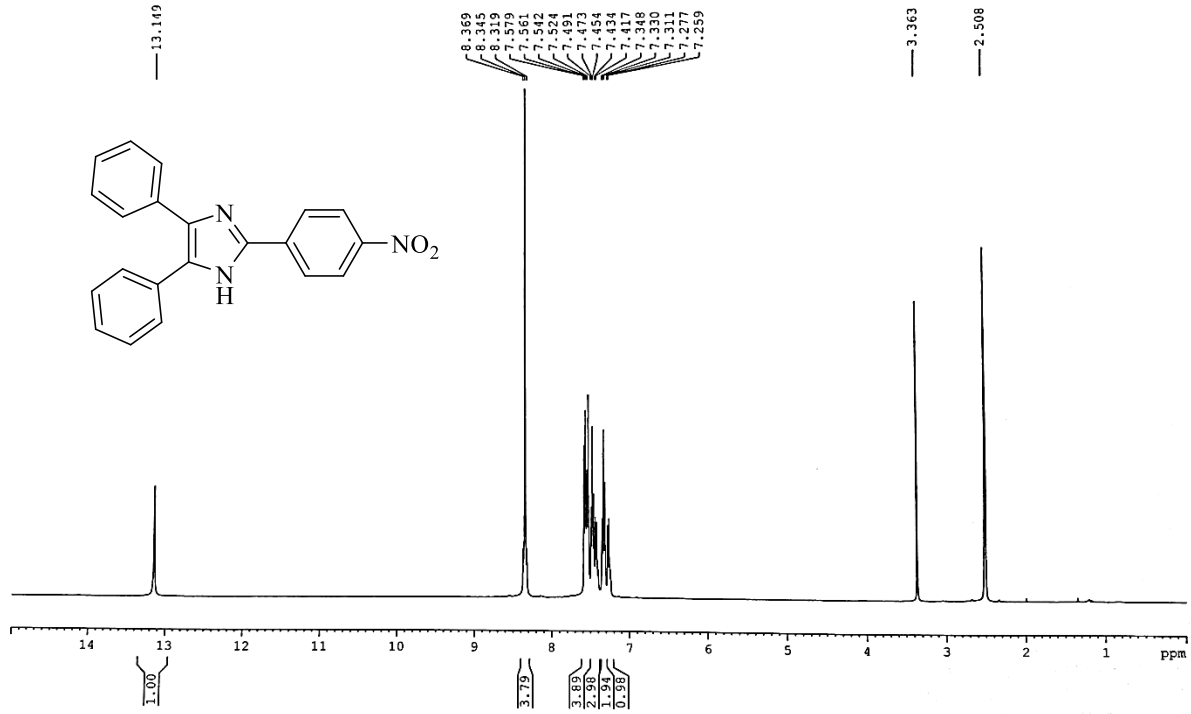
^{1}H NMR (400 MHz, DMSO-d_6) of (**3b**)



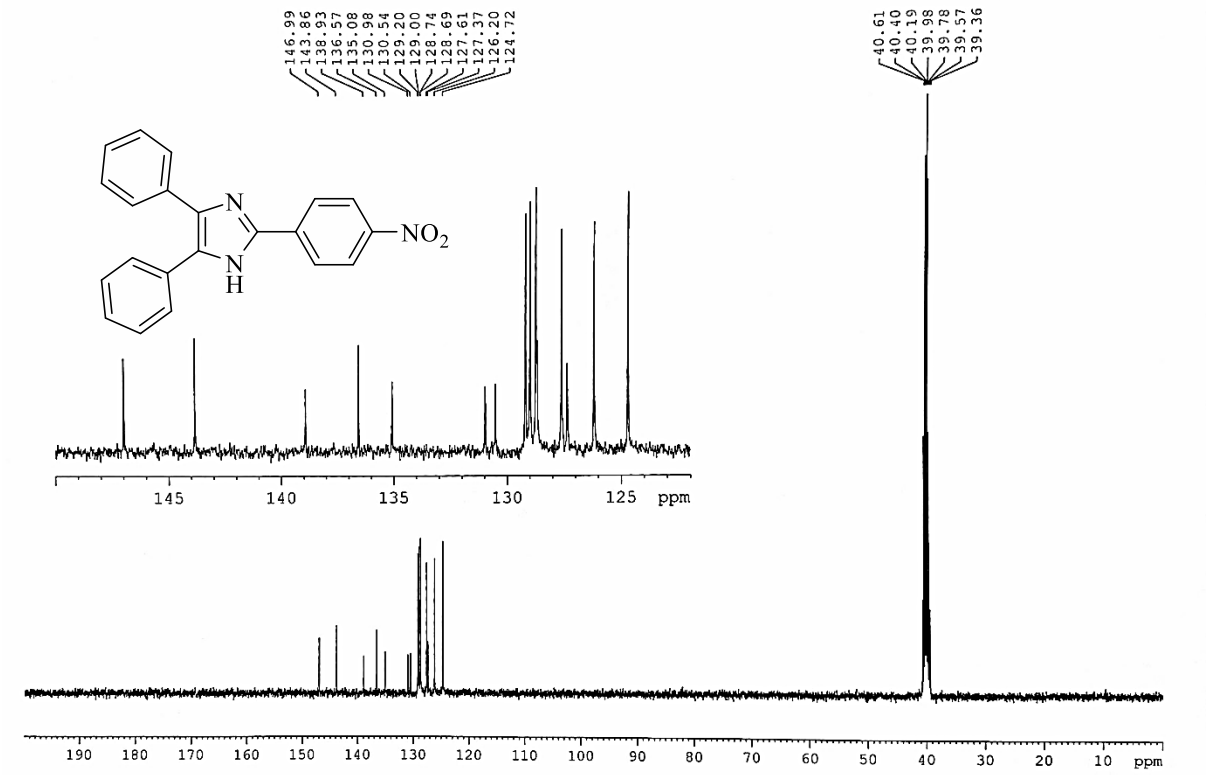
^{13}C NMR (100 MHz, DMSO-d_6) of (**3b**)



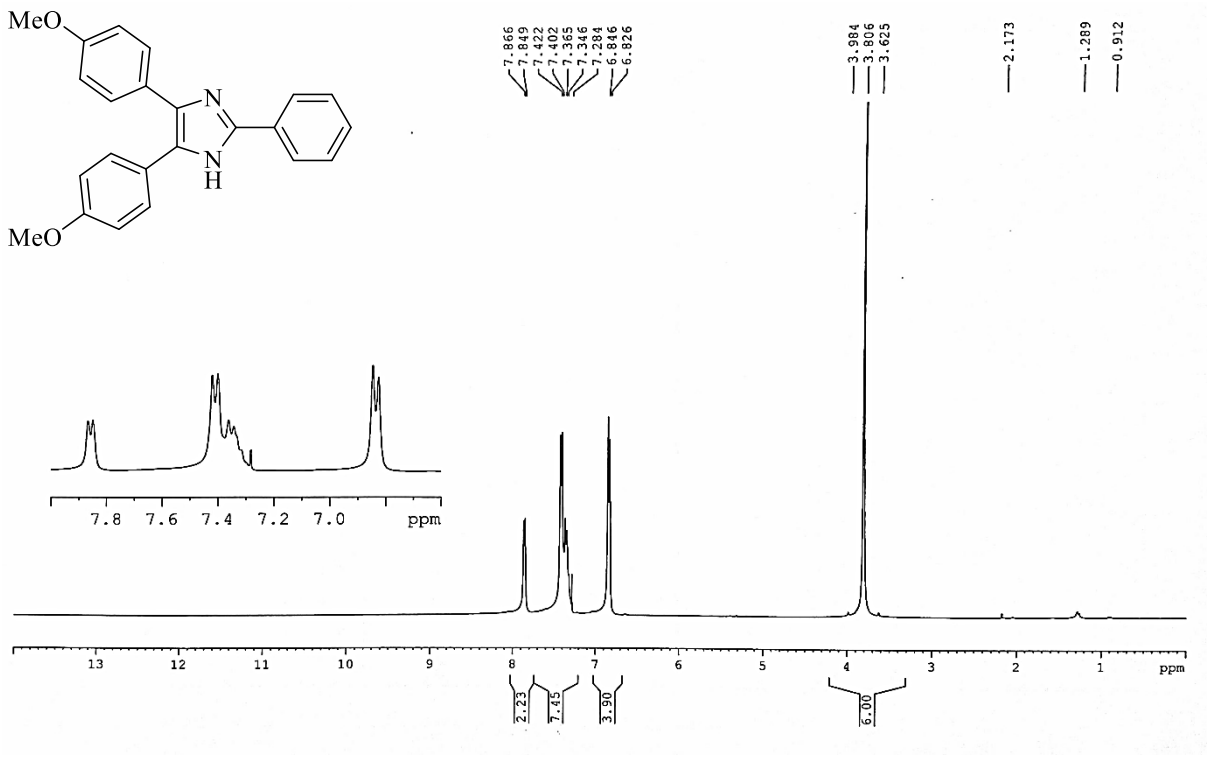
^{1}H NMR (400 MHz, DMSO-d_6) of (**3c**)



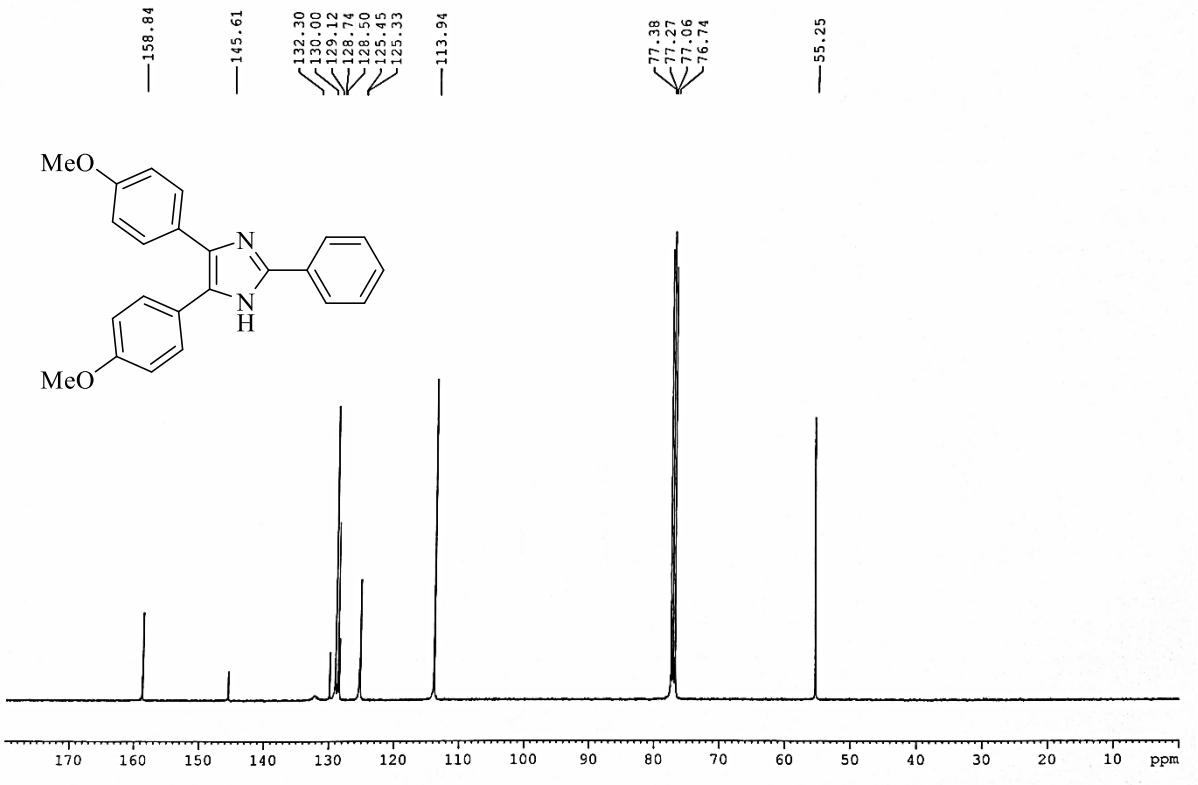
^{13}C NMR (100 MHz, DMSO-d_6) of (**3c**)



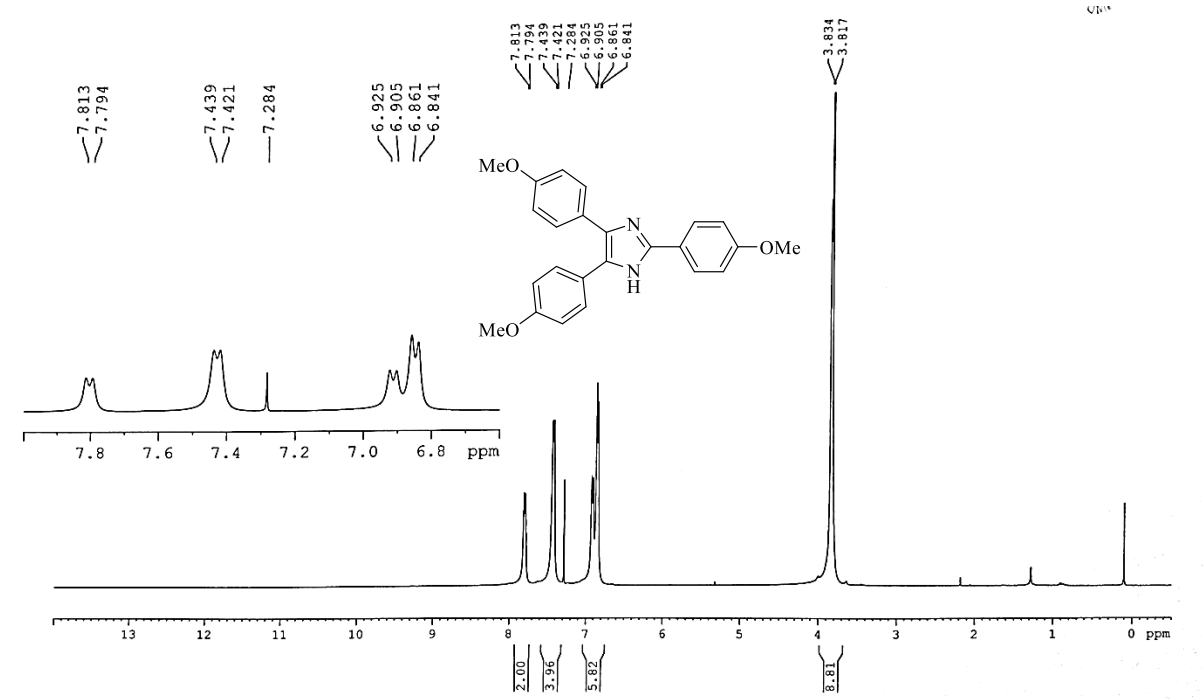
^{1}H NMR (400 MHz, $CDCl_3$) of (**3d**)



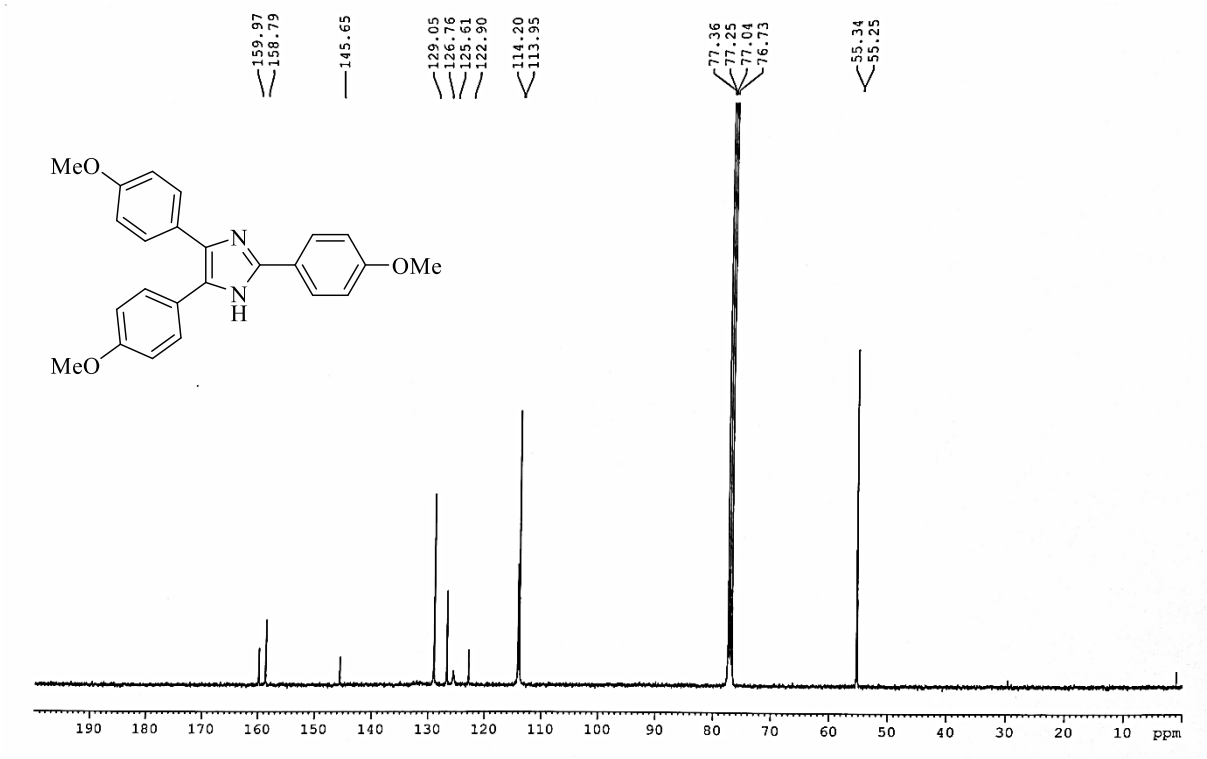
^{13}C NMR (100 MHz, $CDCl_3$) of (**3d**)



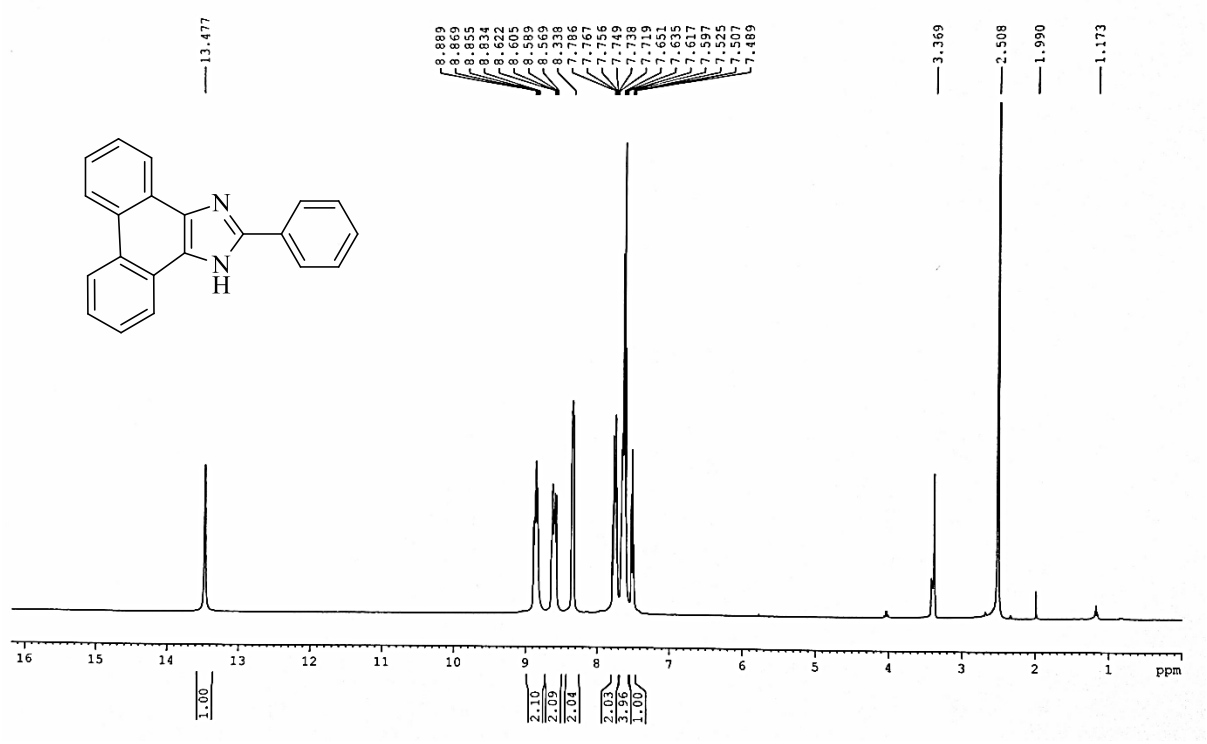
^{1}H NMR (400 MHz, $CDCl_3$) of (**3e**)



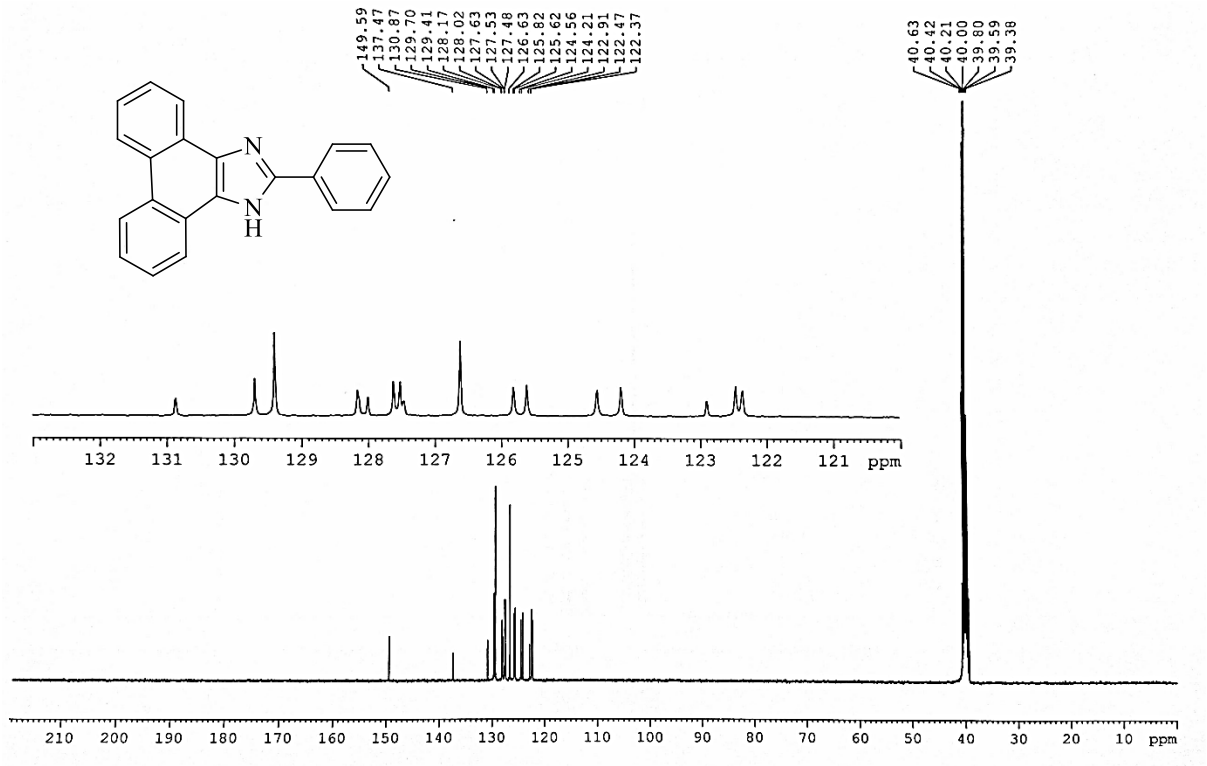
^{13}C NMR (100 MHz, $CDCl_3$) of (**3e**)



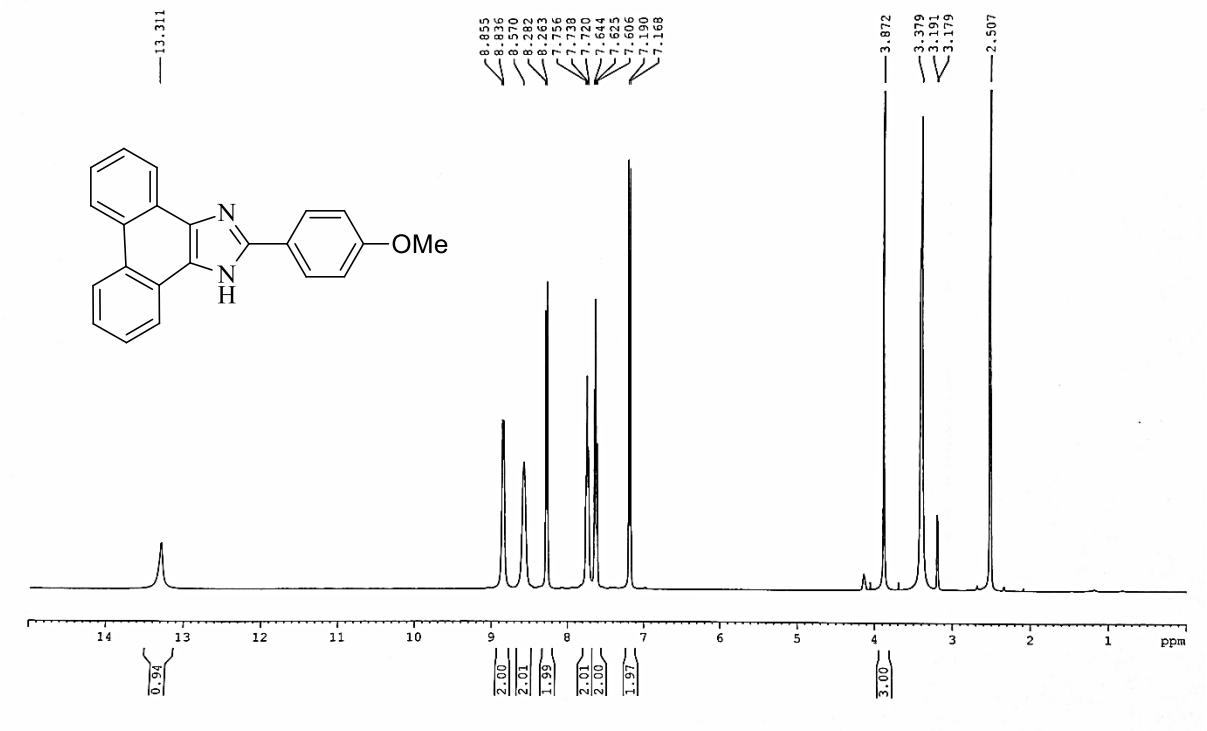
^{1}H NMR (400 MHz, DMSO-d_6) of (**3f**)



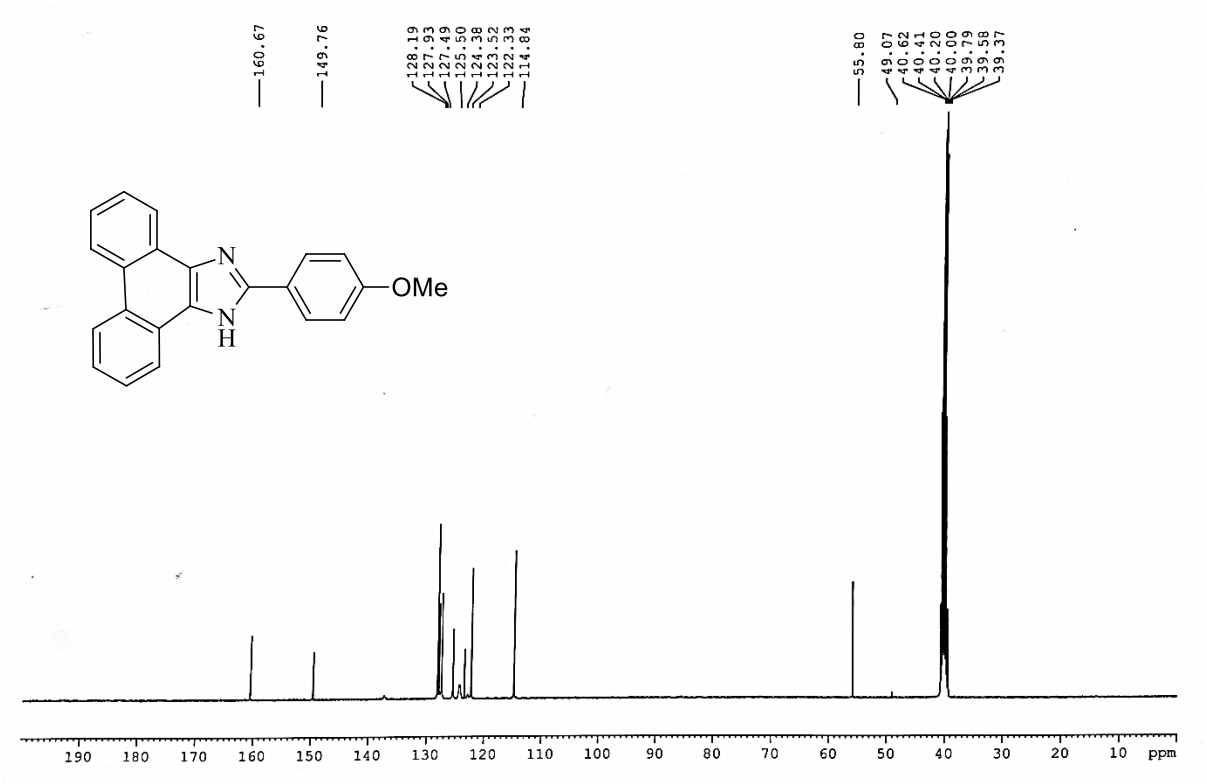
^{13}C NMR (100 MHz, DMSO-d_6) of (**3f**)



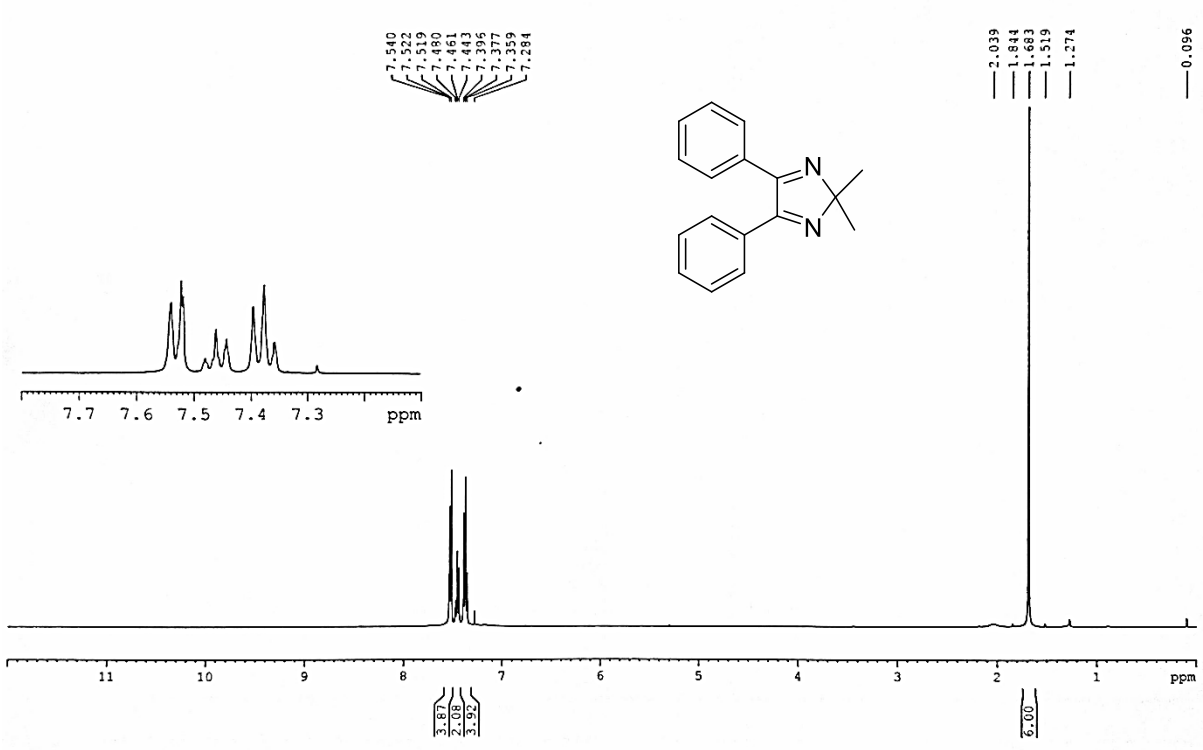
^{1}H NMR (400 MHz, DMSO-d_6) of (**3g**)



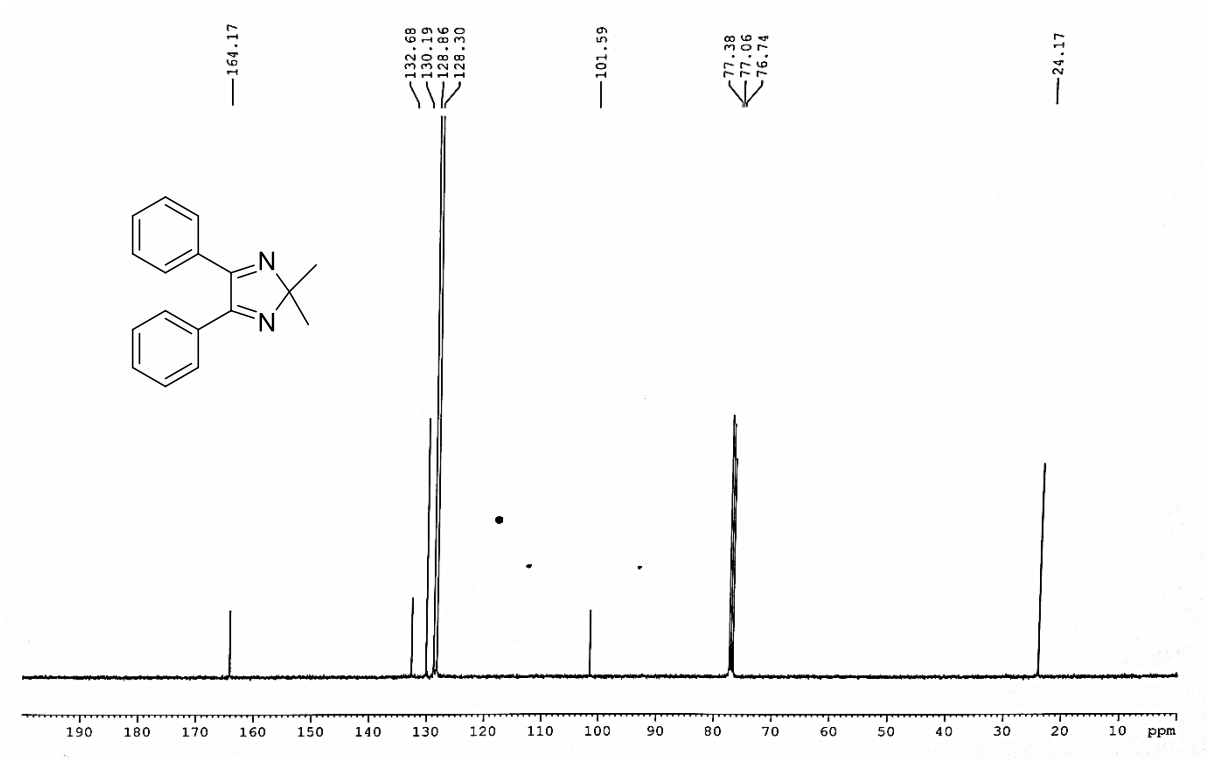
^{13}C NMR (100 MHz, DMSO-d_6) of (**3g**)



^{1}H NMR (400 MHz, $CDCl_3$) of (**3h)**

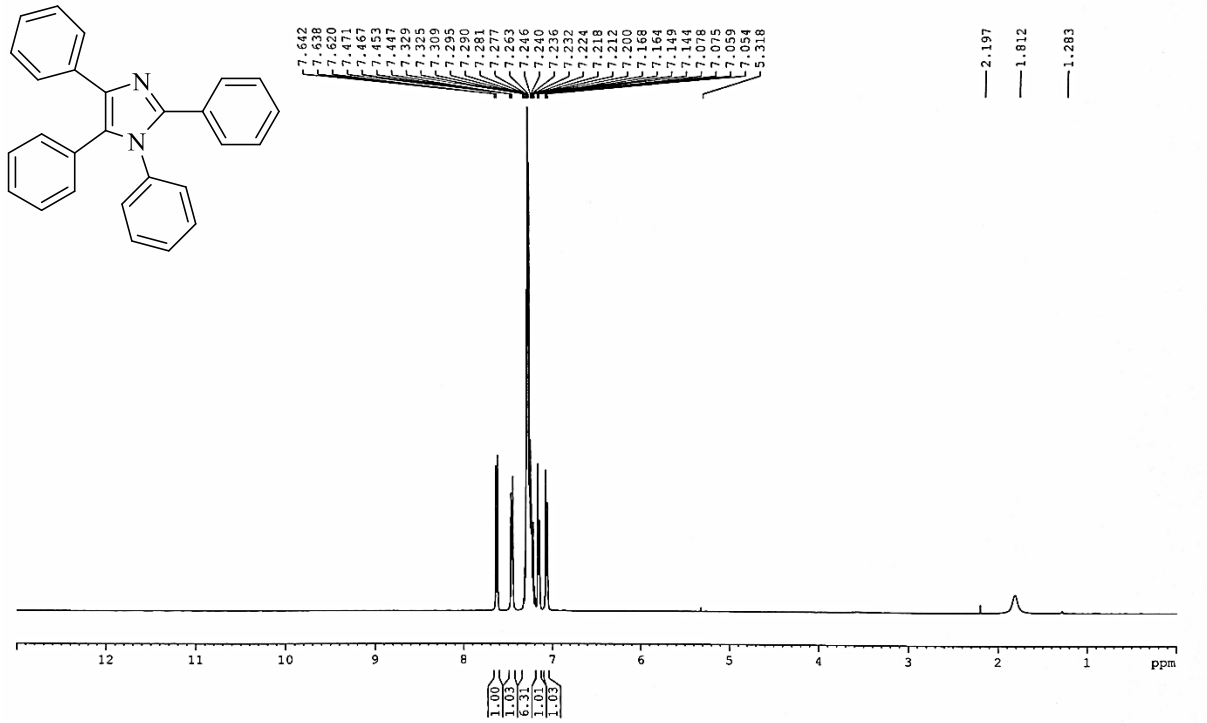


^{13}C NMR (100 MHz, $CDCl_3$) of (**3h**)

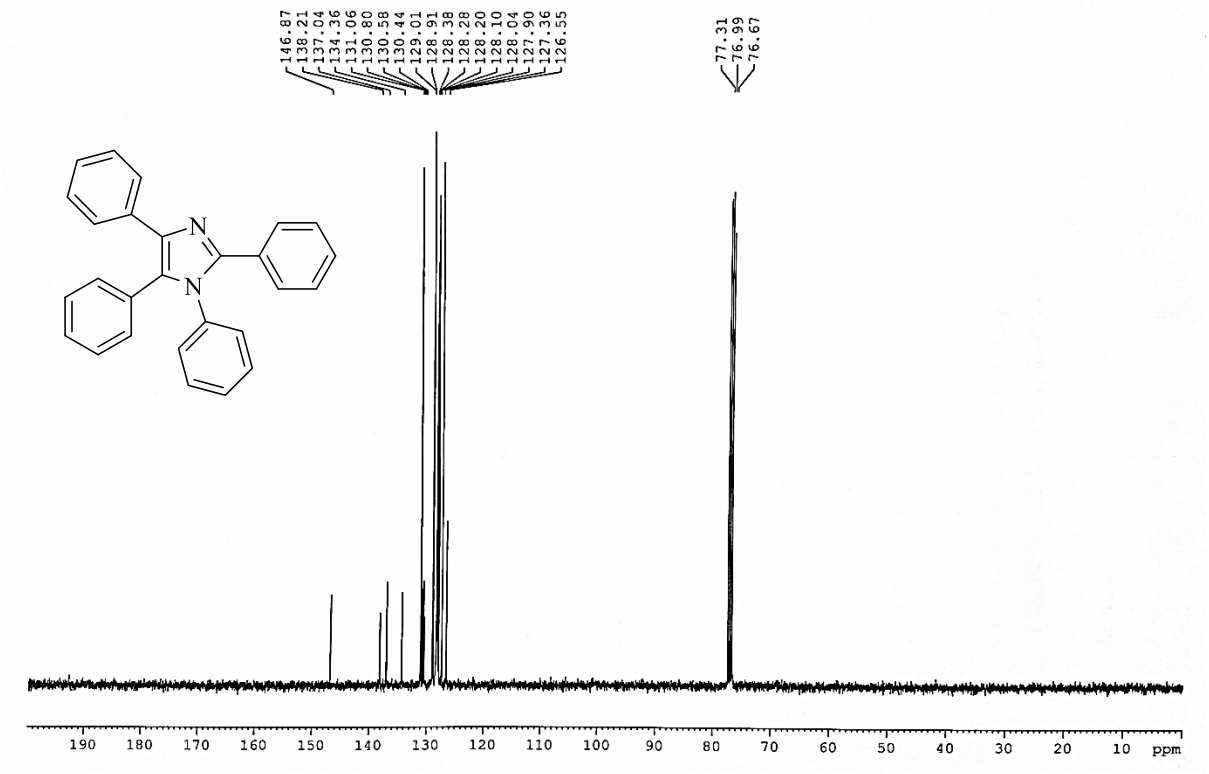


^{1}H and ^{13}C NMR Spectra of 5a-g

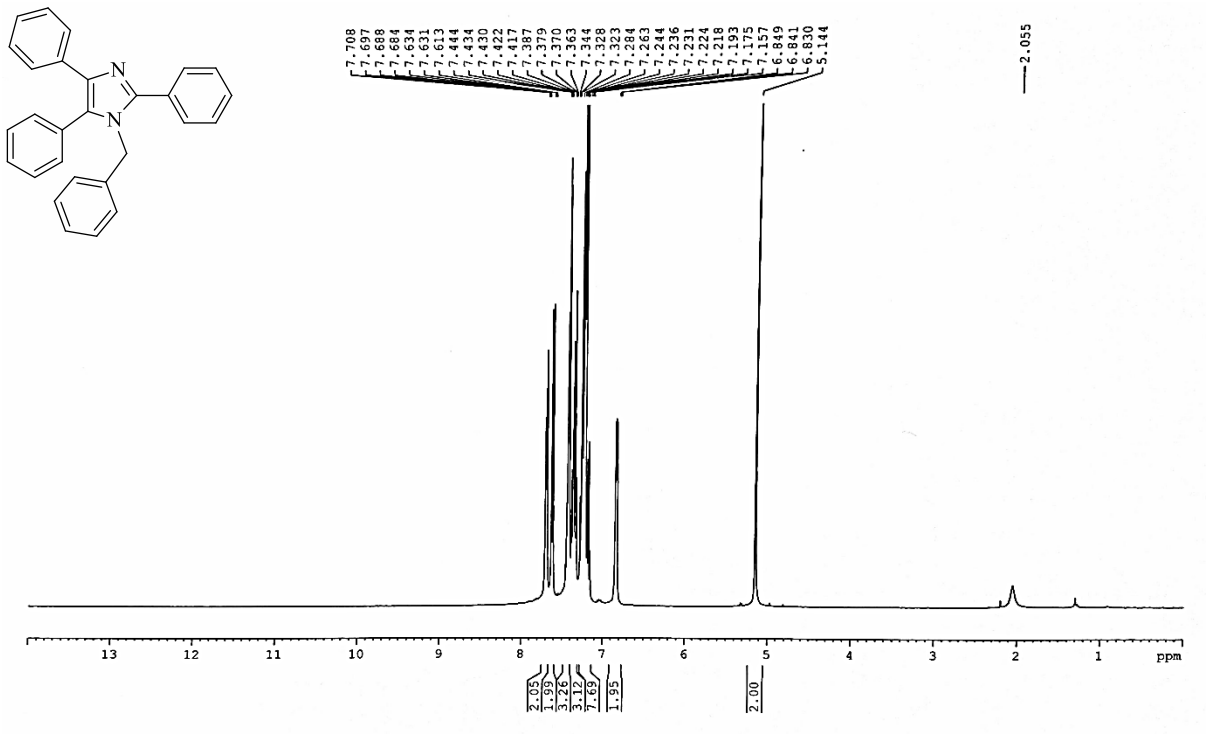
^{1}H NMR (400 MHz, $CDCl_3$) of (**5a**)



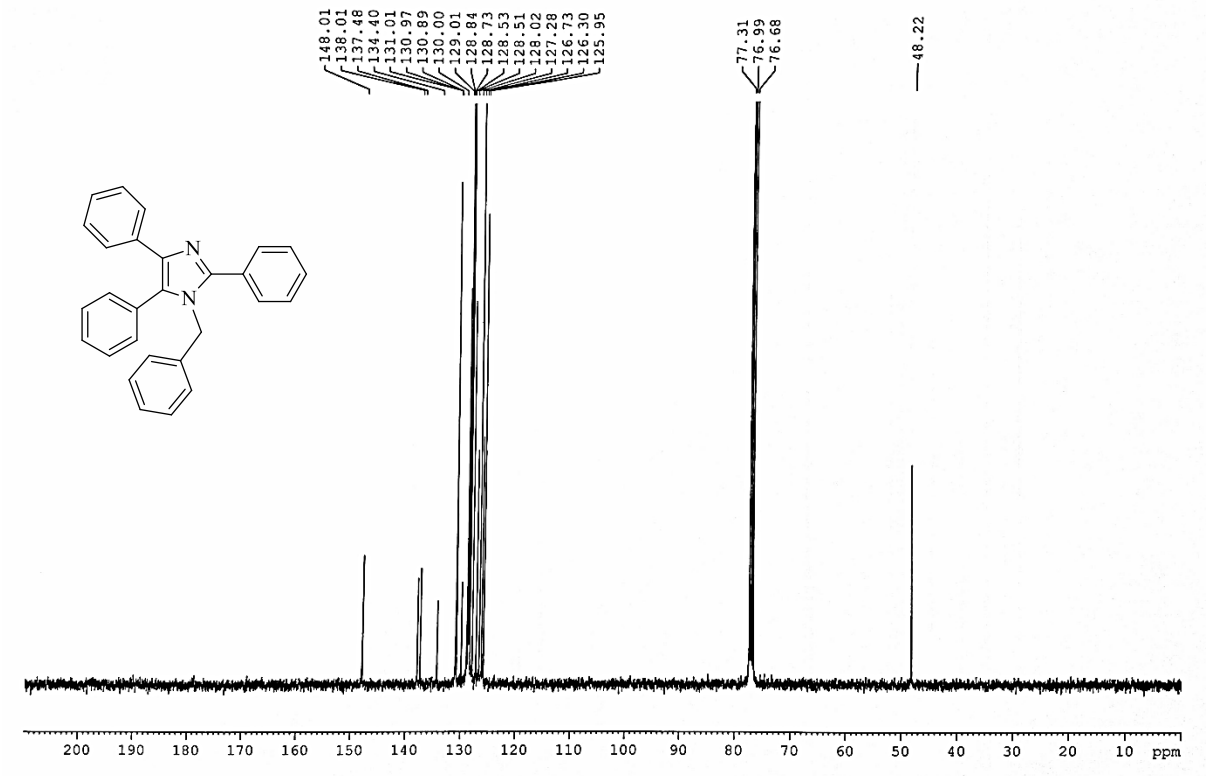
^{13}C NMR (100 MHz, $CDCl_3$) of (**5a**)



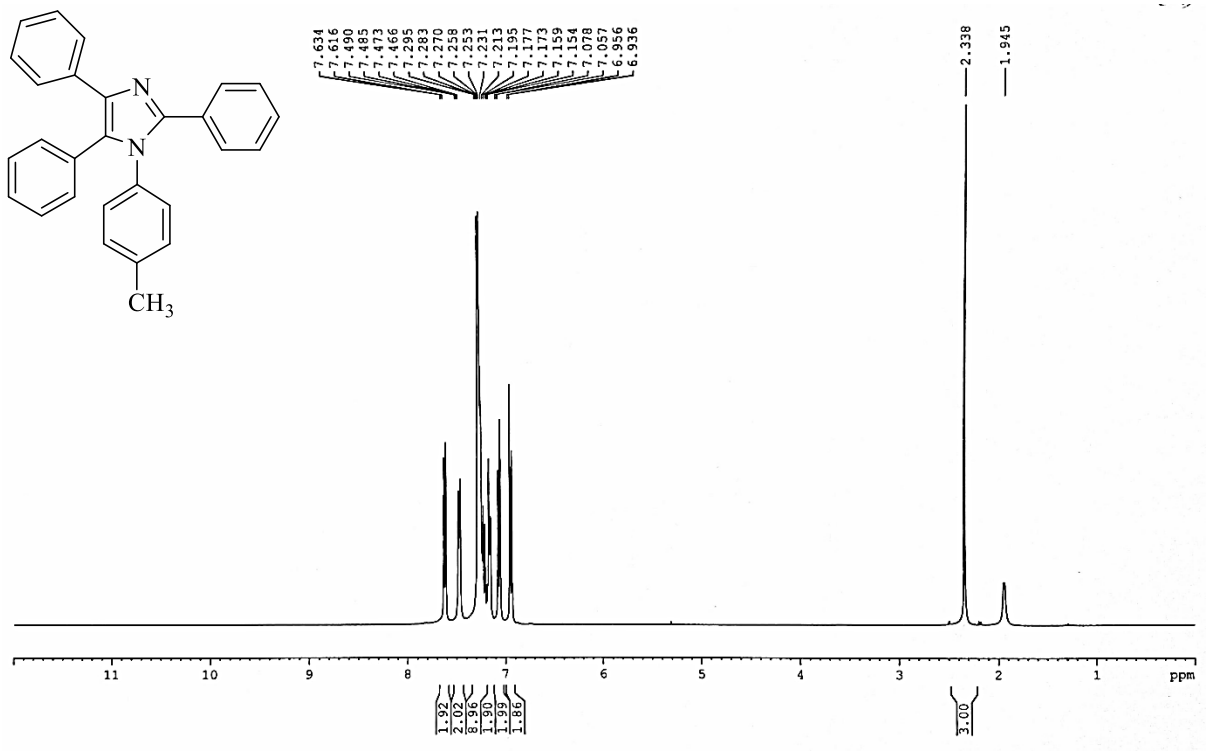
^{1}H NMR (400 MHz, $CDCl_3$) of (**5b**)



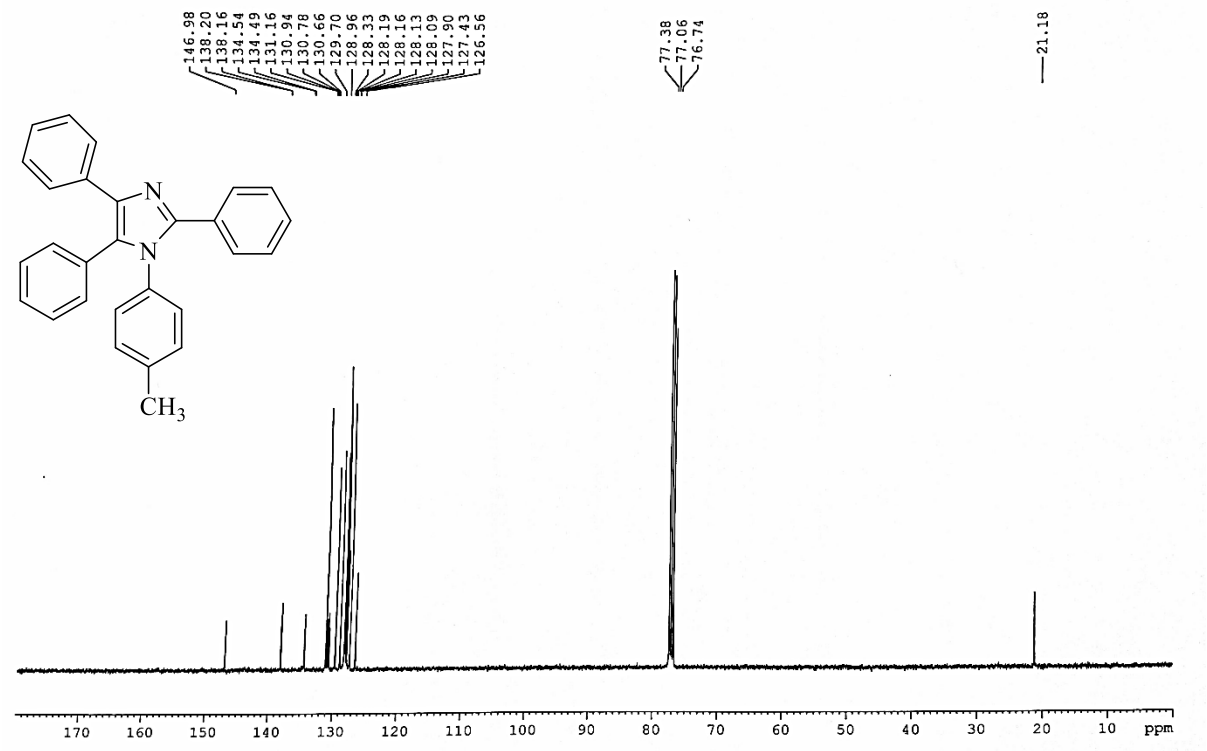
^{13}C NMR (100 MHz, $CDCl_3$) of (**5b**)



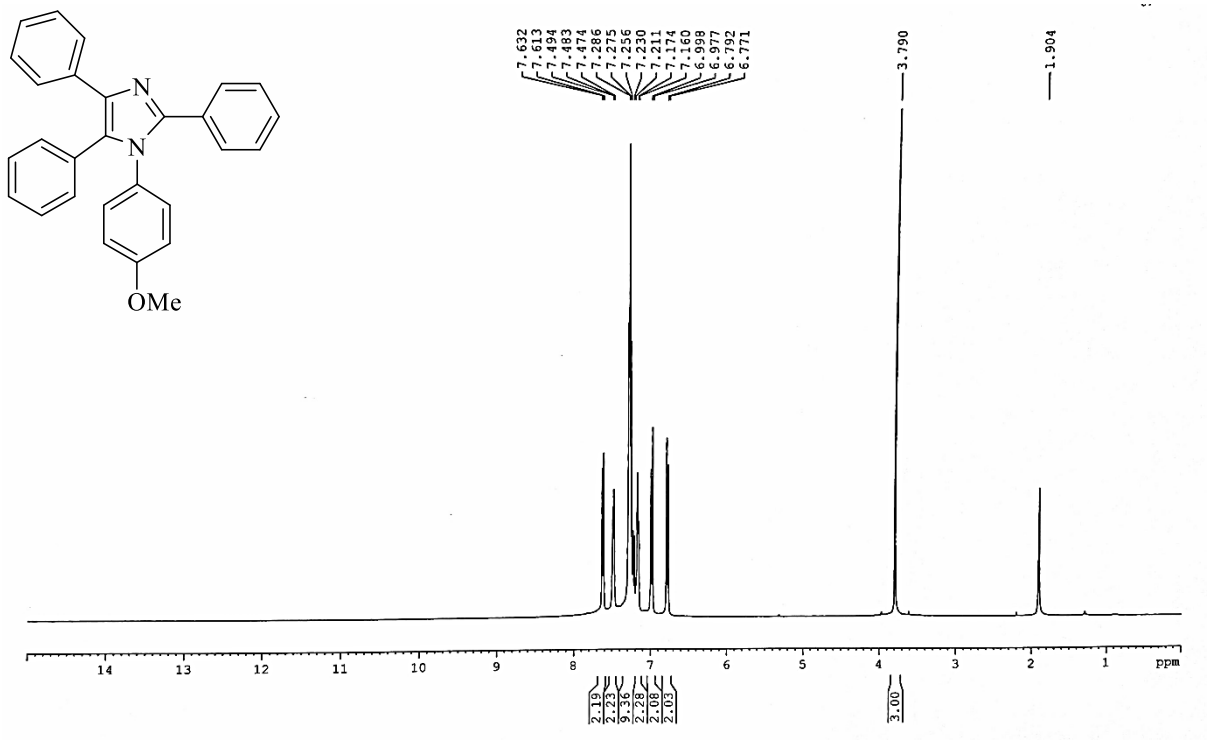
^{1}H NMR (400 MHz, $CDCl_3$) of (**5c**)



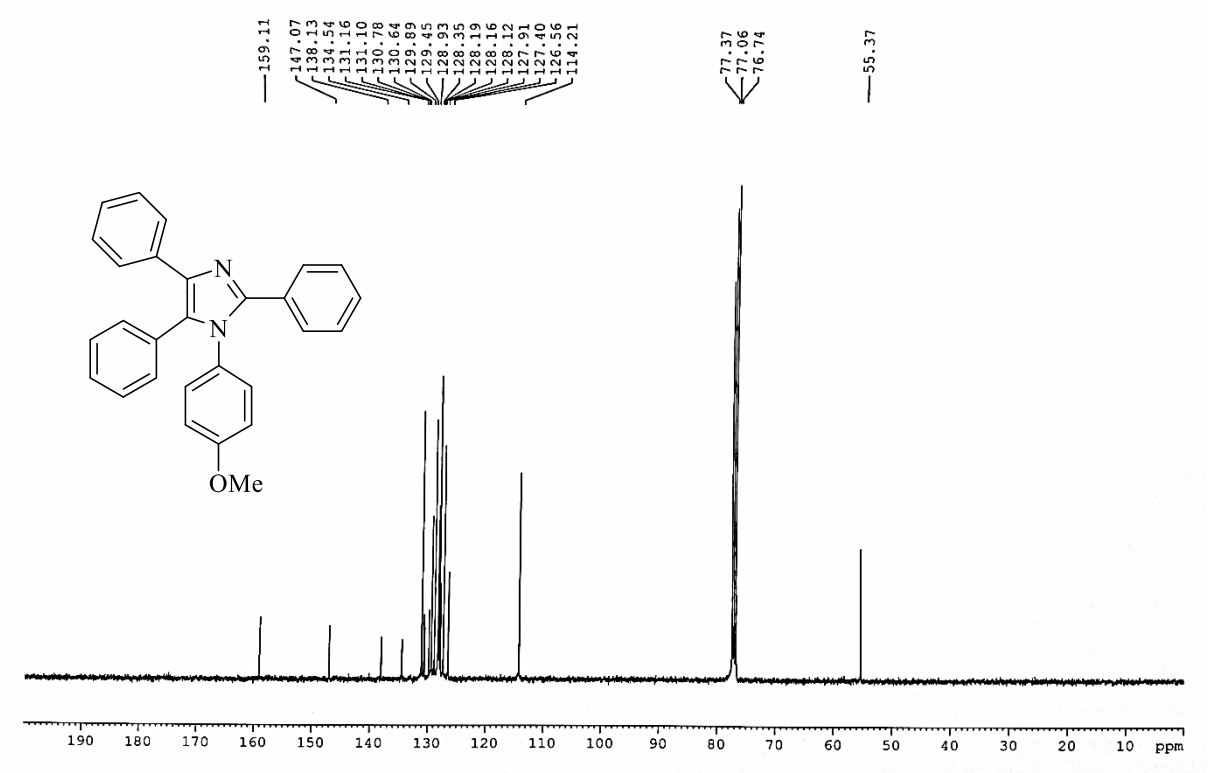
^{13}C NMR (100 MHz, $CDCl_3$) of (**5c**)



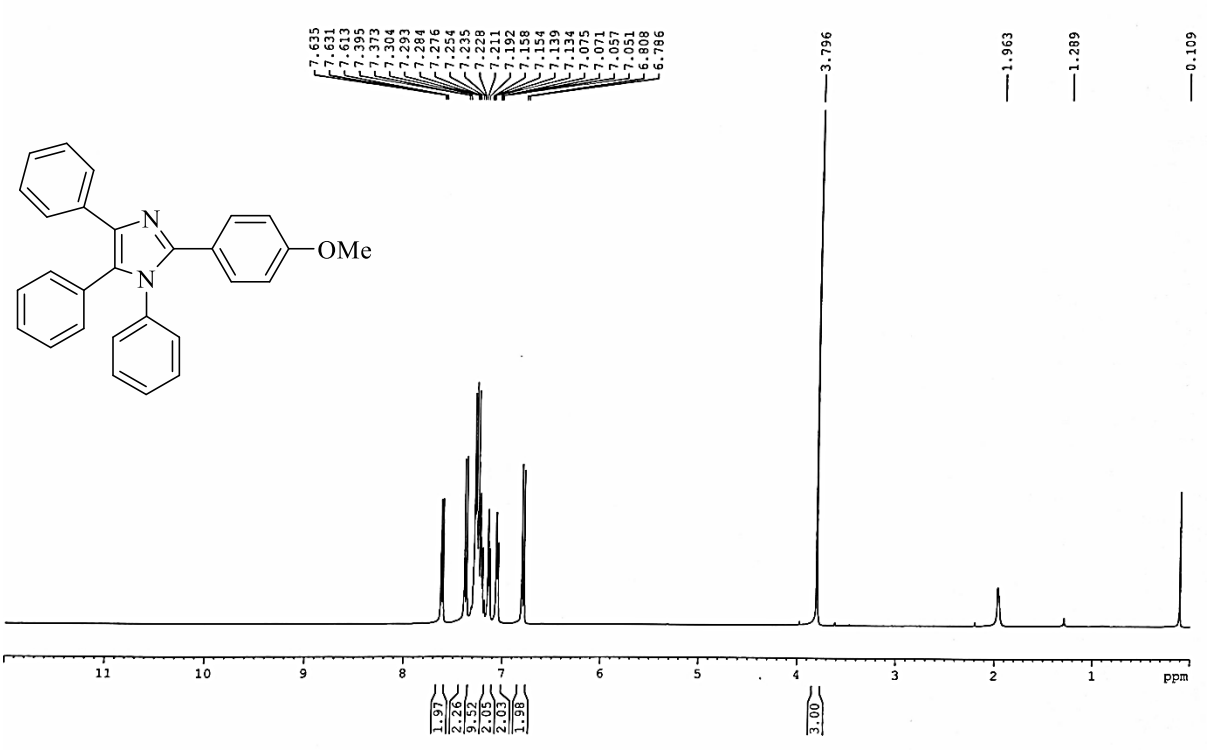
^{1}H NMR (400 MHz, $CDCl_3$) of (**5d**)



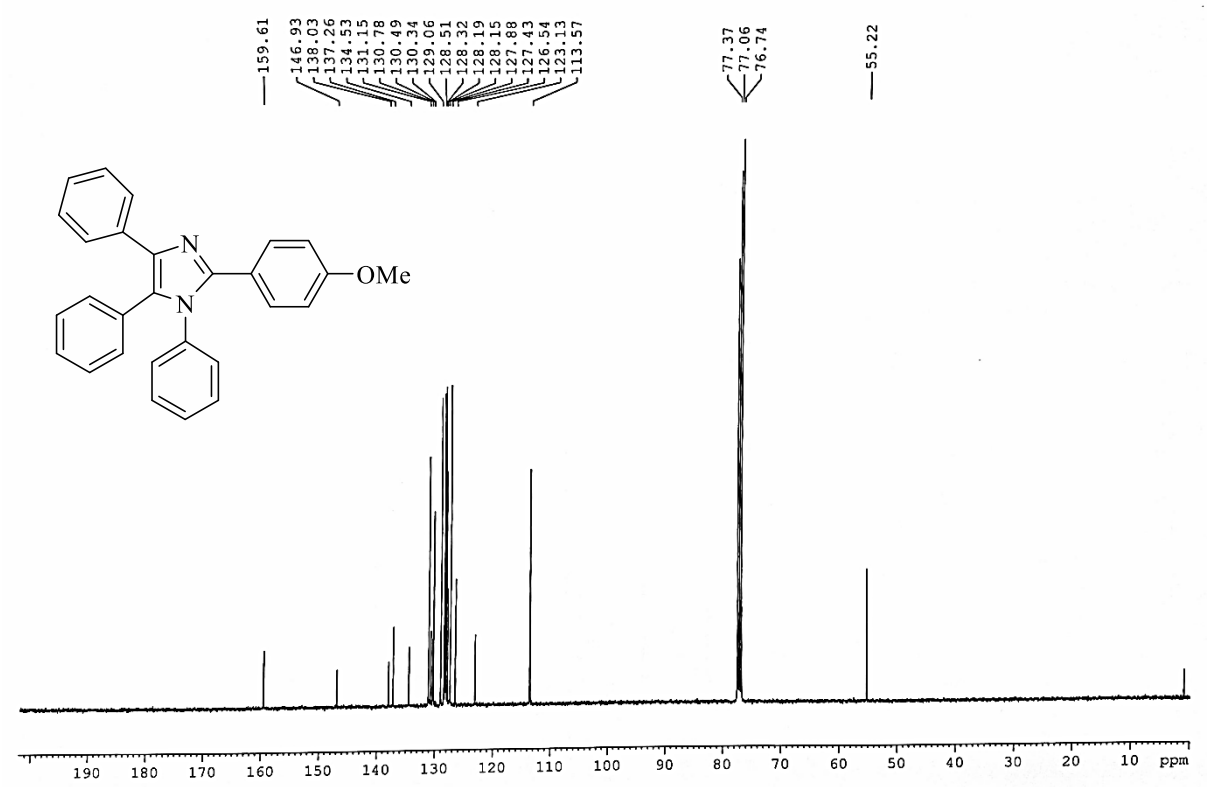
^{13}C NMR (100 MHz, $CDCl_3$) of (**5d**)



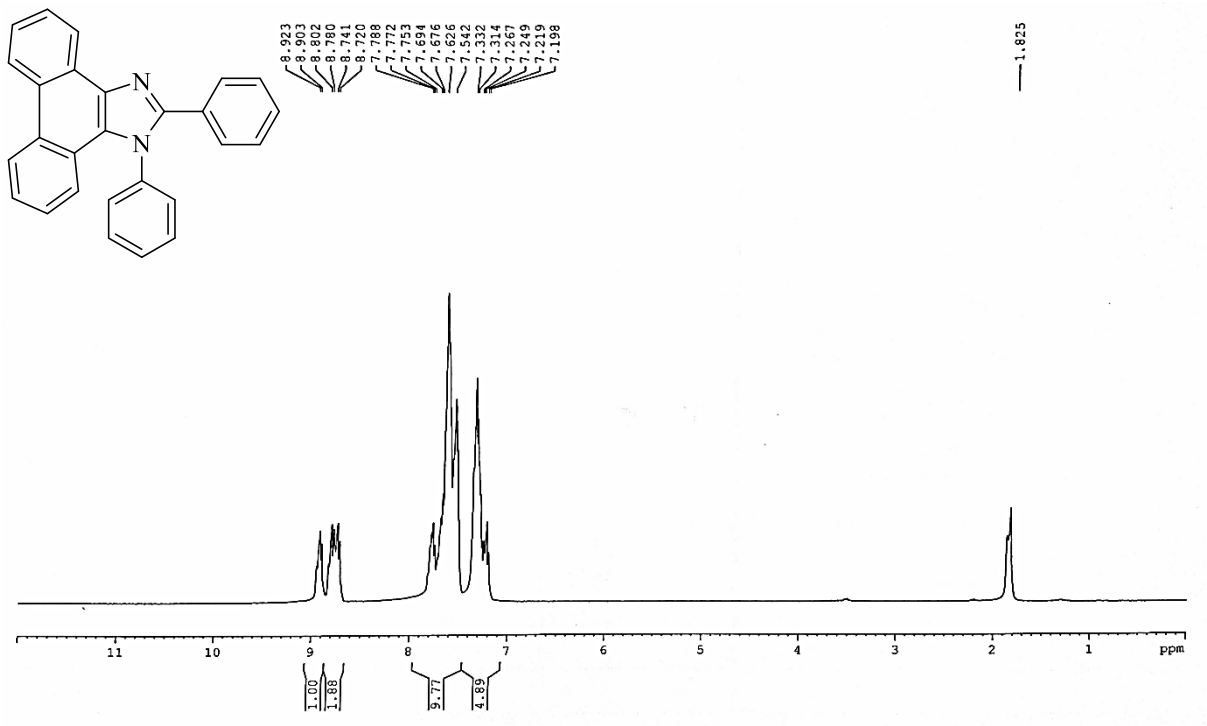
^{1}H NMR (400 MHz, $CDCl_3$) of (**5e**)



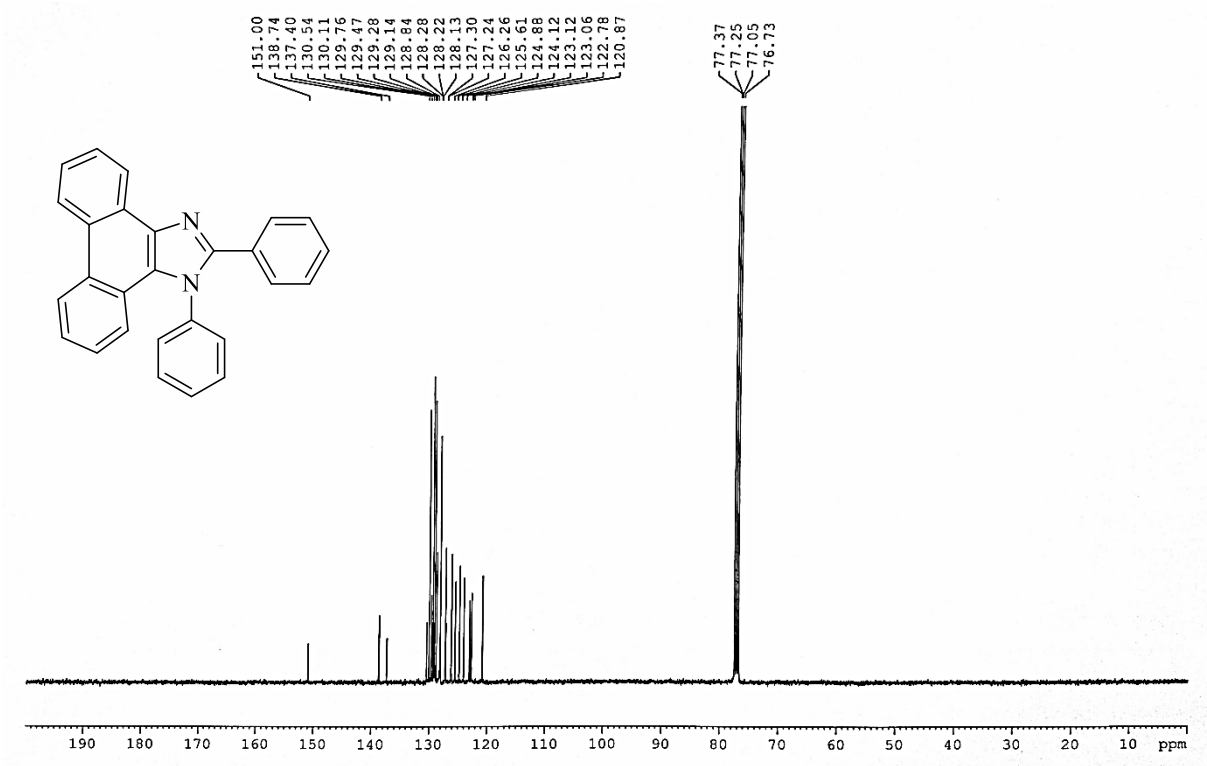
^{13}C NMR (100 MHz, $CDCl_3$) of (**5e**)



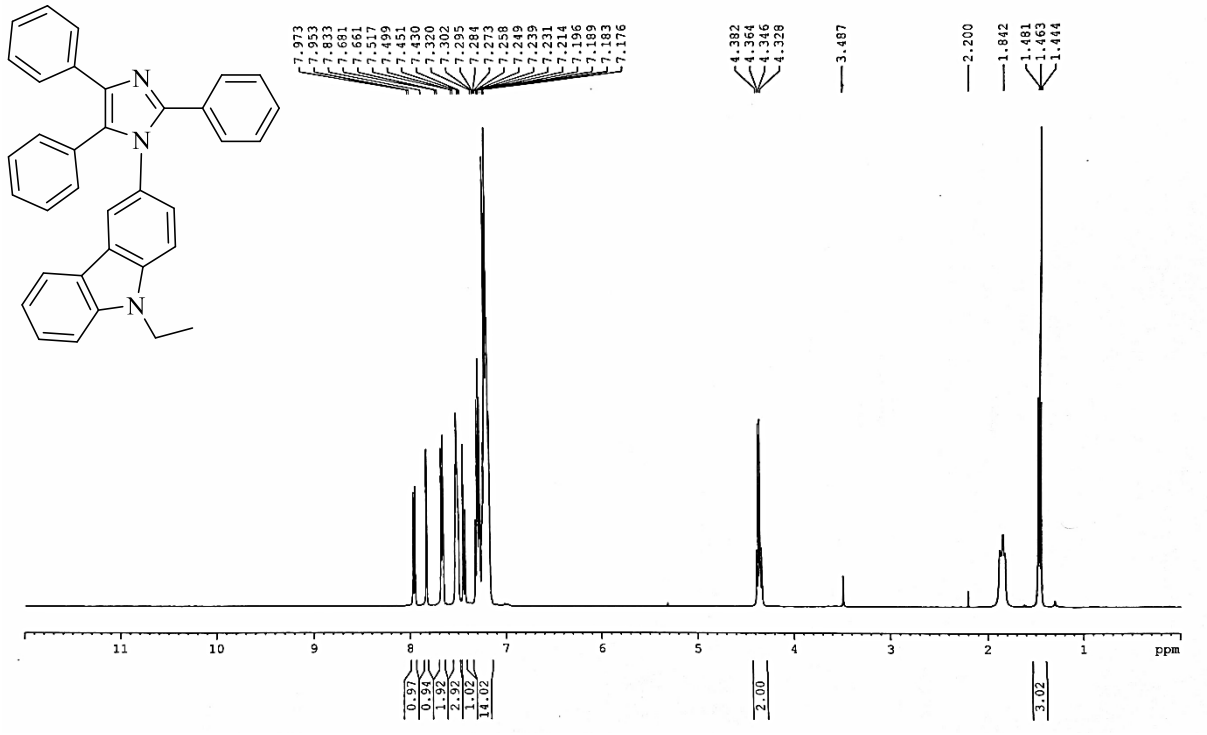
^{1}H NMR (400 MHz, $CDCl_3$) of (**5f**)



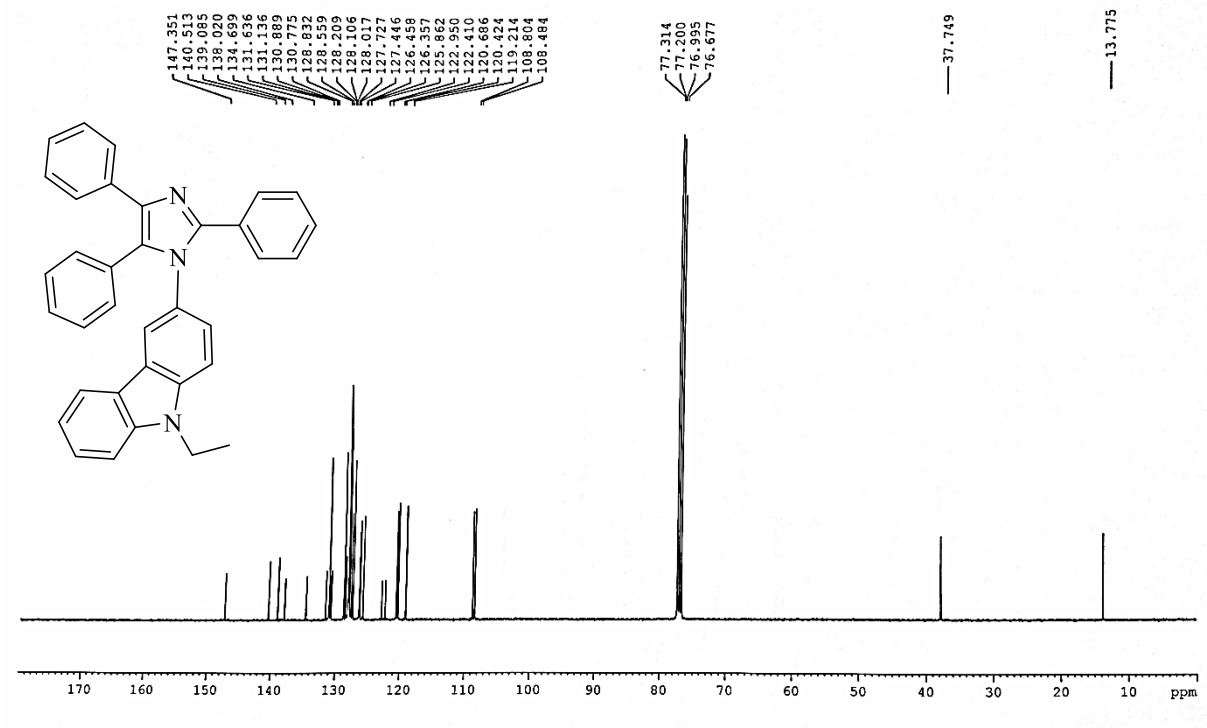
^{13}C NMR (100 MHz, $CDCl_3$) of (**5f)**



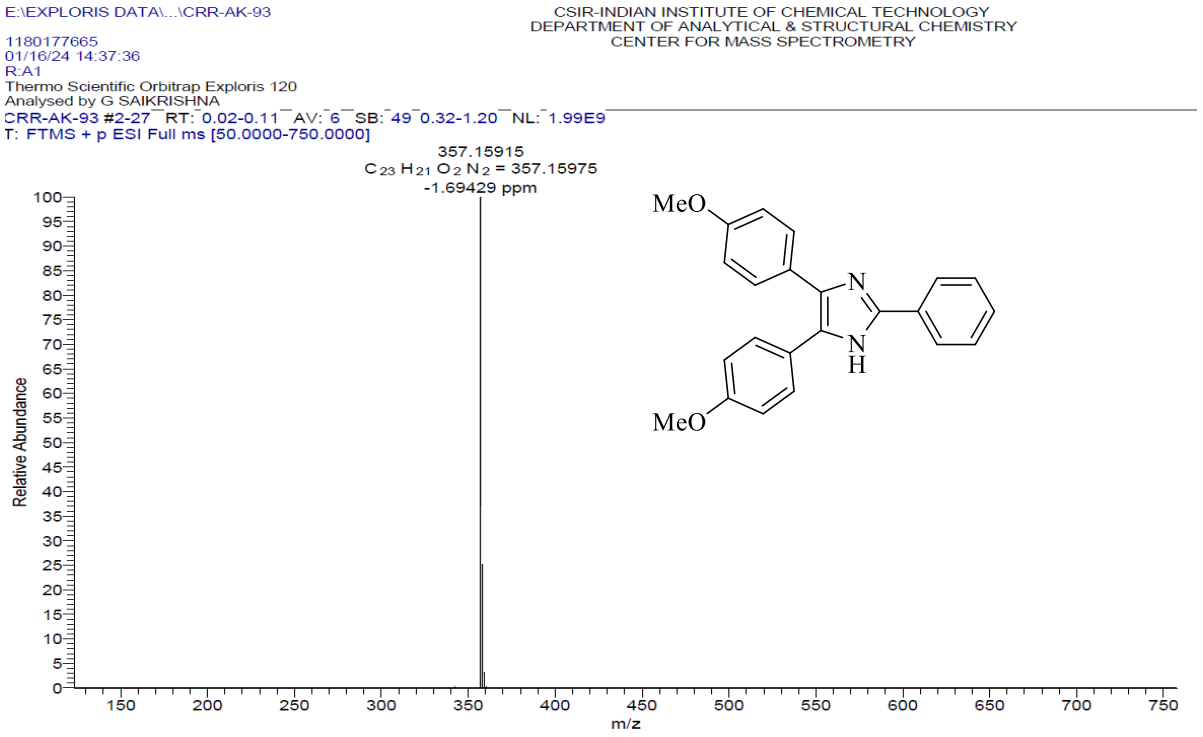
^{1}H NMR (400 MHz, $CDCl_3$) of (**5g**)



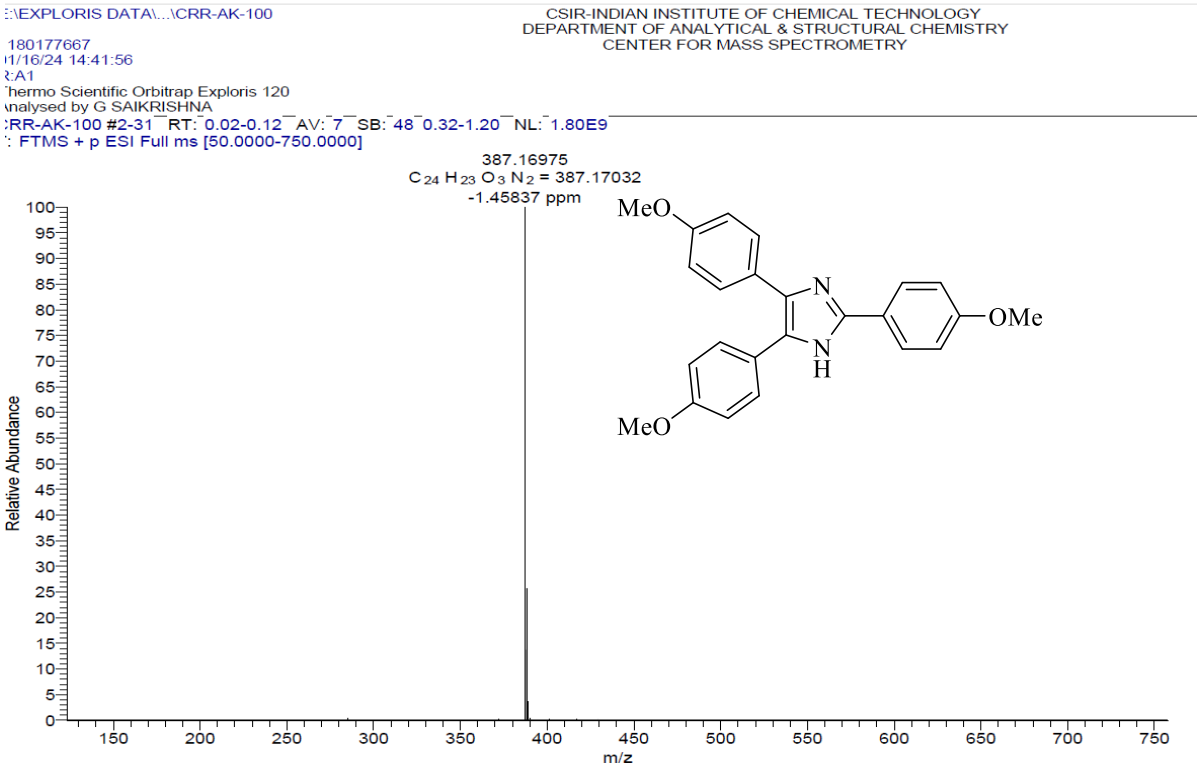
^{13}C NMR (100 MHz, $CDCl_3$) of (**5g**)



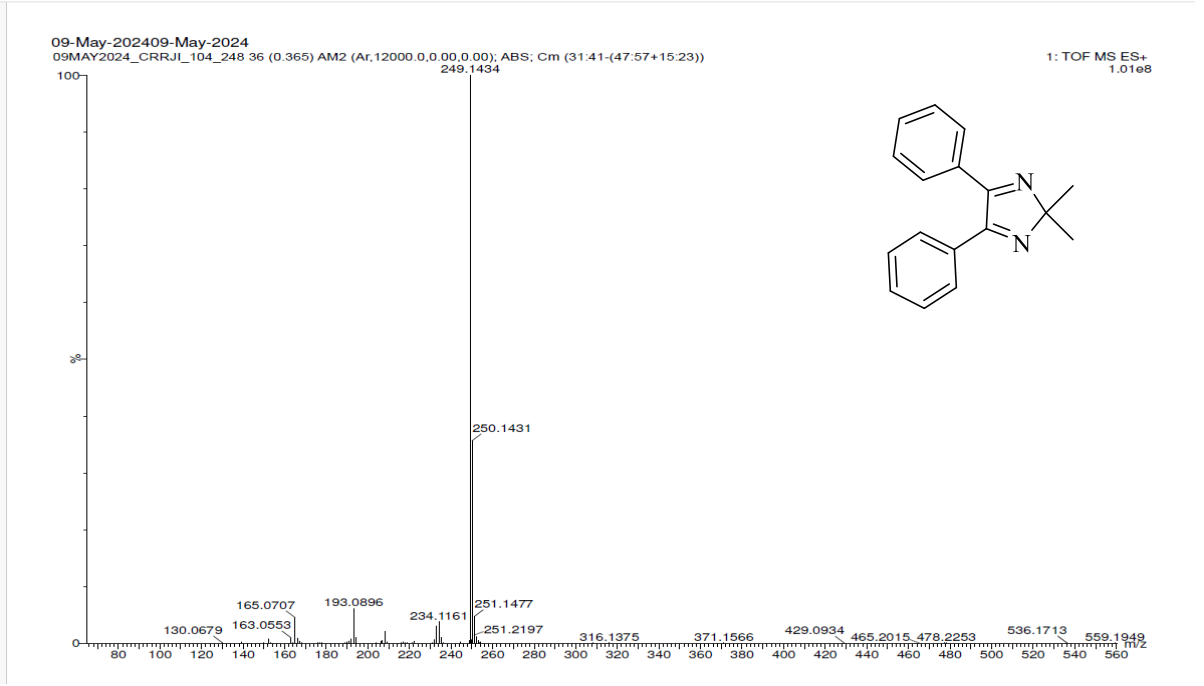
HRMS spectra of 3d



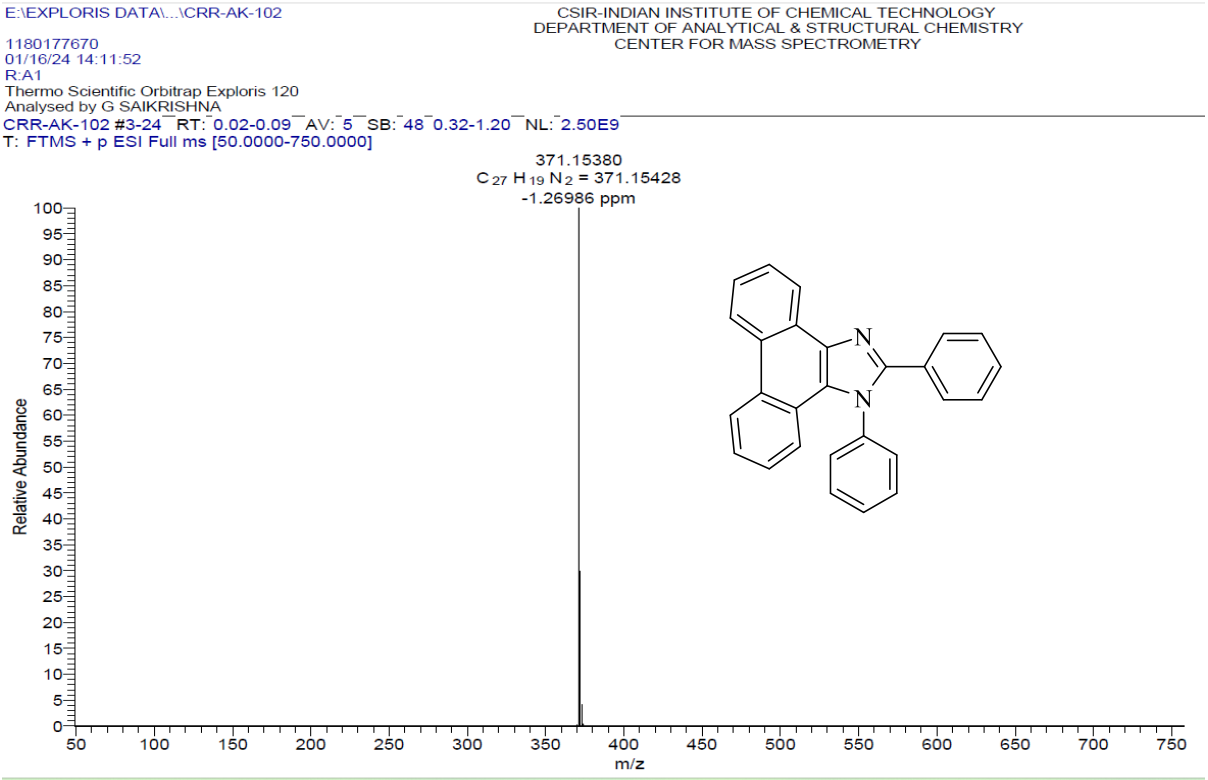
HRMS spectra of 3e



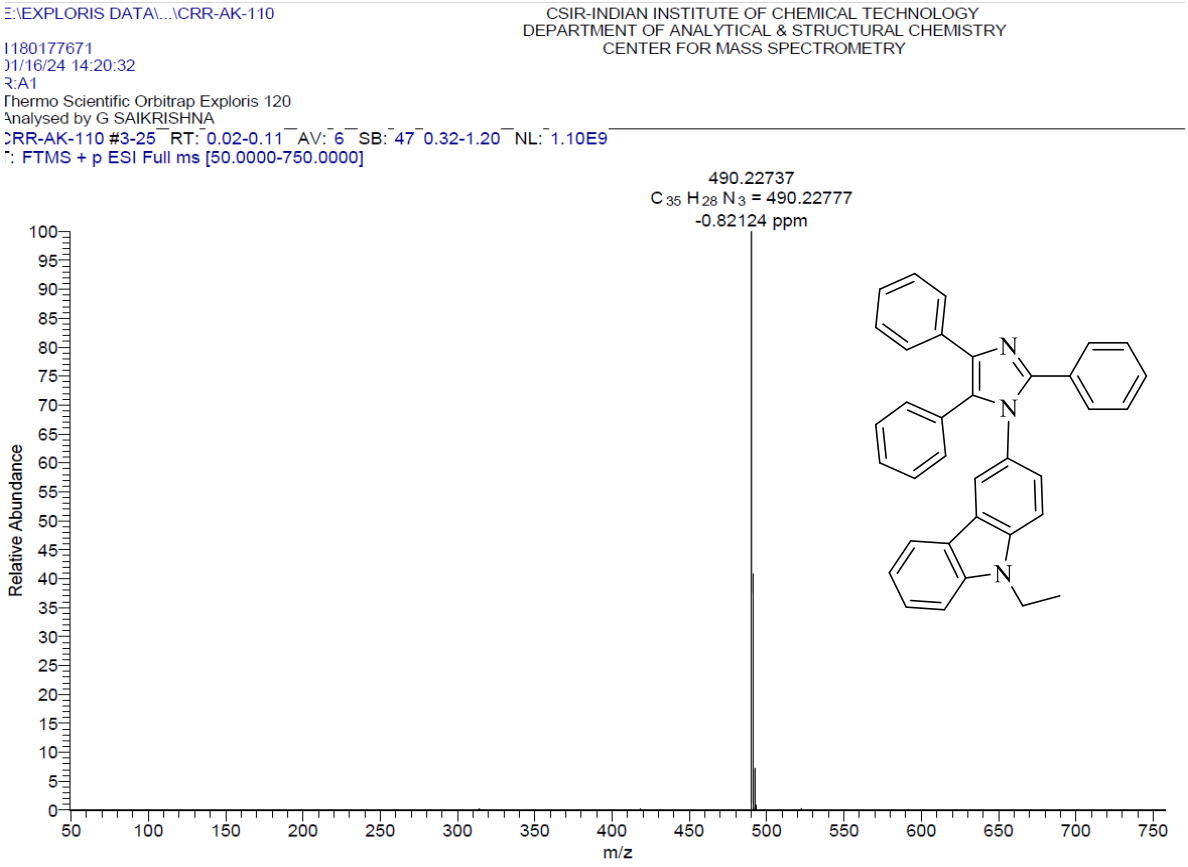
HRMS spectra of 3h



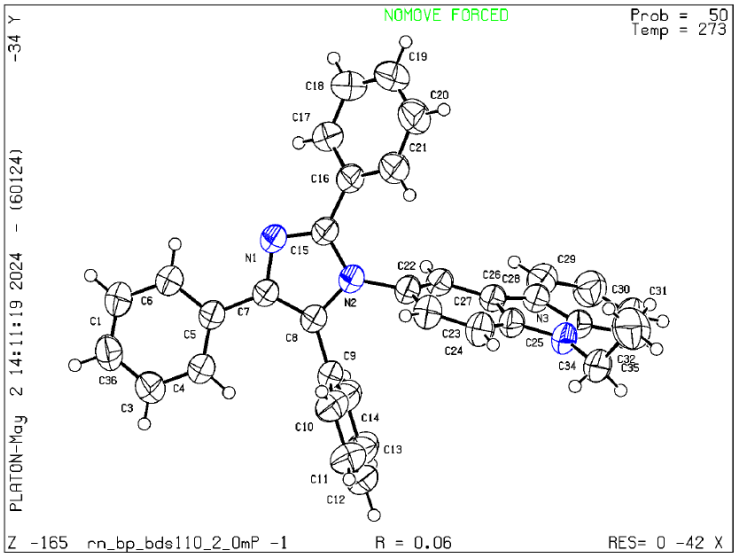
HRMS spectra of 5f



HRMS spectra of 5g



CCDC deposition number of 5g is 2361185



Figures for Supporting Information

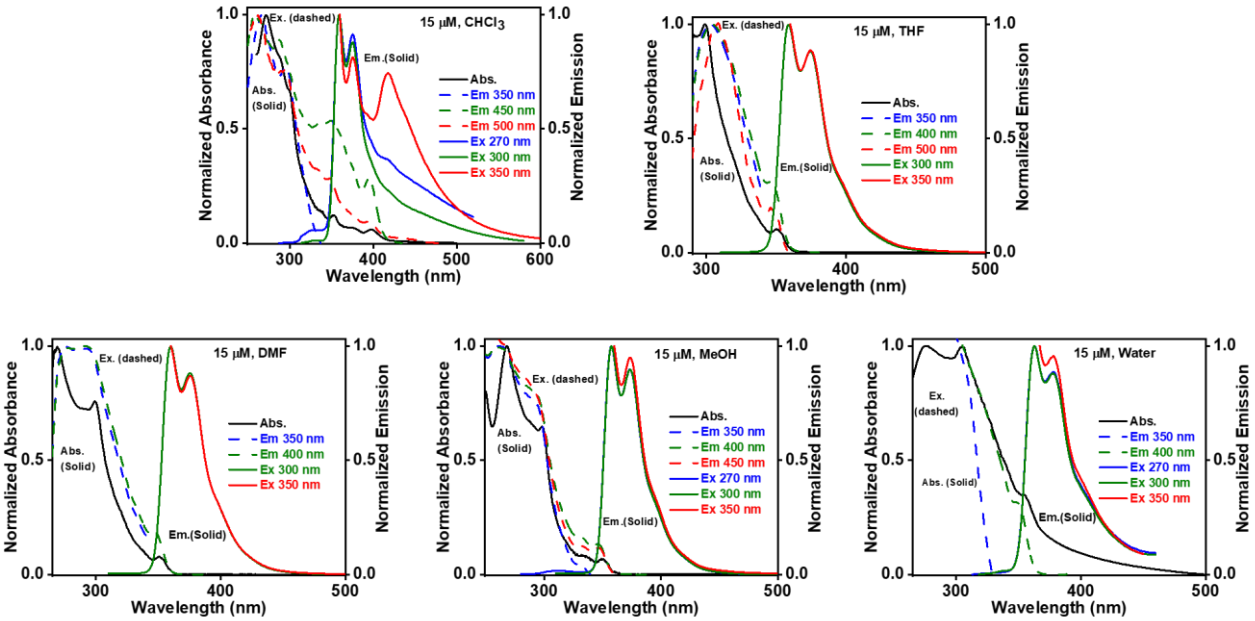


Figure S1. Absoprtion, emission and excitation spectra of **5g** in various solvents

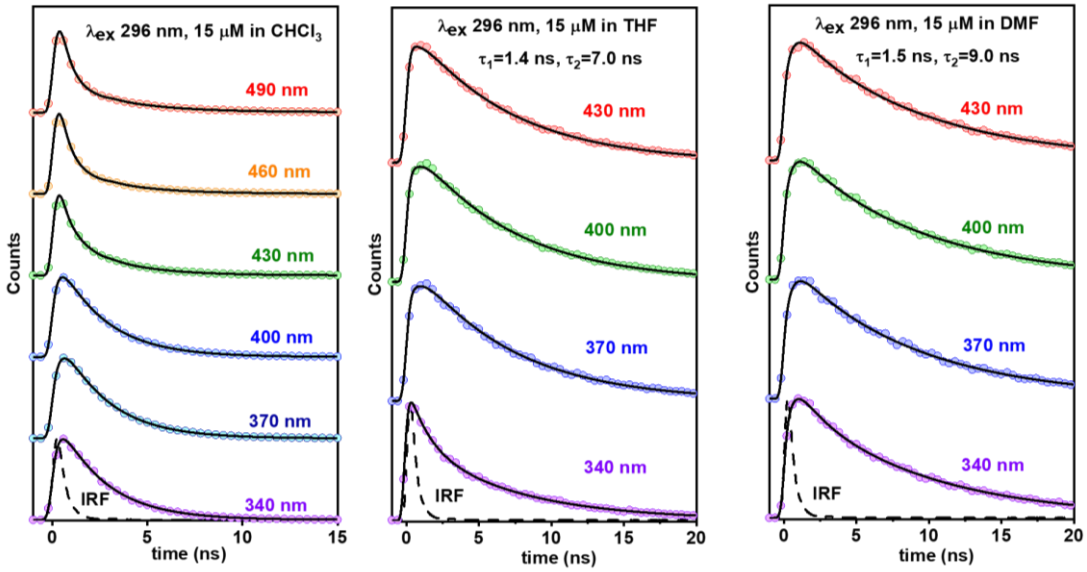


Figure S2. Lifetime emission decays of **5g** in aprotic solvents (15 μM). The excitation and monitoring wavelengths are provided in the insets.

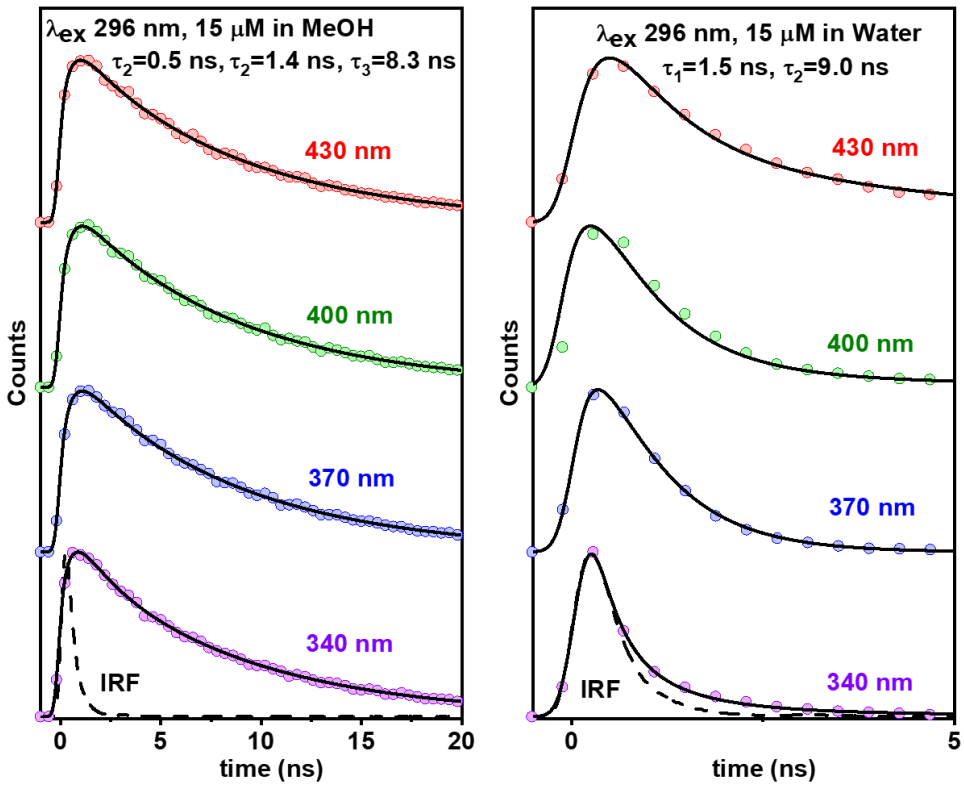


Figure S3. Lifetime emission decays of **5g** in protic solvents (15 μM). The excitation and monitoring wavelengths are provided in the insets.

Reference

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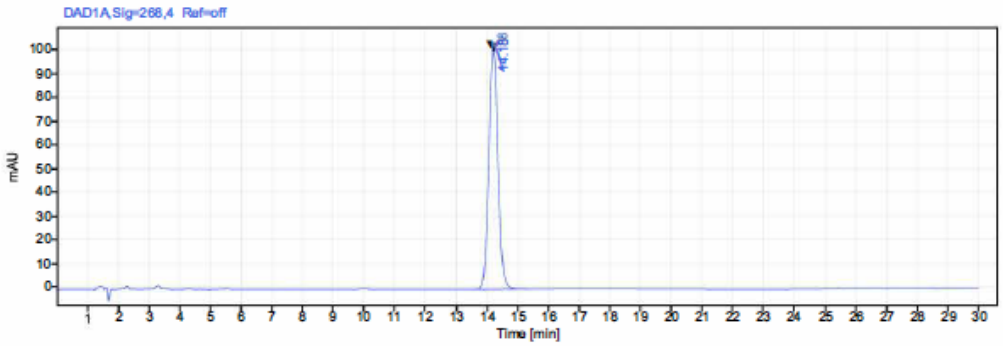
HPLC chromatogram of 5g

Single Injection Report

Agilent

Data file:	BDS-110_Peak Purity_10ug_100%ACN_2024-09-28 14-32-39+05-30.dx		
Sequence Name:	SingleSample	**Project Name:**	AcharyaLab
Sample name:	BDS-110_Peak Purity_10ug_100%ACN_2024-09-28 14:32:39+05:30	**Operator:**	SYSTEM (SYSTEM)
Instrument:	AcharyaLab-HPLC	**Injection date:**	2024-09-28 14:35:44+05:30
Inj. volume:	20.000 uL	**Location:**	
Acq. method:	Compound Screeing_Isocratic.amx	**Type:**	Sample
Processing method:	*3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	10.00 ug/mL
Manually modified:	None		



Signal: DAD1A,Sig=268,4 Ref=off

RT [min]	Type	Width [min]	Peak Purity	Area	Height	Area%	Name
14.188	BB	1.82	999.6	2080.37	99.72	100.00	BDS 110
			Sum	**2080.37**			

XRD data

RN_BP_BDS110_2_0m_a

Table 1 Crystal data and structure refinement for RN_BP_BDS110_2_0m_a.

Identification code	RN_BP_BDS110_2_0m_a
Empirical formula	$C_{36}H_{29}Cl_2N_3$
Formula weight	574.52
Temperature/K	273.15
Crystal system	triclinic
Space group	P-1
a/Å	10.137(2)
b/Å	11.456(3)
c/Å	13.238(3)
α/°	113.447(9)
β/°	91.736(10)
γ/°	95.668(10)
Volume/Å^3	1399.5(6)
Z	2
ρ_{calc}g/cm^3	1.363
μ/mm^{-1}	2.323
F(000)	600.0
Crystal size/mm^3	0.07 × 0.05 × 0.02
Radiation	Cu Kα (λ = 1.54178)
2Θ range for data collection/°	8.474 to 136.828
Index ranges	-12 ≤ h ≤ 11, -13 ≤ k ≤ 13, -15 ≤ l ≤ 15
Reflections collected	45395
Independent reflections	4889 [R_{int} = 0.0612, R_{sigma} = 0.0315]
Data/restraints/parameters	4889/0/344
Goodness-of-fit on F^2	1.033
Final R indexes [I>=2σ (I)]	R_1 = 0.0649, wR_2 = 0.2066
Final R indexes [all data]	R_1 = 0.0692, wR_2 = 0.2120
Largest diff. peak/hole / e Å^{-3}	0.16/-0.21

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for RN_BP_BDS110_2_0m_a. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
N1	6799.0(15)	8794.1(15)	4344.1(12)	55.5(4)
N2	4795.6(15)	8100.6(15)	3464.5(12)	55.9(4)
N3	-147.0(15)	5476.9(17)	1552.2(14)	62.4(4)
C1	10158(2)	10898(3)	3772(3)	89.7(8)
C36	10126(2)	11319(2)	2946(2)	76.5(6)
C3	8971(3)	11082(3)	2302(2)	92.8(8)
C4	7855(2)	10405(3)	2478(2)	89.0(8)
C5	7877.2(17)	9947.9(17)	3296.0(14)	54.6(4)
C6	9042(2)	10223(2)	3947(2)	75.8(6)
C7	6721.8(17)	9195.9(17)	3495.5(14)	53.1(4)
C8	5487.5(18)	8775.6(18)	2937.6(15)	56.3(4)
C9	4868.1(18)	8858(2)	1949.7(16)	61.7(5)
C10	4161(2)	9863(3)	2038(2)	83.0(7)
C11	3560(3)	9935(3)	1125(3)	105.1(10)
C12	3669(3)	8990(4)	109(3)	108.8(12)
C13	4371(3)	7989(4)	-2(2)	102.1(10)
C14	4971(2)	7915(3)	921.0(19)	81.7(7)
C15	5639.1(17)	8139.0(17)	4308.0(14)	54.6(4)
C16	5323.2(19)	7500.3(18)	5053.4(15)	58.6(5)
C17	6351(2)	7021(2)	5435.2(18)	69.6(5)
C18	6119(3)	6454(3)	6169(2)	82.2(7)
C19	4879(3)	6357(3)	6527(2)	87.5(7)
C20	3864(3)	6813(3)	6159(2)	85.9(7)
C21	4069(2)	7391(2)	5424.2(17)	71.1(6)
C22	3479.8(17)	7435.8(19)	3077.2(15)	56.0(4)
C23	2403.2(19)	8154(2)	3238.3(17)	64.9(5)
C24	1145(2)	7575(2)	2766.0(18)	66.5(5)
C25	983.8(17)	6267.8(19)	2144.8(16)	57.5(4)
C26	2050.2(17)	5525.6(18)	2014.9(14)	53.5(4)
C27	3313.5(17)	6133.6(18)	2479.5(15)	54.7(4)
C28	1525.7(18)	4221.5(19)	1324.6(15)	57.5(4)
C29	2079(2)	3073(2)	907.5(19)	71.3(5)
C30	1285(3)	1973(2)	203(2)	82.9(7)
C31	-33(3)	2011(2)	-78(2)	83.0(7)
C32	-623(2)	3132(2)	337.0(18)	71.8(6)
C33	177.4(18)	4239(2)	1046.8(15)	59.5(5)
C34	-1449.3(19)	5885(2)	1470(2)	73.1(6)
C35	-2352(3)	5757(3)	2307(3)	102.9(9)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for RN_BP_BDS110_2_0m_a. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	48.1(8)	64.5(9)	51.6(8)	23.8(7)	-4.9(6)	-2.8(6)
N2	46.2(8)	67.6(9)	52.9(8)	26.7(7)	-5.8(6)	-5.6(6)
N3	40.4(8)	78.7(11)	68.1(9)	32.9(8)	-3.6(7)	-4.2(7)
C1	53.5(12)	115(2)	113.5(19)	67.0(17)	-16.9(12)	-17.4(12)
C36	59.6(12)	83.9(14)	83.9(15)	36.6(12)	4.6(10)	-13.9(10)
C3	81.5(16)	125(2)	80.2(15)	60.8(15)	-10.7(12)	-31.6(14)
C4	66.1(13)	129(2)	80.8(15)	64.2(15)	-22.1(11)	-32.3(13)
C5	47.6(9)	59.1(10)	53.2(9)	21.0(8)	-3.1(7)	-1.9(7)
C6	54.4(11)	96.7(16)	87.9(15)	55.5(13)	-14.5(10)	-11.5(10)
C7	47.2(9)	61.2(10)	49.8(9)	23.5(7)	-2.9(7)	-0.7(7)
C8	48.6(9)	65.8(10)	53.5(9)	26.6(8)	-5.8(7)	-4.7(8)
C9	47.3(9)	78.8(12)	61.2(11)	36.0(9)	-11.1(8)	-12.1(8)
C10	77.0(15)	88.4(15)	88.5(15)	45.5(13)	-20.8(12)	-2.3(12)
C11	91.4(19)	125(2)	122(2)	81(2)	-34.0(17)	-8.9(17)
C12	70.4(16)	177(3)	106(2)	99(2)	-32.8(15)	-32.8(19)
C13	80.1(17)	159(3)	60.3(13)	44.0(16)	-8.9(12)	-15.0(18)
C14	67.4(13)	111.6(18)	61.4(12)	33.5(12)	-5.9(10)	-0.8(12)
C15	49.5(9)	62.9(10)	49.1(9)	22.4(8)	-3.1(7)	-0.7(8)
C16	58.1(10)	63.6(10)	49.7(9)	21.5(8)	-3.9(8)	-4.9(8)
C17	64.4(12)	84.3(14)	65.5(12)	38.5(10)	-3.5(9)	-0.9(10)
C18	90.4(16)	92.1(16)	76.2(14)	49.2(13)	-4.3(12)	2.4(13)
C19	97.8(18)	102.6(18)	73.7(14)	53.0(13)	1.1(13)	-9.0(14)
C20	78.5(15)	105.3(18)	76.9(15)	43.9(14)	13.0(12)	-9.0(13)
C21	64.3(12)	87.6(14)	62.7(12)	34.5(10)	5.2(9)	-2.9(10)
C22	43.8(9)	69.5(11)	53.3(9)	26.4(8)	-3.6(7)	-3.9(8)
C23	54.8(11)	65.2(11)	68.2(12)	21.6(9)	1.7(9)	1.4(8)
C24	49.1(10)	72.5(12)	74.9(13)	27.2(10)	4.0(9)	5.7(8)
C25	41.7(9)	73.3(11)	58.0(10)	29.6(9)	0.3(7)	-2.9(8)
C26	45.4(9)	66.5(10)	49.9(9)	27.0(8)	0.2(7)	-1.0(7)
C27	45.2(9)	68.2(11)	53.2(9)	28.4(8)	-0.6(7)	1.9(7)
C28	50.0(9)	69.1(11)	53.1(9)	27.2(8)	-1.3(7)	-3.6(8)
C29	67.2(12)	70.9(12)	72.9(13)	28.5(10)	-7.3(10)	1.7(10)
C30	87.5(16)	65.6(13)	86.4(15)	24.8(11)	-9.9(12)	-1.8(11)
C31	84.0(16)	77.6(14)	76.2(14)	27.3(11)	-15.0(12)	-19.2(12)
C32	58.8(11)	83.6(14)	69.2(12)	33.6(11)	-9.4(9)	-16.8(10)
C33	50.5(10)	74.2(12)	54.4(10)	30.7(9)	-2.2(8)	-8.5(8)
C34	42.8(10)	97.3(15)	86.0(14)	46.6(12)	-2.7(9)	0.0(9)
C35	69.9(15)	136(2)	116(2)	59.7(19)	35.4(15)	22.2(15)

Table 4 Bond Lengths for RN_BP_BDS110_2_0m_a.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N1	C7	1.376(2)	C13	C14	1.385(4)
N1	C15	1.320(2)	C15	C16	1.469(3)
N2	C8	1.389(2)	C16	C17	1.394(3)
N2	C15	1.370(2)	C16	C21	1.389(3)
N2	C22	1.438(2)	C17	C18	1.380(3)
N3	C25	1.387(2)	C18	C19	1.366(4)
N3	C33	1.385(3)	C19	C20	1.359(4)
N3	C34	1.458(3)	C20	C21	1.388(3)
C1	C36	1.359(3)	C22	C23	1.402(3)
C1	C6	1.385(3)	C22	C27	1.372(3)
C36	C3	1.365(3)	C23	C24	1.383(3)
C3	C4	1.386(3)	C24	C25	1.382(3)
C4	C5	1.378(3)	C25	C26	1.413(3)
C5	C6	1.372(3)	C26	C27	1.392(2)
C5	C7	1.478(2)	C26	C28	1.441(3)
C7	C8	1.373(2)	C28	C29	1.392(3)
C8	C9	1.474(3)	C28	C33	1.409(3)
C9	C10	1.383(3)	C29	C30	1.387(3)
C9	C14	1.375(3)	C30	C31	1.386(4)
C10	C11	1.372(4)	C31	C32	1.386(4)
C11	C12	1.367(5)	C32	C33	1.397(3)
C12	C13	1.370(5)	C34	C35	1.500(3)

Table 5 Bond Angles for RN_BP_BDS110_2_0m_a.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C15	N1	C7	106.61(15)	C17	C16	C15	118.00(18)
C8	N2	C22	122.84(15)	C21	C16	C15	123.36(19)
C15	N2	C8	106.83(15)	C21	C16	C17	118.61(19)
C15	N2	C22	130.17(16)	C18	C17	C16	120.4(2)
C25	N3	C34	125.72(18)	C19	C18	C17	120.2(2)
C33	N3	C25	108.28(16)	C20	C19	C18	120.2(2)
C33	N3	C34	126.01(17)	C19	C20	C21	120.8(2)
C36	C1	C6	120.5(2)	C20	C21	C16	119.8(2)
C1	C36	C3	119.0(2)	C23	C22	N2	118.77(17)
C36	C3	C4	120.4(2)	C27	C22	N2	119.72(17)
C5	C4	C3	121.4(2)	C27	C22	C23	121.27(17)
C4	C5	C7	123.25(17)	C24	C23	C22	120.87(19)
C6	C5	C4	117.00(19)	C25	C24	C23	117.80(19)
C6	C5	C7	119.75(17)	N3	C25	C26	109.08(17)
C5	C6	C1	121.7(2)	C24	C25	N3	129.09(18)
N1	C7	C5	120.36(15)	C24	C25	C26	121.83(17)
C8	C7	N1	109.63(16)	C25	C26	C28	106.64(16)
C8	C7	C5	130.00(16)	C27	C26	C25	119.26(17)
N2	C8	C9	120.19(16)	C27	C26	C28	133.99(17)
C7	C8	N2	105.92(16)	C22	C27	C26	118.89(17)
C7	C8	C9	133.81(17)	C29	C28	C26	133.71(18)
C10	C9	C8	121.1(2)	C29	C28	C33	119.74(18)
C14	C9	C8	120.0(2)	C33	C28	C26	106.53(17)
C14	C9	C10	118.9(2)	C30	C29	C28	118.6(2)
C11	C10	C9	121.5(3)	C31	C30	C29	120.9(2)
C12	C11	C10	118.9(3)	C32	C31	C30	122.0(2)
C11	C12	C13	120.7(2)	C31	C32	C33	117.0(2)
C12	C13	C14	120.2(3)	N3	C33	C28	109.45(16)
C9	C14	C13	119.7(3)	N3	C33	C32	128.81(19)
N1	C15	N2	111.01(16)	C32	C33	C28	121.7(2)
N1	C15	C16	124.03(16)	N3	C34	C35	113.5(2)
N2	C15	C16	124.93(16)				

Table 6 Torsion Angles for RN_BP_BDS110_2_0m_a.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
N1	C7	C8	N2	0.1(2)	C15	N2	C22	C27	-74.3(3)
N1	C7	C8	C9	-176.6(2)	C15	C16	C17	C18	177.6(2)
N1	C15	C16	C17	-31.6(3)	C15	C16	C21	C20	-177.7(2)
N1	C15	C16	C21	146.1(2)	C16	C17	C18	C19	0.1(4)
N2	C8	C9	C10	92.0(2)	C17	C16	C21	C20	0.0(3)
N2	C8	C9	C14	-87.0(2)	C17	C18	C19	C20	0.3(4)
N2	C15	C16	C17	146.0(2)	C18	C19	C20	C21	-0.6(4)
N2	C15	C16	C21	-36.3(3)	C19	C20	C21	C16	0.4(4)
N2	C22	C23	C24	172.13(18)	C21	C16	C17	C18	-0.2(3)
N2	C22	C27	C26	-173.45(15)	C22	N2	C8	C7	-175.94(16)
N3	C25	C26	C27	175.81(15)	C22	N2	C8	C9	1.3(3)
N3	C25	C26	C28	-1.0(2)	C22	N2	C15	N1	175.60(17)
C1	C36	C3	C4	-0.9(4)	C22	N2	C15	C16	-2.3(3)
C36	C1	C6	C5	0.5(4)	C22	C23	C24	C25	0.8(3)
C36	C3	C4	C5	-0.4(5)	C23	C22	C27	C26	0.9(3)
C3	C4	C5	C6	1.8(4)	C23	C24	C25	N3	-176.93(19)
C3	C4	C5	C7	-178.5(2)	C23	C24	C25	C26	1.9(3)
C4	C5	C6	C1	-1.8(4)	C24	C25	C26	C27	-3.2(3)
C4	C5	C7	N1	-178.4(2)	C24	C25	C26	C28	-179.97(18)
C4	C5	C7	C8	2.5(3)	C25	N3	C33	C28	0.8(2)
C5	C7	C8	N2	179.23(18)	C25	N3	C33	C32	-177.42(19)
C5	C7	C8	C9	2.5(4)	C25	N3	C34	C35	-93.7(3)
C6	C1	C36	C3	0.9(4)	C25	C26	C27	C22	1.8(3)
C6	C5	C7	N1	1.4(3)	C25	C26	C28	C29	179.7(2)
C6	C5	C7	C8	-177.7(2)	C25	C26	C28	C33	1.4(2)
C7	N1	C15	N2	-0.3(2)	C26	C28	C29	C30	-176.6(2)
C7	N1	C15	C16	177.67(17)	C26	C28	C33	N3	-1.3(2)
C7	C5	C6	C1	178.4(2)	C26	C28	C33	C32	177.00(17)
C7	C8	C9	C10	-91.7(3)	C27	C22	C23	C24	-2.3(3)
C7	C8	C9	C14	89.3(3)	C27	C26	C28	C29	3.6(4)
C8	N2	C15	N1	0.3(2)	C27	C26	C28	C33	-174.69(19)
C8	N2	C15	C16	-177.60(17)	C28	C26	C27	C22	177.45(19)
C8	N2	C22	C23	-74.2(2)	C28	C29	C30	C31	-0.3(4)
C8	N2	C22	C27	100.3(2)	C29	C28	C33	N3	-179.89(17)
C8	C9	C10	C11	-178.8(2)	C29	C28	C33	C32	-1.6(3)
C8	C9	C14	C13	179.1(2)	C29	C30	C31	C32	-0.9(4)
C9	C10	C11	C12	-0.1(4)	C30	C31	C32	C33	0.8(4)
C10	C9	C14	C13	0.1(3)	C31	C32	C33	N3	178.4(2)
C10	C11	C12	C13	-0.3(4)	C31	C32	C33	C28	0.4(3)
C11	C12	C13	C14	0.6(4)	C33	N3	C25	C24	179.1(2)
C12	C13	C14	C9	-0.5(4)	C33	N3	C25	C26	0.1(2)
C14	C9	C10	C11	0.2(4)	C33	N3	C34	C35	86.8(3)
C15	N1	C7	C5	-179.13(16)	C33	C28	C29	C30	1.5(3)
C15	N1	C7	C8	0.1(2)	C34	N3	C25	C24	-0.5(3)
C15	N2	C8	C7	-0.2(2)	C34	N3	C25	C26	-179.38(17)
C15	N2	C8	C9	177.01(18)	C34	N3	C33	C28	-179.72(18)
C15	N2	C22	C23	111.2(2)	C34	N3	C33	C32	2.1(3)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for RN_BP_BDS110_2_0m_a.

Atom	*x*	*y*	*z*	U(eq)
H1	10935.7	11065	4222.24	108
H36	10879.43	11762.39	2822.39	92
H3	8933.12	11375.76	1741.54	111
H4	7074.52	10257.23	2035.19	107
H6	9082.28	9949.78	4519.58	91
H10	4091.22	10503.77	2731.7	100
H11	3086.45	10615.19	1197.52	126
H12	3261.63	9027.81	-514.99	131
H13	4444.2	7357.28	-699.07	123
H14	5440.59	7231.82	845.64	98
H17	7196.74	7082.8	5194.11	84
H18	6809.56	6137.18	6420.31	99
H19	4730.07	5978.92	7024.29	105
H20	3021.45	6735.35	6401.91	103
H21	3368.35	7704.31	5181.01	85
H23	2536.91	9030.29	3668.5	78
H24	430.64	8050.3	2863.26	80
H27	4032.71	5665	2386.2	66
H29	2962.13	3043.65	1097.04	86
H30	1642.4	1199.88	-84.16	99
H31	-537.73	1260.2	-560.63	100
H32	-1511.5	3147.63	151.42	86
H34A	-1871.44	5377.31	736.42	88
H34B	-1330.82	6773.61	1563.26	88
H35A	-2371.2	4912.22	2294.41	154
H35B	-3232.89	5897.29	2132.95	154
H35C	-2028.94	6380.49	3028.34	154

Table 8 Solvent masks information for RN_BP_BDS110_2_0m_a.

Number	X	Y	Z	Volume	Electron count	Content
1	0.000	0.500	0.500	121.5	59.9	2 CH2Cl2
2	0.500	0.500	0.000	28.7	7.5	?

Experimental

Single crystals of $C_{36}H_{29}Cl_2N_3$ **[RN_BP_BDS110_2_0m_a]** were **[]**. A suitable crystal was selected and **[]** on a **Bruker APEX3_IMuS Diamond_Photon III** diffractometer. The crystal was kept at 273.15 K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [RN_BP_BDS110_2_0m_a]

Crystal Data for $C_{36}H_{29}Cl_2N_3$ (M =574.52 g/mol): triclinic, space group P-1 (no. 2), a = 10.137(2) Å, b = 11.456(3) Å, c = 13.238(3) Å, α = 113.447(9)°, β = 91.736(10)°, γ = 95.668(10)°, V = 1399.5(6) Å^3, Z = 2, T = 273.15 K, μ(Cu Kα) = 2.323 mm^{-1}, *Dcalc* = 1.363 g/cm^3, 45395 reflections measured (8.474° ≤ 2Θ ≤ 136.828°), 4889 unique (R_{int} = 0.0612, R_{sigma} = 0.0315) which were used in all calculations. The final R_1 was 0.0649 (I > 2σ(I)) and wR_2 was 0.2120 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.
Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All C(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Secondary CH2 refined with riding coordinates:
 C34(H34A,H34B)
2.b Aromatic/amide H refined with riding coordinates:
 C1(H1), C36(H36), C3(H3), C4(H4), C6(H6), C10(H10), C11(H11), C12(H12),
 C13(H13), C14(H14), C17(H17), C18(H18), C19(H19), C20(H20), C21(H21), C23(H23),
  C24(H24), C27(H27), C29(H29), C30(H30), C31(H31), C32(H32)
2.c Idealised Me refined as rotating group:
 C35(H35A,H35B,H35C)
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