Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

Synthesis of Novel Series of 7,7'-(substituted methylene)bis-imidazo[1,2-*b*]pyrazoles via an Acid Catalyzed One-pot Three-component Reaction

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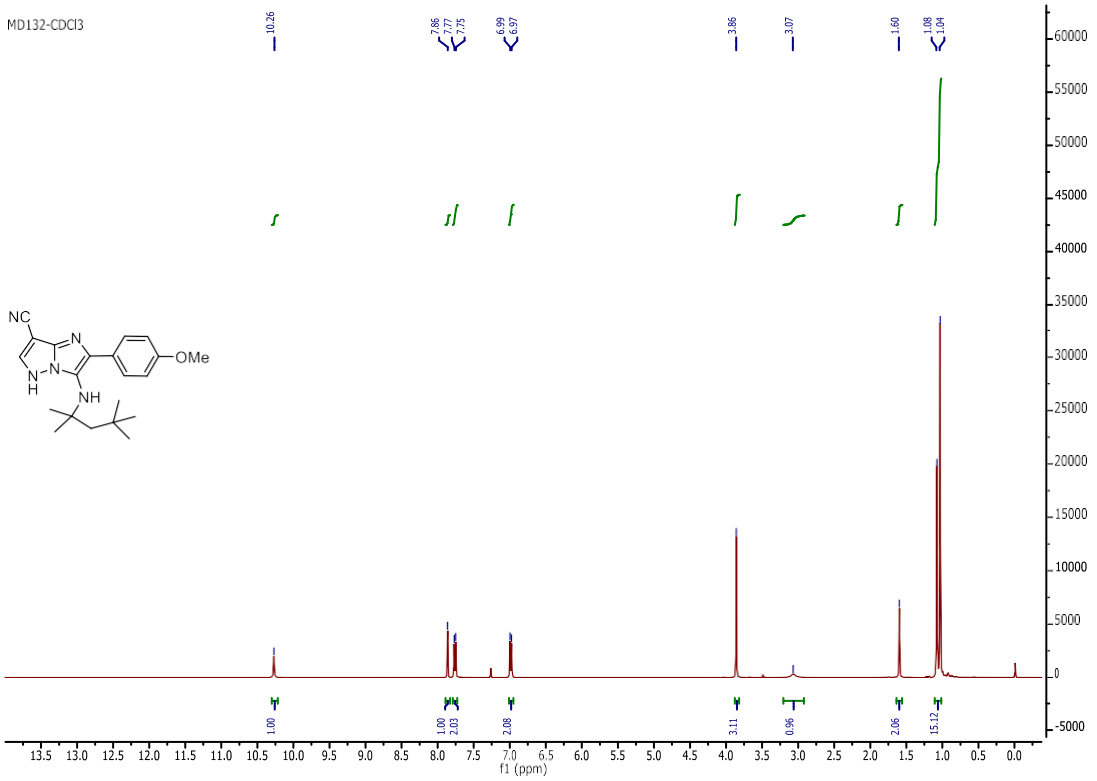
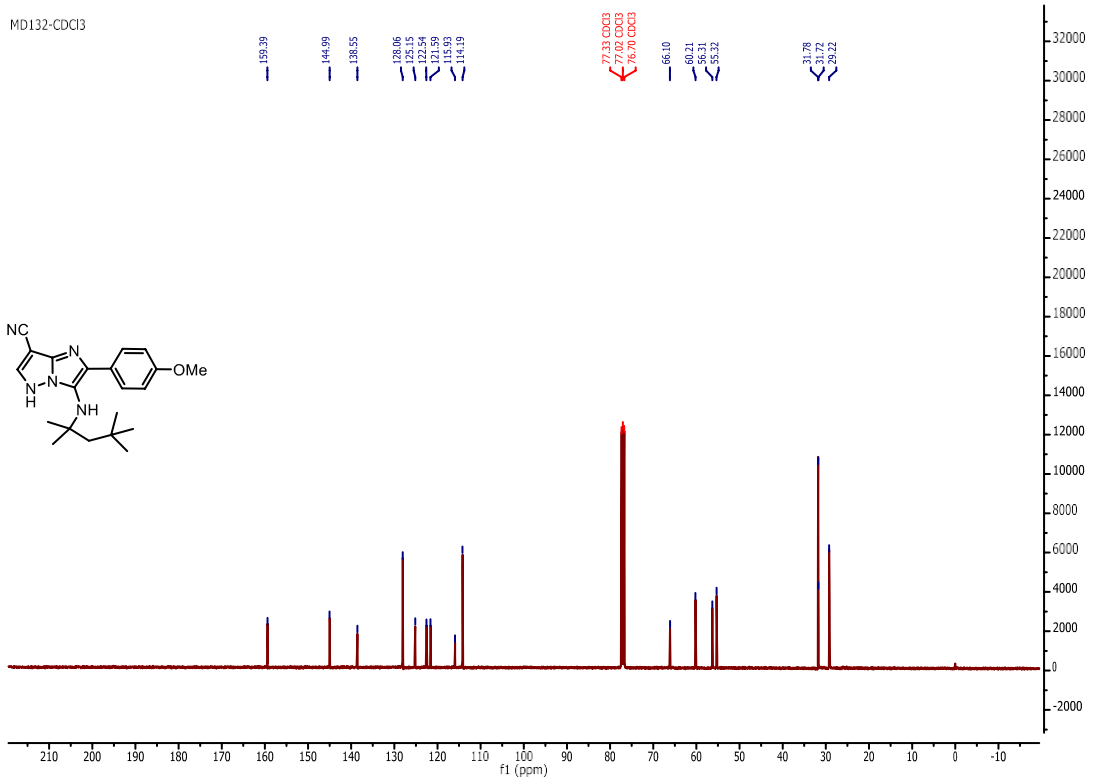
2-(4-Methoxyphenyl)-3-((2,4,4-trimethylpentan-2-yl)amino)-5*H*-imidazo[1,2-*b*]pyrazole-7-carbonitrile (**4a**):

^{1}H NMR (400 MHz, $CDCl_3$)

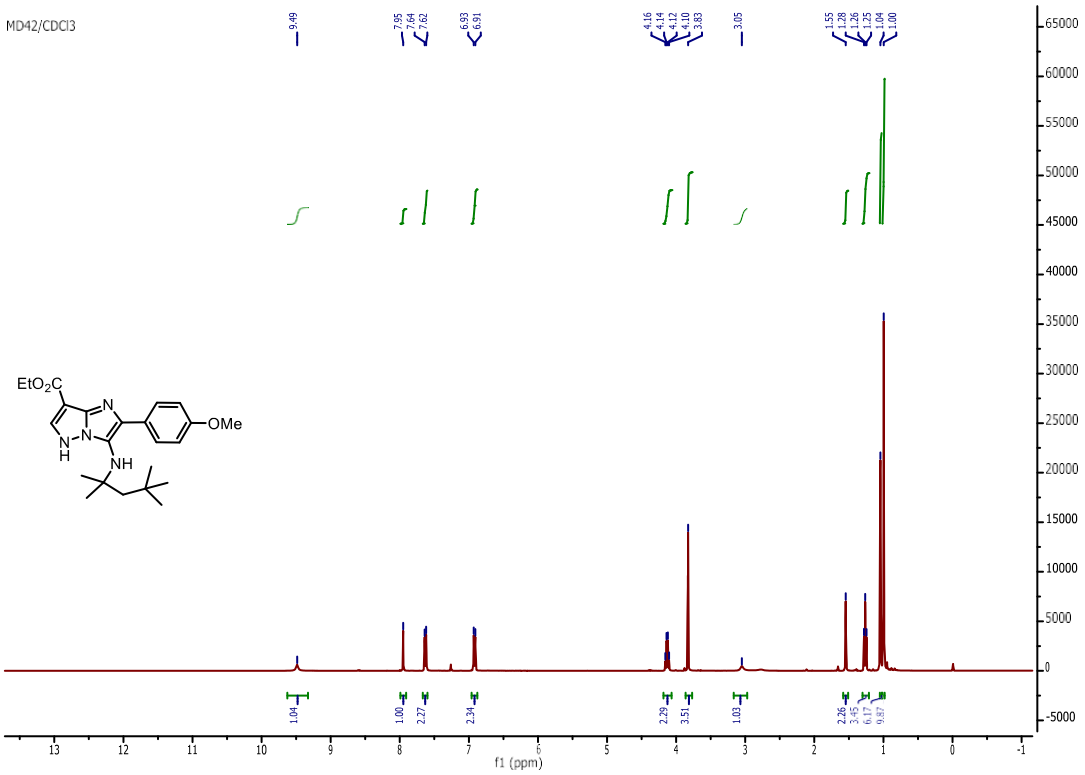


^{13}C NMR (100 MHz, $CDCl_3$)

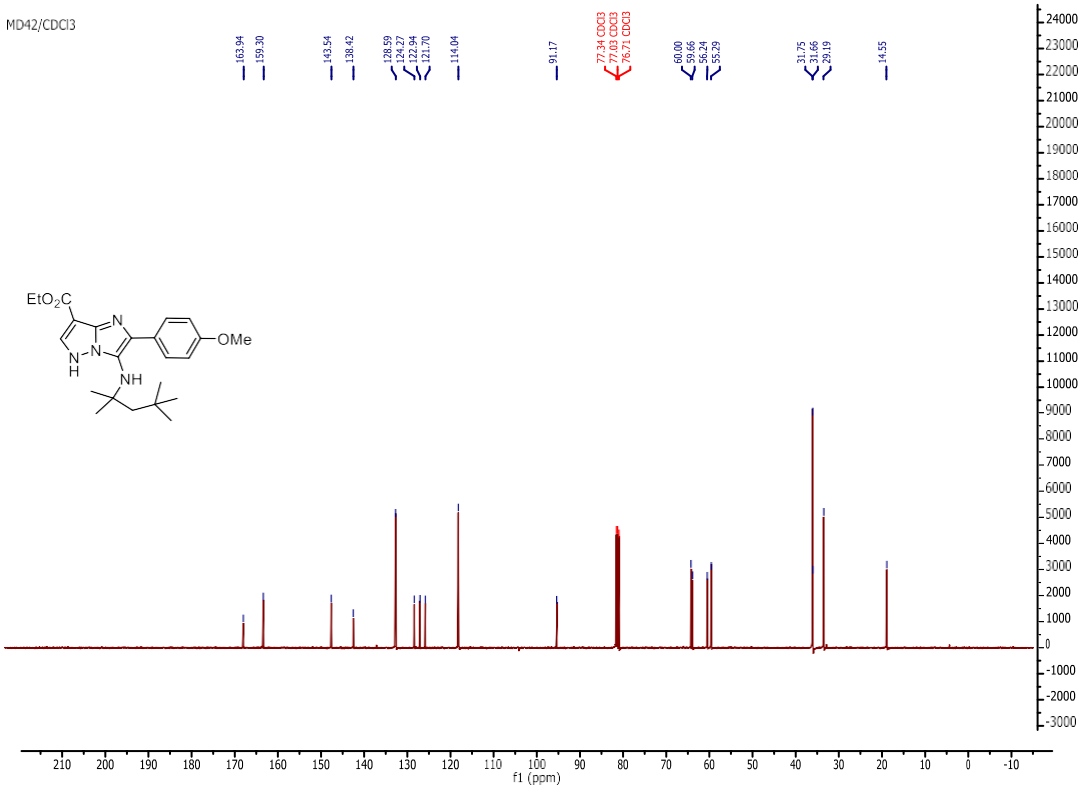


Ethyl 2-(4-methoxyphenyl)-3-((2,4,4-trimethylpentan-2-yl)amino)-5*H*-imidazo[1,2-*b*]pyrazole-7-carboxylate (**4b**):

^{1}H NMR (400 MHz, $CDCl_3$)

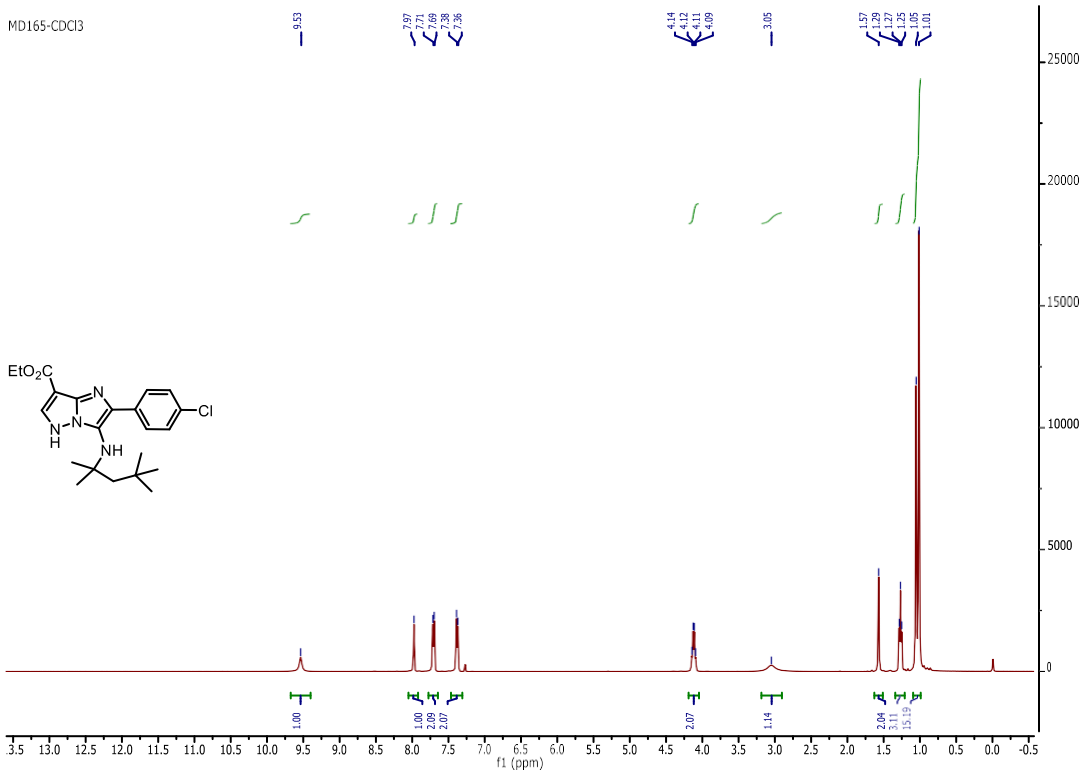


^{13}C NMR (100 MHz, $CDCl_3$)

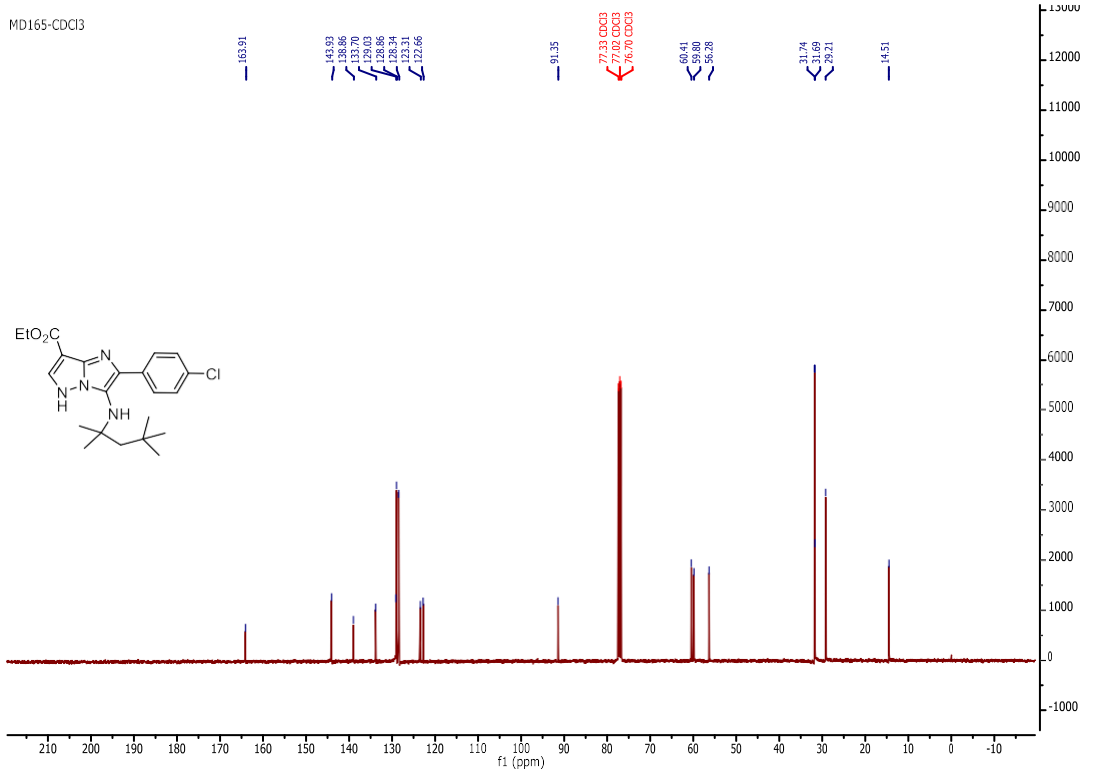


Ethyl 2-(4-chlorophenyl)-3-((2,4,4-trimethylpentan-2-yl)amino)-5*H*-imidazo[1,2-*b*]pyrazole-7-carboxylate (**4c**):

^{1}H NMR (400 MHz, $CDCl_3$)

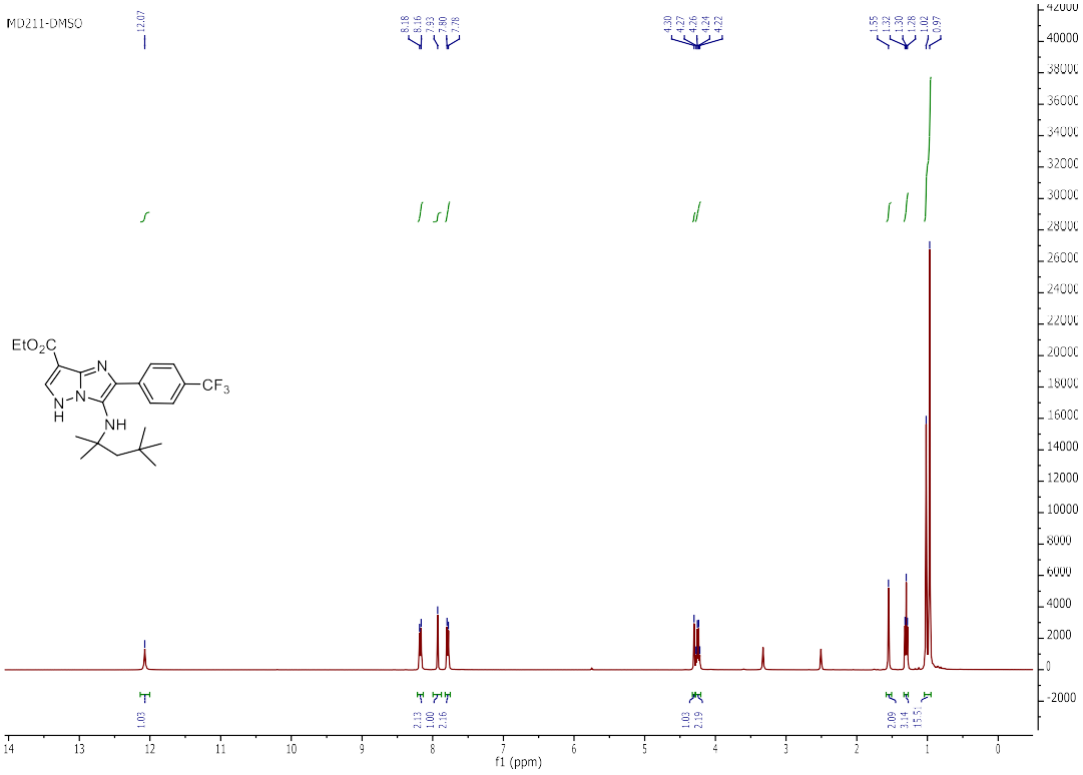


^{13}C NMR (100 MHz, $CDCl_3$)

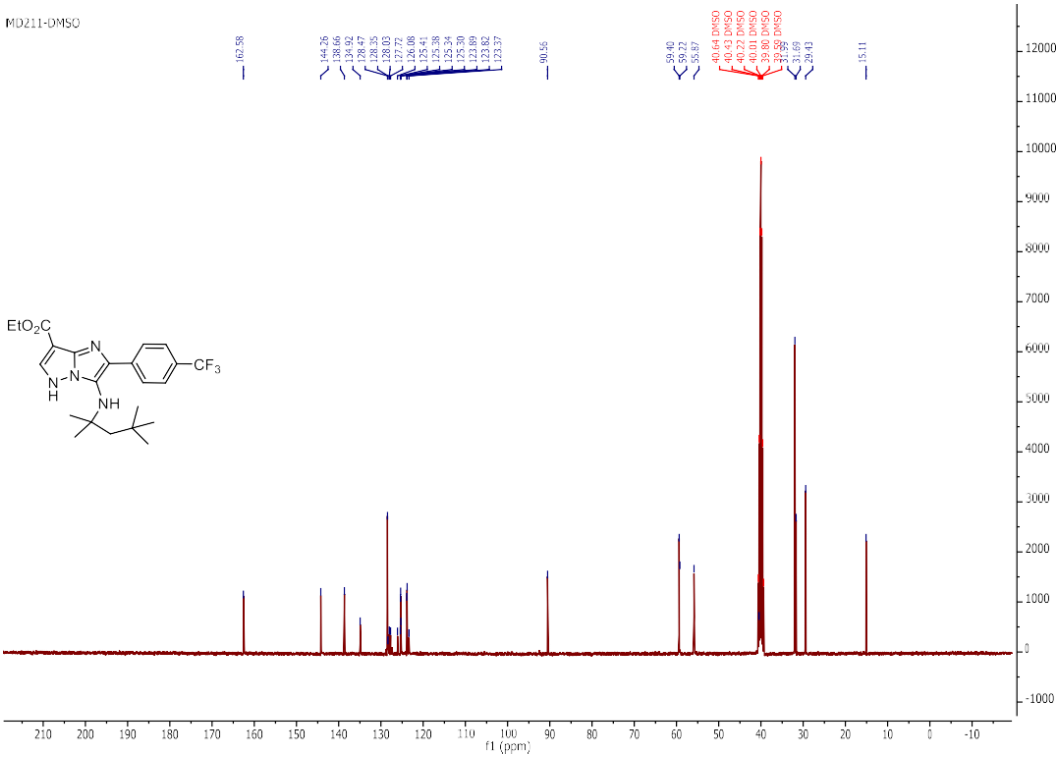


Ethyl 2-(4-(trifluoromethyl)phenyl)-3-((2,4,4-trimethylpentan-2-yl)amino)-5*H*-imidazo[1,2-*b*]pyrazole-7-carboxylate (**4d**):

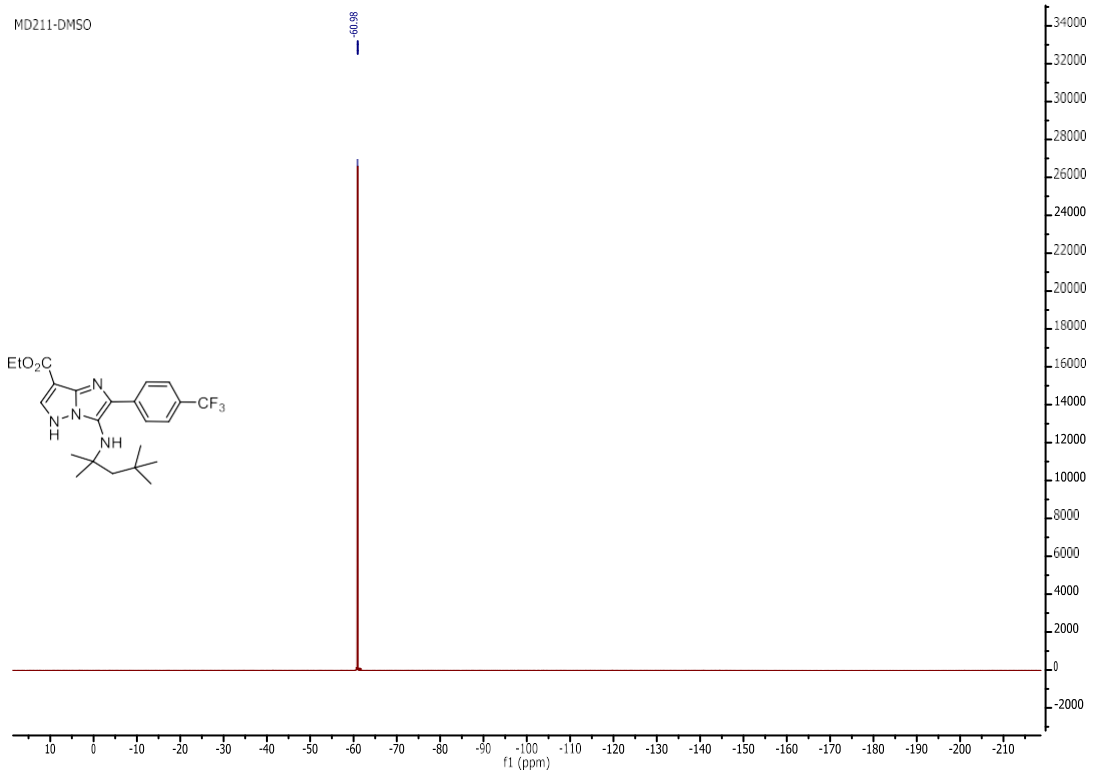
^{1}H NMR (400 MHz, DMSO-d_6)



^{13}C NMR (100 MHz, DMSO-d_6)

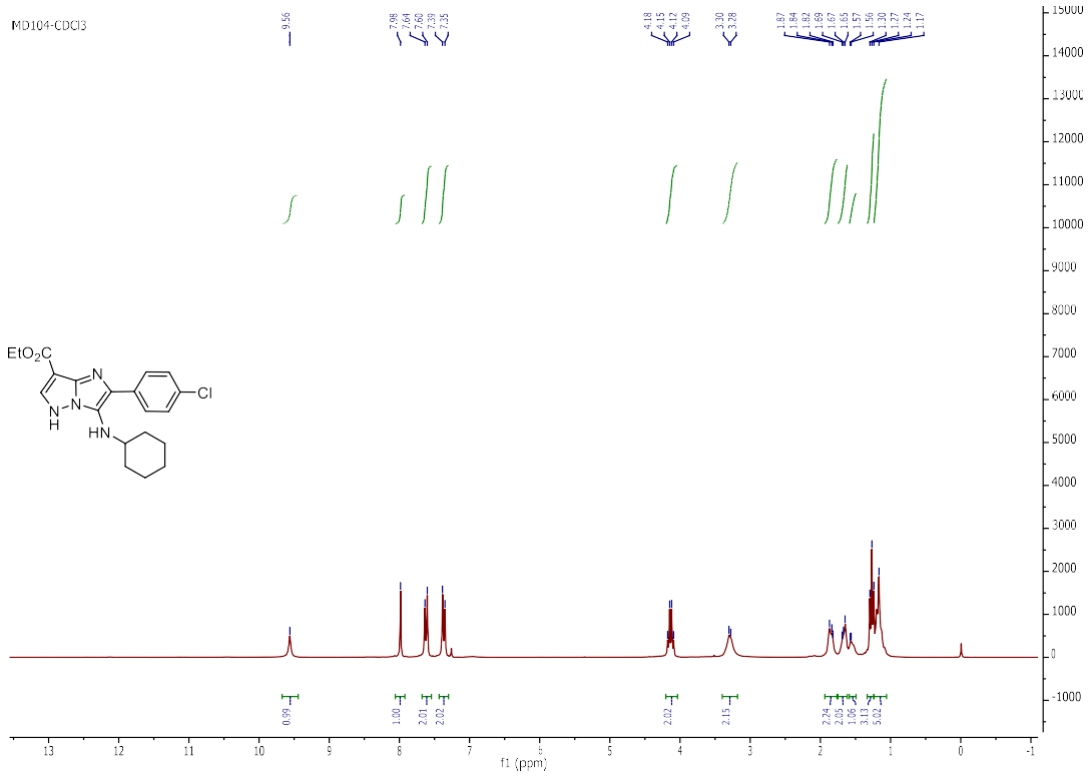


^{19}F NMR (376 MHz, DMSO-d_6)

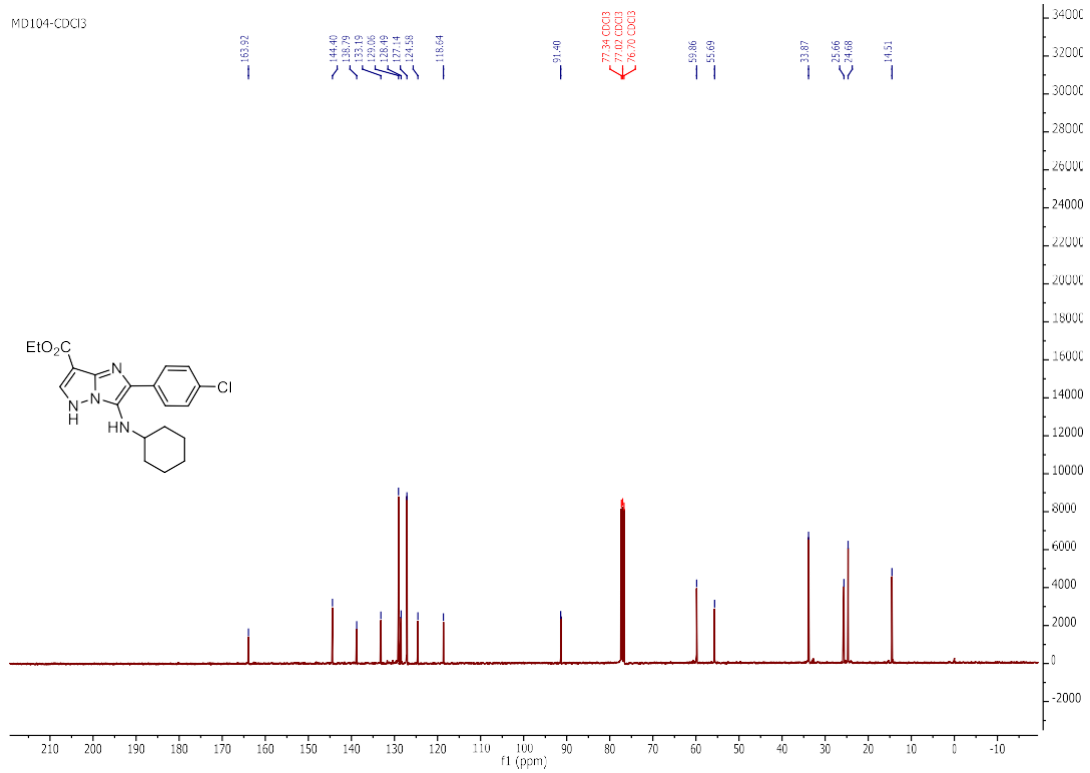


Ethyl 2-(4-chlorophenyl)-3-(cyclohexylamino)-5*H*-imidazo[1,2-*b*]pyrazole-7-carboxylate (**4e**):

^{1}H NMR (400 MHz, $CDCl_3$)

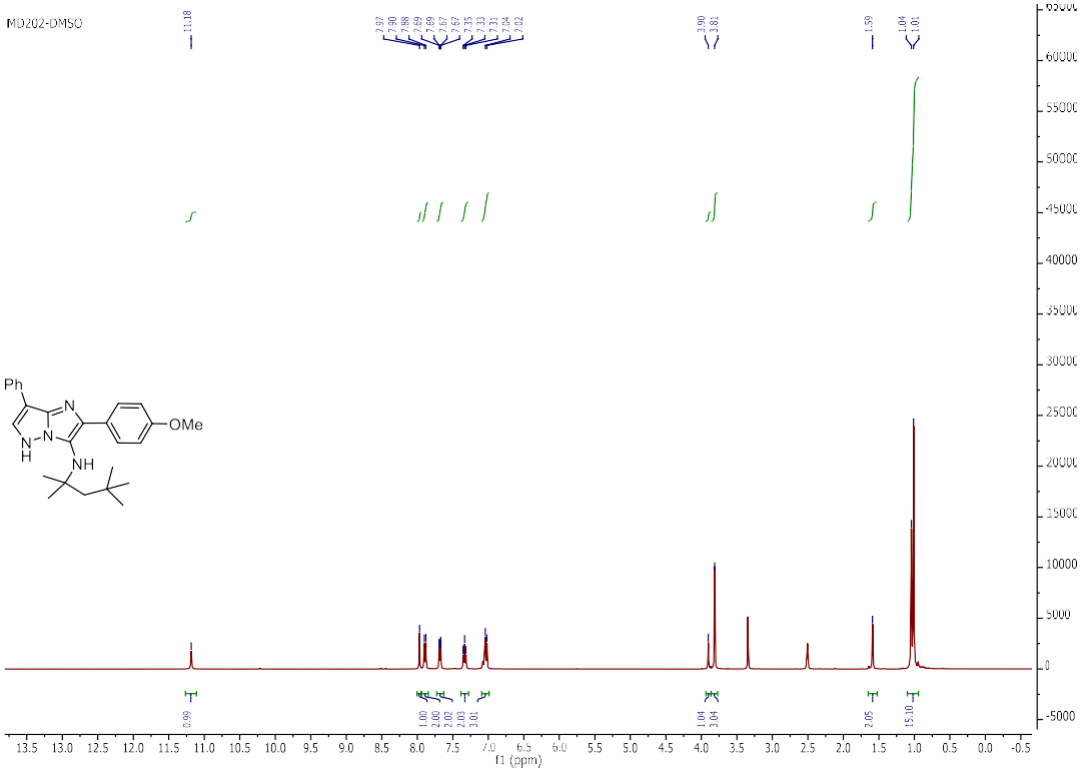


^{13}C NMR (100 MHz, $CDCl_3$)

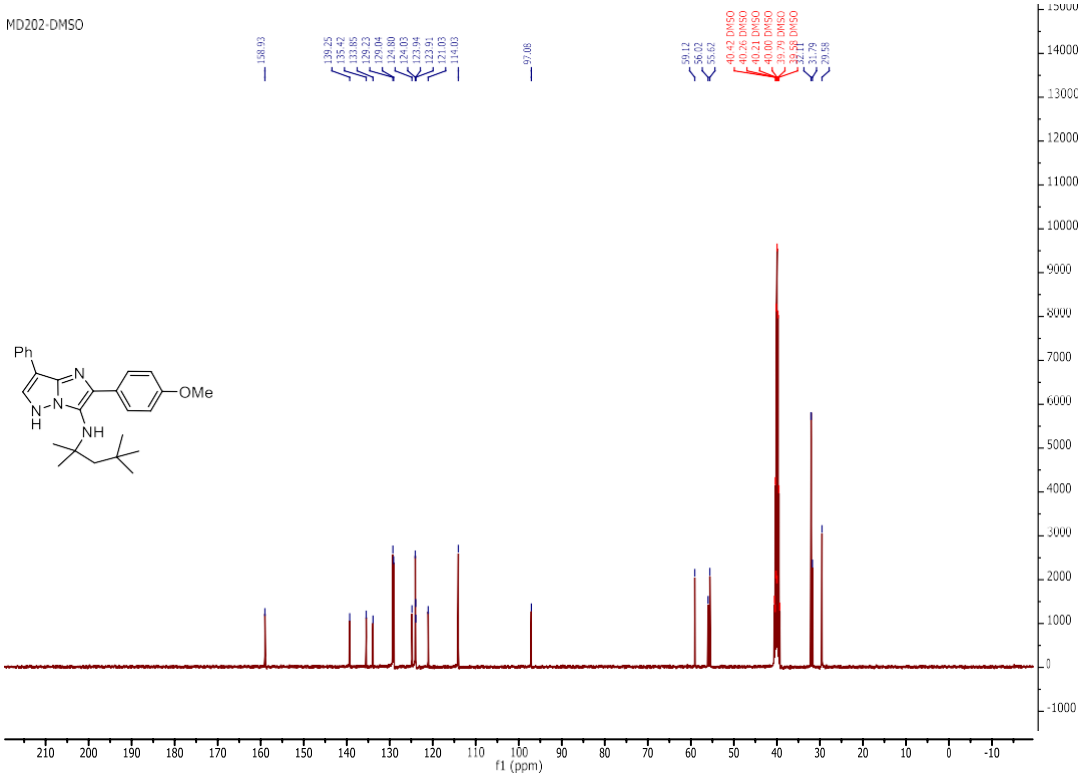


2-(4-Methoxyphenyl)-7-phenyl-*N*-(2,4,4-trimethylpentan-2-yl)-5*H*-imidazo[1,2-*b*]pyrazol-3-amine (**4f**):

^{1}H NMR (400 MHz,DMSO-d_6)

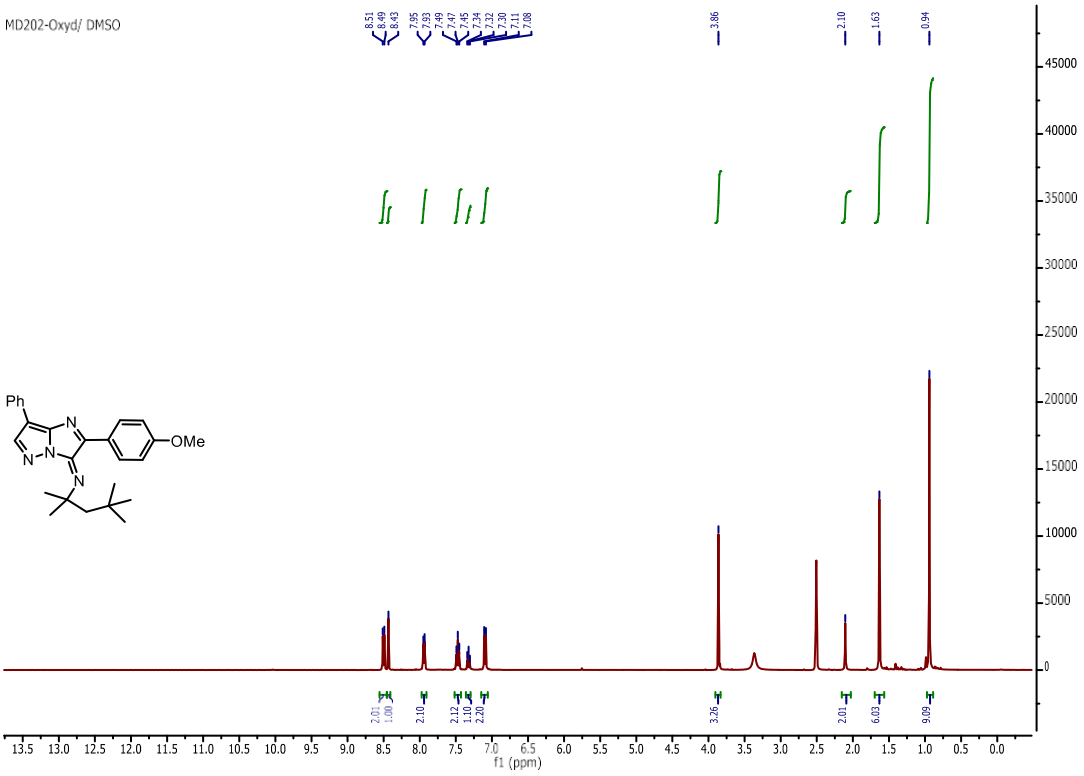


^{13}C NMR (100 MHz, DMSO-d_6)

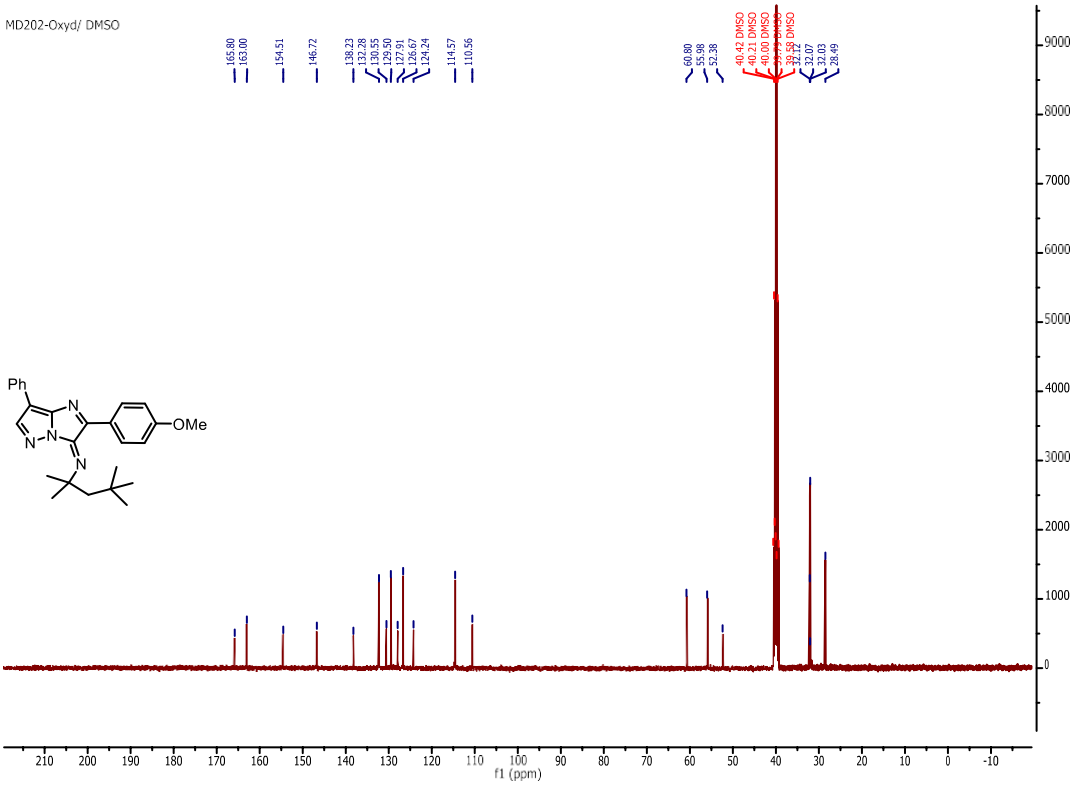


(4-Methoxyphenyl)-7-phenyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine (**4f'**):

^{1}H NMR (400 MHz,DMSO-d_6)

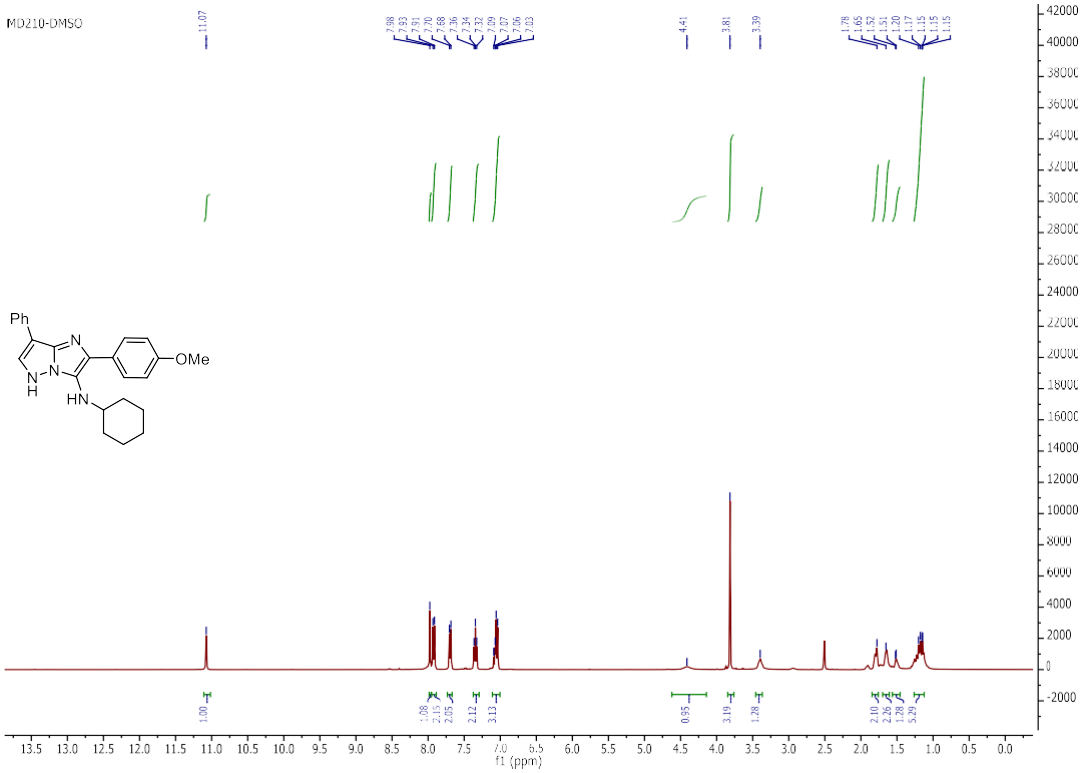


^{13}C NMR (100 MHz, DMSO-d_6)

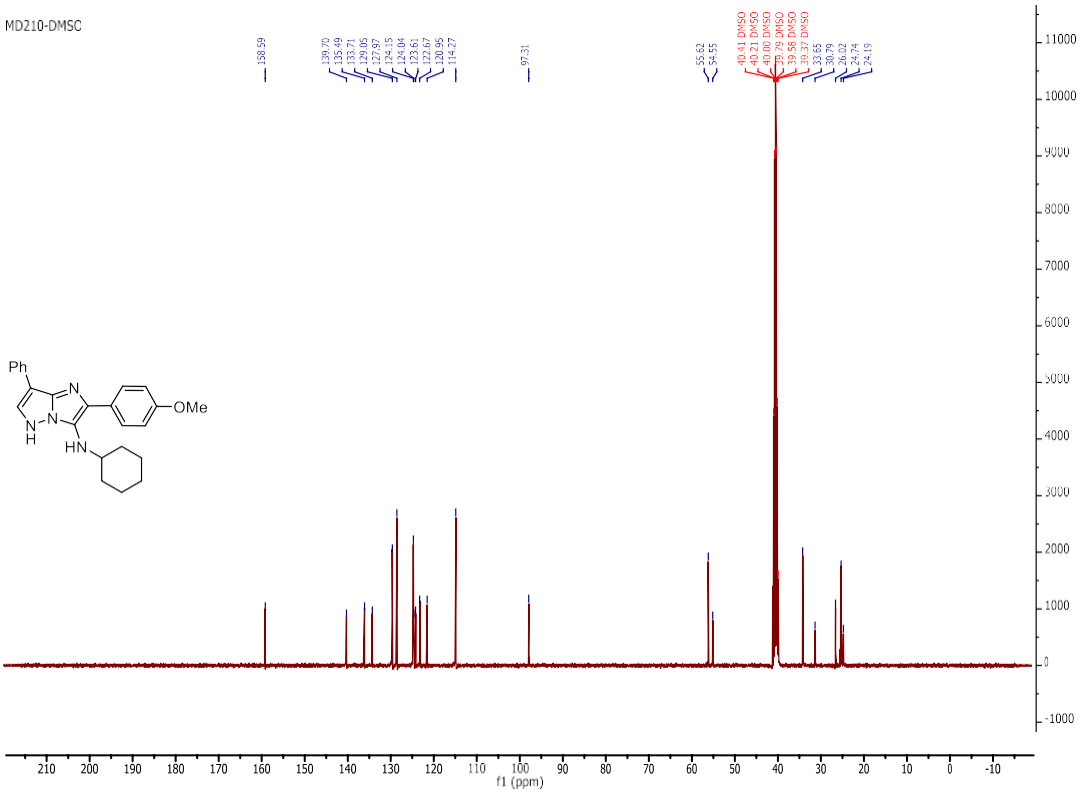


N-Cyclohexyl-2-(4-methoxyphenyl)-7-phenyl-5*H*-imidazo[1,2-*b*]pyrazol-3-amine (**4g**):

^{1}H NMR (400 MHz,DMSO-d_6)

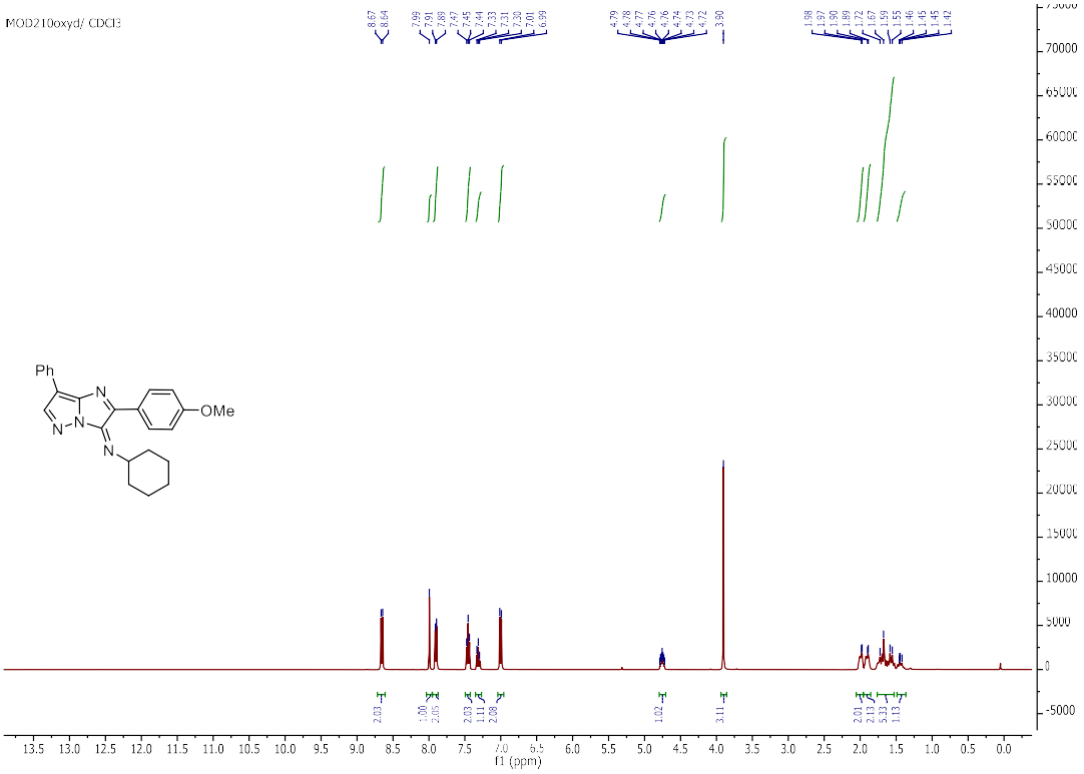


^{13}C NMR (100 MHz, DMSO-d_6)

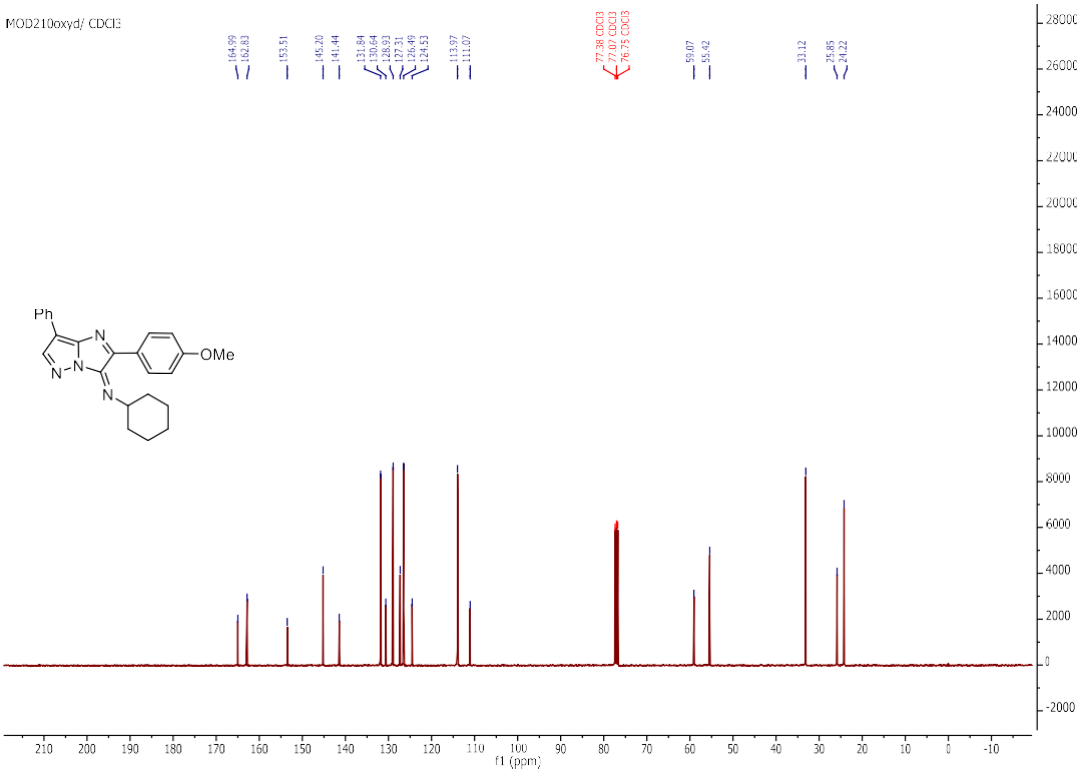


N-cyclohexyl-2-(4-methoxyphenyl)-7-phenyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine (**4g'**):

^{1}H NMR (400 MHz, $CDCl_3$)

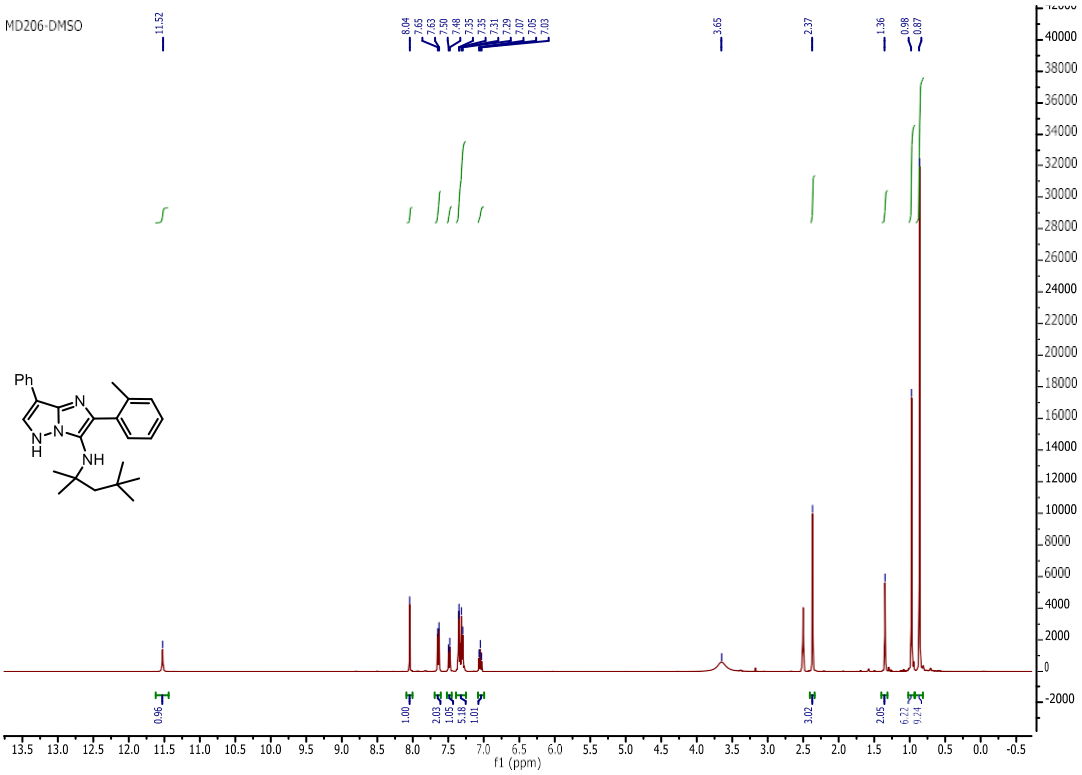


^{13}C NMR (100 MHz, $CDCl_3$)

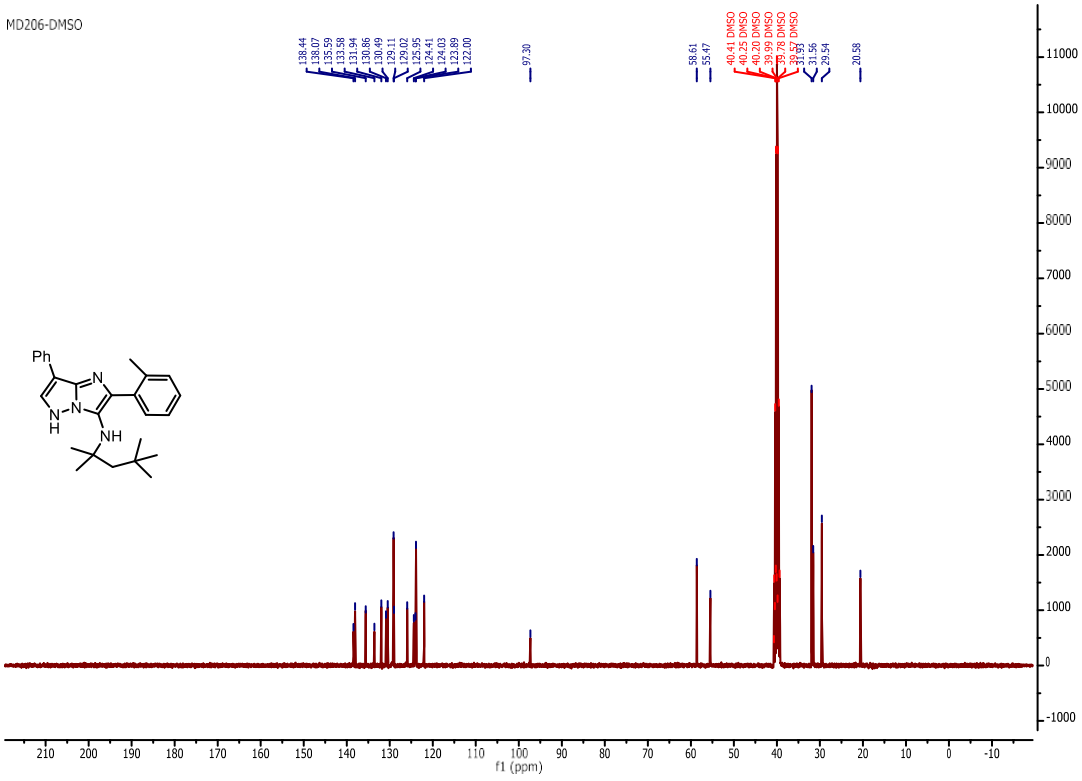


7-Phenyl-2-(*o*-tolyl)-*N*-(2,4,4-trimethylpentan-2-yl)-5*H*-imidazo[1,2-*b*]pyrazol-3-amine (**4h**):

^{1}H NMR (400 MHz,DMSO-d_6)

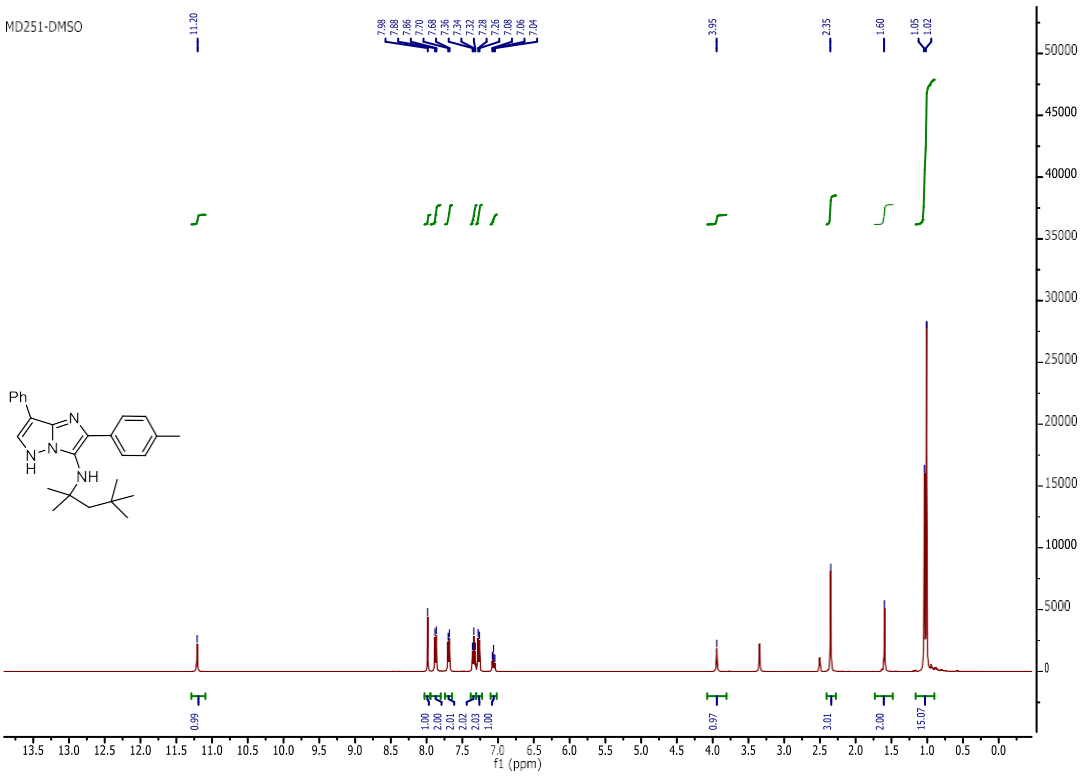


^{13}C NMR (100 MHz, DMSO-d_6)

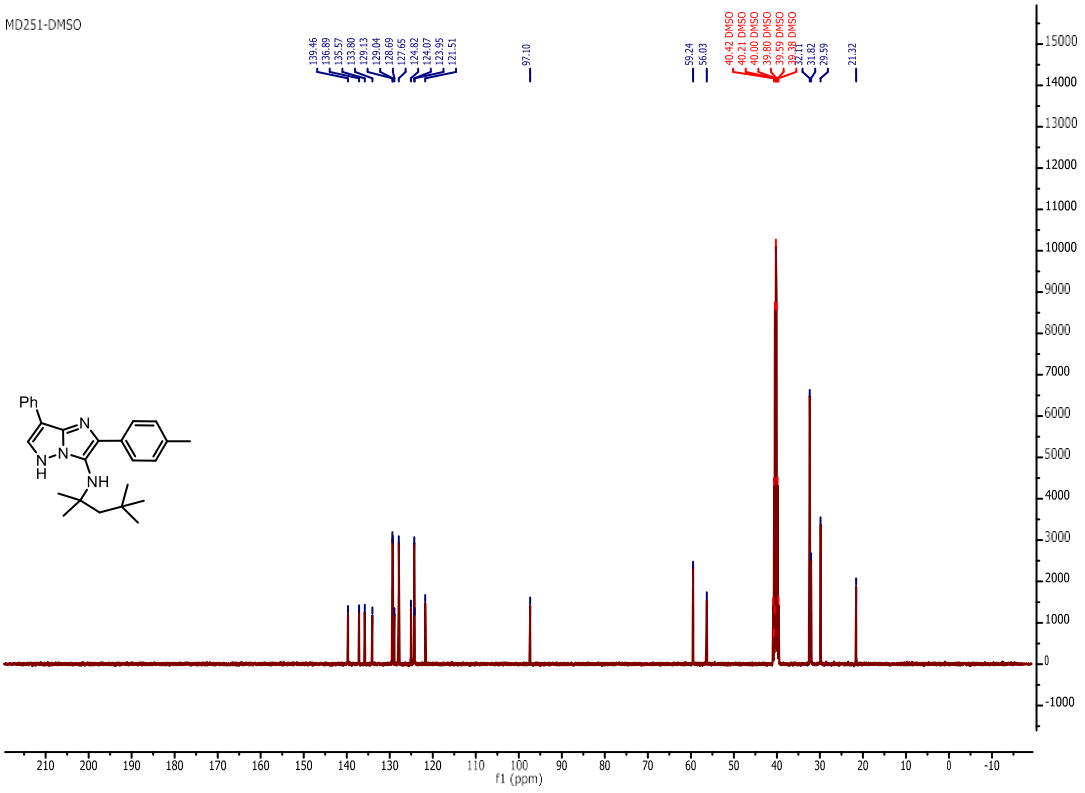


7-Phenyl-2-(*p*-tolyl)-*N*-(2,4,4-trimethylpentan-2-yl)-5*H*-imidazo[1,2-*b*]pyrazol-3-amine (**4i**):

^{1}H NMR (400 MHz,DMSO-d_6)

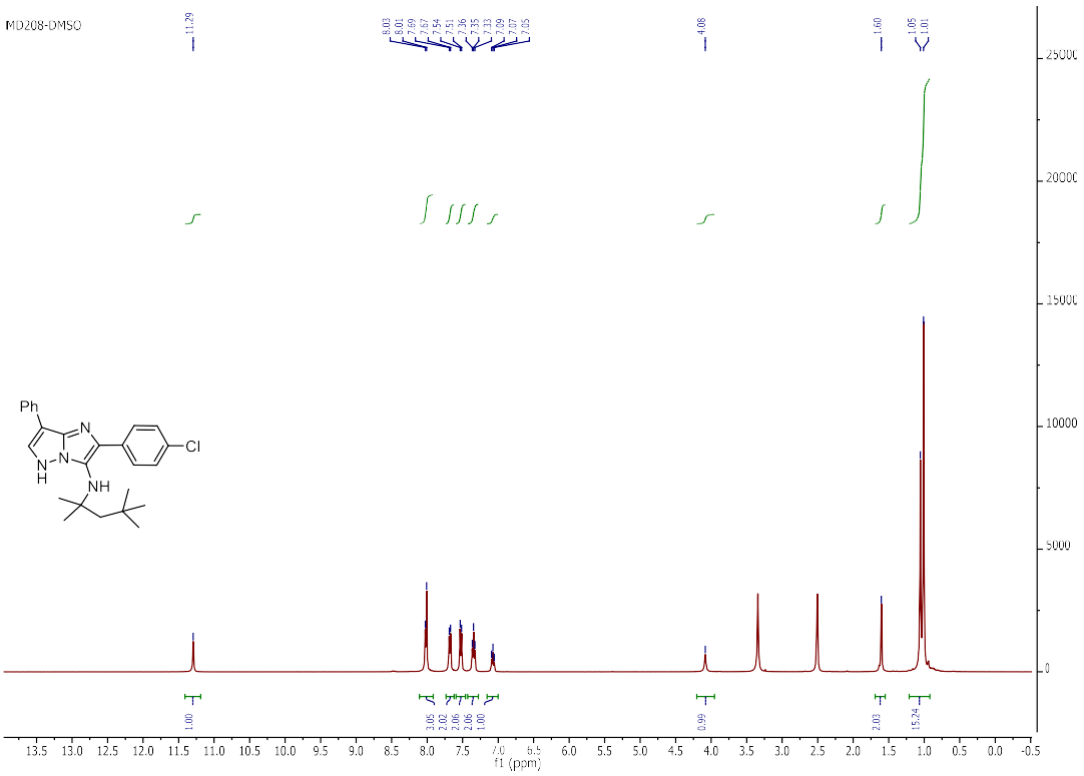


^{13}C NMR (100 MHz, DMSO-d_6)

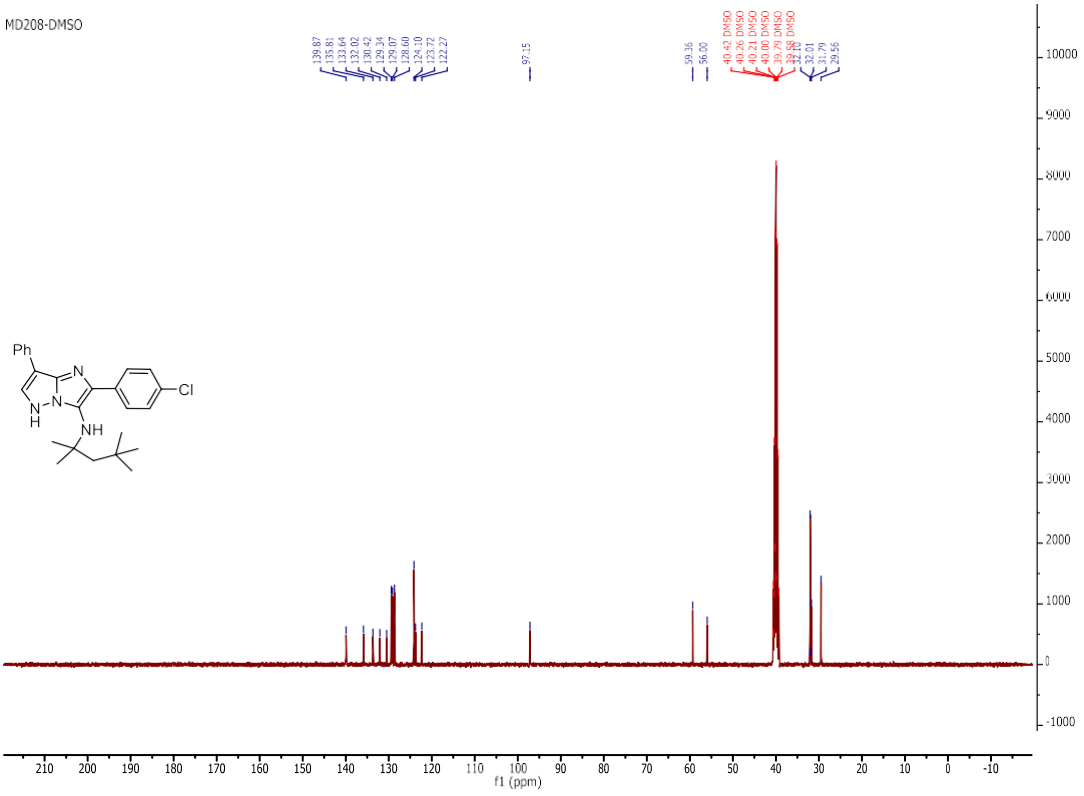


2-(4-Chlorophenyl)-7-phenyl-*N*-(2,4,4-trimethylpentan-2-yl)-5*H*-imidazo[1,2-*b*]pyrazol-3-amine (**4j**):

^{1}H NMR (400 MHz,DMSO-d_6)

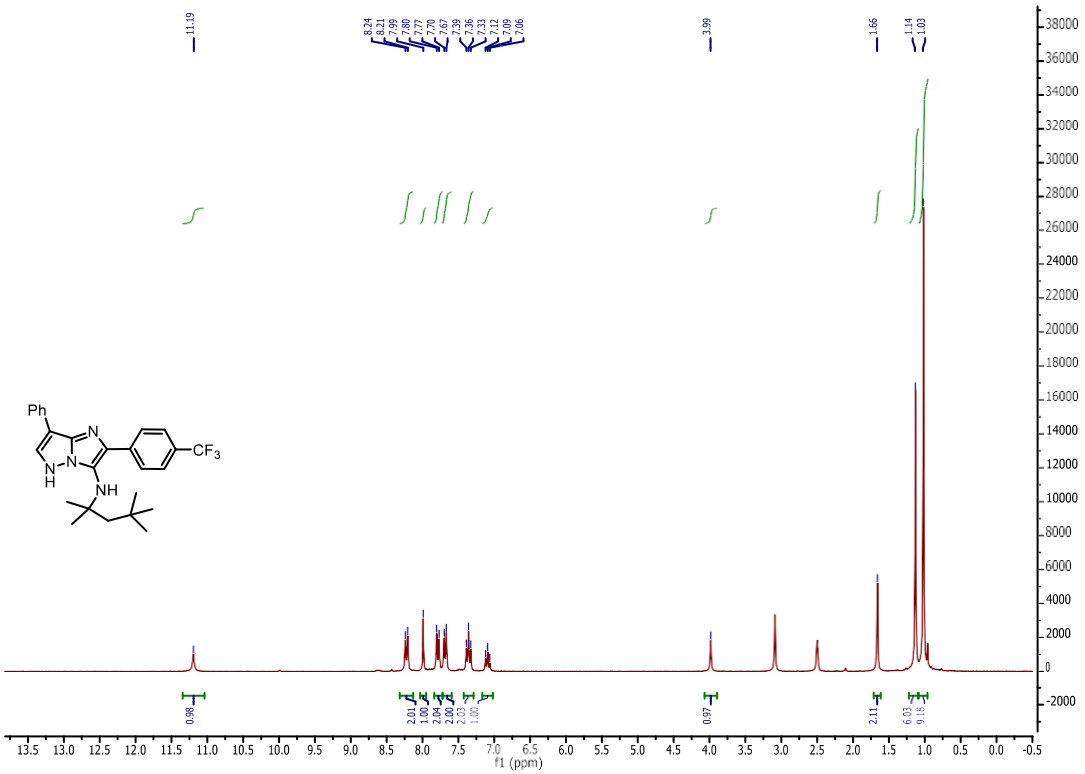


^{13}C NMR (100 MHz, DMSO-d_6)

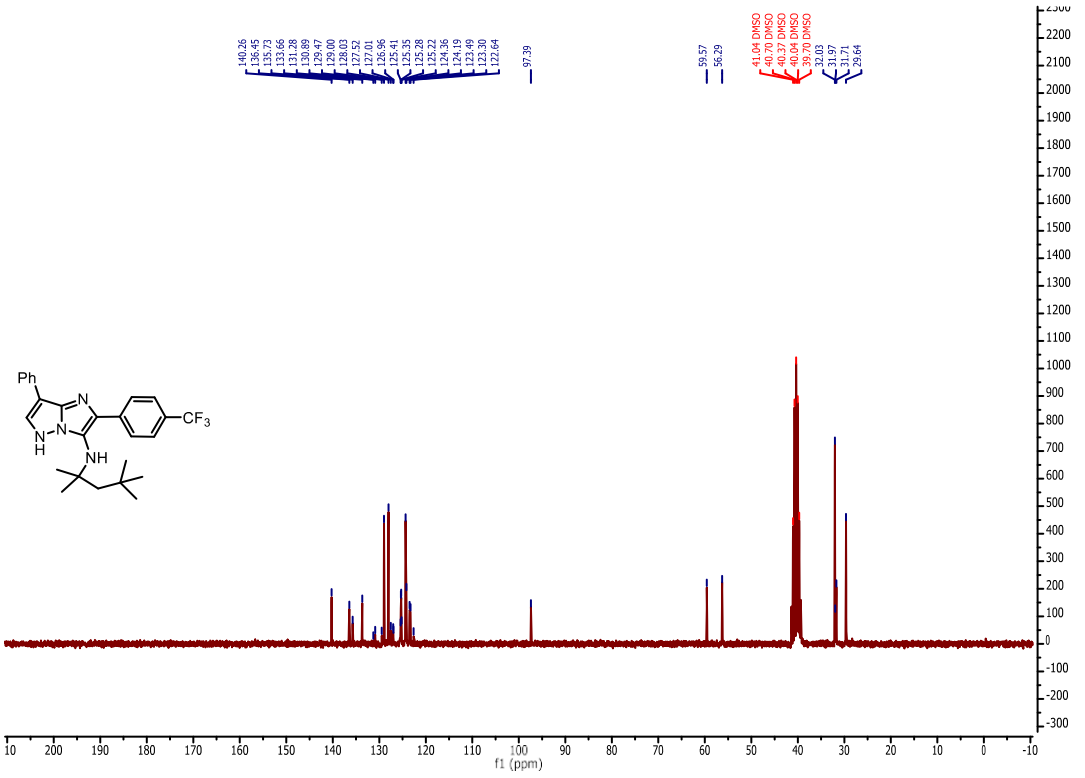


7-Phenyl-2-(4-(trifluoromethyl)phenyl)-*N*-(2,4,4-trimethylpentan-2-yl)-5*H*-imidazo[1,2-*b*]pyrazol-3-amine (**4k**):

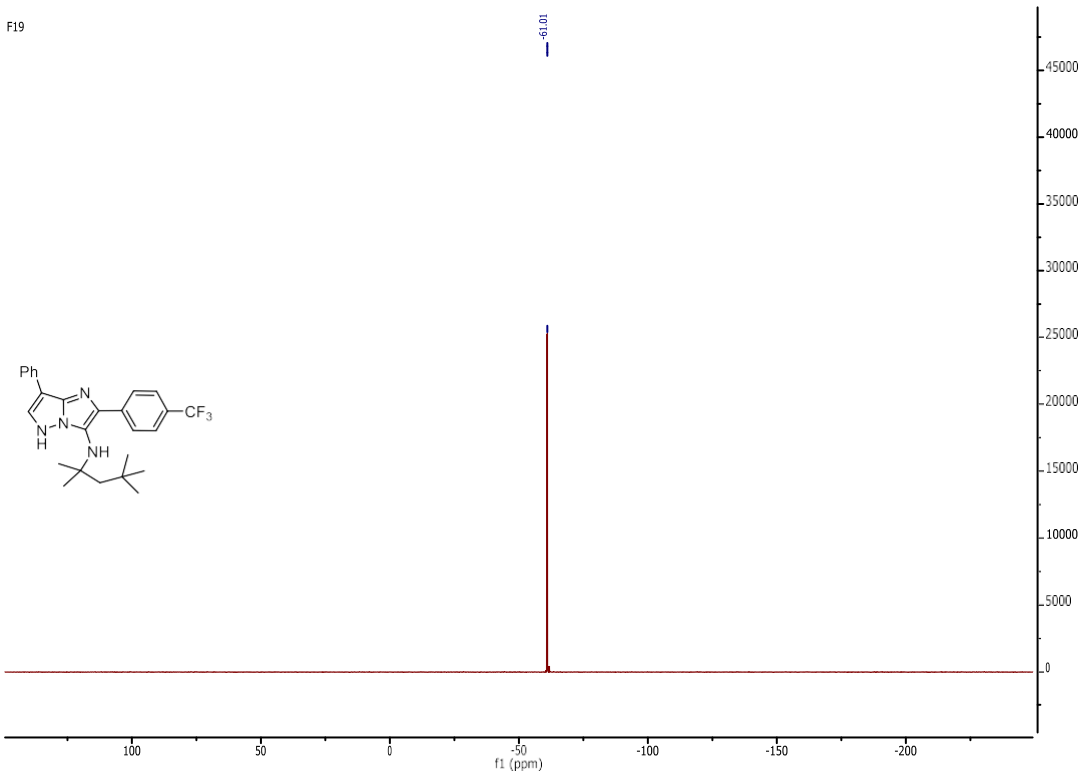
^{1}H NMR (250 MHz, DMSO-d_6, 70°C)



^{13}C NMR (62.9 MHz, DMSO-d_6, 70°C)

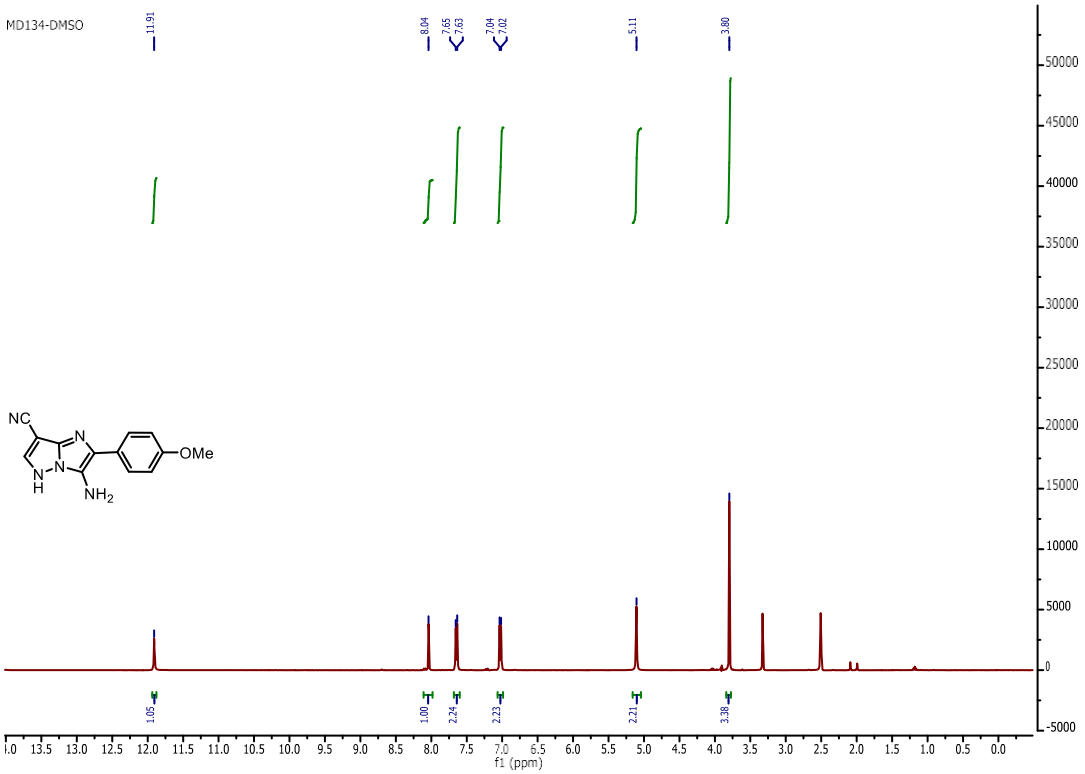


^{19}F NMR (235 MHz, DMSO-d_6, 70°C)

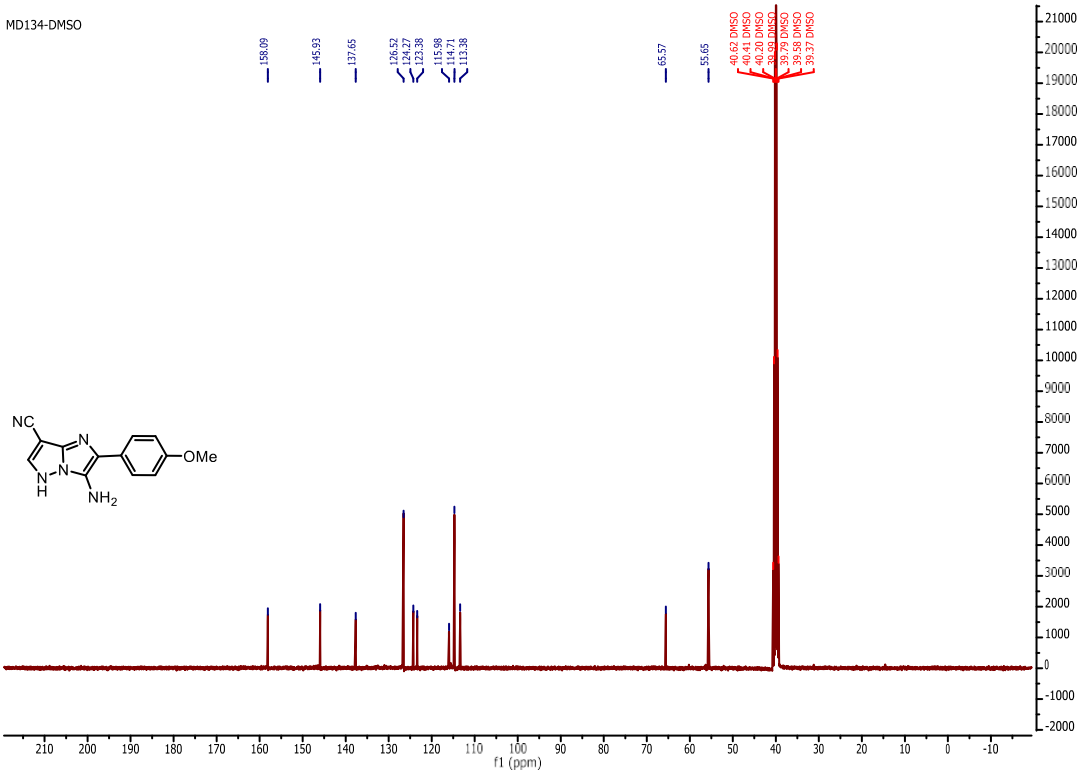


3-Amino-2-(4-methoxyphenyl)-5*H*-imidazo[1,2-*b*]pyrazole-7-carbonitrile (**5a**):

^{1}H NMR (400 MHz, DMSO-d_6)

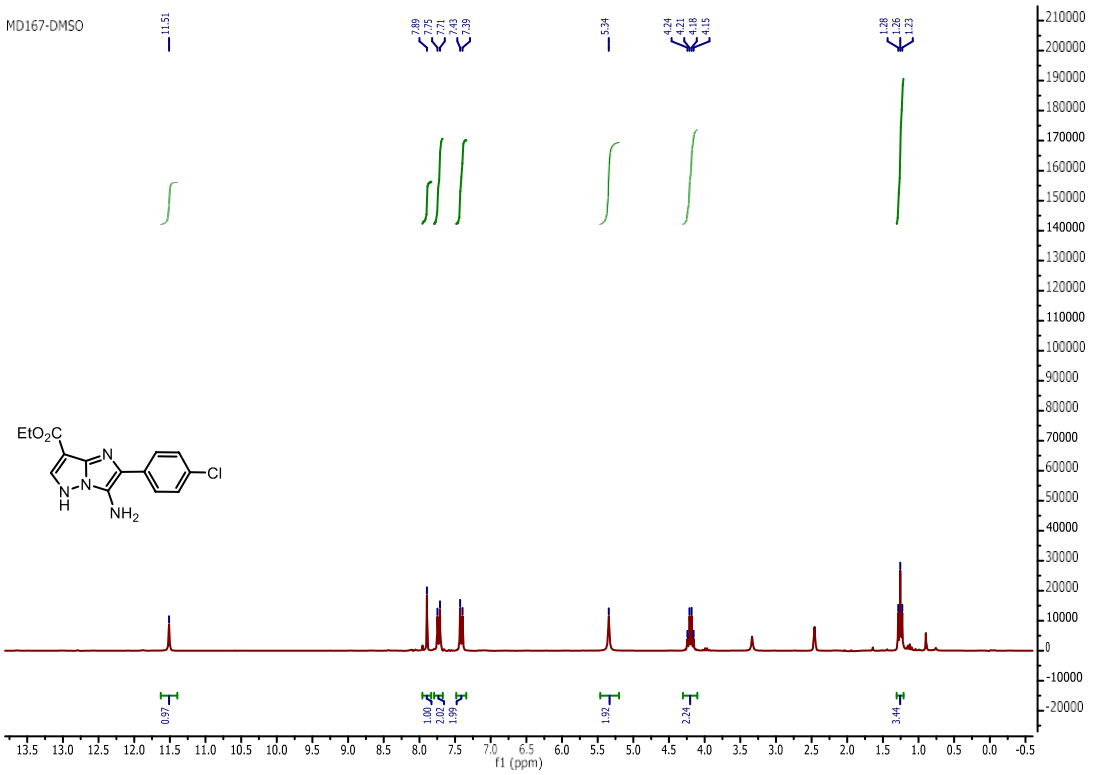


^{13}C NMR (100 MHz, DMSO-d_6)

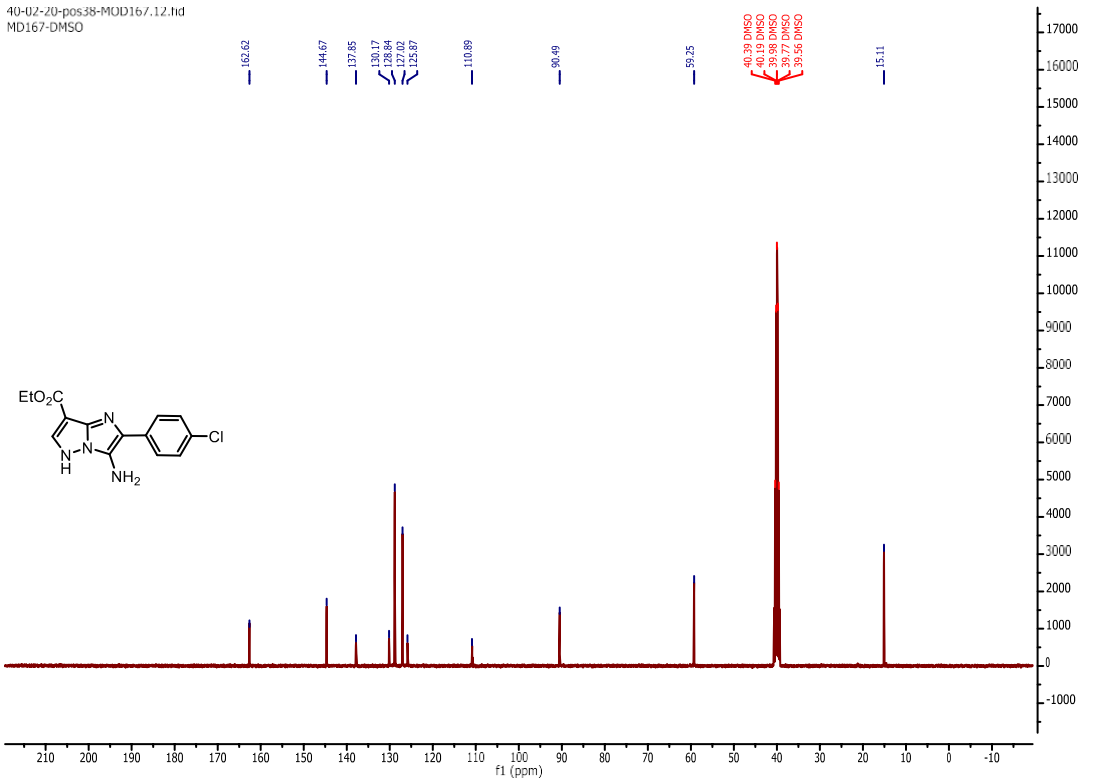


Ethyl 3-amino-2-(4-chlorophenyl)-5*H*-imidazo[1,2-*b*]pyrazole-7-carboxylate (**5c**):

^{1}H NMR (250 MHz, DMSO-d_6)

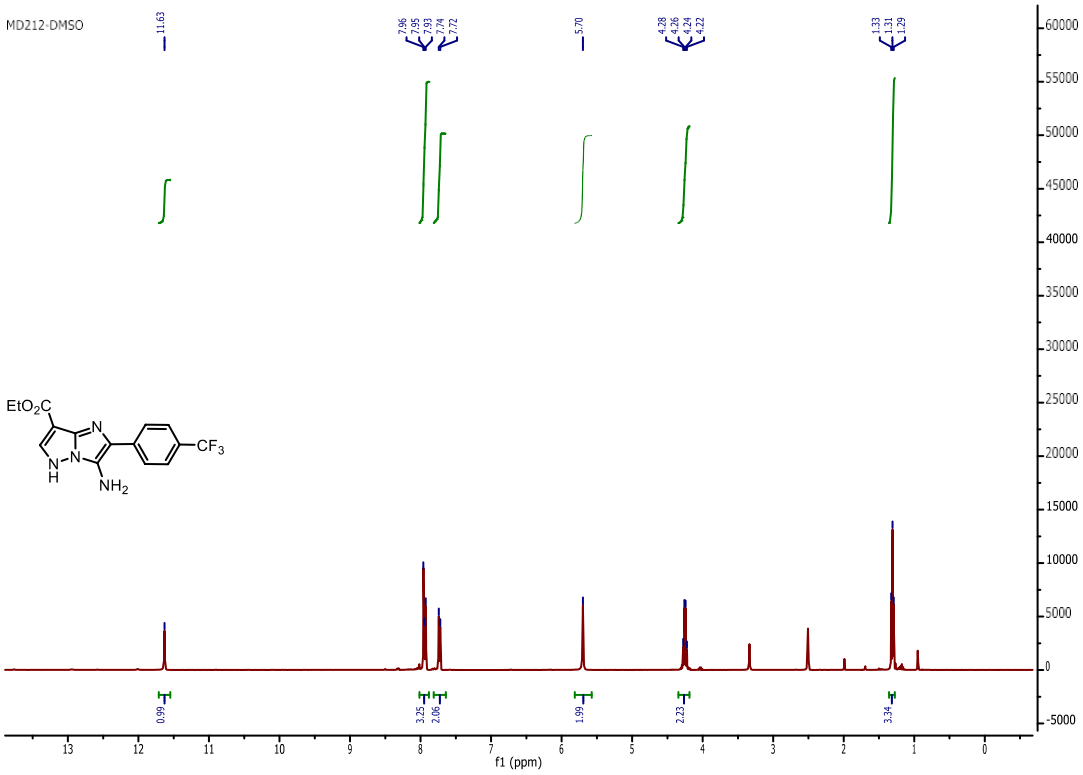


^{13}C NMR (62.9 MHz, DMSO-d_6)

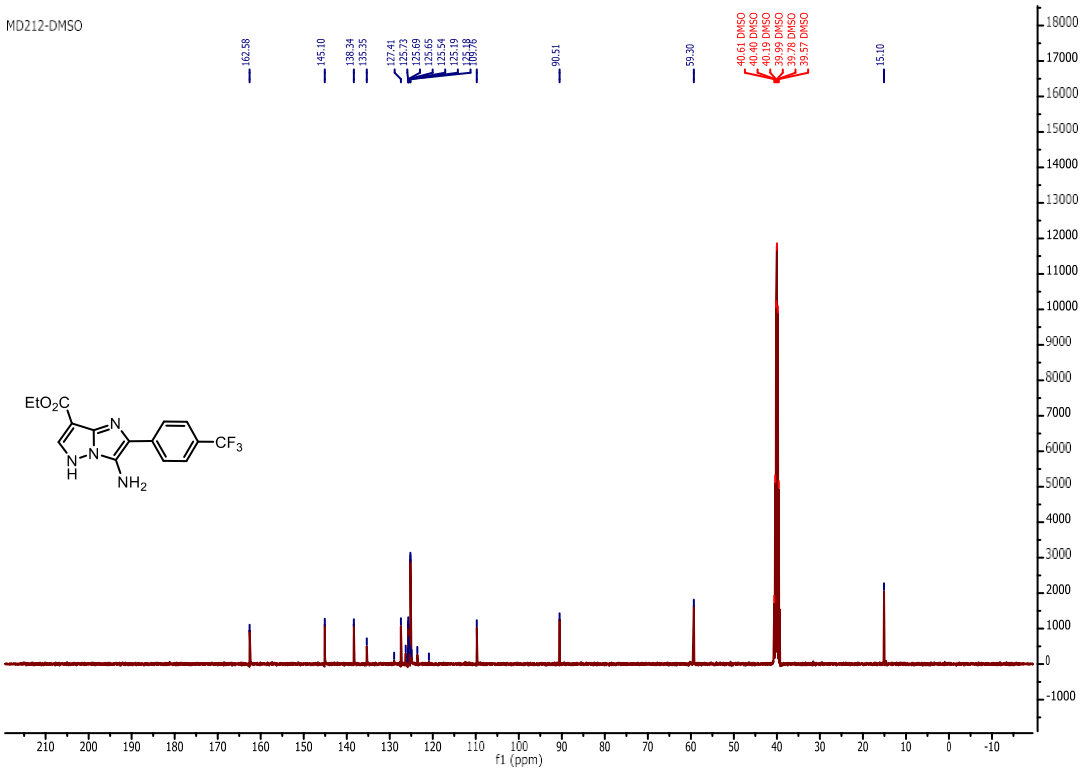


Ethyl3-amino-2-(4-(trifluoromethyl)phenyl)-5*H*-imidazo[1,2-*b*]pyrazole-7-carboxylate (**5d**):

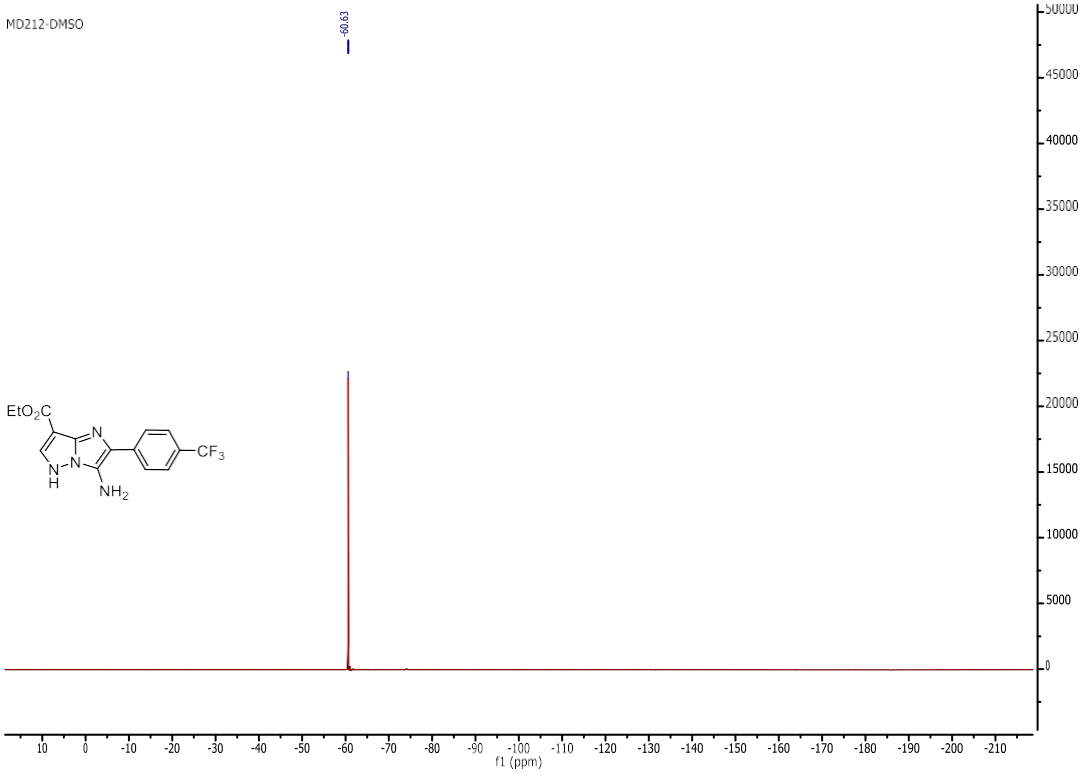
^{1}H NMR (400 MHz,DMSO-d_6)



^{13}C NMR (100 MHz, DMSO-d_6)

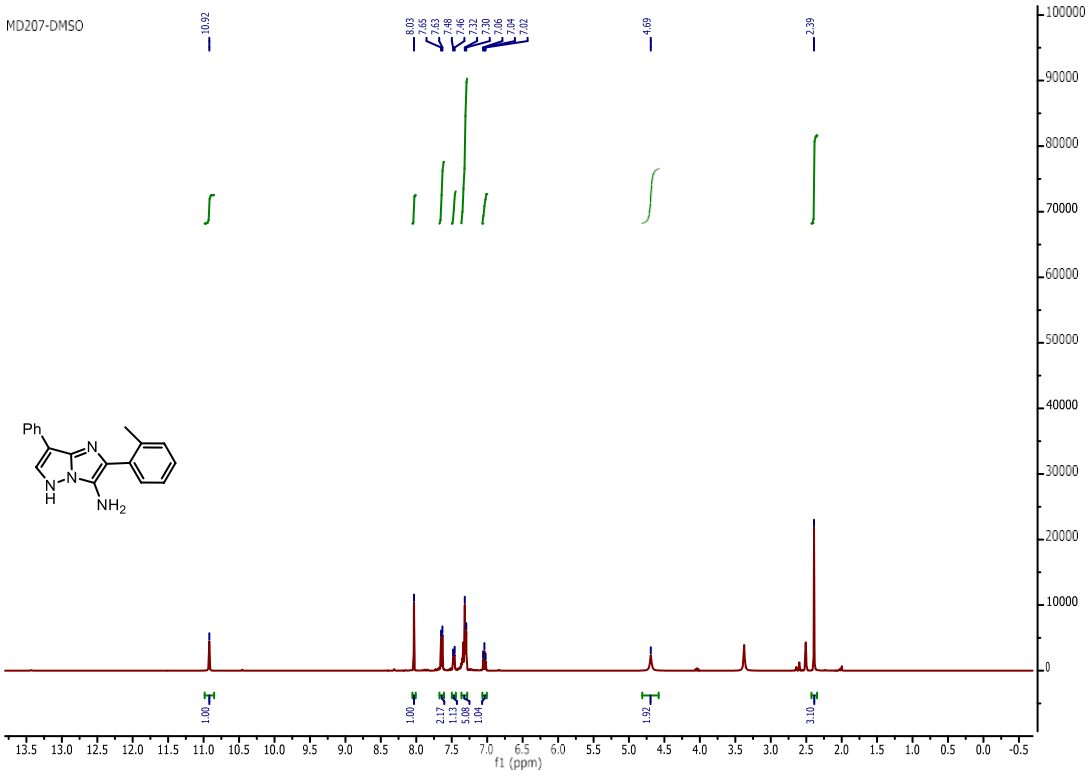


^{19}F NMR (376 MHz, DMSO-d_6)

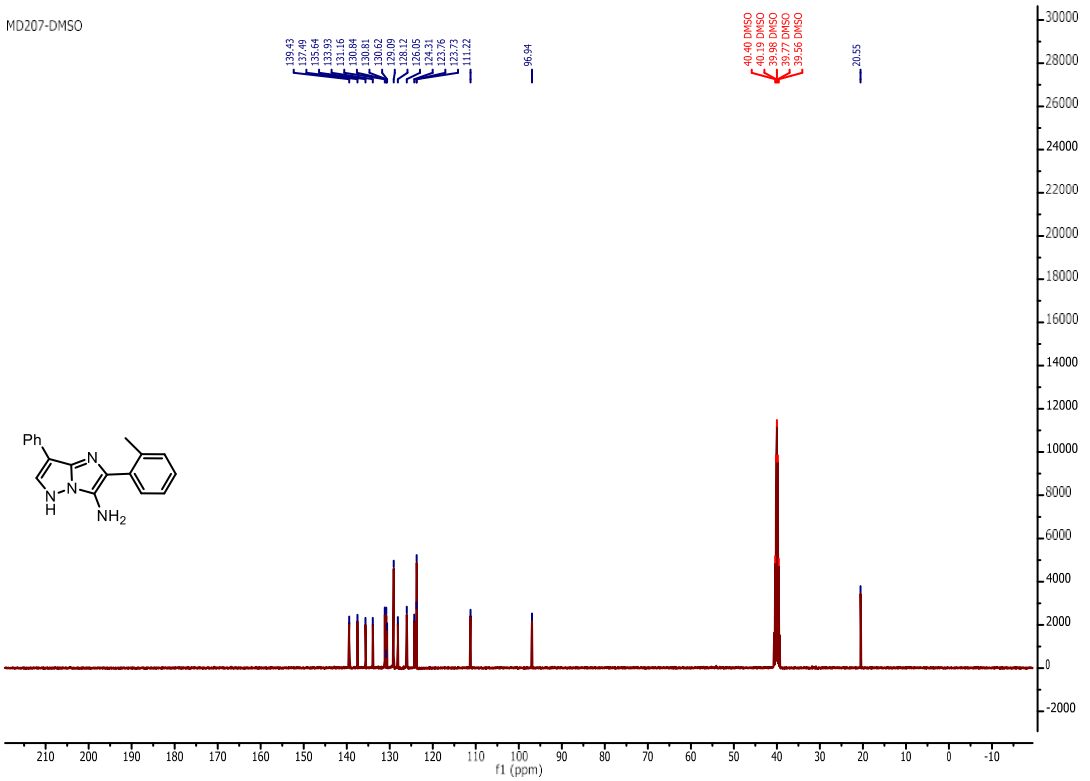


7-Phenyl-2-(*o*-tolyl)-5*H*-imidazo[1,2-*b*]pyrazol-3-amine (**5h**):

^{1}H NMR (400 MHz,DMSO-d_6)

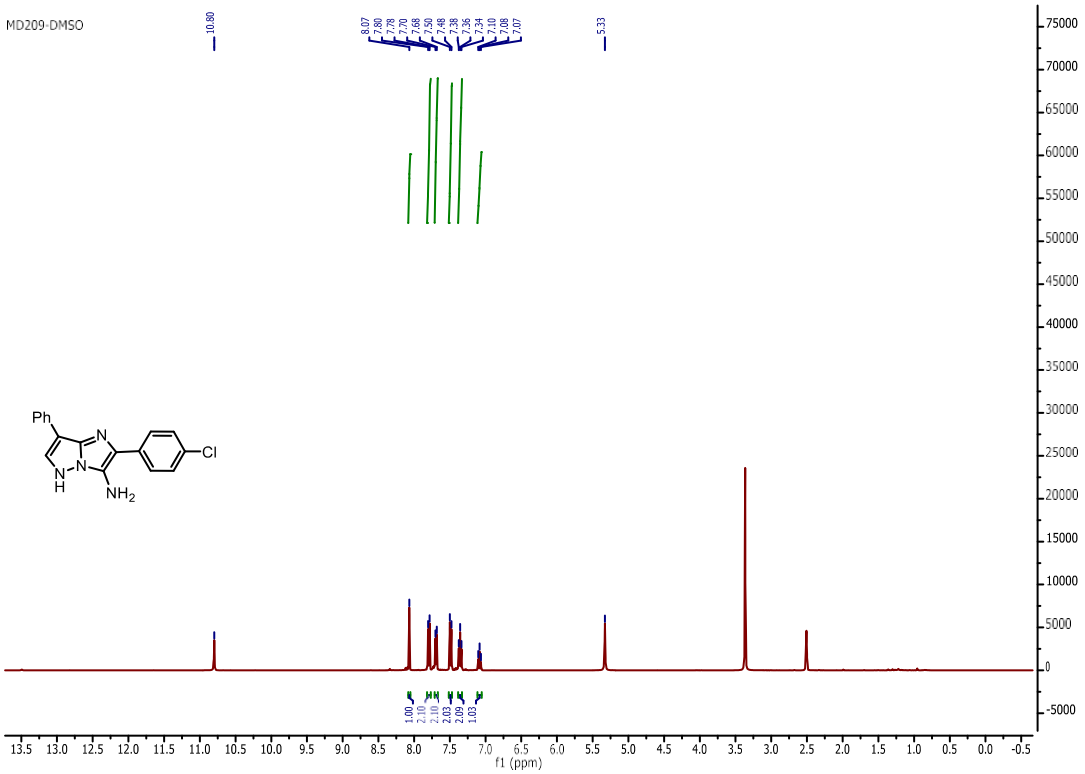


^{13}C NMR (100 MHz, DMSO-d_6)

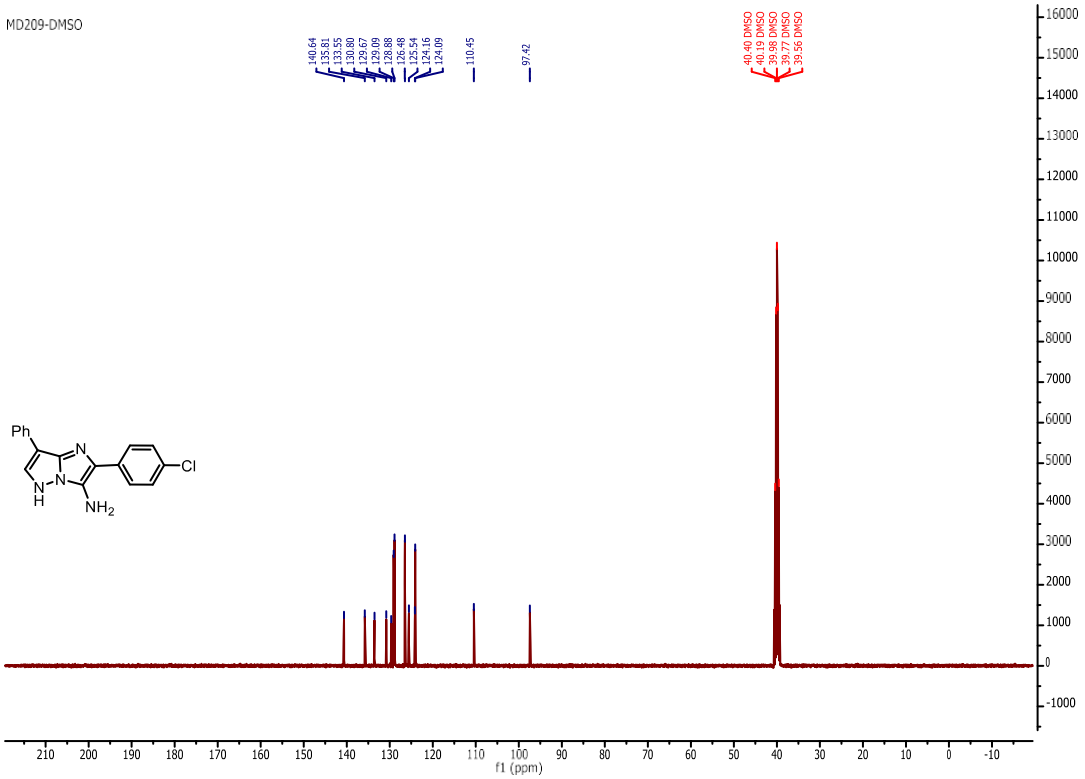


2-(4-Chlorophenyl)-7-phenyl-5*H*-imidazo[1,2-*b*]pyrazol-3-amine (**5j**):

^{1}H NMR (400 MHz,DMSO-d_6)

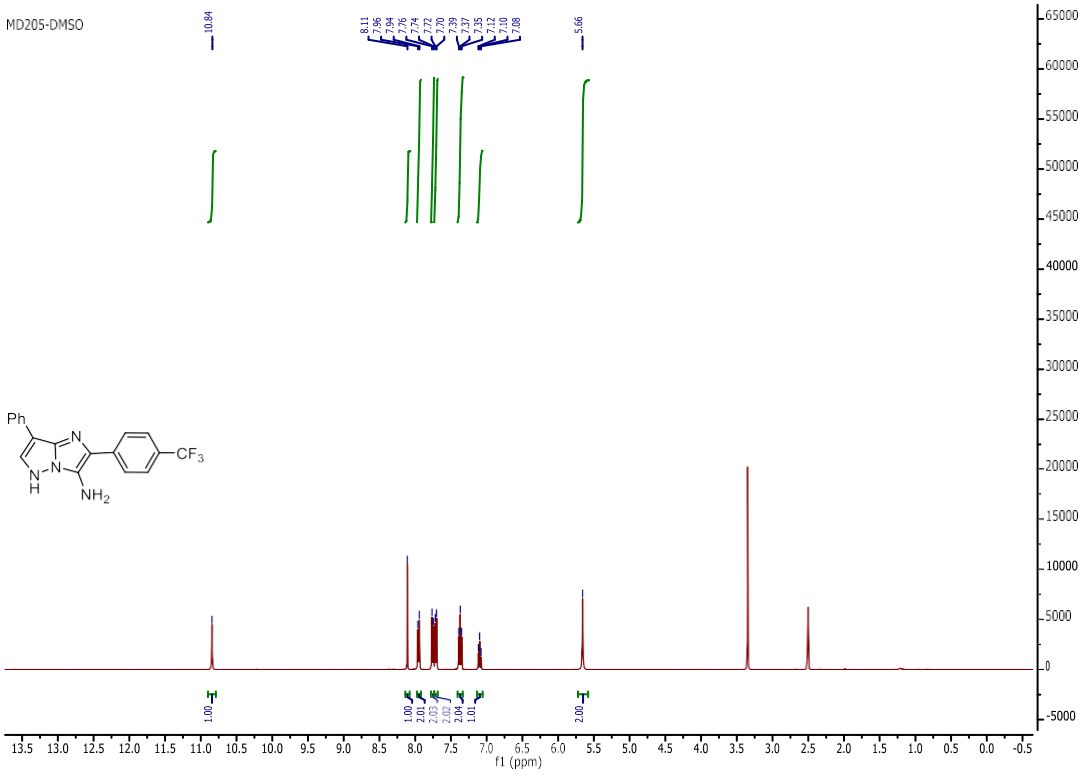


^{13}C NMR (100 MHz, DMSO-d_6)

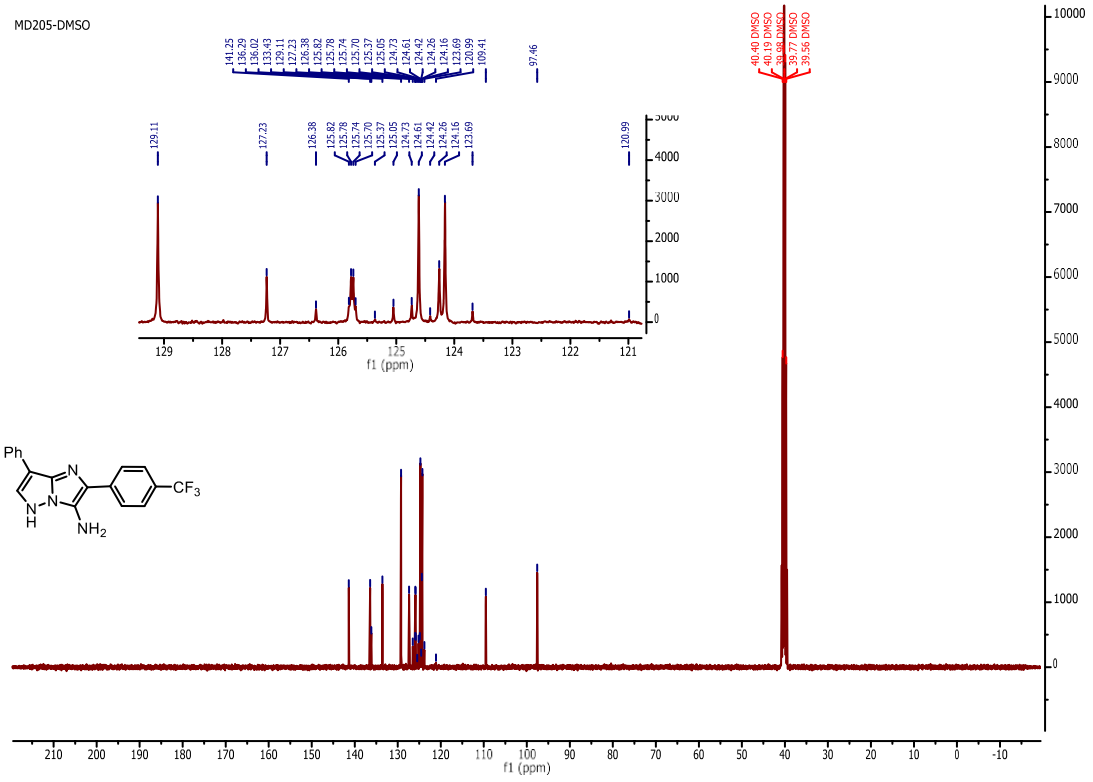


7-Phenyl-2-(4-(trifluoromethyl)phenyl)-5*H*-imidazo[1,2-*b*]pyrazol-3-amine **(5k)**:

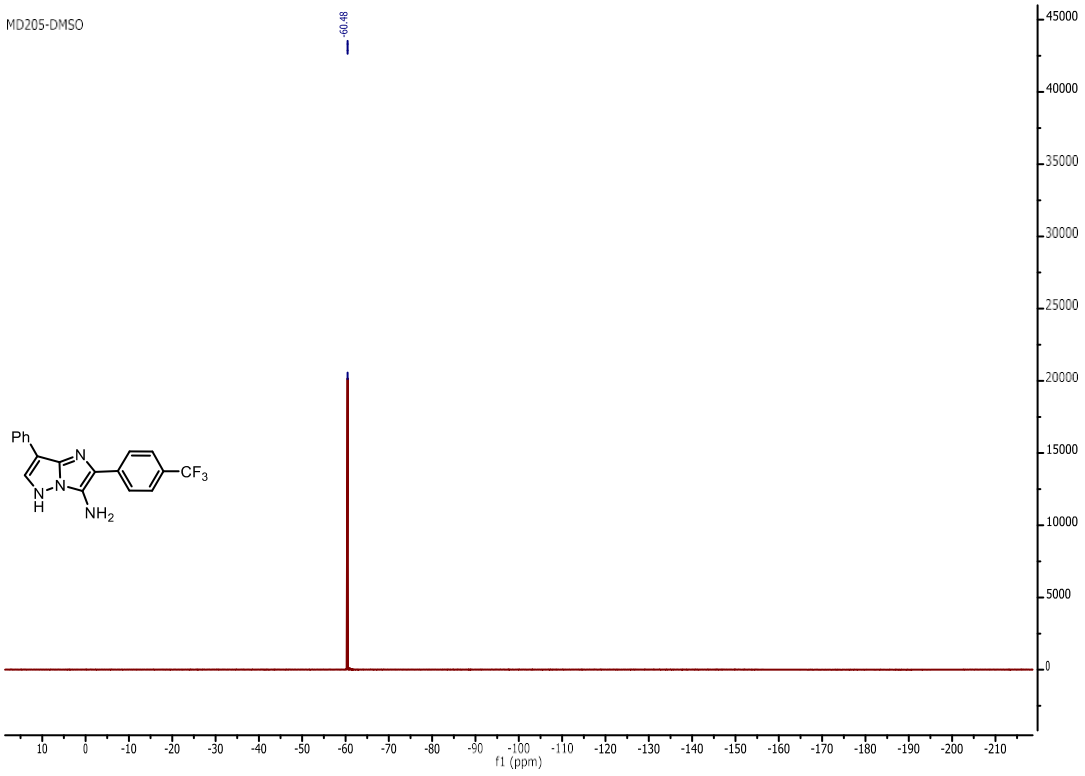
^{1}H NMR (400 MHz,DMSO-d_6)



^{13}C NMR (100 MHz, DMSO-d_6)

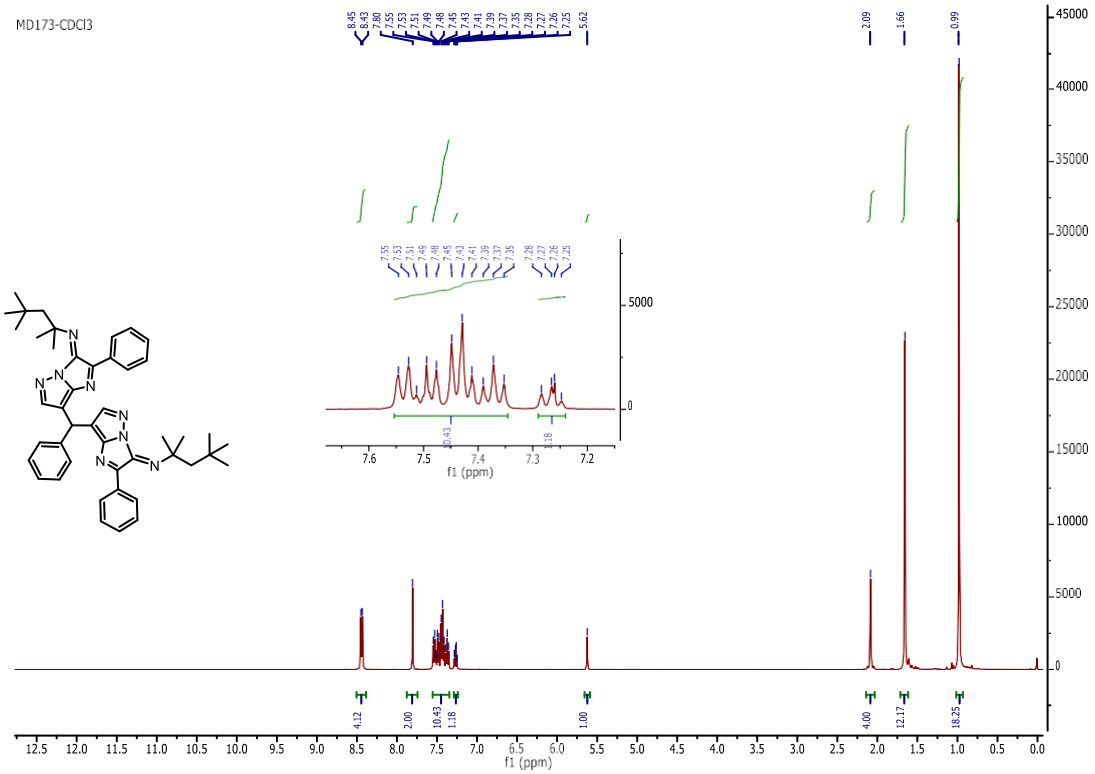


^{19}F NMR (376 MHz, DMSO-d_6)

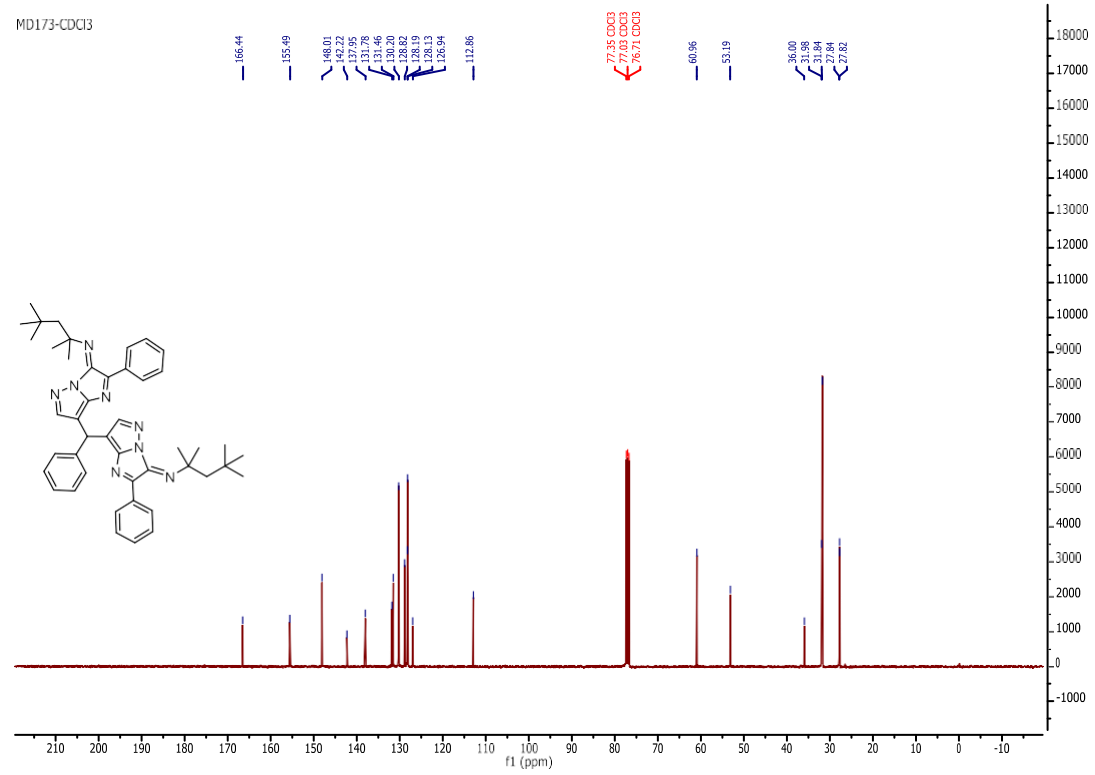


7,7'-(Phenylmethylene)bis(2-phenyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8a**):

^{1}H NMR (400 MHz, $CDCl_3$)

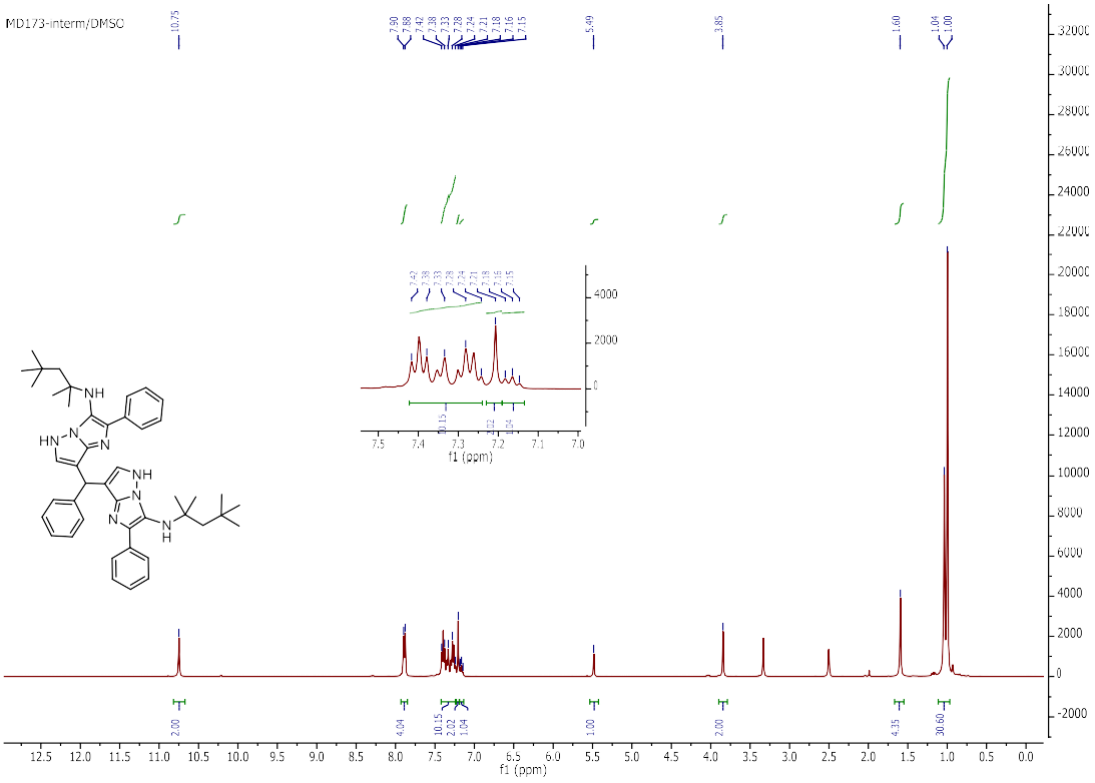


^{13}C NMR (100 MHz, $CDCl_3$)

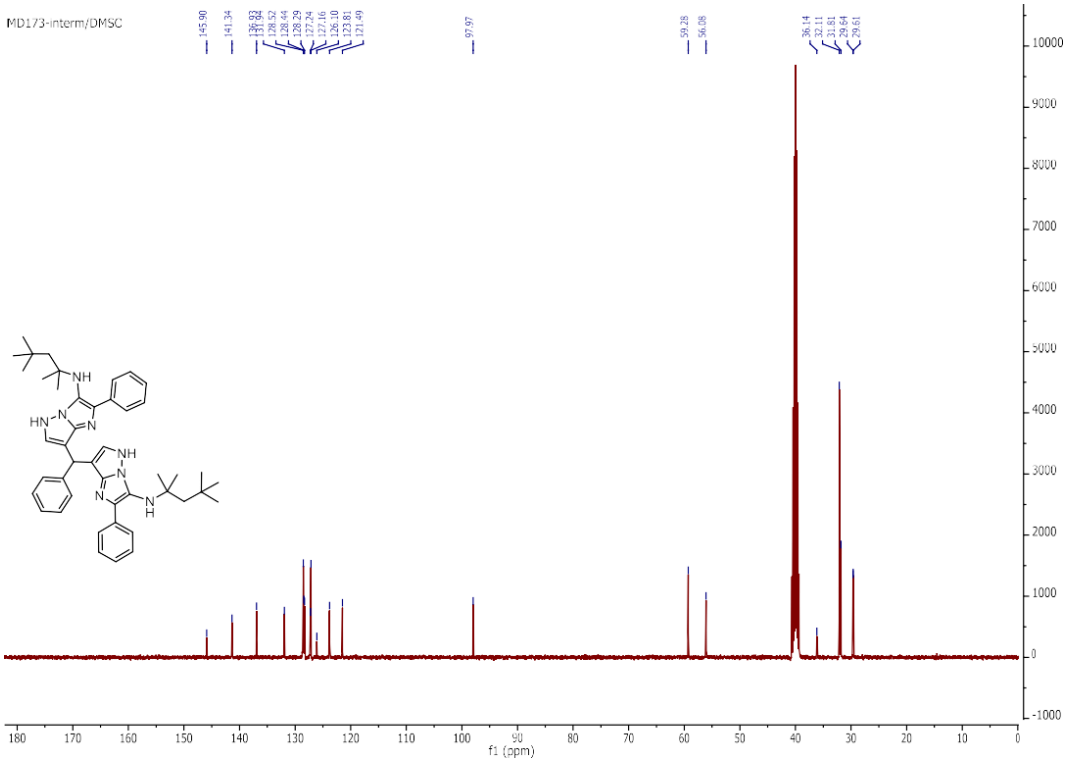


7,7'-(Phenylmethylene)bis(2-phenyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-amine) (**8a'**):

^{1}H NMR (400 MHz,DMSO-d_6)

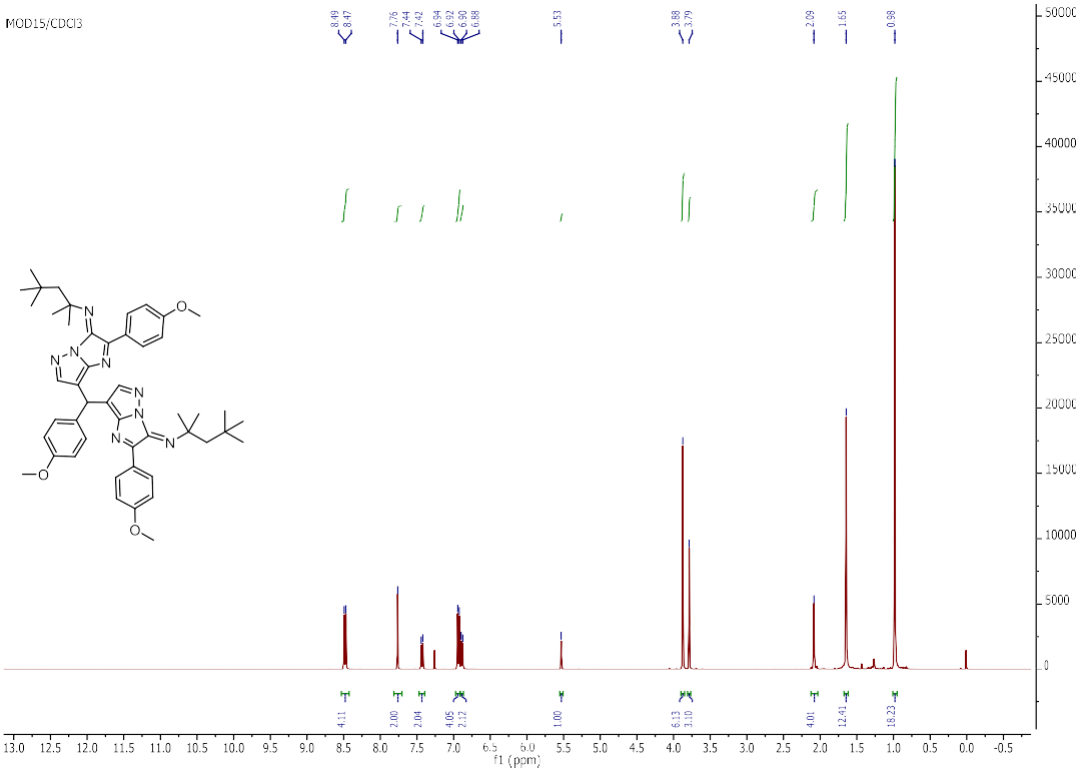


^{13}C NMR (100 MHz, DMSO-d_6)

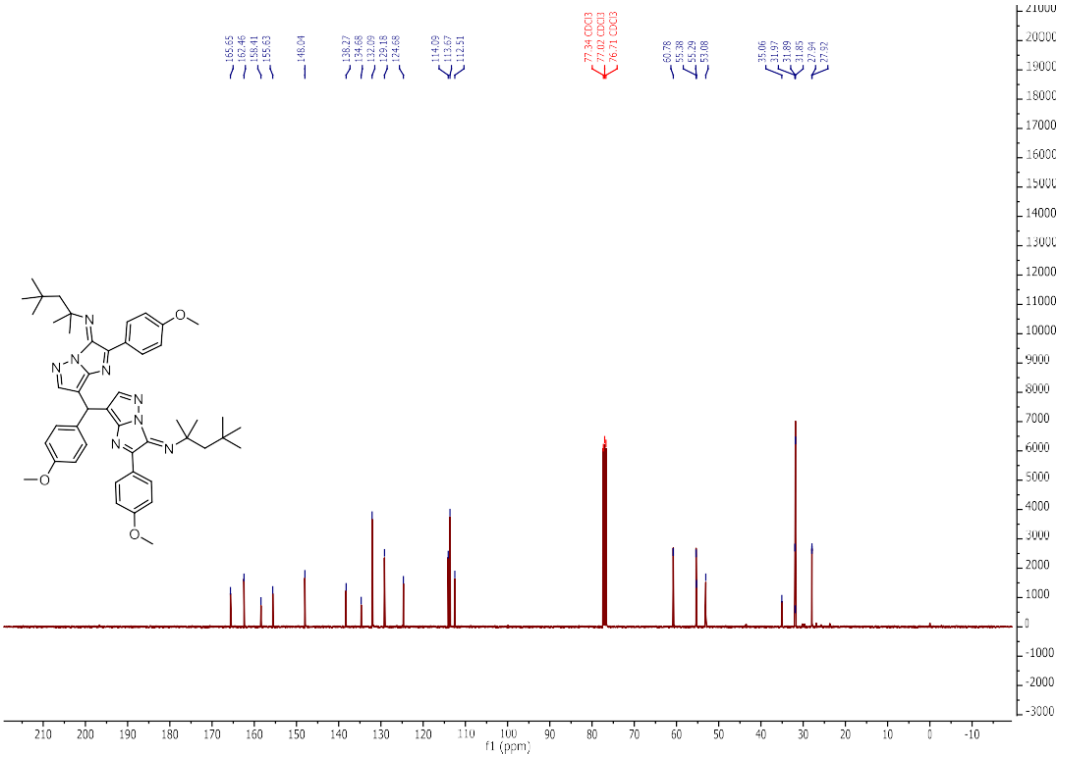


7,7'-((4-Methoxyphenyl)methylene)bis(2-(4-methoxyphenyl)-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8b**):

^{1}H NMR (400 MHz, $CDCl_3$)

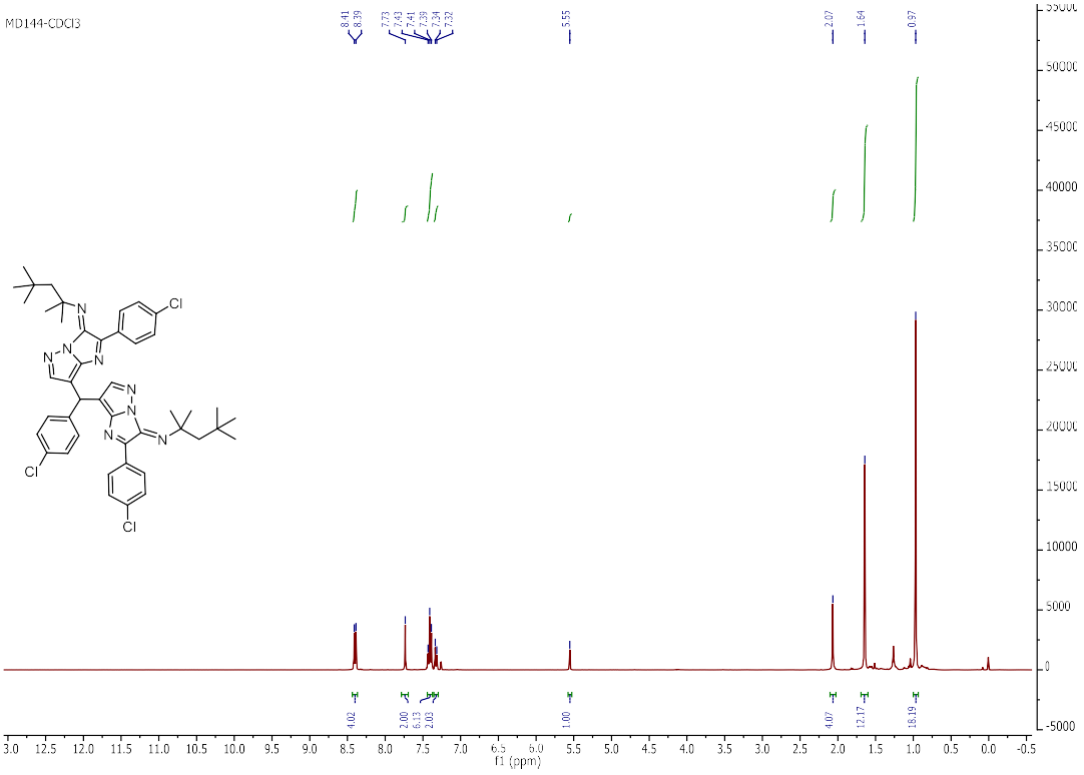


^{13}C NMR (100 MHz, $CDCl_3$)

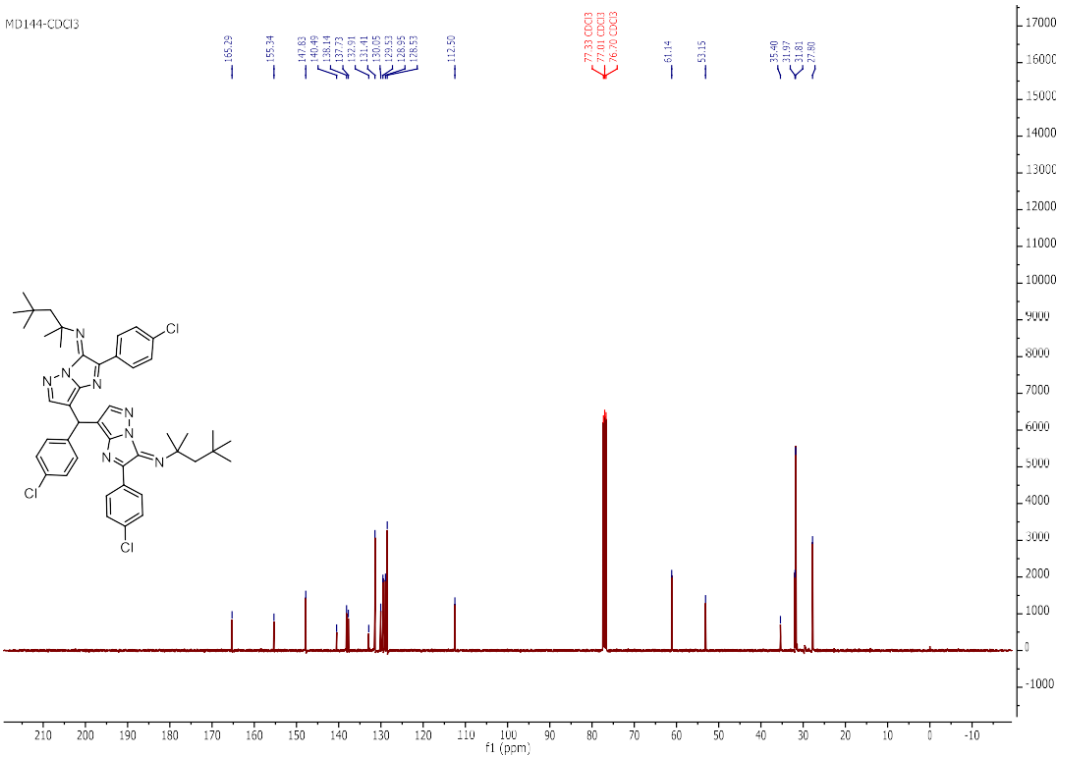


7,7'-((4-Chlorophenyl)methylene)bis(2-(4-chlorophenyl)-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8c**):

^{1}H NMR (400 MHz, $CDCl_3$)

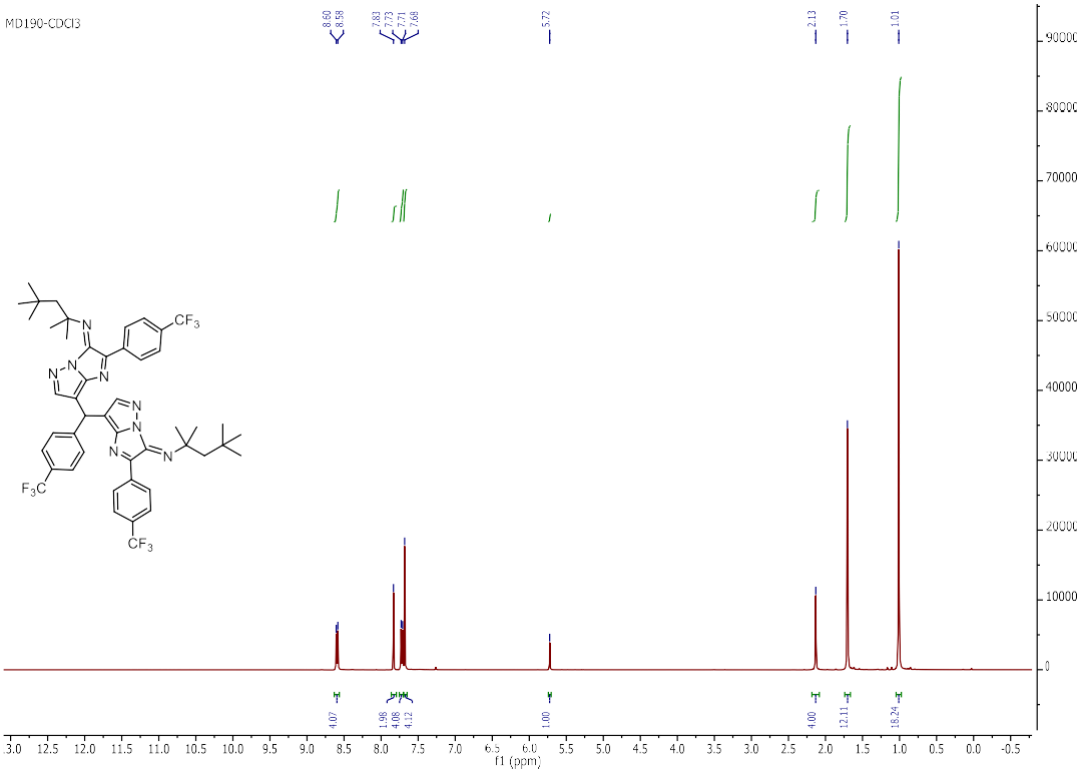


^{13}C NMR (100 MHz, $CDCl_3$)

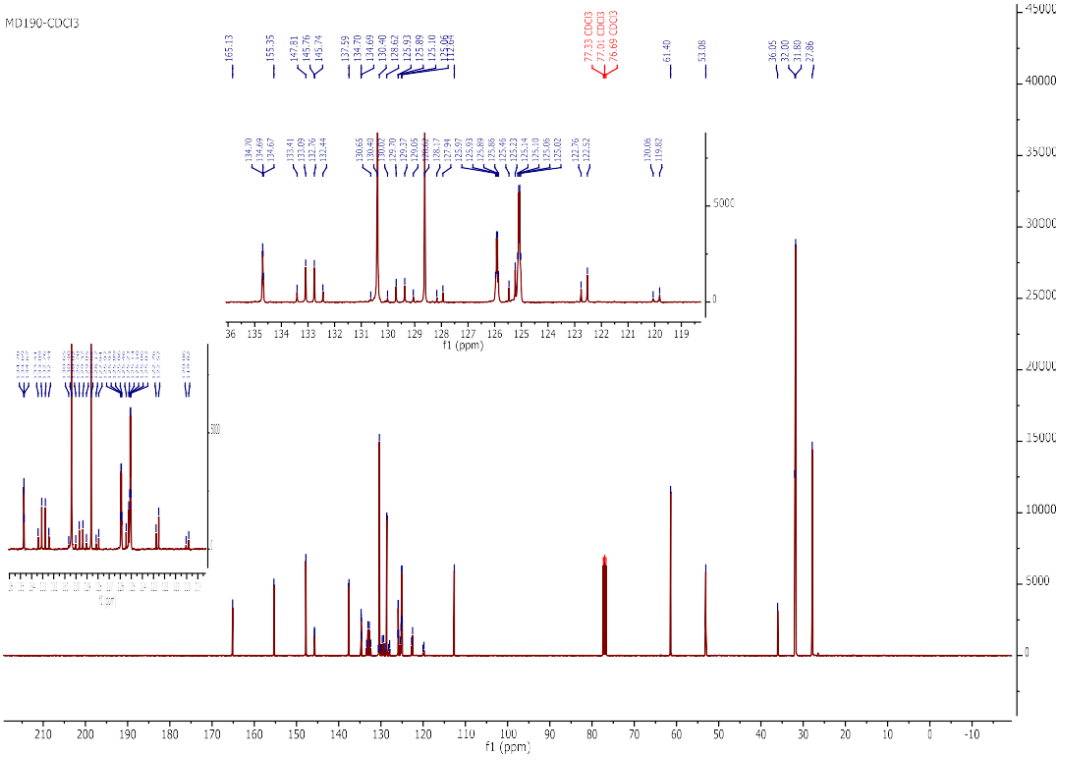


7,7'-((4-(Trifluoromethyl)phenyl)methylene)bis(2-(4-(trifluoromethyl)phenyl)-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8d**):

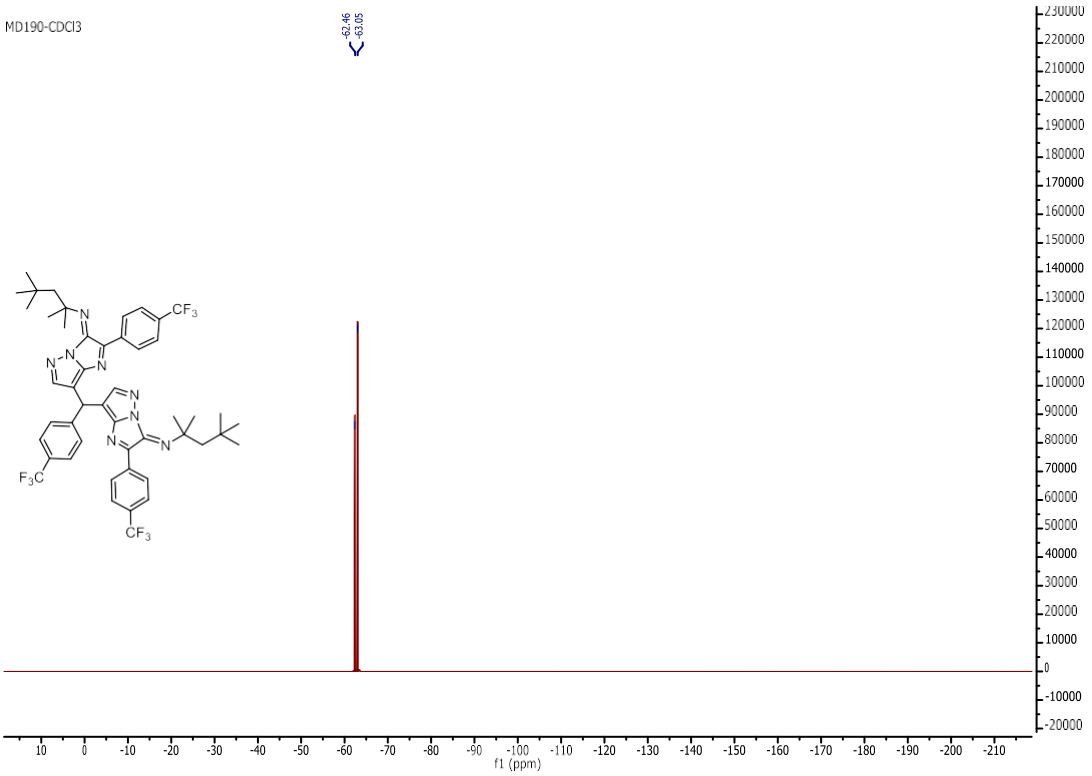
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)

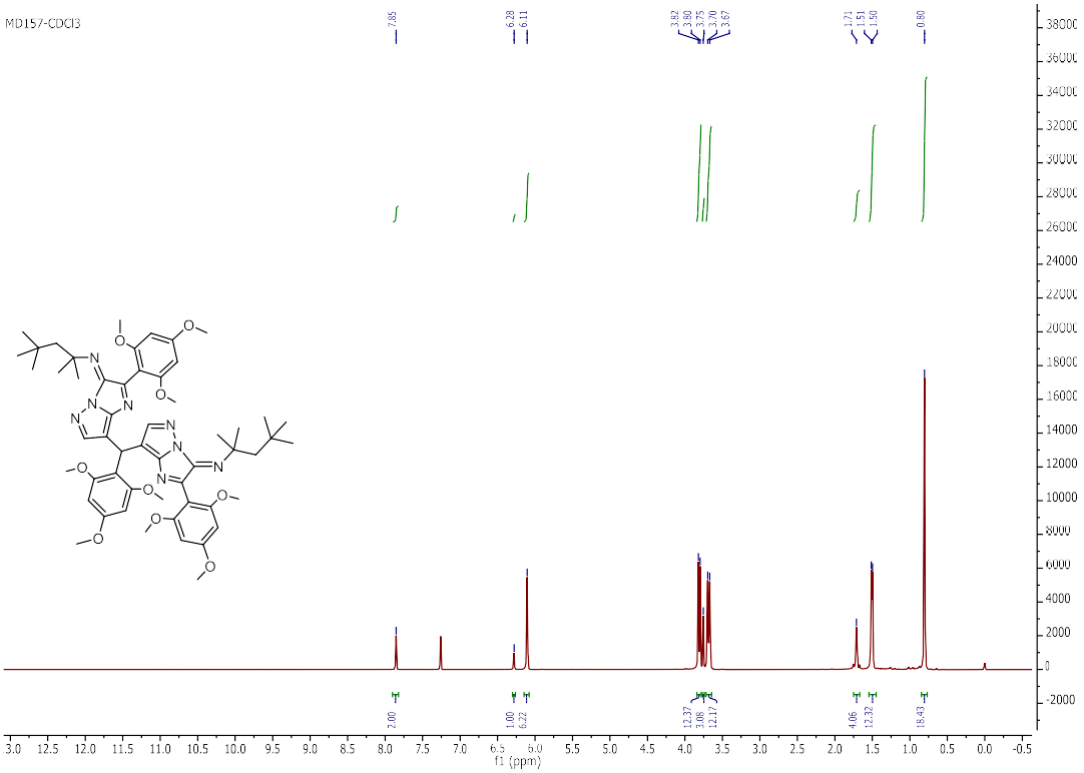


^{19}F NMR (376 MHz, $CDCl_3$)

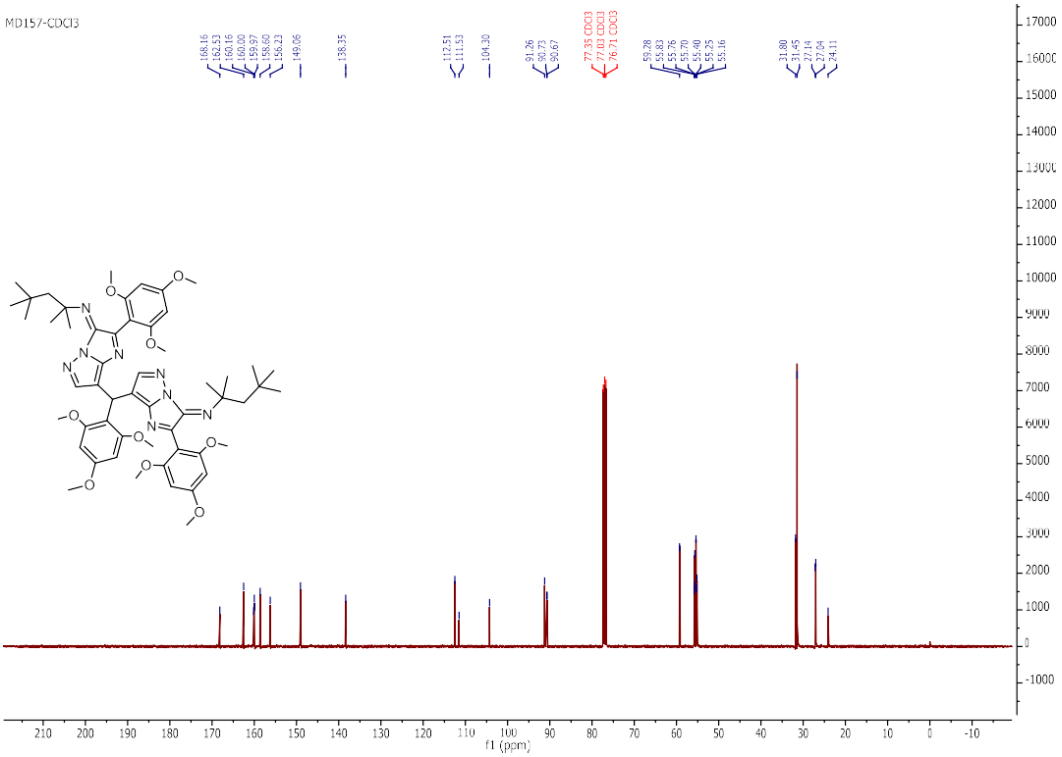


7,7'-((2,4,6-Trimethoxyphenyl)methylene)bis(2-(2,4,6-trimethoxyphenyl)-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8e**):

^{1}H NMR (400 MHz, $CDCl_3$)

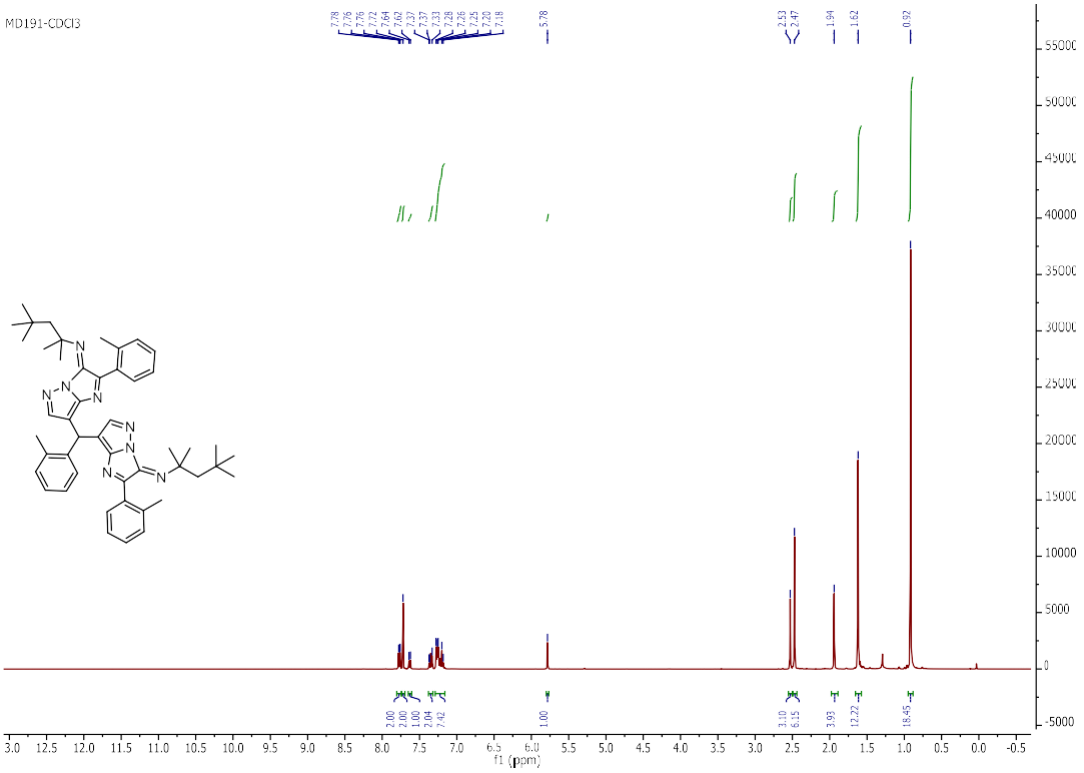


^{13}C NMR (100 MHz, $CDCl_3$)

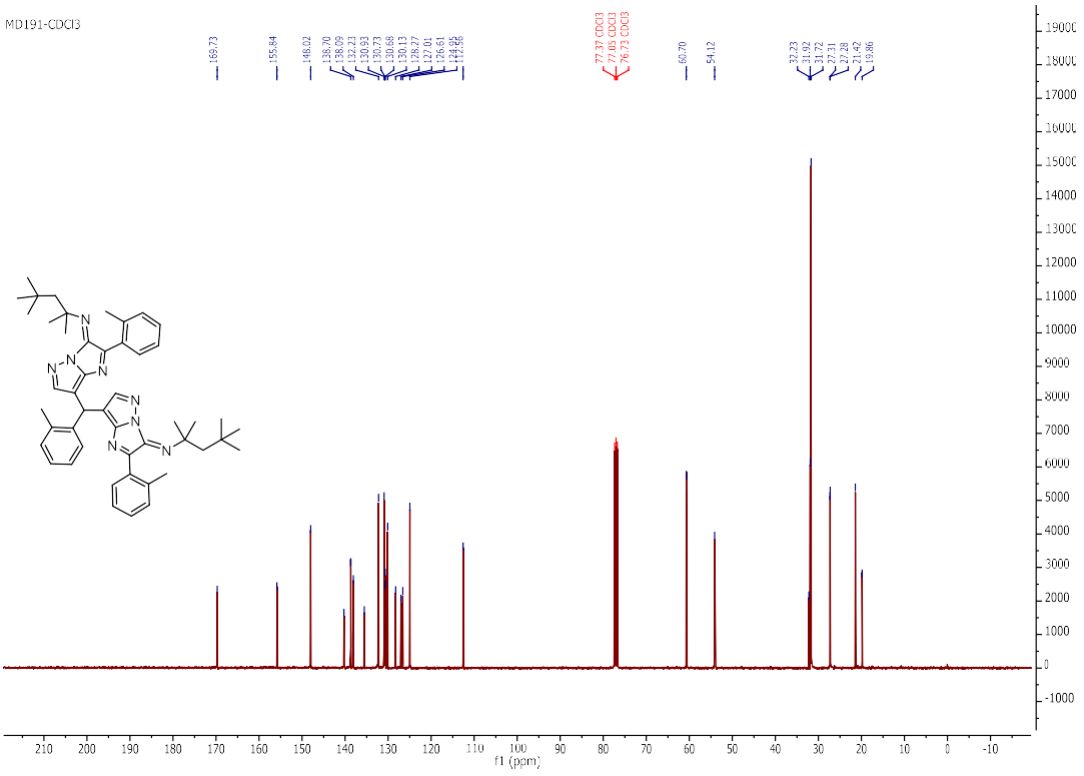


7,7'-(*o*-Tolylmethylene)bis(2-(*o*-tolyl)-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8f**):

^{1}H NMR (400 MHz, $CDCl_3$)

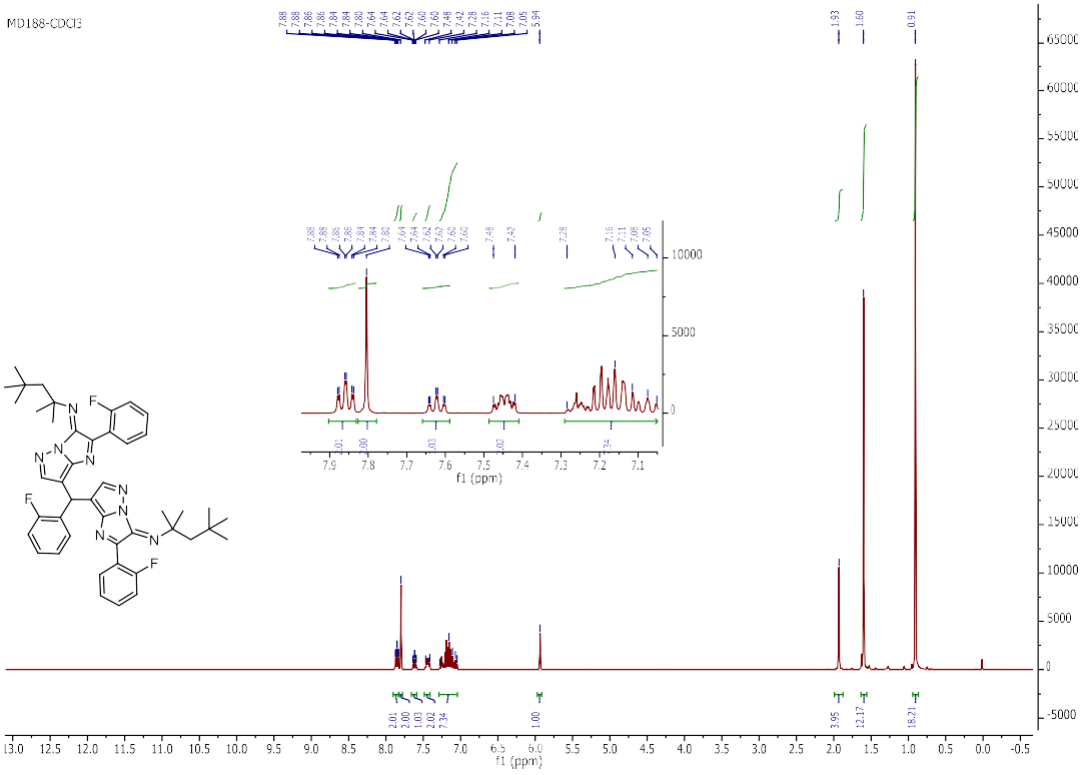


^{13}C NMR (100 MHz, $CDCl_3$)

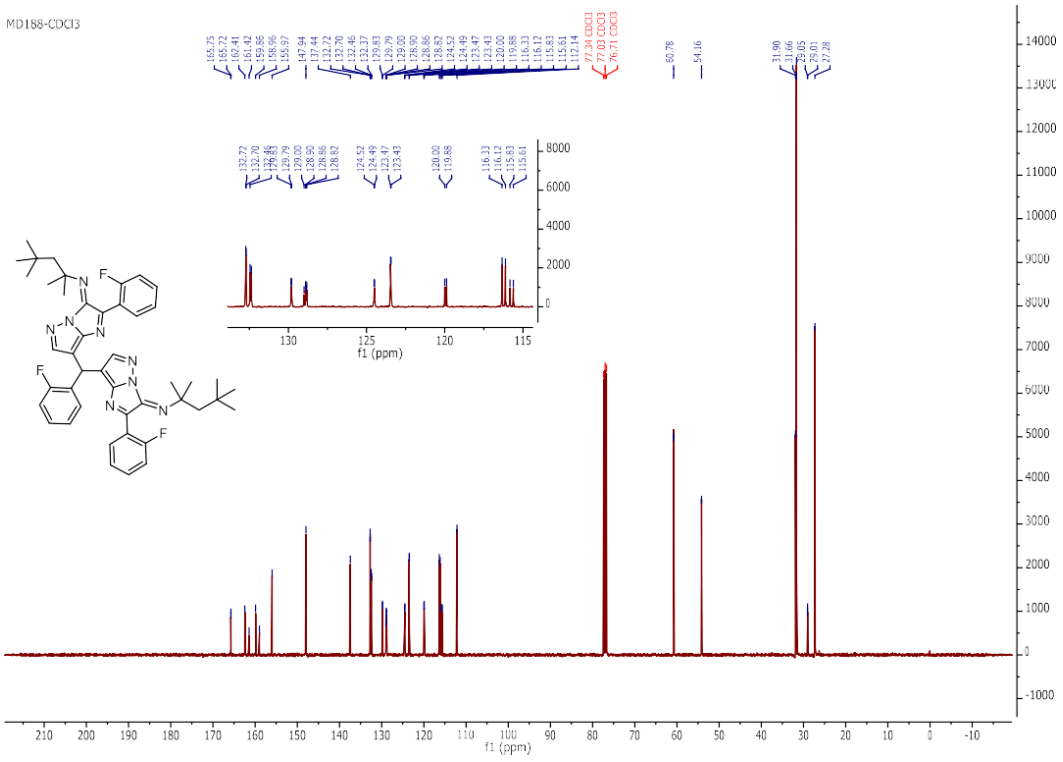


7,7'-((2-Fluorophenyl)methylene)bis(2-(2-fluorophenyl)-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8g**):

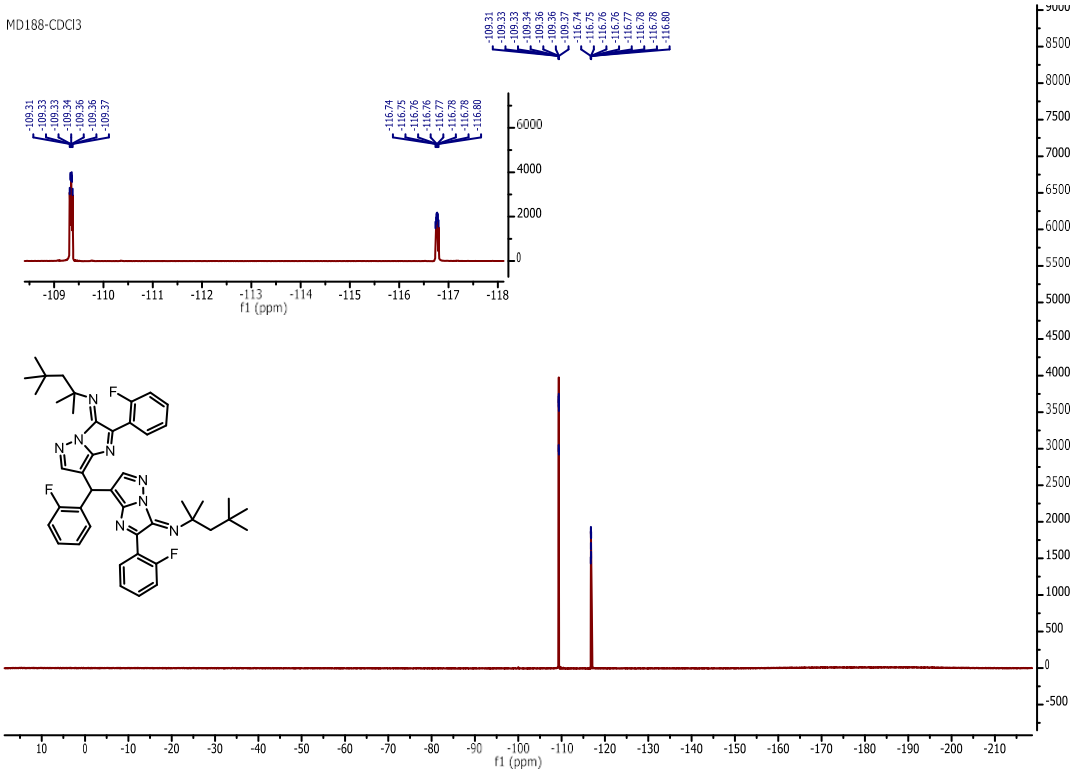
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)

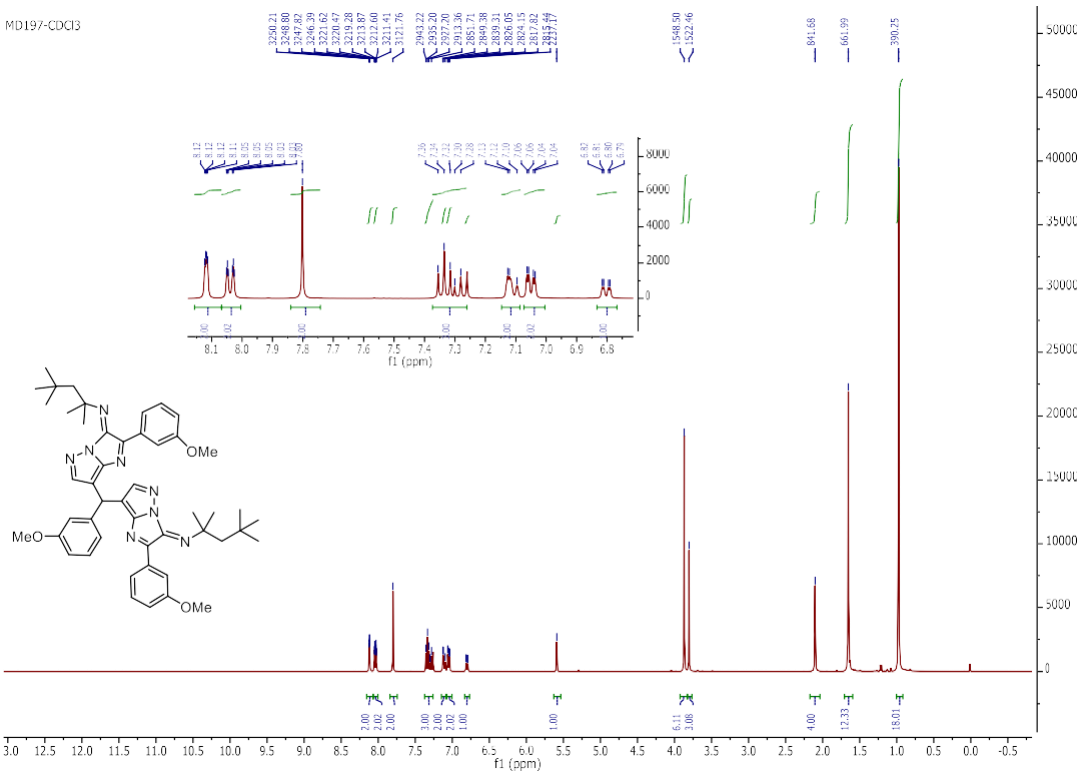


^{19}F NMR (376 MHz, $CDCl_3$)

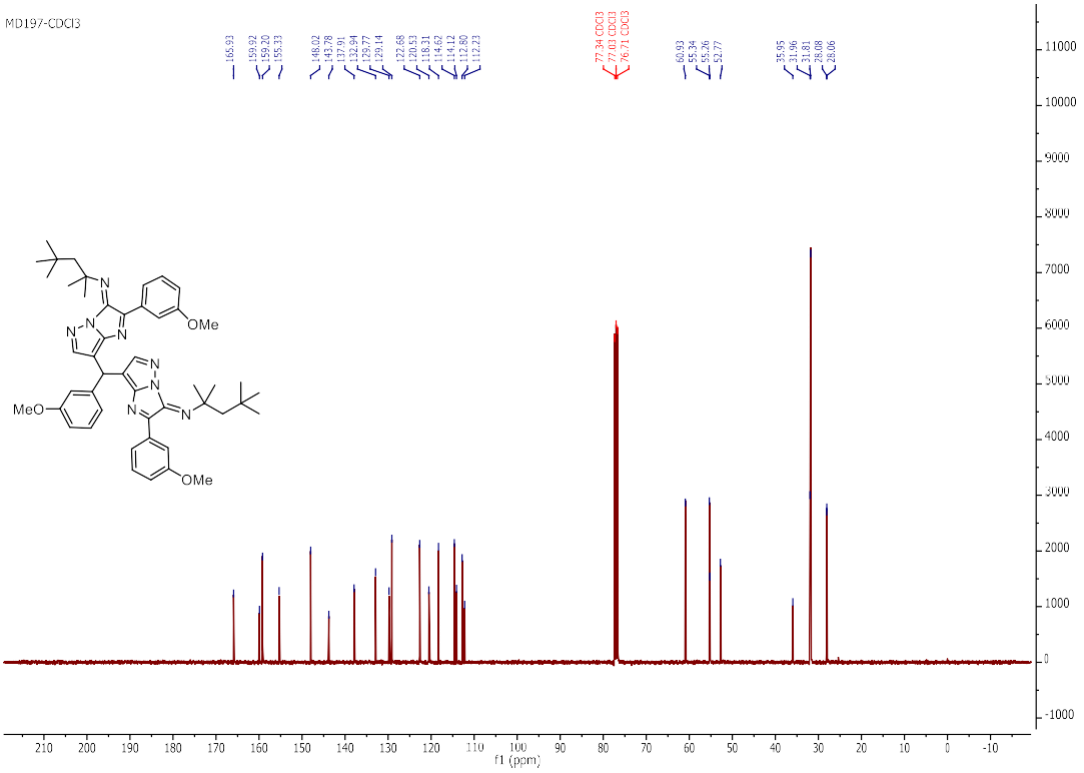


7,7'-((3-Methoxyphenyl)methylene)bis(2-(3-methoxyphenyl)-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8h**):

^{1}H NMR (400 MHz, $CDCl_3$)

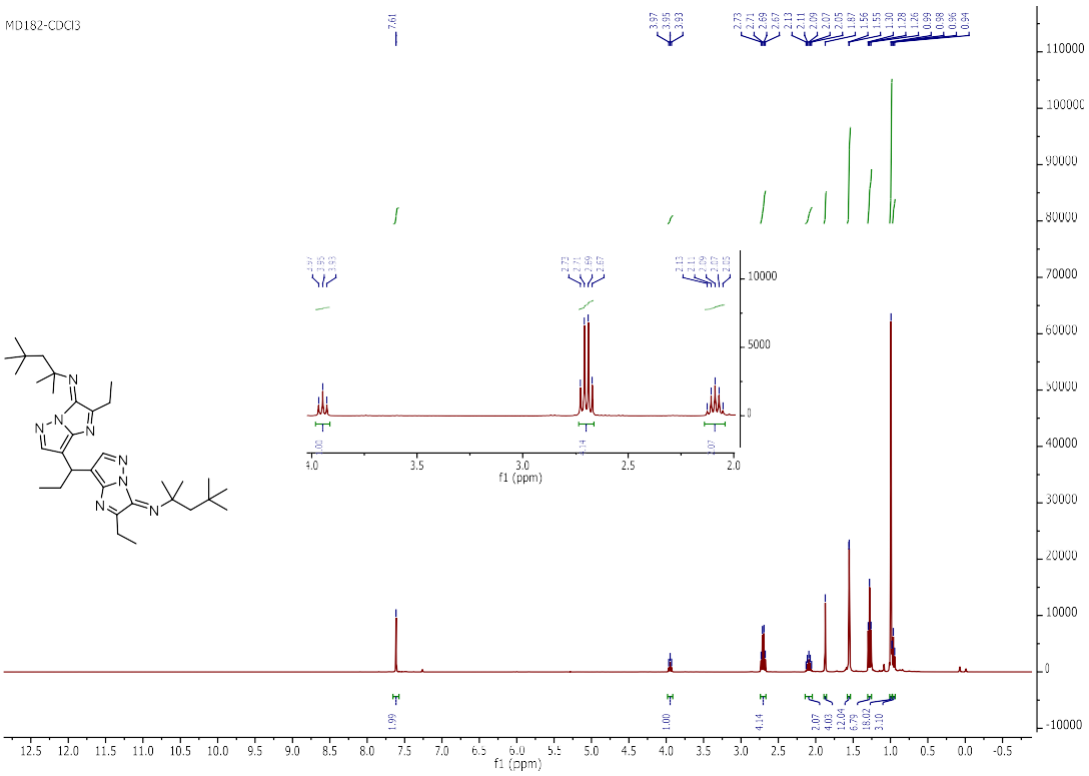


^{13}C NMR (100 MHz, $CDCl_3$)

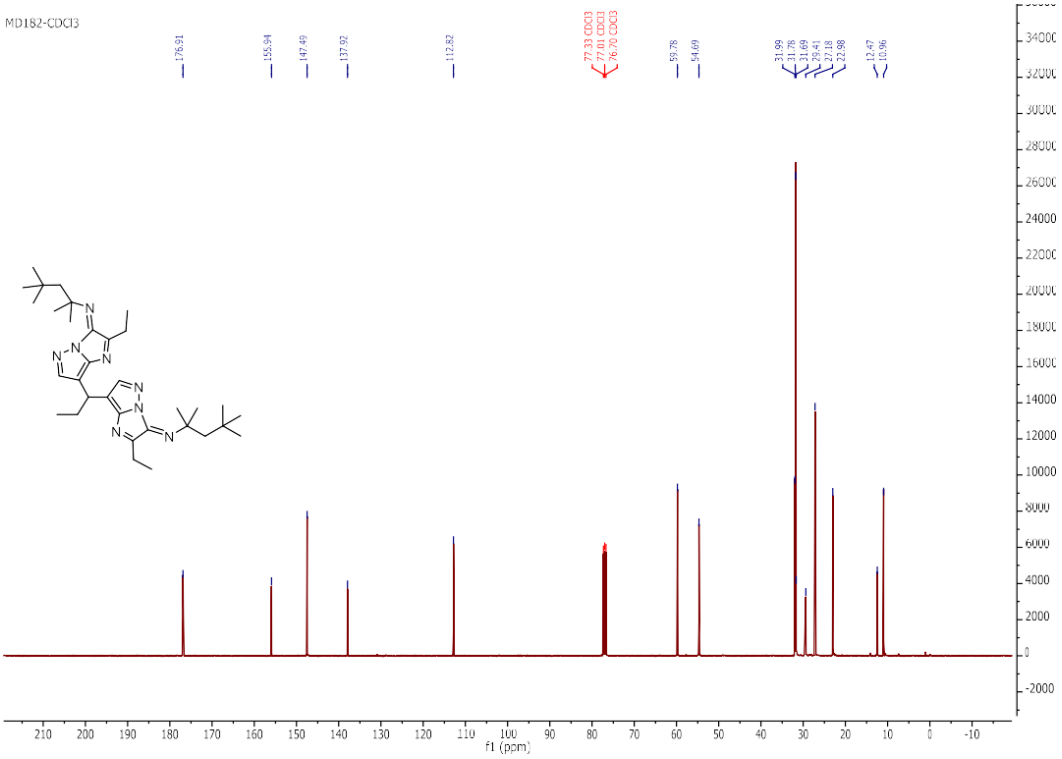


7,7'-(Propane-1,1-diyl)bis(2-ethyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8i**):

^{1}H NMR (400 MHz, $CDCl_3$)

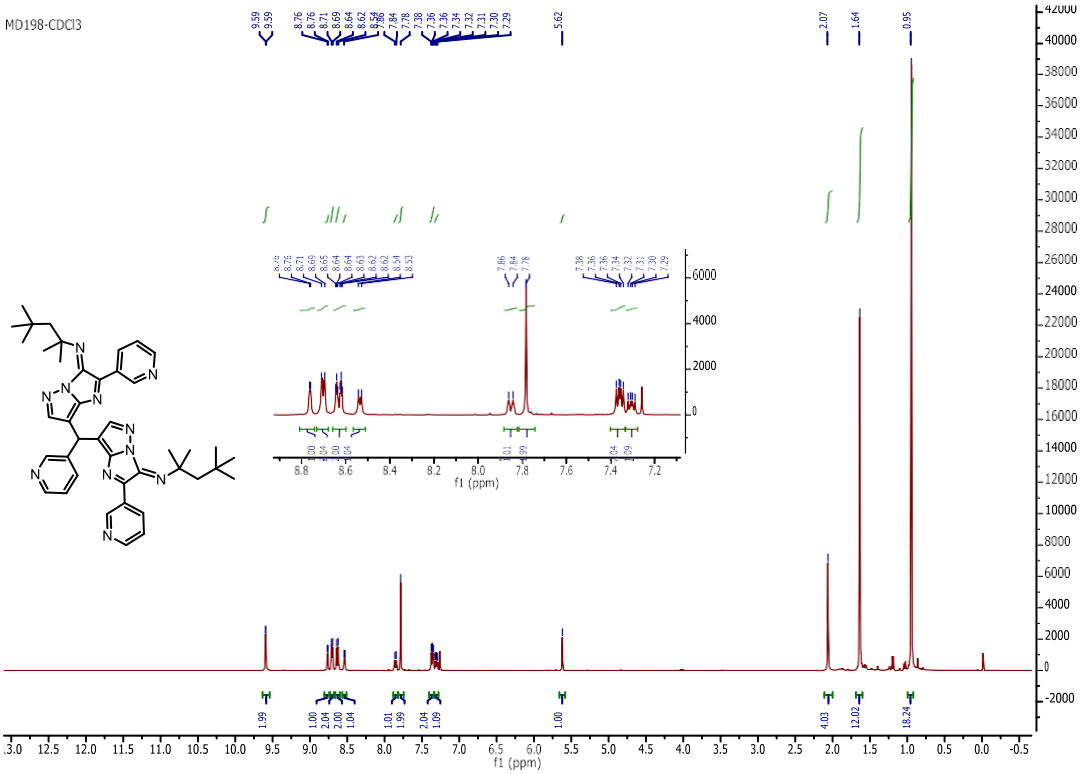


^{13}C NMR (100 MHz, $CDCl_3$)

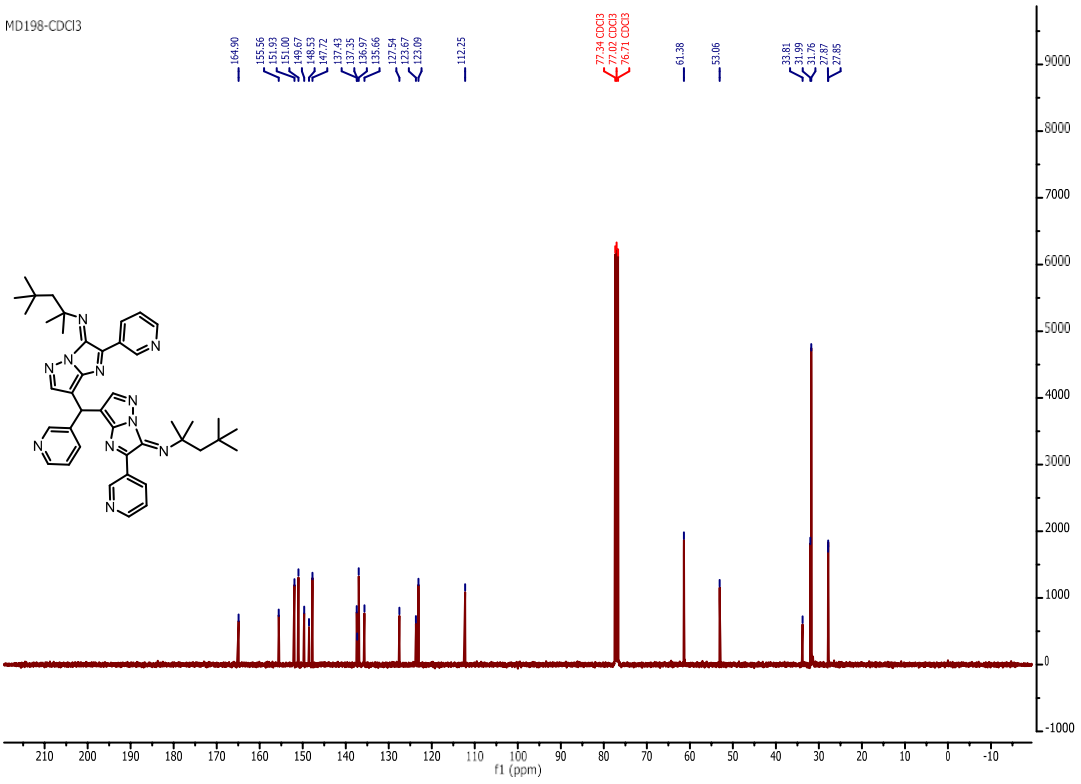


7,7'-(Pyridin-3-ylmethylene)bis(2-(pyridin-3-yl)-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8j**):

^{1}H NMR (400 MHz, $CDCl_3$)

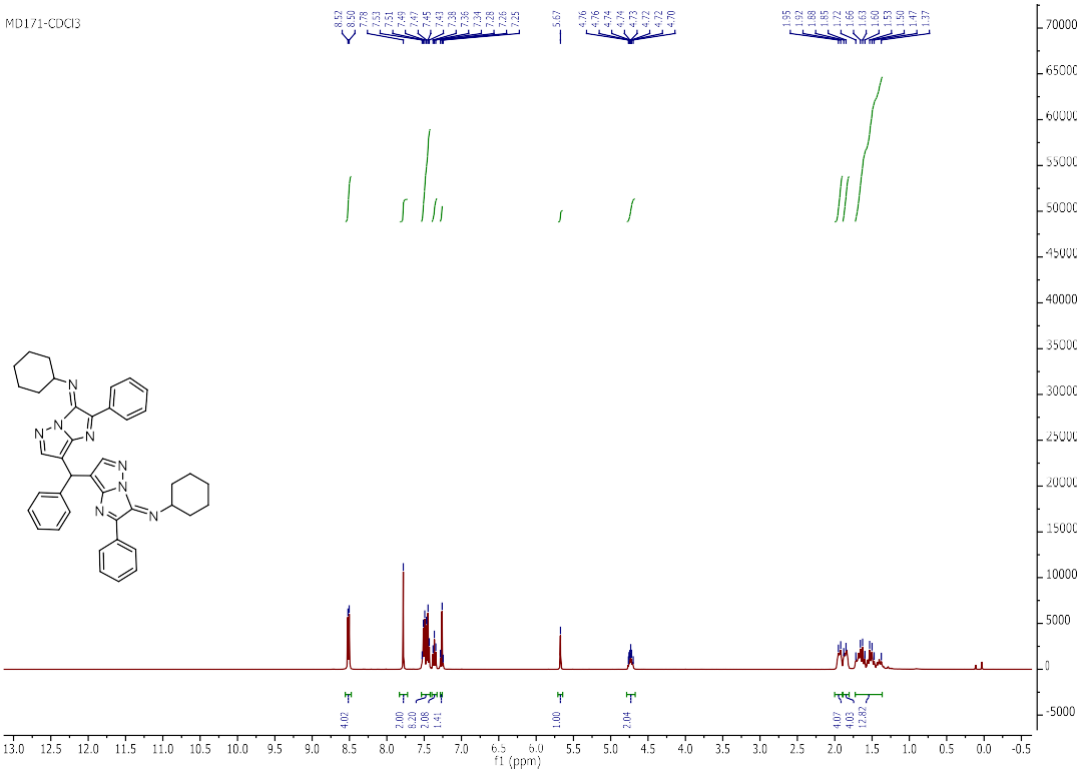


^{13}C NMR (100 MHz, $CDCl_3$)

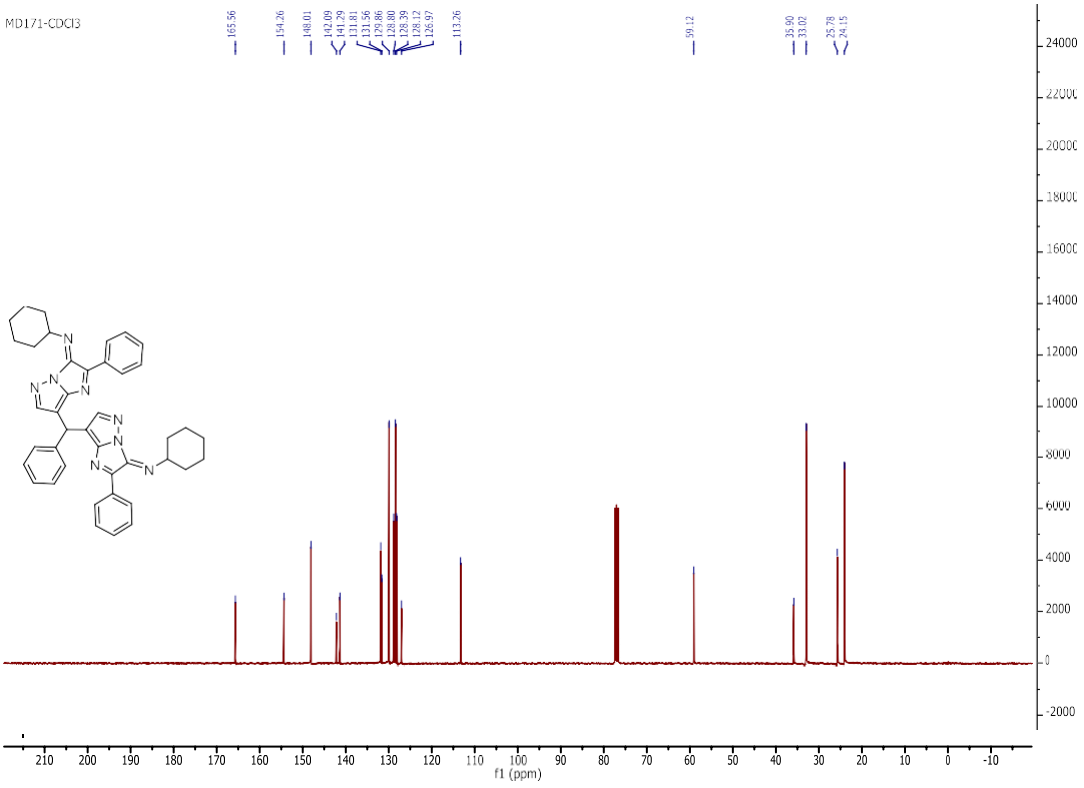


7,7'-(Phenylmethylene)bis(*N*-cyclohexyl-2-phenyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8k**):

^{1}H NMR (400 MHz, $CDCl_3$)

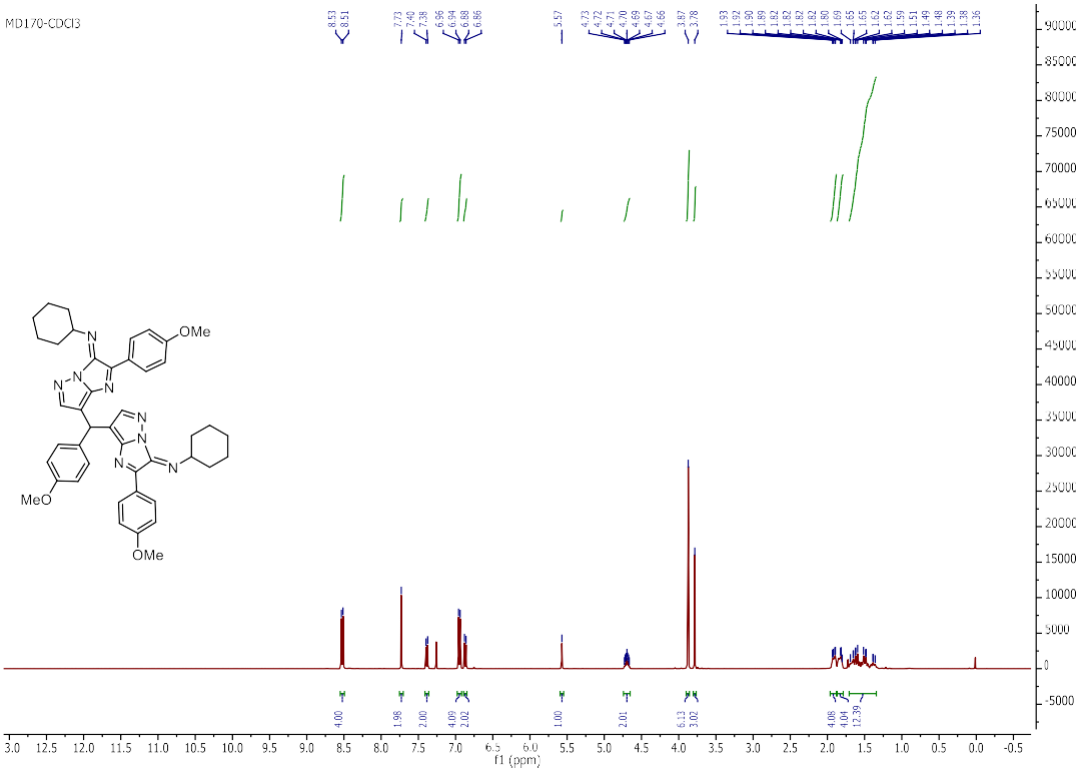


^{13}C NMR (100 MHz, $CDCl_3$)

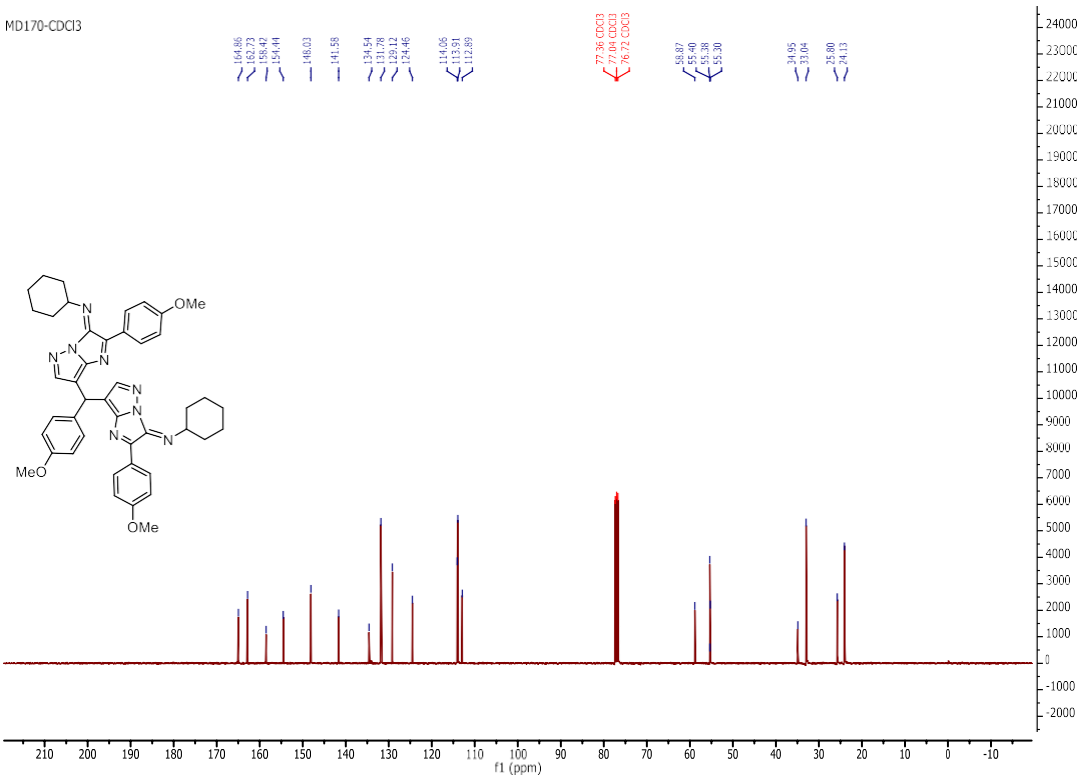


7,7'-((4-Methoxyphenyl)methylene)bis(*N*-cyclohexyl-2-(4-methoxyphenyl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8l**):

^{1}H NMR (400 MHz, $CDCl_3$)

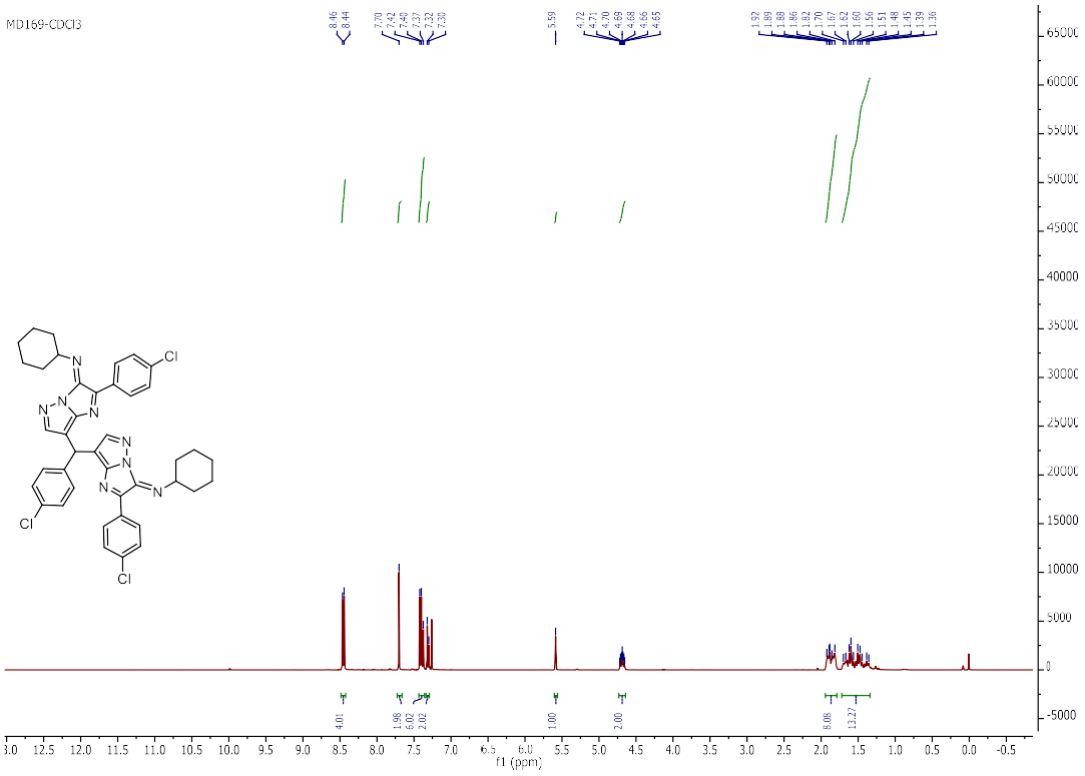


^{13}C NMR (100 MHz, $CDCl_3$)

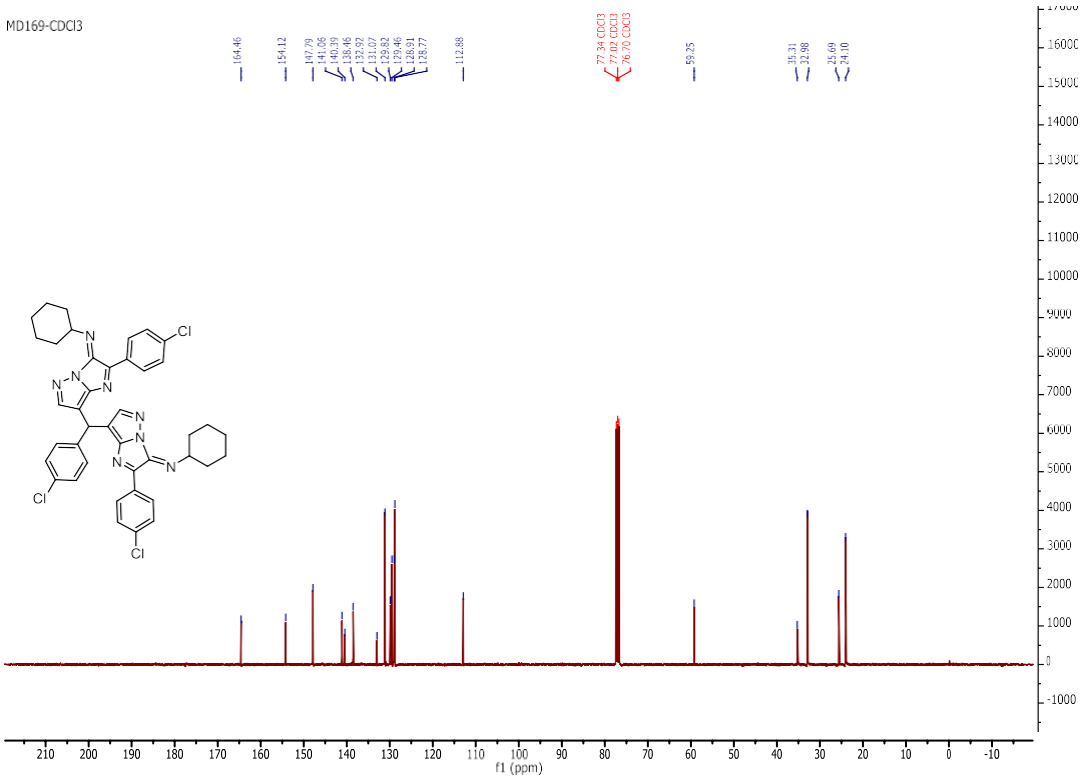


7,7'-((4-Chlorophenyl)methylene)bis(2-(4-chlorophenyl)-*N*-cyclohexyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8m**):

^{1}H NMR (400 MHz, $CDCl_3$)

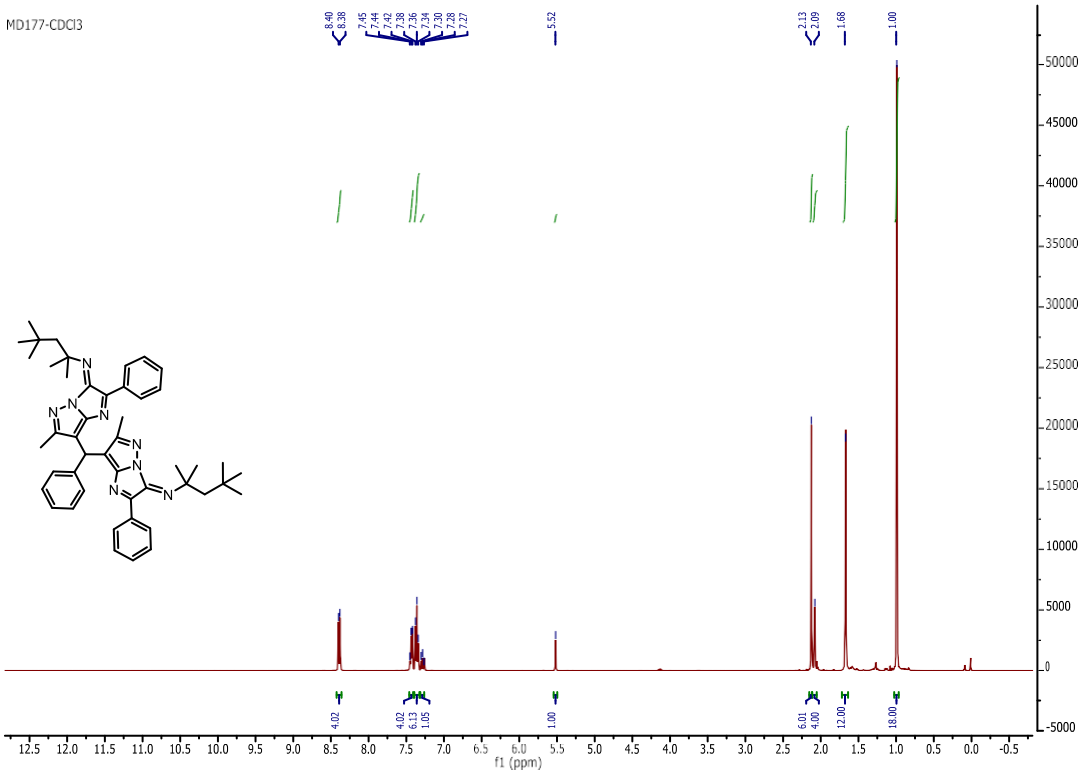


^{13}C NMR (100 MHz, $CDCl_3$)

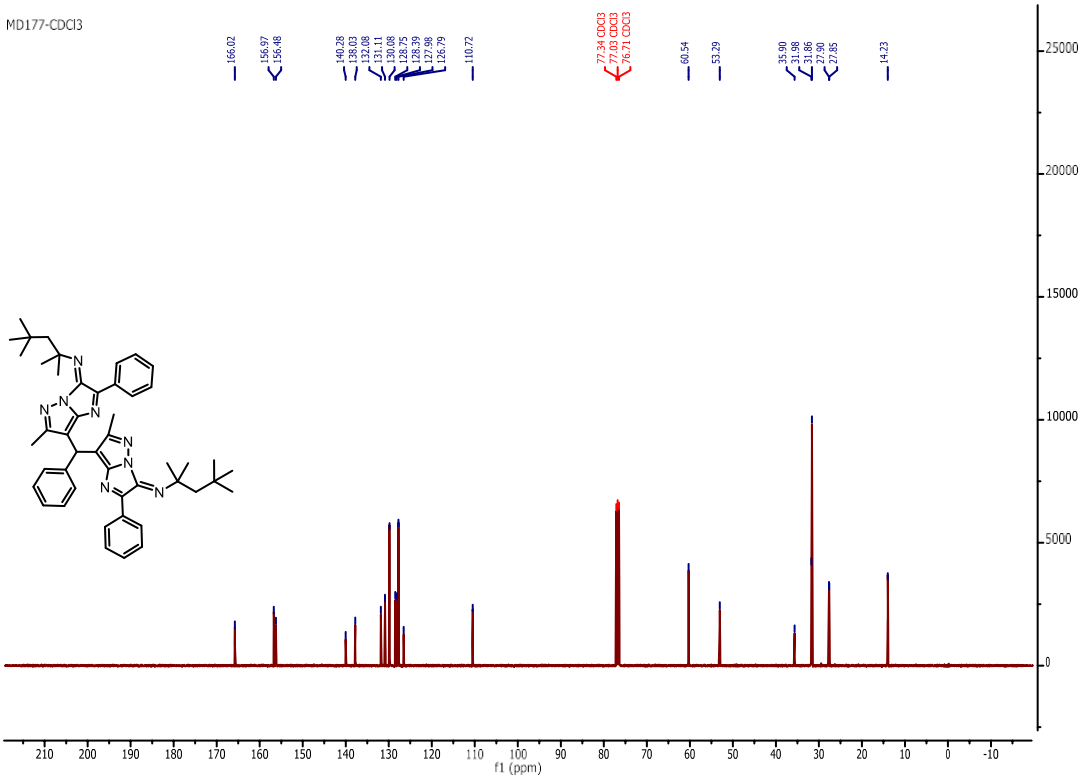


7,7'-(Phenylmethylene)bis(6-methyl-2-phenyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8n**):

^{1}H NMR (400 MHz, $CDCl_3$)

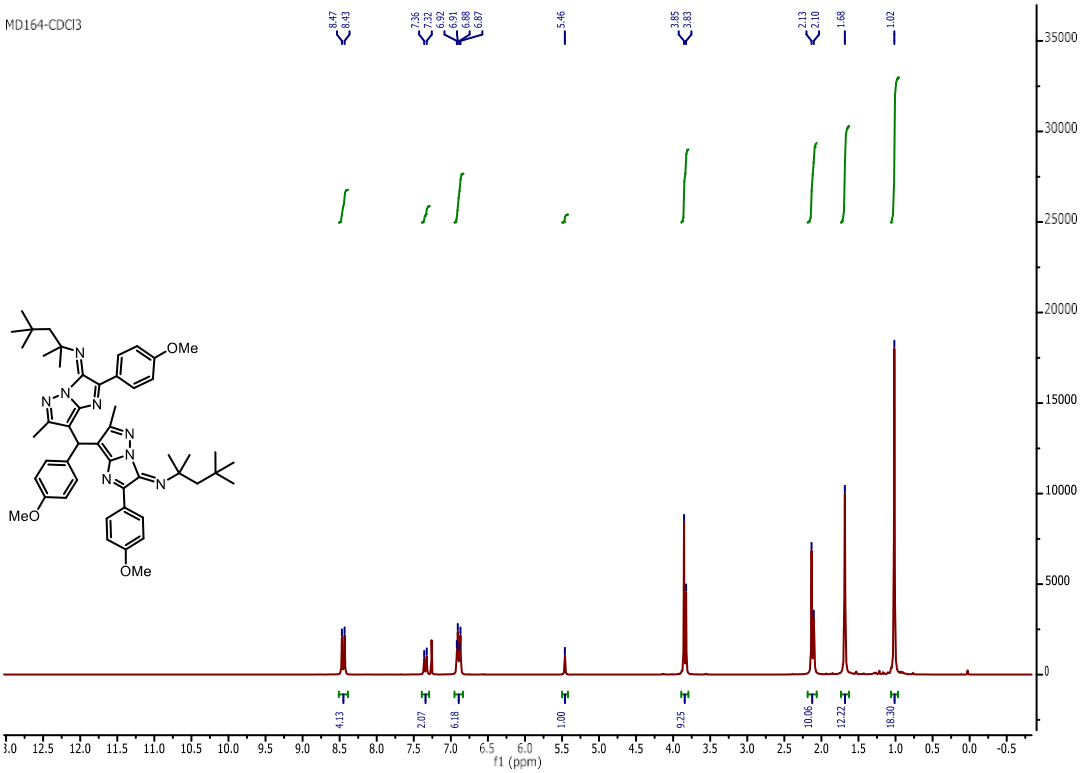


^{13}C NMR (100 MHz, $CDCl_3$)

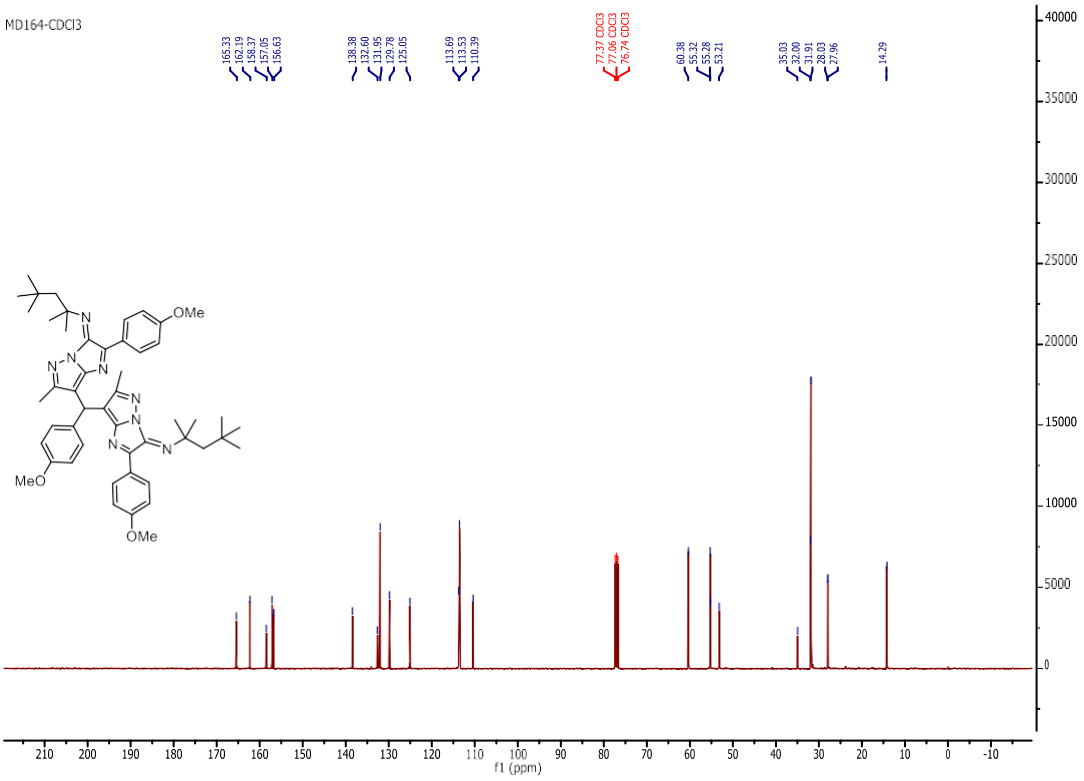


7,7'-((4-Methoxyphenyl)methylene)bis(2-(4-methoxyphenyl)-6-methyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8o**):

^{1}H NMR (250 MHz, $CDCl_3$)

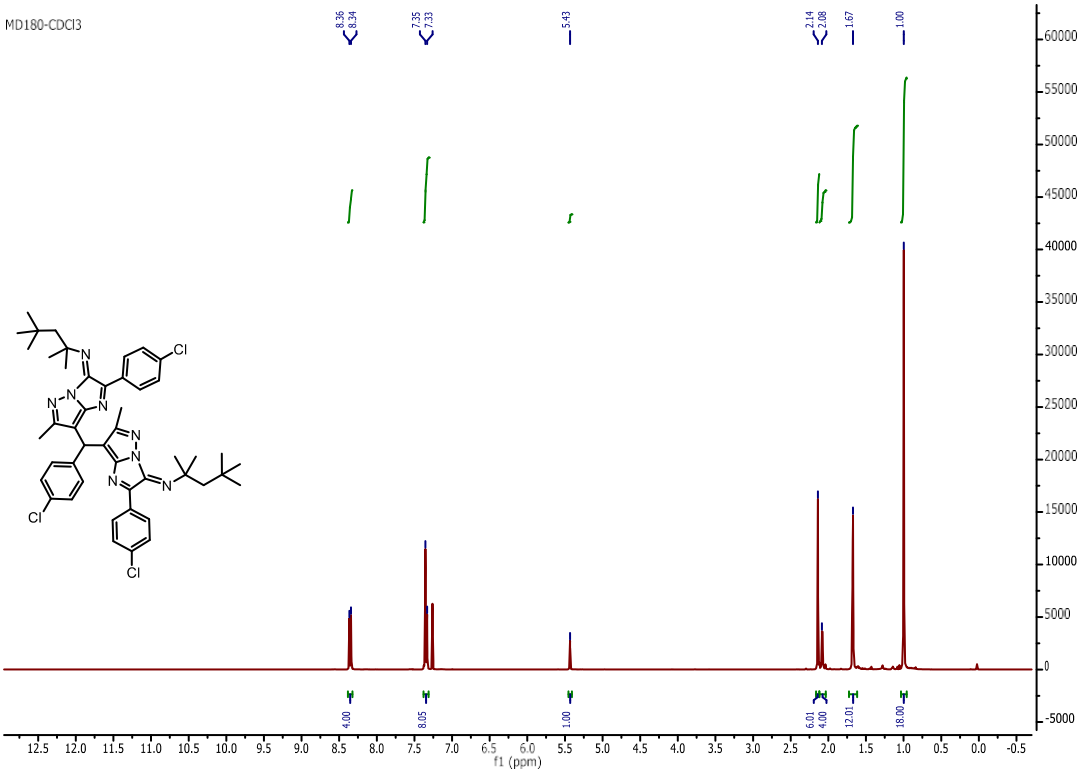


^{13}C NMR (100 MHz, $CDCl_3$)

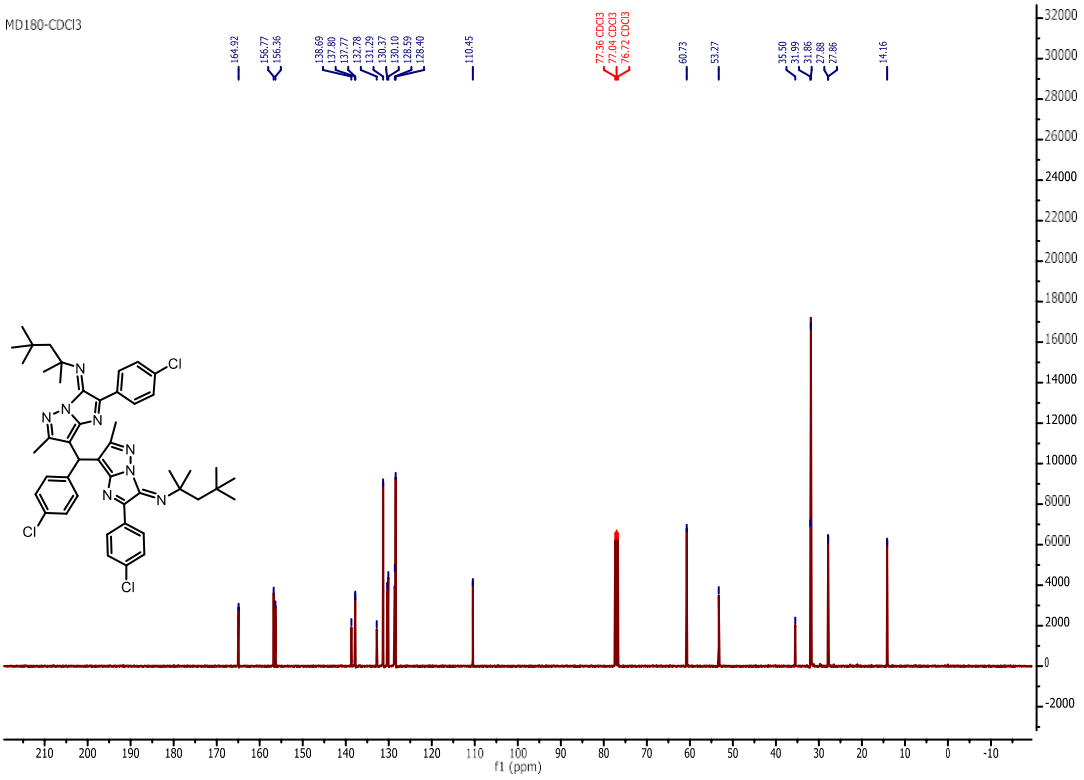


7,7'-((4-Chlorophenyl)methylene)bis(2-(4-chlorophenyl)-6-methyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8p**):

^{1}H NMR (400 MHz, $CDCl_3$)

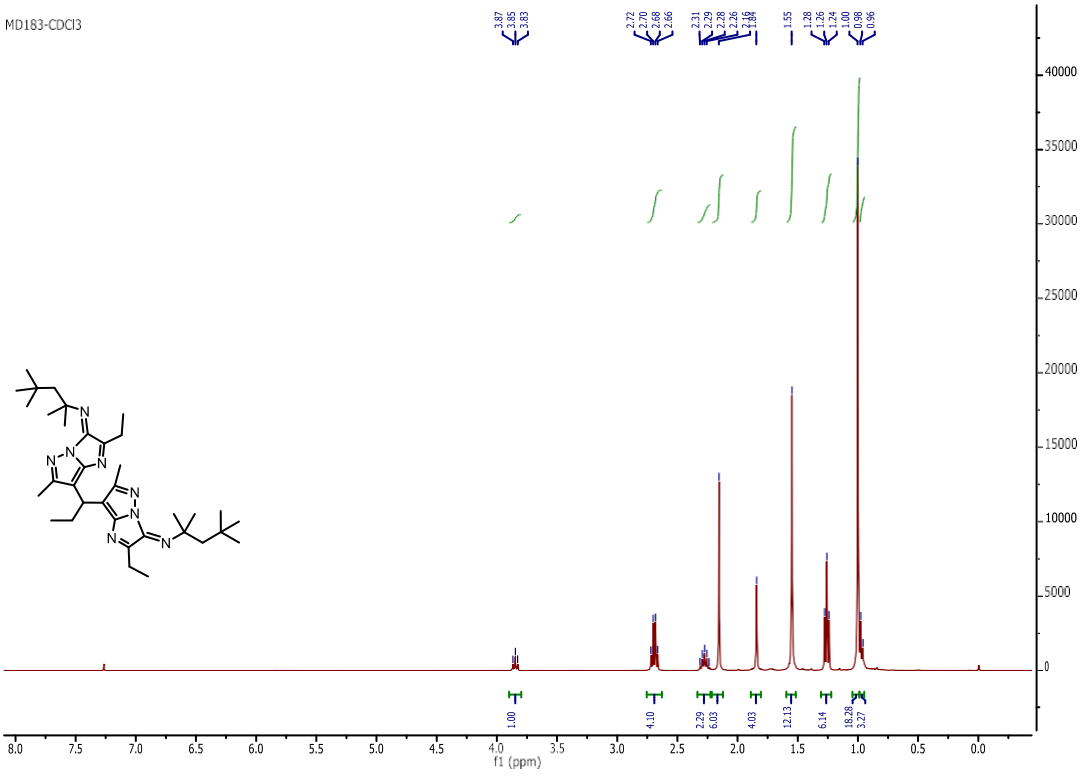


^{13}C NMR (100 MHz, $CDCl_3$)

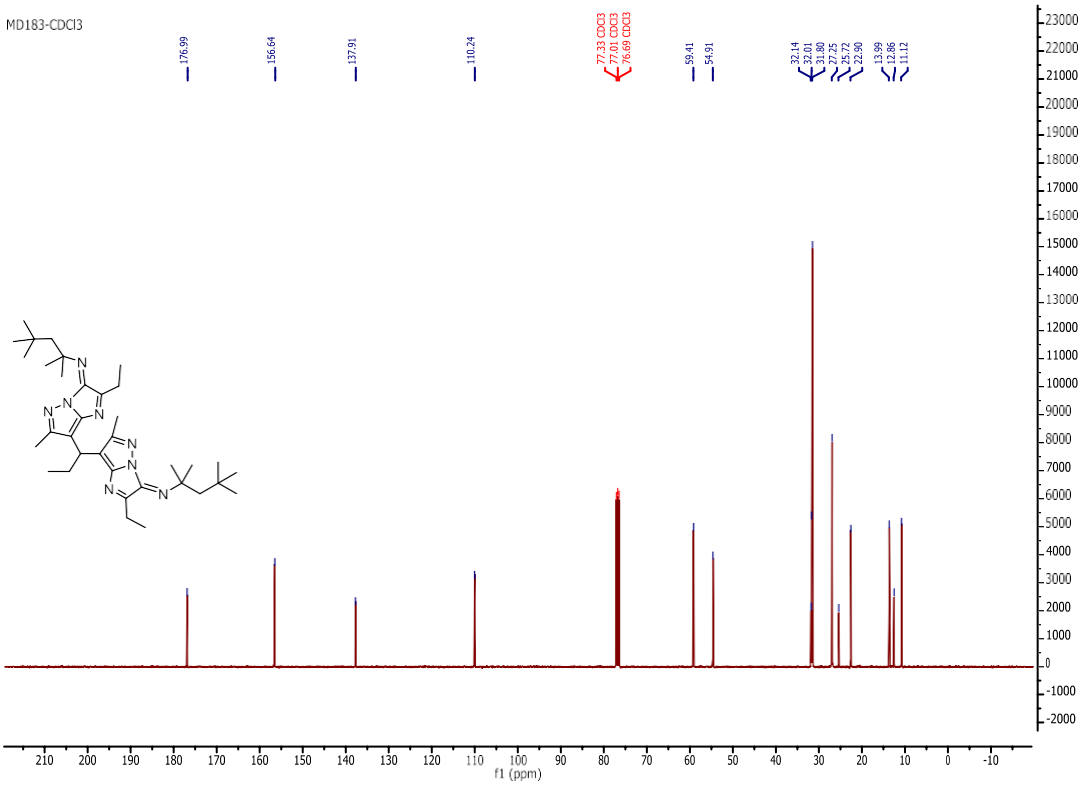


7,7'-(Propane-1,1-diyl)bis(2-ethyl-6-methyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8q**):

^{1}H NMR (400 MHz, $CDCl_3$)

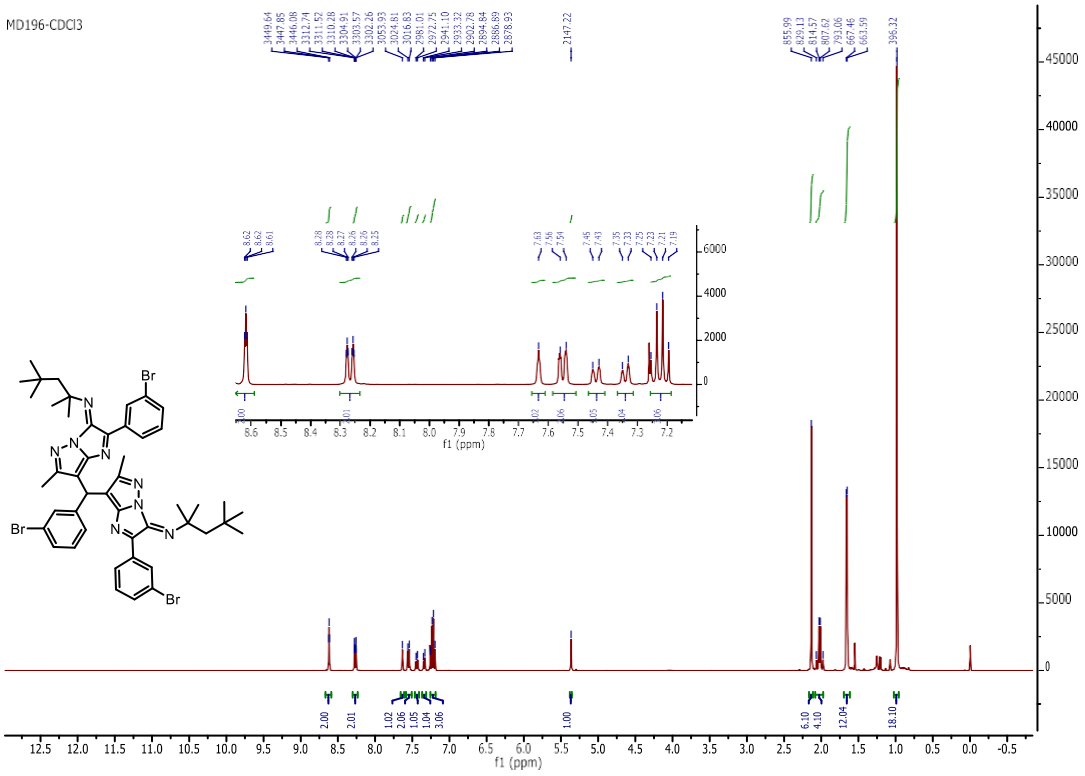


^{13}C NMR (100 MHz, $CDCl_3$)

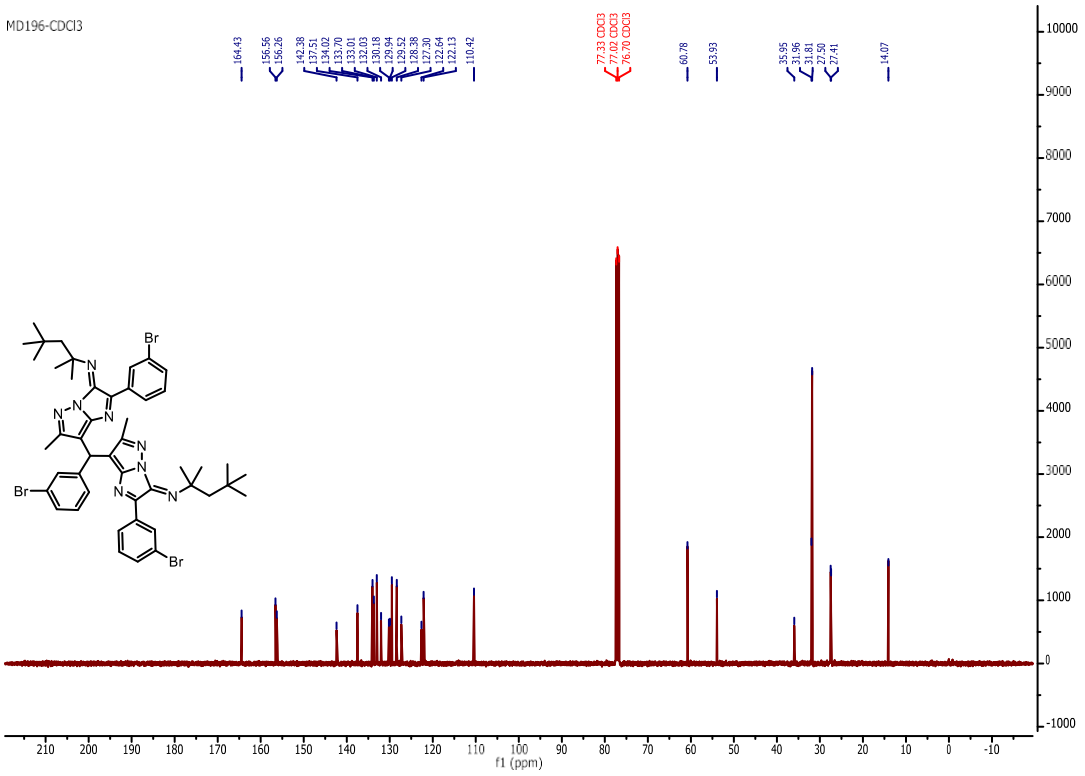


7,7'-((3-Bromophenyl)methylene)bis(2-(3-bromophenyl)-6-methyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8r**):

^{1}H NMR (400 MHz, $CDCl_3$)

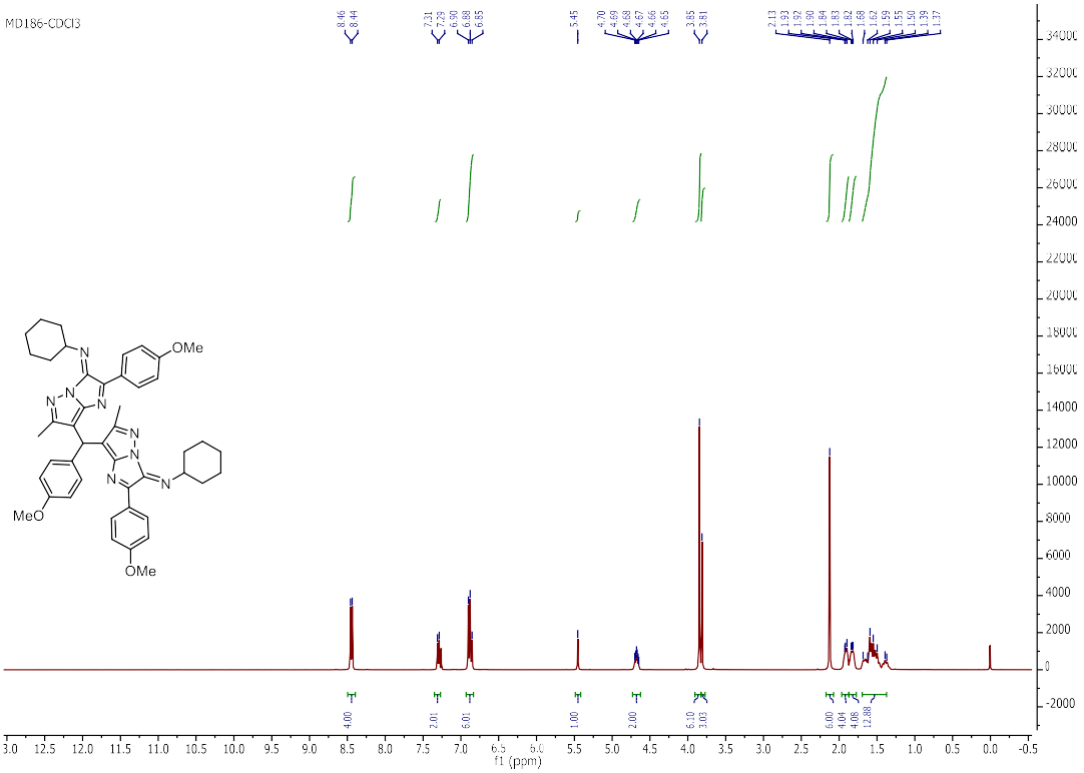


^{13}C NMR (100 MHz, $CDCl_3$)

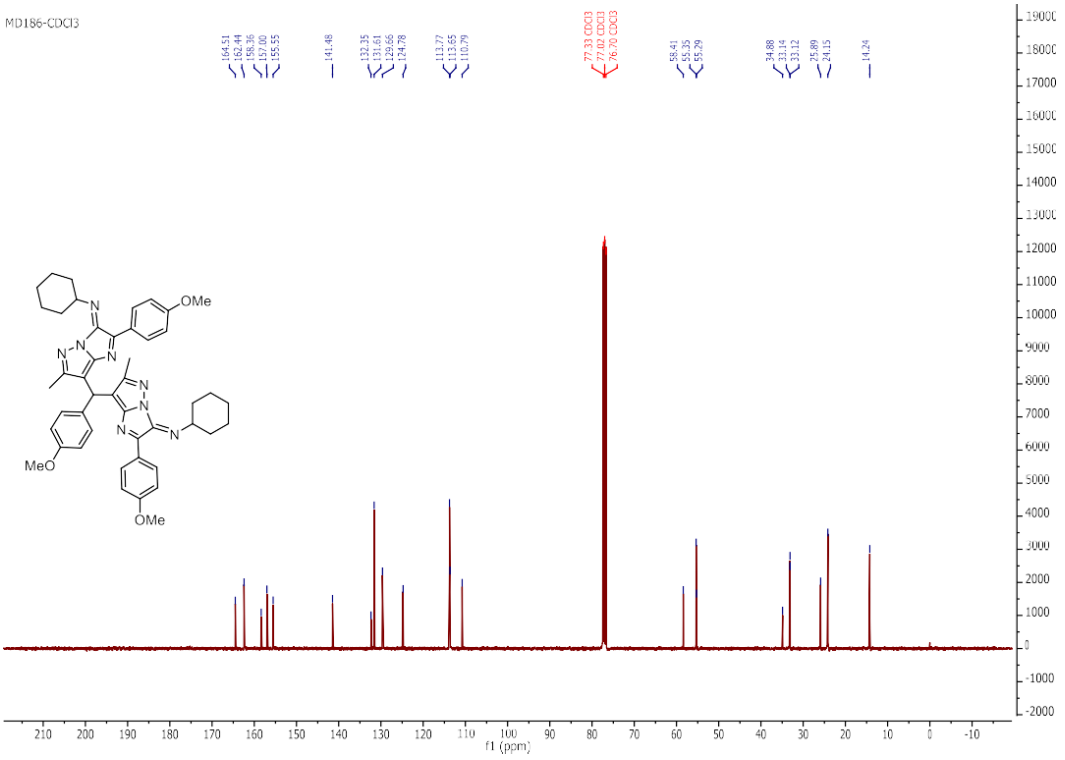


7,7'-((4-Methoxyphenyl)methylene)bis(*N*-cyclohexyl-2-(4-methoxyphenyl)-6-methyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8s**):

^{1}H NMR (400 MHz, $CDCl_3$)

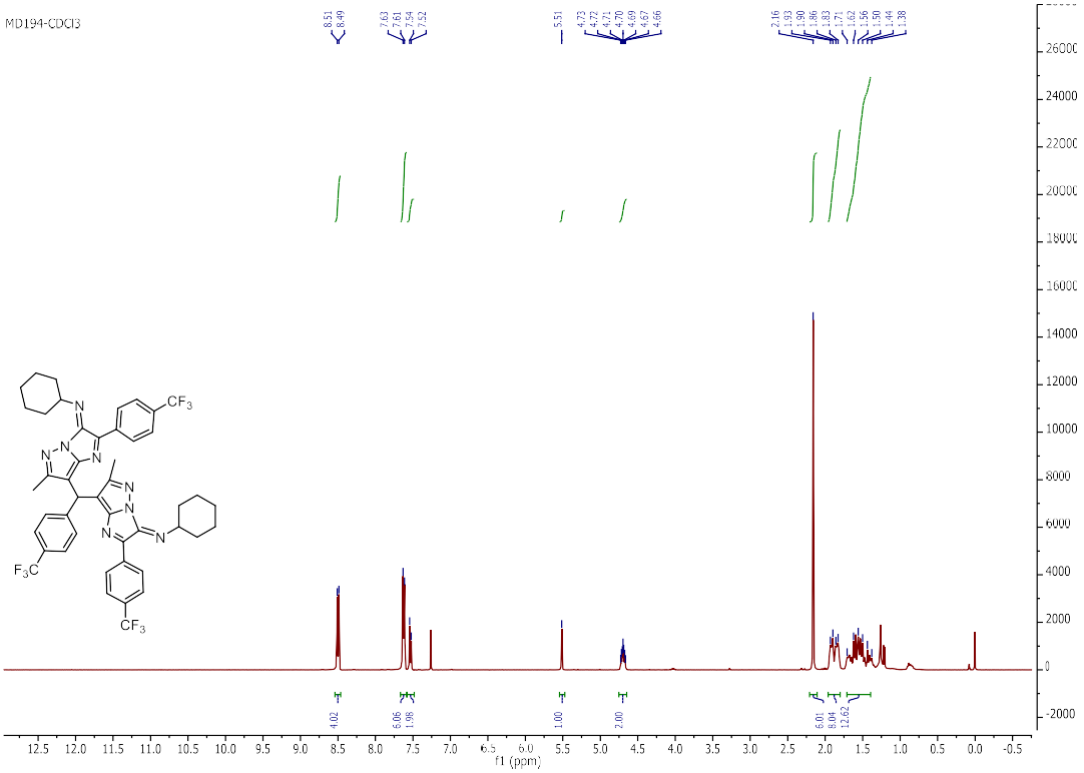


^{13}C NMR (100 MHz, $CDCl_3$)

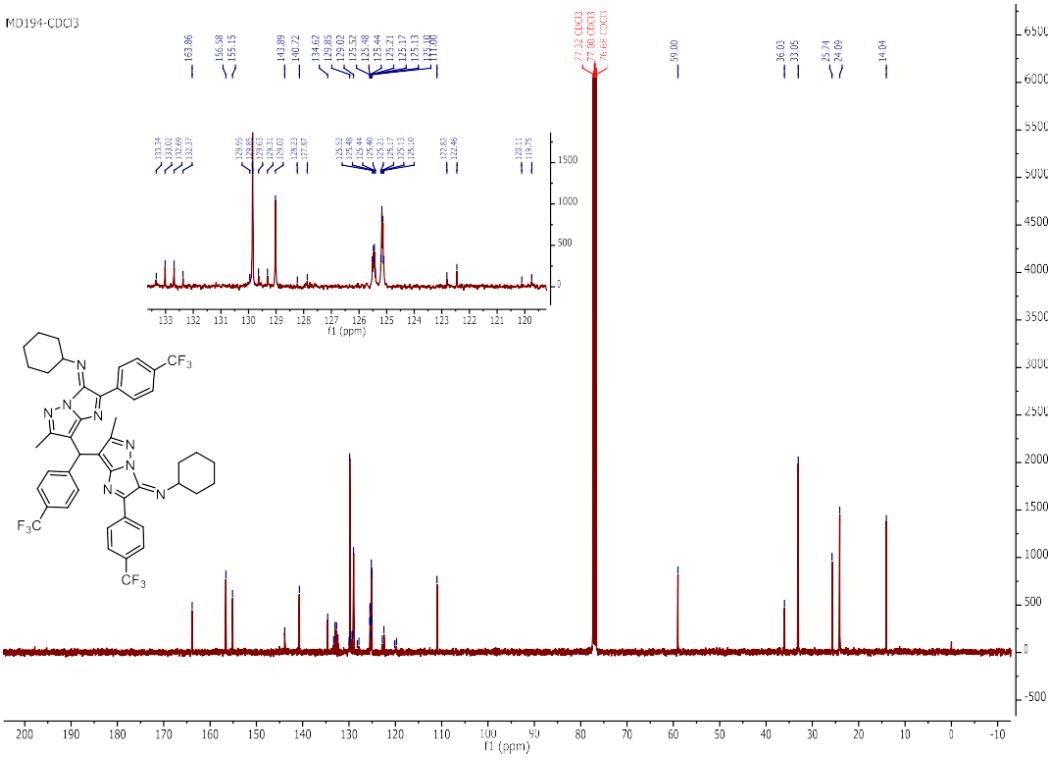


7,7'-((4-(Trifluoromethyl)phenyl)methylene)bis(*N*-cyclohexyl-6-methyl-2-(4-(trifluoromethyl)phenyl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8t**):

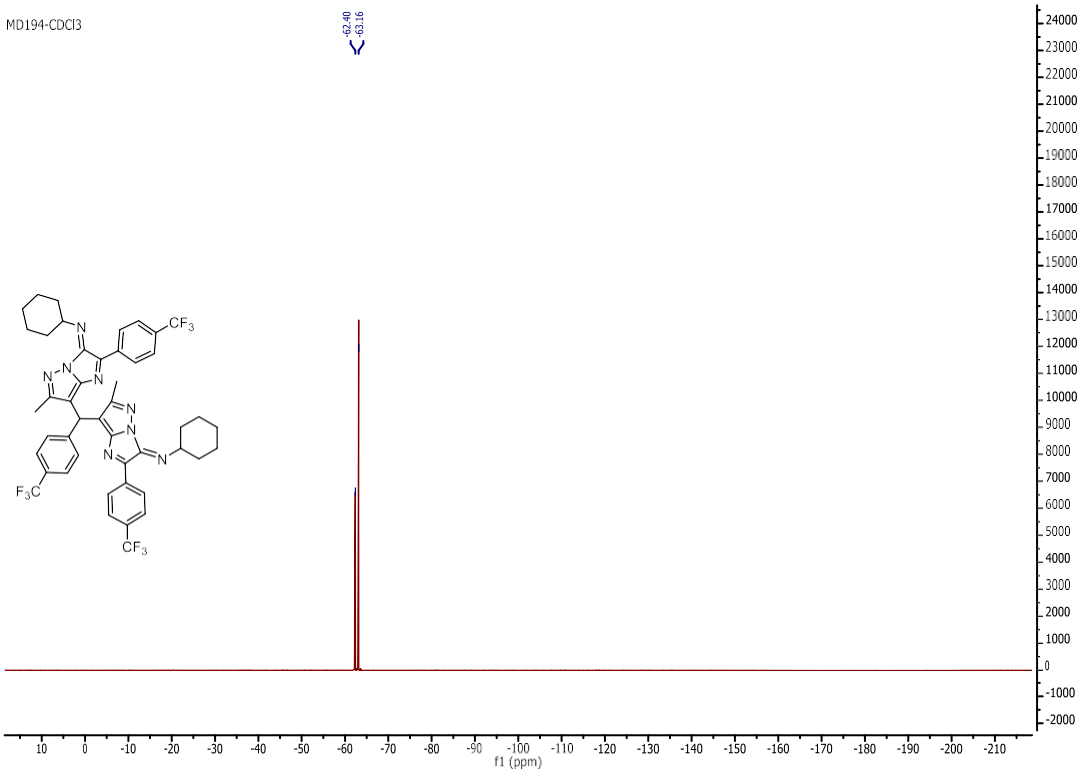
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)

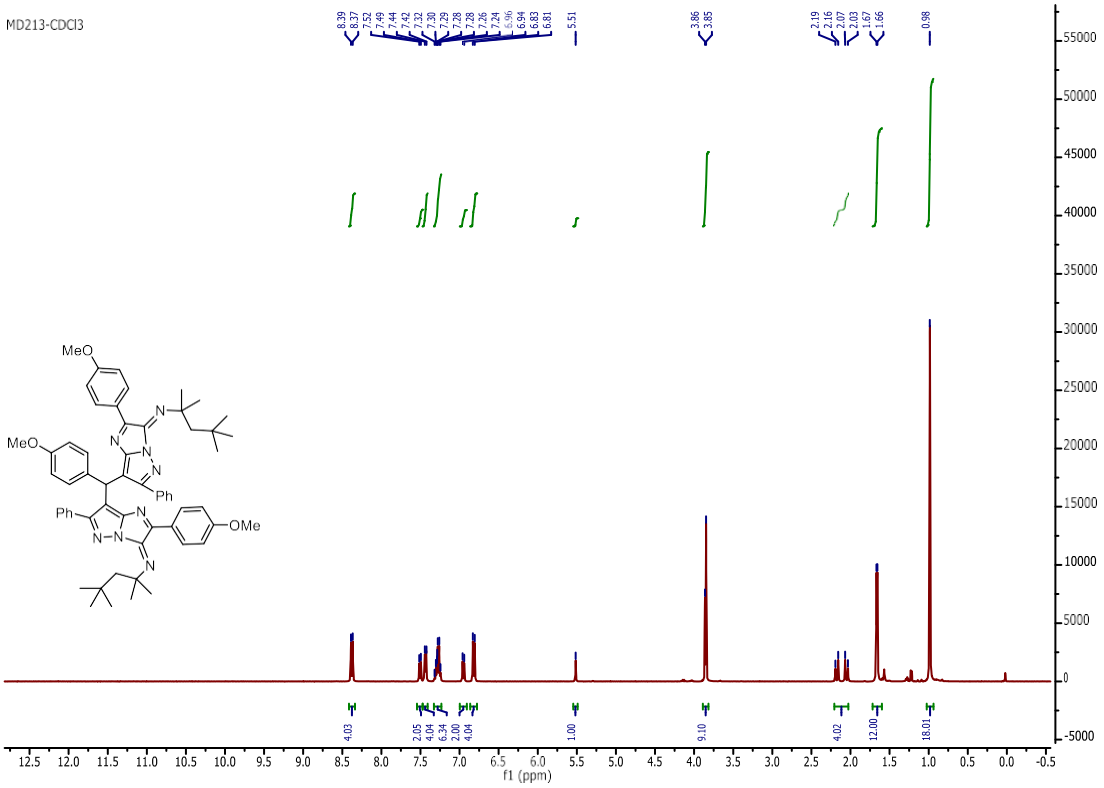


^{19}F NMR (376 MHz, $CDCl_3$)

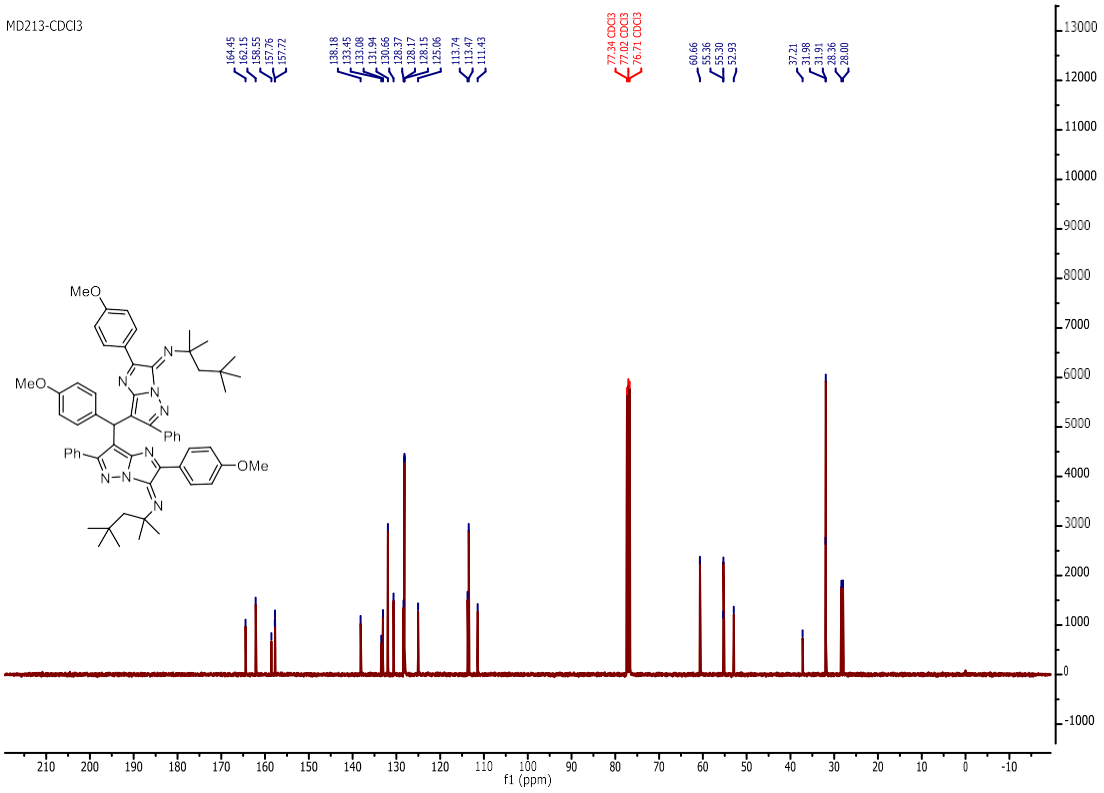


7,7'-((4-Methoxyphenyl)methylene)bis(2-(4-methoxyphenyl)-6-phenyl-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8u**):

^{1}H NMR (400 MHz, $CDCl_3$)

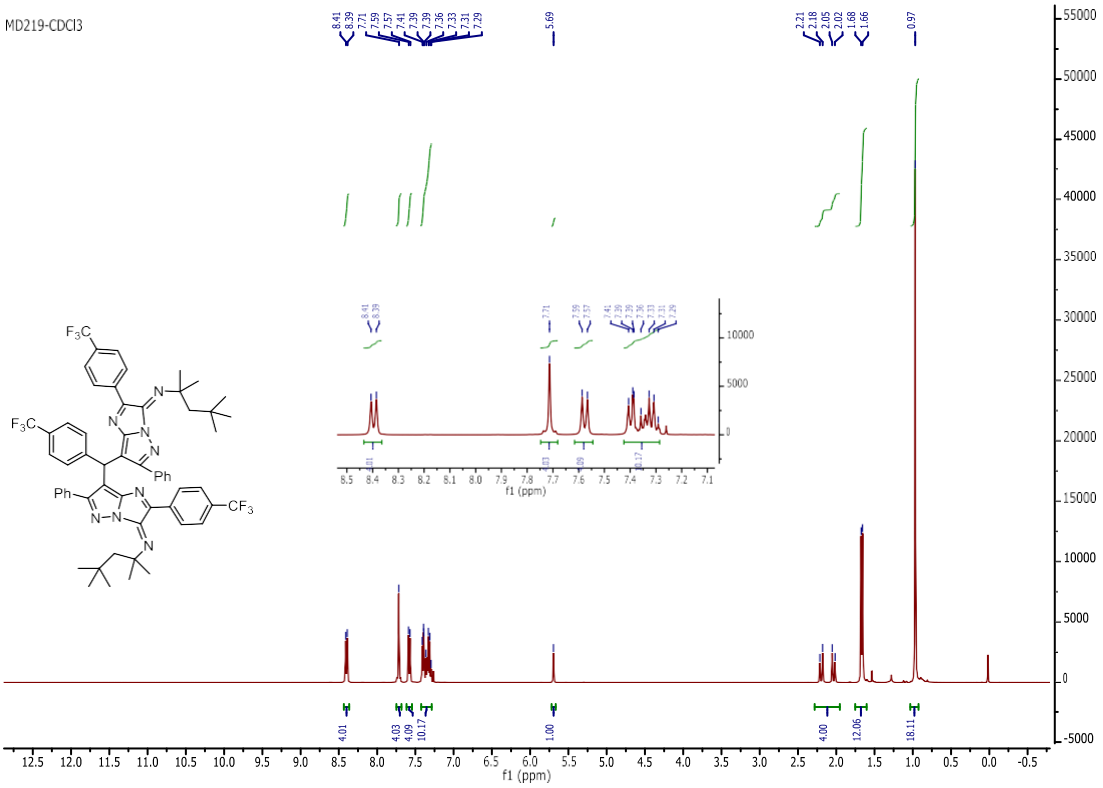


^{13}C NMR (100 MHz, $CDCl_3$)

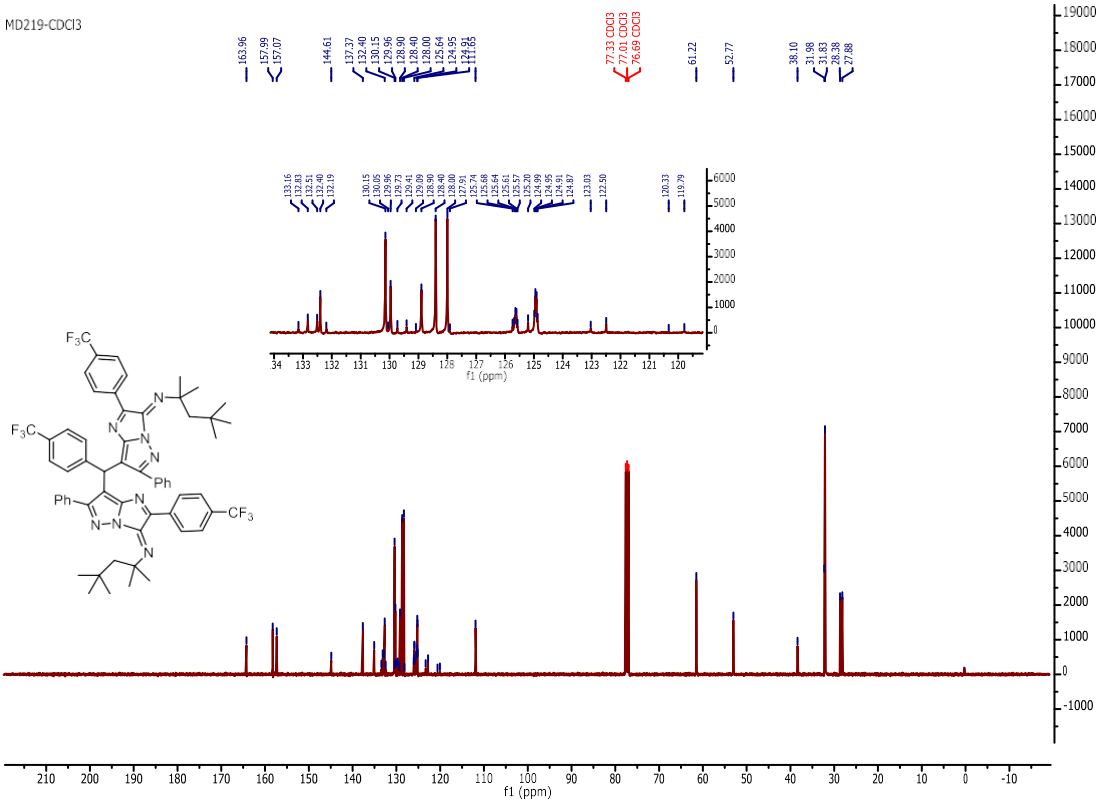


7,7'-((4-(Trifluoromethyl)phenyl)methylene)bis(6-phenyl-2-(4-(trifluoromethyl)phenyl)-*N*-(2,4,4-trimethylpentan-2-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**8v**):

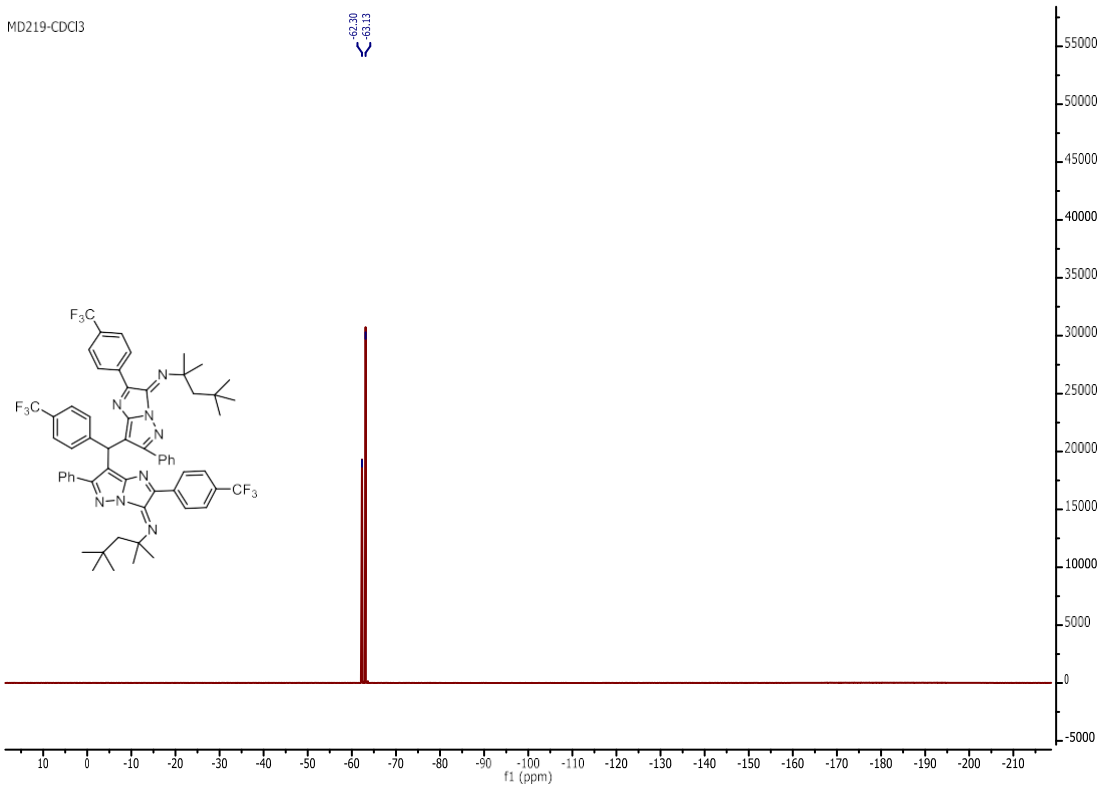
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)

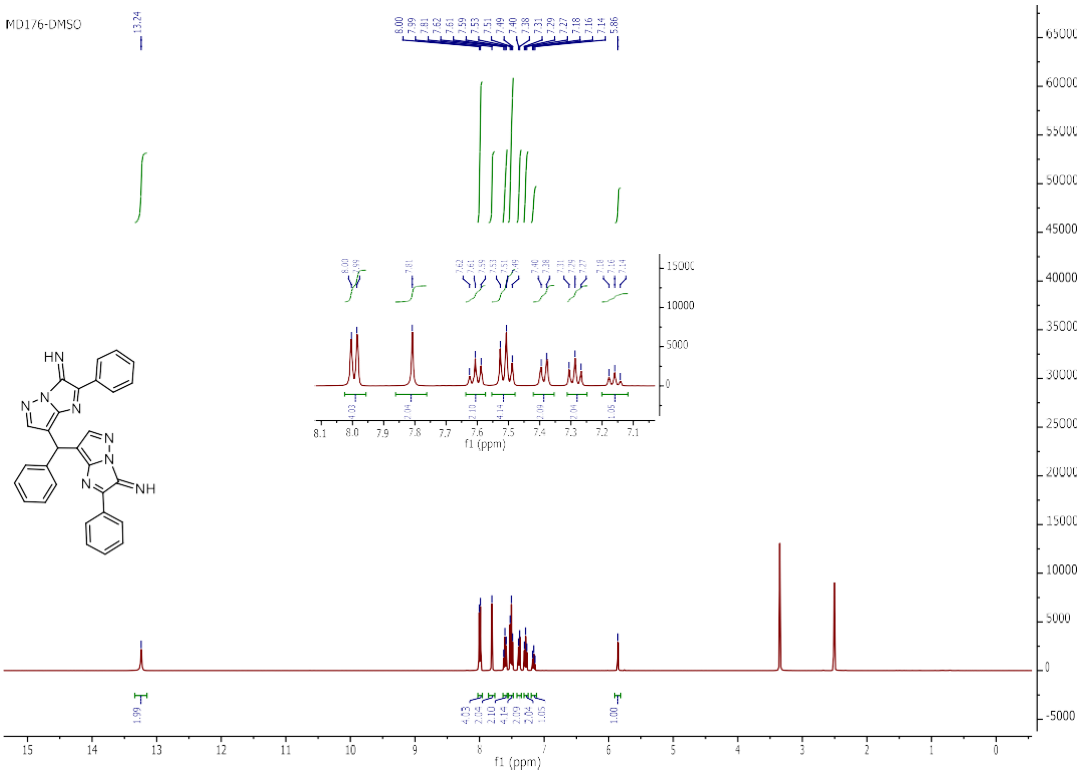


^{19}F NMR (376 MHz, $CDCl_3$)

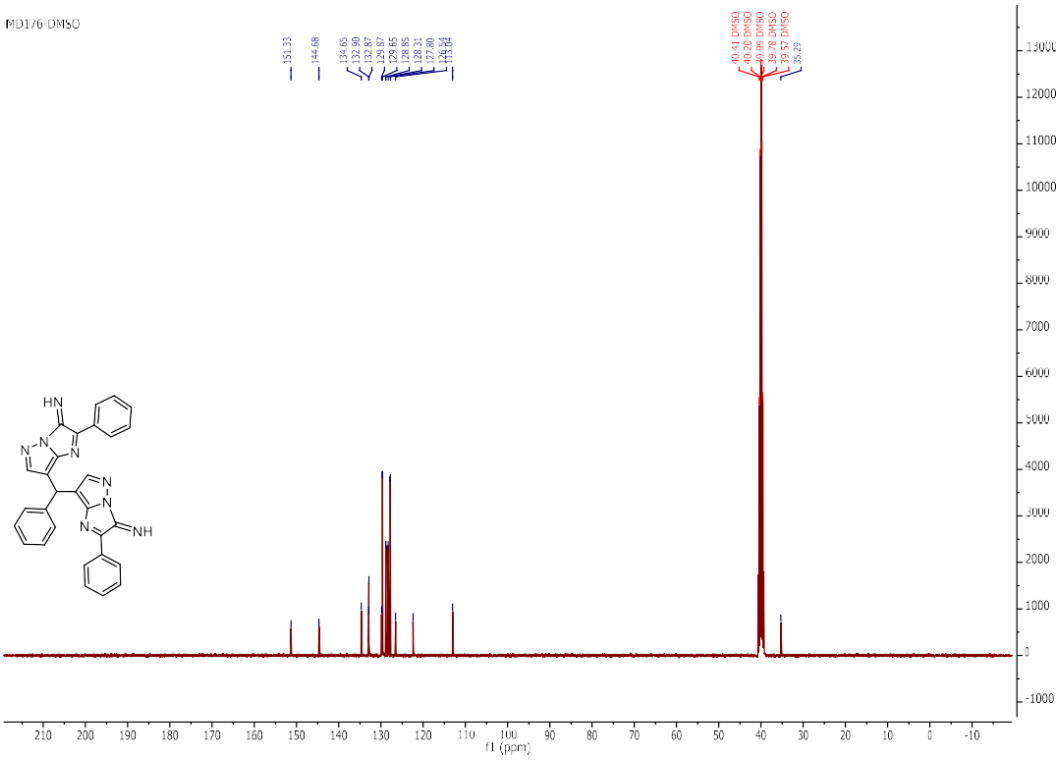


7,7'-(Phenylmethylene)bis(2-phenyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9a**):

^{1}H NMR (400 MHz,DMSO-d_6)

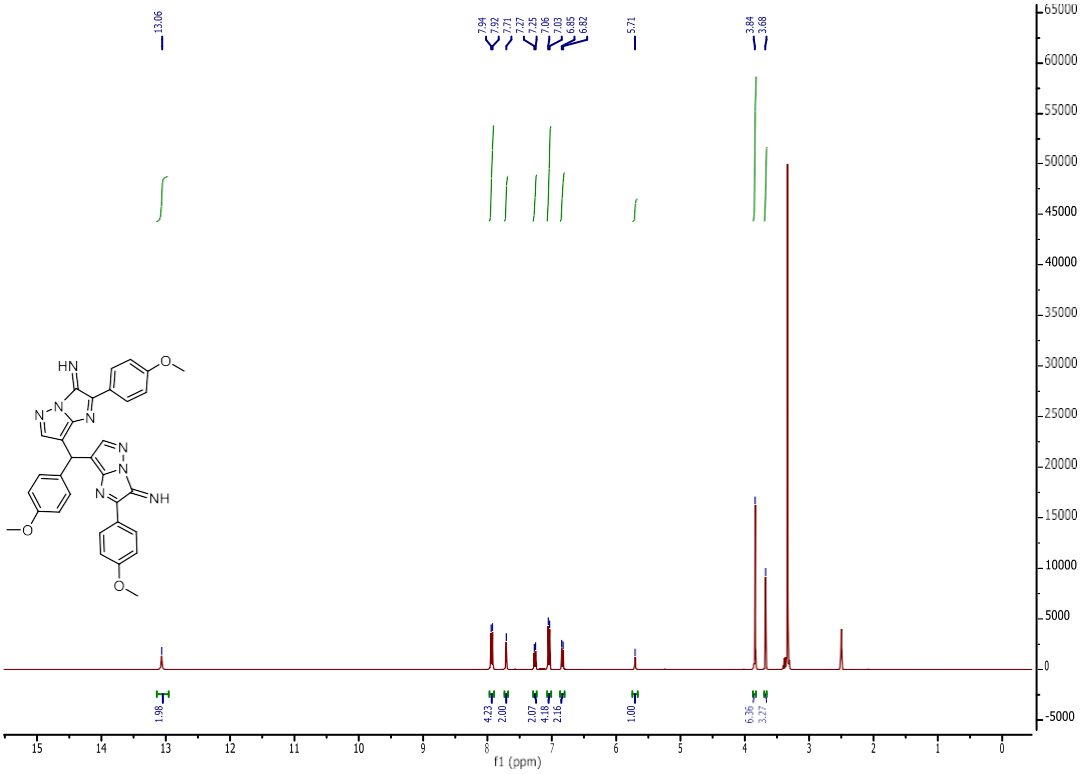


^{13}C NMR (100 MHz, DMSO-d_6)

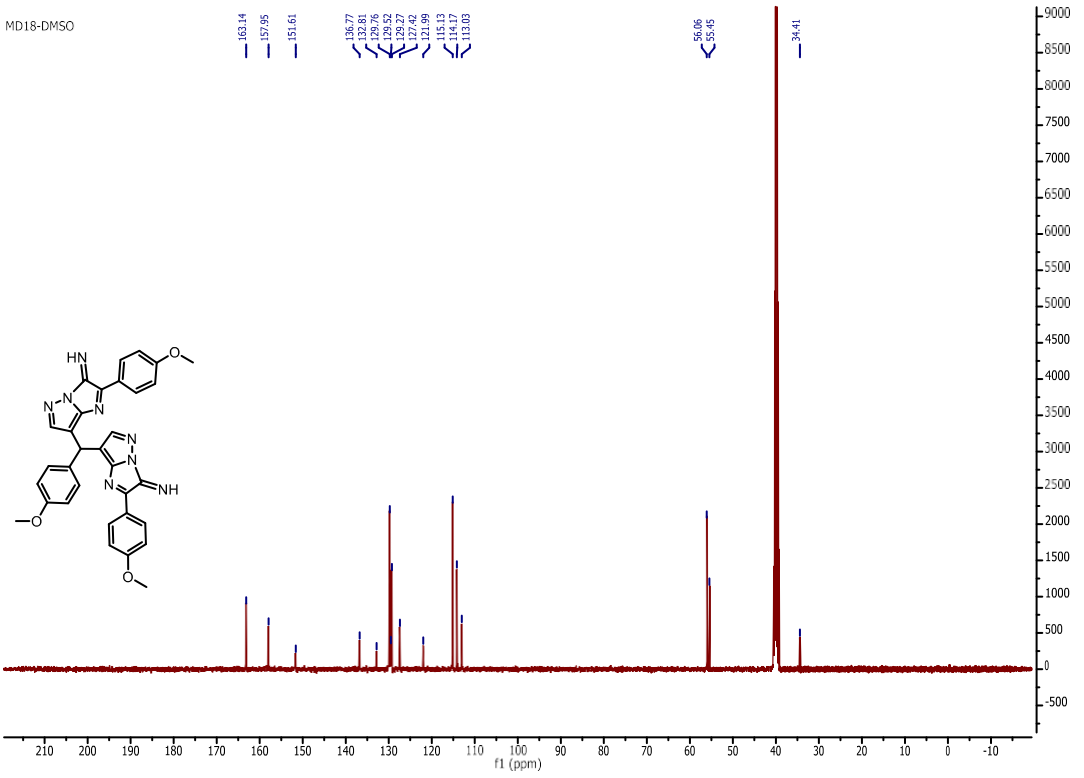


7,7'-((4-Methoxyphenyl)methylene)bis(2-(4-methoxyphenyl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9b**):

^{1}H NMR (400 MHz,DMSO-d_6)

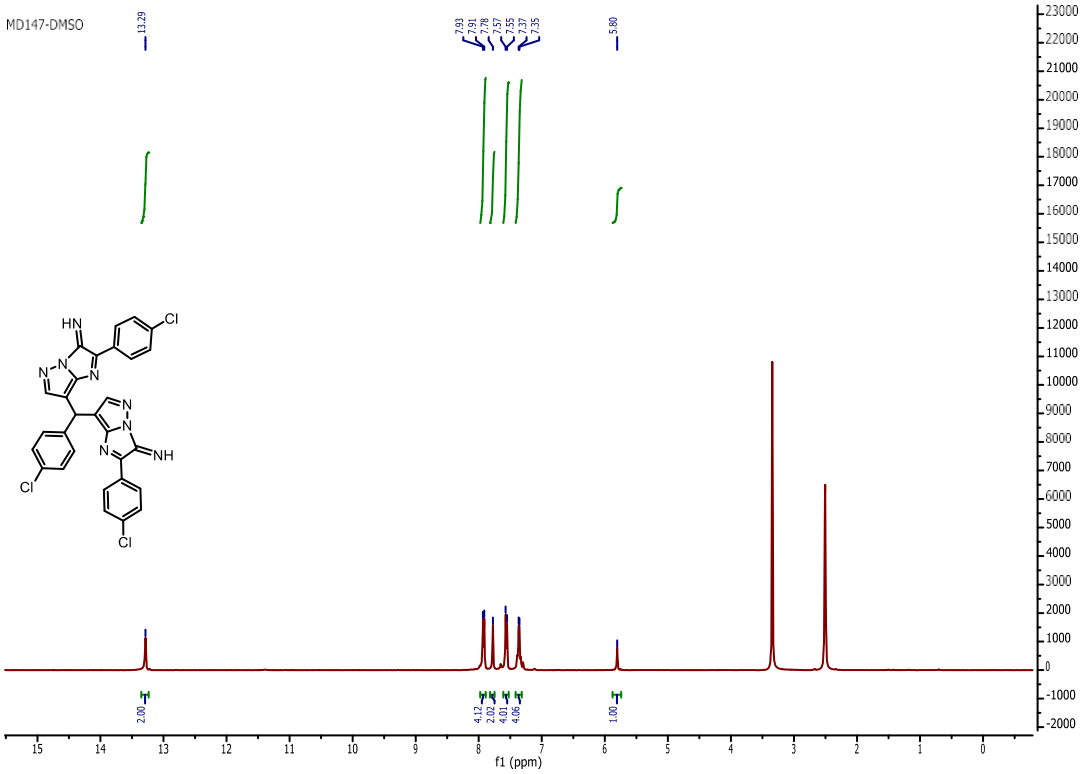


^{13}C NMR (100 MHz, DMSO-d_6)

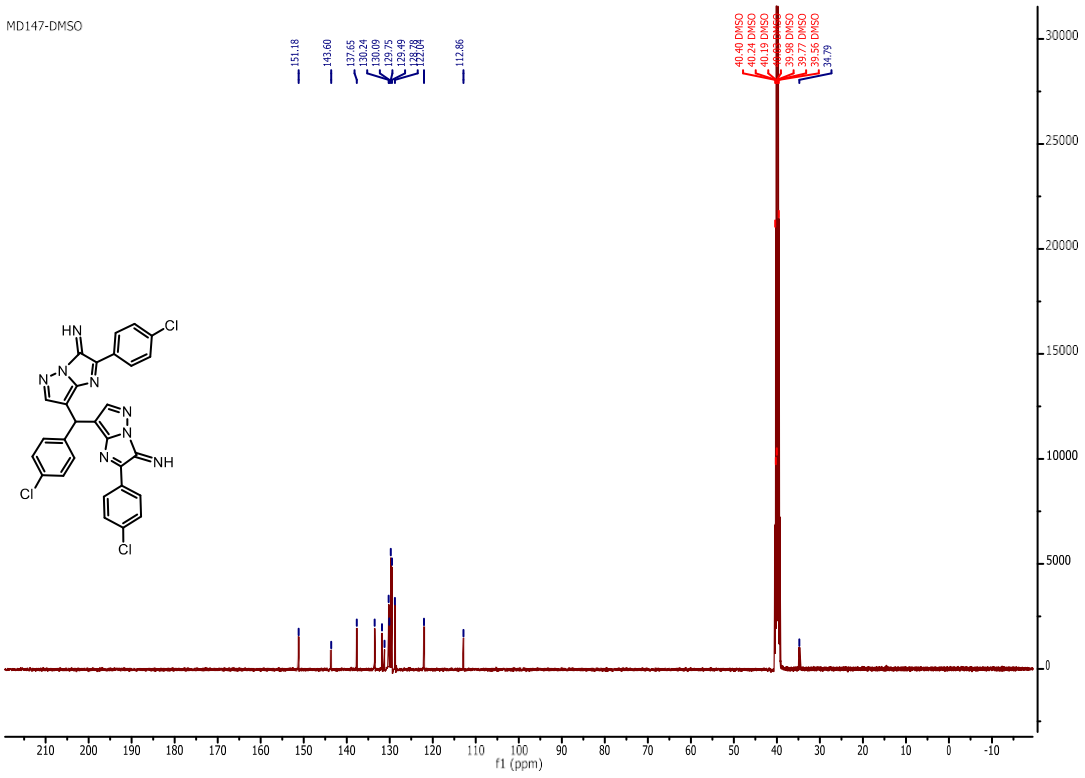


7,7'-((4-Chlorophenyl)methylene)bis(2-(4-chlorophenyl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9c**):

^{1}H NMR (400 MHz,DMSO-d_6)

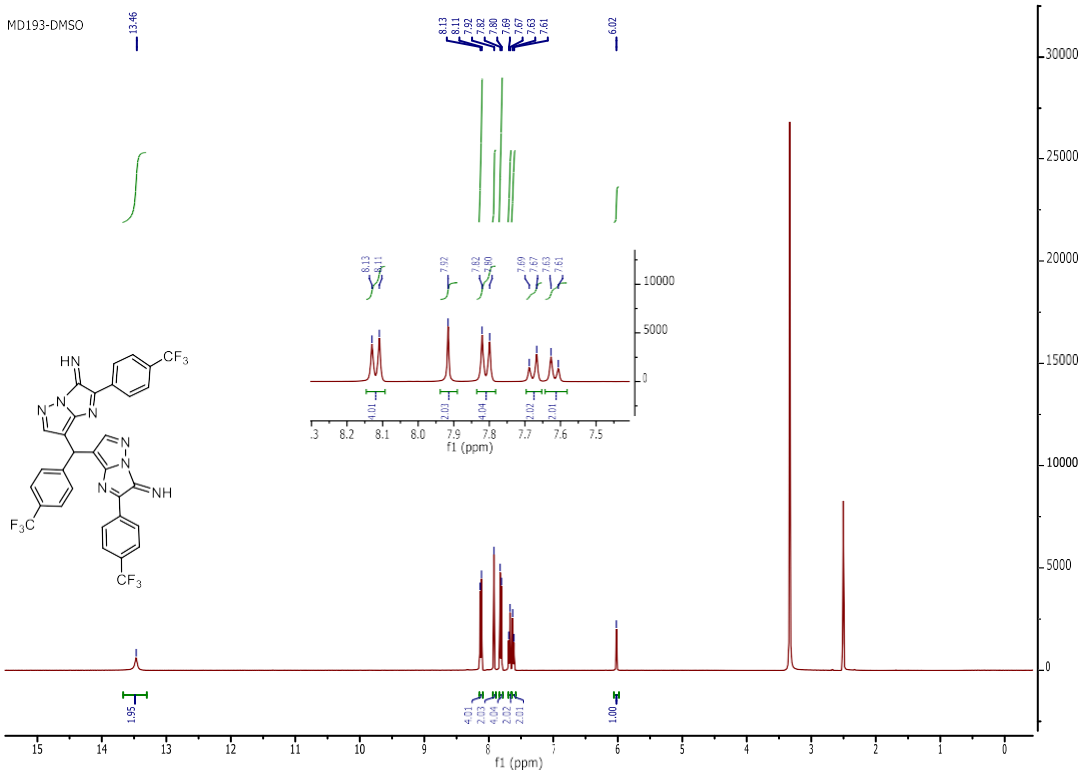


^{13}C NMR (100 MHz, DMSO-d_6)

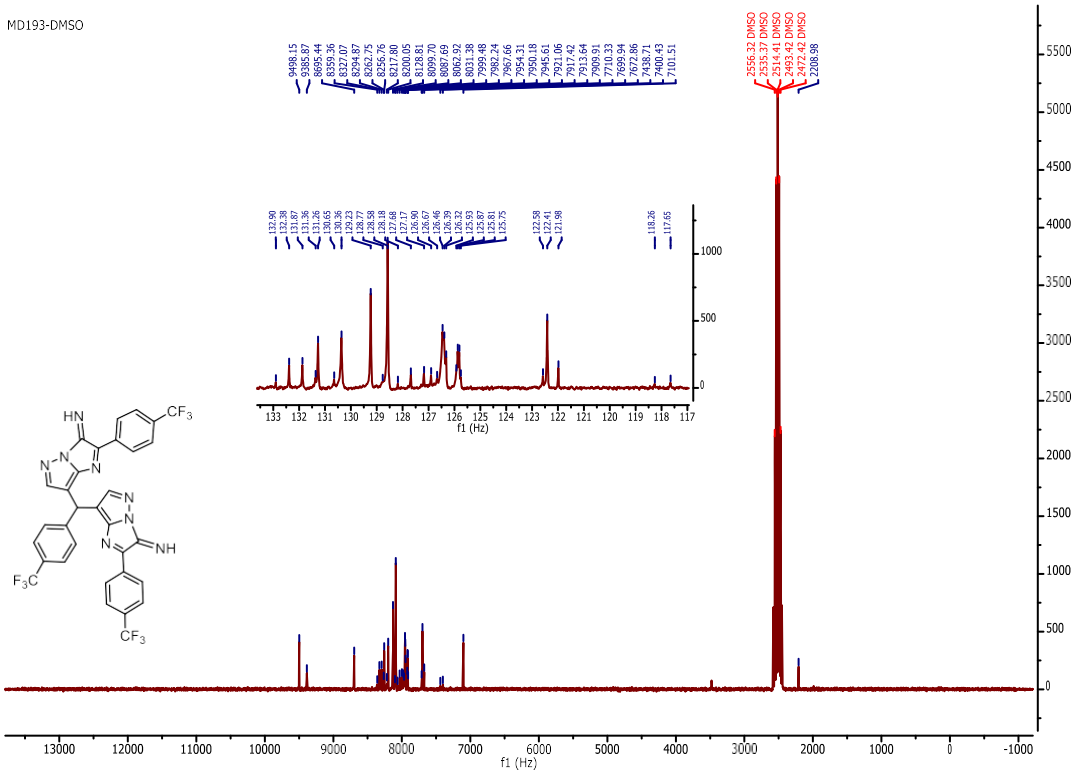


7,7'-((4-(Trifluoromethyl)phenyl)methylene)bis(2-(4-(trifluoromethyl)phenyl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9d**):

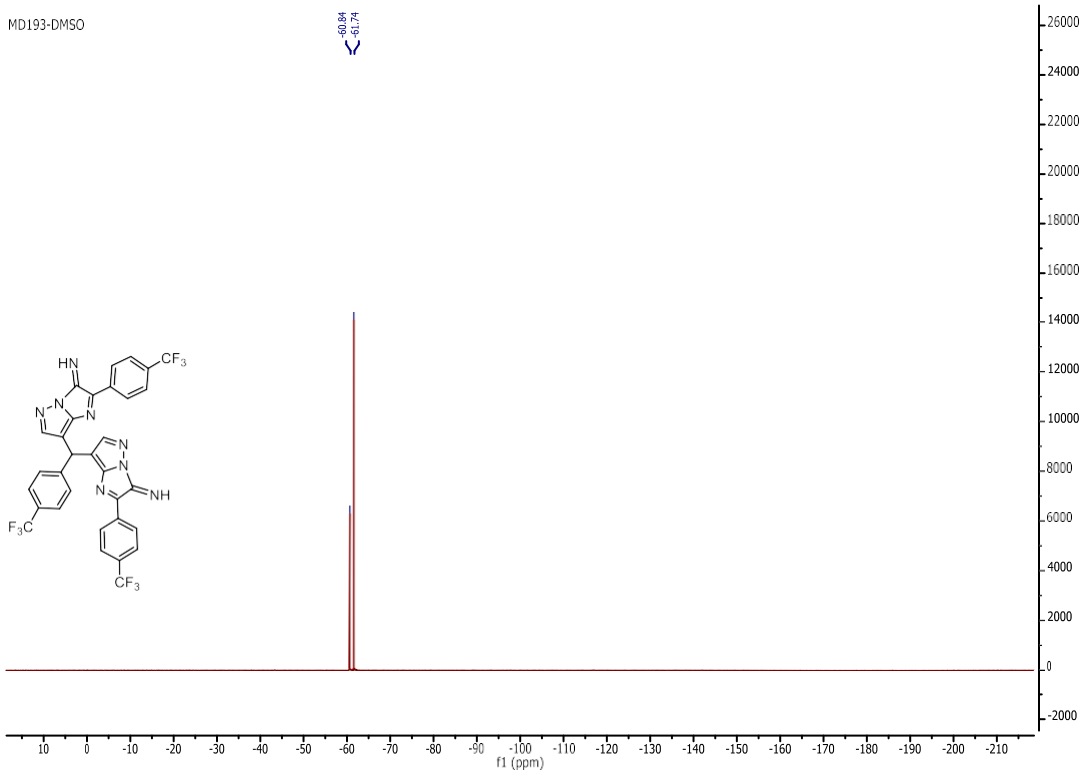
^{1}H NMR (400 MHz,DMSO-d_6)



^{13}C NMR (62.9 MHz, DMSO-d_6)

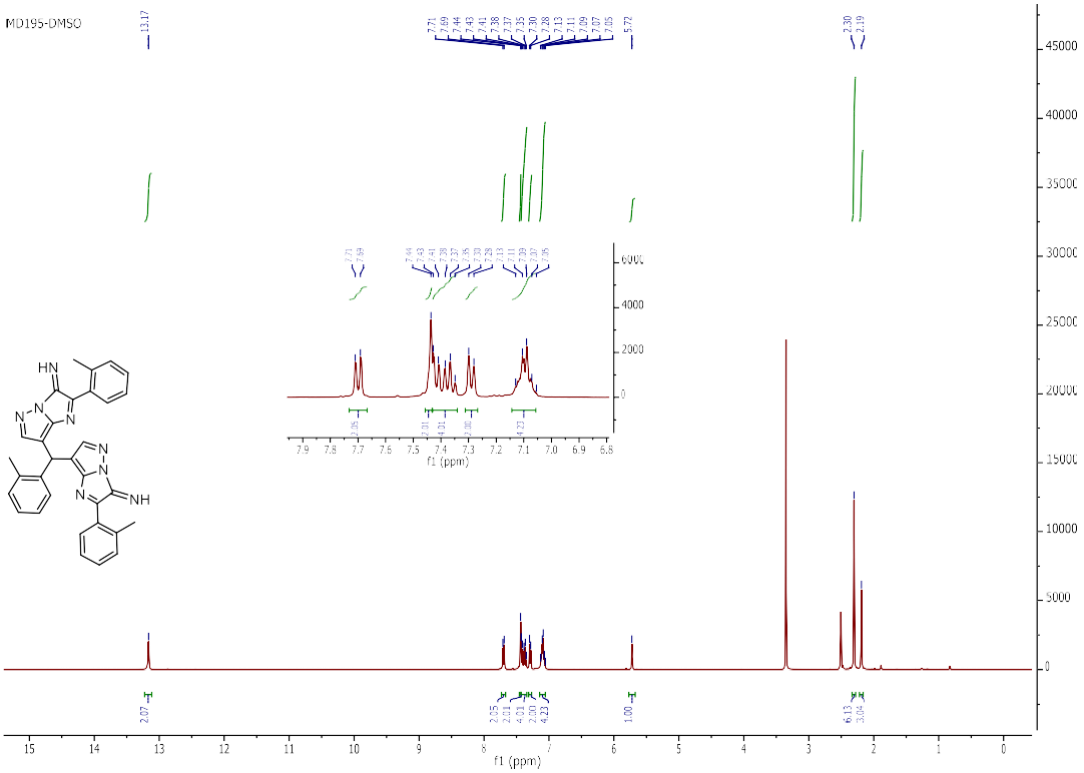


^{19}F NMR (376 MHz, DMSO-d_6)

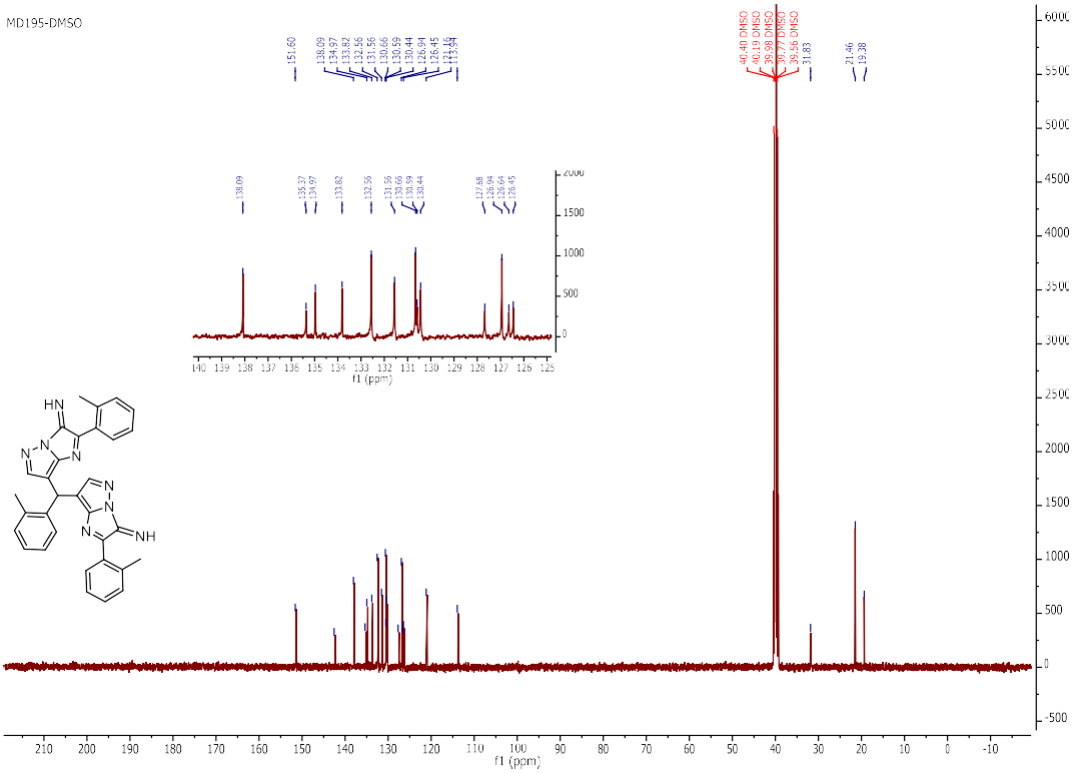


7,7'-(*o*-Tolylmethylene)bis(2-(*o*-tolyl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9f**):

^{1}H NMR (400 MHz,DMSO-d_6)

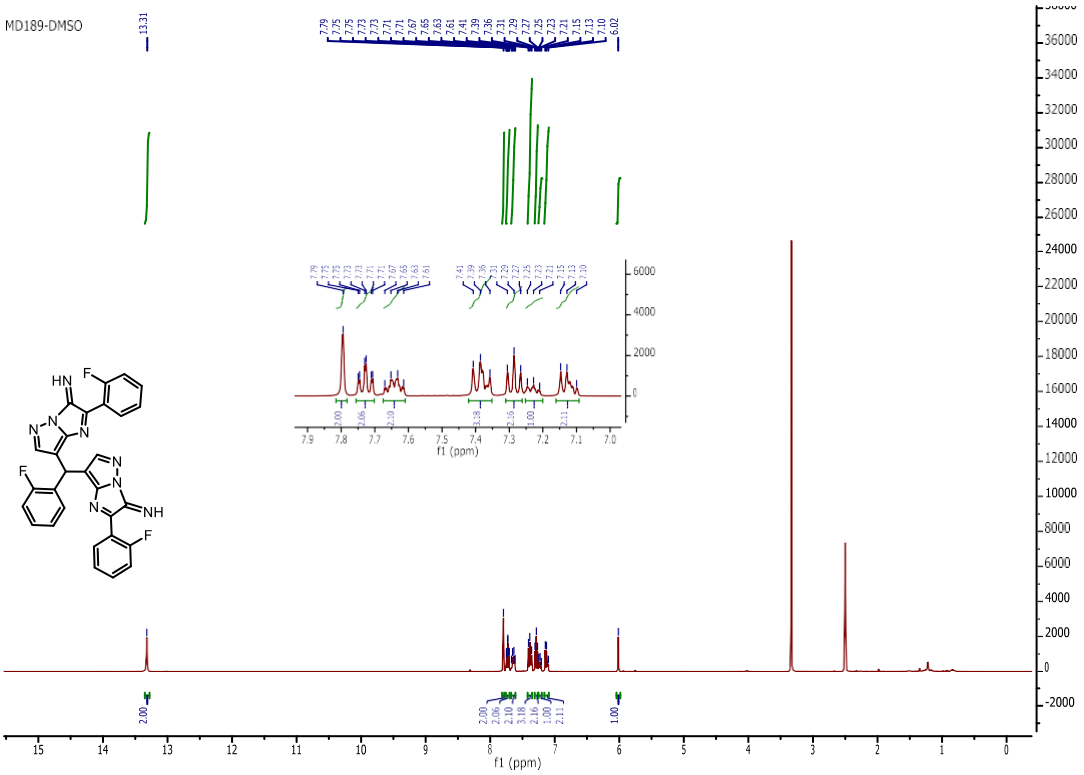


^{13}C NMR (100 MHz, DMSO-d_6)

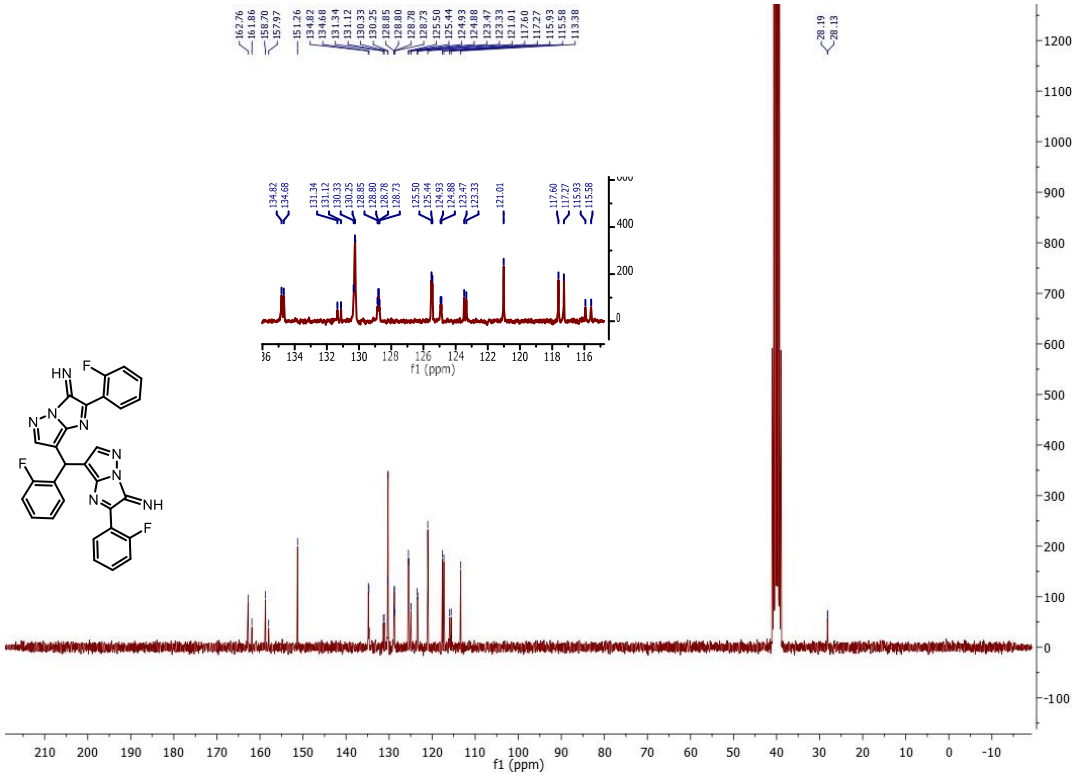


7,7'-((2-Fluorophenyl)methylene)bis(2-(2-fluorophenyl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9g**):

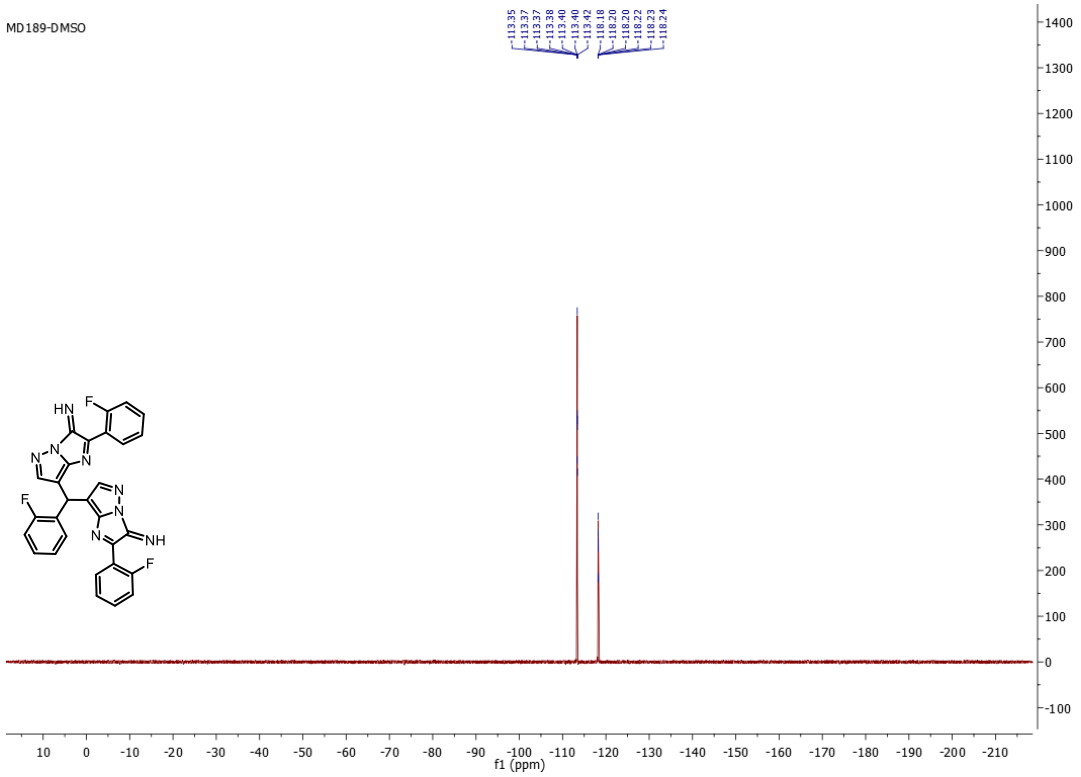
^{1}H NMR (400 MHz,DMSO-d_6)



^{13}C NMR (62.9 MHz, DMSO-d_6)

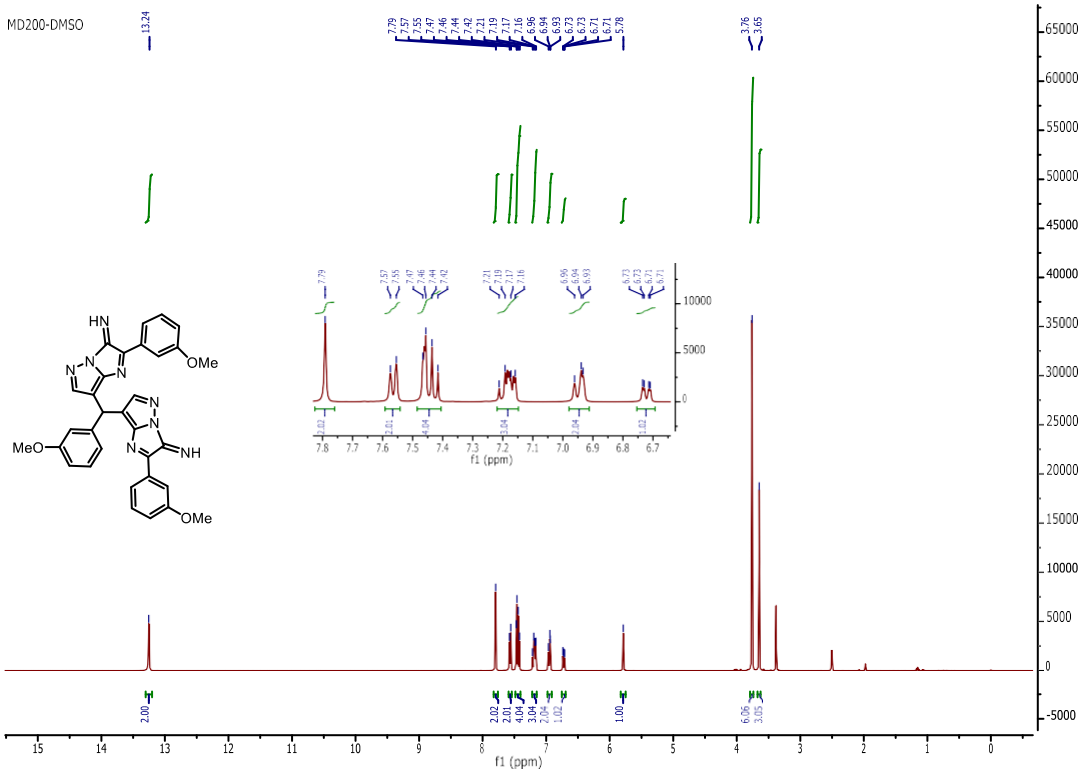


^{19}F NMR (376 MHz, DMSO-d_6)

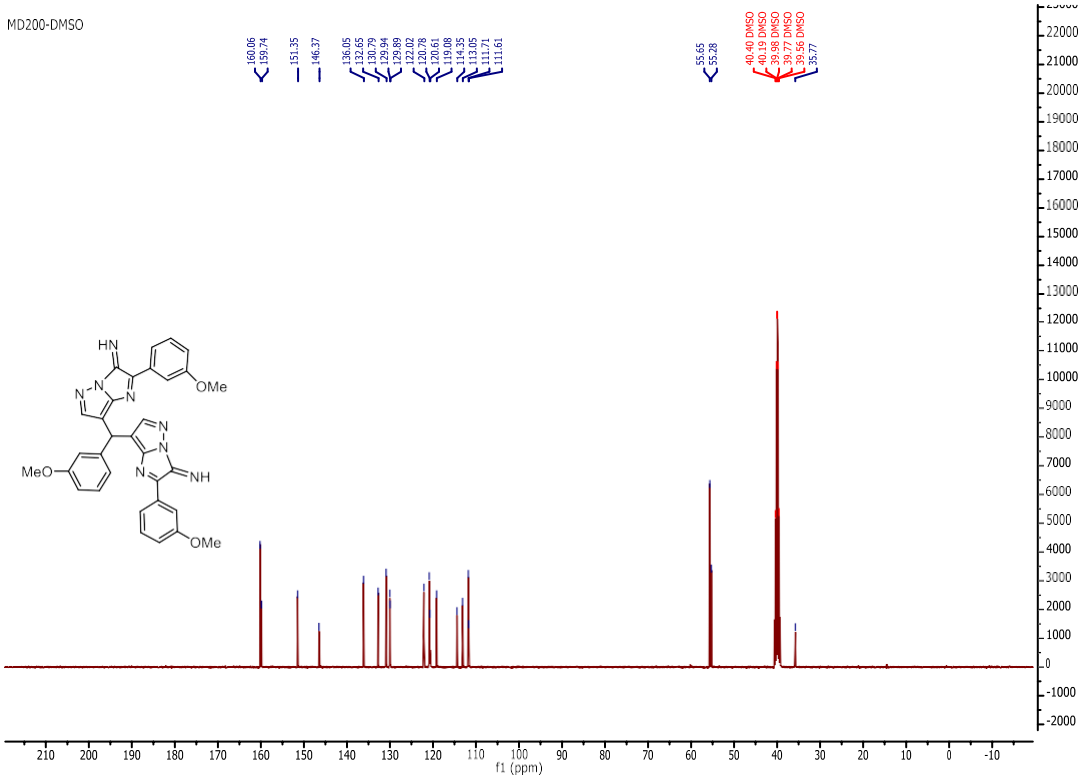


7,7'-((3-Methoxyphenyl)methylene)bis(2-(3-methoxyphenyl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9h**):

^{1}H NMR (400 MHz,DMSO-d_6)

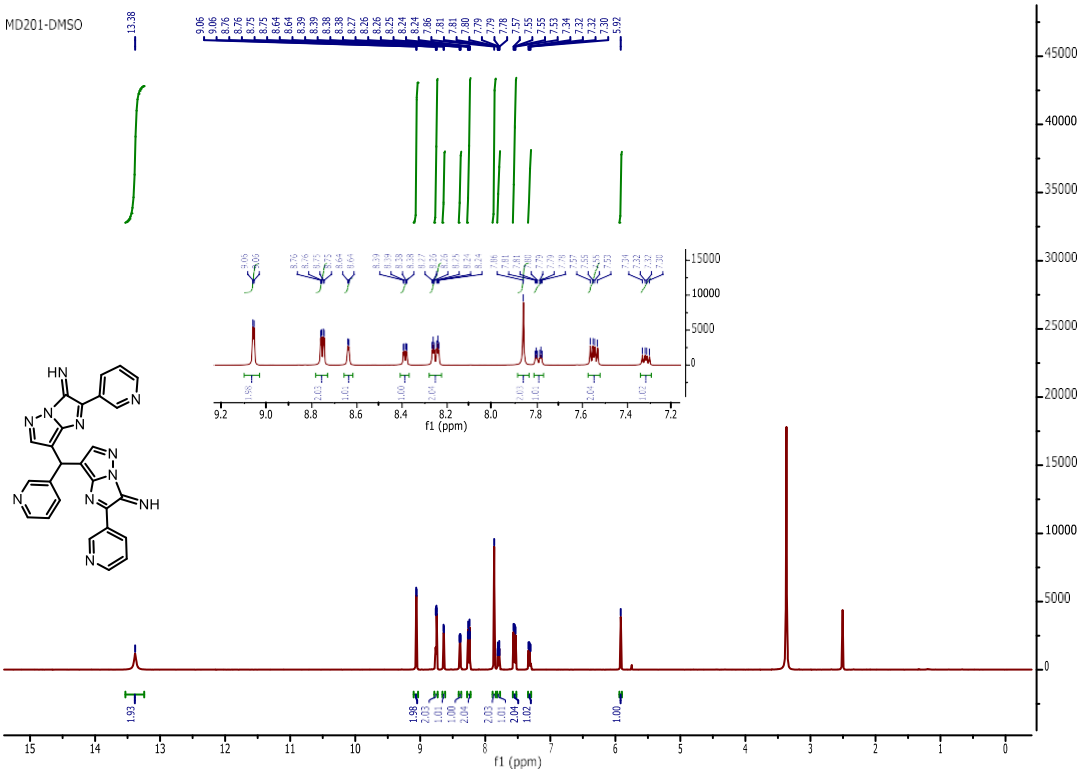


^{13}C NMR (100 MHz, DMSO-d_6)

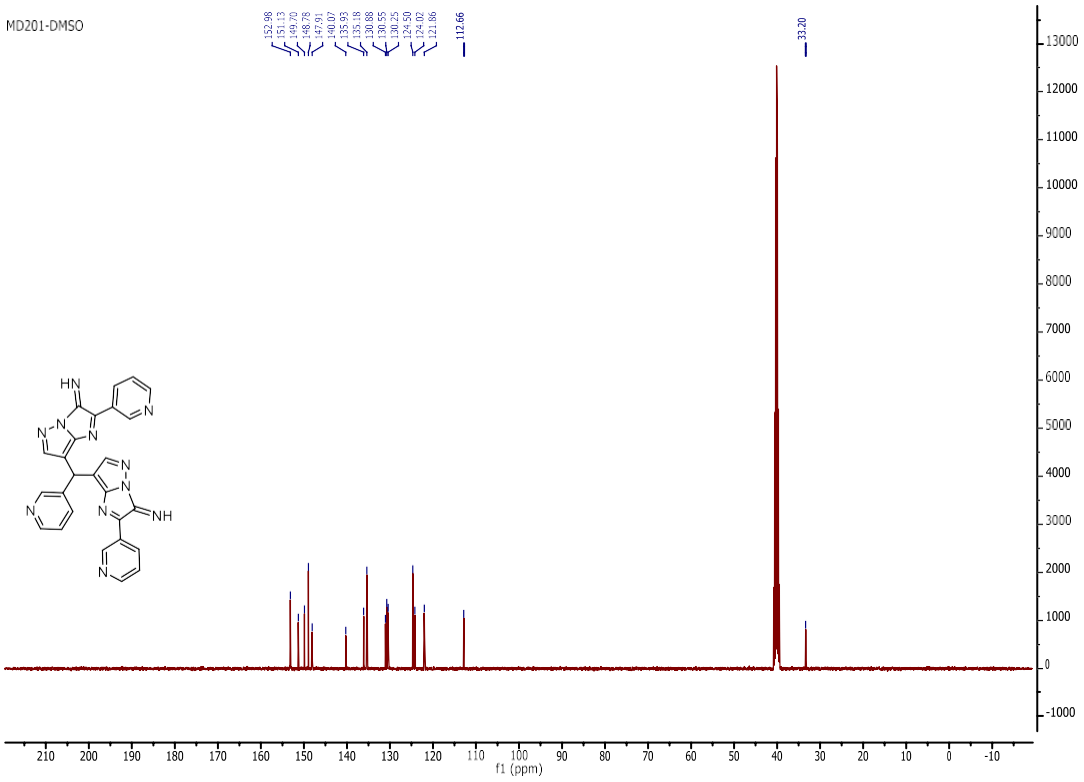


7,7'-(Pyridin-3-ylmethylene)bis(2-(pyridin-3-yl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9j**):

^{1}H NMR (400 MHz,DMSO-d_6)

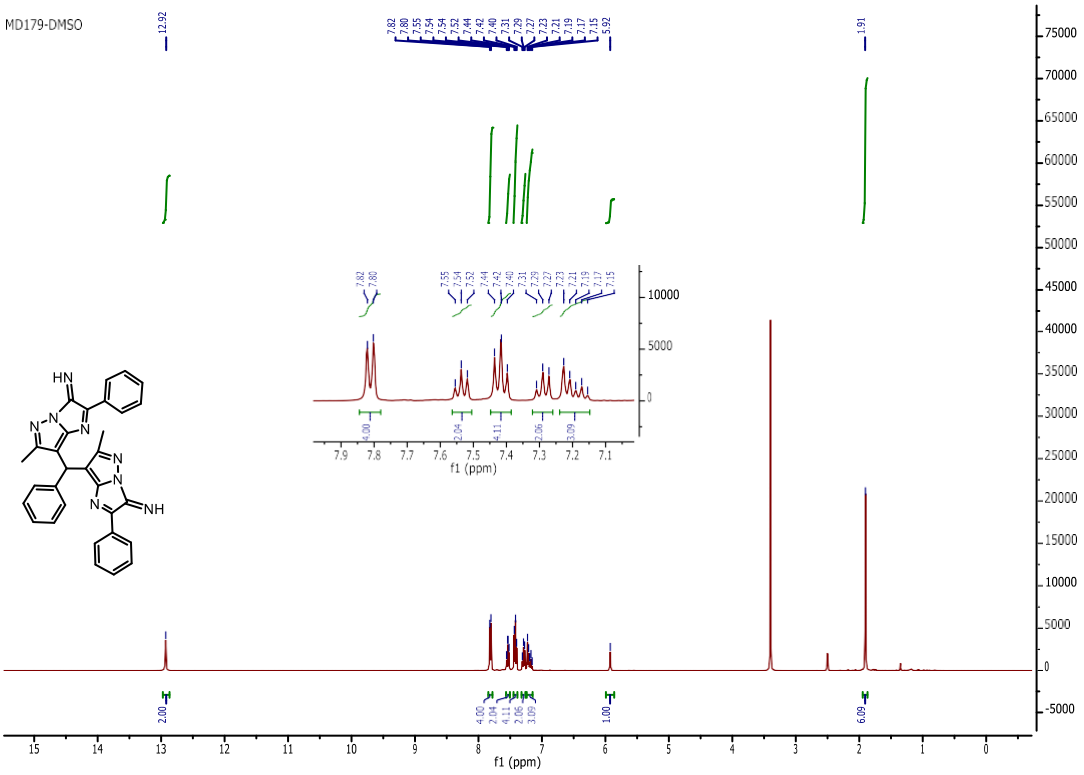


^{13}C NMR (100 MHz, DMSO-d_6)

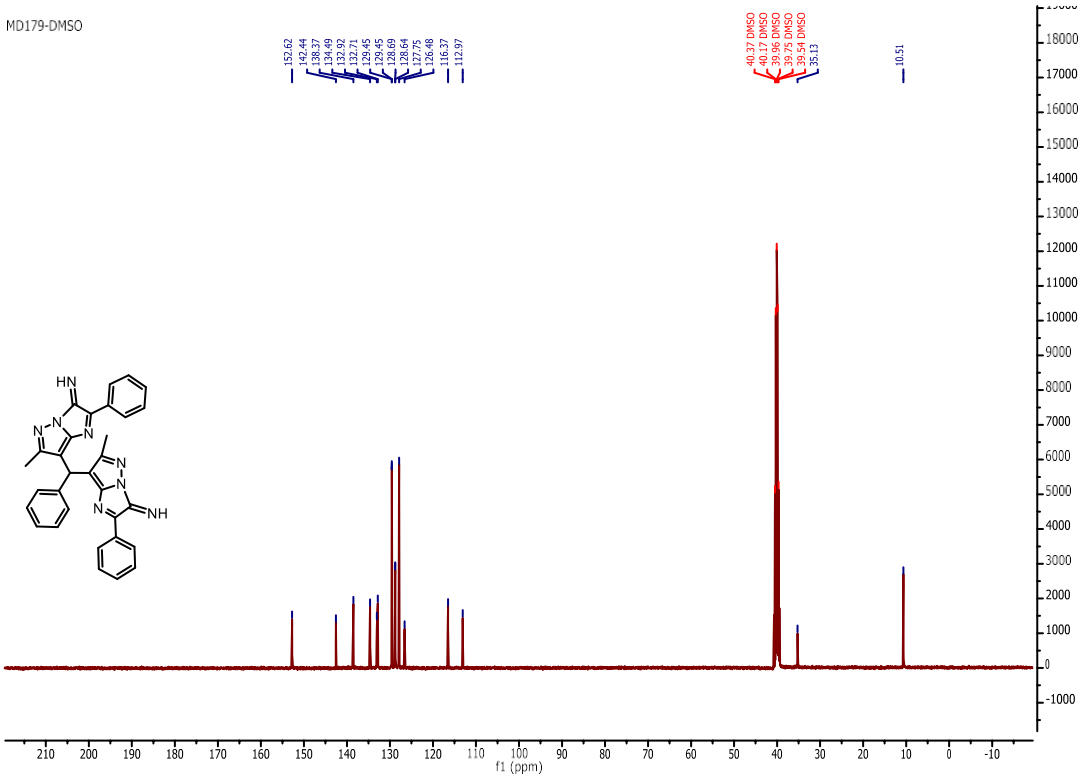


7,7'-(Phenylmethylene)bis(6-methyl-2-phenyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9n**):

^{1}H NMR (400 MHz,DMSO-d_6)

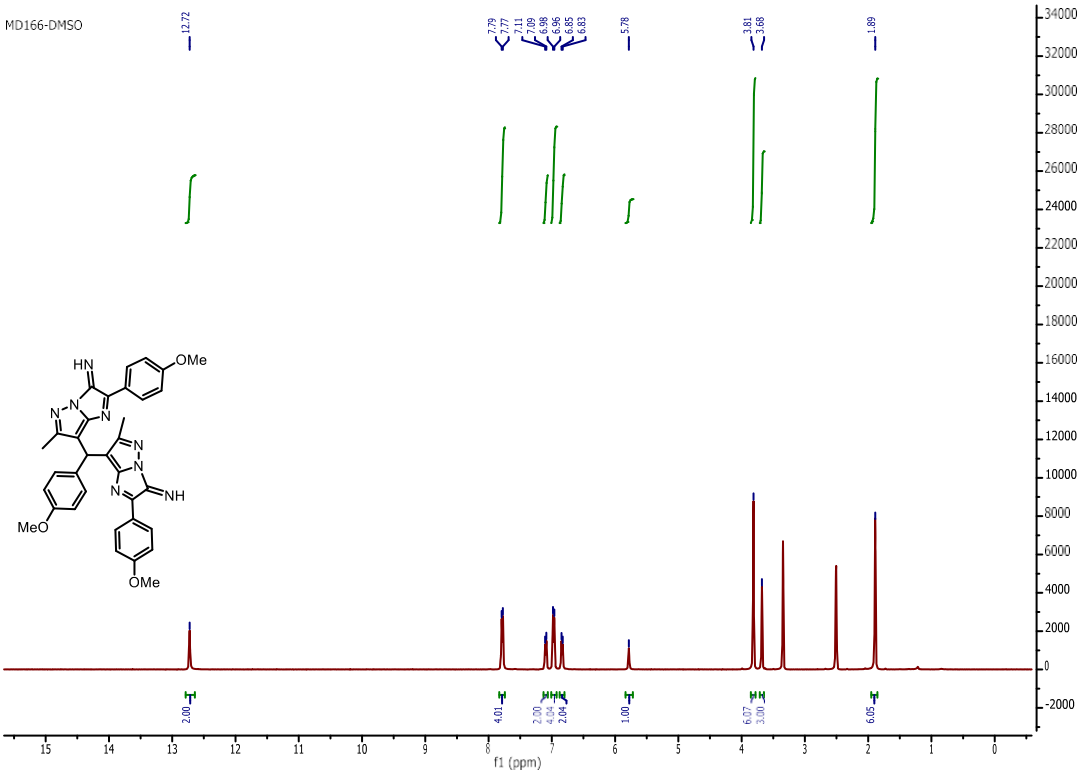


^{13}C NMR (100 MHz, DMSO-d_6)

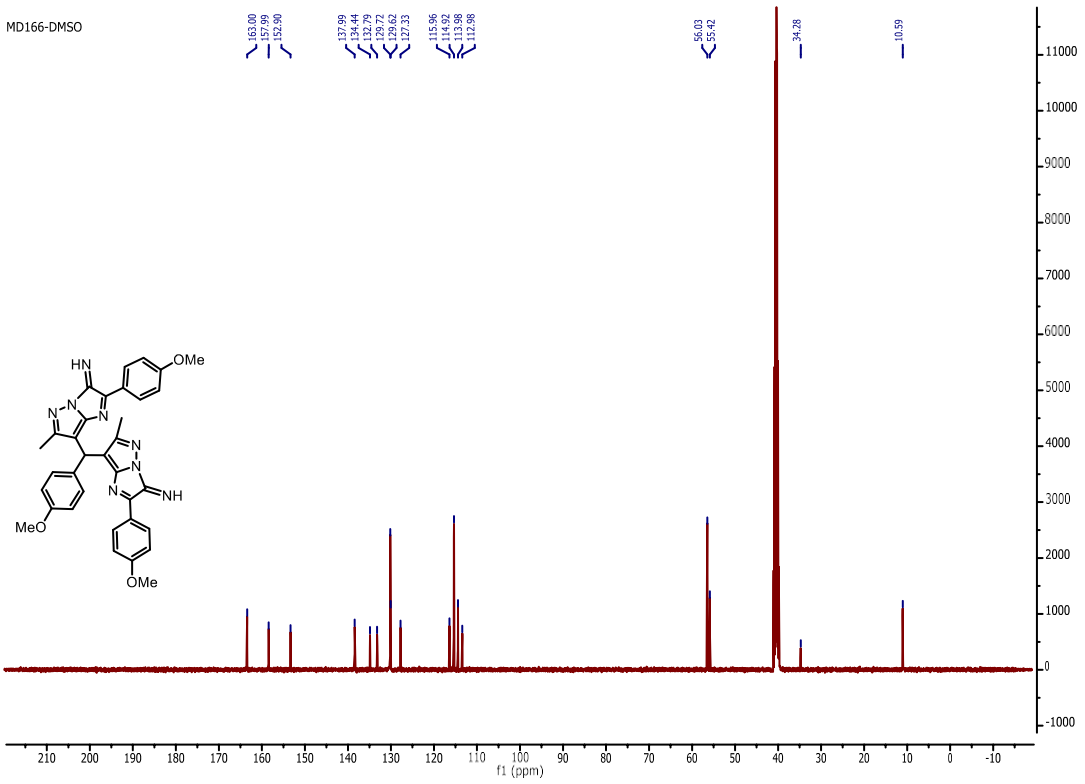


7,7'-((4-Methoxyphenyl)methylene)bis(2-(4-methoxyphenyl)-6-methyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9o**):

^{1}H NMR (400 MHz,DMSO-d_6)

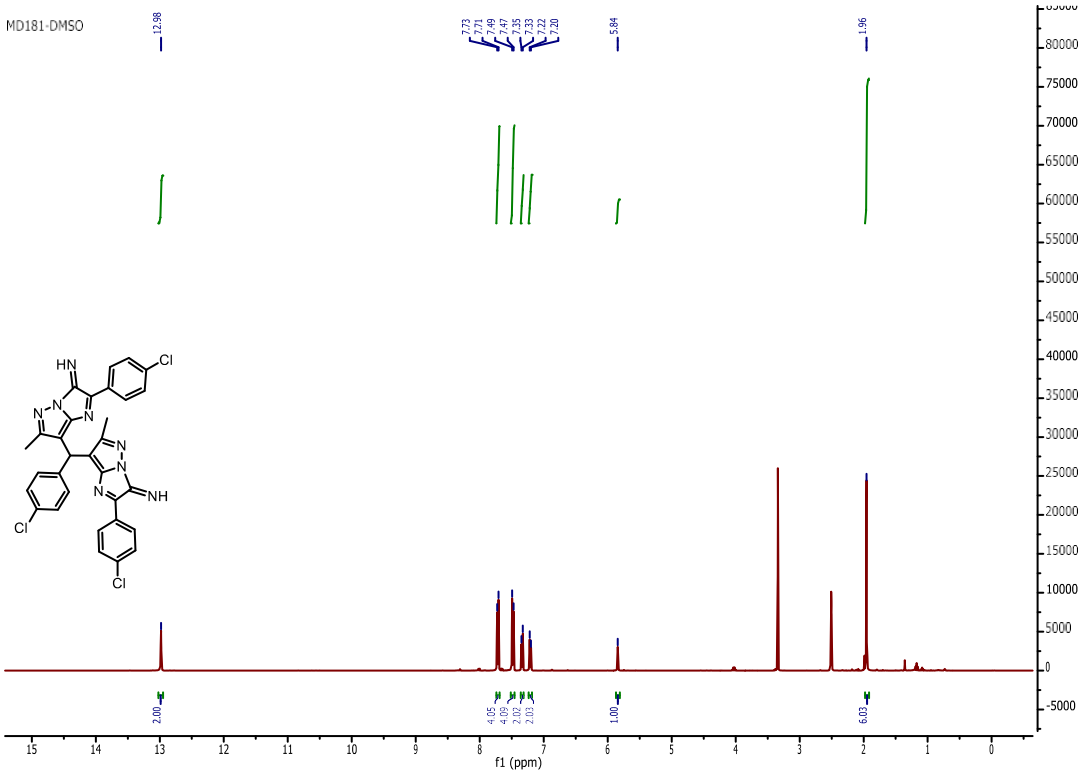


^{13}C NMR (100 MHz, DMSO-d_6)

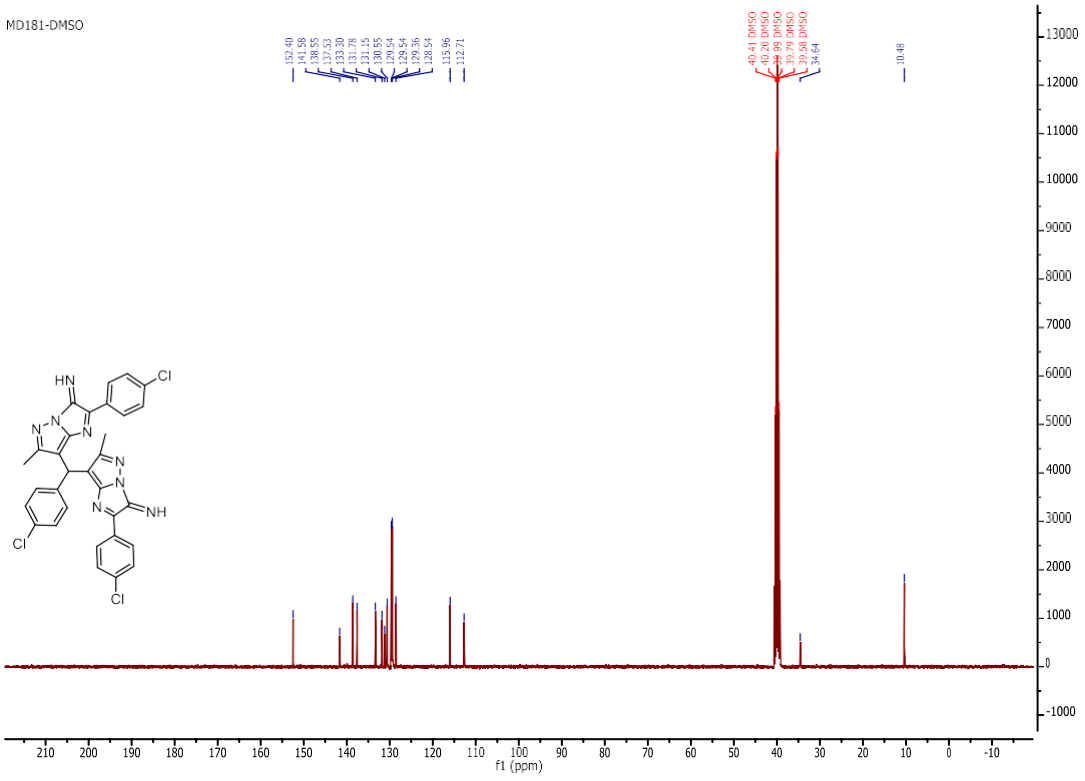


7,7'-((4-Chlorophenyl)methylene)bis(2-(4-chlorophenyl)-6-methyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9p**):

^{1}H NMR (400 MHz,DMSO-d_6)

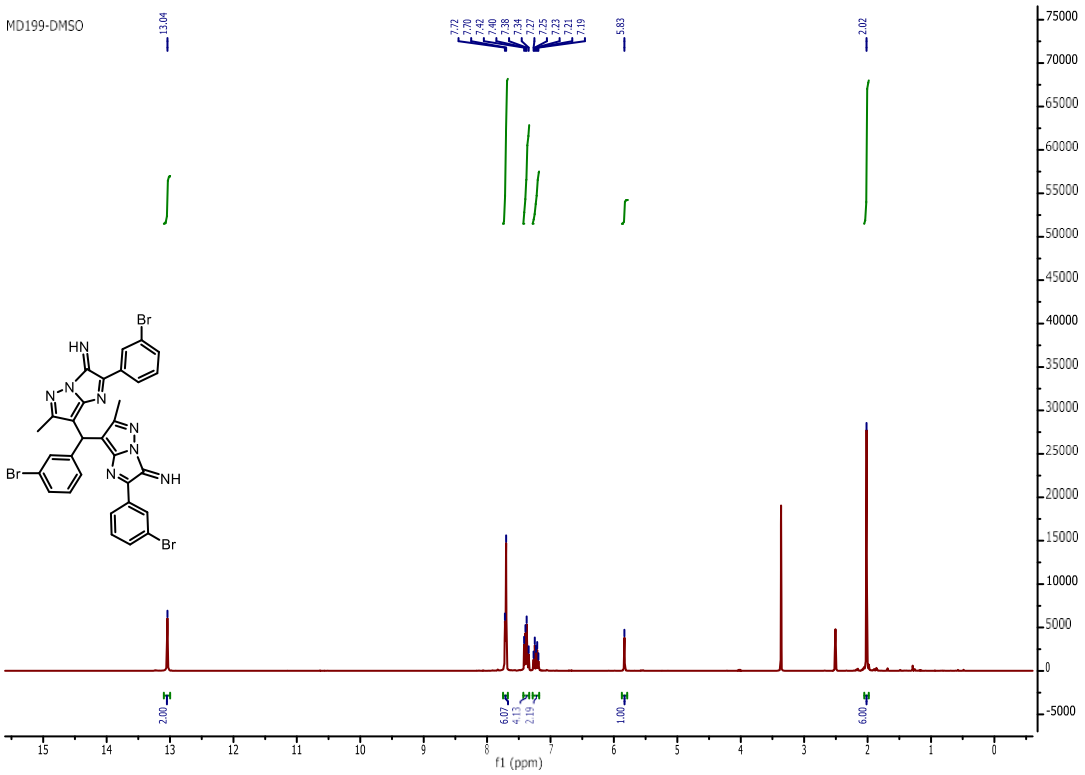


^{13}C NMR (100 MHz, DMSO-d_6)

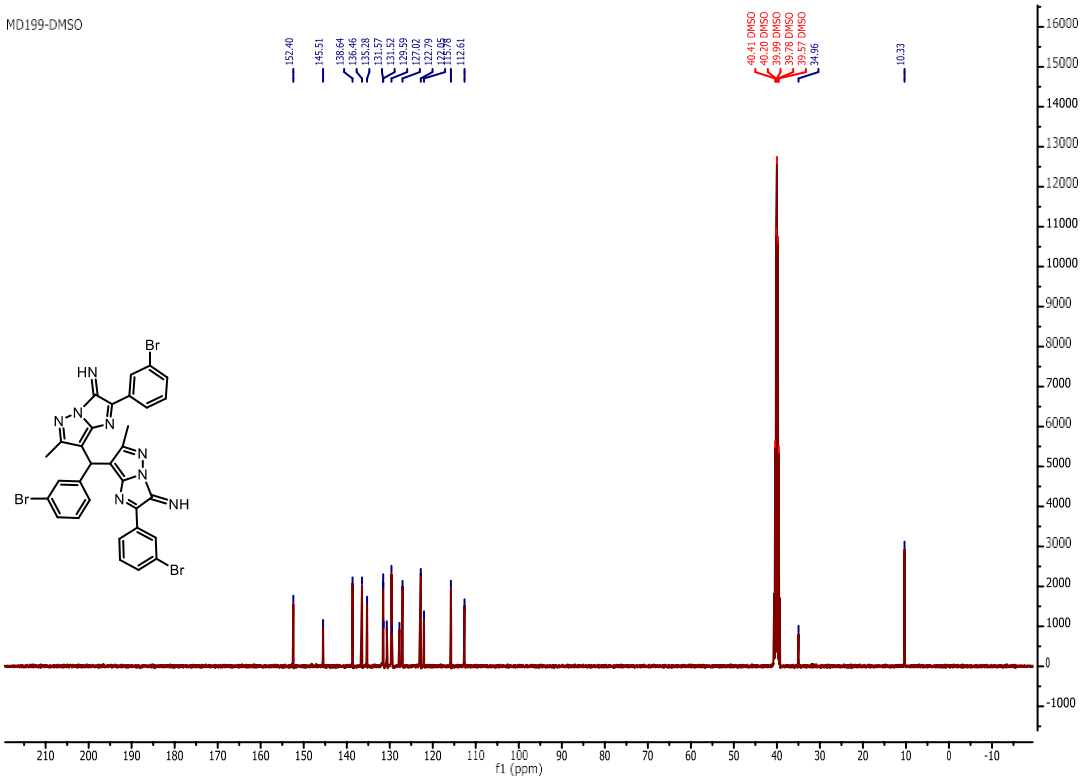


7,7'-((3-Bromophenyl)methylene)bis(2-(3-bromophenyl)-6-methyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9r**):

^{1}H NMR (400 MHz,DMSO-d_6)

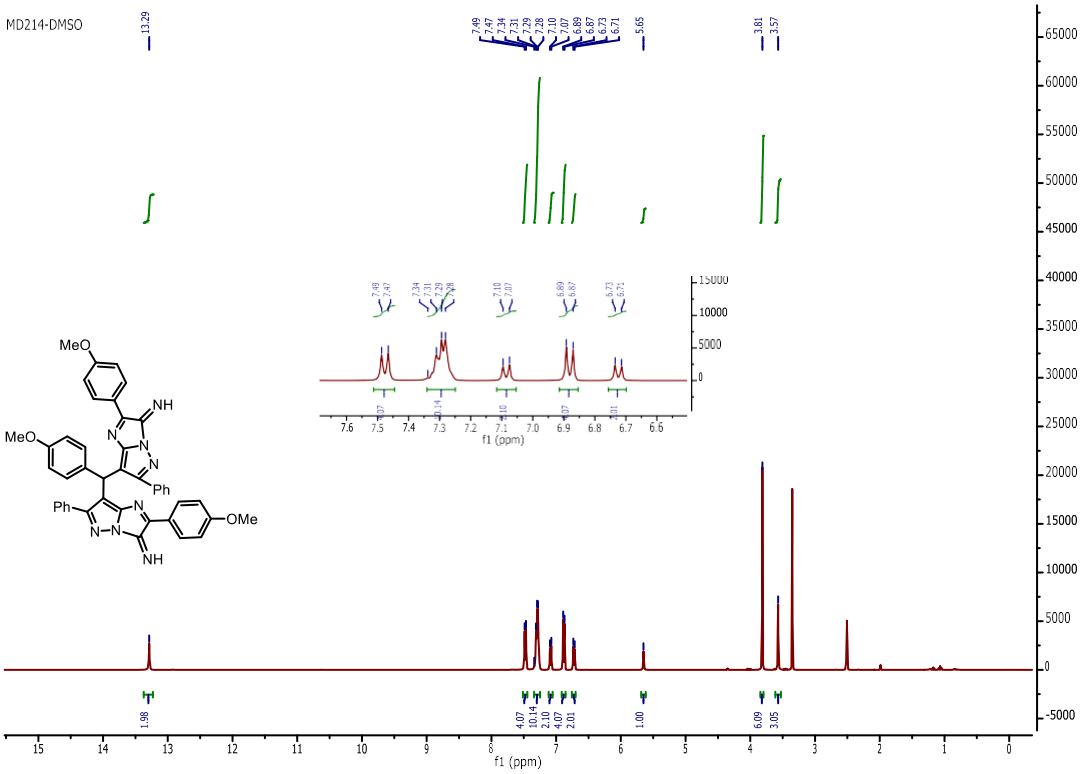


^{13}C NMR (100 MHz, DMSO-d_6)

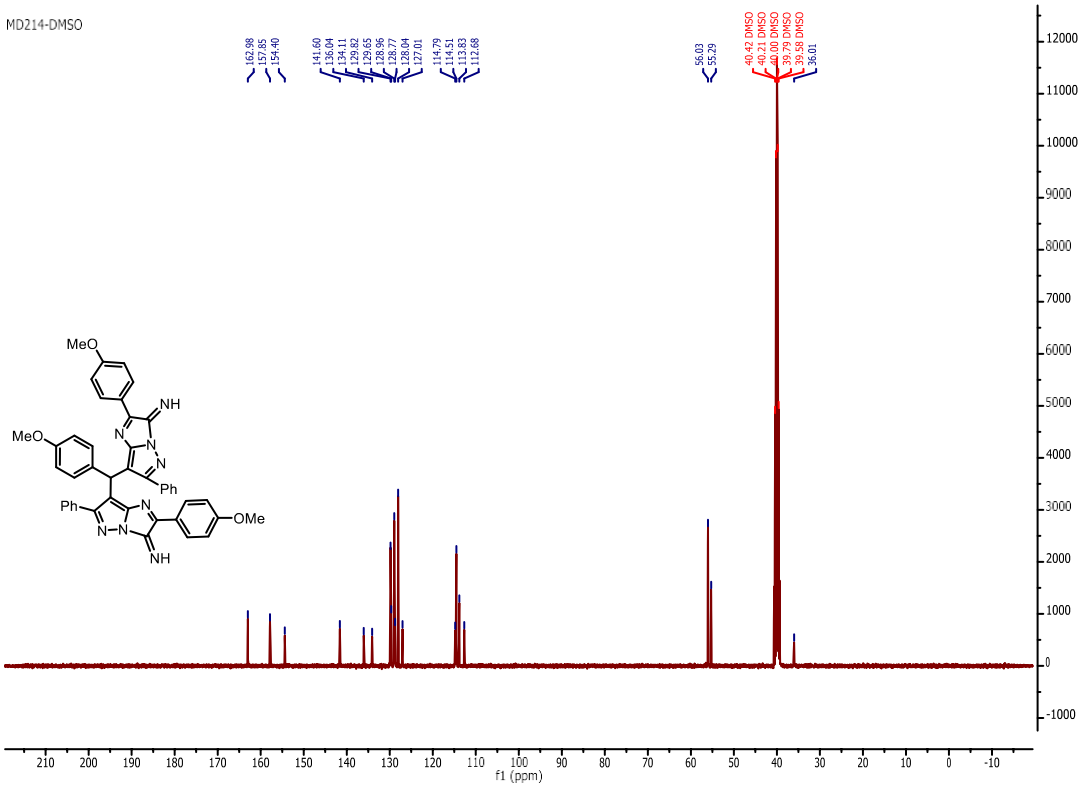


7,7'-((4-Methoxyphenyl)methylene)bis(2-(4-methoxyphenyl)-6-phenyl-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9u**):

^{1}H NMR (400 MHz, DMSO-d_6)

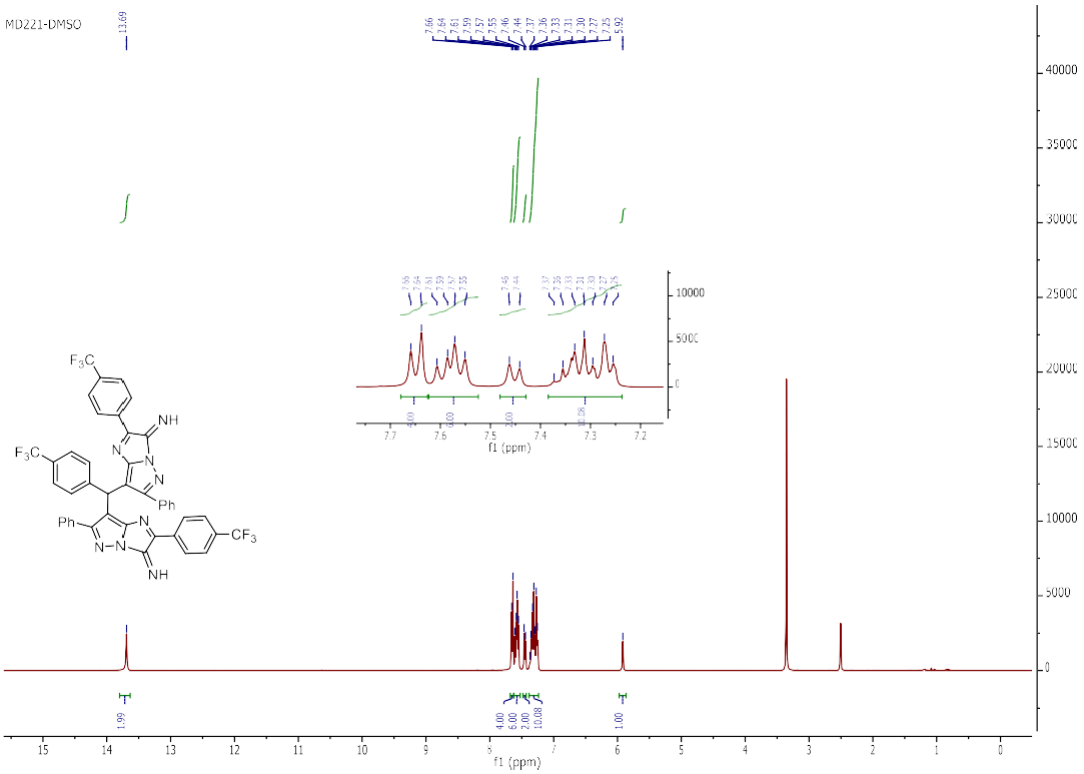


^{13}C NMR (100 MHz, DMSO-d_6)

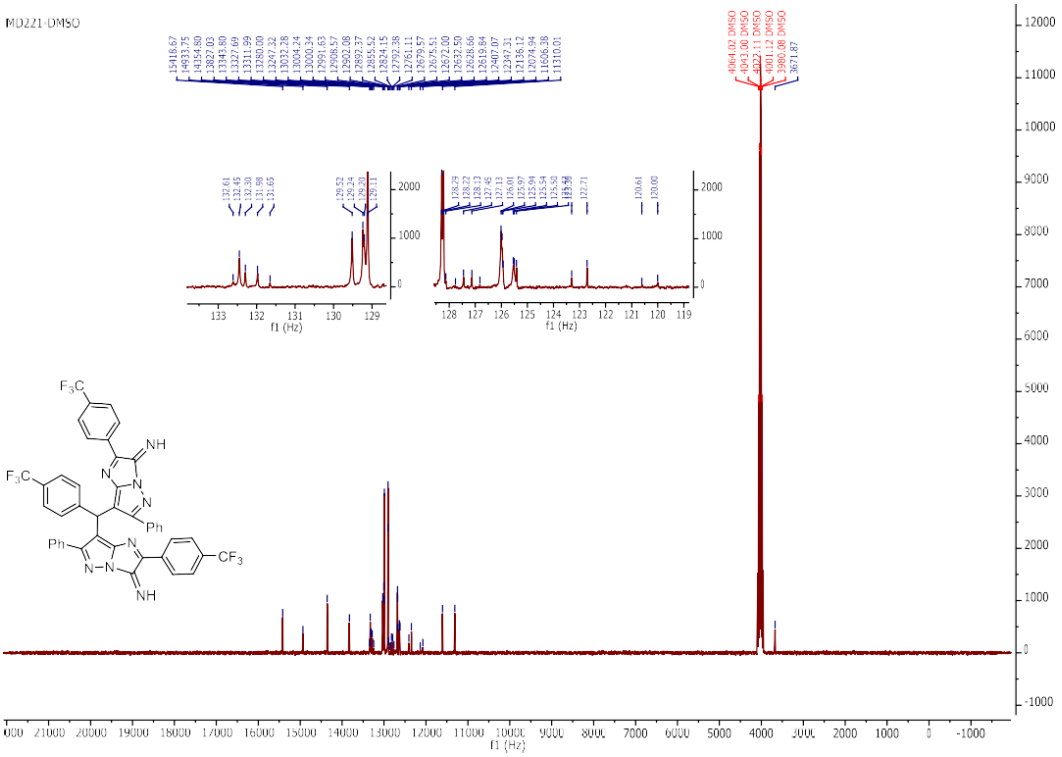


7,7'-((4-(Trifluoromethyl)phenyl)methylene)bis(6-phenyl-2-(4-(trifluoromethyl)phenyl)-3*H*-imidazo[1,2-*b*]pyrazol-3-imine) (**9v**):

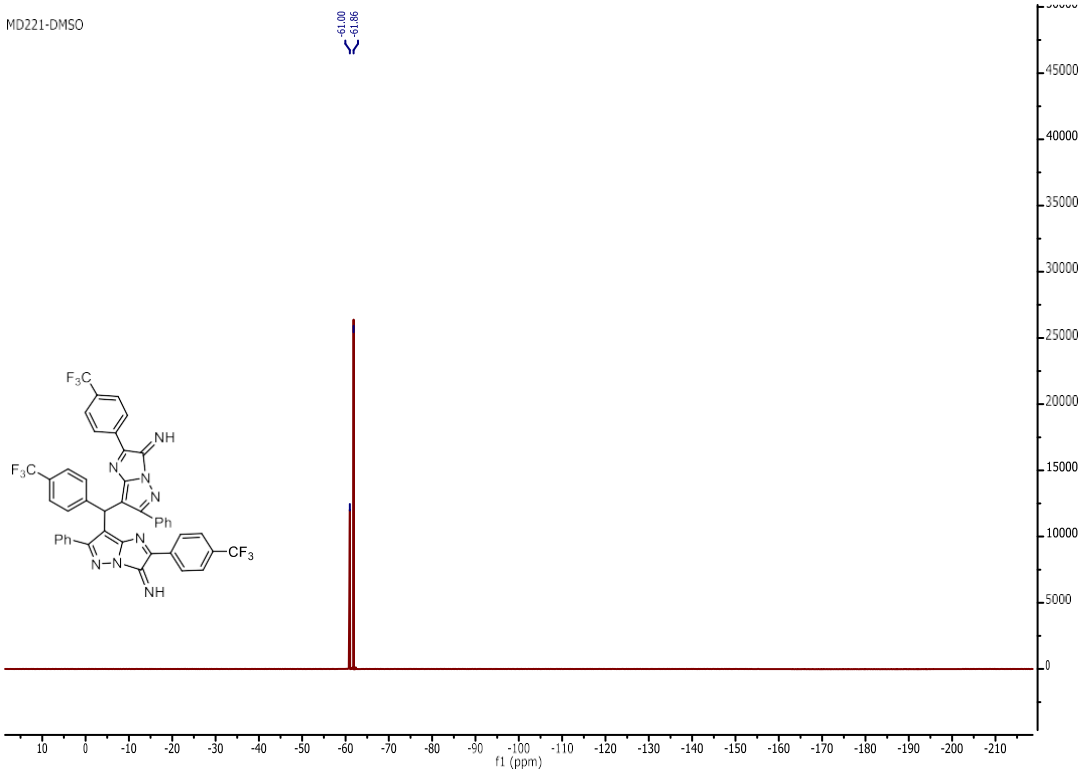
^{1}H NMR (400 MHz,DMSO-d_6)



^{13}C NMR (100 MHz, DMSO-d_6)



^{19}F NMR (376 MHz, DMSO-d_6)



Crystallographic data collections and structural determinations :

Suitable crystals for diffraction experiments of compound **8b** and **8o** were obtained by using a mixture of DCM/MeOH (1:2 v/v). Crystallographic studies were performed at **170** K. Single crystals were mounted on a Xcalibur equipped with monochromatized Mo-Kα radiation (0.71073 Å). The data collection, unit cell refinement, and data reduction were performed using the CrysAlis CCD, Oxford Diffraction Ltd. software package[1].
The structural determination was carried out by direct methods, and the refinement of atomic parameters based on full-matrix least-squares on F^2 were performed using the SHELX-2014 programs[2] within the WINGX package[3]. The positions of non-H atoms were determined and refinement by SHELX-2014 program[3]. A summary of crystallographic data for **8b** and **8o** were resumed in table 1.
The positions of the H atoms were deduced from coordinates of the non-H atoms and Fourier synthesis. H atoms were included for structure factor calculations but not refined. Supplementary crystallographic data can be found in the CCDC deposit (CCDC 1812145-1812146), and obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif/.

1 CrysAlis CCD, Oxford Diffraction Ltd., Version 1.171.33.46.
2 G. M. Sheldrick, *Programs for Crystal Structure Analysis*; University of Göttingen: Göttingen, Germany, 2014.
3 L. J. Farrugia, WinGX, *J. Appl. Cryst.* 2012, **45**, 849-854.

Table 1. Crystal data and structure refinement for compound **8b** and **8o**.

Identification code	**8b**	**8o**
Empirical formula	C48.75 H59.5 Cl1.5 N11	C50 H62 N11
Formula weight	852.74	817.10
Temperature	170(2) K	170(2) K
Wavelength	0.71073 Å	0.71073 Å
Crystal system	Triclinic	Monoclinic
Space group	P -1	P 21/n
Unit cell dimensions	a = 11.8594(6) Å ; α= 105.058(4)°	a = 24.6041(16) Å ; α= 90°
	b = 13.3077(7) Å ; β= 101.304(4)°	b = 6.0490(7) Å ; β= 92.106(6)°
	c = 15.7675(8) Å ; γ = 96.287(4)°	c = 31.305(3) Å ; γ = 90°
Volume	2322.3(2) Å^3	4656.0(7) Å^3
Z	2	4
Density (calculated)	1.219 g/cm^3	1.166 g/cm^3
Absorption coefficient	0.158 mm^{-1}	0.071 mm^{-1}
F(000)	909	1756
Crystal size	0.170 x 0.110 x 0.090 mm^3	0.340 x 0.140 x 0.070 mm^3
Theta range for data collection	3.217 to 26.372°	3.330 to 26.371°
Index ranges	-14≤h≤13, -16≤k≤15, -12≤l≤19	-30≤h≤30, -7≤k≤3, -39≤l≤22
Reflections collected	16767	19938
Independent reflections	9476 [R(int) = 0.1147]	9527 [R(int) = 0.0993]
Completeness to θ = 25.242°	99.7 %	99.8 %
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	9476 / 7 / 590	9527 / 0 / 566
Goodness-of-fit on F^2	1.012	0.993

Final R indices [I>2σ (I)]	R1 = 0.0770, wR2 = 0.1827	R1 = 0.0731, wR2 = 0.1666
R indices (all data)	R1 = 0.1502, wR2 = 0.2404	R1 = 0.1463, wR2 = 0.2154
Extinction coefficient	0.0000(10)	0.0008(4)
Largest diff. peak and hole	0.434 and -0.305 e.Å^{-3}	0.354 and -0.236 e.Å^{-3}