Supporting Information

for

Visible-light-promoted decarboxylative addition cyclization of *N*-aryl glycines and azobenzenes to access 1,2,4-triazolidines

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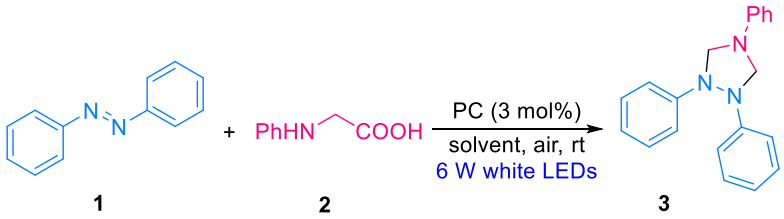
1. General Information

^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on a Varian Mercury-400 Plus spectrometer or an Agilent Technologies DD2 (600 MHz) in $CDCl_3$. Chemical shifts (δ) for NMR were quoted in ppm relative to the solvent peak (7.26 ppm for ^{1}H and 77.00 ppm for ^{13}C in $CDCl_3$). Coupling constants J are recorded in Hz. High-resolution mass spectra (HRMS) were reported from the Thermo Orbitrap Elite with an ESI source. UV-Visible absorption spectra were recorded on an Agilent 8453 spectrophotometer. Photoluminescence spectra were obtained with a HORIBA FluroMax-4 Spectrofluorometer. Melting points (m.p.) were measured on an XT4A apparatus (uncorrected). The X-ray single-crystal diffraction was performed on an Agilent SuperNOVA instrument. Reactions were monitored by thin layer chromatography (TLC) using pre-coated silica gel plates (GF254). Flash column chromatography was performed on silica gel 60 (particle size 200–400 mesh ASTM, purchased from Liangchen, China) and eluted with petroleum ether /ethylacetate. The symmetrical azobenzenes were prepared according to the literature procedure.[1] The unsymmetrical azobenzenes were synthesized according to the literature procedure.[2] The other materials and solvents obtained from commercial suppliers were used directly without further purification.

Unless otherwise noted, all photochemical reactions were carried out in Pyrex glass tube with magnetic stirring bar. The LED white lamps employed in this work were bought from Wuhan Jiushang Technology Co. LTD; Power (6 W); The setup of photocatalytic reaction as illustrated in Figure S1. The distance from the light source to the irradiation vessel is about 1.5 cm. The temperature is controlled by a fan. No filter was used in this reaction.

Figure S1. Setup of Photocatalytic Reaction and Light Characteristics

2. Optimization of Reaction Conditions[a]

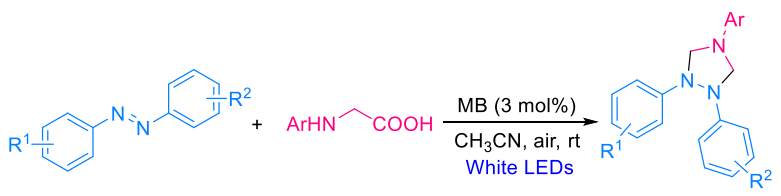


1 2 3

Entry	Photocatalyst	Solvent	Yield(%) [b]
1	MB	CH_3CN	95
2	Rose Bengal	CH_3CN	48
3	Eosin Y	CH_3CN	0
4	Mes-Acr$^+$$ClO_4^-$	CH_3CN	trace
5	Rhodamine B	CH_3CN	0
6	Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6	CH_3CN	0
7	MB	CH_3OH	12
8	MB	toluene	0
9	MB	acetone	44
10	MB	THF	0
11	MB	DMF	trace
12	MB	dioxane	0
13	MB	DMSO	0
14	MB	CH_2Cl_2	84
15[c]	–	CH_3CN	0
16[d]	MB	CH_3CN	0
17[e]	MB	CH_3CN	83
18[f]	MB	CH_3CN	92
19[g]	MB	CH_3CN	81
20[h]	MB	CH_3CN	72

[a]Reaction conditions: A solution of **1** (0.2 mmol), **2** (0.5 mmol, 2.5 equiv.) and MB (3.0 mol%) in CH_3CN (3.0 mL) is irradiated by 6 W white LEDs at room temperature for 4 h. [b]Isolated yield. [c]Without photocatalyst. [d]In the dark. [e]**2** (0.4 mmol, 2 equiv.). [f]**2** (0.6 mmol, 3 equiv.). [g]6 W bule LEDs, [h]6 W green LEDs

3. General Procedure for the Synthesis of 1,2,4-Triazolidines



Azobenzenes (0.2 mmol), ***N***-aryl glycines (0.5 mmol, 2.5 equiv.), MB (1.9 mg, 0.006 mmol,

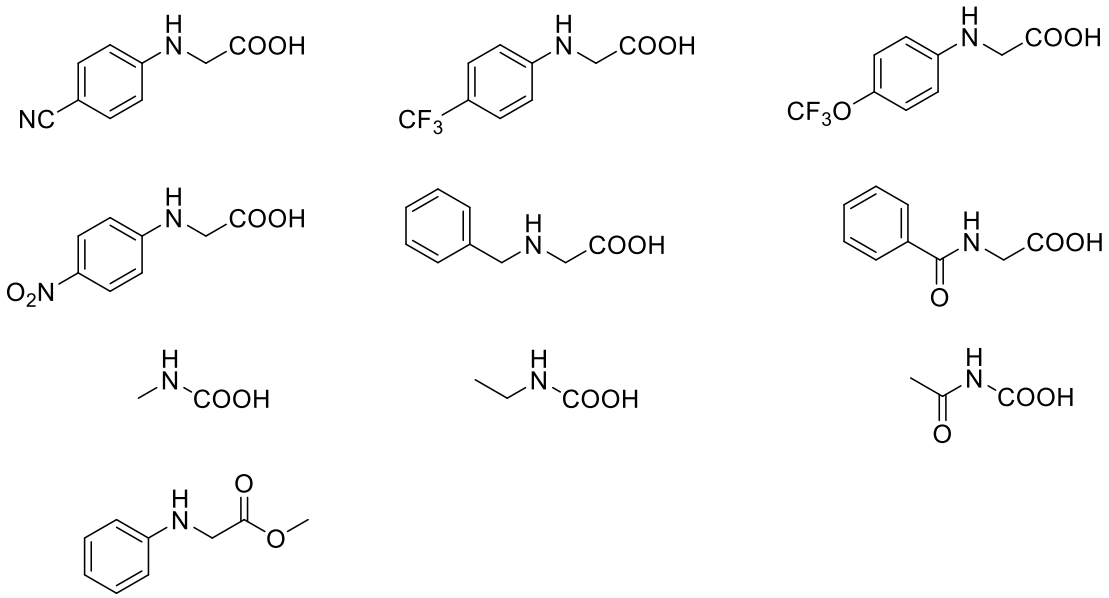
3.0 mol%) and CH_3CN (3 mL) were added into a 10 mL Pyrex glass tube equipped with a magnetic stir bar. Then, the reaction mixture was stirred under irradiation of 6 W white LEDs at room temperature. After azobenzenes were consumed completely (monitored by TLC), the reaction mixture was concentrated under vacuum and the residue was purified by column chromatography on silica gel (PE/EtOAc = 200/1 to 50/1) to afford the desired product.

4. Procedure for the Gram-Scale Synthesis of 3

1 (4 mmol) **2** (10 mmol) **3** (0.855 g, 71%)

Azobenzene **1** (0. 728 g, 4.0 mmol), *N*-phenyl glycine **2** (1.512 g, 10 mmol, 2.5 equiv.), MB (38.4 mg，0.12 mmol, 3.0 mol%) and CH_3CN (50.0 mL) were added into a 100 mL round flask equipped with a magnetic stirring bar. The mixture was then irradiated by two 15 W white LEDs and stirred at room temperature for 24 hours. Upon completion (monitored by TLC), the reaction mixture was concentrated under vacuum and the residue was purified by column chromatography (petrol ether/EtOAc = 200:1) to give the desired product **3** (0.855 g, 71%) as a white solid.

5. Unsuccessful Substrates



6. Crystallographic Structure Determination

The colorless single crystal of product **15** was obtained by recrystallization using a $CHCl_3$/acetone solvent system at room temperature. The X-ray single-crystal diffraction was performed on an Agilent SuperNOVA instrument (**Figure S2**).

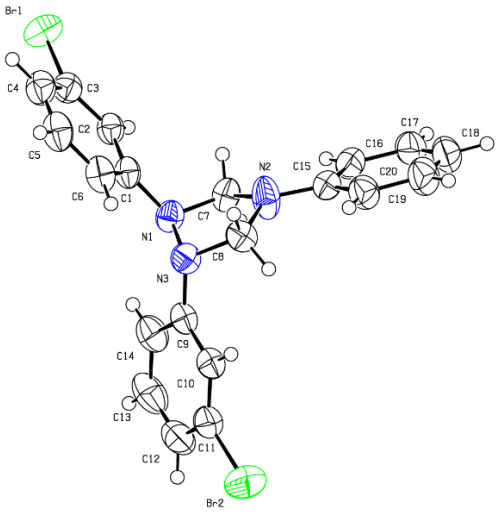


Figure S2. Thermal ellipsoid plot of compound **15** at the 30% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Table S1. Crystal data and structure refinement for songmh_1210.

Identification code	songmh_1210
Empirical formula	$C_{20}H_{17}Br_2N_3$
Formula weight	459.18
Temperature/K	294.65(10)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	14.62006(16)
b/Å	5.80584(6)
c/Å	22.6165(2)
α/°	90
β/°	104.1994(11)

γ/°	90
Volume/Å^3	1861.08(4)
Z	4
ρ_{calc}g/cm^3	1.639
μ/mm^{-1}	5.573
F(000)	912.0
Crystal size/mm^3	0.11 × 0.05 × 0.03
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	6.542 to 152.118
Index ranges	-17 ≤ h ≤ 17, -7 ≤ k ≤ 5, -27 ≤ l ≤ 26
Reflections collected	11202
Independent reflections	3576 [R_{int} = 0.0198, R_{sigma} = 0.0164]
Data/restraints/parameters	3576/0/226
Goodness-of-fit on F^2	1.037
Final R indexes [I>=2σ (I)]	R_1 = 0.0458, wR_2 = 0.1208
Final R indexes [all data]	R_1 = 0.0481, wR_2 = 0.1229
Largest diff. peak/hole / e Å^{-3}	0.68/-0.84

Table S2. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for songmh_1210. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br1	3782.3(2)	6187.7(7)	2078.5(2)	64.64(16)
Br2	-4161.5(3)	7659.9(13)	-252.8(2)	100.5(2)
N1	108.1(17)	6898(4)	1404.2(11)	45.1(5)
N2	-618(2)	7999(6)	2136.6(13)	65.9(8)
N3	-702.5(17)	8209(4)	1111.8(11)	46.3(5)
C1	983(2)	8008(5)	1422.2(12)	42.0(6)
C2	1810(2)	6869(5)	1710.7(12)	43.0(6)
C3	2668(2)	7849(5)	1707.5(13)	46.8(6)
C4	2740(2)	9943(5)	1434.5(14)	54.5(7)
C5	1917(3)	11041(5)	1147.2(15)	58.0(8)
C6	1037(2)	10110(5)	1132.0(14)	52.0(7)
C7	-63(2)	6149(5)	1981.0(14)	46.2(6)
C8	-1015(2)	9366(5)	1604.2(13)	46.7(6)
C9	-1374(2)	6903(5)	680.8(12)	44.8(6)
C10	-2283(2)	7736(6)	463.4(13)	49.5(7)
C11	-2930(3)	6473(7)	34.4(14)	60.3(8)
C12	-2697(3)	4432(7)	-191.8(15)	68.9(10)
C13	-1788(4)	3647(6)	10.9(17)	73.1(11)
C14	-1119(3)	4856(6)	444.8(15)	59.0(8)
C15	-1004(2)	7909(6)	2642.5(13)	48.1(6)
C16	-780(2)	6098(6)	3056.6(14)	53.0(7)
C17	-1132(3)	6092(7)	3575.4(15)	62.3(9)
C18	-1688(3)	7868(7)	3682.3(16)	68.8(10)
C19	-1911(3)	9632(7)	3273.1(17)	68.7(9)
C20	-1581(2)	9683(6)	2750.0(15)	56.9(7)

Table S3. Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for songmh_1210. 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}
Br1	44.9(2)	62.2(3)	81.9(3)	0.60(17)	6.16(16)	-5.34(14)
Br2	56.0(3)	155.5(6)	80.7(3)	1.7(3)	-0.8(2)	-4.5(3)
N1	43.3(12)	43.6(12)	50.6(12)	5.6(10)	15.7(10)	7.7(10)
N2	75.0(19)	74.0(18)	58.5(15)	23.6(14)	35.2(14)	36.0(16)
N3	46.6(13)	44.5(12)	48.0(12)	-1.2(10)	11.8(10)	9.3(10)
C1	49.3(15)	37.7(13)	43.5(13)	-1.7(10)	20.2(11)	2.5(11)
C2	49.2(15)	38.2(13)	45.1(14)	-0.9(11)	18.2(11)	-0.9(11)
C3	50.4(16)	43.6(14)	48.3(14)	-8.0(12)	15.6(12)	-3.2(12)
C4	62.4(19)	48.1(16)	58.6(17)	-6.2(13)	25.4(14)	-11.8(14)
C5	80(2)	39.9(15)	62.3(18)	4.4(13)	33.6(17)	-1.9(14)
C6	65.9(19)	43.0(15)	52.3(15)	6.8(12)	24.6(14)	9.7(14)
C7	46.7(15)	39.2(14)	56.8(16)	8.6(11)	20.7(12)	4.5(11)
C8	52.7(16)	38.5(13)	48.1(14)	-2.5(11)	11.0(12)	10.0(12)
C9	54.7(16)	43.9(14)	38.8(13)	1.6(11)	17.4(11)	0.6(12)
C10	52.1(16)	56.4(17)	41.5(14)	1.3(12)	14.5(12)	0.0(13)
C11	60.3(19)	79(2)	43.6(15)	3.4(15)	16.5(14)	-12.3(17)
C12	89(3)	75(2)	43.8(16)	-6.8(16)	18.9(16)	-27(2)
C13	116(4)	55(2)	54.6(19)	-13.4(15)	32(2)	-7(2)
C14	75(2)	53.0(18)	51.7(16)	-3.3(14)	21.7(15)	10.0(16)
C15	39.8(14)	59.5(17)	46.0(14)	-0.5(12)	12.2(11)	0.7(13)
C16	41.6(15)	65.0(19)	53.8(16)	3.3(14)	14.3(12)	1.3(13)
C17	64(2)	75(2)	49.9(17)	3.7(15)	17.7(15)	-13.2(17)
C18	76(2)	84(3)	54.3(18)	-15.8(18)	30.5(17)	-19(2)
C19	74(2)	72(2)	68(2)	-19.5(18)	32.2(18)	-1.0(19)
C20	61.4(19)	58.5(18)	53.4(16)	-5.8(14)	19.2(14)	4.4(15)

Table S4. Bond Lengths for songmh_1210.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br1	C3	1.902(3)	C4	C5	1.376(5)
Br2	C11	1.888(4)	C5	C6	1.388(5)
N1	N3	1.427(3)	C9	C10	1.385(4)
N1	C1	1.424(4)	C9	C14	1.391(4)
N1	C7	1.454(4)	C10	C11	1.387(5)
N2	C7	1.441(4)	C11	C12	1.367(6)
N2	C8	1.440(4)	C12	C13	1.373(6)
N2	C15	1.396(4)	C13	C14	1.393(6)
N3	C8	1.466(4)	C15	C16	1.392(4)
N3	C9	1.421(4)	C15	C20	1.390(5)
C1	C2	1.392(4)	C16	C17	1.392(4)
C1	C6	1.397(4)	C17	C18	1.371(6)
C2	C3	1.379(4)	C18	C19	1.365(6)
N3	C9	1.421(4)	C15	C20	1.390(5)
C1	C2	1.392(4)	C16	C17	1.392(4)
C1	C6	1.397(4)	C17	C18	1.371(6)
C2	C3	1.379(4)	C18	C19	1.365(6)
C3	C4	1.380(4)	C19	C20	1.383(5)

Table S5. Bond Angles for songmh_1210.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom
N3	N1	C7	105.6(2)	N2	C8	N3
C1	N1	N3	114.2(2)	C10	C9	N3
C1	N1	C7	117.5(2)	C10	C9	C14
C8	N2	C7	110.0(2)	C14	C9	N3
C15	N2	C7	122.5(3)	C9	C10	C11
C15	N2	C8	123.0(3)	C10	C11	Br2
N1	N3	C8	105.7(2)	C12	C11	Br2
C9	N3	N1	112.9(2)	C12	C11	C10
C9	N3	C8	118.3(2)	C11	C12	C13
C2	C1	N1	118.0(2)	C12	C13	C14
C2	C1	C6	119.5(3)	C9	C14	C13
C6	C1	N1	122.4(3)	C16	C15	N2
C3	C2	C1	119.2(3)	C20	C15	N2
C2	C3	Br1	118.0(2)	C20	C15	C16
C2	C3	C4	122.4(3)	C17	C16	C15
C4	C3	Br1	119.5(2)	C18	C17	C16
C5	C4	C3	117.7(3)	C19	C18	C17
C4	C5	C6	122.0(3)	C18	C19	C20
C5	C6	C1	119.2(3)	C19	C20	C15
N2	C7	N1	103.1(2)			

Table S6. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for songmh_1210.

Atom	*x*	*y*	*z*	U(eq)
H2	1783.81	5463.2	1903.25	52
H4	3325.77	10591.14	1444.26	65
H5	1951.25	12449.19	957.62	70
H6	490.24	10876.13	931.06	62
H7A	-408.82	4707.31	1934.4	55
H7B	524.26	5961.27	2288.85	55
H8A	-780.05	10934.63	1656.86	56
H8B	-1697.83	9394.11	1520.56	56
H10	-2457.3	9133.71	604.32	59
H12	-3142.21	3597.08	-475.4	83
H13	-1615.24	2276.73	-144.9	88
H14	-506.67	4297.63	575.62	71
H16	-397.97	4899.61	2986.82	64
H17	-988.64	4877.83	3850.8	75
H18	-1913.34	7871.32	4032.86	83
H19	-2292.89	10824.65	3347.48	82
H20	-1743.36	10888.78	2473.48	68

7. Mechanistic Studies

7.1 UV-Vis Absorption Spectra

The UV-visible absorption spectra of azobenzene **1**, *N*-phenyl glycine **2**, methylene bule (MB) in acetonitrile show that **1** especially MB has significant absorption in the visible-light region, while **2** has no obvious absorption in the visible-light region (Figure S3).

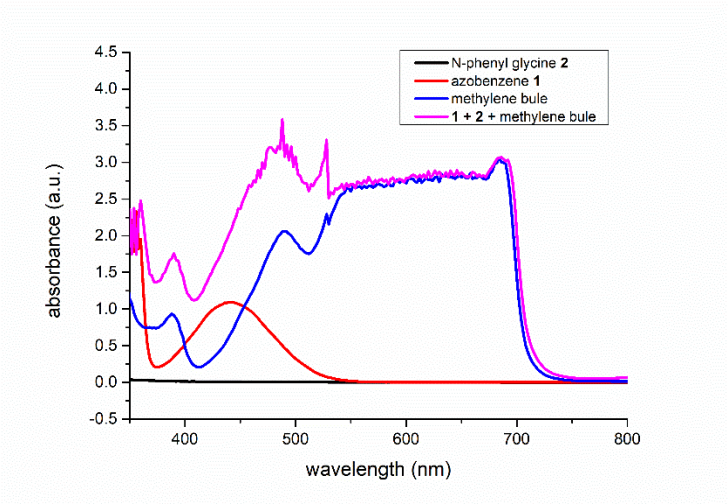


Figure S3. The UV-Vis absorption spectra in CH_3CN (2 mM)

7.2 Stern-Volmer Quenching Experiments

Stern-Volmer luminescence quenching experiments were run with freshly prepared solutions of 1.0×10^{-4} M MB and the appropriate amount of quencher in CH_3CN at room temperature. The solutions were irradiated at 425 nm and luminescence was measured at 702 nm. Then, appropriate amount of quencher was added to the measured solution, the emission spectrum of the sample was collected (Figures S4–S6).

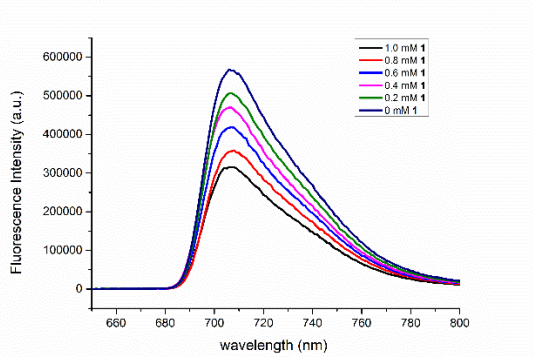


Figure S4. Fluorescence quenching of MB with **1** (λ_{ex} = 425 nm)

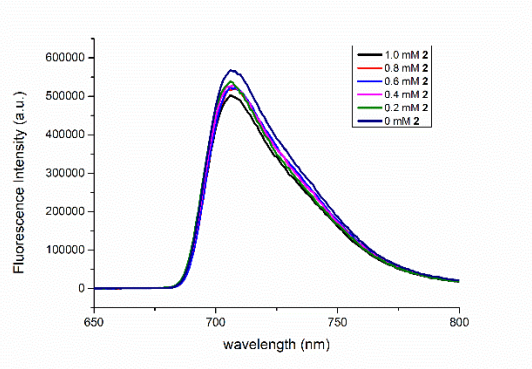


Figure S5. Fluorescence quenching of MB with **2** (λ_{ex} = 425 nm)

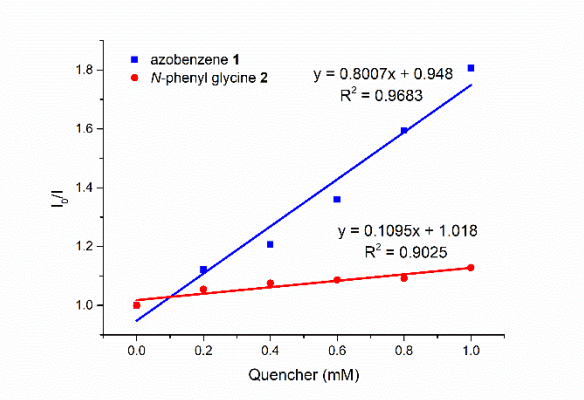
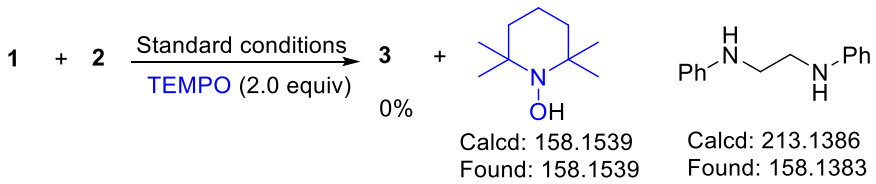


Figure S6. Stern-Volmer analysis

7.3 Radical Inhibiting Experiment with TEMPO



Azobenzene **1** (36.4 mg, 0.2 mmol), *N*-phenyl glycine **2** (75.6 mg, 0.5 mmol, 2.5equiv.), MB (1.9 mg, 0.006 mmol, 3.0 mol%), TEMPO (62.5 mg, 0.4 mmol, 2 equiv.) and CH_3CN (3 mL) were added into a 10 mL Pyrex glass tube. The reaction mixture was continually stirred at room temperature under 6 W white LEDs irradiation for 4 hours. There, **3** was not detected by TLC and the reduced TEMPO-H and a dimerization product *N*,*N*'-diphenylethane-1,2-diamine were detected by HRMS (Figures S7–S8, data of $[M+H]^+$are showed).

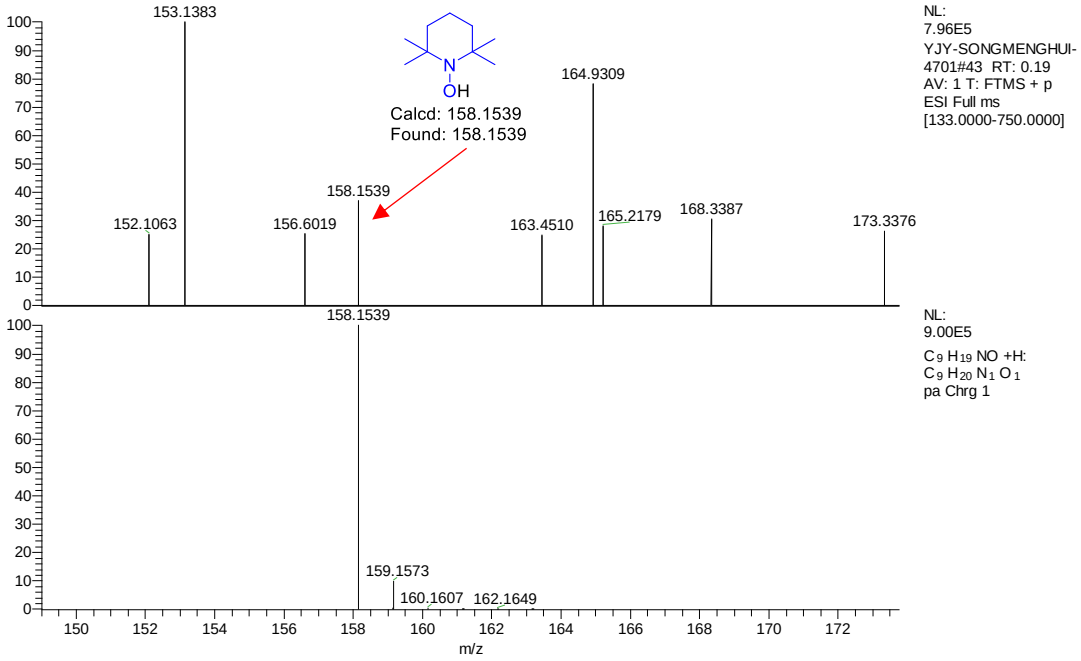


Figure S7

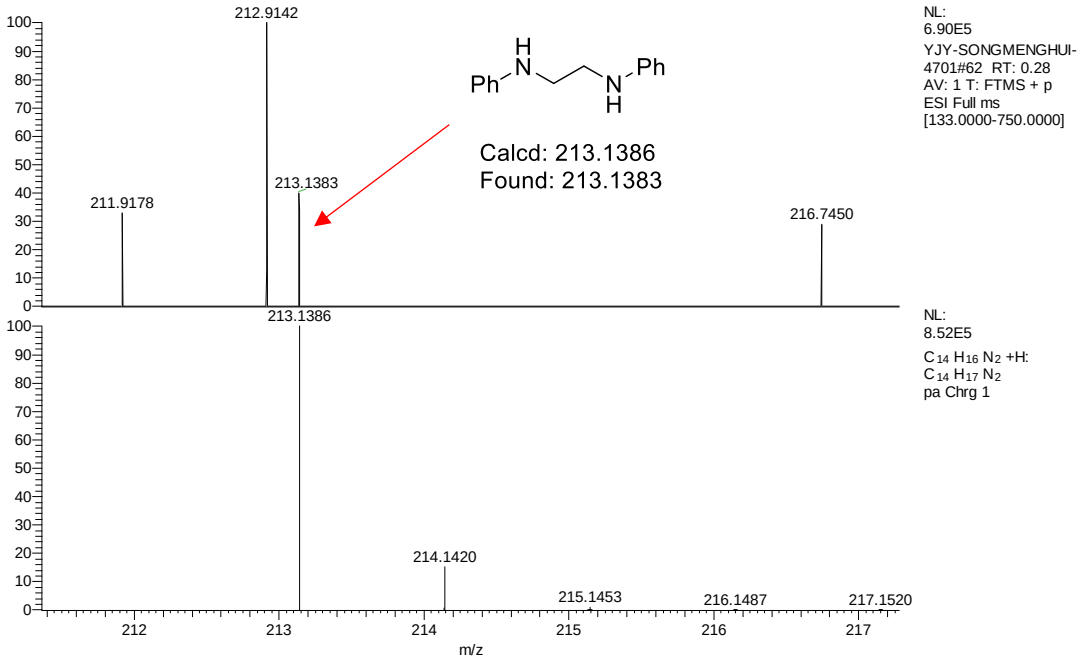


Figure S8

7.4 Detecting the Reaction Mixture by HRMS

Azobenzene **1** (36.4 mg, 0.2 mmol), *N*-phenyl glycine **2** (75.6 mg, 0.5 mmol, 2.5equiv.), MB (1.9 mg, 0.006 mmol, 3.0 mol%) and CH_3CN (3 mL) were added into a 10 mL Pyrex glass tube. The reaction mixture was continually stirred at room temperature under 6 W white LEDs irradiation for 1.5 hours. And then, without any treatment, the reaction mixture was detected by HRMS (Figures S9–S11, data of $[M+H]^+$are showed).

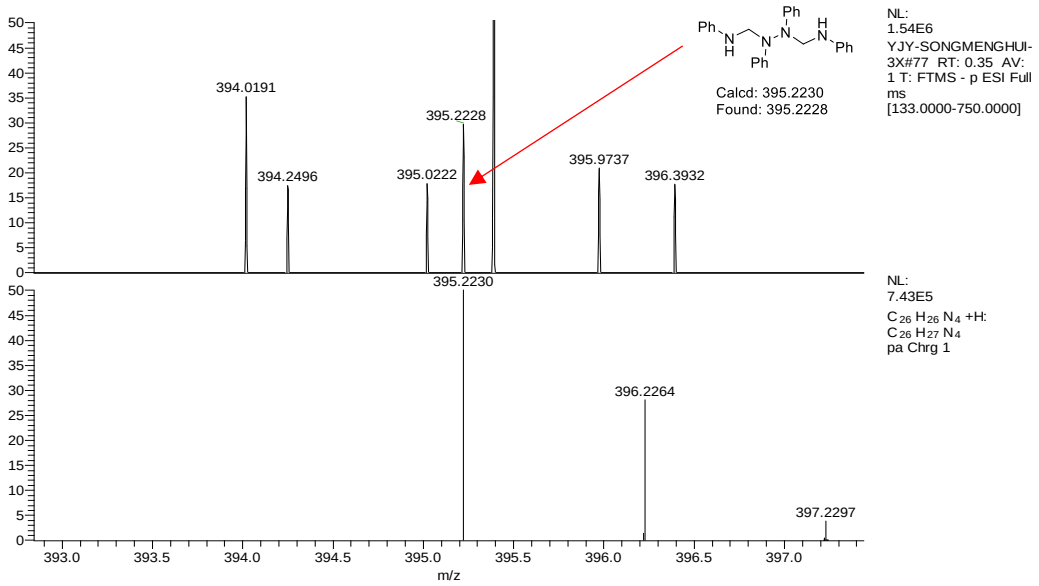


Figure S9

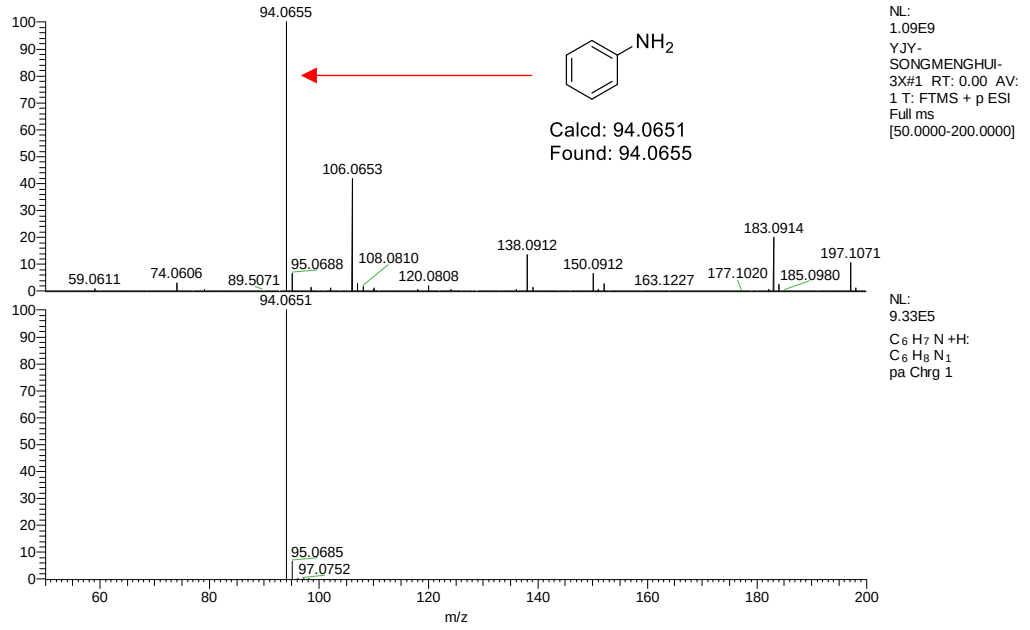


Figure S10

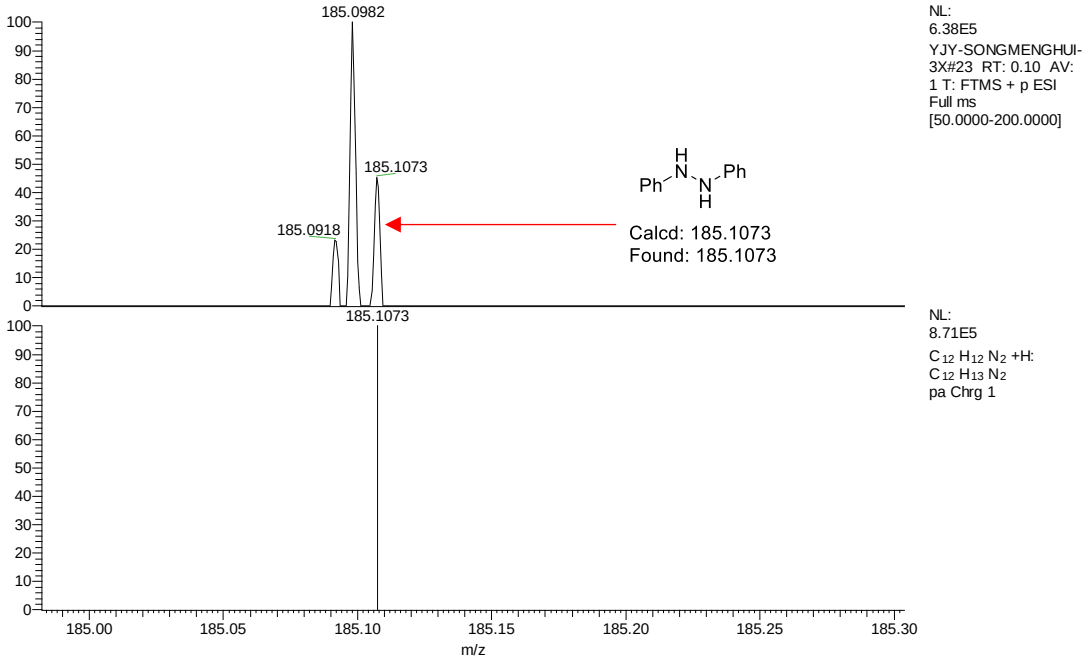
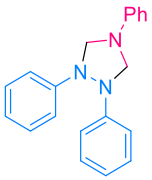


Figure S11

8. Characterization Data of Products

1) 1,2,4-Triphenyl-1,2,4-triazolidine (3)[3]



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product **3**.

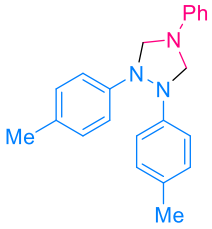
White solid, m.p. 140–141 °C; yield: 57.2 mg (95%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33–7.28 (m, 4H), 7.27–7.19 (m, 6H), 6.98 (t, J = 7.4 Hz, 2H), 6.80 (t, J = 7.4 Hz, 1H), 6.61 (d, J = 8.0 Hz, 2H), 4.89 (s, 2H), 4.71 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.2, 145.2, 129.4, 129.2, 121.6, 118.4, 115.0, 113.3, 67.1.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{20}N_3^+$: 302.1652; found: 302.1651.

2) 4-Phenyl-1,2-di-*p*-tolyl-1,2,4-triazolidine (4)



Prepared according to the general procedure from (*E*)-1,2-di-*p*-tolyldiazene (42.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product **4**.

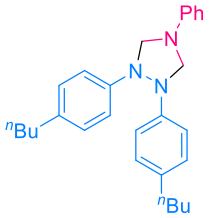
White solid, m.p. 134–136 ℃; yield: 61.3 mg (93%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27–7.20 (m, 2H), 7.10–7.08 (m, 8H), 6.76 (t, J = 7.2 Hz, 1H), 6.57 (d, J = 7.6 Hz, 2H), 4.82 (s, 2H), 4.65 (s, 2H), 2.28 (s, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 148.0, 145.3, 130.9, 129.7, 129.3, 118.2, 115.2, 113.2, 67.2, 20.4.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{22}H_{24}N_3^+$: 330.1965; found: 330.1965.

3) 1,2-Bis(4-butylphenyl)-4-phenyl-1,2,4-triazolidine (5)



Prepared according to the general procedure from (*E*)-1,2-bis(4-butylphenyl)diazene (58.9 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product **5**.

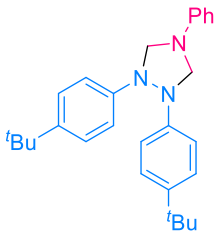
White solid, m.p. 104–105 ℃; yield: 76.1 mg (92%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26–7.20 (m, 2H), 7.15–7.07 (m, 8H), 6.78–6.74 (m, 1H), 6.61–6.54 (m, 2H), 4.84 (s, 2H), 4.66 (s, 2H), 2.54 (t, J = 7.8 Hz, 4H), 1.60–1.52 (m, 4H), 1.39–1.30 (m, 4H), 0.92 (t, J = 7.2 Hz, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 148.1, 145.1, 136.0, 129.3, 129.0, 118.1, 115.0, 113.1, 67.1, 34.8, 33.8, 22.3, 13.9.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{28}H_{36}N_3^+$: 414.2904; found: 414.2907.

4) 1,2-Bis(4-(*tert*-butyl)phenyl)-4-phenyl-1,2,4-triazolidine (6)



Prepared according to the general procedure from (*E*)-1,2-bis(4-(*tert*-butyl)phenyl)diazene (58.9 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product **6**.

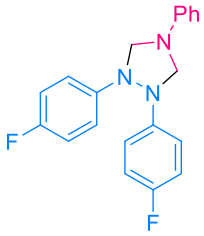
White solid, m.p. 158–160 ℃; yield: 73.6 mg (89%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.29 (m, 4H), 7.25–7.20 (m, 2H), 7.16–7.12 (m, 4H), 8.79–6.74 (m, 1H), 6.59–6.54 (m, 2H), 4.86 (s, 2H), 4.67 (s, 2H), 1.30 (s, 18H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 147.8, 145.1, 144.3, 129.3, 125.9, 118.0, 114.7, 113.0, 67.0, 34.1, 31.5.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{28}H_{36}N_3^+$: 414.2904; found: 414.2906.

5) 1,2-Bis(4-fluorophenyl)-4-phenyl-1,2,4-triazolidine (7)



Prepared according to the general procedure from (*E*)-1,2-bis(4-fluorophenyl)diazene (43.6 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to a afford the pure product **7**.

White solid, m.p. 125–126 ℃; yield: 60.0 mg (89%).

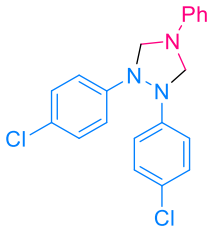
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28–7.22 (m, 2H), 7.15–7.11 (m, 4H), 7.06–6.94 (m, 4H), 6.81 (t, J = 7.2 Hz, 1H), 6.64–6.56 (m, 2H), 4.77 (s, 2H), 4.70 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 159.2 (d, J = 238.5 Hz), 146.4 (d, J = 1.5 Hz), 145.1, 129.4, 118.8, 116.6 (d, J = 7.5 Hz), 115.7 (d, J = 22.5 Hz), 113.5, 67.8.

^{19}F NMR (376 MHz, $CDCl_3$): δ -123.2 – -123.3.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{18}F_2N_3^+$: 338.1463; found: 338.1464.

6) 1,2-Bis(4-chlorophenyl)-4-phenyl-1,2,4-triazolidine (8)



Prepared according to the general procedure from (*E*)-1,2-bis(4-chlorophenyl)diazene (50.2 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product **8**.

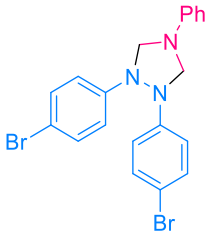
White solid, m.p. 155–156 ℃; yield: 64.4 mg (87%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29–7.21 (m, 6H), 7.11–7.05 (m, 4H), 6.82 (t, J = 7.4 Hz, 1H), 6.64–6.55 (m, 2H), 4.79 (s, 2H), 4.68 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 148.5, 144.8 129.4, 129.1, 126.7, 118.9, 116.3, 113.5, 67.4.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{18}Cl_2N_3^+$: 370.0872; found: 370.0876.

7) 1,2-Bis(4-bromophenyl)-4-phenyl-1,2,4-triazolidine (9)



Prepared according to the general procedure from (*E*)-1,2-bis(4-bromophenyl)diazene (68.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product **9**.

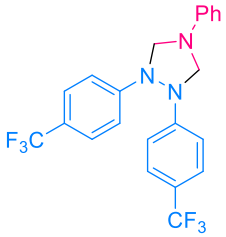
White solid, m.p. 160–161 ℃; yield: 80.8 mg (88%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41–7.38 (m, 4H), 7.29–7.24 (m, 2H), 7.05–7.02 (m, 4H), 6.86–6.20 (m, 1H), 6.62–6.60 (m, 2H), 4.79 (s, 2H), 4.68 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 148.9, 144.8, 132.0, 129.4, 118.9, 116.7, 114.1, 113.5, 67.2.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{18}Br_2N_3^+$: 459.9842; found: 459.9843.

8) 4-Phenyl-1,2-bis(4-(trifluoromethyl)phenyl)-1,2,4-triazolidine (10)



Prepared according to the general procedurefrom (*E*)-1,2-bis(4-(trifluoromethyl)phenyl)diazene (63.6 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 100:1) to afford the pure product **10**.

White solid, m.p. 82–83C ℃; yield: 66.5 mg (76%).

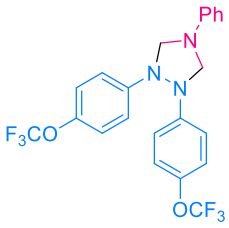
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57–7.54 (m, 4H), 7.30–7.26 (m, 2H), 7.19 (d, J = 8.8 Hz, 4H), 6.85 (t, J = 7.4 Hz, 1H), 6.66–6.63 (m, 2H), 4.91 (s, 2H), 4.76 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 152.2, 144.7, 129.5, 126.6 (q, J = 4.5 Hz), 124.6 (d, J = 190.5 Hz), 123.6 (d, J = 48.0 Hz) 119.4, 114.5, 113.7, 67.1.

^{19}F NMR (376 MHz, $CDCl_3$): δ = -62.0.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{22}H_{18}F_6N_3^+$: 438.1399; found: 438.1399.

9) 4-Phenyl-1,2-bis(4-(trifluoromethoxy)phenyl)-1,2,4-triazolidine (11)



Prepared according to the general procedure from (*E*)-1,2-bis(4-(trifluoromethoxy)phenyl)diazene (70.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 100:1) to afford the pure product **11**.

Colorless oil; yield: 80.7 mg (86%).

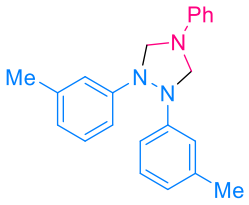
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30–7.25 (m, 2H), 7.14–7.20 (m, 8H), 6.84 (t, J = 7.4 Hz, 1H), 6.63 (d, J = 8.0 Hz, 2H), 4.83 (s, 2H), 4.73 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 148.6, 144.8, 143.8 (d, J =1.5 Hz), 129.5, 122.2, 121.4 (d, J = 255.0 Hz), 119.0, 115.9, 113.5, 67.6.

^{19}F NMR (376 MHz, $CDCl_3$): δ = -58.7.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{22}H_{18}F_6O_2N_3^+$: 470.1298; found: 470.1299.

10) 4-Phenyl-1,2-di-*m*-tolyl-1,2,4-triazolidine (12)



Prepared according to the general procedure from (*E*)-1,2-di-*m*-tolyldiazene (42.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE : EtOAc = 200:1) to afford the pure product **12**.

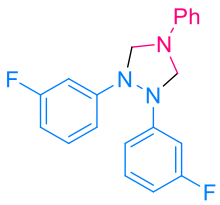
White solid, m.p. 58–59 ℃; yield: 56.2 mg (85%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29–7.19 (m, 4H), 7.06–7.00 (m, 4H), 6.83–6.78 (m, 3H), 6.65–6.60 (m, 2H), 4.90 (s, 2H), 4.70 (s, 2H), 2.37 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2, 145.1, 139.0, 129.3, 129.0, 122.3, 118.2, 115.5, 113.1, 112.0, 67.0, 21.7.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{22}H_{24}N_3^+$: 330.1965; found: 330.1964.

11) 1,2-Bis(3-fluorophenyl)-4-phenyl-1,2,4-triazolidine (13)



Prepared according to the general procedure from (*E*)-1,2-bis(3-fluorophenyl)diazene (43.6 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **13**.

White solid, m.p. 106–107 ℃; yield: 56.0 mg (83%).

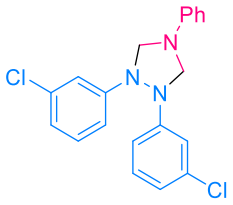
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28–7.23 (m, 4H), 6.94–6.87 (m, 4H), 6. 84–6.80 (m, 1H), 6.70–6.64 (m, 2H), 6.63–6.59 (m, 2H), 4.83 (s, 2H), 4.69 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 164.6 (d, J = 244.5 Hz), 151.9 (d, J = 9.0 Hz), 144.8, 130.5 (d, J = 9.0 Hz), 129.4, 118.9, 113.5, 110.5 (d, J = 1.5 Hz), 108.5 (d, J = 6.0 Hz), 102.5 (d, J = 27.0 Hz), 67.3.

^{19}F NMR (376 MHz, $CDCl_3$): δ = -112.26– -112.33.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{18}F_2N_3^+$: 338.1463; found: 338.1467.

12) 1,2-Bis(3-chlorophenyl)-4-phenyl-1,2,4-triazolidine (14)



Prepared according to the general procedure from (*E*)-1,2-bis(3-chlorophenyl)diazene (50.2mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **14**.

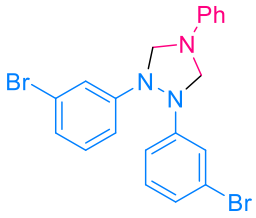
White solid, m.p. 90–92 ℃; yield: 62.2 mg (84%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29–7.20 (m, 6H), 7.03–6.94 (m, 4H), 6.84 (t, J = 7.2 Hz, 1H), 6.61 (d, J = 8.0 Hz, 2H), 4.82 (s, 2H), 4.69 (s, 2H)

^{13}C NMR (150 MHz, $CDCl_3$): δ 151.1, 144.7, 135.1, 130.2, 129.4, 121.8, 119.0, 115.1, 113.5, 113.2, 67.3.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{18}Cl_2N_3^+$: 370.0872; found: 370.0870.

13) 1,2-Bis(3-bromophenyl)-4-phenyl-1,2,4-triazolidine (15)



Prepared according to the general procedure from (*E*)-1,2-bis(3-bromophenyl)diazene (68.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **15**.

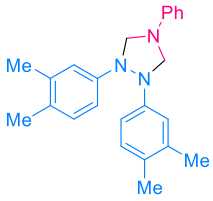
White solid, m.p. 135–136 ℃; yield: 74.4 mg (81%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37–7.35 (m, 2H), 7.29–7.24 (m, 2H), 7.19–7.10 (m, 4H), 7.08–7.03 (m, 2H), 6.84 (t, J = 7.2 Hz, 1H), 6.61 (d, J = 8.0 Hz, 2H), 4.80 (s, 2H), 4.68 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 151.2, 144.7, 130.5, 129.4, 124.8, 123.3, 119.0, 118.0, 113.6, 113.5, 67.3.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{18}Br_2N_3^+$: 459.9842; found: 459.9844.

14) 1,2-Bis(3,4-dimethylphenyl)-4-phenyl-1,2,4-triazolidine (16)



Prepared according to the general procedure from (*E*)-1,2-bis(3,4-dimethylphenyl)diazene (47.6 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product **16**.

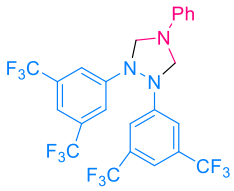
White solid, m.p. 145–147 ℃; yield: 58.6 mg (82%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26–7.21 (m, 2H), 7.07–7.01 (m, 4H), 6.96–6.93 (m, 2H), 6.77 (t, J = 7.2 Hz, 1H), 6.59 (d, J = 8.0 Hz, 2H), 4.84 (s, 2H), 4.65 (s, 2H), 2.26 (s, 6H), 2.21 (s, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 148.4, 145.3, 137.3, 130.2, 129.5, 129.3, 118.1, 116.5, 113.2, 112.5, 67.0, 20.1, 18.8.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{24}H_{28}N_3^+$: 358.2278; found: 358.2279.

15) 1,2-Bis(3,5-bis(trifluoromethyl)phenyl)-4-phenyl-1,2,4-triazolidine (17)



Prepared according to the general procedure from (*E*)-1,2-bis(3,5-bis(trifluoromethyl)phenyl)diazene (90.8 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 100:1) to afford the pure product **17**.

White solid, m.p. 136–137 ℃; yield: 58.5 mg (51%).

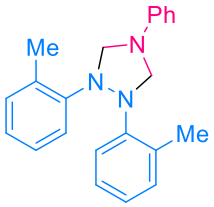
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60–7.56 (m, 4H), 7.56–7.51 (m, 2H), 7.33–7.27 (m, 2H), 6.90 (t, J = 7.6 Hz, 1H), 6.8 (d, J = 7.6 Hz, 2H), 4.90 (s, 2H), 4.87 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.9, 144.2, 133.0 (q, J = 33.0 Hz), 129.7, 124.1 (d, J = 271.5 Hz), 120.2, 116.1 (quint, J = 3.0 Hz), 115.0 (d, J = 4.5 Hz), 114.0, 68.3.

^{19}F NMR (376 MHz, $CDCl_3$): δ = -63.3.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{24}H_{16}F_{12}N_3^+$: 574.1147; found: 574.1150.

16) 4-Phenyl-1,2-di-*o*-tolyl-1,2,4-triazolidine (18)



Prepared according to the general procedure from (*E*)-1,2-di-*o*-tolyldiazene (42.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product **18**.

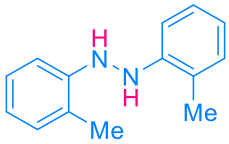
White solid, m.p. 80–81 ℃; yield: 19.8 mg (30%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58–7.55 (m, 2H), 7.30–7.25 (m, 2H), 7.24–7.16 (m, 4H), 7.08–7.00 (m, 2H), 6.83–6.78 (m, 1H), 6.59–6.55 (m, 2H), 4.96 (s, 2H), 4.57 (s, 2H), 2.42 (s, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.1, 144.7, 131.2, 129.8, 129.4, 126.3, 123.6, 117.9, 117.8, 112.8, 69.1, 19.2.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{22}H_{24}N_3^+$: 330.1965; found: 330.1965.

17) 1,2-Di-*o*-tolylhydrazine (19)[4]



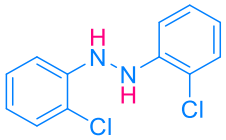
Prepared according to the general procedure from (*E*)-1,2-di-*o*-tolyldiazene (42.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product **19**.

White solid; yield: 22.5 mg (53%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15–7.10 (m, 4H), 6.96–6.89 (m, 2H), 6.87–6.75 (m, 2H), 5.54 (s, 2H), 2.29 (s, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 146.2, 130.4, 127.2, 121.1, 119.4, 111.0, 17.1.

18) 1,2-Bis(2-chlorophenyl)hydrazine (20)[4]



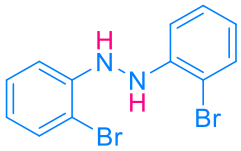
Prepared according to the general procedure from (*E*)-1,2-bis(2-chlorophenyl)diazene (50.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product **20**.

White solid; yield: 21.8 mg (43%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34–7.31 (m, 2H), 7.19–7.12 (m, 2H), 7.00–6.96 (m, 2H), 6.86–6.77 (m, 2H), 6.22 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 143.8, 129.3, 127.9, 120.2, 117.9, 112.8.

19) 1,2-Bis(2-bromophenyl)hydrazine (21)[4]



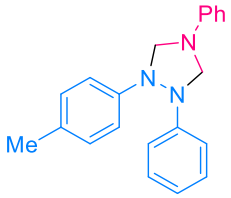
Prepared according to the general procedure from (*E*)-1,2-bis(2-bromophenyl)diazene (67.6 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on neutral alumina (PE: EtOAc = 200:1) to afford the pure product **21**.

White solid; yield: 26.7 mg (39%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48–7.45 (m, 2H), 7.20–7.15 (m, 2H), 6.96–6.91 (m, 2H), 6.77–6.70 (m, 2H), 6.23 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 144.7, 132.5, 128.6, 120.8, 113.1, 107.5.

20) **1,4-Diphenyl-2-(*p*-tolyl)-1,2,4-triazolidine (22)**[3]



Prepared according to the general procedure from (*E*)-1-phenyl-2-(*p*-tolyl)diazene (39.2 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **22**.

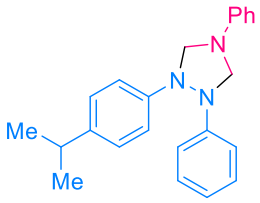
White solid, m.p. 106–107 ℃; yield: 58.7 mg (93%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.18 (m, 6H), 7.11–7.10 (m, 4H), 6.99–6.94 (m, 1H), 6.79 (t, J = 7.2 Hz, 1H), 6.61–6.56 (m, 2H), 4.89–4.63 (m, 4H), 2.30 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.1, 147.9, 145.1, 131.1, 129.7, 129.3, 129.1, 121.3, 118.2, 115.2, 114.8, 113.2, 67.3, 66.9, 20.5.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{22}N_3^+$: 316.1808; found: 316.1808.

21) 1-(4-Isopropylphenyl)-2,4-diphenyl-1,2,4-triazolidine (23)



Prepared according to the general procedure from (*E*)-1-(4-isopropylphenyl)-2-phenyldiazene (44.8 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **23**.

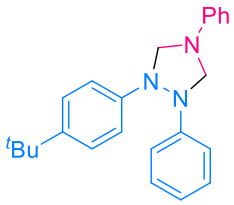
White solid, m.p. 100–101 ℃; yield: 61.8 mg (90%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31–7.12 (m, 10H), 6.97–6.92 (m, 1H), 6.80–6.75 (m, 1H), 6.61–6.56 (m, 2H), 4.94–4.58 (m, 4H), 2.90–2.81 (m, 1H), 1.22 (d, J = 6.8 Hz, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.2, 148.2, 145.1, 142.2, 129.3, 129.1, 127.0, 121.3, 118.2, 115.2, 114.9, 113.1, 67.3, 66.9, 33.3, 24.1.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H_{26}N_3^+$: 344.2121; found: 344.2121.

22) 1-(4-(*Tert*-butyl)phenyl)-2,4-diphenyl-1,2,4-triazolidine (24)



Prepared according to the general procedure from (*E*)-1-(4-(*tert*-butyl)phenyl)-2-phenyldiazene (47.6 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **24**.

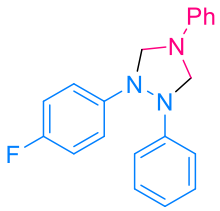
White solid, m.p. 121–122 ℃; yield: 65.1 mg (91%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37–7.20 (m, 8H), 7.18–7.13 (m, 2H), 7.00–6.95 (m, 1H), 6.81–6.77 (m, 1H), 6.63–6.56 (m, 2H), 4.97–4.58 (m, 4H), 1.32 (s, 9H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.2, 147.8, 145.1, 144.4, 129.3, 129.1, 126.0, 121.4, 118.2, 114.9, 114.8, 113.1, 67.2, 66.9, 34.1, 31.5.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{24}H_{28}N_3^+$: 358.2278; found: 358.2284.

23) 1-(4-Fluorophenyl)-2,4-diphenyl-1,2,4-triazolidine (25)[3]



Prepared according to the general procedure from (*E*)-1-(4-fluorophenyl)-2-phenyldiazene (40.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **25**.

White solid, m.p. 96–97 ℃; yield: 54.3 mg (85%).

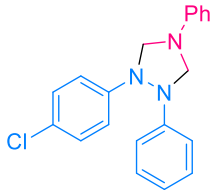
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37–7.24 (m, 4H), 7.23–7.13 (m, 4H), 7.03–6.98 (m, 3H), 6.86–6.80 (m, 1H), 6.64–6.60 (m, 2H), 4.89–4.61 (m, 4H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 158.3 (d, J = 238.5 Hz), 149.9, 146.5 (d, J = 1.5 Hz), 145.0, 129.3, 129.2, 121.6, 118.5, 116.6 (d, J = 7.5 Hz), 115.6 (d, J =22.5 Hz), 114.8, 113.3, 67.7, 67.2.

^{19}F NMR (376 MHz, $CDCl_3$): δ = -123.3 – -123.4.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}FN_3^+$: 320.1558; found: 320.1560.

24) 1-(4-Chlorophenyl)-2,4-diphenyl-1,2,4-triazolidine (26)[3]



Prepared according to the general procedure from (*E*)-1-(4-chlorophenyl)-2-phenyldiazene (43.3 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **26**.

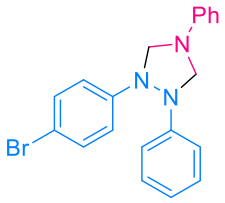
White solid, m.p. 120–121 ℃; yield: 58.4 mg (87%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36–7.30 (m, 2H), 7.30–7.24 (m, 4H), 7.21–7.16 (m, 2H), 7.16–7.11 (m, 2H), 7.04–6.97 (m, 1H), 6.86–6.80 (t, J = 7.4 Hz, 1H), 6.64–6.60 (m, 2H), 4.89–4.68 (m, 4H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 149.8, 148.7, 144.9, 129.4, 129.2, 129.0, 126.4, 121.8, 118.6, 116.2, 115.0, 113.3, 67.3, 67.1.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}ClN_3^+$: 336.1262; found: 336.1262.

25) 1-(4-Bromophenyl)-2,4-diphenyl-1,2,4-triazolidine (27)



Prepared according to the general procedure from (*E*)-1-(4-bromophenyl)-2-phenyldiazene (52.2 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **27**.

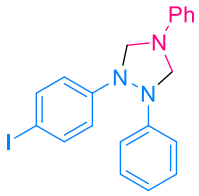
White solid, m.p. 155–156 ℃; yield: 63.1 mg (83%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42–7.35 (m, 2H), 7.33–7.22 (m, 4H), 7.17–7.13 (m, 2H), 7.09–7.03 (m, 2H), 7.02–6.95 (m, 1H), 6.84–6.79 (m, 1H), 6.63–6.59 (m, 2H), 4.75 (s, 4H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 149.8, 149.2, 144.9, 131.9, 129.4, 129.2, 121.8, 118.7, 116.6, 115.0, 113.7, 113.3, 67.3, 67.0.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}BrN_3^+$: 380.0757; found: 380.0760.

26) 1-(4-Iodophenyl)-2,4-diphenyl-1,2,4-triazolidine (28)



Prepared according to the general procedure from (*E*)-1-(4-iodophenyl)-2-phenyldiazene (61.6 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **28**.

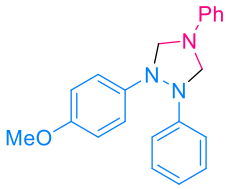
White solid, m.p. 178–179 ℃; yield: 70.1 mg (82%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61–7.50 (m, 2H), 7.33–7.22 (m, 4H), 7.14 (d, J = 8.0 Hz, 2H), 7.00–6.93 (m, 3H), 6.80 (t, J = 7.2 Hz, 1H), 6.60 (d, J = 8.0 Hz, 2H), 4.87–4.65 (m, 4H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 149.9, 149.8, 144.9, 137.9, 129.4, 129.2, 121.9, 118.7, 117.1, 115.1, 113.4, 83.6, 67.3, 66.9.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}IN_3^+$: 428.0618; found: 420.0619.

27) 1-(4-Methoxyphenyl)-2,4-diphenyl-1,2,4-triazolidine (29)



Prepared according to the general procedure from (*E*)-1-(4-methoxyphenyl)-2-phenyldiazene (42.4 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 50:1) to afford the pure product **29**.

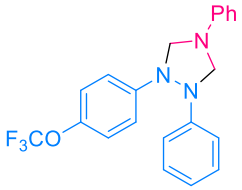
White solid, m.p. 92–93 ℃; yield: 58.3 mg (88%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33–7.28 (m, 2H), 7.25–7.14 (m, 6H), 6.98–6.93 (m, 1H), 6.88–6.83 (m, 2H), 6.81–6.76 (m, 1H), 6.62–6.57 (m, 2H), 4.86–4.77 (m, 3H), 4.59 (s, 1H), 3.78 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 155.1, 150.1, 145.1, 144.1, 129.3, 129.1, 121.2, 118.2, 117.0, 114.7, 114.5, 113.2, 67.8, 66.9, 55.6.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{22}ON_3^+$: 332.1757; found: 332.1761.

28) 1,4-Diphenyl-2-(4-(trifluoromethoxy)phenyl)-1,2,4-triazolidine (30)



Prepared according to the general procedure from (*E*)-1-phenyl-2-(4-(trifluoromethoxy)phenyl)diazene (53.2 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **30**.

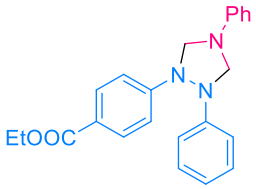
Colorless oil; yield: 62.4 mg (81%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34–7.29 (m, 2H), 7.28–7.23 (m, 2H), 7.21–7.15 (m, 6H), 7.02–6.97 (m, 1H), 7.85–6.79 (m, 1H), 6.64–6.59 (m, 2H), 4.89–4.72 (m, 4H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 149.9, 148.8, 144.9, 143.5 (d, J = 3.0 Hz), 129.3 (d, J = 12.0 Hz), 122.1, 121.9, 120.5 (d, J = 255.0 Hz), 118.7, 115.8, 115.0, 113.4, 67.4, 67.3.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{19}F_3ON_3^+$: 386.1475; found: 386.1474.

29) Ethyl 4-(2,4-diphenyl-1,2,4-triazolidin-1-yl)benzoate (31)



Prepared according to the general procedure from (*E*)-4-(phenyldiazenyl)phenyl propionate (50.8 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **31**.

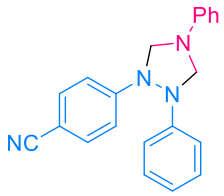
Colorless oil; yield: 59.8 mg (80%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, J = 8.8 Hz, 2H), 7.35–7.25 (m, 4H), 7.18–7.12 (m, 4H), 7.02 (t, J = 7.2 Hz, 1H), 6.83 (t, J = 7.2 Hz, 1H), 6.63 (d, J = 7.6 Hz, 2H), 4.97–4.85 (s, 3H), 4.60 (s, 1H), 4.36 (q, J = 7.2 Hz, 2H), 1.39 (t, J = 7.2 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 166.4, 153.0, 149.8, 144.9, 131.2, 129.4, 129.3, 122.8, 122.3, 118.8, 115.4, 113.4, 113.3, 67.8, 66.0, 60.5, 14.4.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H_{24}O_2N_3^+$: 374.1863; found: 374.1862.

30) 4-(2,4-Diphenyl-1,2,4-triazolidin-1-yl)benzonitrile (32)



Prepared according to the general procedure from (*E*)-4-(phenyldiazenyl)benzonitrile (41.4 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 100:1) to afford the pure product **32**.

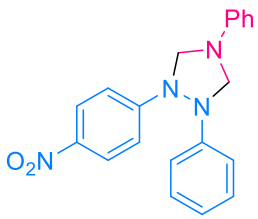
White solid, m.p. 143–144 ℃; yield: 45.0 mg (69%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58–7.53 (m, 2H), 7.35–7.30 (m, 2H), 7.29–7.25 (m, 2H), 7.16–7.11 (m, 4H), 7.07–7.02 (m, 1H), 6.89–6.82 (m, 1H), 6.66–6.62 (m, 2H), 5.02–4.94 (m, 1H), 4.89–4.81 (m, 2H), 4.57 (s, 1H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 152.4, 149.4, 144.7, 133.5, 129.4, 129.3, 122.7, 119.5, 119.2, 115.5, 113.9, 113.6, 103.1, 68.3, 65.8.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{19}N_4^+$: 327.1604; found: 327.1605.

31) 1-(4-Nitrophenyl)-2,4-diphenyl-1,2,4-triazolidine (33)



Prepared according to the general procedure from (*E*)-*1*-(4-nitrophenyl)-2-phenyldiazene (45.4 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 50:1) to afford the pure product **33**.

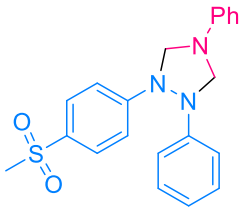
Yellow solid, m.p. 130–131 ℃; yield: 53.3 mg (77%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20–8.16 (m, 2H), 7.36–7.26 (m, 4H), 7.15–7.11 (m, 2H), 7.10–7.04 (m, 3H), 6.87 (t, *J* = 7.4 Hz, 1H), 6.66 (d, *J* = 8.0 Hz, 2H), 4.99 (s, 2H), 4.84 (s, 1H), 4.57 (s, 1H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 153.6, 149.3, 144.6, 140.9, 129.5, 129.4, 125.8, 123.1, 119.4, 115.8, 113.7, 112.7, 68.8, 65.6.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}O_2N_4^+$: 347.1503; found: 347.1505.

32) 1-(4-(Methylsulfonyl)phenyl)-2,4-diphenyl-1,2,4-triazolidine (34)



Prepared according to the general procedure from (*E*)-1-(4-(methylsulfonyl)phenyl)-2-phenyldiazene (52.0 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 50:1) to afford the pure product **34**.

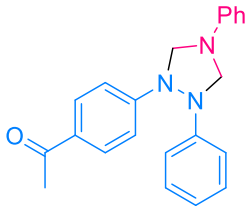
White solid, m.p. 183–184 ℃; yield: 39.7 mg (76%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.8 Hz, 2H), 7.26–7.22 (m, 2H), 7.21–7.15 (m, 2H), 7.14–7.10 (m, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 6.96 (t, *J* = 7.4 Hz, 1H), 6.76 (t, *J* = 7.2 Hz, 1H), 6.55 (d, *J* = 8.0 Hz, 2H), 4.91–4.76 (m, 3H), 4.50 (s, 1H), 2.94 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 153.4, 149.4, 144.7, 131.7, 129.5, 129.3, 129.1, 122.7, 119.1, 115.6, 113.7, 113.6, 68.3, 66.0, 44.9.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{22}O_2SN_3^+$: 380.1427; found: 380.1434.

33) 1-(4-(2,4-Diphenyl-1,2,4-triazolidin-1-yl)phenyl)ethan-1-one (35)



Prepared according to the general procedure from (*E*)-1-(4-(phenyldiazenyl)phenyl)ethan-1-one (44.8 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 100:1) to afford the pure product **35**.

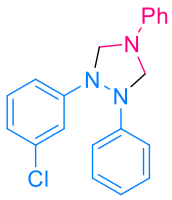
White solid, m.p. 117–118 ℃; yield: 43.27 mg (63%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95–7.90 (m, 2H), 7.36–7.29 (m, 2H), 7.28–7.23 (m, 2H), 7.18–7.11 (m, 4H), 7.02 (s, 1H), 6.83 (t, J = 7.2 Hz, 1H), 6.63 (d, J = 8.0 Hz, 2H), 4.98 (s, 1H), 4.87 (s, 2H), 4.59 (s, 1H), 2.55 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 196.6, 153.1, 149.7, 144.8, 130.3, 130.1, 129.4, 129.3, 122.4, 118.9, 115.5, 113.5, 113.2, 68.0, 65.9, 26.2.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}FN_3^+$: 344.1757; found: 344.1754.

34) 1-(3-Chlorophenyl)-2,4-diphenyl-1,2,4-triazolidine (36)



Prepared according to the general procedure from (*E*)-1-(3-chlorophenyl)-2-phenyldiazene (43.3 mg, 0.2 mmol) and *N*-phenyl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **36**.

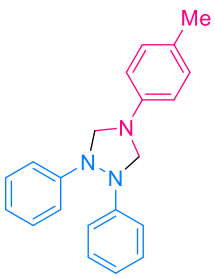
White solid, m.p. 95–96 ℃; yield: 54.4 mg (81%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35–7.22 (m, 6H), 7.21–7.17 (m, 2H), 7.05–6.99 (m, 2H), 6.97–6.92 (m, 1H), 6.83 (t, J = 7.2 Hz, 1H), 6.62 (d, J = 8.0 Hz, 2H), 4.90–4.67 (m, 4H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 151.3, 149.8, 144.9, 135.1, 130.2, 129.4, 129.2, 122.0, 121.4, 118.7, 115.1, 114.9, 113.3, 113.0, 67.4, 66.9.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}ClN_3^+$: 336.1262; found: 336.1263.

35) 1,2-Diphenyl-4-(*p*-tolyl)-1,2,4-triazolidine (37)



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and *p*-tolylglycine (82.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **37**.

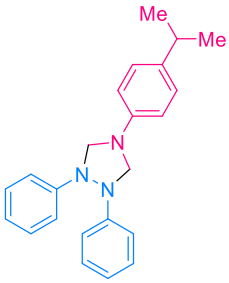
White solid, m.p. 94–95 ℃; yield: 56.1 mg (89%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.27 (m, 4H), 7.20–7.16 (m, 4H), 7.07–7.03 (m, 2H), 6.99–6.94 (m, 2H), 6.56–6.52 (m, 2H), 4.85 (s, 2H), 4.68 (s, 2H), 2.25 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.2, 143.1, 129.8, 129.1, 127.8, 121.4, 114.9, 113.5, 67.6, 20.4.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{22}N_3^+$: 316.1808; found: 316.1806.

36) 4-(4-Isopropylphenyl)-1,2-diphenyl-1,2,4-triazolidine (38)



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (4-isopropylphenyl)glycine (96.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **38**.

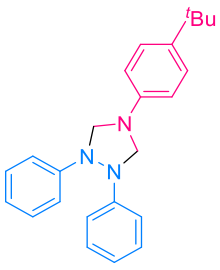
White solid, m.p. 88–90 ℃; yield: 57.7 mg (84%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36–7.29 (m, 4H), 7.24–7.19 (m, 4H), 7.16–7.12 (m, 2H), 7.01–6.96 (m, 2H), 6.62–6.57 (m, 2H), 4.89 (s, 2H), 4.71 (s, 2H), 2.90–280 (m, 1H), 1.24 (d, J = 7.2 Hz, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.2, 143.3, 139.0, 129.2, 127.3, 121.5, 115.0, 113.4, 67.5, 33.2, 24.2.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{23}H_{26}N_3^+$: 344.2121; found: 344.2122.

37) 4-(4-(*Tert*-butyl)phenyl)-1,2-diphenyl-1,2,4-triazolidine (39)



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and

(4-(*tert*-butyl)phenyl)glycinee (103.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **39**.

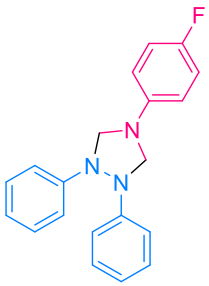
Colorless oil; yield: 59.3 mg (83%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30–7.25 (m, 6H), 7.18–7.15 (m, 4H), 6.97–6.92 (m, 2H), 6.57–6.54 (m, 2H), 4.85 (s, 2H), 4.68 (s, 2H), 1.26 (s, 9H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.2, 142.8, 141.2, 129.1, 126.1, 121.4, 115.0, 113.0, 67.3, 33.9, 31.4.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{24}H_{28}N_3^+$: 358.2278; found: 358.2277.

38) 4-(4-Fluorophenyl)-1,2-diphenyl-1,2,4-triazolidine (40)



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (4-fluorophenyl)glycine (84.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **40**.

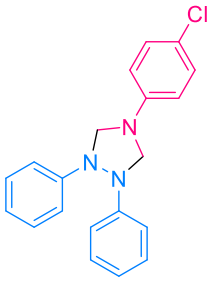
White solid, m.p. 114–115 ℃; yield: 53.7 mg (84%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33–7.27 (m, 4H), 7.20–7.16 (m, 4H), 7.01–6.92 (m, 4H), 6.57–6.52 (m, 2H), 4.82 (s, 2H), 4.68 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 156.5 (d, J = 235.5 Hz), 150.1, 141.9 (d, J = 1.5 Hz), 129.2, 121.6, 115.8 (d, J = 22.5 Hz), 114.5 (d, J = 7.5 Hz), 114.4, 67.9.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}FN_3^+$: 320.1558; found: 320.1555.

39) 4-(4-Chlorophenyl)-1,2-diphenyl-1,2,4-triazolidine (41)



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and

(4-chlorophenyl)glycine (92.8 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **41**.

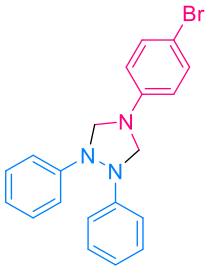
White solid, m.p. 132–133 ℃; yield: 57.1 mg (85%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 (t, J = 7.8 Hz, 4H), 7.22–7.15 (m, 6H), 7.00–6.95 (m, 2H), 6.52–6.48 (m, 2H), 4.83 (s, 2H), 4.66 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 149.9, 143.6, 129.21, 129.19, 123.2, 121.7, 115.0, 114.3, 67.1.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}ClN_3^+$: 336.1262; found: 336.1258.

40) 4-(4-Bromophenyl)-1,2-diphenyl-1,2,4-triazolidine (42)



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (4-bromophenyl)glycine (115.0 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **42**.

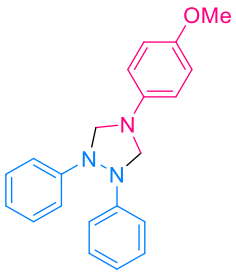
White solid, m.p. 137–138 ℃; yield: 62.4 mg (82%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33–7.28 (m, 6H), 7.20–7.15 (m, 4H), 7.01–6.95 (m, 2H), 6.49–6.42 (m, 2H), 4.83 (s, 2H), 4.65 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 149.9, 143.9, 132.1, 129.2, 121.7, 115.0, 114.7, 110.3, 67.0.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}BrN_3^+$: 380.0757; found: 380.0759.

41) 4-(4-Methoxyphenyl)-1,2-diphenyl-1,2,4-triazolidine (43)



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (4-methoxyphenyl)glycine (90.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on

silica gel (PE: EtOAc = 200:1) to afford the pure product **43**.

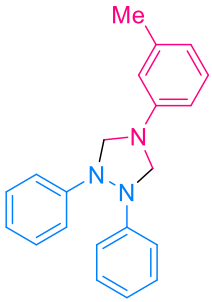
White solid, m.p. 70–71 ℃; yield: 47.1 mg (71%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30–7.26 (m, 4H), 7.17–7.11 (m, 4H), 6.98–6.91 (m, 2H), 6.83–6.78 (m, 2H), 6.63–6.58 (m, 2H), 4.79 (s, 2H), 4.67 (s, 2H), 3.74 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 152.9, 150.3, 140.0, 129.1, 121.3, 115.1, 114.9, 114.9, 68.4, 55.7.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{22}ON_3^+$: 332.1757; found: 332.1758.

42) 1,2-Diphenyl-4-(*m*-tolyl)-1,2,4-triazolidine (44)



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and *m*-tolylglycine (82.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **44**.

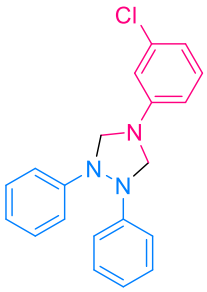
White solid, m.p. 118–119 ℃; yield: 47.9 mg (76%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32–7.26 (m, 4H), 7.20–7.16 (m, 4H), 7.15–7.08 (m, 1H), 6,98–6.92 (m, 2H), 6.63–6.58 (m, 1H), 6.43–6.39 (m, 2H), 4.86 (s, 2H), 4.68 (s, 2H), 2.30 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 150.1, 145.1, 139.2, 129.2, 129.1, 121.5, 119.3, 115.0, 114.0, 110.4, 67.1, 21.7.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{21}H_{22}N_3^+$: 316.1808; found: 316.1810.

43) 4-(3-Chlorophenyl)-1,2-diphenyl-1,2,4-triazolidine (45)



Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (3-chlorophenyl)glycine (92.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **45**.

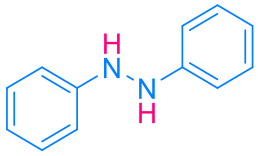
White solid, m.p. 99–101 ℃; yield: 49.0 mg (73%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33–7.28 (m, 4H), 7.20–7.17 (m, 4H), 7.13 (t, J = 8.0 Hz, 1H), 7.02–6.94 (m, 2H), 6.77–6.73 (m, 1H), 6.58–6.55 (m, 1H), 6.47–6.42 (m, 1H), 4.88–6.63 (m, 4H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 149.8, 145.8, 135.1, 130.3, 129.2, 121.8, 118.1, 115.0, 112.9, 111.2, 66.7.

HRMS (ESI): *m/z* $[M+H]^+$ calcd for $C_{20}H_{19}ClN_3^+$: 336.1262; found: 336.1263.

44) **1,2-Diphenylhydrazine (46)**[4]



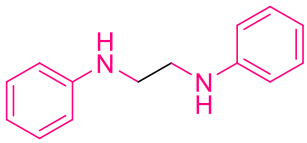
Prepared according to the general procedure from azobenzene (36.4 mg, 0.2 mmol) and (2-chlorophenyl)glycine (92.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 200:1) to afford the pure product **46**.

White solid; yield: 12.9 mg (35%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22–7.13 (m, 4H), 7.02–6.95 (m, 4H), 6.84–6.76 (m, 2H), 5.39 (s, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 147.7, 129.1, 120.0, 114.1.

45) N^1,N^2-Diphenylethane-1,2-diamine(47)[5]



Prepared according to the general procedure from *N*-aryl glycine (75.6 mg, 0.5 mmol, 2.5 equiv.). Purified by column chromatography on silica gel (PE: EtOAc = 50:1) to afford the pure product **47**.

White solid; yield: 7.9 mg (15%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34–7.28 (m, 4H), 6.87–6.74 (m, 2H), 6.72–6.65 (m, 4H), 4.67 (s, 2H), 3.66 (s, 4H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 146.4, 129.3, 117.6, 112.4, 46.5.

9. References

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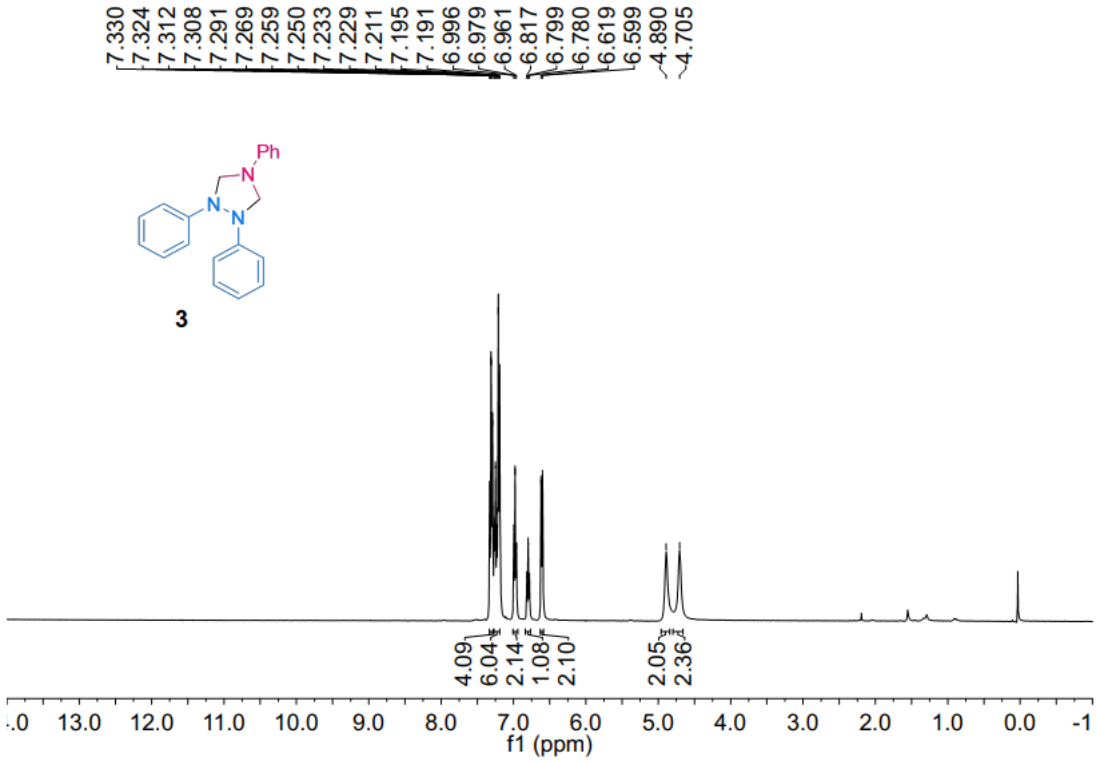
[3] Matsui M.; Shibara, K. *J. Jpn. Soc. Colour Mater. (SHIKIZAI)*, **2001**, *74*, 607.

[4] Song, M.; Zhou, H.; Wang, G.; Ma, B.; Jiang, Y.; Yang, J.; Huo, C.; Wang, X.-C. *J. Org. Chem*. **2021**, *86*, 4804.

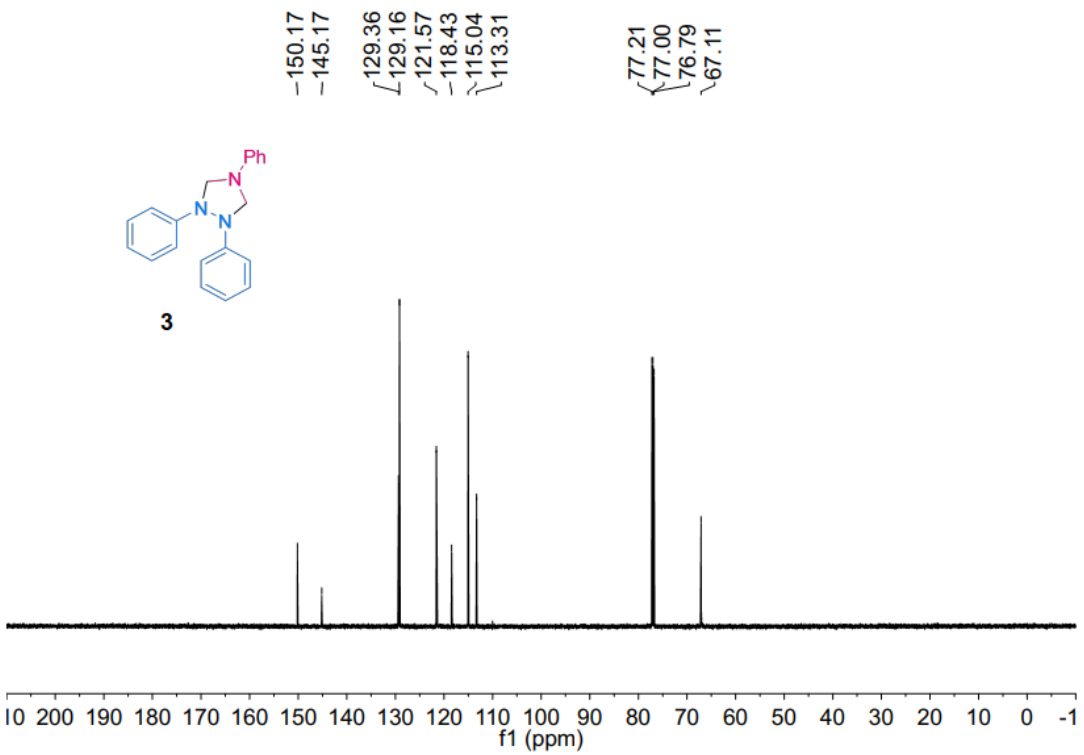
[5] Lavoie, C. M.; MacQueen, P. M.; Rotta-Loria, N. L.; Sawatzky, R. S.; Borzenko, A.; Chisholm, A. J.; Hargreaves, B. K. V.; McDonald, R.; Ferguson, M. J.; Stradiotto, M. *Nat. Commun*. **2016**, *7*, 11073.

10. Copy of NMR Spectra

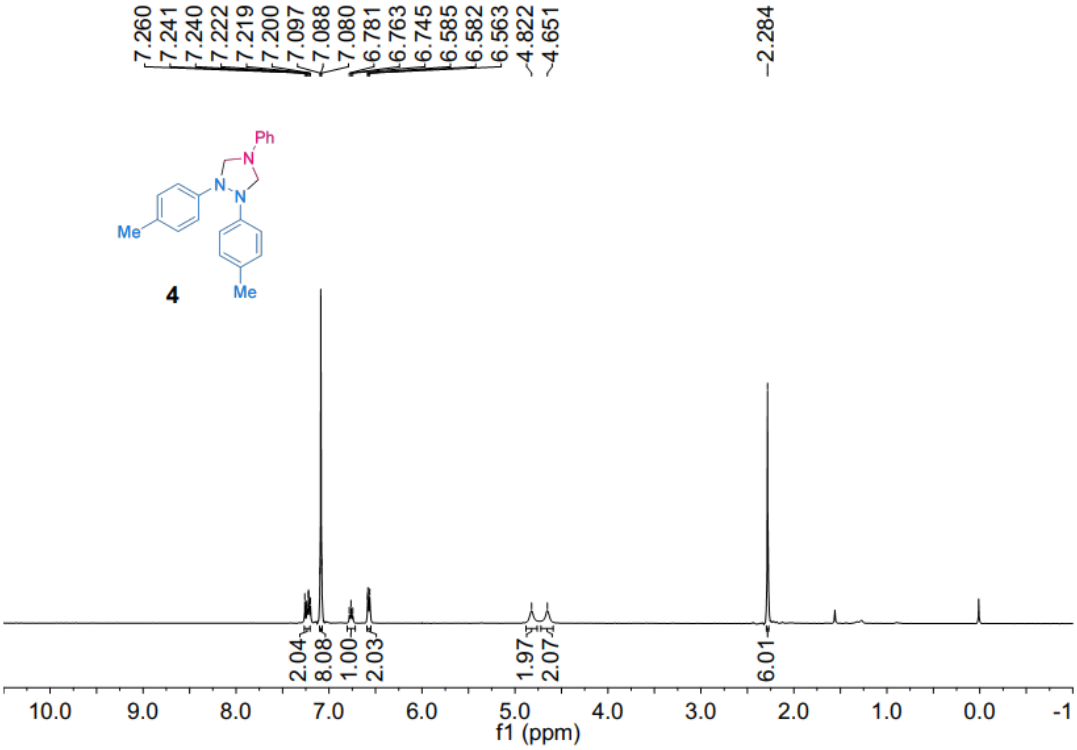
^{1}H NMR spectrum of **3** (400 MHz, $CDCl_3$):



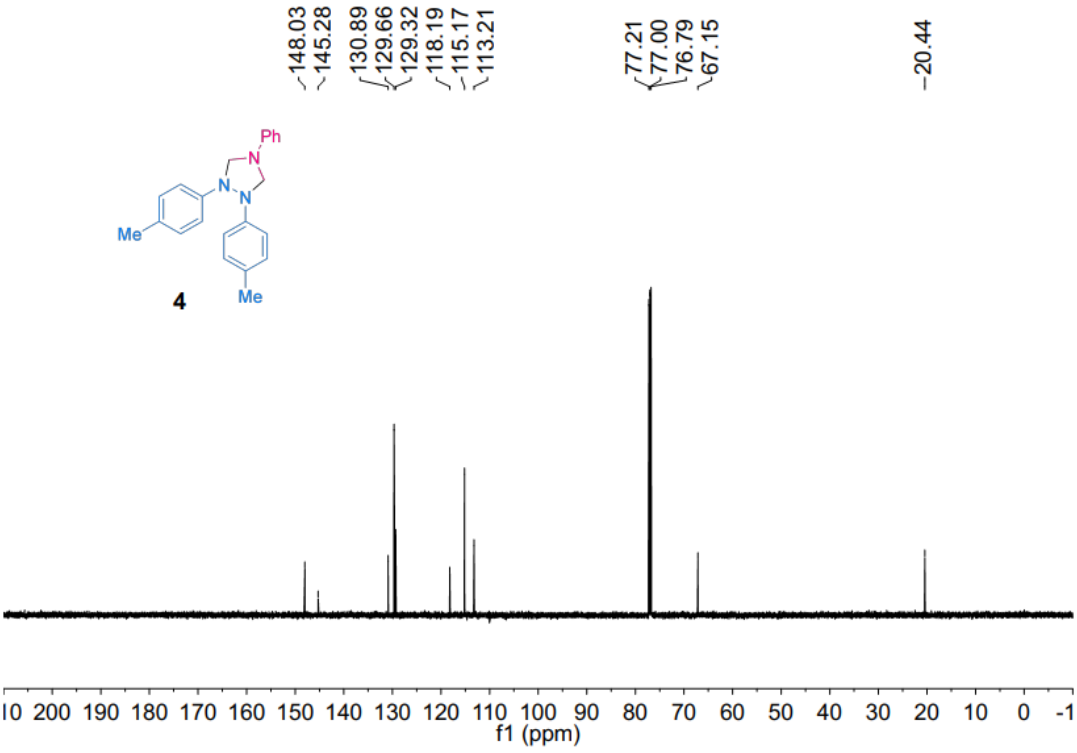
^{13}C NMR spectrum of **3** (150 MHz, $CDCl_3$):



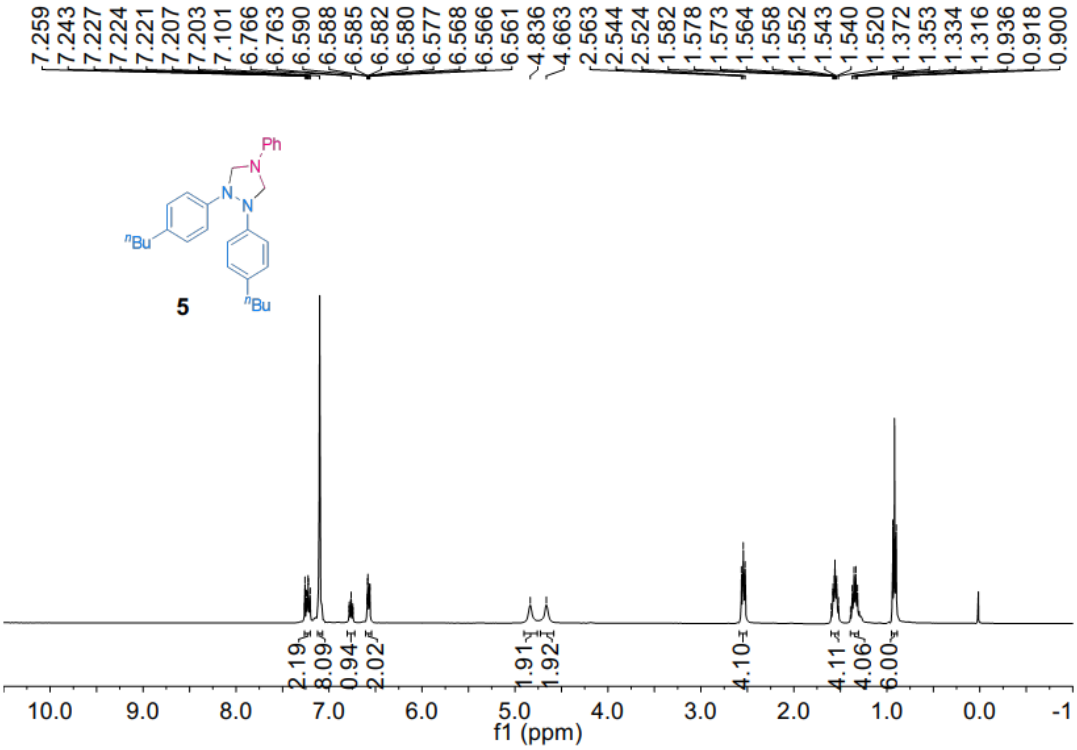
^{1}H NMR spectrum of **4** (400 MHz, $CDCl_3$):



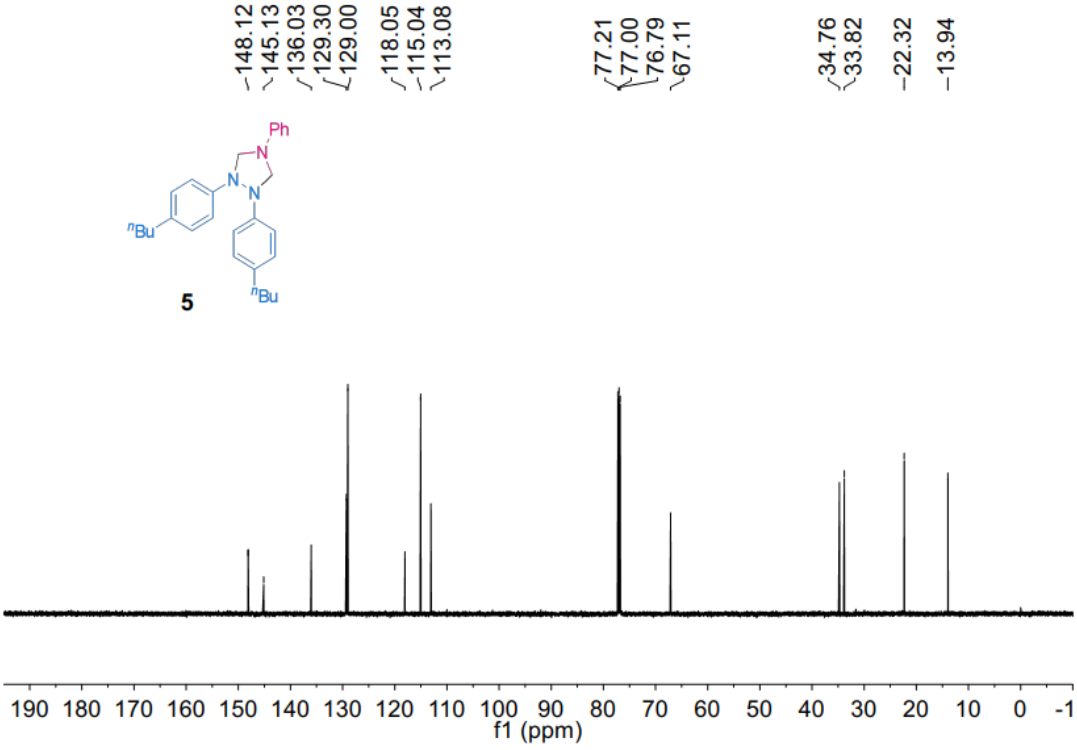
^{13}C NMR spectrum of **4** (150 MHz, $CDCl_3$):



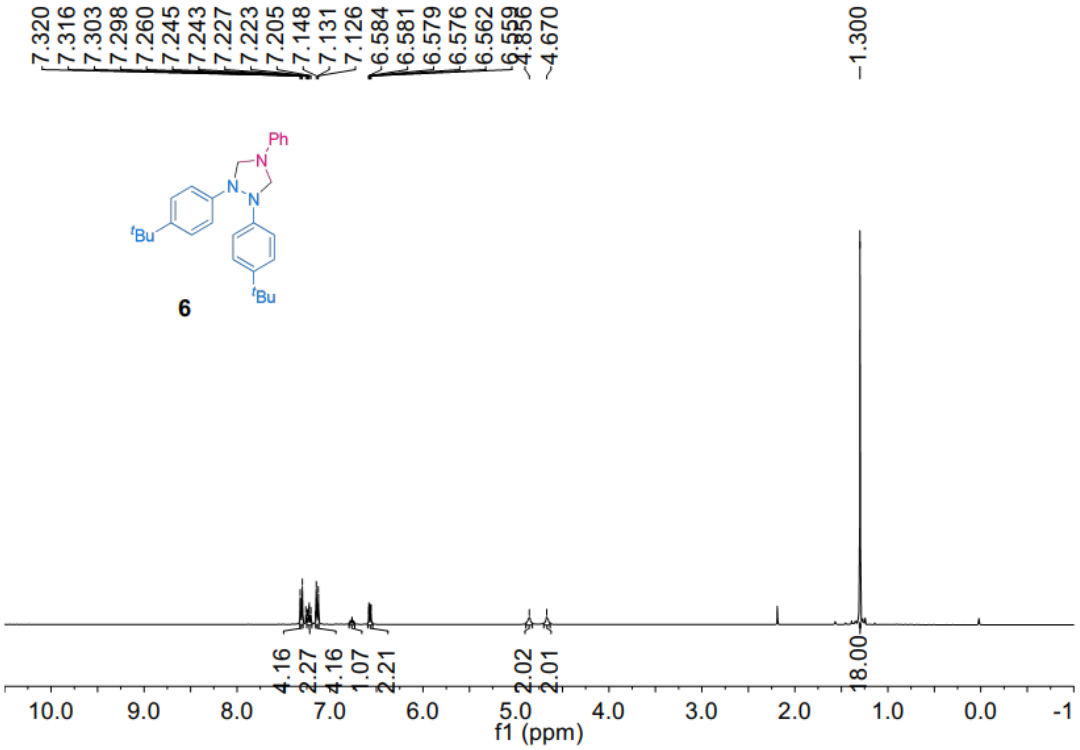
^{1}H NMR spectrum of **5** (400 MHz, $CDCl_3$):



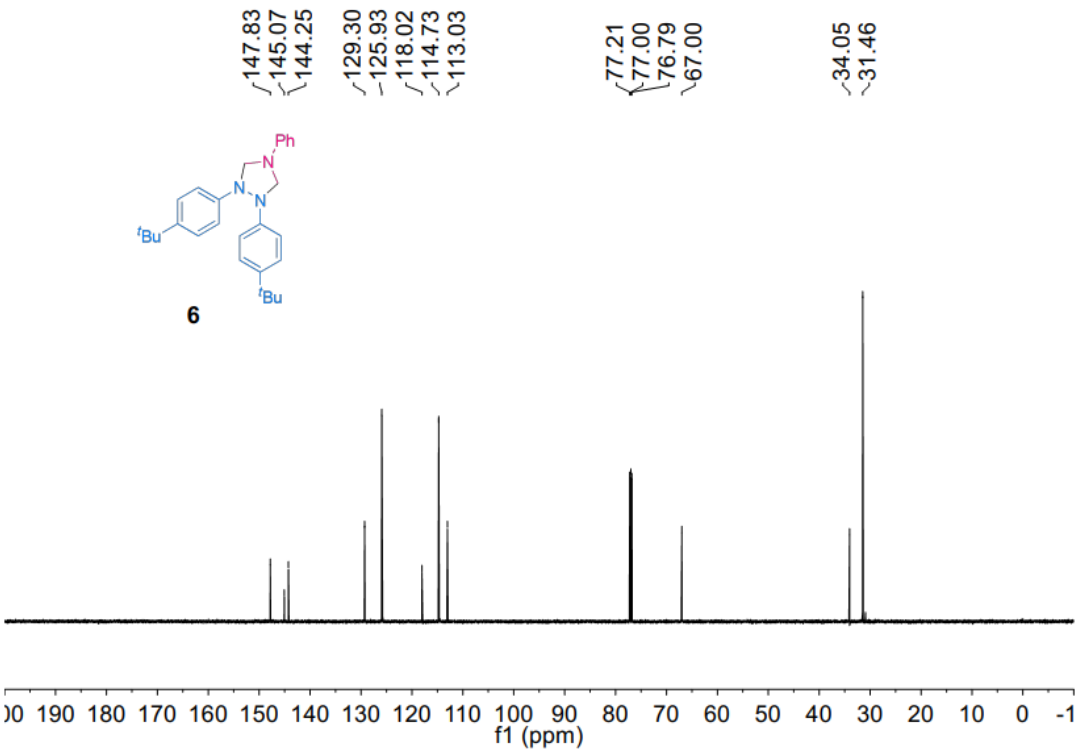
^{13}C NMR spectrum of **5** (150 MHz, $CDCl_3$):



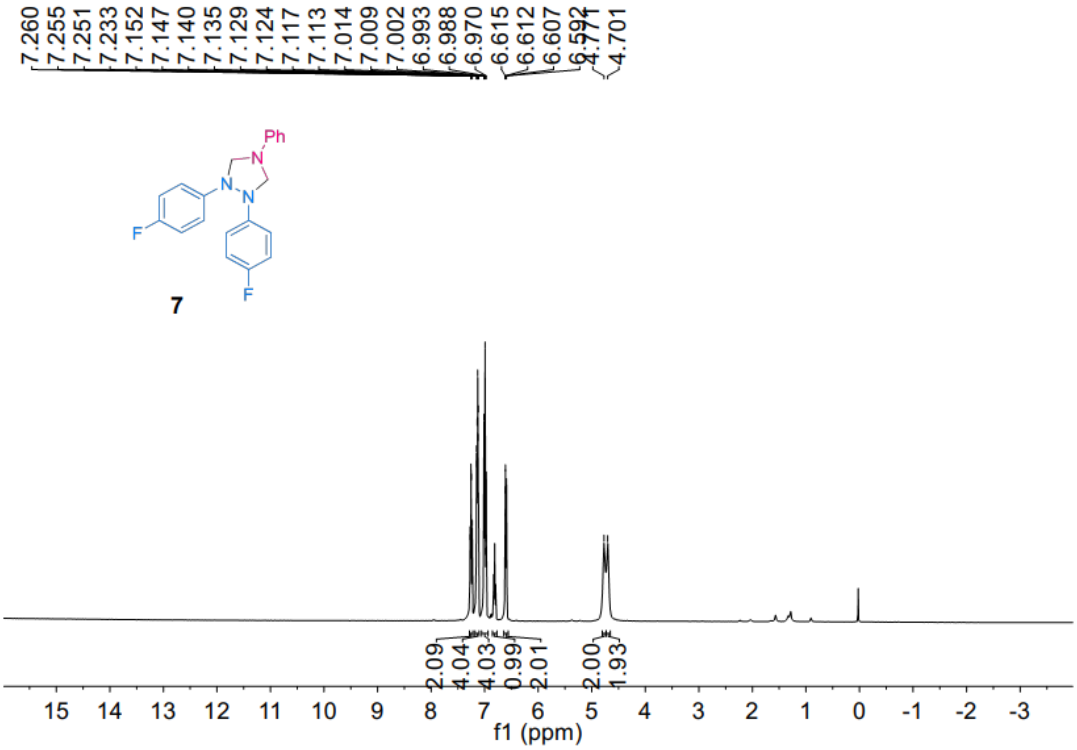
^{1}H NMR spectrum of **6** (400 MHz, $CDCl_3$):



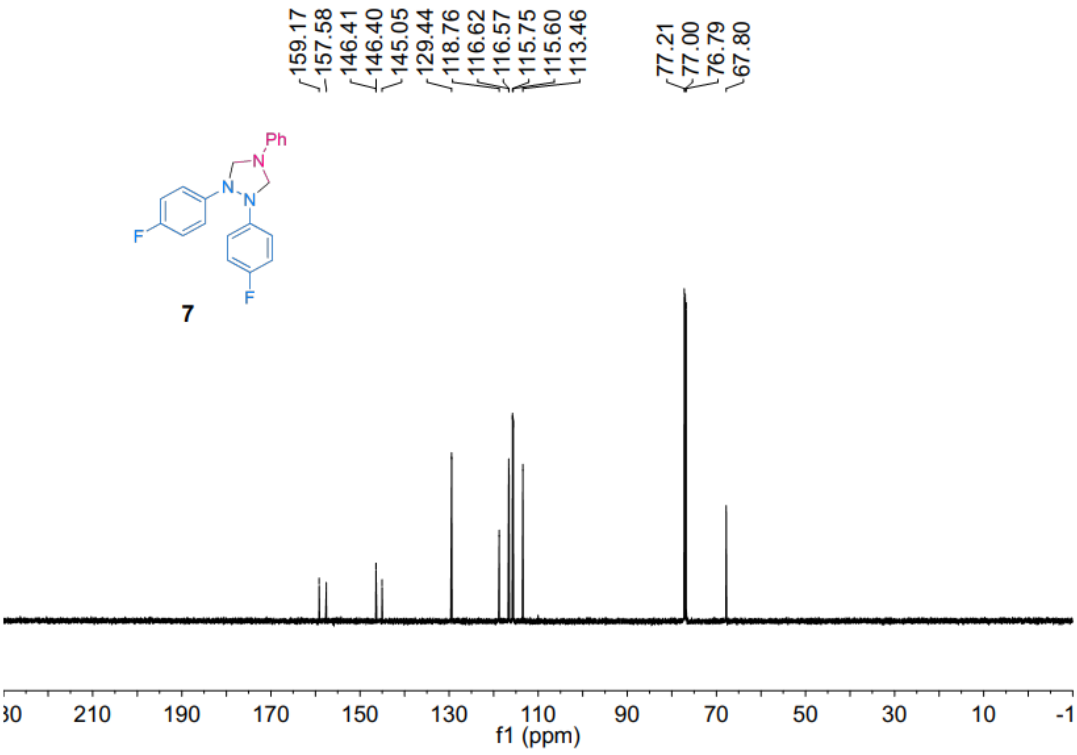
^{13}C NMR spectrum of **6** (150 MHz, $CDCl_3$):



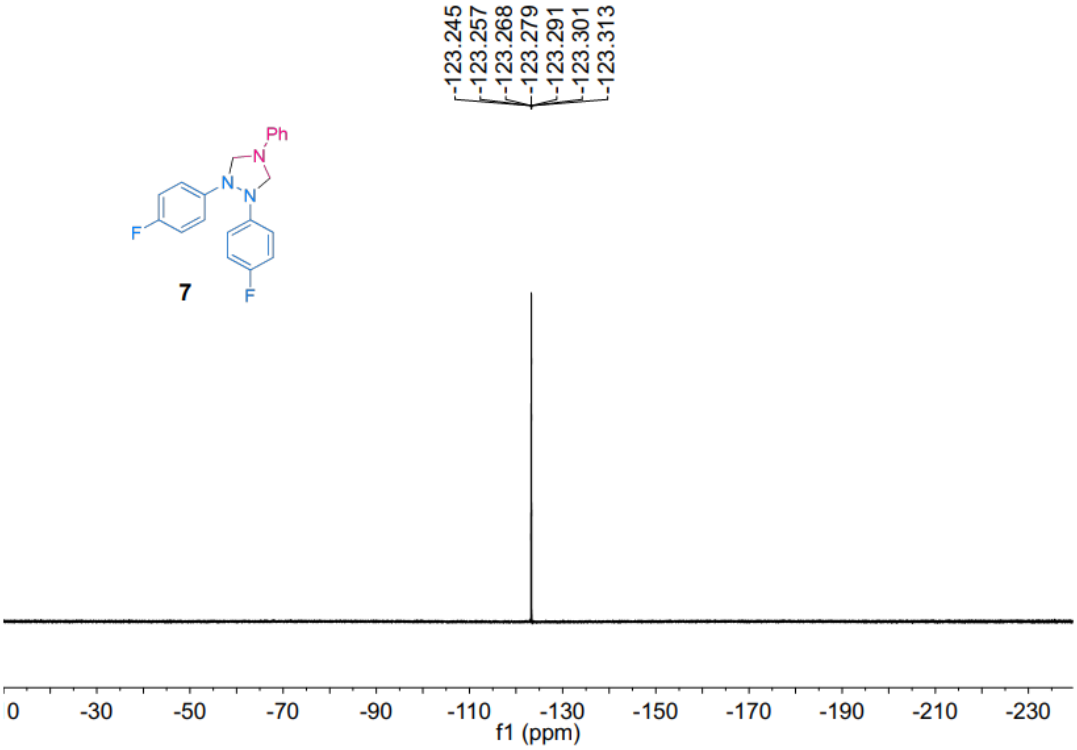
^{1}H NMR spectrum of **7** (400 MHz, $CDCl_3$):



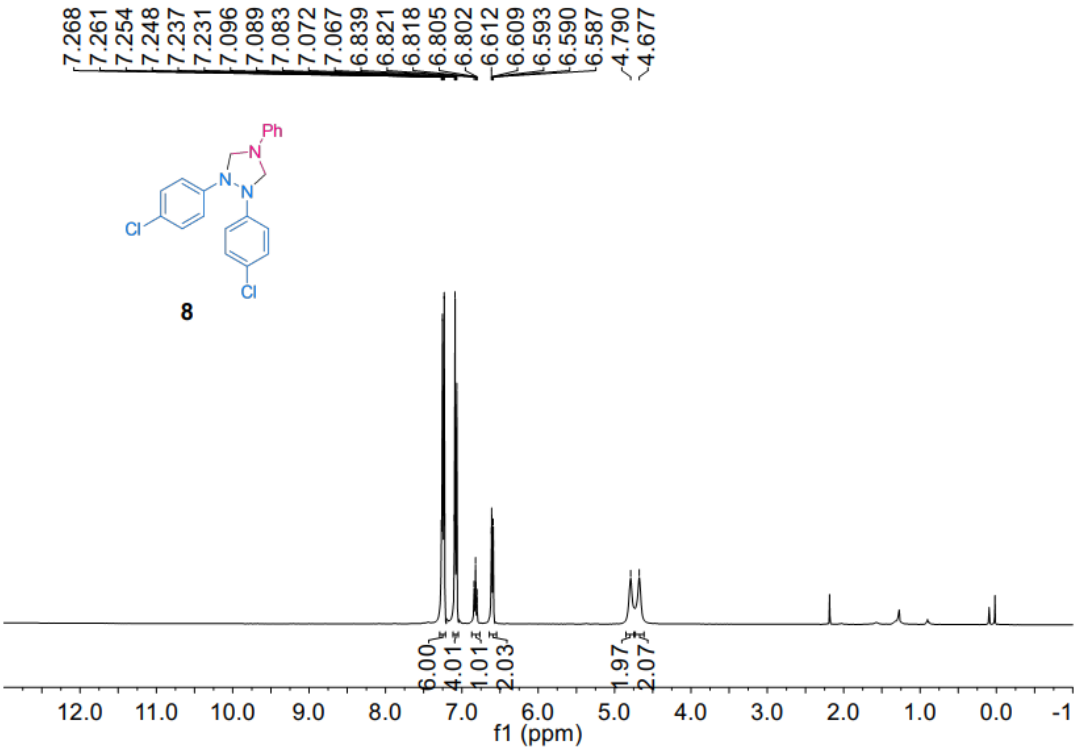
^{13}C NMR spectrum of **7** (150 MHz, $CDCl_3$):



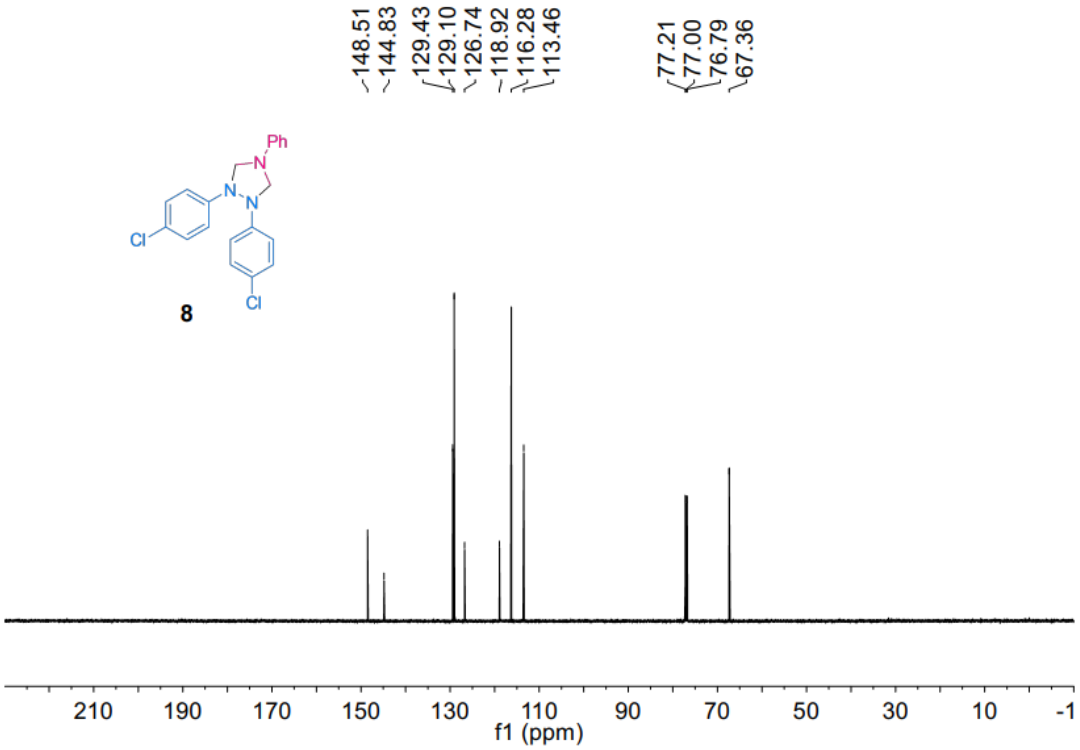
^{19}F NMR spectrum of **7** (376 MHz, $CDCl_3$):



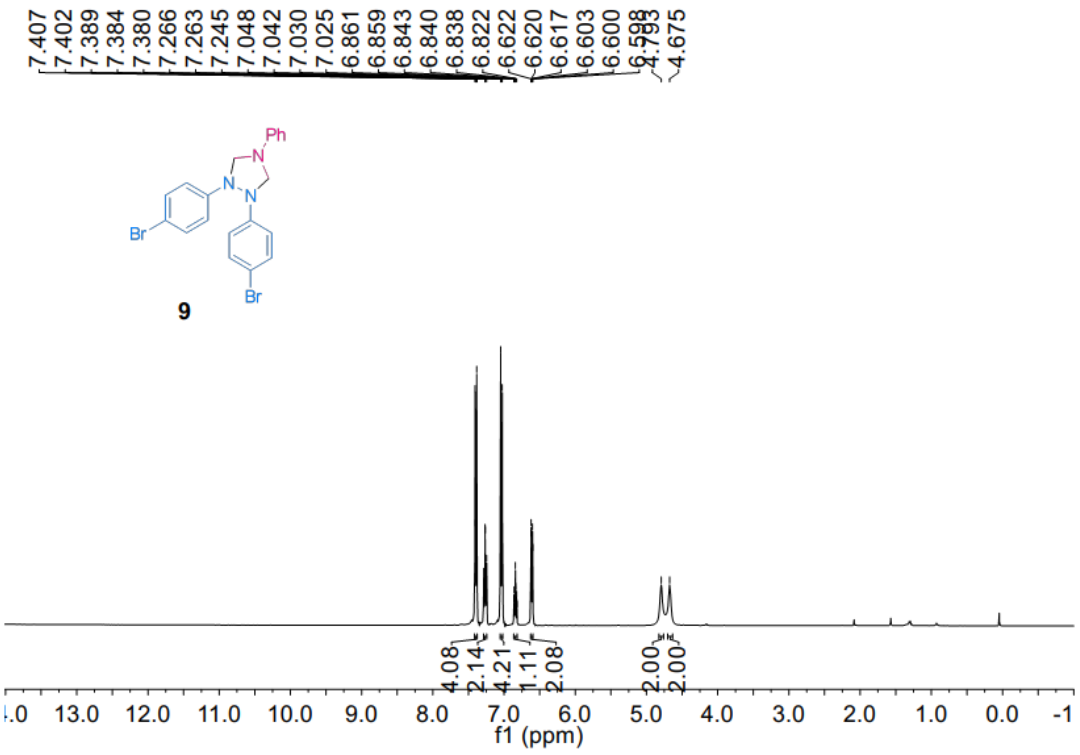
^{1}H NMR spectrum of **8** (400 MHz, $CDCl_3$):



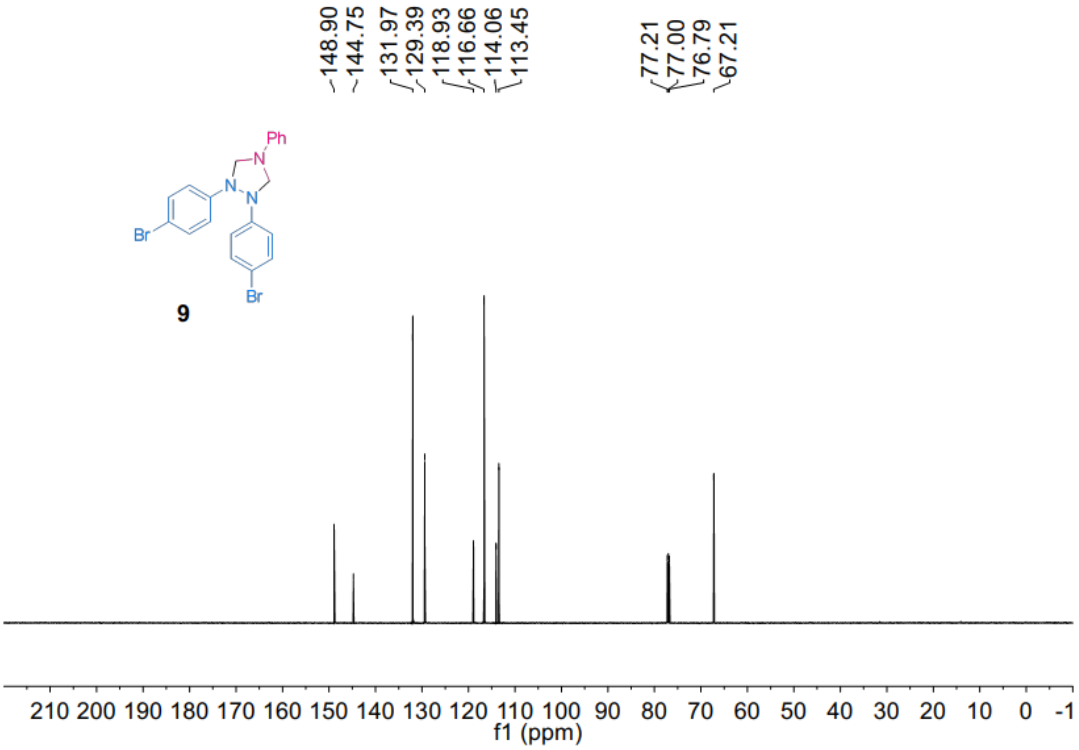
^{13}C NMR spectrum of **8** (150 MHz, $CDCl_3$):



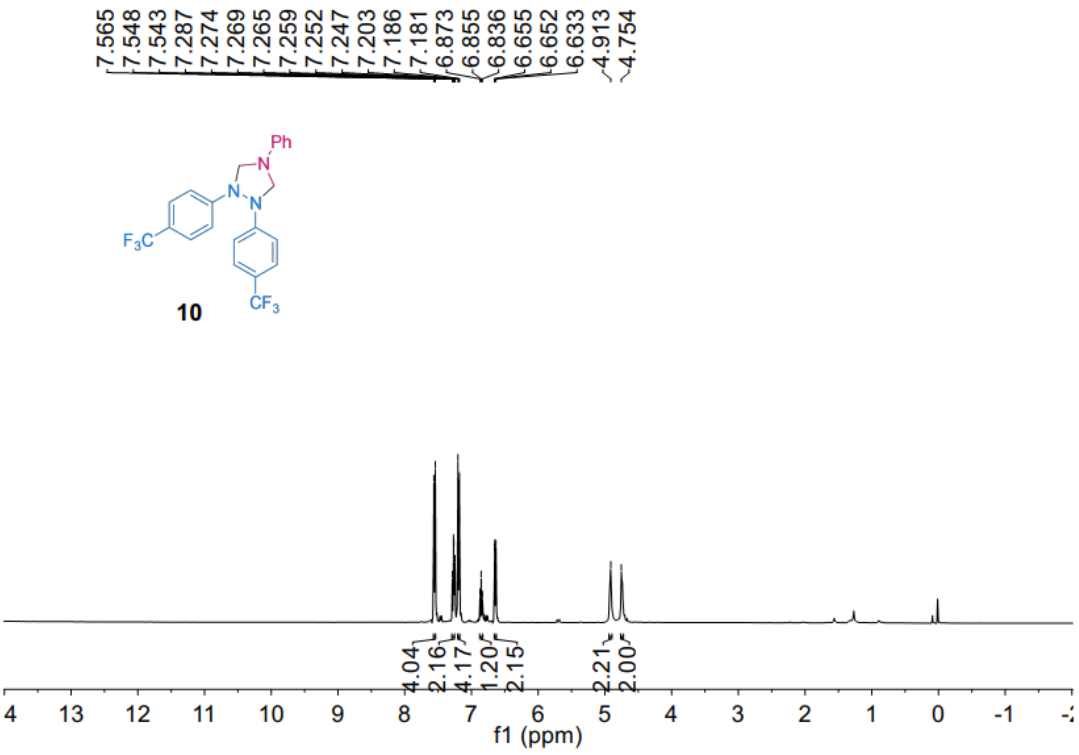
^{1}H NMR spectrum of **9** (400 MHz, $CDCl_3$):



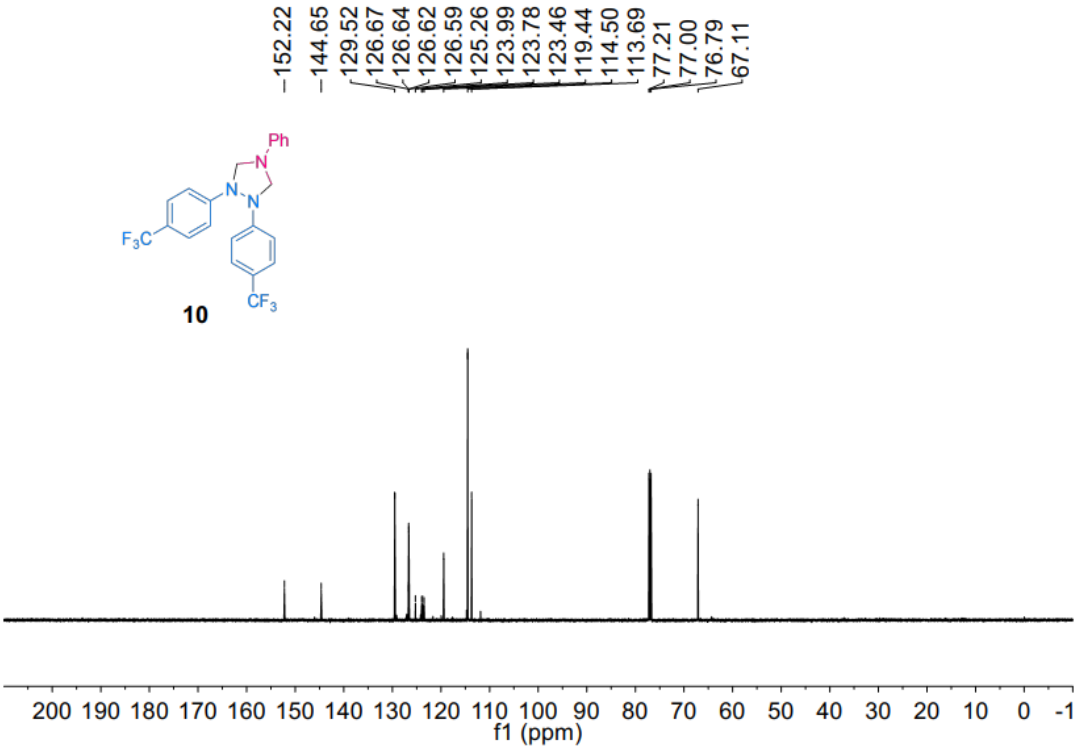
^{13}C NMR spectrum of **9** (150 MHz, $CDCl_3$):



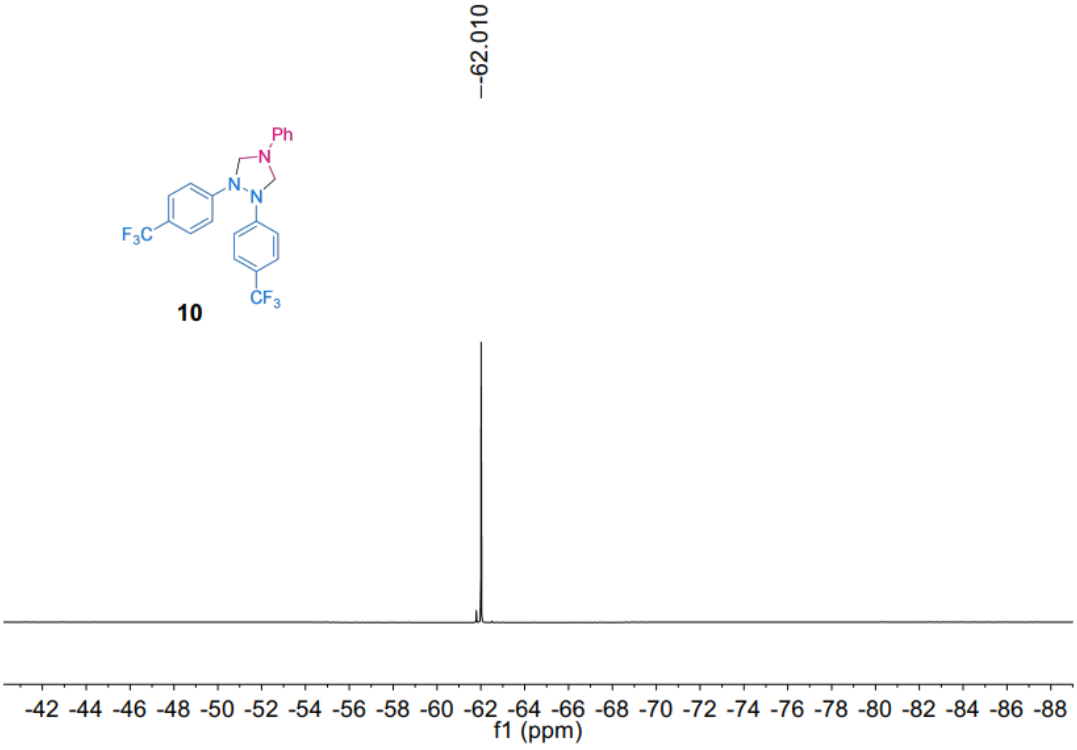
^{1}H NMR spectrum of **10** (400 MHz, $CDCl_3$):



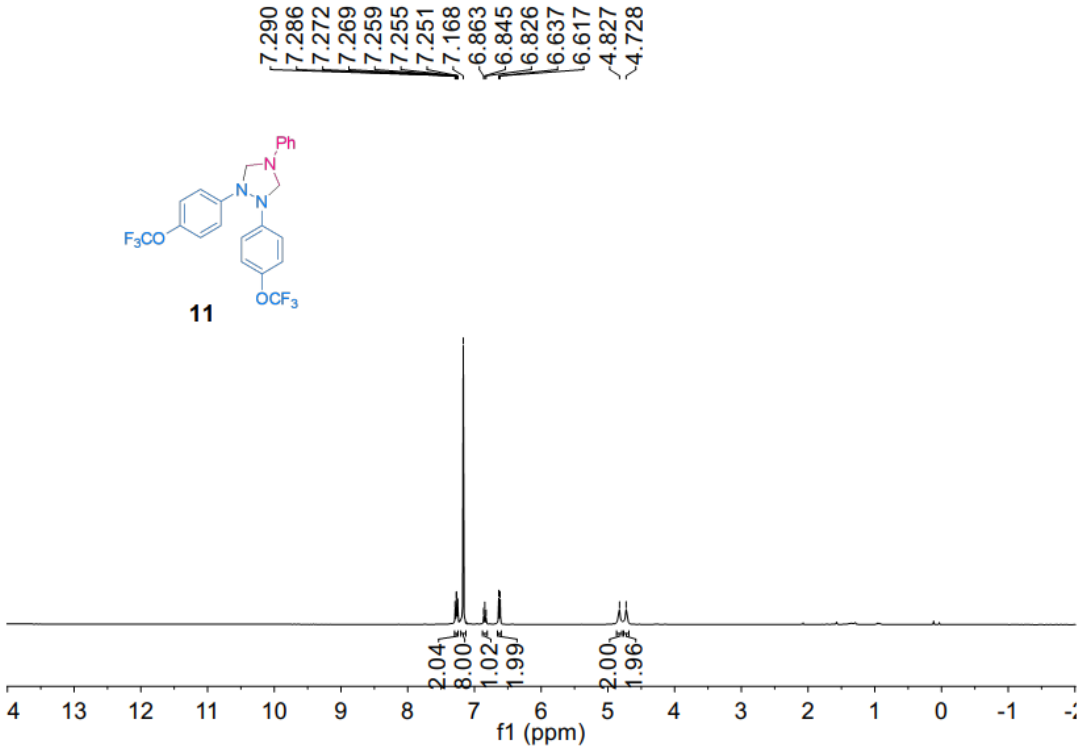
^{13}C NMR spectrum of **10** (150 MHz, $CDCl_3$):



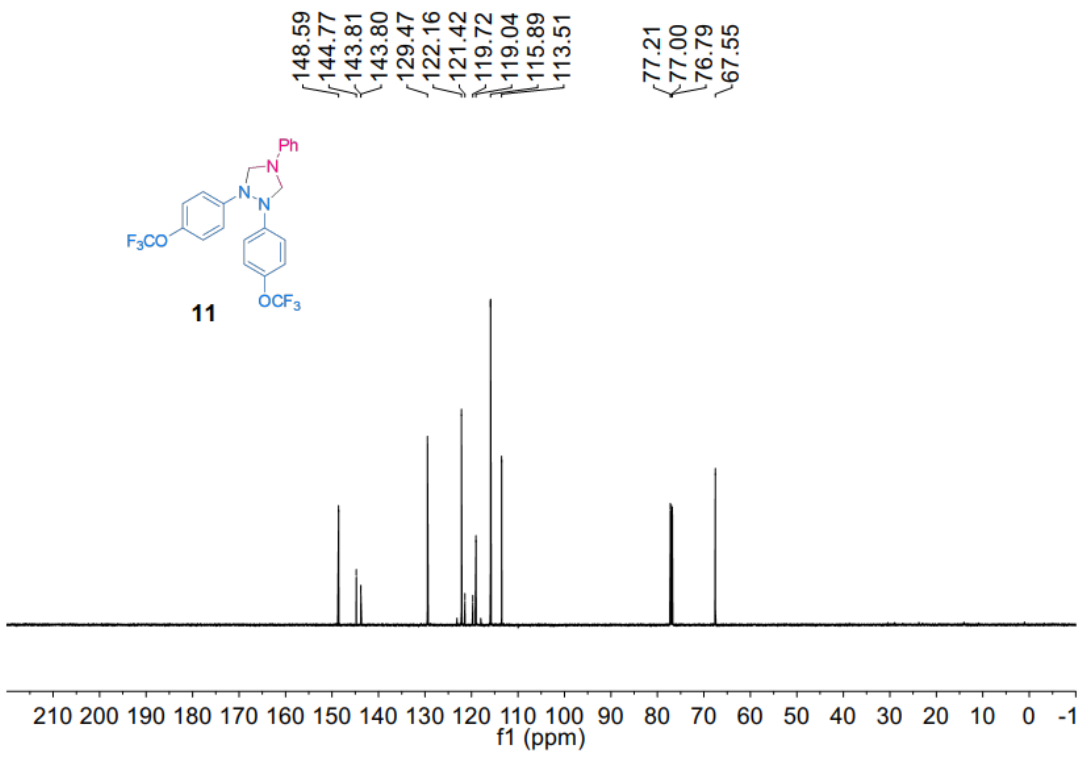
^{19}F NMR spectrum of **10** (376 MHz, $CDCl_3$):



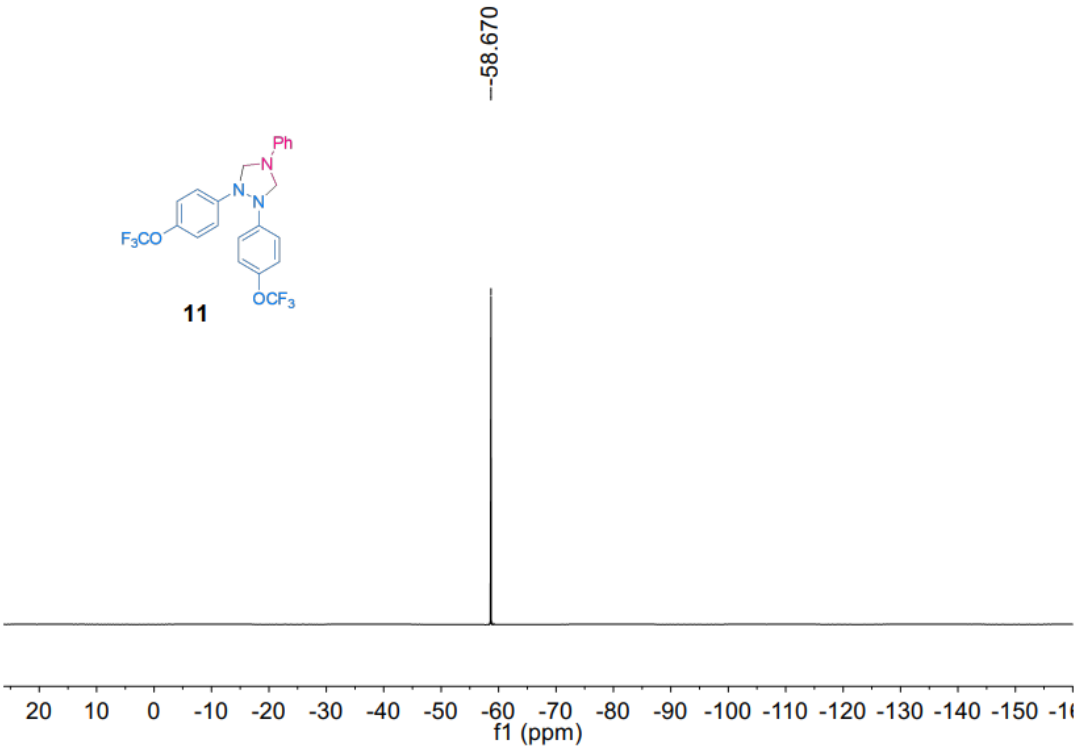
^{1}H NMR spectrum of **11** (400 MHz, $CDCl_3$):



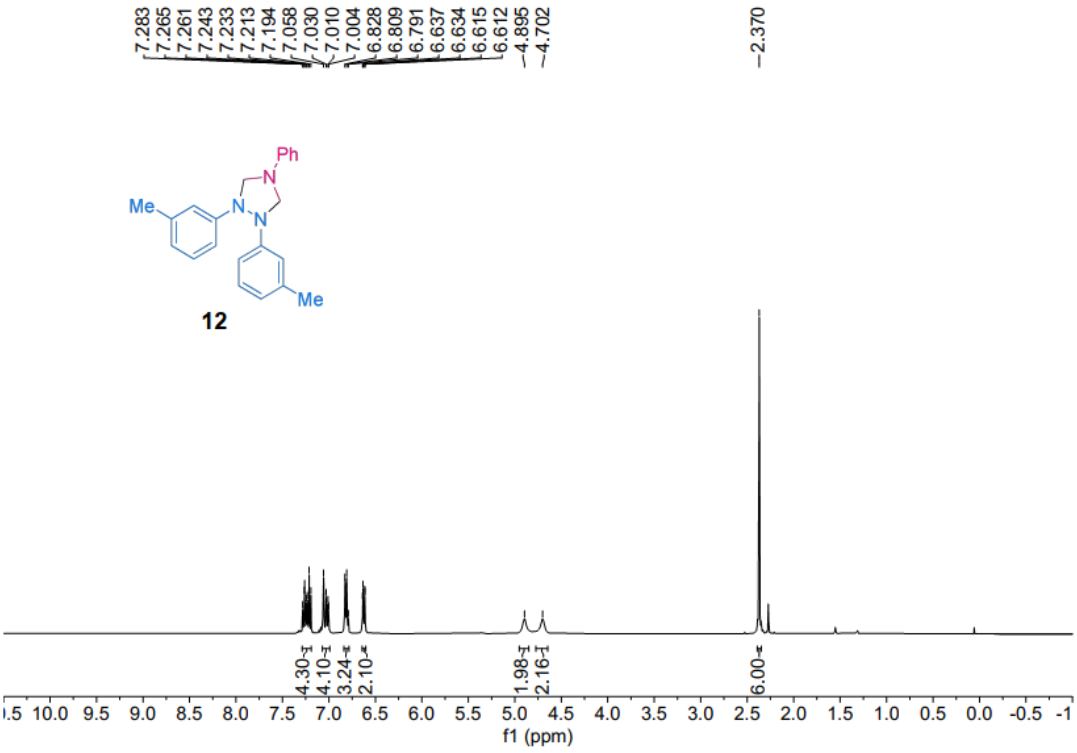
^{13}C NMR spectrum of **11** (150 MHz, $CDCl_3$):



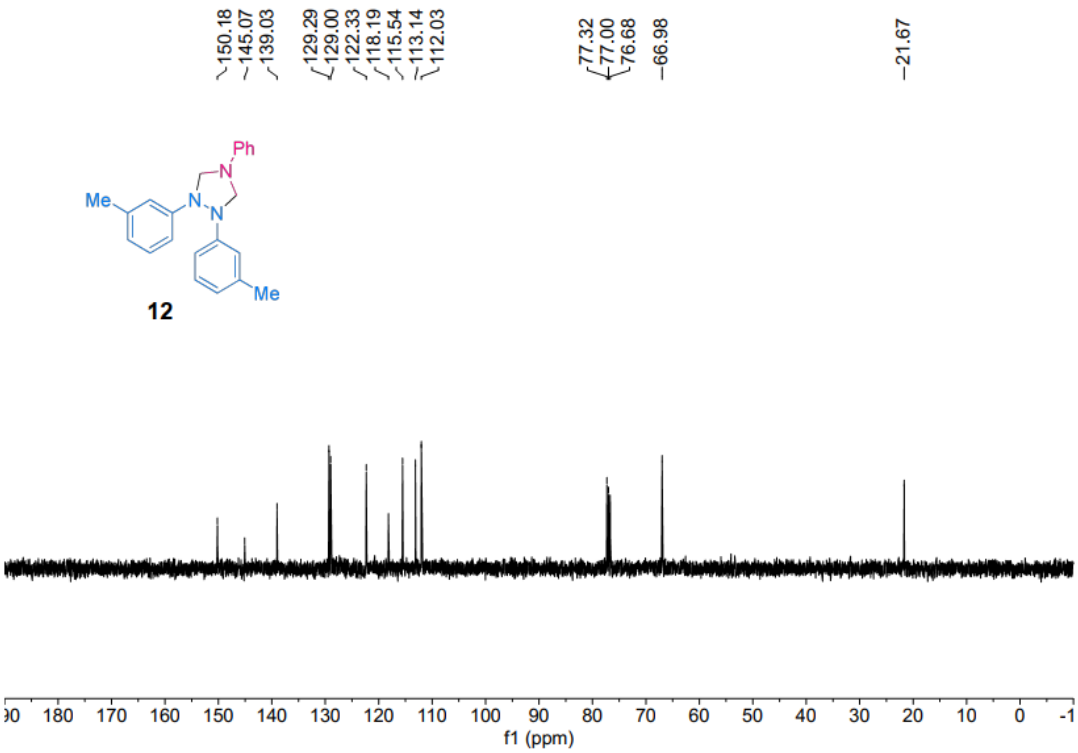
^{19}F NMR spectrum of **11** (376 MHz, $CDCl_3$):



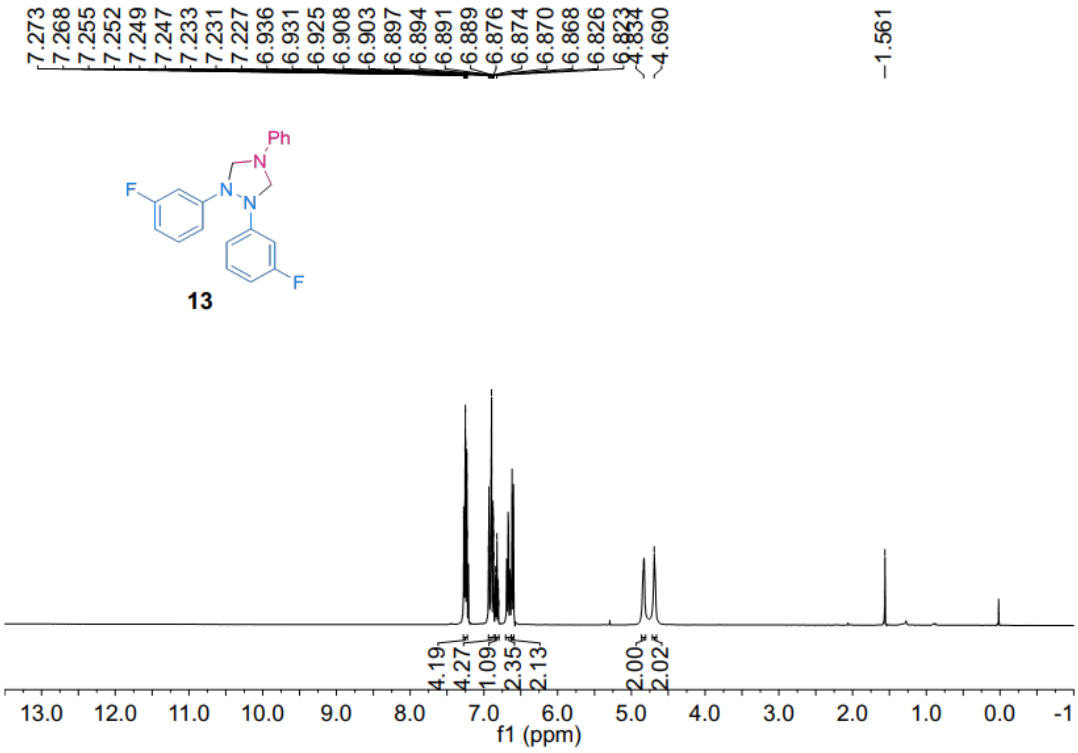
^{1}H NMR spectrum of **12** (400 MHz, $CDCl_3$):



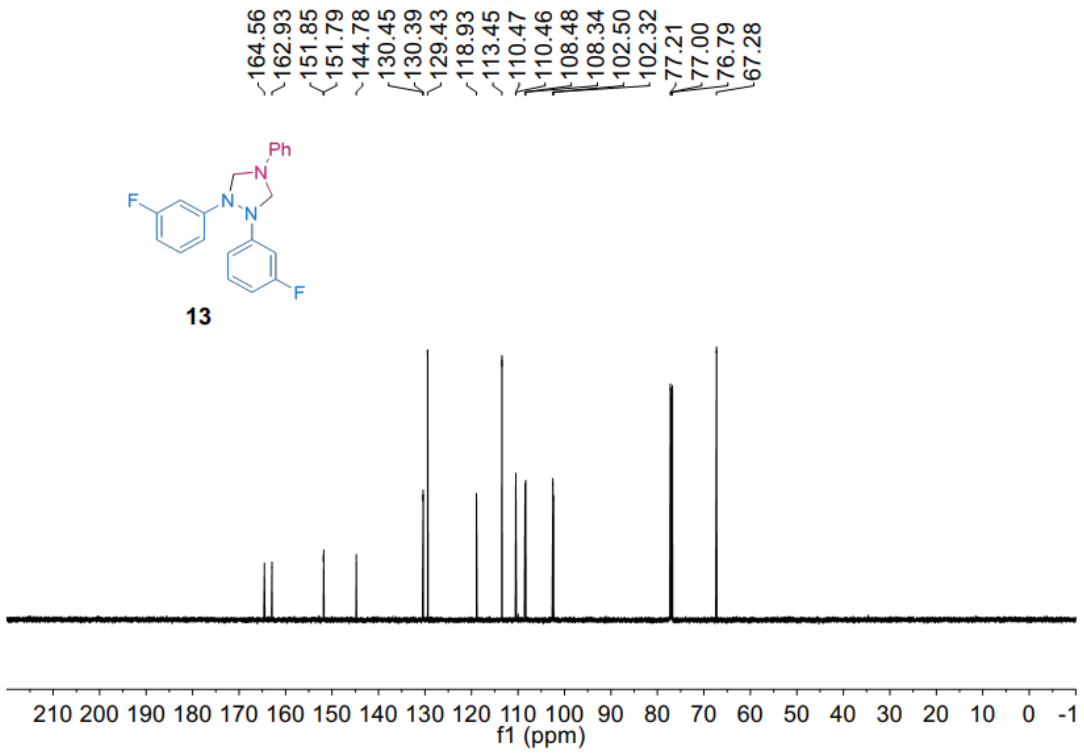
^{13}C NMR spectrum of **12** (100 MHz, $CDCl_3$):



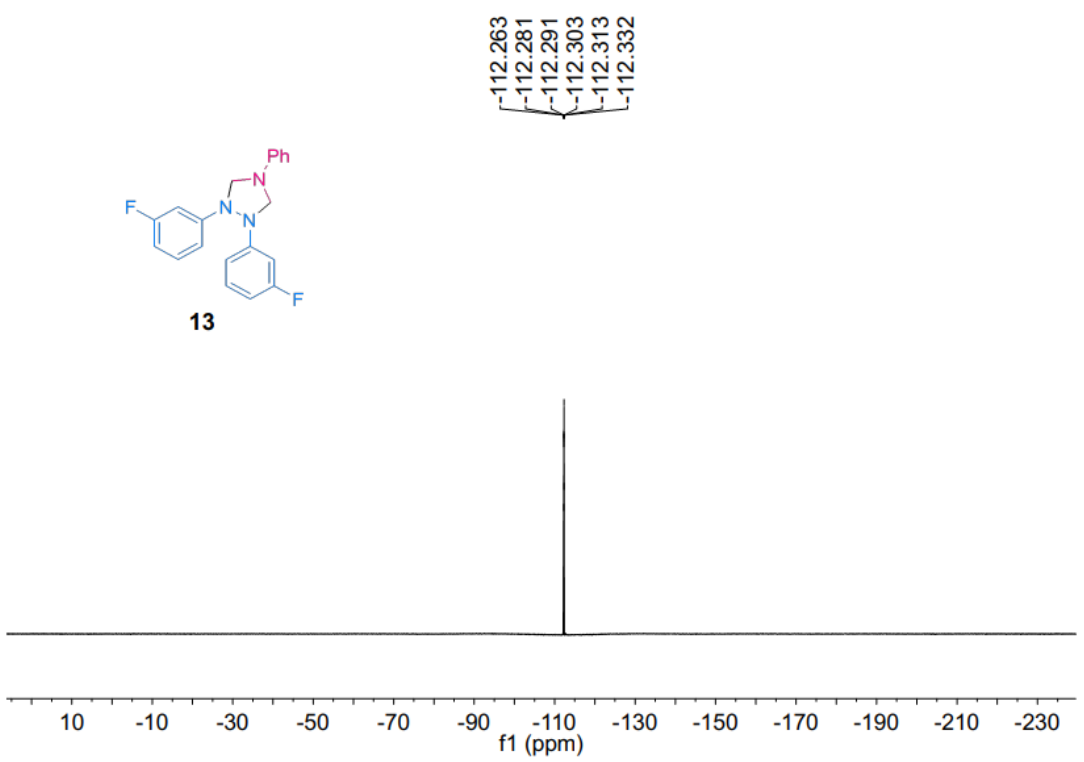
^{1}H NMR spectrum of **13** (400 MHz, $CDCl_3$):



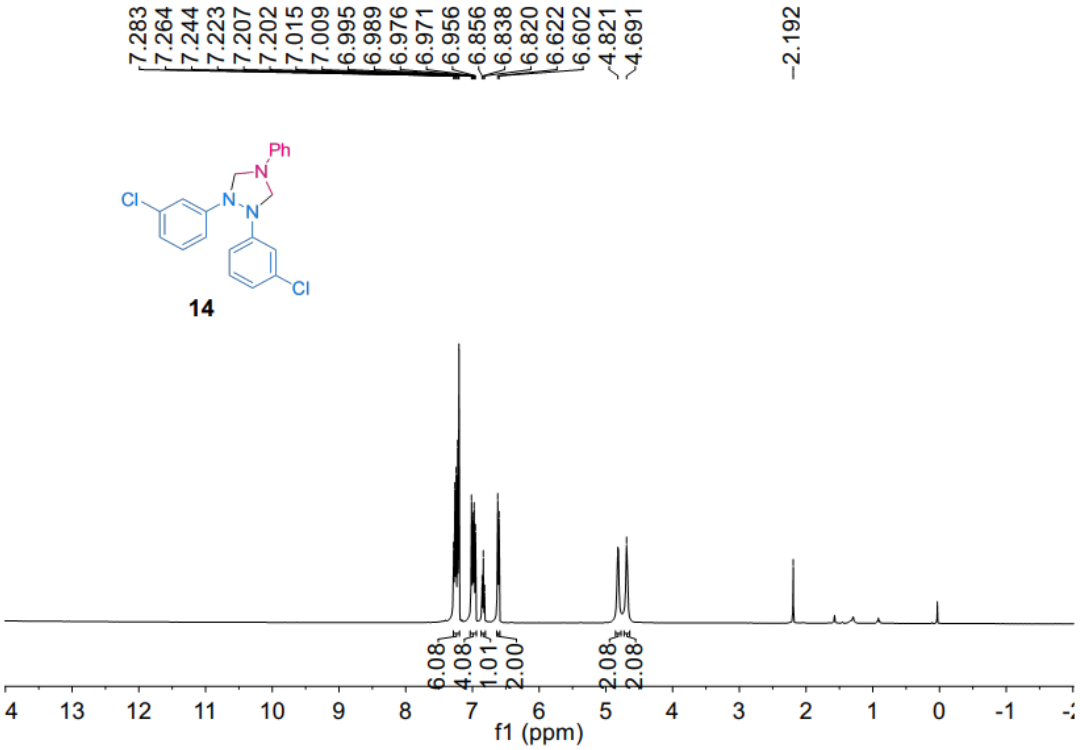
^{13}C NMR spectrum of **13** (150 MHz, $CDCl_3$):



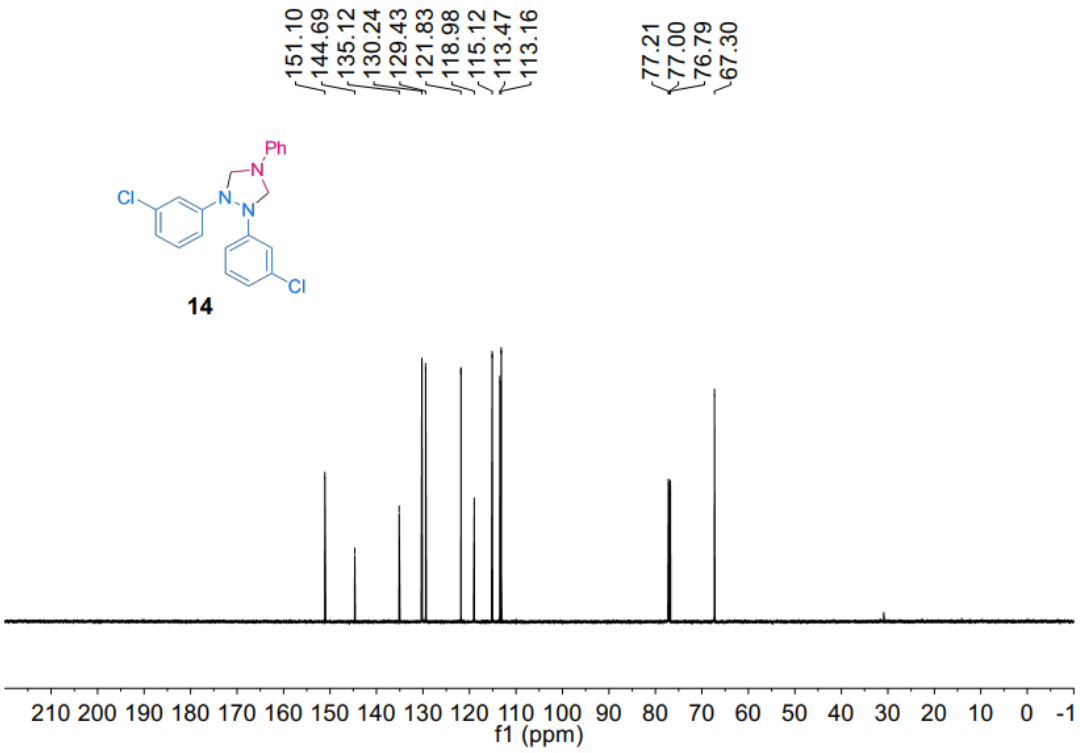
^{19}F NMR spectrum of **13** (376 MHz, $CDCl_3$):



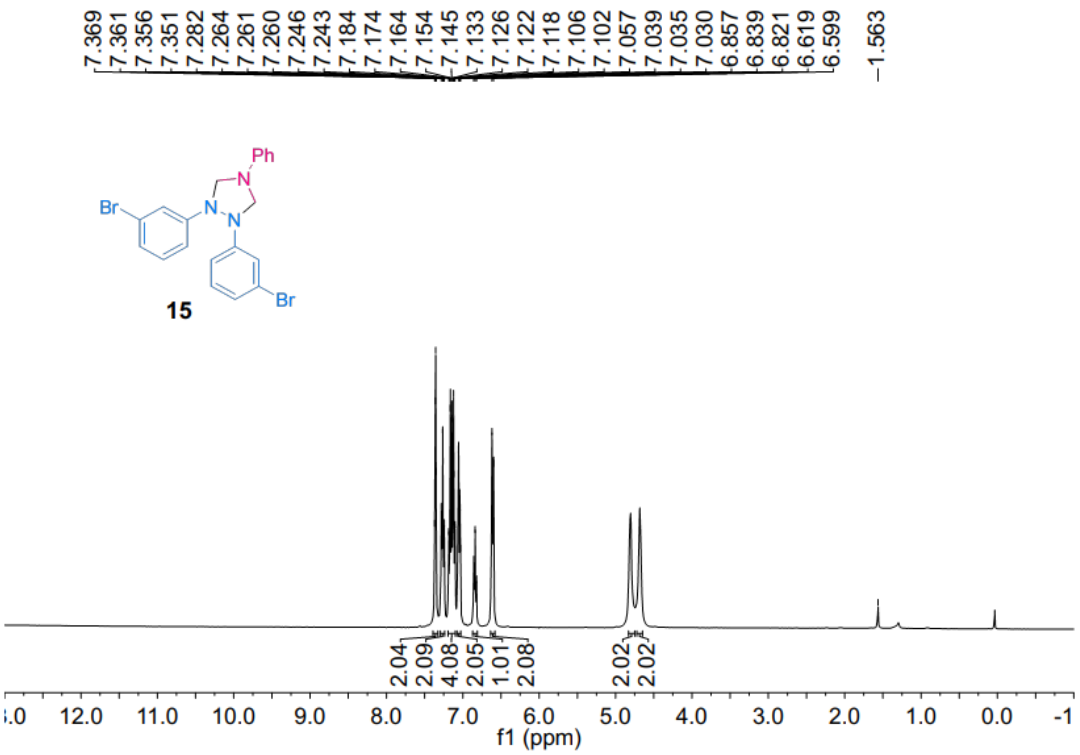
^{1}H NMR spectrum of **14** (400 MHz, $CDCl_3$):



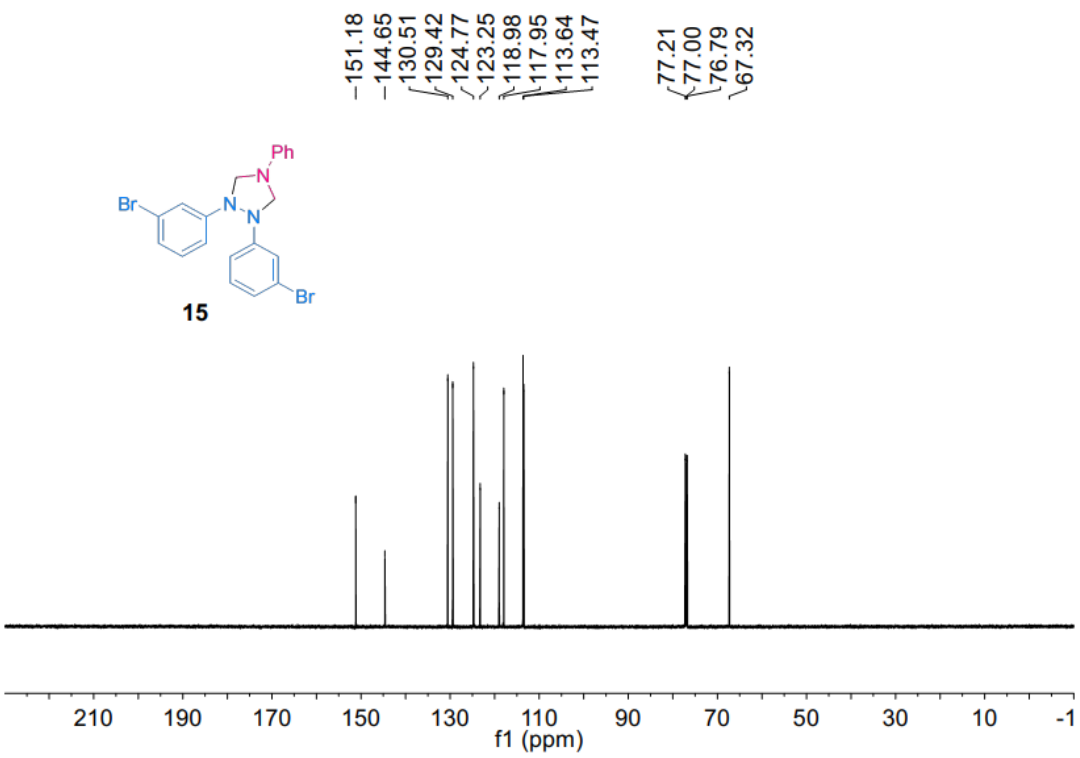
^{13}C NMR spectrum of **14** (150 MHz, $CDCl_3$):



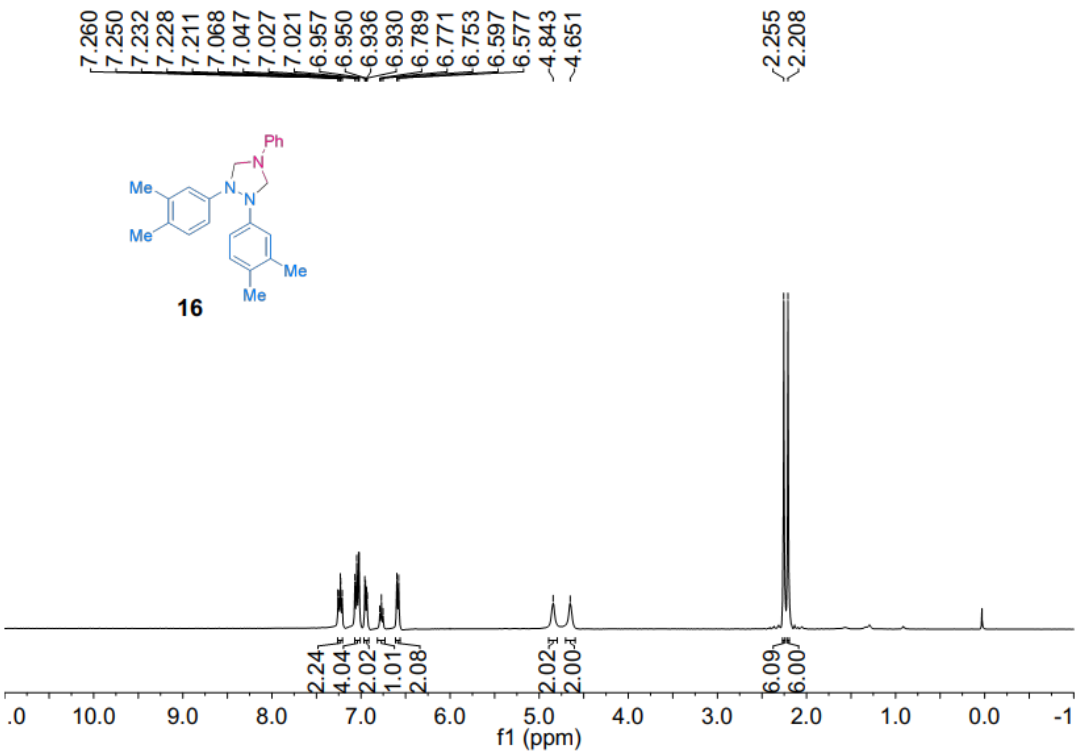
^{1}H NMR spectrum of **15** (400 MHz, $CDCl_3$):



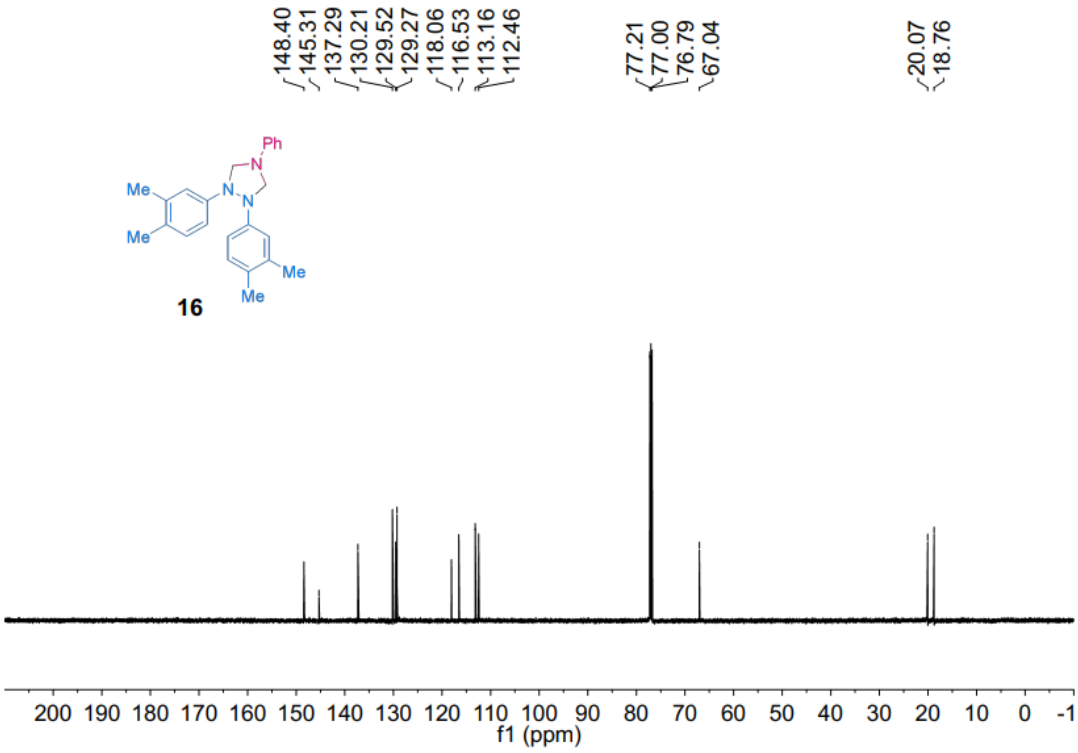
^{13}C NMR spectrum of **15** (150 MHz, $CDCl_3$):



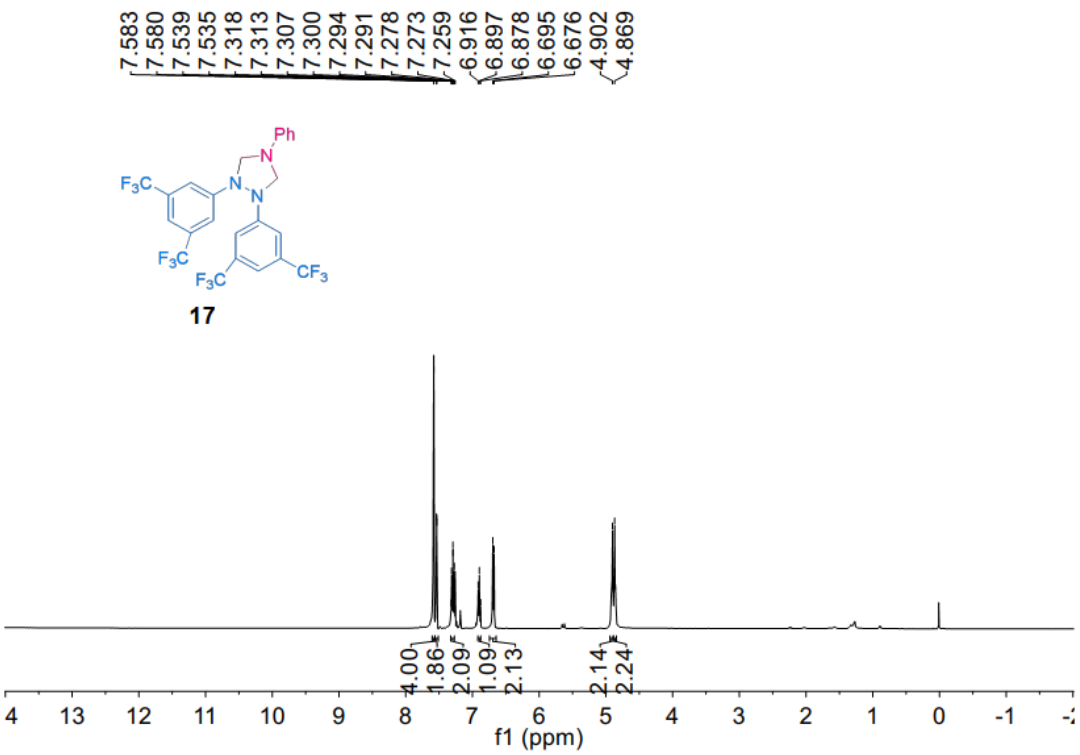
^{1}H NMR spectrum of **16** (400 MHz, $CDCl_3$):



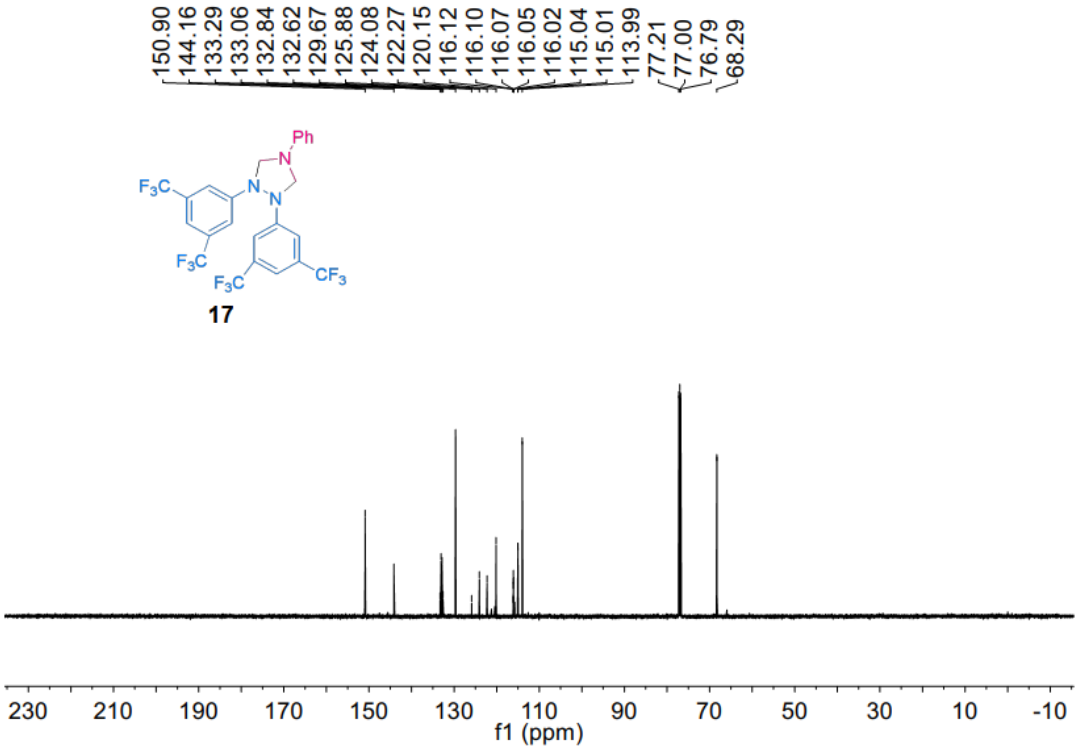
^{13}C NMR spectrum of **16** (150 MHz, $CDCl_3$):



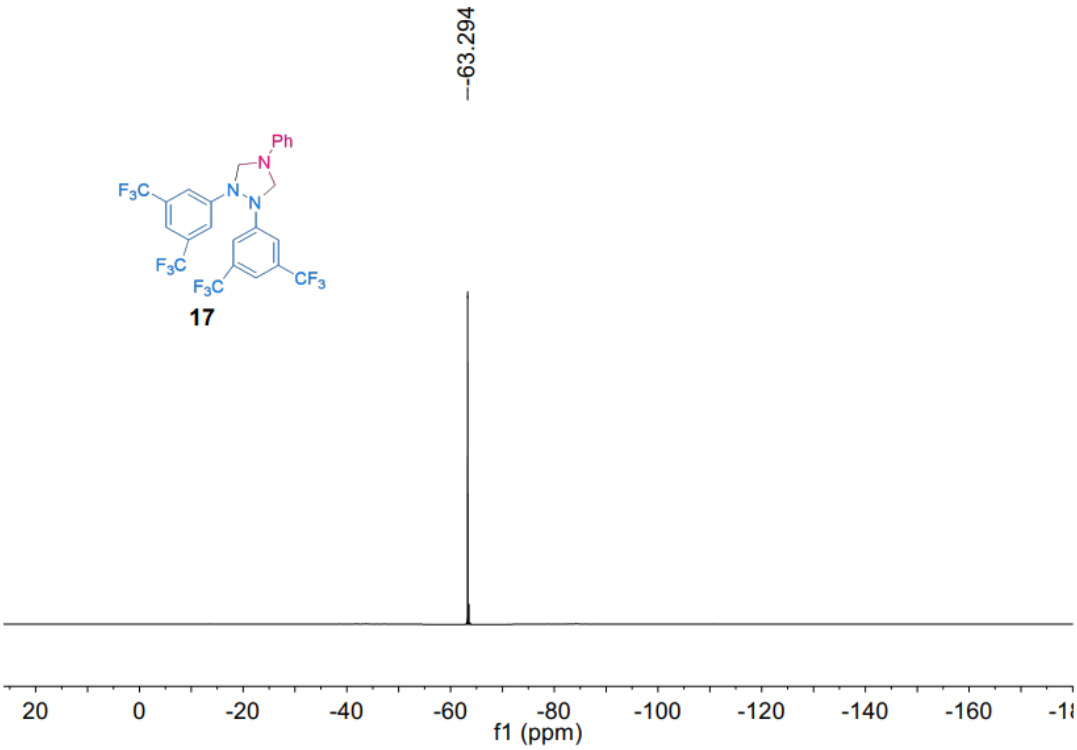
^{1}H NMR spectrum of **17** (400 MHz, $CDCl_3$):



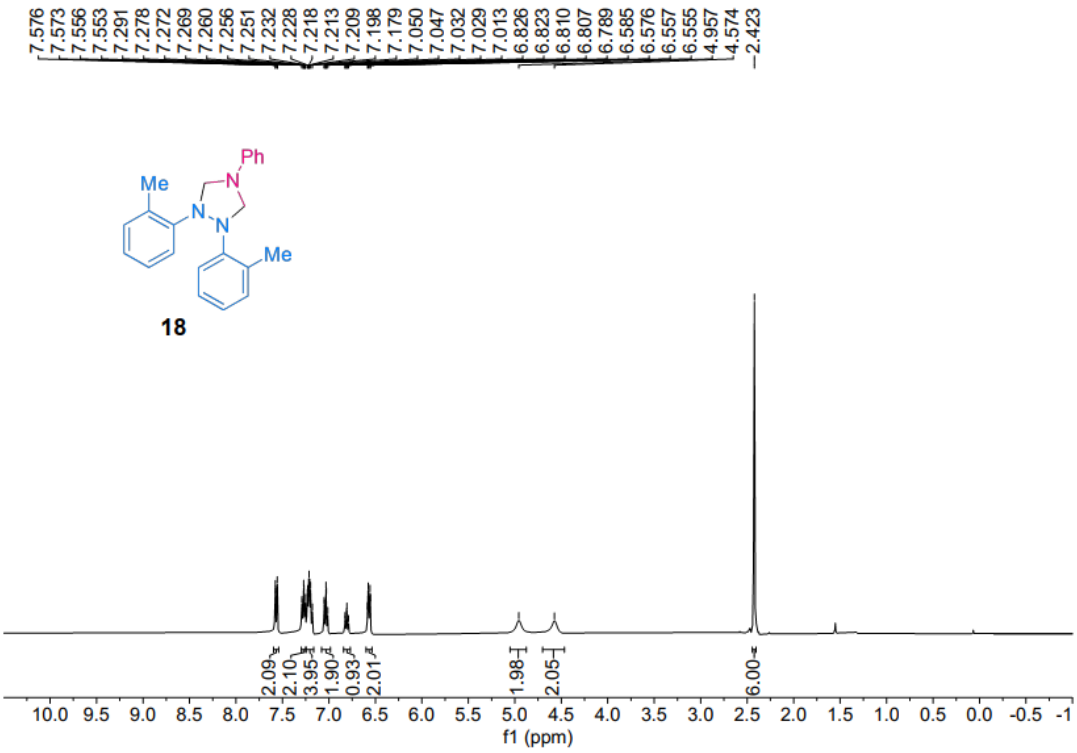
^{13}C NMR spectrum of **17** (150 MHz, $CDCl_3$):



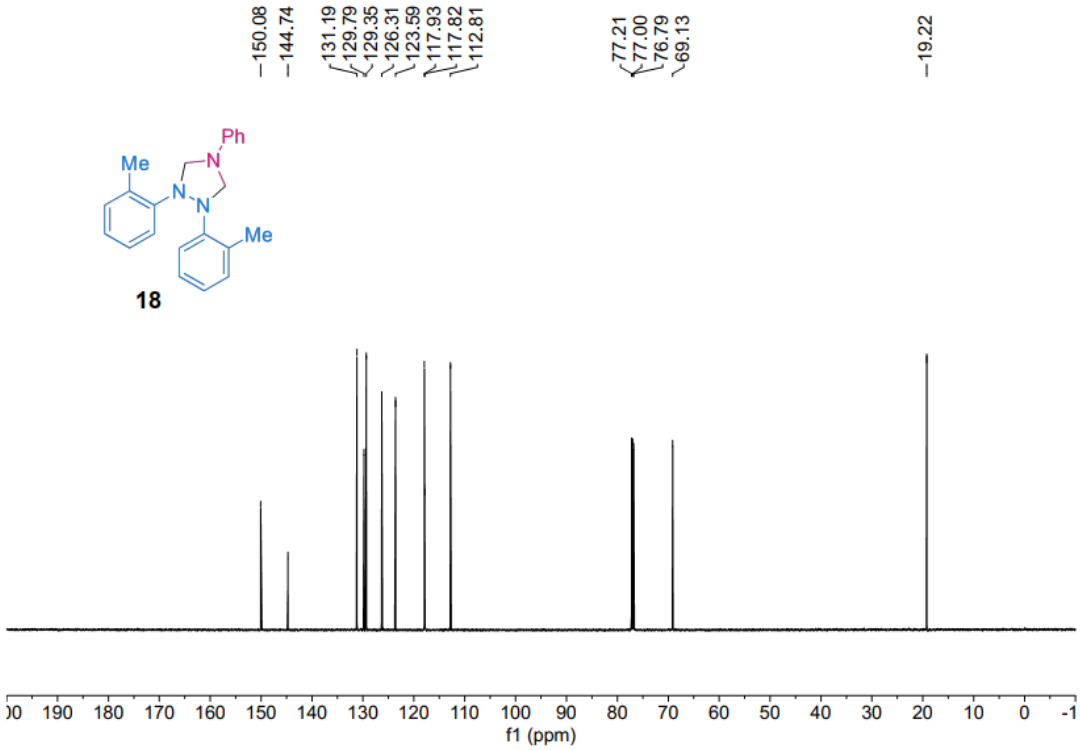
^{19}F NMR spectrum of **17** (376 MHz, $CDCl_3$):



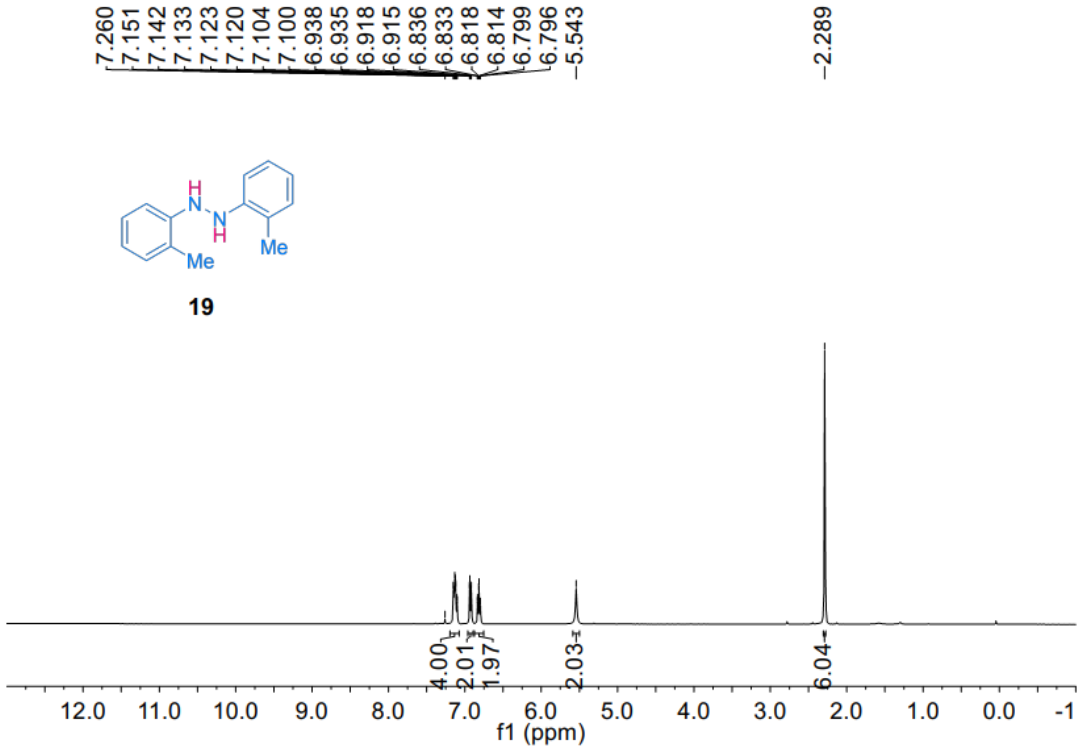
^{1}H NMR spectrum of **18** (400 MHz, $CDCl_3$):



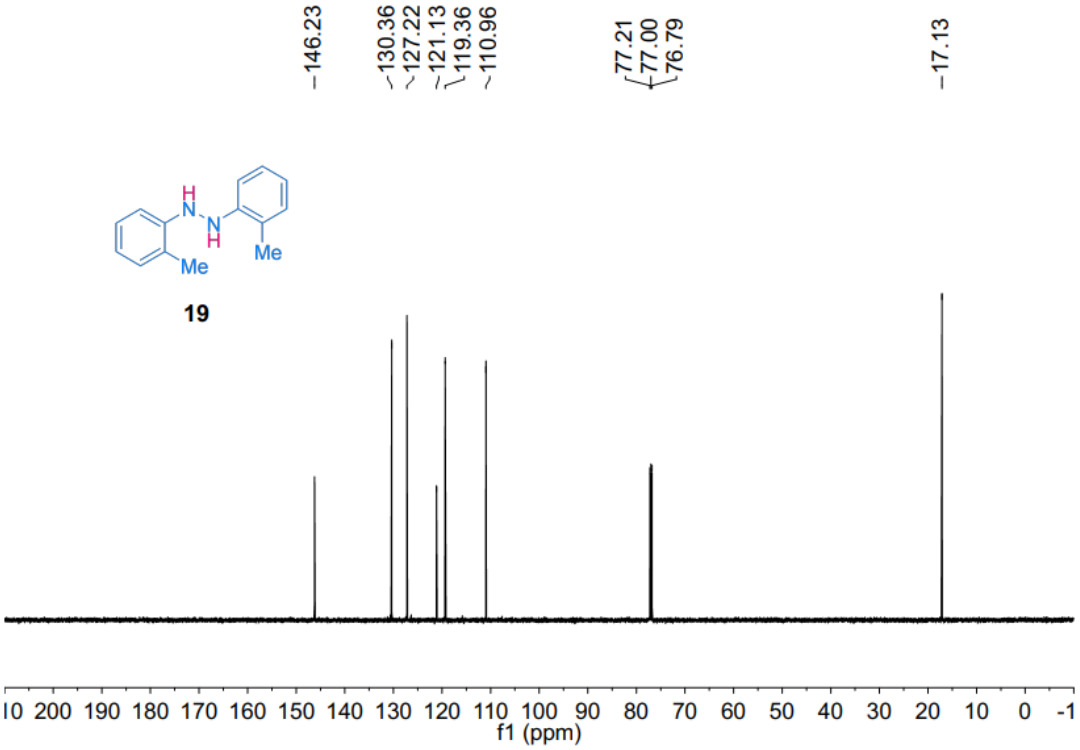
^{13}C NMR spectrum of **18** (150 MHz, $CDCl_3$):



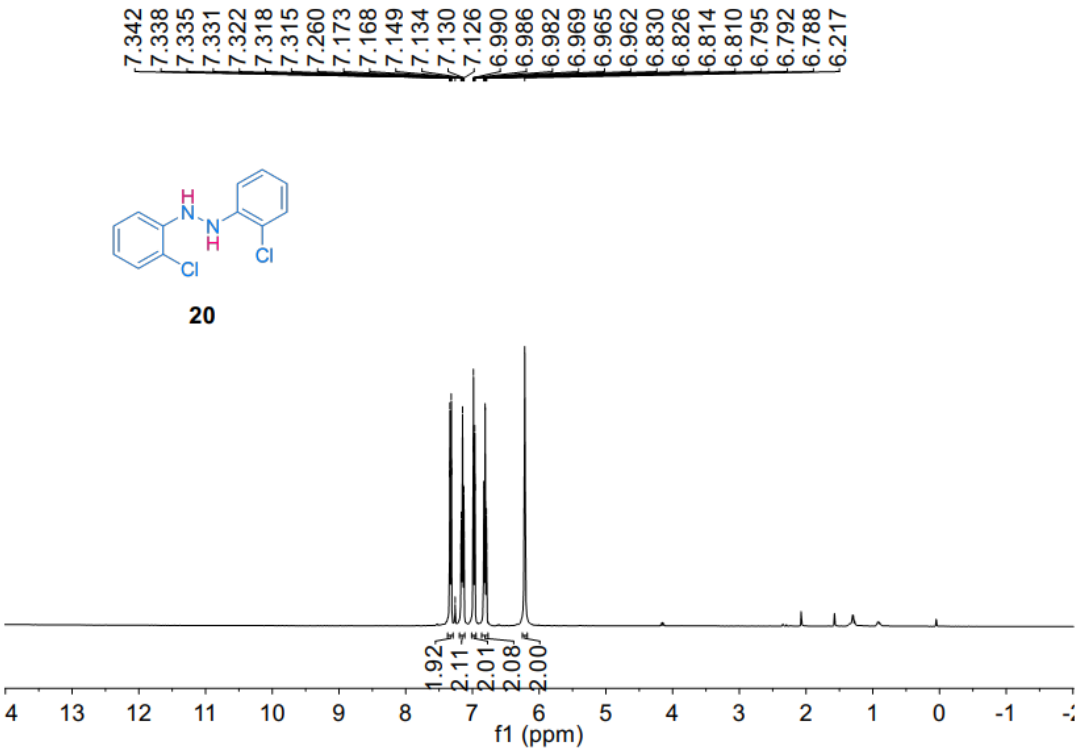
^{1}H NMR spectrum of **19** (400 MHz, $CDCl_3$):



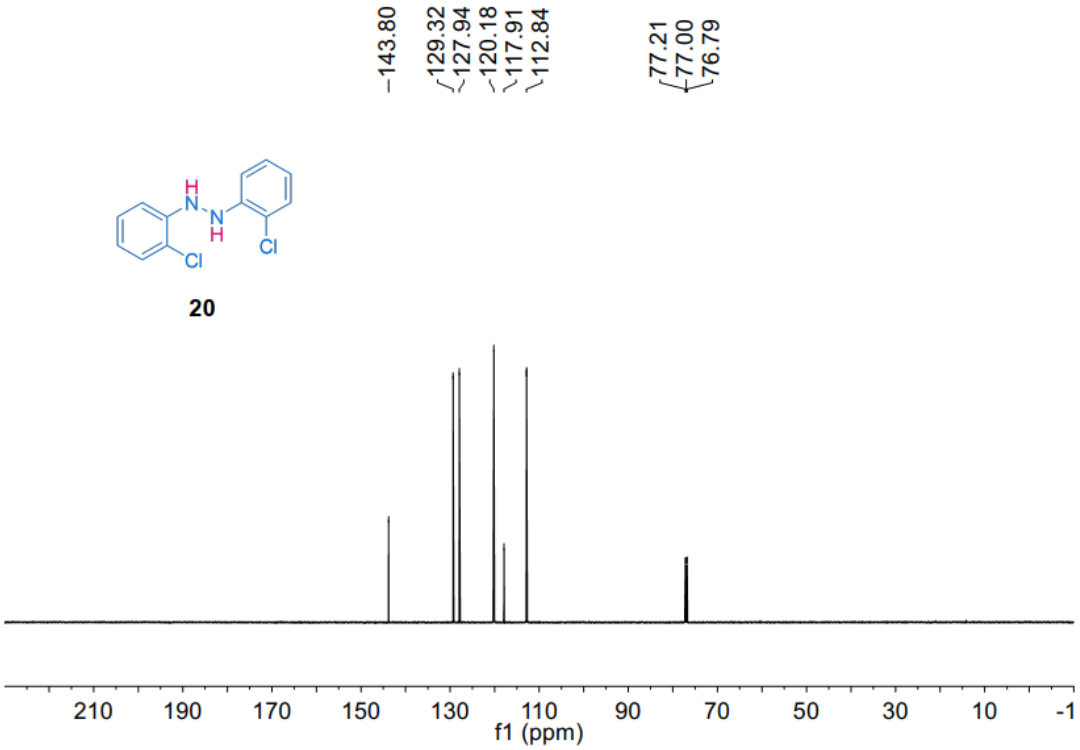
^{13}C NMR spectrum of **19** (150 MHz, $CDCl_3$):



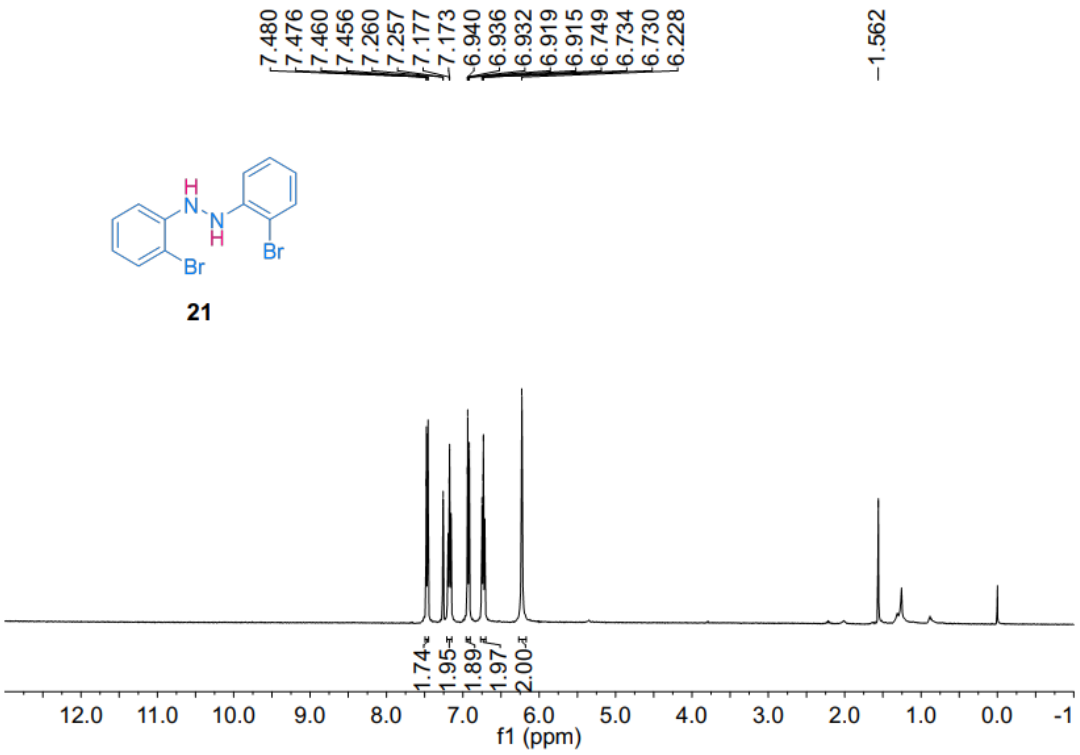
^{1}H NMR spectrum of **20** (400 MHz, $CDCl_3$):



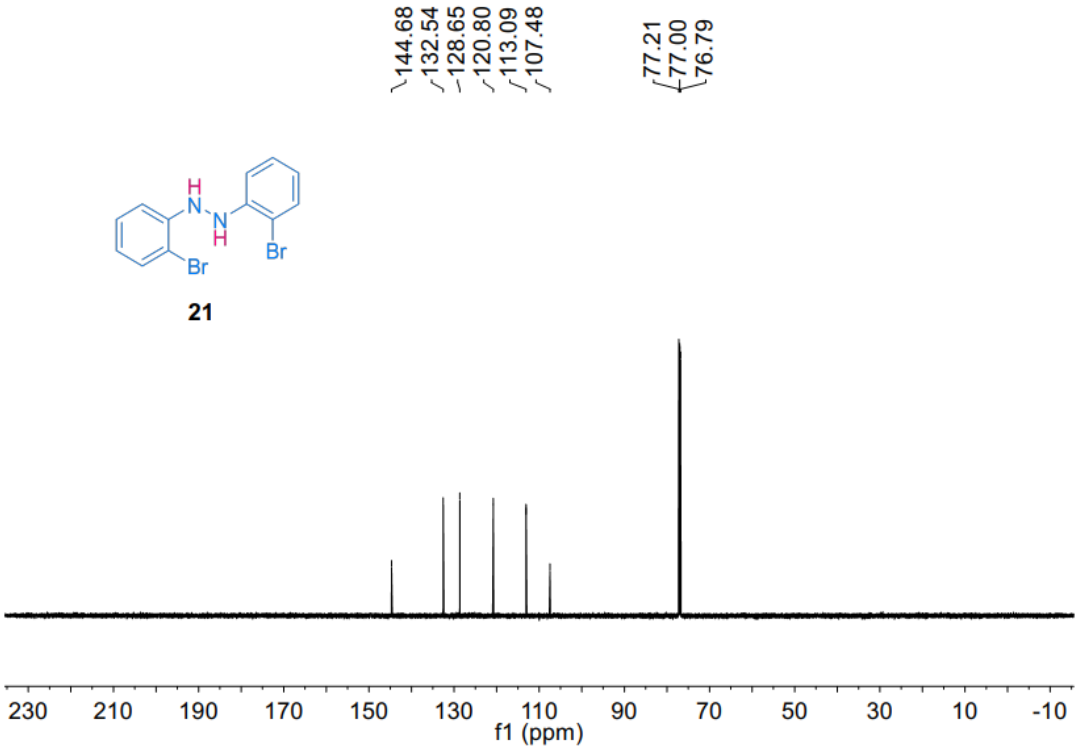
^{13}C NMR spectrum of **20** (150 MHz, $CDCl_3$):



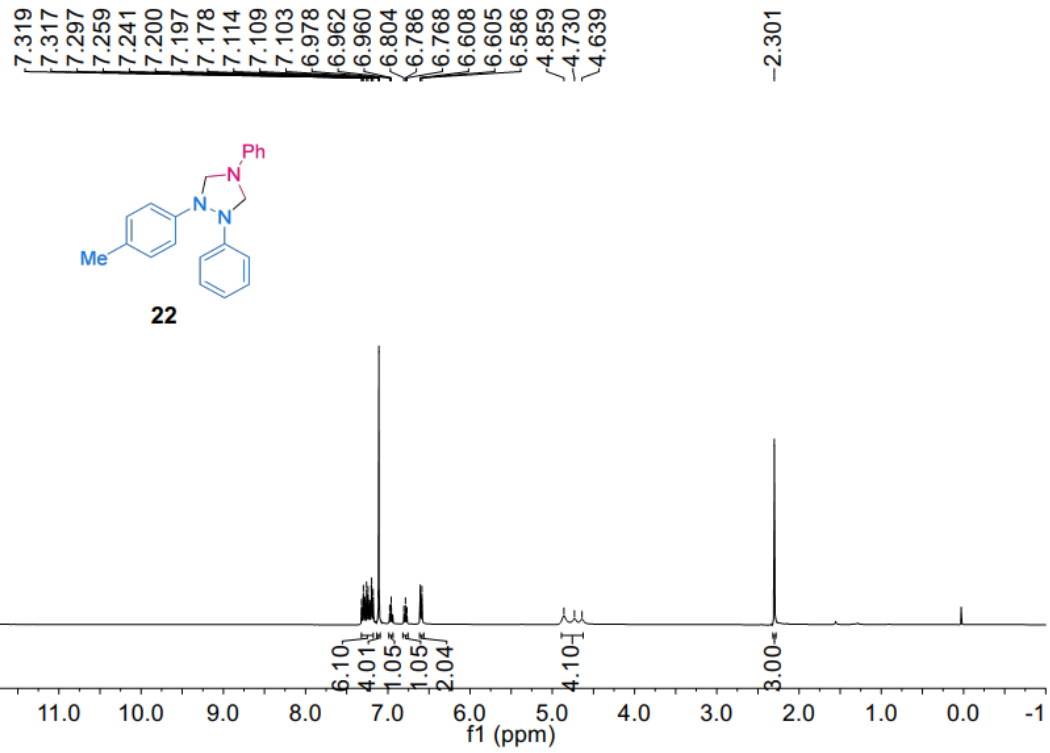
^{1}H NMR spectrum of **21** (400 MHz, $CDCl_3$):



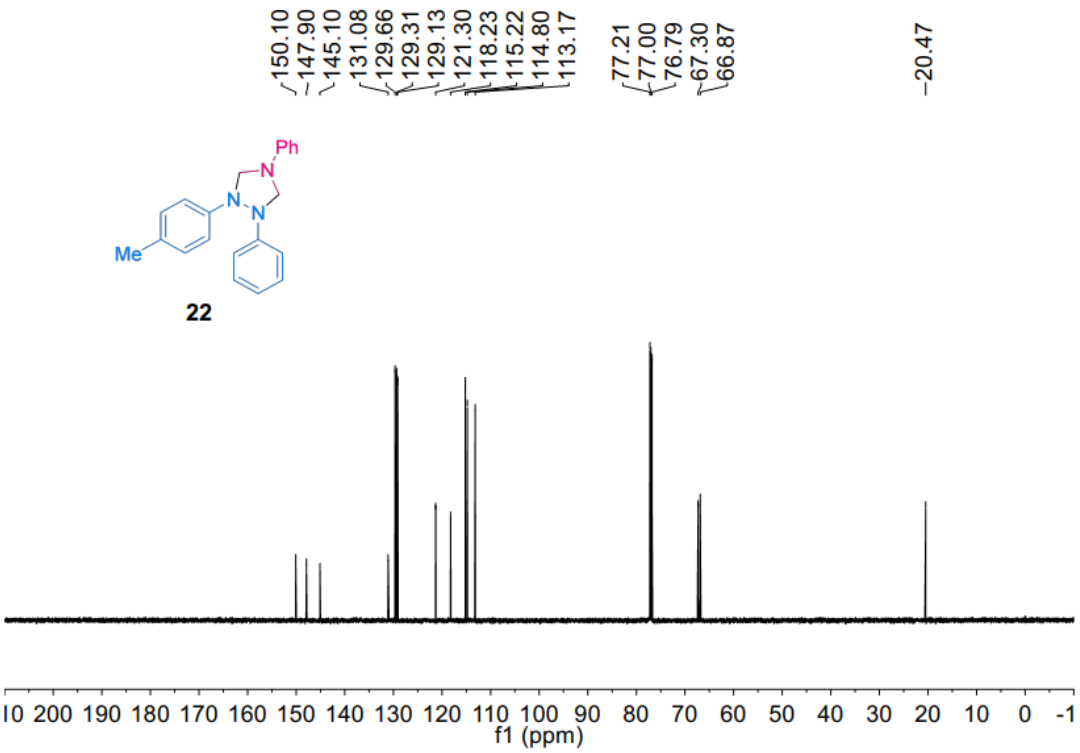
^{13}C NMR spectrum of **21** (150 MHz, $CDCl_3$):



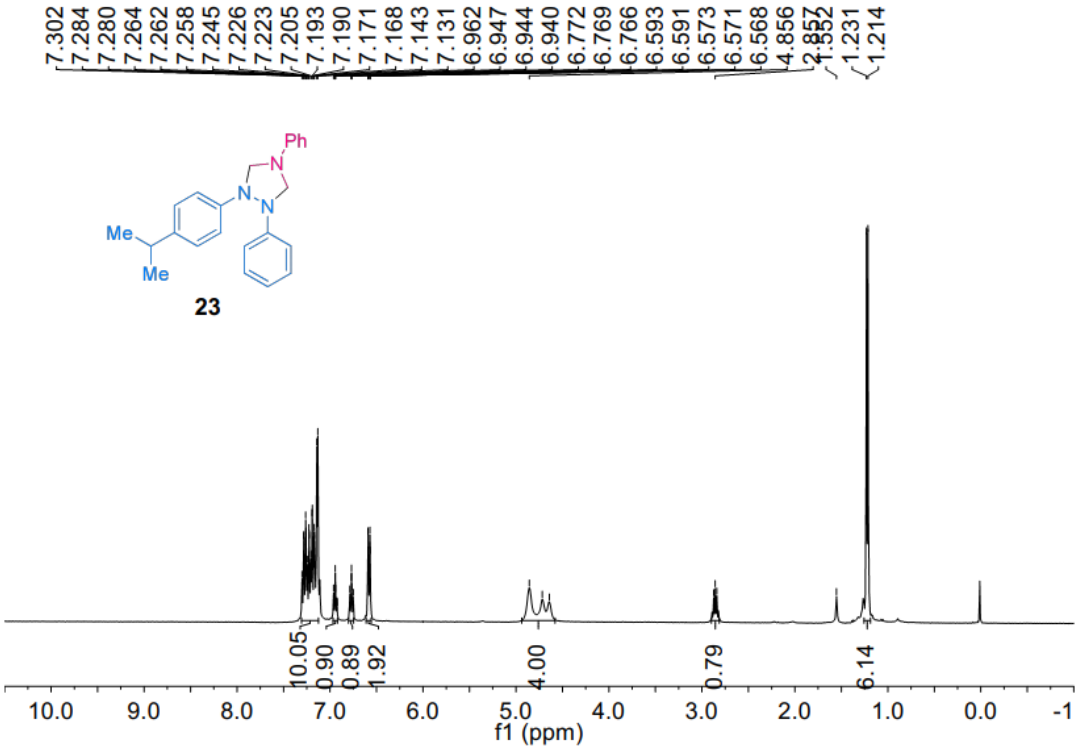
^{1}H NMR spectrum of **22** (400 MHz, $CDCl_3$):



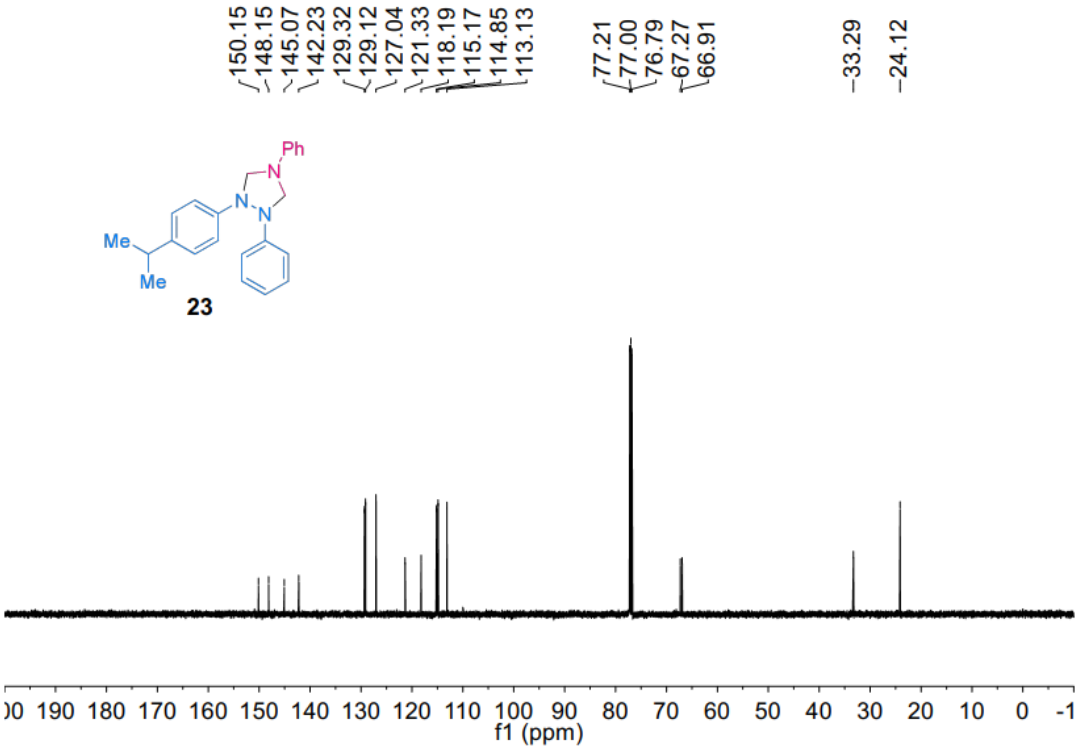
^{13}C NMR spectrum of **22** (150 MHz, $CDCl_3$):



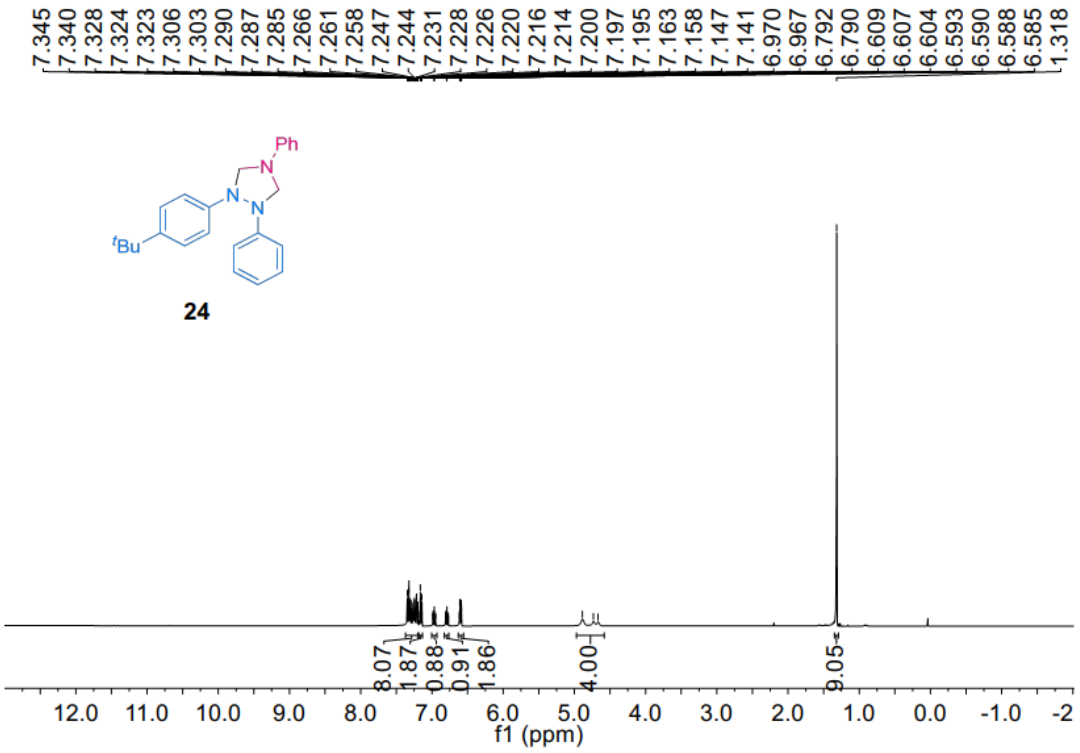
^{1}H NMR spectrum of **23** (400 MHz, $CDCl_3$):



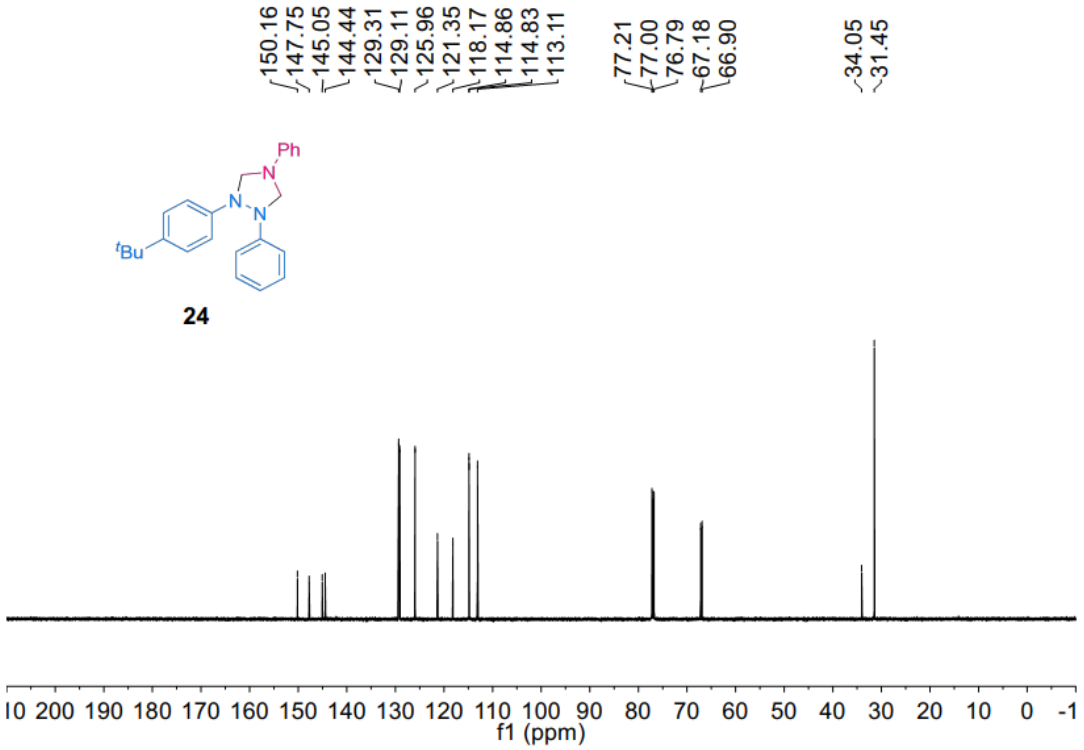
^{13}C NMR spectrum of **23** (150 MHz, $CDCl_3$):



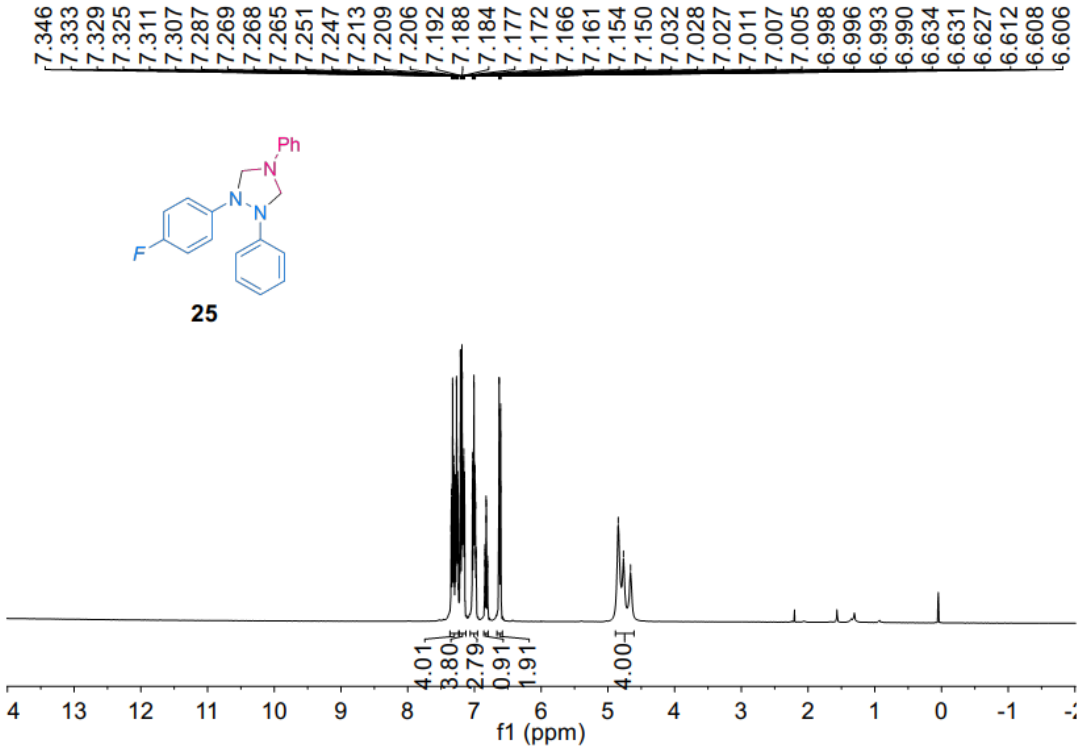
^{1}H NMR spectrum of **24** (400 MHz, $CDCl_3$):



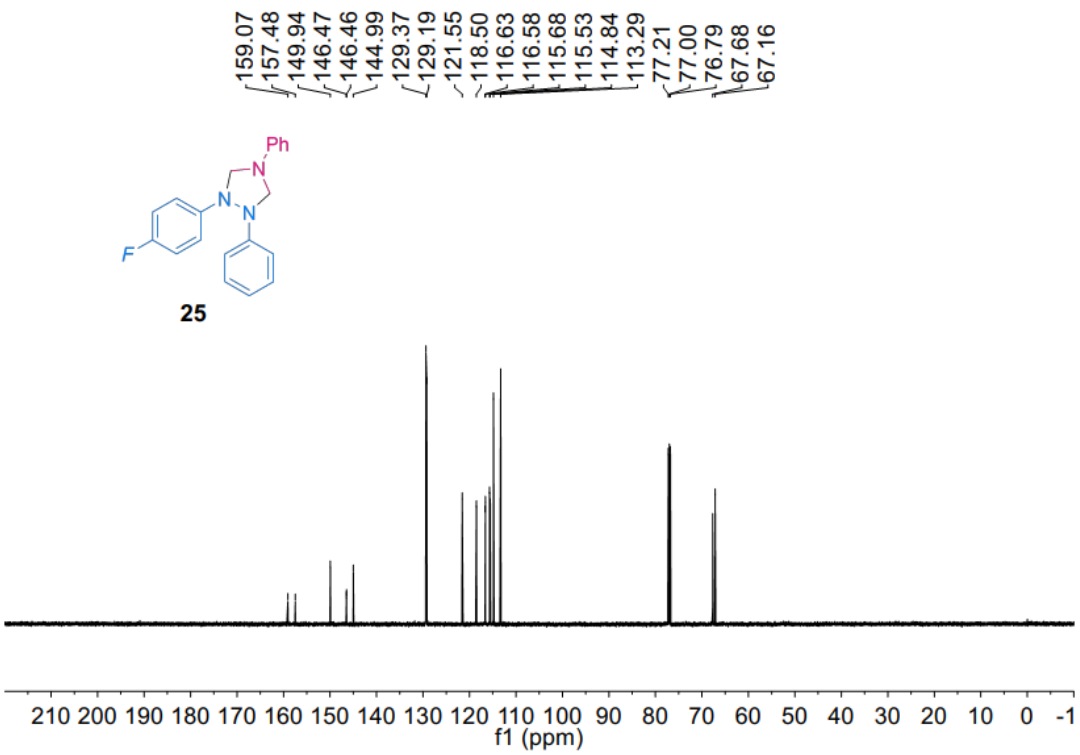
^{13}C NMR spectrum of **24** (150 MHz, $CDCl_3$):



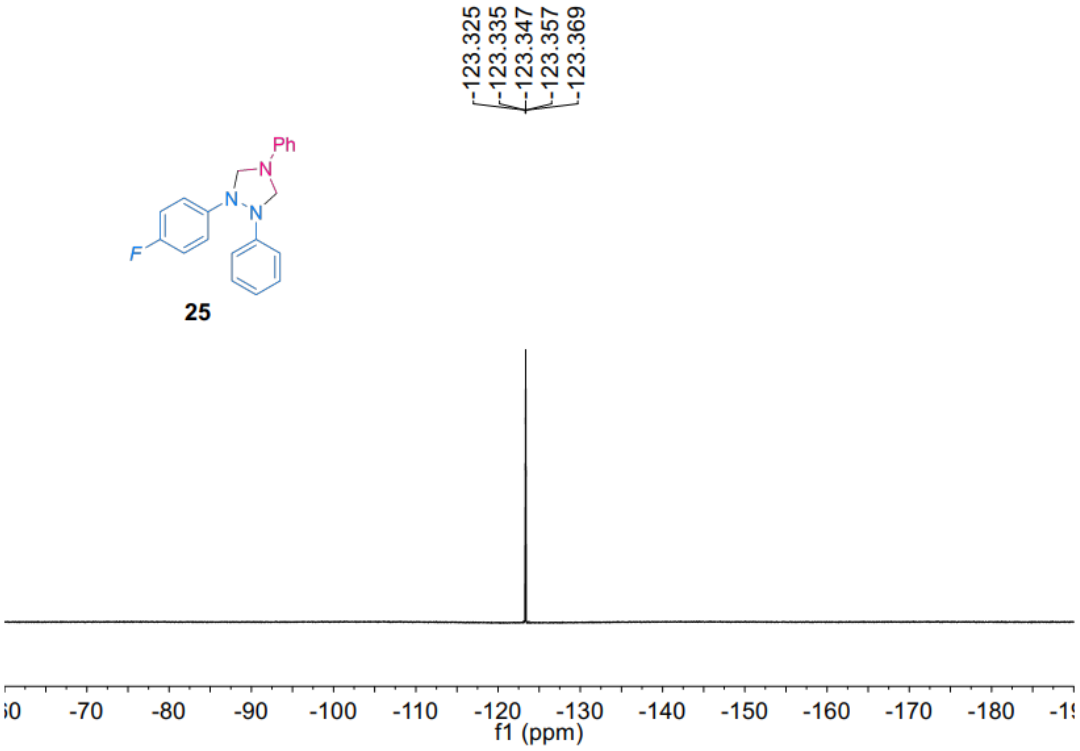
^{1}H NMR spectrum of **25** (400 MHz, $CDCl_3$):



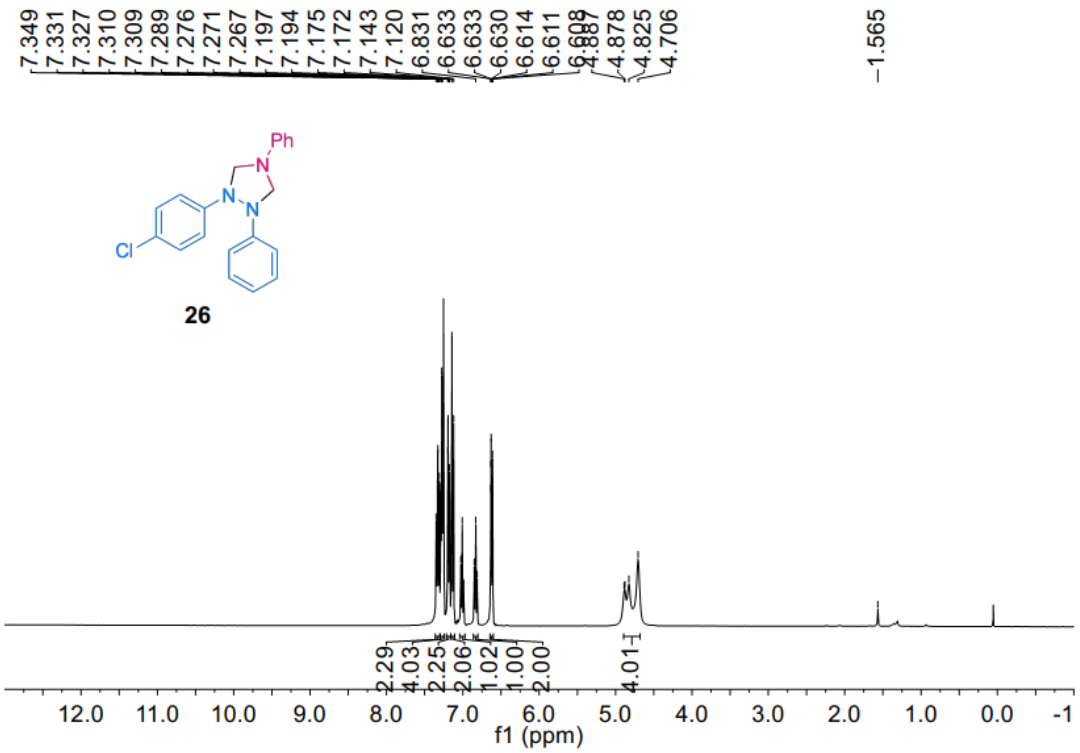
^{13}C NMR spectrum of **25** (150 MHz, $CDCl_3$):



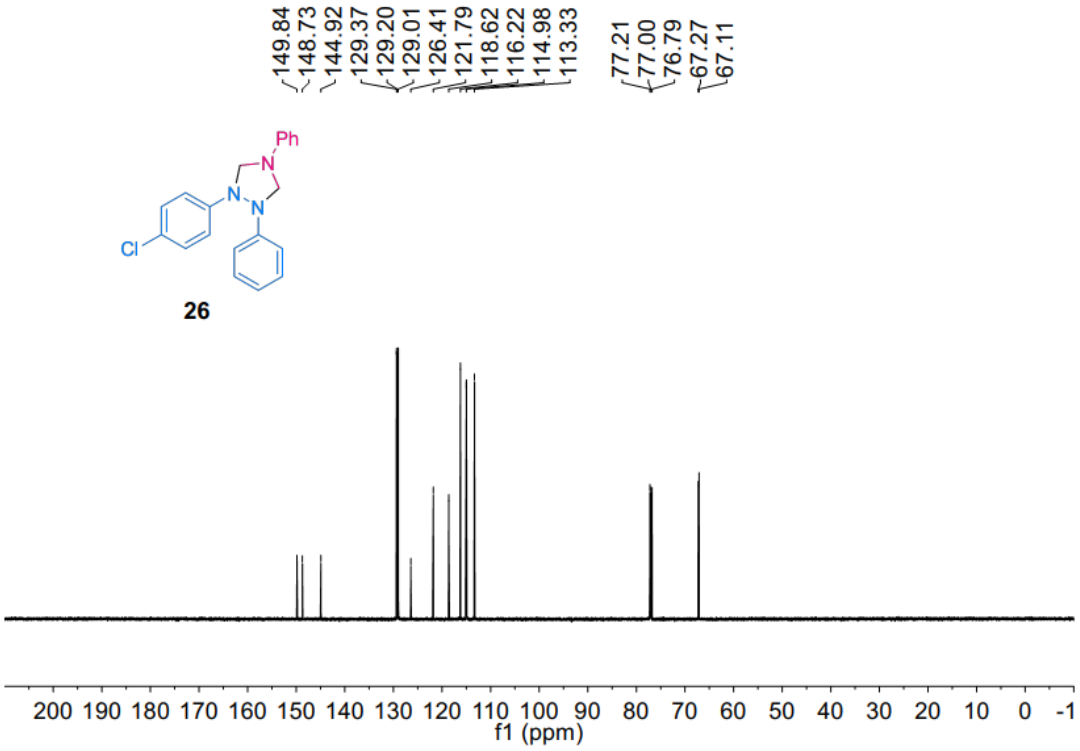
^{19}F NMR spectrum of **25** (376 MHz, $CDCl_3$):



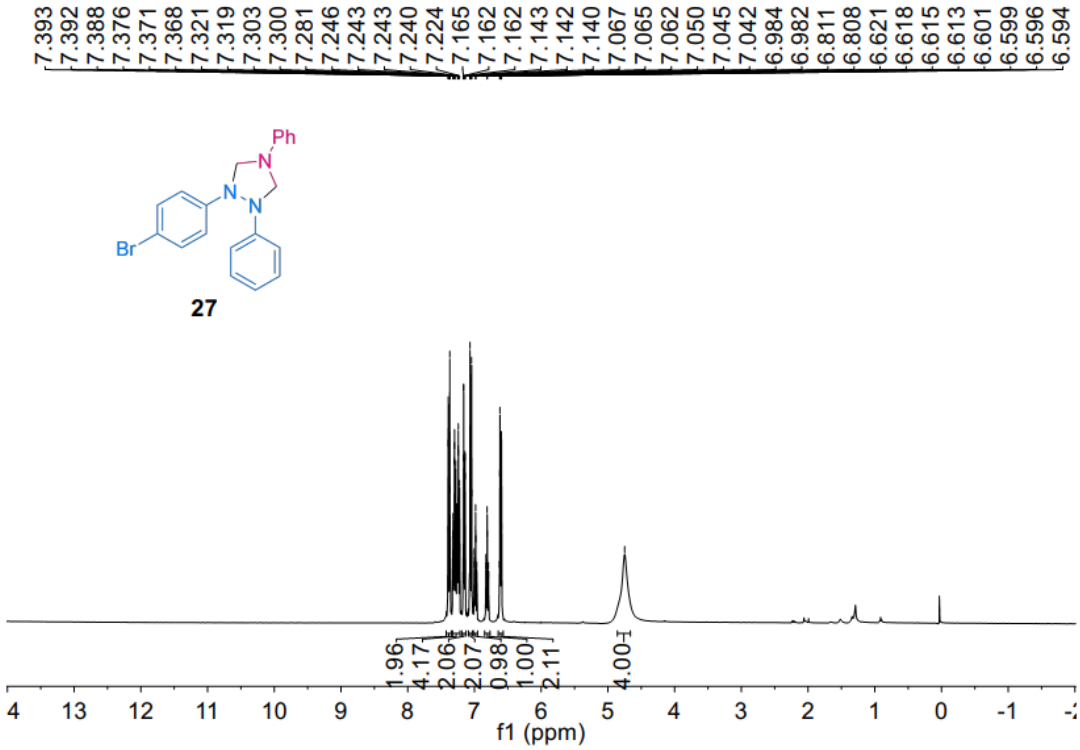
^{1}H NMR spectrum of **26** (400 MHz, $CDCl_3$):



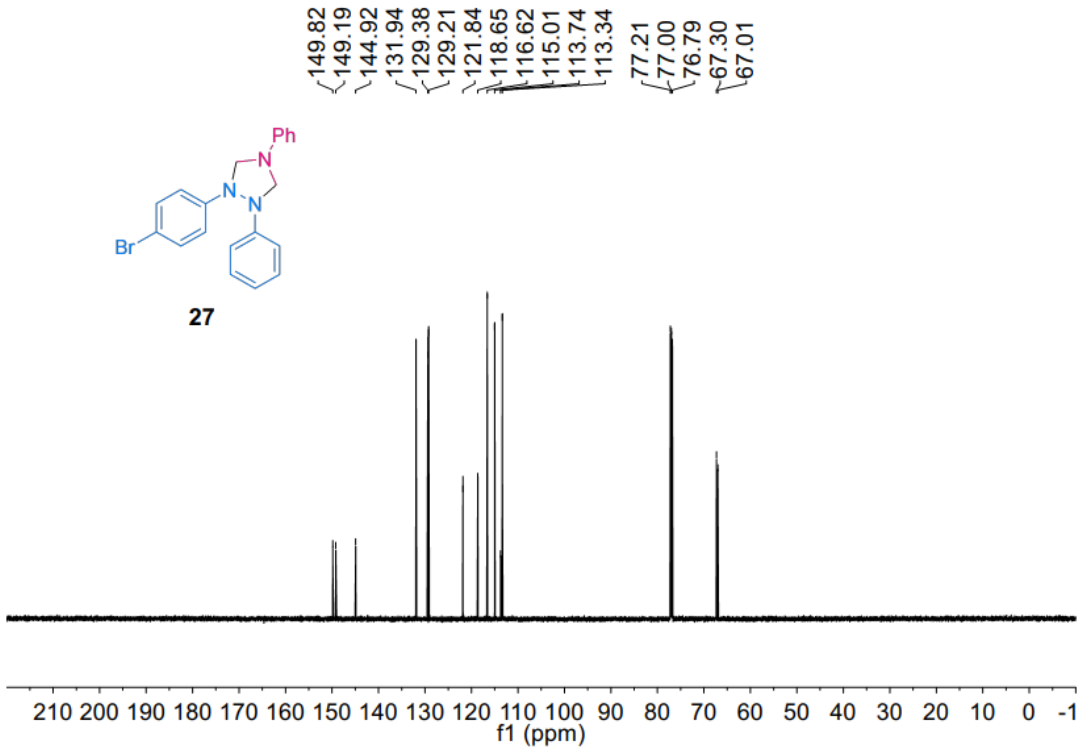
^{13}C NMR spectrum of **26** (150 MHz, $CDCl_3$):



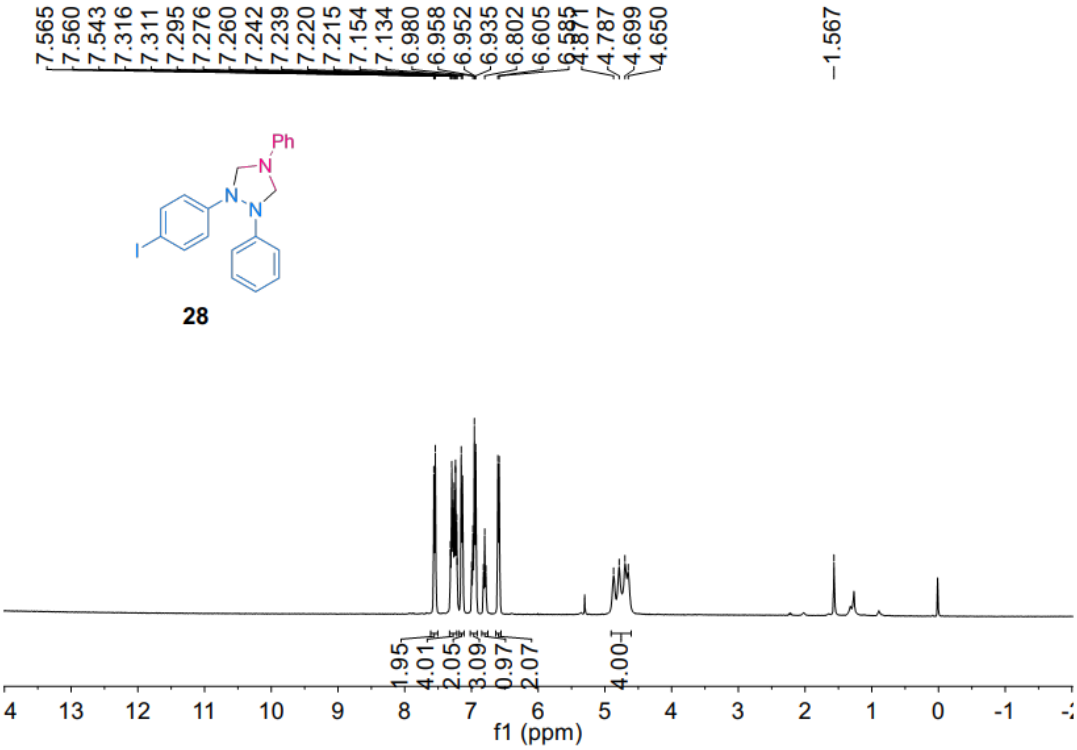
^{1}H NMR spectrum of **27** (400 MHz, $CDCl_3$):



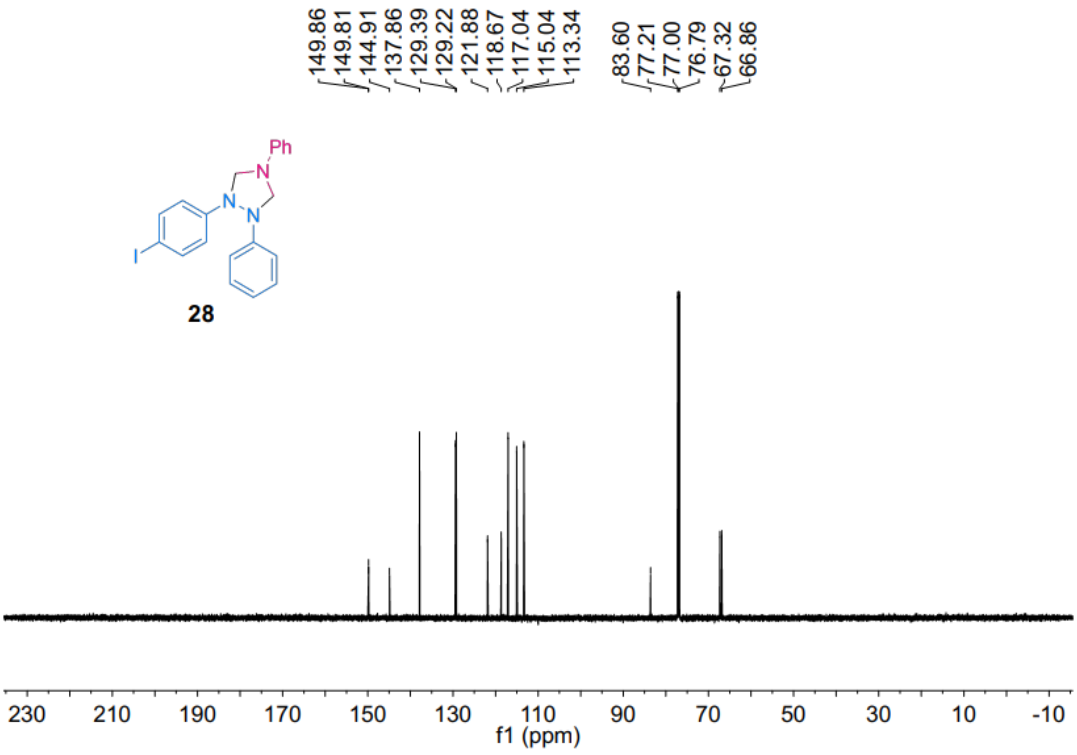
^{13}C NMR spectrum of **27** (150 MHz, $CDCl_3$):



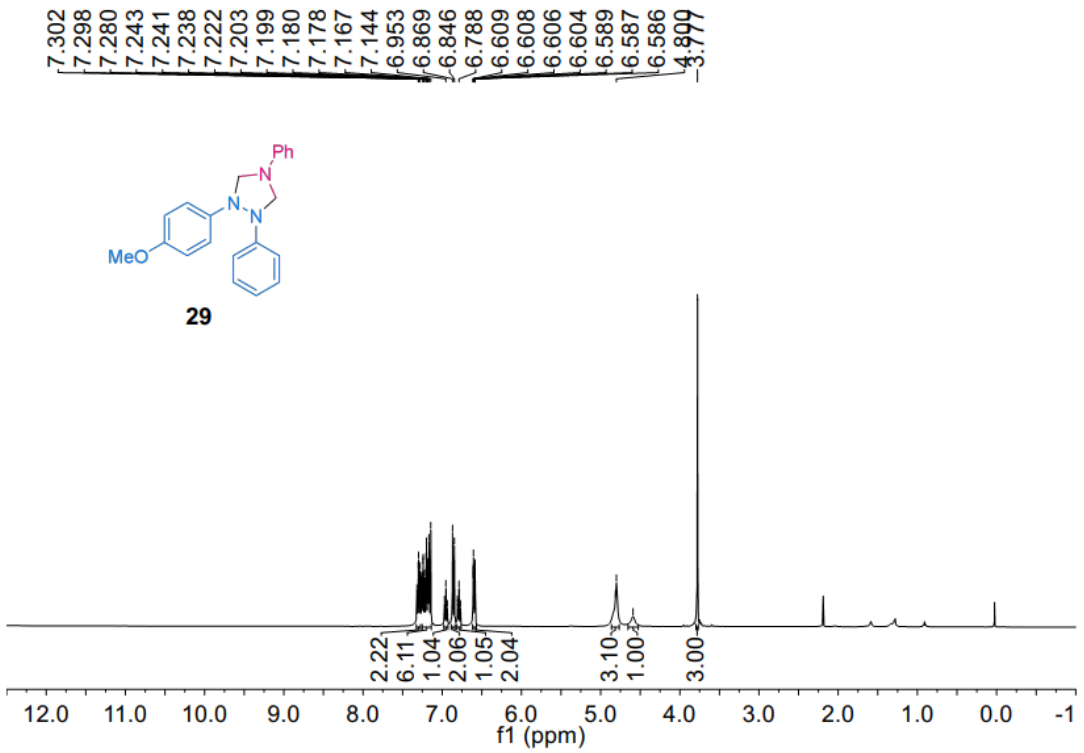
^{1}H NMR spectrum of **28** (400 MHz, $CDCl_3$):



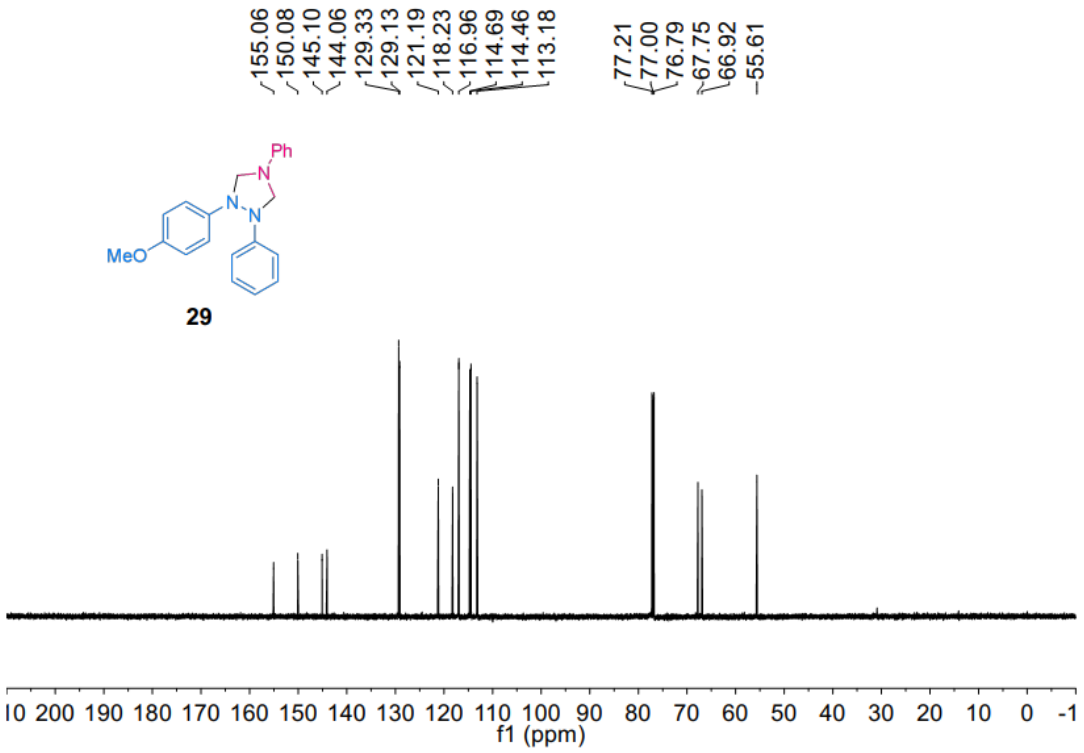
^{13}C NMR spectrum of **28** (150 MHz, $CDCl_3$):



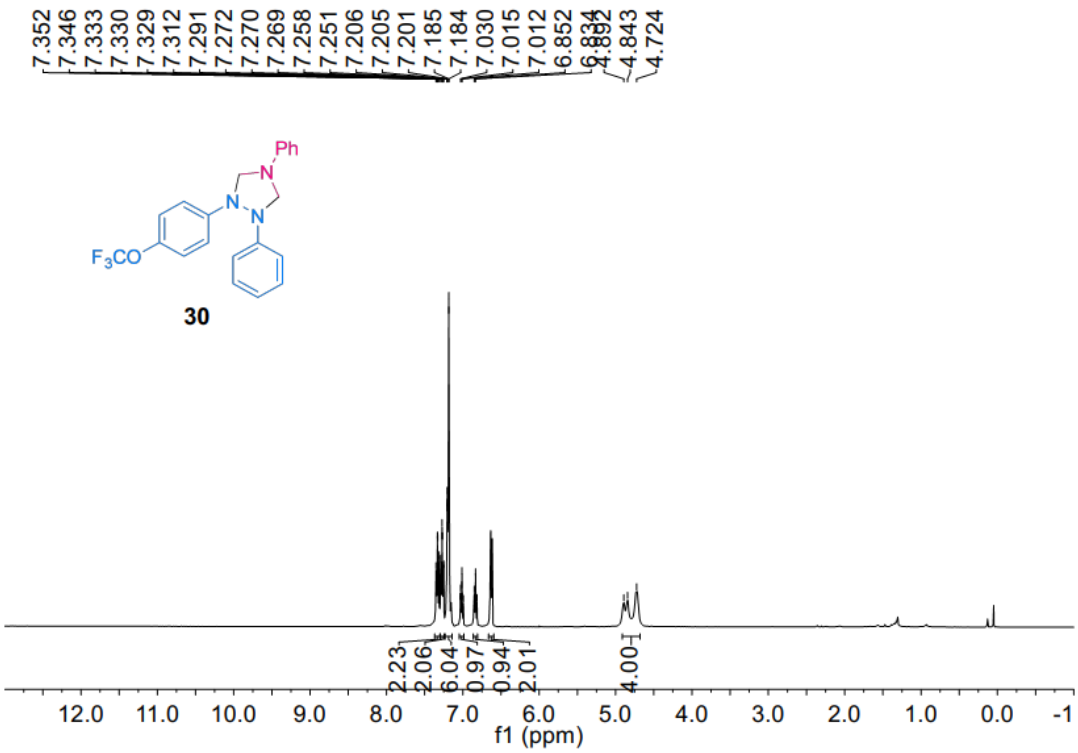
^{1}H NMR spectrum of **29** (400 MHz, $CDCl_3$):



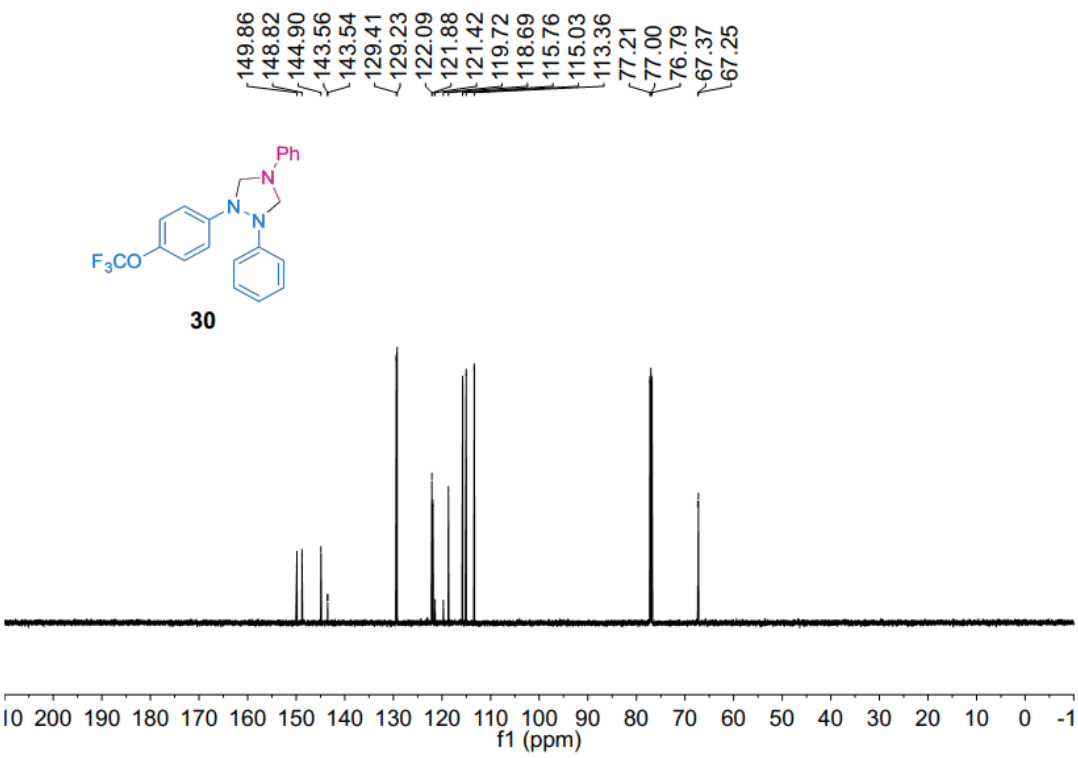
^{13}C NMR spectrum of **29** (150 MHz, $CDCl_3$):



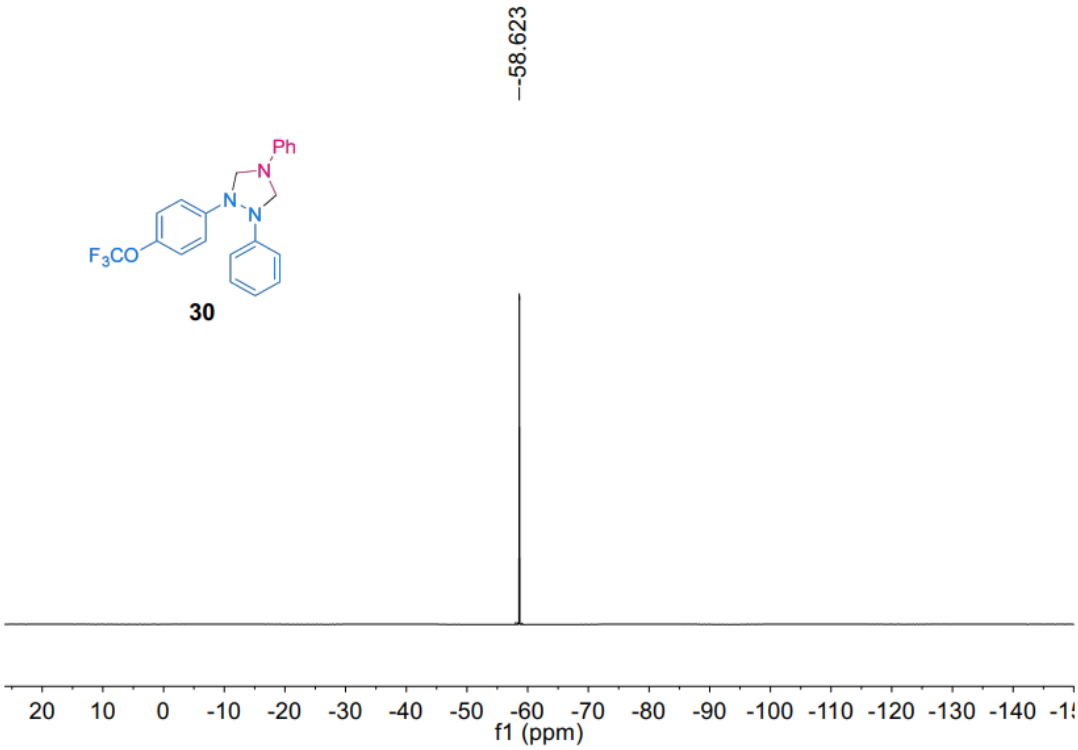
^{1}H NMR spectrum of **30** (400 MHz, $CDCl_3$):



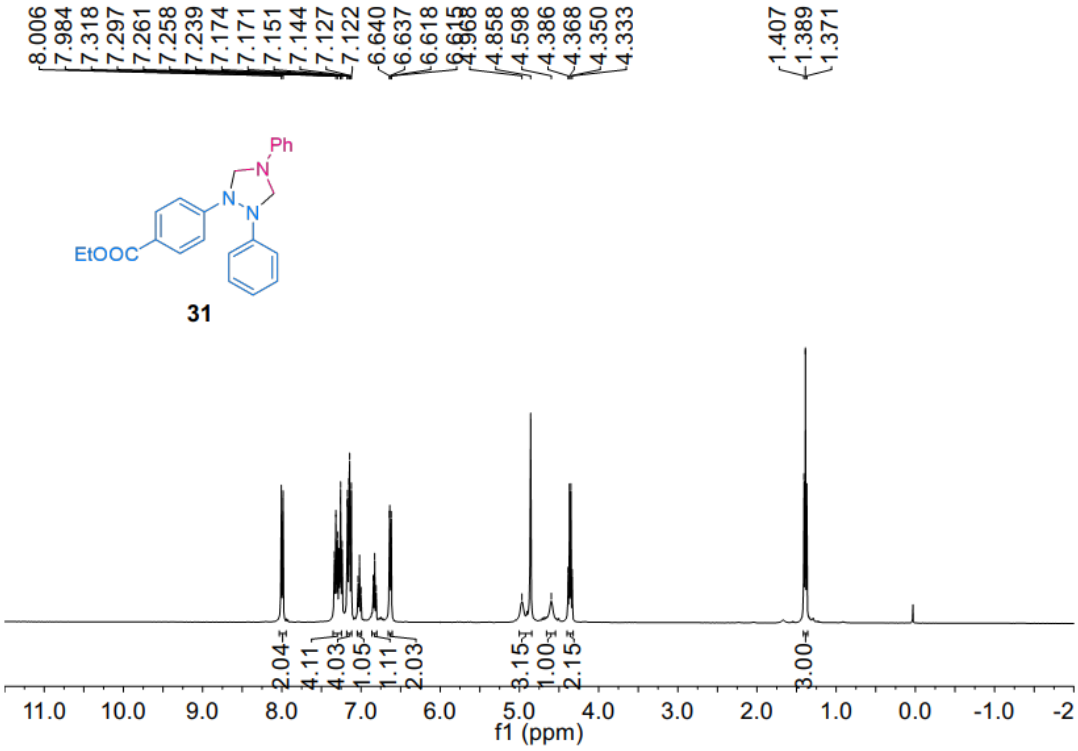
^{13}C NMR spectrum of **30** (150 MHz, $CDCl_3$):



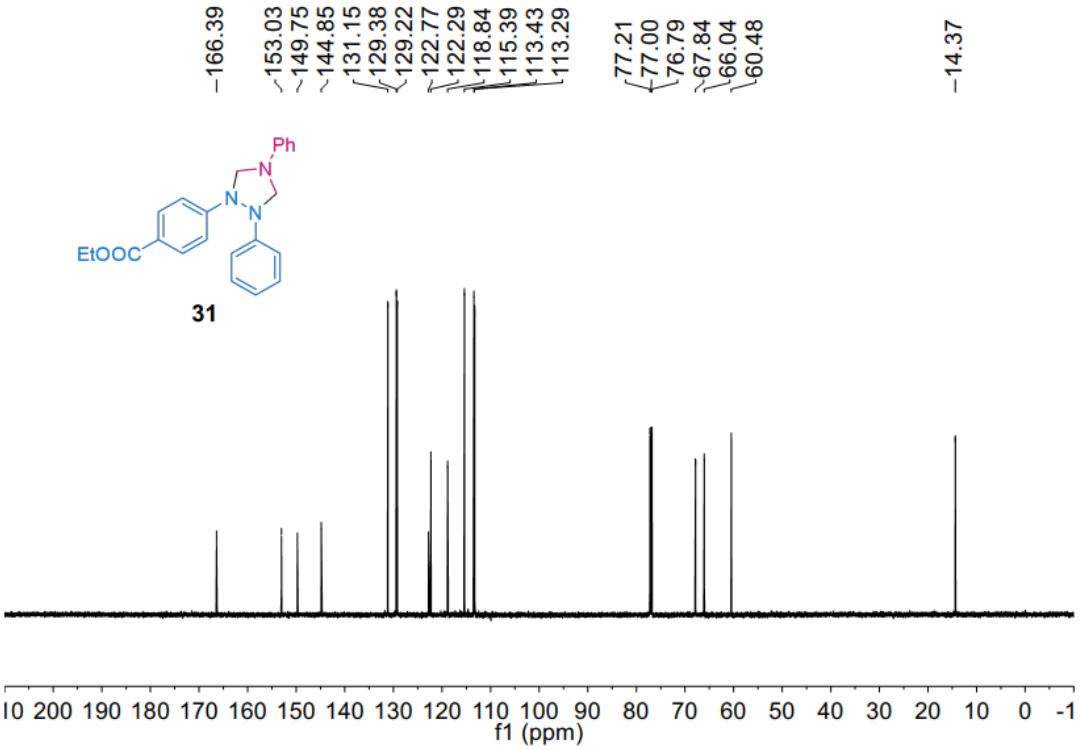
^{19}F NMR spectrum of **30** (376 MHz, $CDCl_3$):



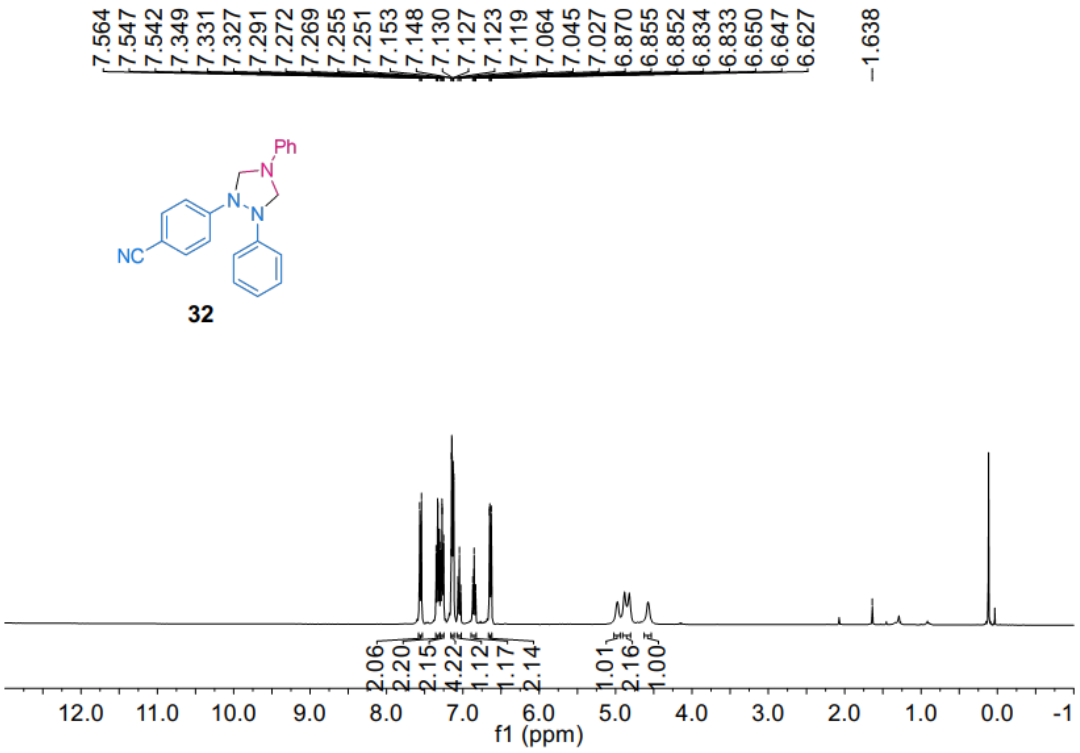
^{1}H NMR spectrum of **31** (400 MHz, $CDCl_3$):



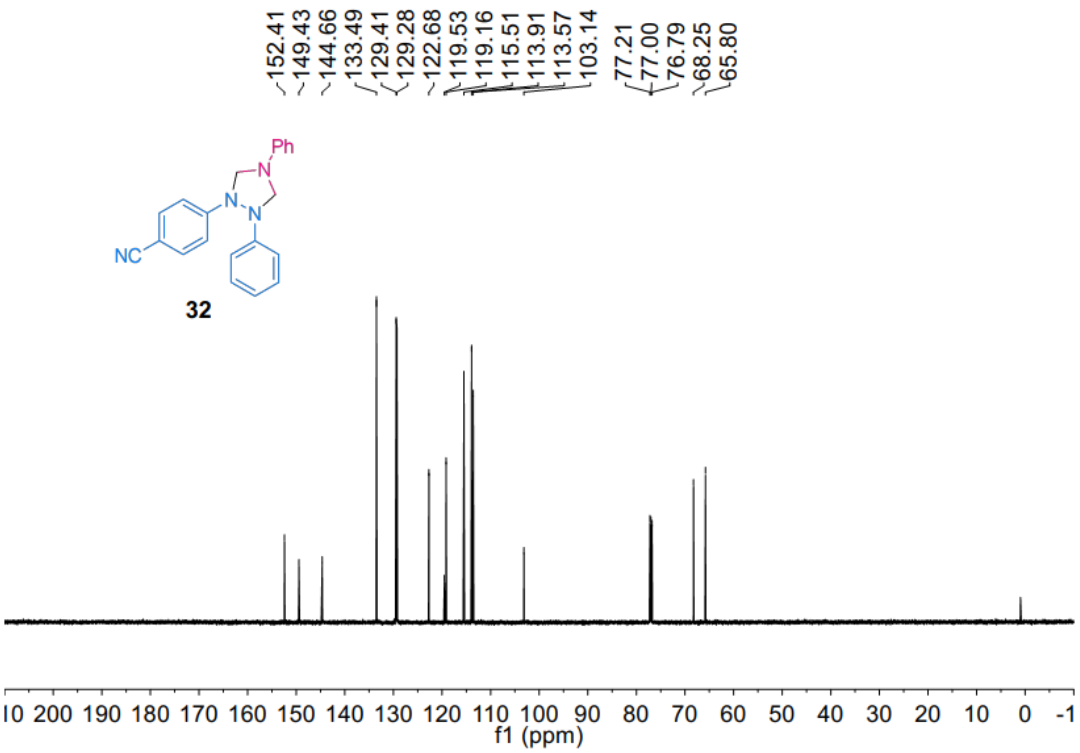
^{13}C NMR spectrum of **31** (150 MHz, $CDCl_3$):



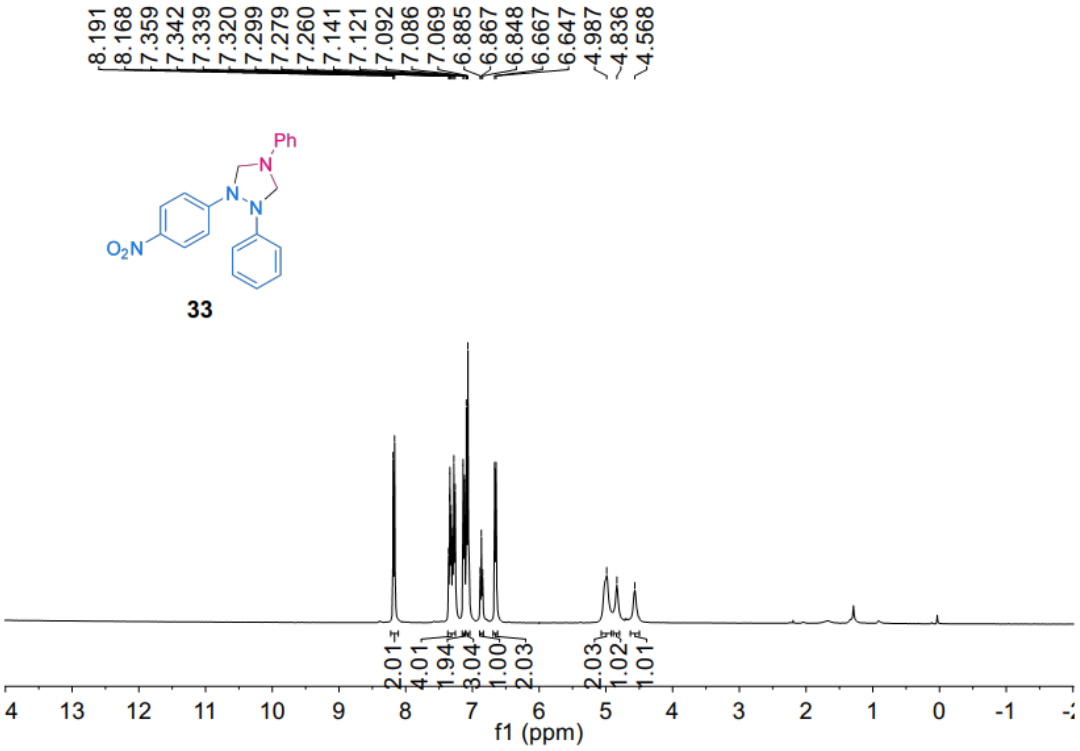
^{1}H NMR spectrum of **32** (400 MHz, $CDCl_3$):



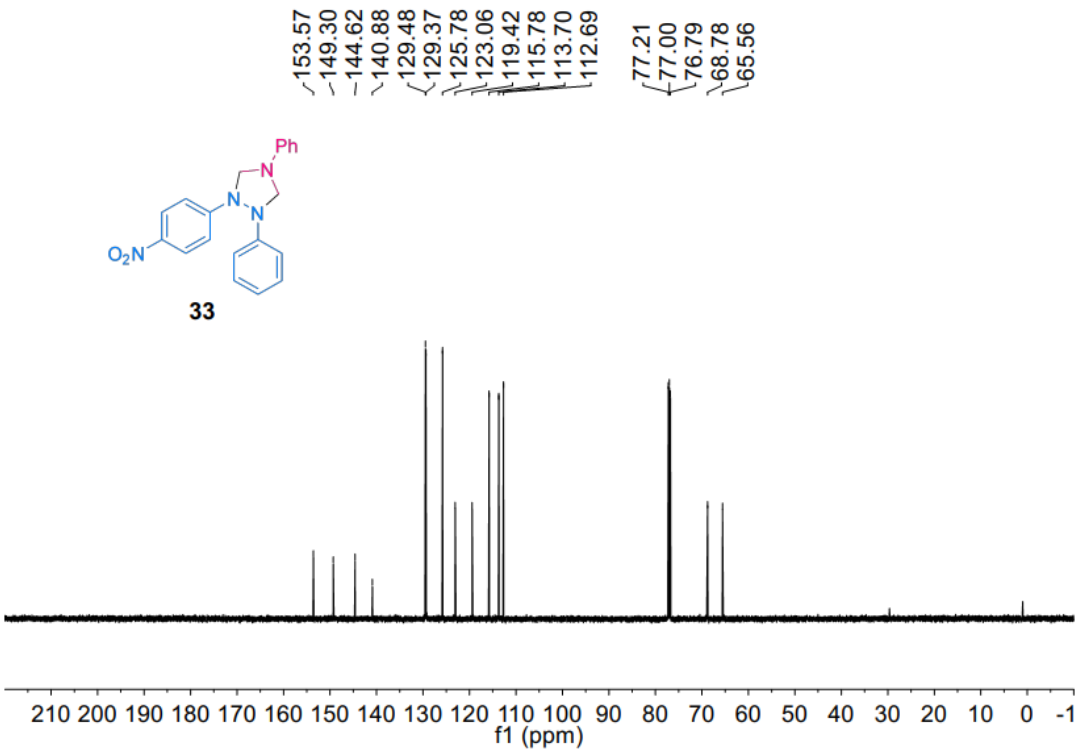
^{13}C NMR spectrum of **32** (150 MHz, $CDCl_3$):



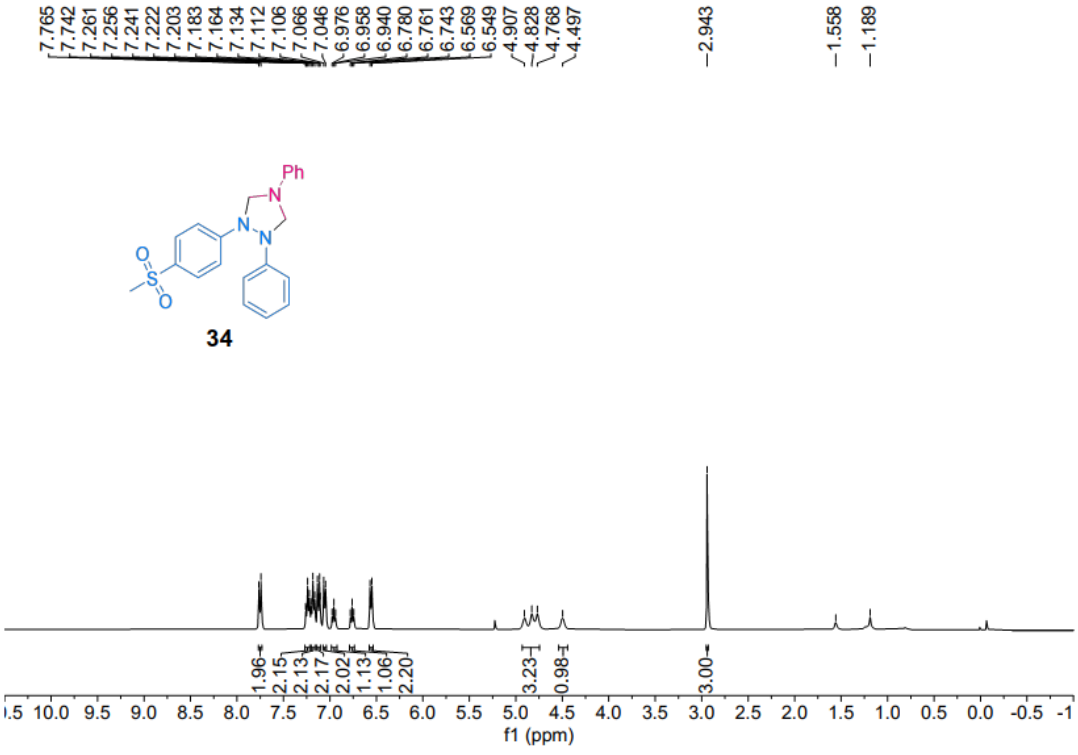
^{1}H NMR spectrum of **33** (400 MHz, $CDCl_3$):



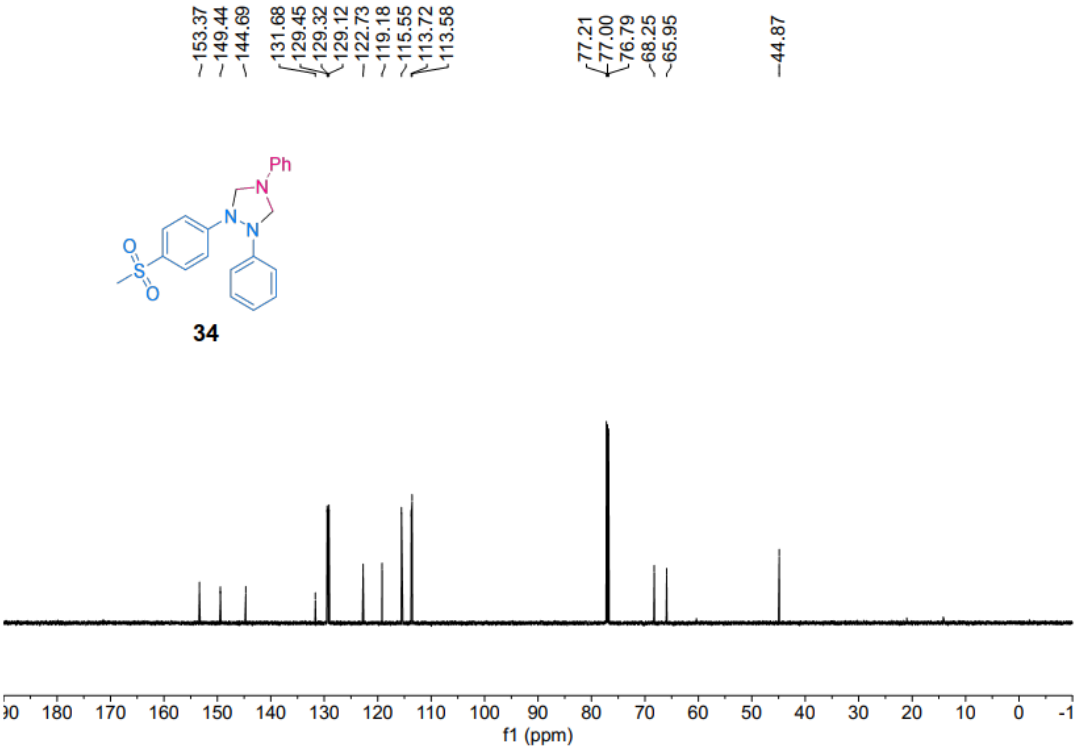
^{13}C NMR spectrum of **33** (150 MHz, $CDCl_3$):



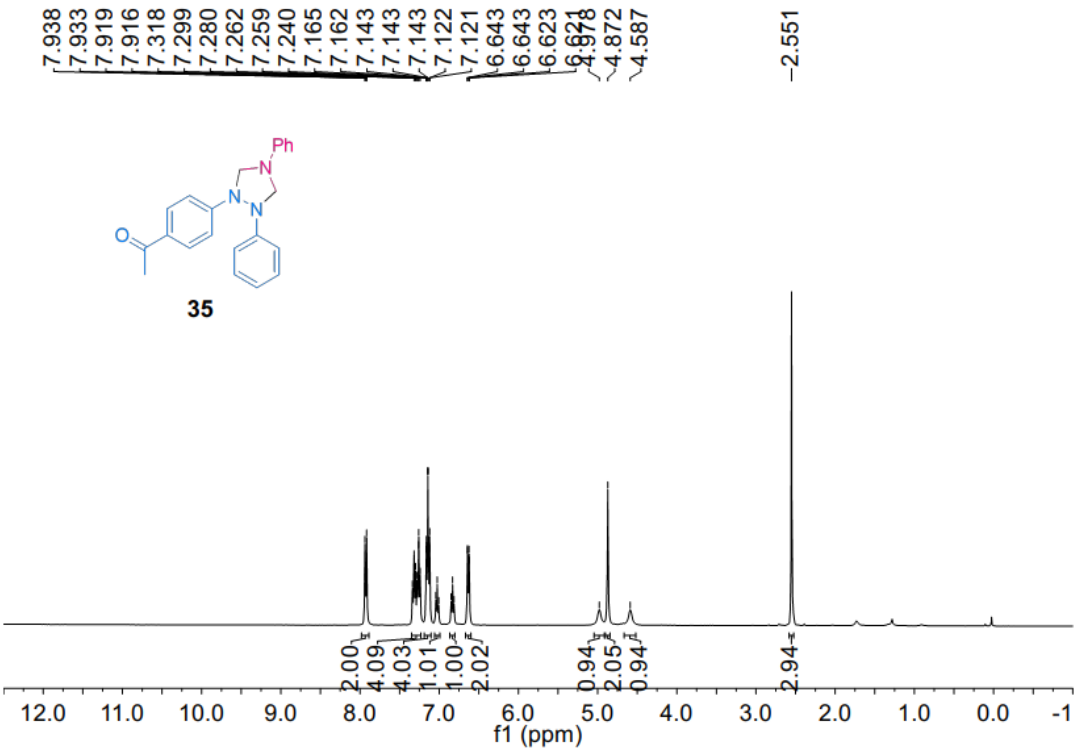
^{1}H NMR spectrum of **34** (400 MHz, $CDCl_3$):



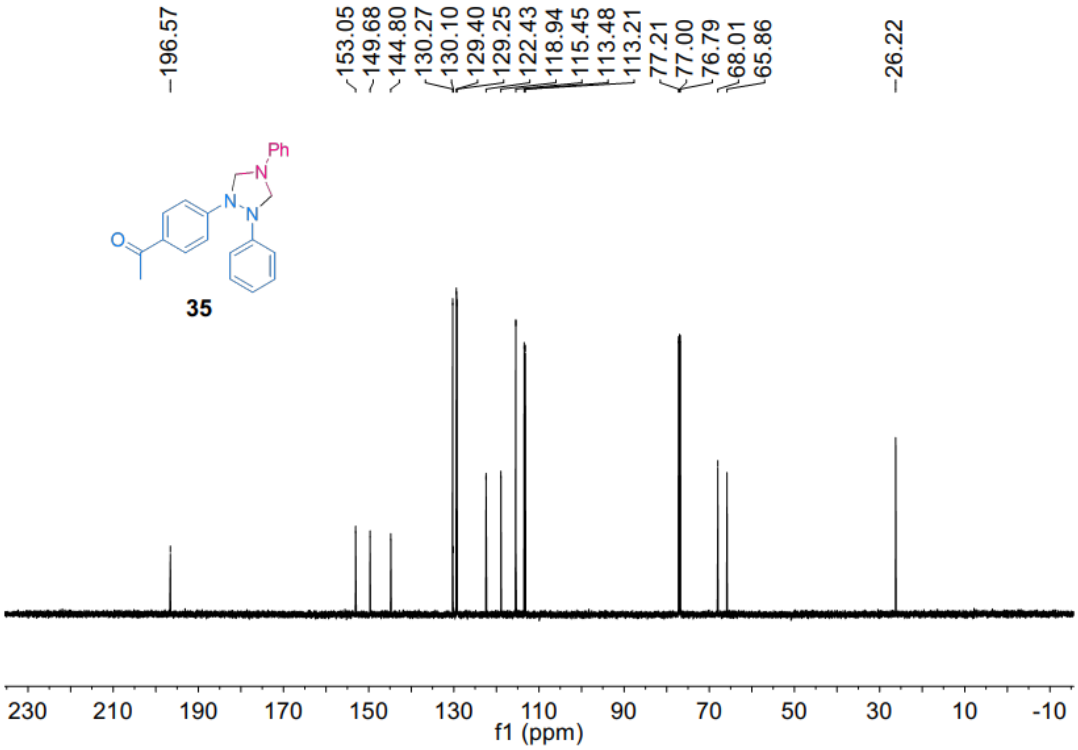
^{13}C NMR spectrum of **34** (150 MHz, $CDCl_3$):



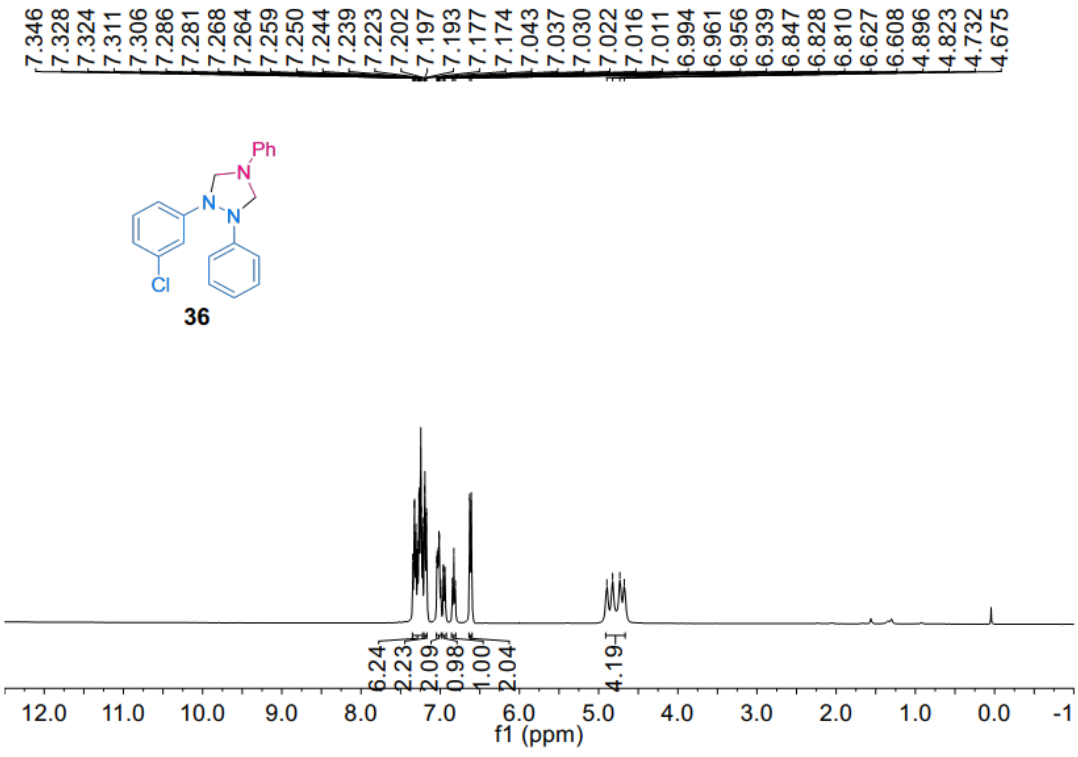
^{1}H NMR spectrum of **35** (400 MHz, $CDCl_3$):



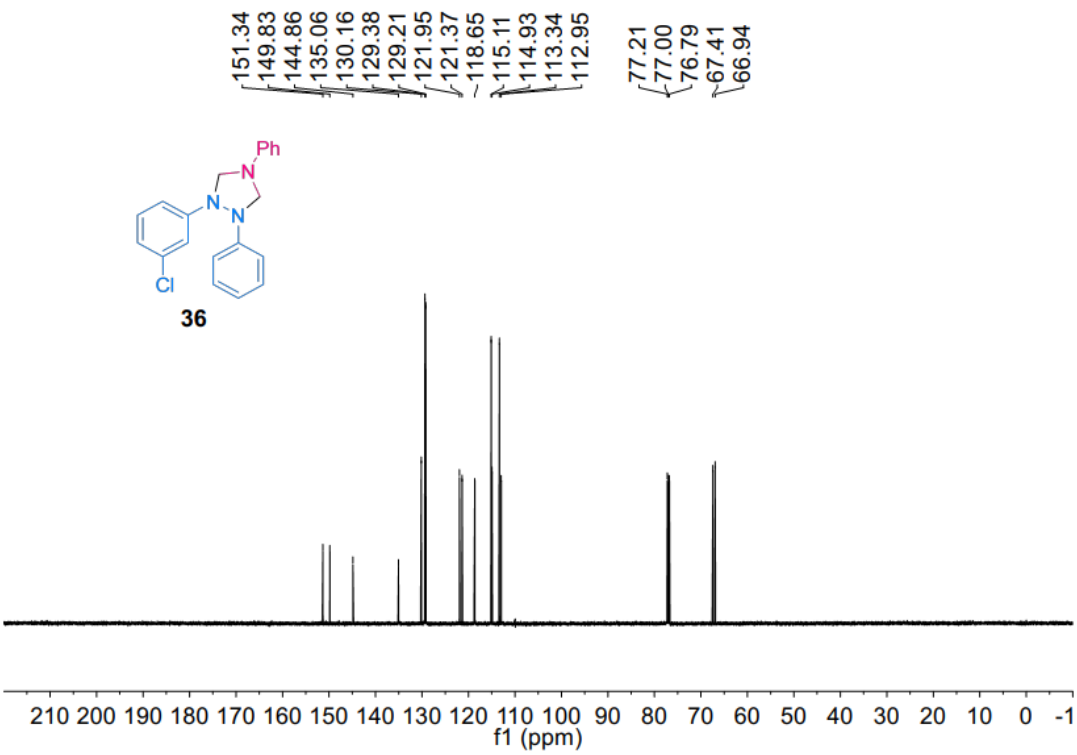
^{13}C NMR spectrum of **35** (150 MHz, $CDCl_3$):



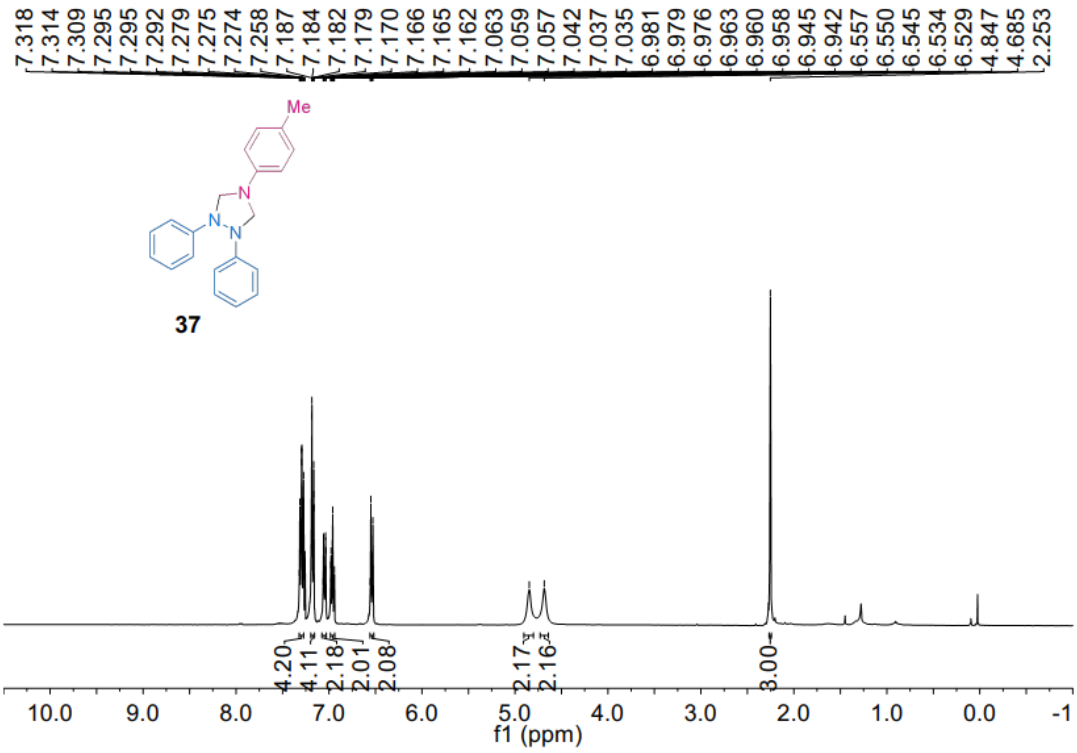
^{1}H NMR spectrum of **36** (400 MHz, $CDCl_3$):



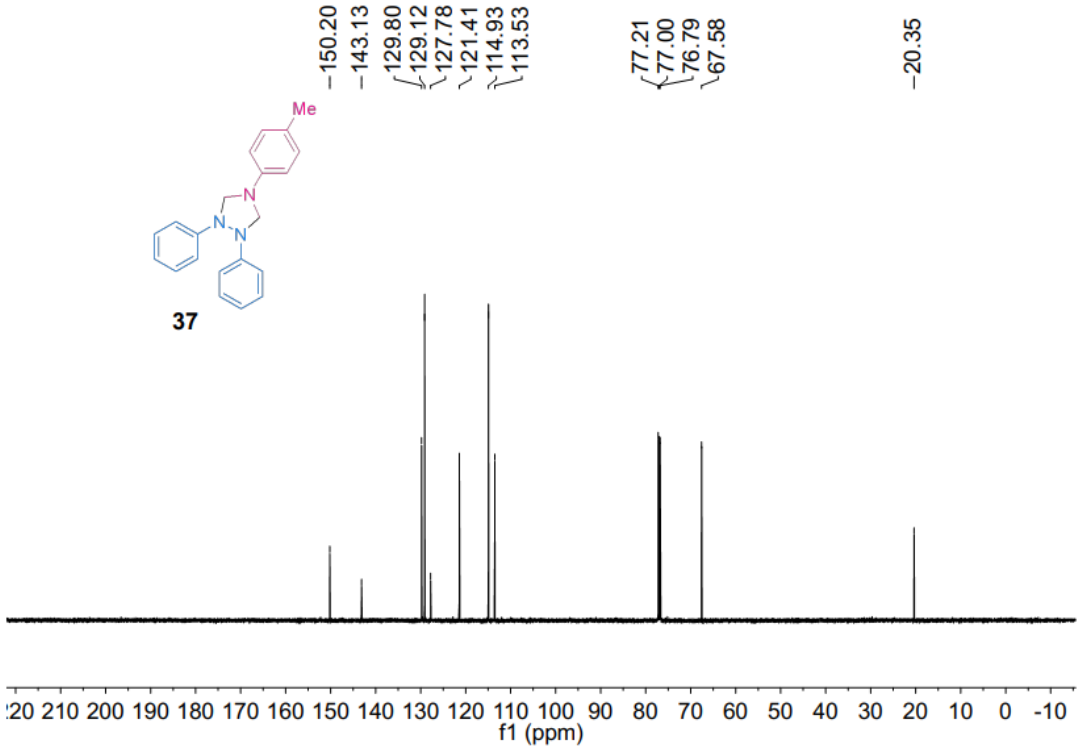
^{13}C NMR spectrum of **36** (150 MHz, $CDCl_3$):



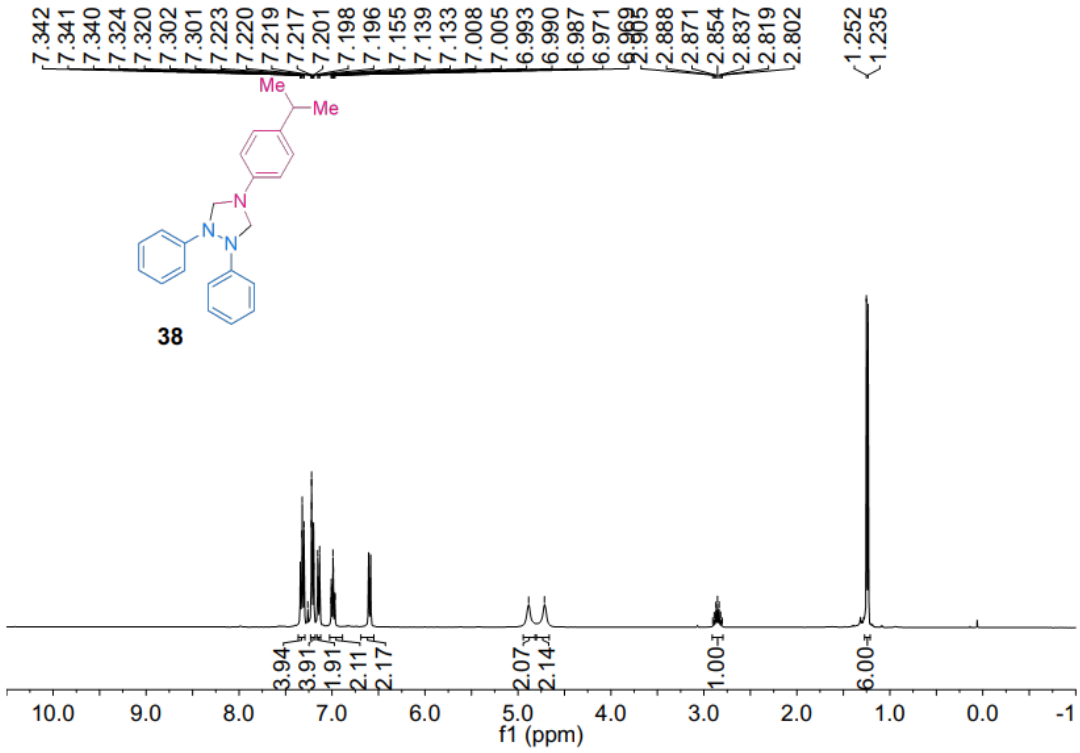
^{1}H NMR spectrum of **37** (400 MHz, $CDCl_3$):



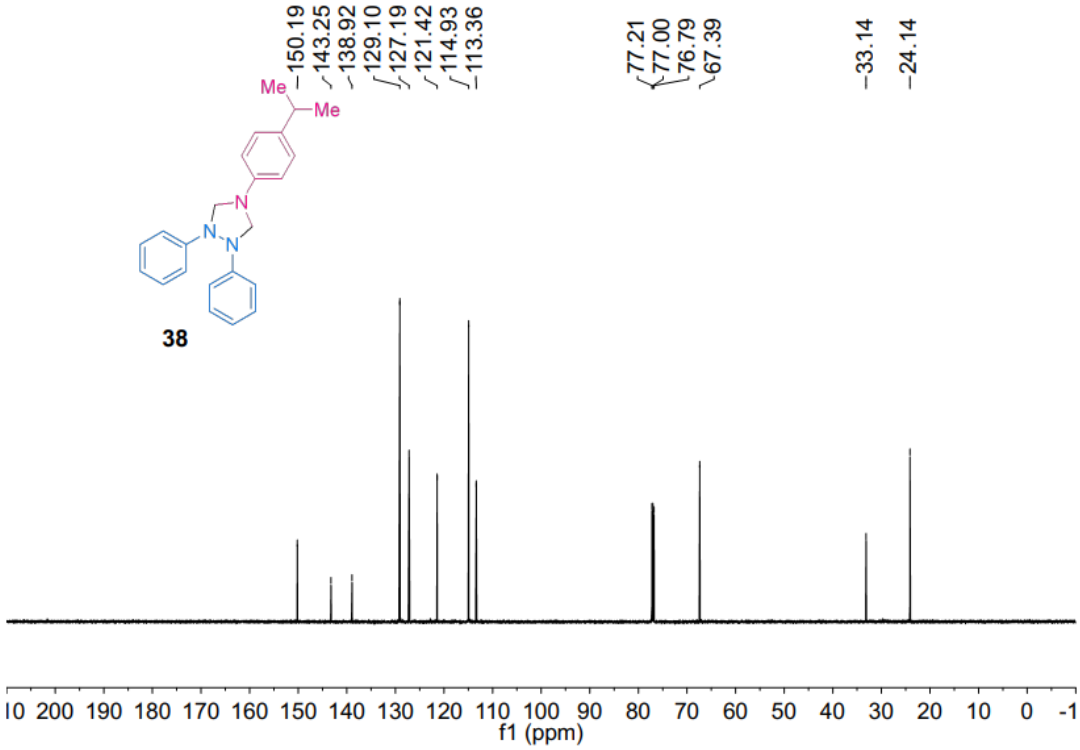
^{13}C NMR spectrum of **37** (150 MHz, $CDCl_3$):



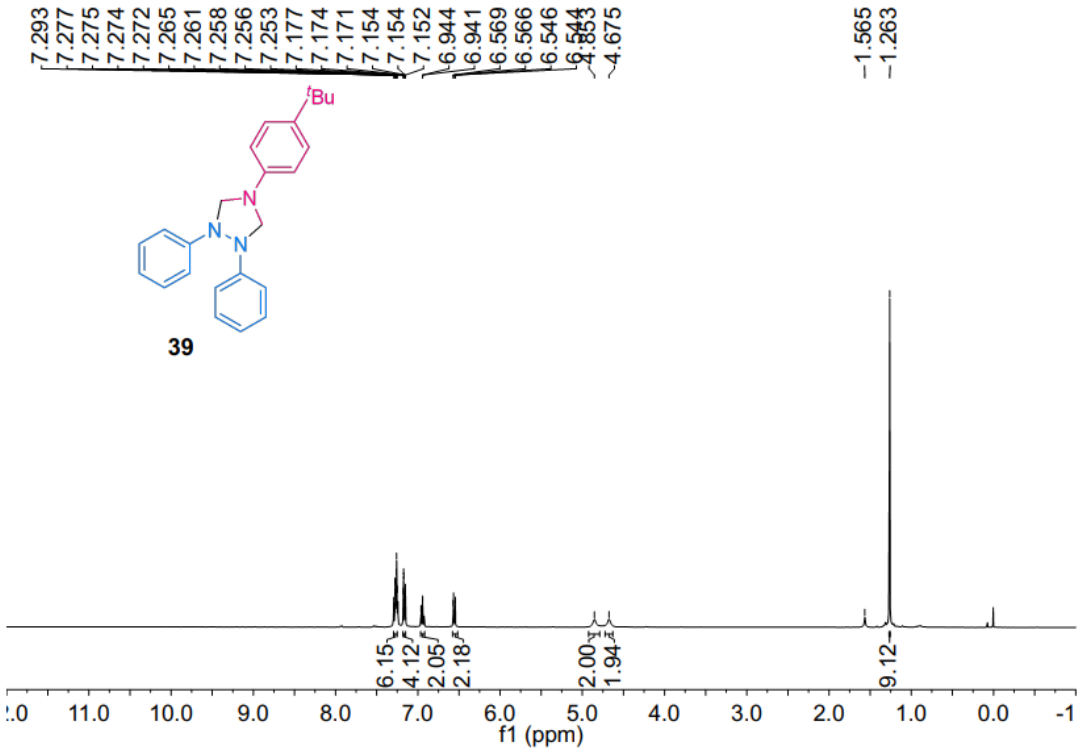
^{1}H NMR spectrum of **38** (400 MHz, $CDCl_3$):



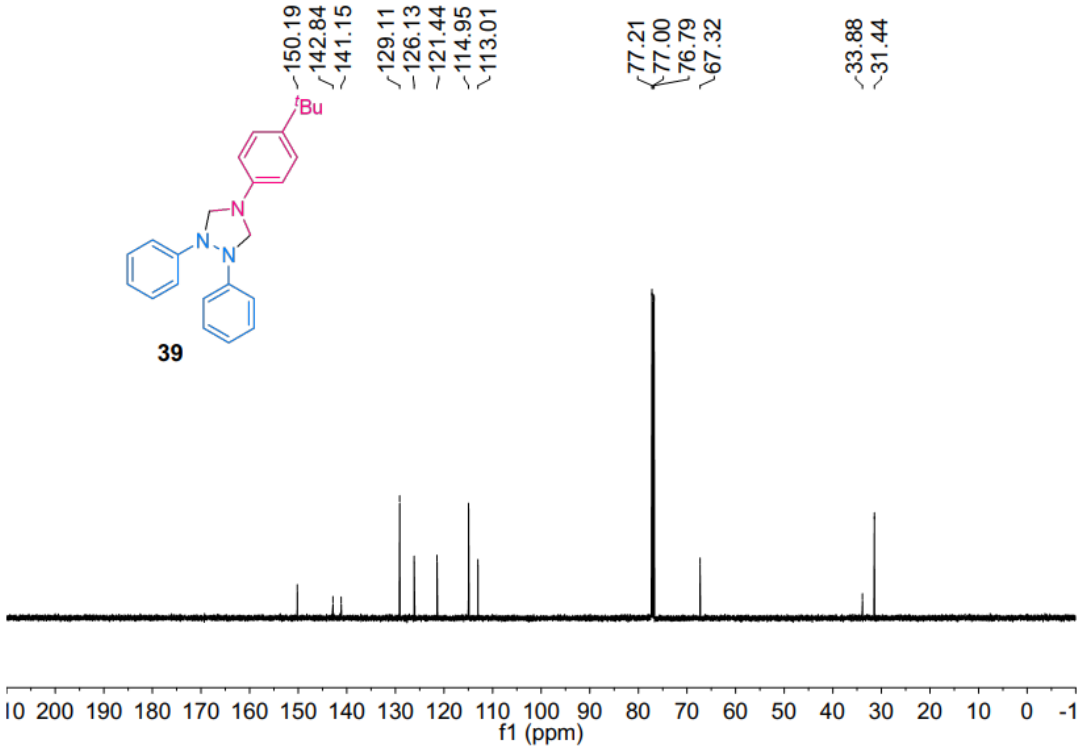
^{13}C NMR spectrum of **38** (150 MHz, $CDCl_3$):



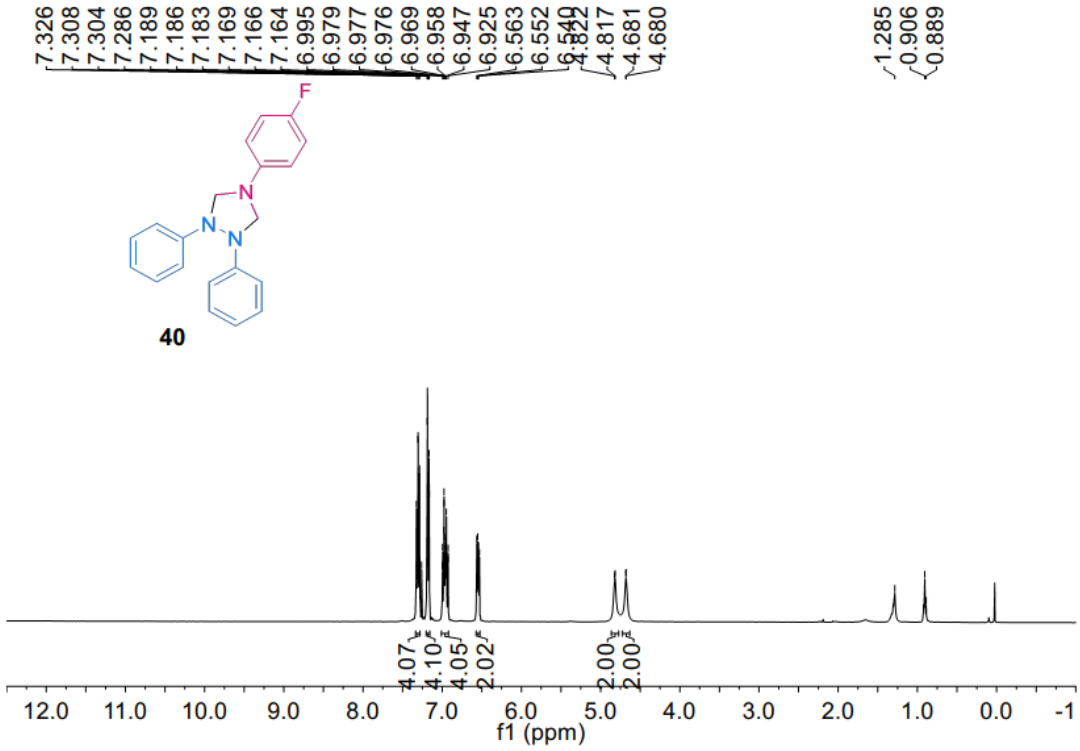
^{1}H NMR spectrum of **39** (400 MHz, $CDCl_3$):



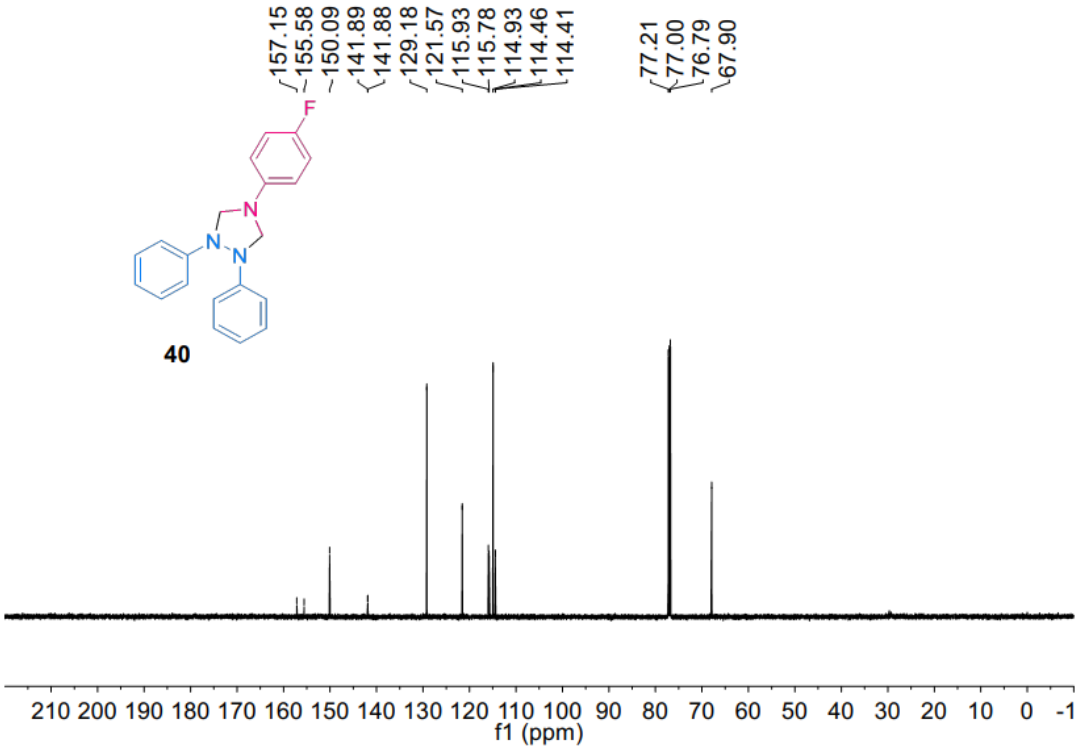
^{13}C NMR spectrum of **39** (150 MHz, $CDCl_3$):



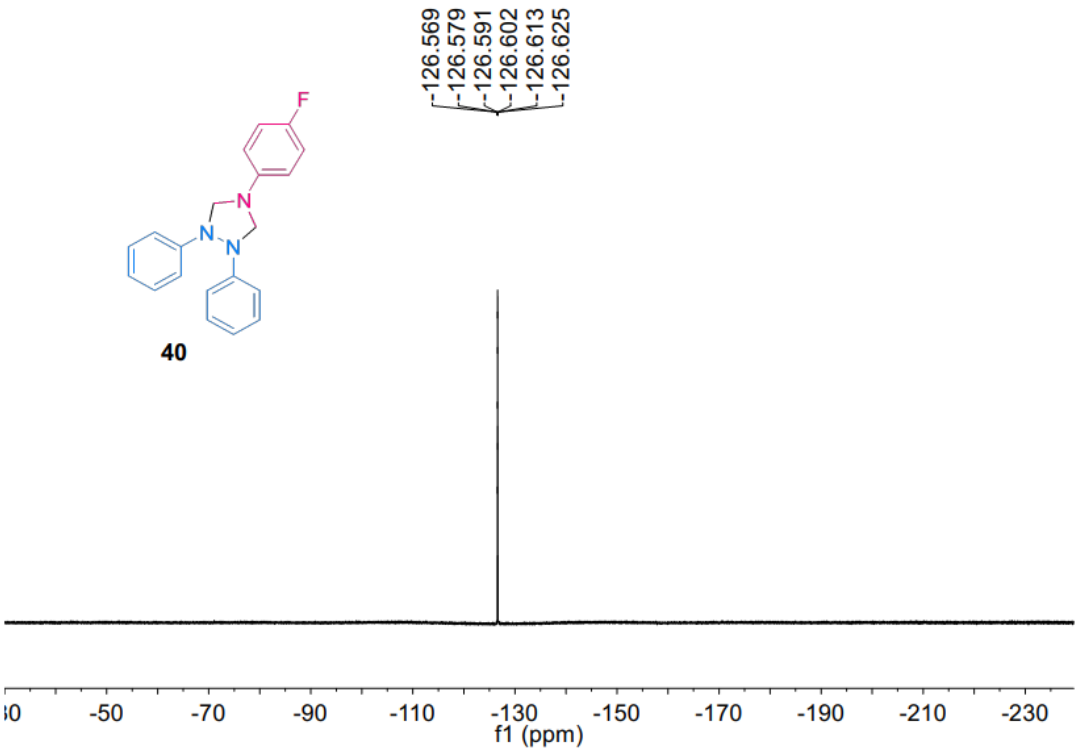
^{1}H NMR spectrum of **40** (400 MHz, $CDCl_3$):



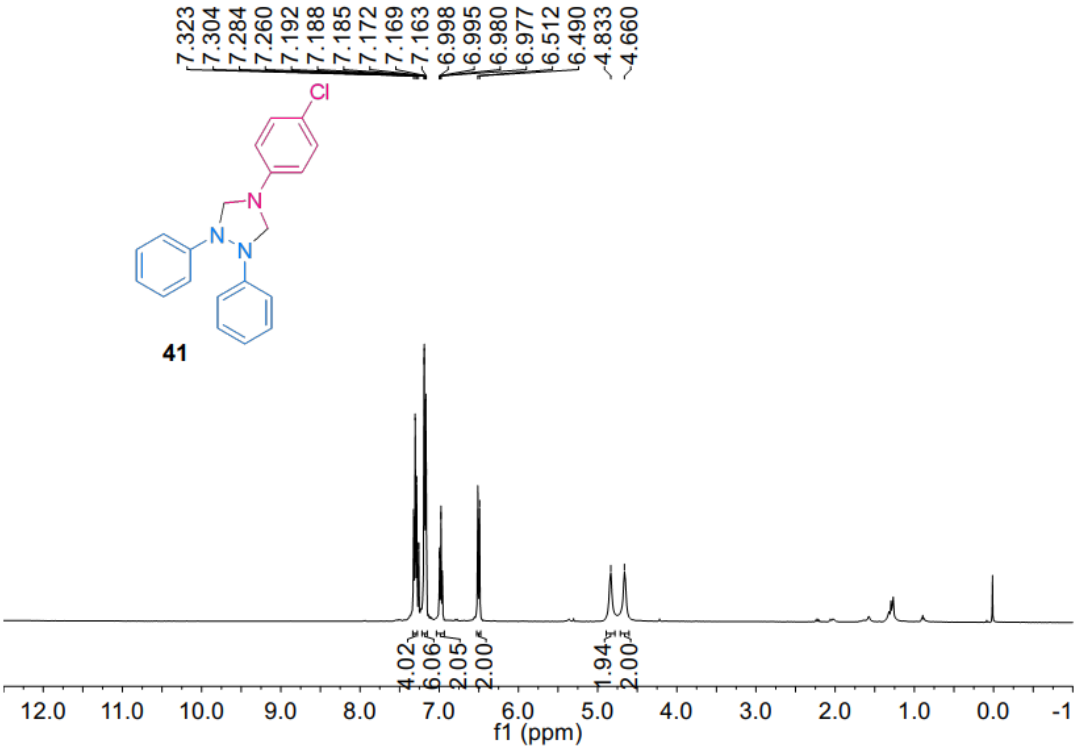
^{13}C NMR spectrum of **40** (150 MHz, $CDCl_3$):



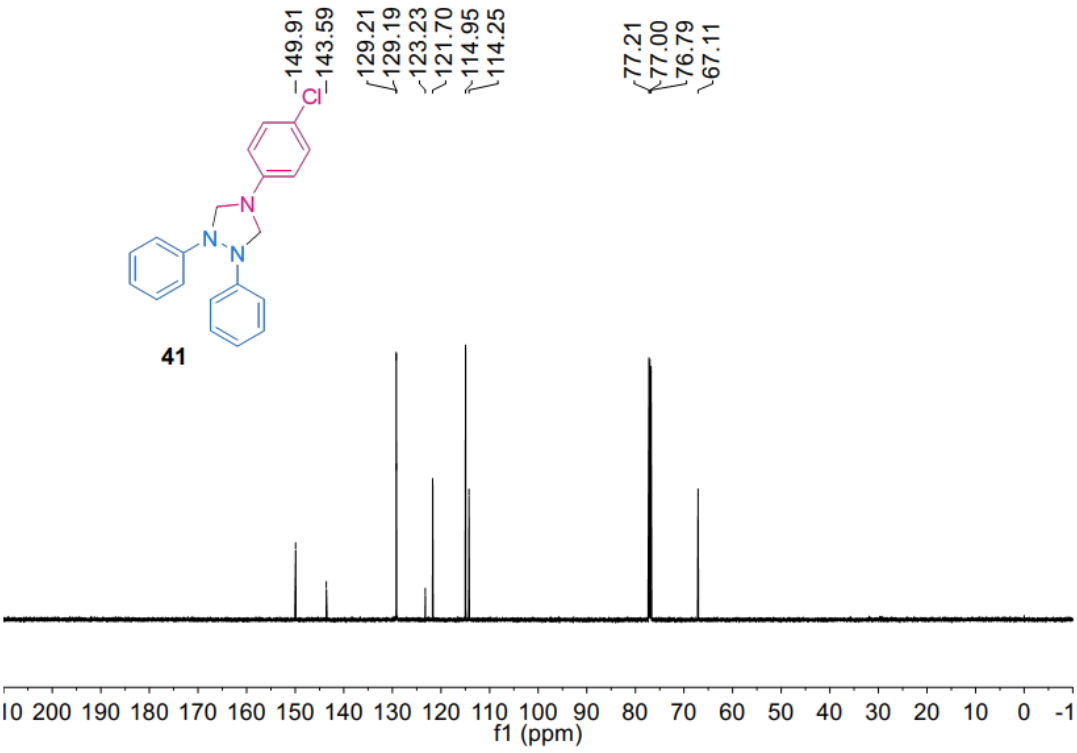
^{19}F NMR spectrum of **40** (376 MHz, $CDCl_3$):



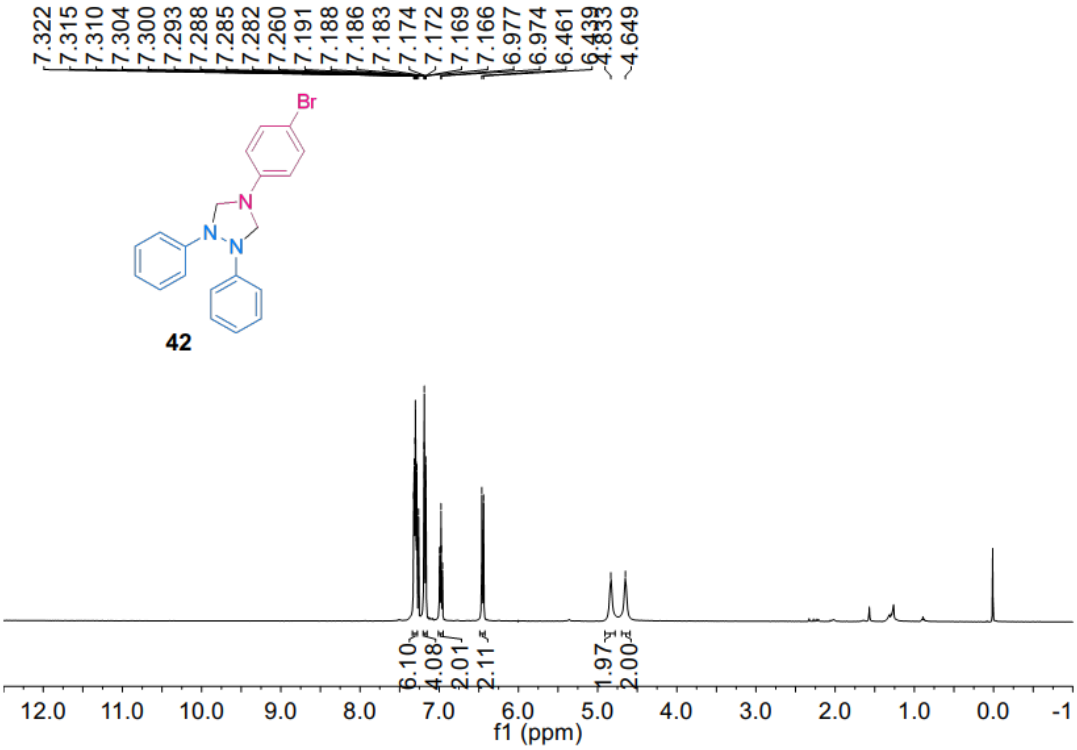
^{1}H NMR spectrum of **41** (400 MHz, $CDCl_3$):



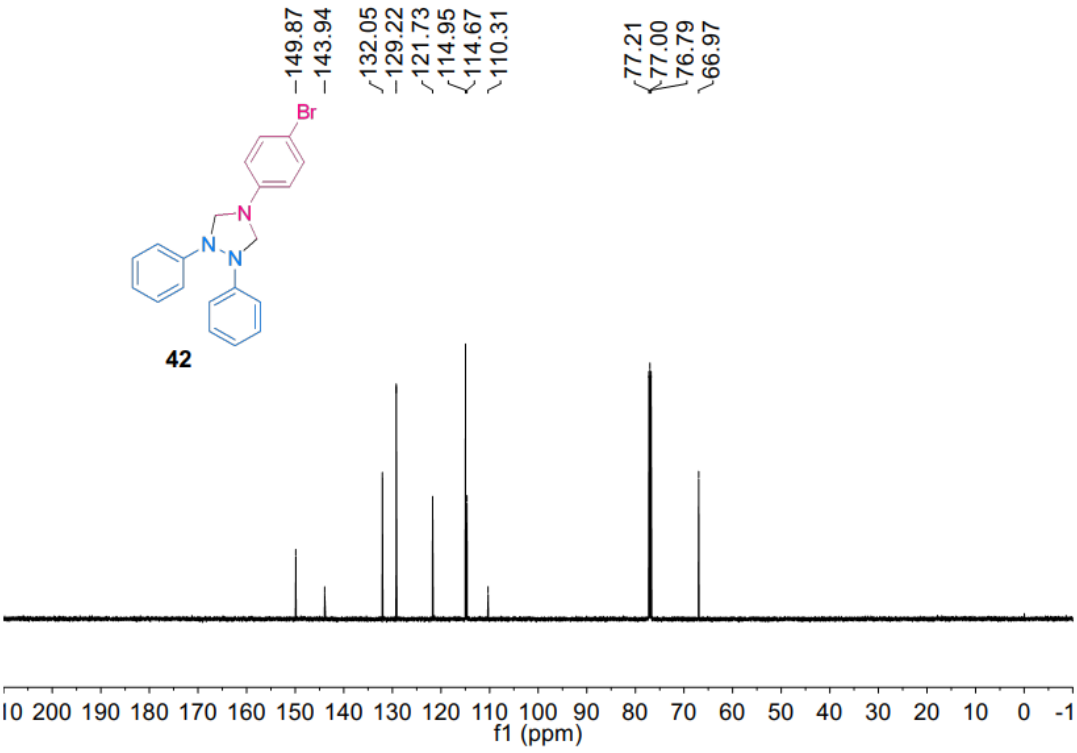
^{13}C NMR spectrum of **41** (150 MHz, $CDCl_3$):



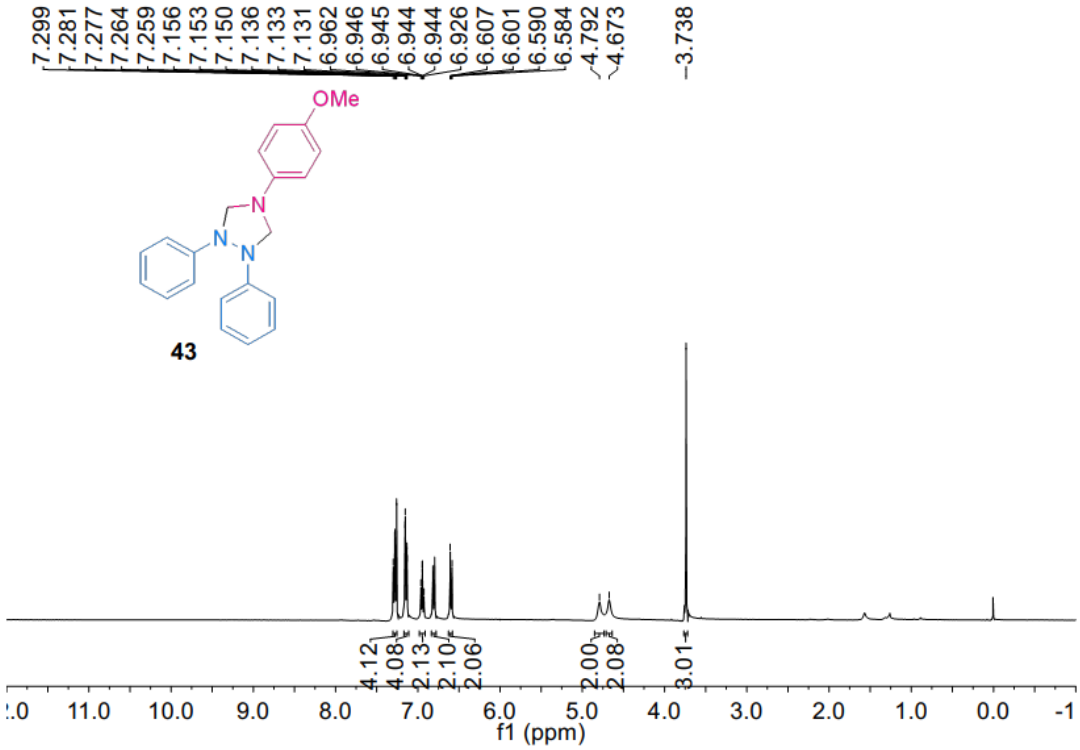
^{1}H NMR spectrum of **42** (400 MHz, $CDCl_3$):



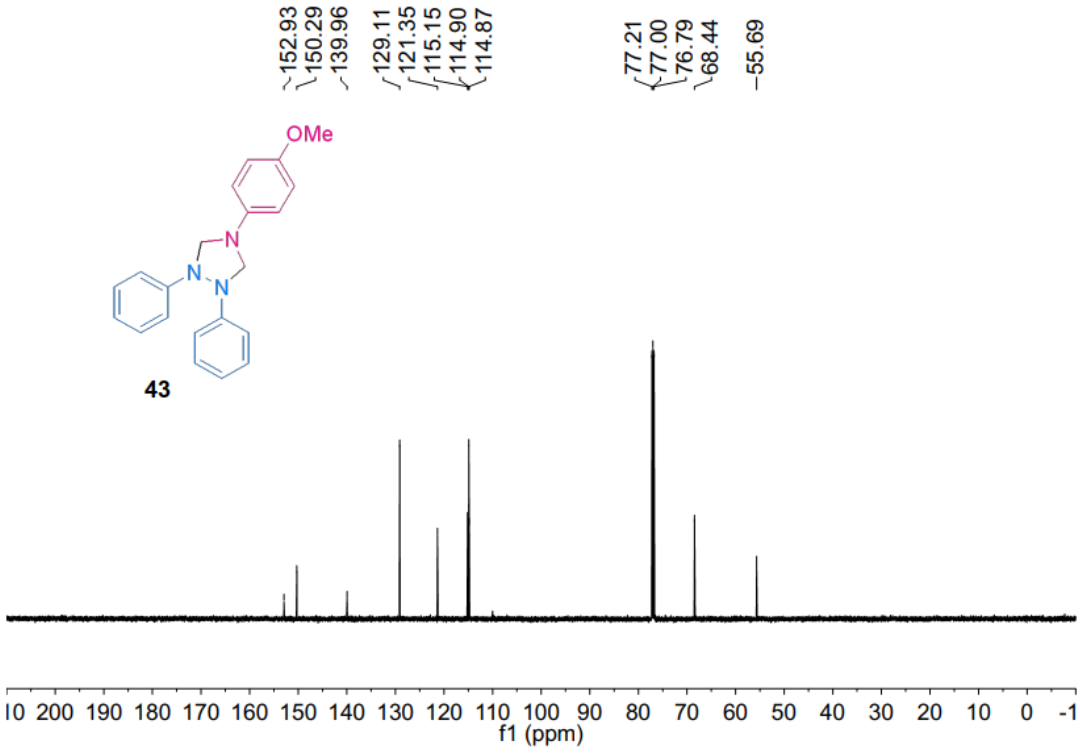
^{13}C NMR spectrum of **42** (150 MHz, $CDCl_3$):



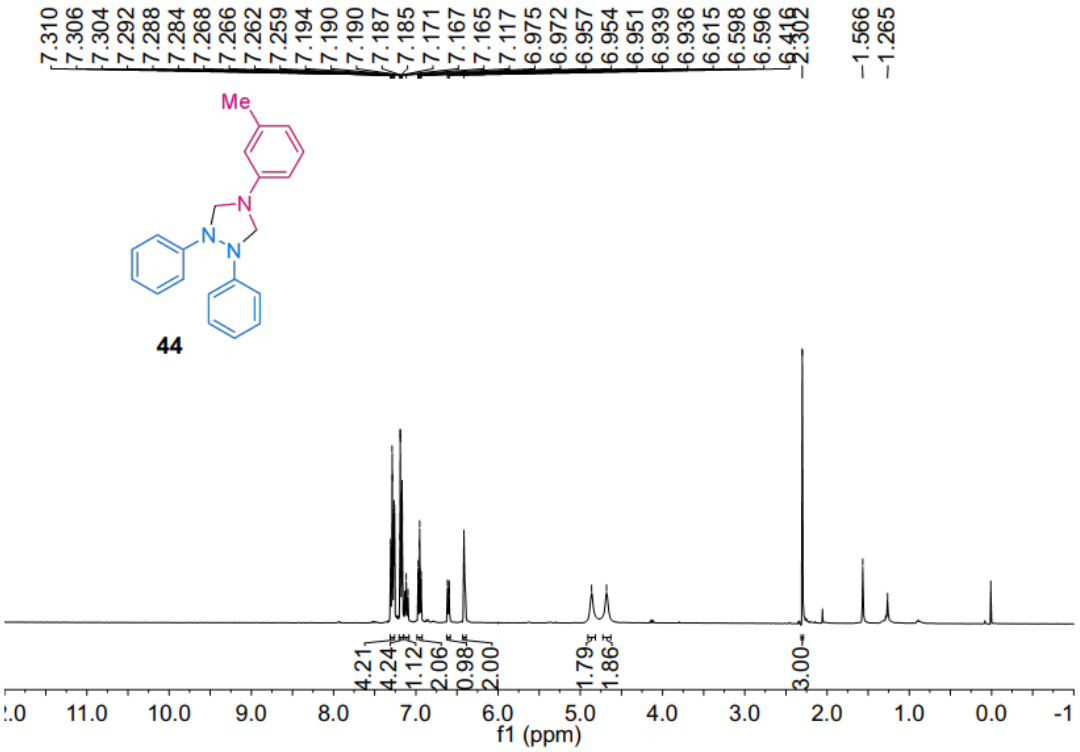
^{1}H NMR spectrum of **43** (400 MHz, $CDCl_3$):



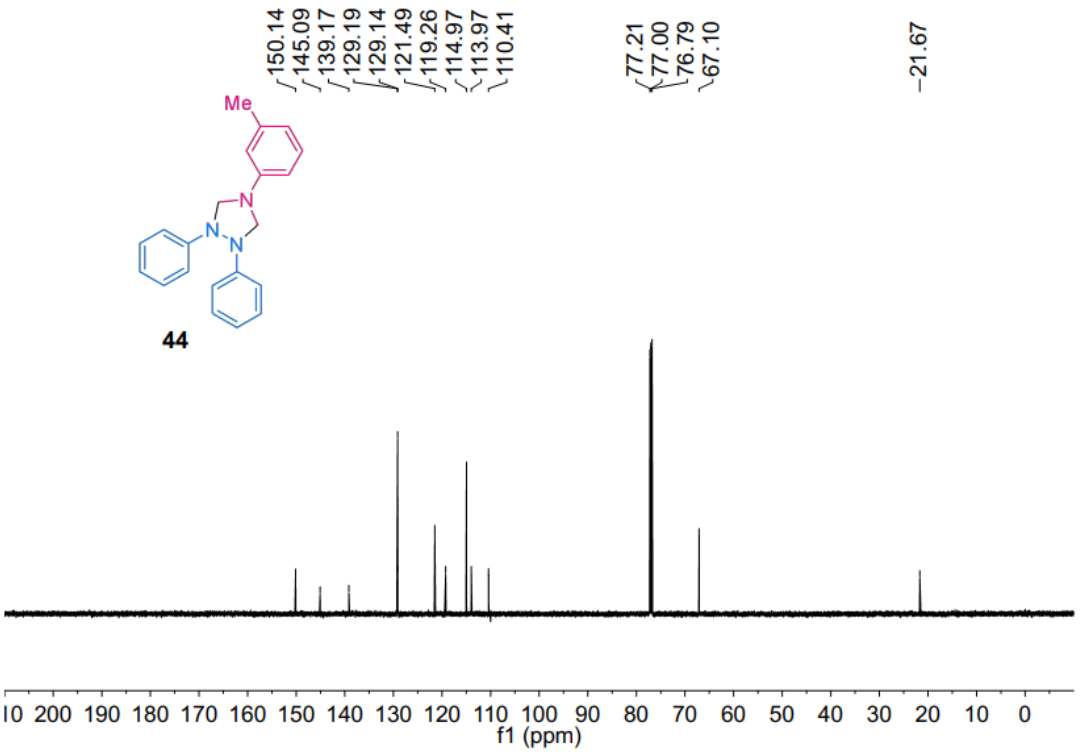
^{13}C NMR spectrum of **43** (150 MHz, $CDCl_3$):



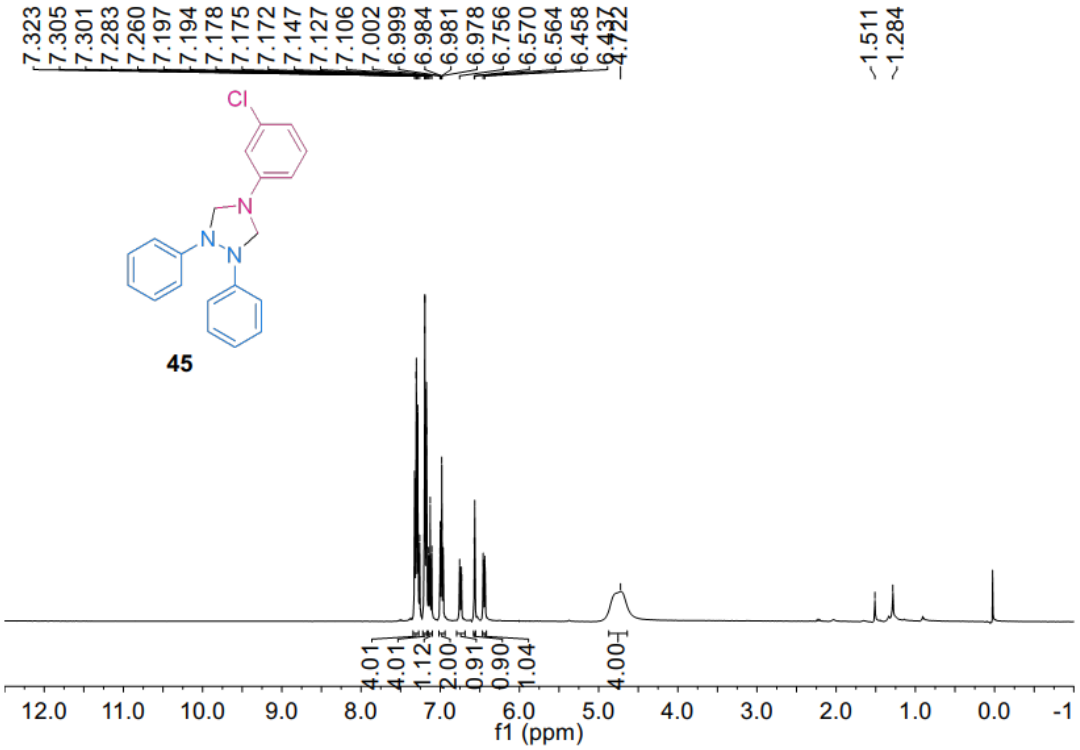
^{1}H NMR spectrum of **44** (400 MHz, $CDCl_3$):



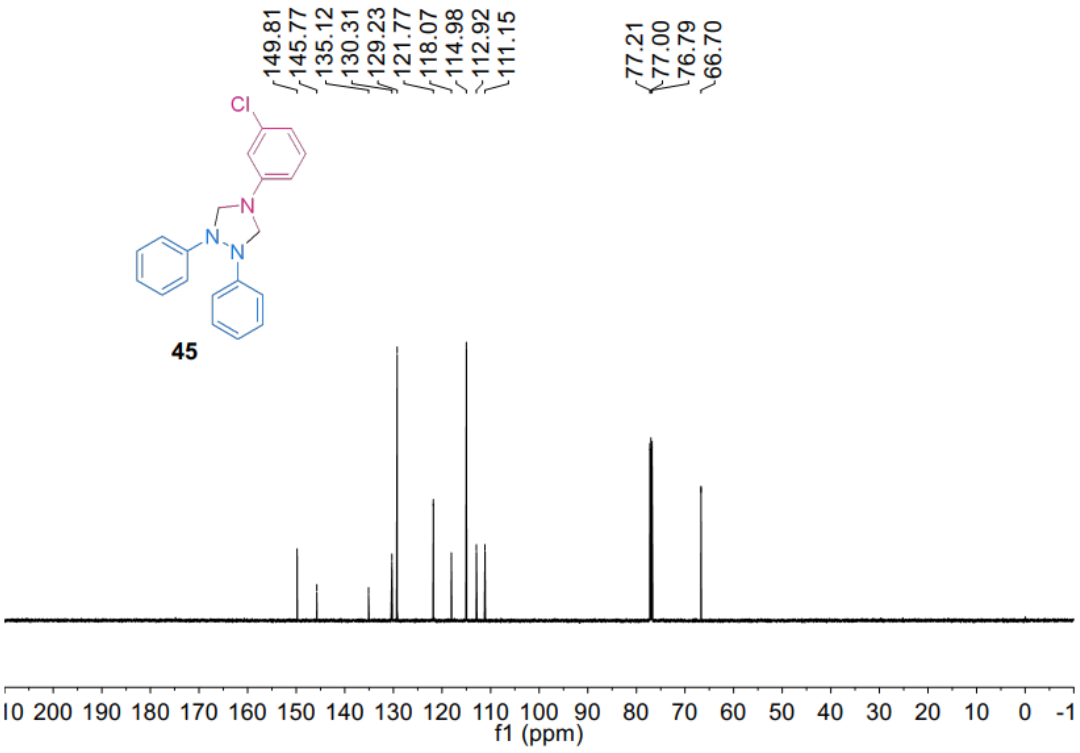
^{13}C NMR spectrum of **44** (150 MHz, $CDCl_3$):



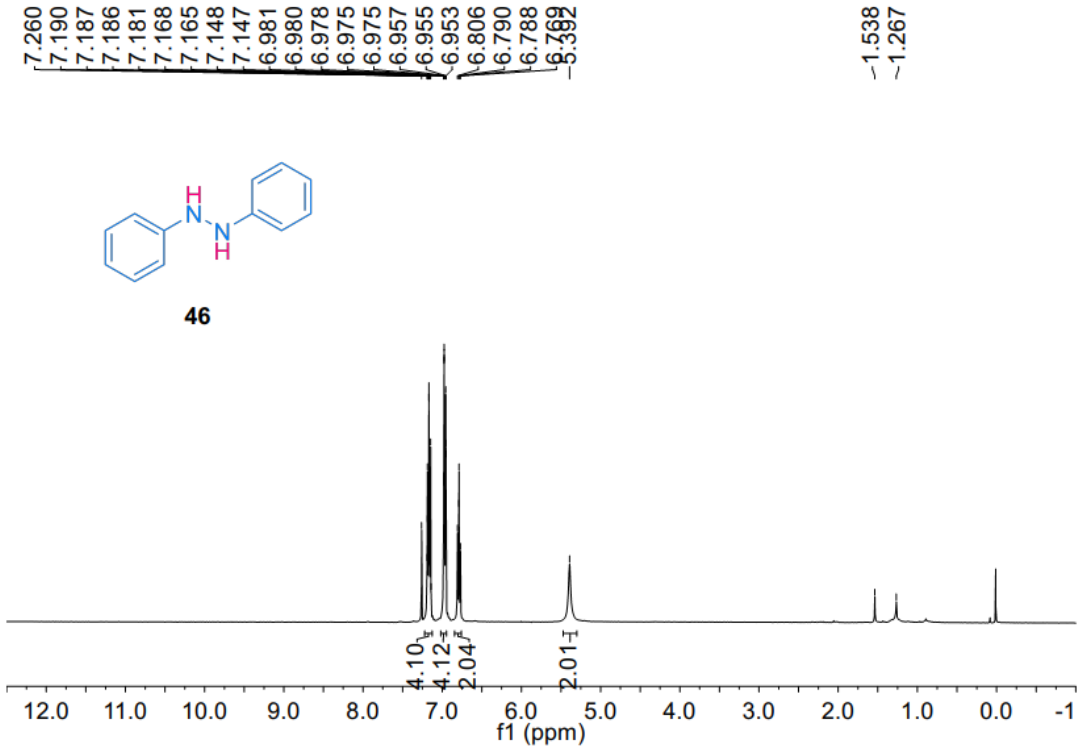
^{1}H NMR spectrum of **45** (400 MHz, $CDCl_3$):



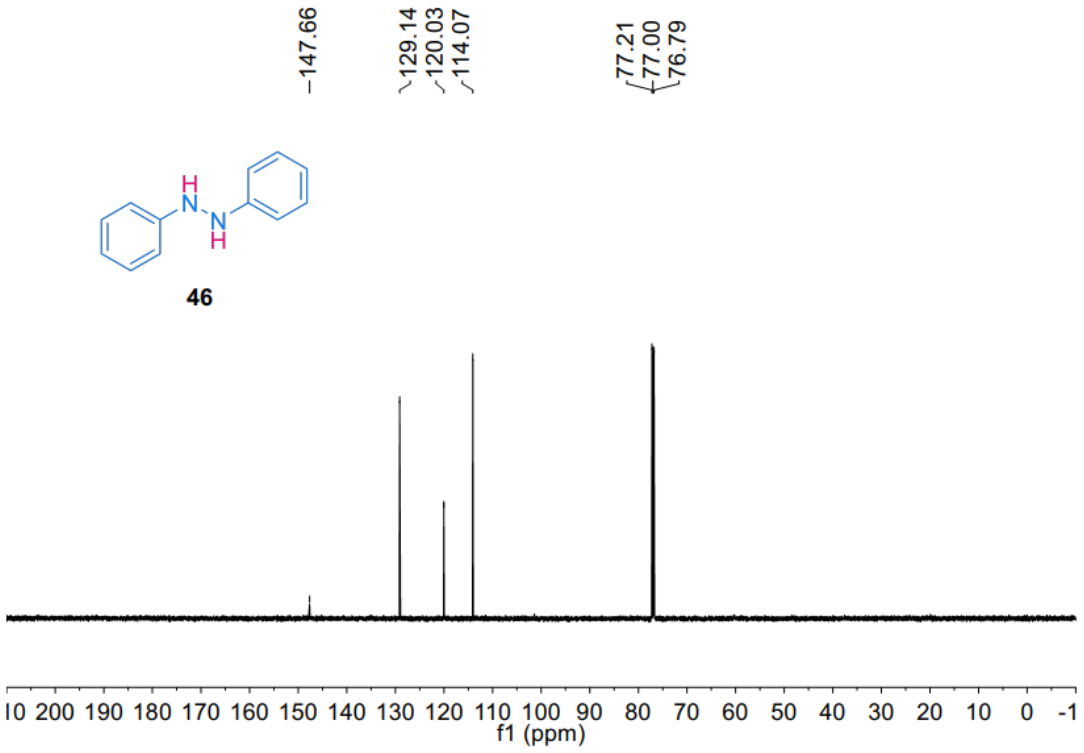
^{13}C NMR spectrum of **45** (150 MHz, $CDCl_3$):



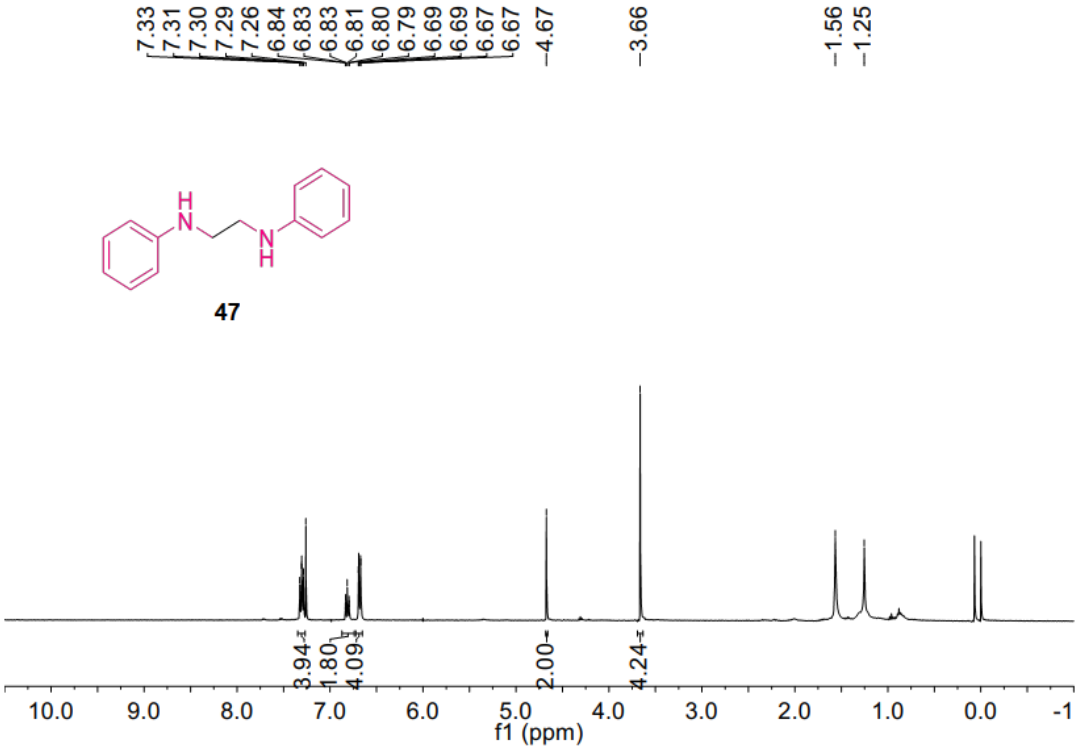
^{1}H NMR spectrum of **46** (400 MHz, $CDCl_3$):



^{13}C NMR spectrum of **46** (150 MHz, $CDCl_3$):



^{1}H NMR spectrum of **47** (400 MHz, $CDCl_3$):



^{13}C NMR spectrum of **47** (150 MHz, $CDCl_3$):