

**Electrochemical C(sp²)-H/N-H “Formal” Cross-Dehydrogenative
Coupling of Olefins with Benzotriazoles for Synthesis of *N*-Vinyl
Benzotriazoles**

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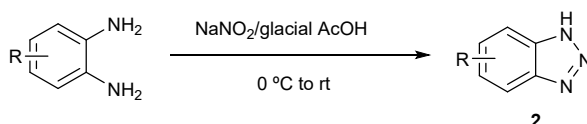
1. General methods

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without further purification. Reactions were monitored by thin-layer chromatography (TLC) with Haiyang GF 254 silica gel plates (Qingdao Haiyang chemical industry Co Ltd, Qingdao, China) using UV light and vanillic aldehyde or phosphomolybdic acid as visualizing agents. Flash column chromatography was performed using 200–300 mesh silica gel at increased pressure. ^1H NMR spectra, ^{19}F NMR spectra and ^{13}C NMR spectra were respectively recorded on 600 MHz, 565 MHz, 400 MHz, 151 MHz and 101 MHz NMR spectrometers. Chemical shifts (δ) were expressed in ppm with TMS as the internal standard, and coupling constants (J) were reported in Hz. High-resolution mass spectra were obtained by using ESI ionization sources (quadrupole time-of-flight mass spectrometer, Bruker Impact II, Bremen, Germany). Electron paramagnetic resonance (EPR) spectra were recorded on a Bruker EMXplus-9.5/12 spectrometer. Cyclic voltammograms were obtained on a CHI 700E potentiostat (CH Instruments, Inc.).

Abbreviations: DMF = *N,N*-dimethylformamide, DMSO = dimethyl sulfoxide, DCE = dichloroethane, DCM = dichloromethane, MeCN = acetonitrile, THF = tetrahydrofuran, EA = ethyl acetate, HFIP = 1,1,1,3,3,3-hexafluoro-2-propanol, AcOH = Acetic acid, DMPO = 3,4-dihydro-2,2-dimethyl-2*H*-pyrrole 1-oxide, n.r. = no reaction, n.d. = not detected.

2. Experimental procedures

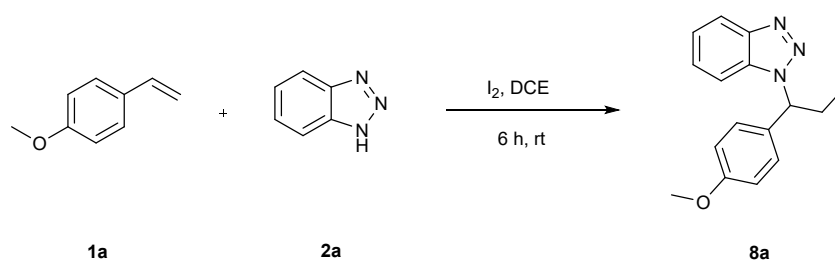
2.1. General procedure for the preparation of substituted benzotriazoles **2**¹



1,2-phenylenediamine derivative (3.26 mmol) was dissolved in a mixture of 0.45 mL of glacial acetic acid and 1.2 mL of water and cooled to 4 °C. A solution of sodium nitrite (0.26 g, 3.76 mmol) in 1 mL of water was added. The reaction temperature was raised to 50 °C for 30 min, then allowed

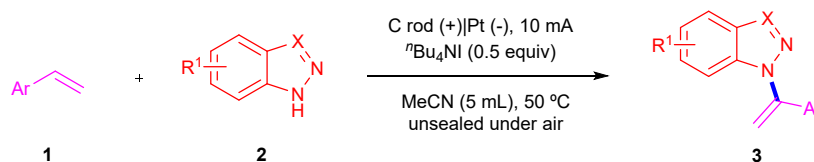
to cool to r.t. and stirred at this temperature for 12 h. The mixture was cooled to 0 °C for 1 h. Produced precipitate was collected by suction filtration, and washed with water, and dried to provide substituted benzotriazole **2**.

2.2. General procedure for the preparation of **8a**²



1-methoxy-4-vinylbenzene **1a** (0.5 mmol), benzotriazole **2a** (1.0 mmol), I₂ (0.5 mmol) were added to DCE (2 mL). The mixture was stirred at 20 °C for 6 h (monitored by TLC), quenched with water (10 mL), extracted with dichloromethane (5×3 mL), and dried over anhydrous Na₂SO₄. The solvent was removed under reduced pressure, and the residue was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate as the eluent to give the target product **8a**.

2.3. General procedure for the electrochemical synthesis of products **3**



To an undivided beaker-type electrolysis cell (10 mL) equipped with a magnetic stirring bar was added olefin **1** (0.2 mmol, 1 equiv), azole **2** (0.8 mmol, 4 equiv) and ^tBu₄NI (0.1 mmol, 0.5 equiv) in MeCN (5 mL). A carbon rod electrode (Φ = 0.5 cm) was used as the anode and a platinum plate (1 cm × 1 cm × 0.2 mm) was used as the cathode (the electrodes were immersed 1 cm in the reaction mixture). The reaction mixture was stirred and electrolyzed with a constant current of 10 mA at 50 °C (water bath) in air. After reaction completion (monitored by TLC), the reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate as the eluent to give the target product **3**.

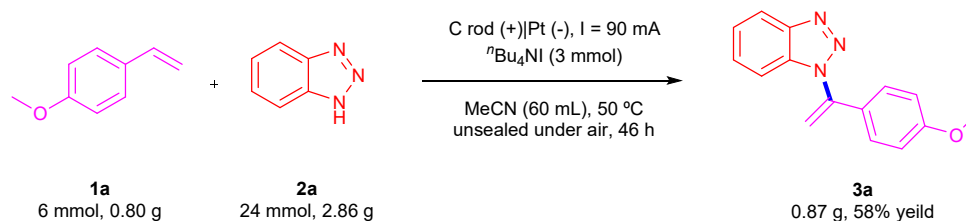


Figure S1 Electrochemical setup used.

The experimental setup consisted of a carbon rod electrode ($\Phi = 0.5$ cm) and a platinum plate (1 cm \times 1 cm \times 0.2 mm), a tube (10 mL) with perforated rubber plugs, an adjustable DC regulated power supply (MS-150V 100 mA), and a magnetic stirrer.

Although the reaction vessel is equipped with a cover, there are holes on the cover for the electrodes to pass through. These holes are not sealed around the electrodes. Additionally, there are additional gaps between the screw outer cap and the disc-shaped inner cover. As a result, the reaction system is not sealed, allowing it to be open to the atmosphere, and therefore, enabling the escape of the hydrogen gas produced during the reaction.

2.4. Scale-up experiment for the synthesis of **3a**



1a (6 mmol, 1 equiv), **2a** (24 mmol, 4 equiv), $n\text{Bu}_4\text{NI}$ (3 mmol, 0.5 equiv) and MeCN (60 mL) were added to an undivided beaker-type electrolysis cell (100 mL) equipped with a magnetic stir bar. Three carbon rod electrodes ($\Phi = 0.5$ cm each, 0.5 cm apart from each other and connected in parallel) were used as the anode and a platinum plate (3 cm \times 3 cm \times 0.2 mm) was used as cathode (the electrodes were immersed 3 cm in the reaction solution). The reaction mixture was stirred and electrolyzed at a constant current of 90 mA at 50 $^\circ\text{C}$ (in a water bath) under atmospheric conditions. Every 12 h, 5 mL of MeCN was added (a total of 3 additions) to maintain the reaction volume. After 46 h, the reaction mixture was filtered and concentrated under reduced pressure. The residue was

purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (15:1, v/v) as the eluent to give the target product **3a** (0.87 g, 58% yield).

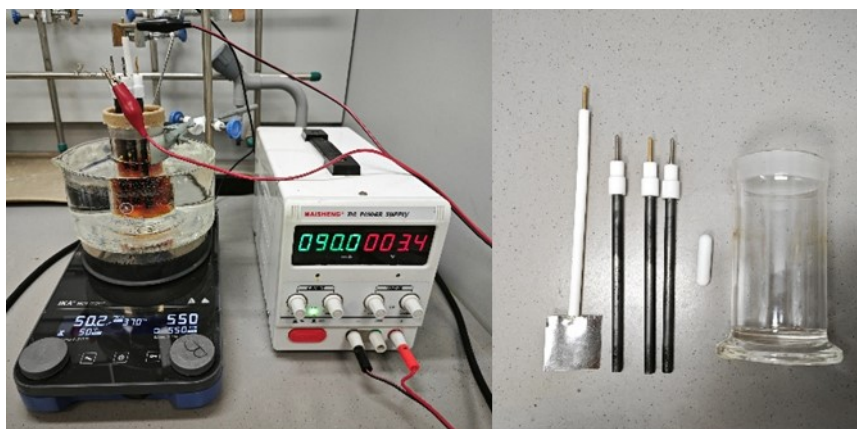


Figure S2 Electrochemical setup used for the scale-up experiment.

The experimental setup for scale-up experiment consisted of three carbon rod electrodes ($\Phi = 0.5$ cm each) and a platinum plate ($3\text{ cm} \times 3\text{ cm} \times 0.2\text{ mm}$), a beaker-type electrolysis cell (100 mL), an adjustable DC regulated power supply (MS-150V 100 mA), and a magnetic stirrer.

3. Optimization of reaction conditions

Table S1. Screening of the amount of $n\text{Bu}_4\text{NI}$ ^a

Entry	Amount of $n\text{Bu}_4\text{NI}$ (equiv)	Yield (%) ^b	Time (h)
1	0.3	30	5
2	0.4	51	6
3	0.5	64	6
4	0.6	60	6.5
5	0.7	58	7.5
6	0.8	55	8
7	1.2	50	16

^a Reaction conditions: A mixture of **1a** (0.2 mmol, 1 equiv), **2a** (0.8 mmol, 4 equiv) and $n\text{Bu}_4\text{NI}$ (x equiv) in MeCN (5 mL) under a constant current of 15 mA (C-rod anode: $\Phi = 0.5$ cm; Pt-plate cathode: $1\text{ cm} \times 1\text{ cm} \times 0.2\text{ mm}$) in an undivided cell at 50 °C (water bath) unsealed under air. ^b Isolated yield.

In the reaction, $n\text{Bu}_4\text{NI}$ functions as both an electrolyte and an iodine mediator. When $n\text{Bu}_4\text{NI}$

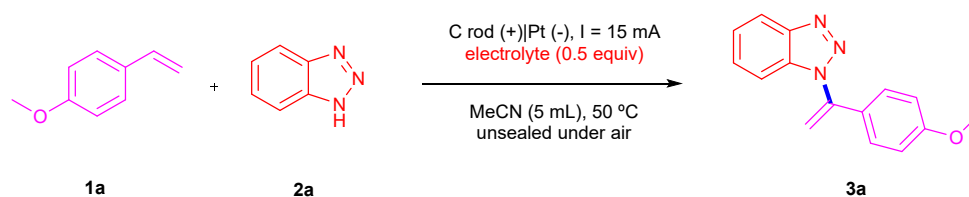
was employed at 0.5 equiv, the voltage range of the reaction spanned from 2.7 to 5.8V, whereas with 1.2 equiv of ⁿBu₄NI, the voltage range of the reaction was from 1.9 to 2.3V. During our experimental procedures, it was observed that an excessive addition of ⁿBu₄NI leads to an increased presence of elemental iodine within the system. With 1.2 equiv of ⁿBu₄NI, even after extending the reaction time to 16 h, unreacted starting material remained.

Table S2. Solvent screening ^a

Entry	Solvent	Yield (%) ^b	Time (h)
1	DCM	7	6
2	DCE	Voltage overload	0.5
3	EA	Voltage overload	0.3
4	THF	Voltage overload	0.3
5	DMSO	10	6
6	HFIP	n.d.	6
7	MeOH	n.d.	6
8	DMF	32	6.5
9	MeNO ₂	trace	6
10	acetone	49	8.5
11	MeCN	64	6
12	MeCN:acetone = 4:1	56	6
13	MeCN:acetone = 3:2	60	6
14	MeCN:acetone = 1:1	60	7
15	MeCN:acetone = 2:3	54	8
16	MeCN:acetone = 1:4	55	10
17	MeCN:DMF = 4:1	38	7

^a Reaction conditions: A mixture of **1a** (0.2 mmol, 1 equiv), **2a** (0.8 mmol, 4 equiv) and ⁿBu₄NI (0.1 mmol, 0.5 equiv) in a solvent (5 mL) under a constant current of 15 mA (C-rod anode: Φ = 0.5 cm; Pt-plate cathode: 1 cm × 1 cm × 0.2 mm) in an undivided cell at 50 °C (water bath) unsealed under air. ^b Isolated yield.

Table S3. Electrolyte screening ^a



Entry	Electrolyte	Yield (%) ^b
1	ⁿ Bu ₄ NBF ₄	n.d.
2	ⁿ Bu ₄ NPF ₆	n.d.
3	ⁿ Bu ₄ NOAc	n.r.
4	NaI	41
5	KI	29
6	NH ₄ I	n.d.
7	ⁿ Bu ₄ NClO ₄	n.d.
8	KClO ₄	n.d.
9	ⁿ Et ₄ NI	53
10	ⁿ Et ₄ NCl	Trace
11	ⁿ Bu ₄ NBr	40
12	LiBr	Voltage overload
13	ⁿ Bu ₄ NI	64

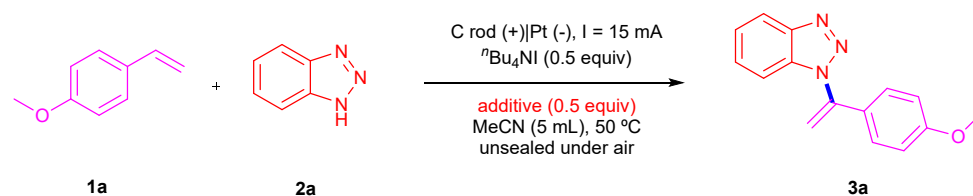
^a Reaction conditions: A mixture of **1a** (0.2 mmol, 1 equiv), **2a** (0.8 mmol, 4 equiv) and electrolyte (0.1 mmol, 0.5 equiv) in MeCN (5 mL) under a constant current of 15 mA (C-rod anode: $\Phi = 0.5$ cm; Pt-plate cathode: 1 cm \times 1 cm \times 0.2 mm) in an undivided cell at 50 °C (water bath) unsealed under air for 6 h. ^b Isolated yield.

Table S4. Mediator screening ^a

Entry	Mediator	Yield (%) ^b	Time (h)
1	--	64	6
2	4-Acetamido-2,2,6,6-tetramethylpiperidine 1-Oxyl	64	6
3	2,2,6,6-Tetramethylpiperidinoxy	63	6
4	Ferrocene	54	6
5	Triphenylamine	56	8
6	9-Azabicyclo[3.3.1]nonane N-oxyl	63	7
7	1,4-Diaza[2.2.2]bicyclooctane	55	10

^a Reaction conditions: A mixture of **1a** (0.2 mmol, 1 equiv), **2a** (0.8 mmol, 4 equiv), ⁿBu₄NI (0.1 mmol, 0.5 equiv) and mediator (5 mol%) in MeCN (5 mL) under a constant current of 15 mA (C-rod anode: $\Phi = 0.5$ cm; Pt-plate cathode: 1 cm \times 1 cm \times 0.2 mm) in an undivided cell at 50 °C (water bath) unsealed under air. ^b Isolated yield.

Table S5. Additive screening ^a



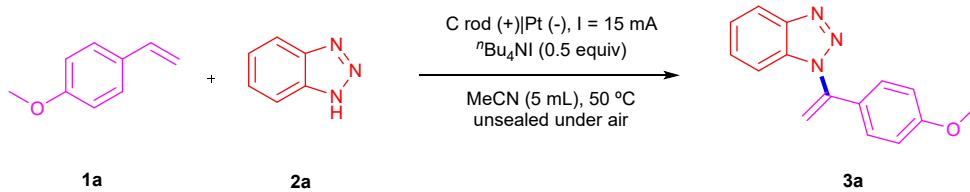
Entry	Additive	Yield (%) ^b	Time (h)
1	--	64	6
2	AcOH	n.d.	6
3	KHCO ₃	trace	6
4	2,6-lutidine	55	4.5
5	K ₂ CO ₃	44	6
6	Et ₃ N	48	6
7	1,8-Diazabicyclo[5.4.0]undecane-7-ene	50	8
8	4-Dimethylaminopyridine	49	8
9	KOH	44	6
10	K ₃ PO ₄	39	5
11	Cs ₂ CO ₃	42	6
12	^t BuOK	33	6.5
13	2,4,6-collidine	50	5
14	N,N-Diisopropylethylamine	50	5
15	Propylamine	60	6.5

^a Reaction conditions: A mixture of **1a** (0.2 mmol, 1 equiv), **2a** (0.8 mmol, 4 equiv), ⁿBu₄NI (0.1 mmol, 0.5 equiv) and additive (0.1 mmol, 0.5 equiv) in MeCN (5 mL) under a constant current of 15 mA (C-rod anode: Φ = 0.5 cm; Pt-plate cathode: 1 cm × 1 cm × 0.2 mm) in an undivided cell at 50 °C (water bath) unsealed under air. ^b Isolated yield.

Table S6. Electrode material screening ^a

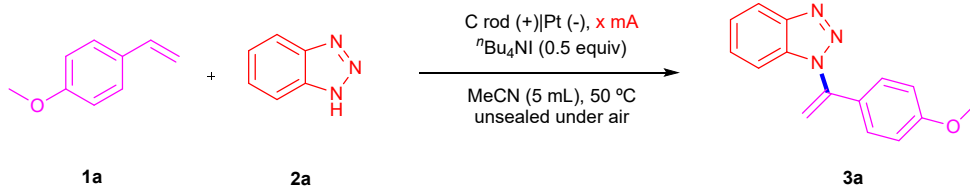
Entry	Electrode material	Yield (%) ^b
1	C(+) Pt(-)	64
2	C(+) C(-)	44
3	Pt(+) Pt(-)	37
4	Pt(+) C(-)	36
5	RVC(+) Pt(-)	n.d.
6	Pt(+) RVC(-)	n.d.

^a Reaction conditions: A mixture of **1a** (0.2 mmol, 1 equiv), **2a** (0.8 mmol, 4 equiv) and ⁿBu₄NI (0.1 mmol, 0.5 equiv) in MeCN (5 mL) under a constant current of 15 mA (C-rod: Φ = 0.5 cm; Pt-plate: 1 cm × 1 cm × 0.2 mm; RVC-plate: 0.5cm × 0.5 cm × 0.5 cm) in an undivided cell at 50 °C (water bath) unsealed under air for 6 h. ^b Isolated yield.

Table S7. Screening of substrate molar ratio ^a


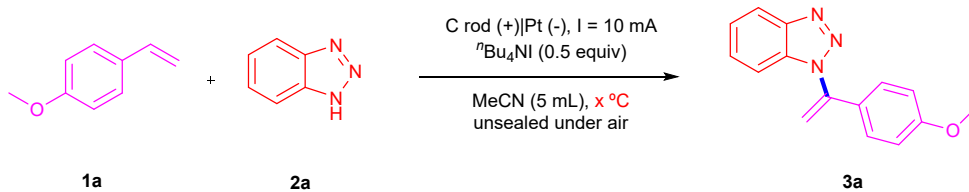
Entry	Amount of 2a (equiv)	1a : 2a	Yield (%) ^b
1	1	1:1	35
2	2	1:2	49
3	3	1:3	60
4	4	1:4	64
5	5	1:5	59
6	6	1:6	52
7	0.5	2:1	30
8	0.25	4:1	37

^a Reaction conditions: A mixture of **1a** (0.2 mmol, 1 equiv), **2a** (x equiv) and ^tBu₄NI (0.1 mmol, 0.5 equiv) in MeCN (5 mL) under a constant current of 15 mA (C-rod anode: Φ = 0.5 cm; Pt-plate cathode: 1 cm × 1 cm × 0.2 mm) in an undivided cell at water bath (50 °C) unsealed under air for 6 h. ^b Isolated yield.

Table S8. Current screening ^a


Entry	Current (mA)	Yield (%) ^b
1	--	n.r.
2	5	20
3	8	43
4	10	68
5	12	67
6	15	64
7	20	39

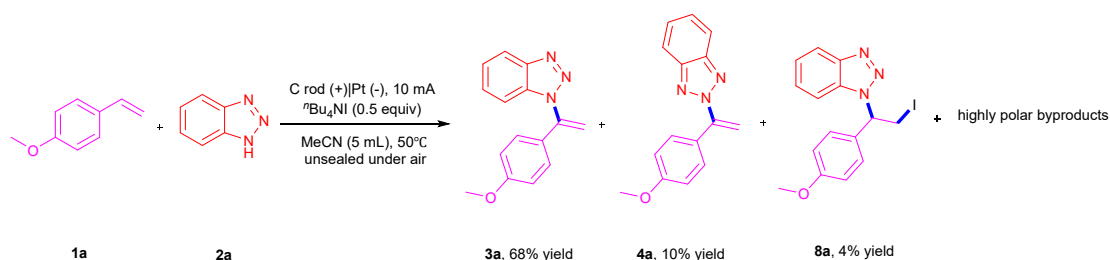
^a Reaction conditions: A mixture of **1a** (0.2 mmol, 1 equiv), **2a** (0.8 mmol, 4 equiv) and ^tBu₄NI (0.1 mmol, 0.5 equiv) in MeCN (5 mL) under a constant current of x mA (C-rod anode: Φ = 0.5 cm; Pt-plate cathode: 1 cm × 1 cm × 0.2 mm) in an undivided cell at 50 °C (water bath) unsealed under air for 6 h. ^b Isolated yield.

Table S9. Temperature screening^a

Entry	Temperature (°C)	Yield (%) ^b
1	25	44
2	40	50
3	50	68
4	60	65

^a Reaction conditions: A mixture of **1a** (0.2 mmol, 1 equiv), **2a** (0.8 mmol, 4 equiv) and ^tBu₄NI (0.1 mmol, 0.5 equiv) in MeCN (5 mL) under a constant current of 10 mA (C-rod anode: Φ = 0.5 cm; Pt-plate cathode: 1 cm × 1 cm × 0.2 mm) in an undivided cell at water bath (x °C) unsealed under air for 6 h. ^b Isolated yield.

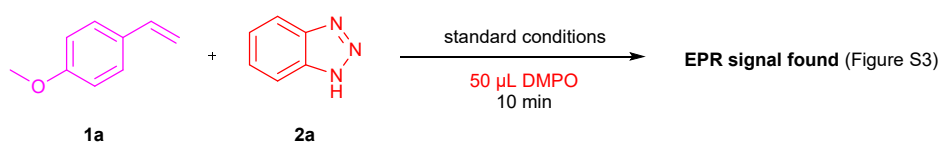
4. Main by-products



In the model reaction, the desired product **3a** was obtained in 68% isolated yield, while byproduct **4a** was isolated in 10% yield, and the iodinated compound **8a** was isolated in 4% yield. Additionally, nearly all reactions generated unidentified highly polar byproducts. For the ¹H NMR and ¹³C NMR of the secondary products (**4a** and **8a**), see SI, §7 and §9.

5. Mechanistic investigation

5.1. EPR experiments



The model reaction was carried out under standard conditions in the presence of 50 μL 3,4-dihydro-

2,2-dimethyl-2*H*-pyrrole 1-oxide (DMPO). After 10 minutes, the reaction solution was taken out with capillary and analyzed by EPR at room temperature. Radical signals were detected (Figure S3), indicating the possible generation of radicals.

EPR spectra were recorded with a modified Bruker EMXplus-9.5/12 spectrometer operated at 9.8293 GHz. The scan range was 150.0 G, with a resolution of 1500 points and a center field of 3511.15 G. The time constant was 81.92 ms, the conversion time was 66.68 ms, and the scan time was 100.02 s. A modulation amplitude of 10.0 G, the modulation frequency of 100.00 kHz, and microwave power of 6.325 mW were used for the experiments.

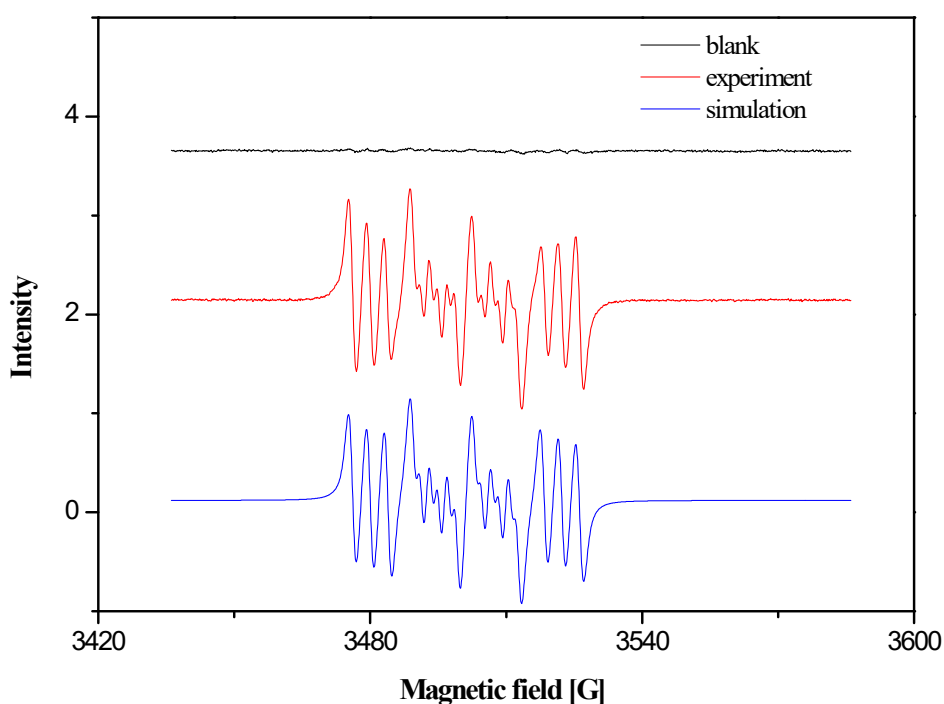


Figure S3 EPR experiments.

5.2. Cyclic voltammetry experiments

The electrochemical measurement was performed by a computer-controlled electrochemical analyzer. Cyclic voltammetry experiments were performed in a three-electrode cell with MeCN (15 mL) as solvent, $n\text{Bu}_4\text{NClO}_4$ (0.05 M) as supporting electrolyte, and the concentration of the tested compound was 2.0 mM. Glassy carbon (diameter 3 mm) was used as working electrode, platinum wire as auxiliary electrode, and Ag/AgCl (3 M KCl) as reference electrode. The scanning speed was $100 \text{ mV}\cdot\text{s}^{-1}$. For $n\text{Bu}_4\text{NI}$, 1-methoxy-4-vinylbenzene (**1a**), benzotriazole (**2a**) and 1-(2-iodo-1-(4-methoxyphenyl)ethyl)-1*H*-benzo[*d*][1,2,3]triazole (**8a**), the oxidation potential range studied was 0.0 V to +3.0 V vs. Ag/AgCl (3 M KCl). The oxidation potentials were determined as: The onset

potential for the oxidation of $n\text{Bu}_4\text{NI}$ is around +0.73 V, 1-methoxy-4-vinylbenzene (**1a**) is around +1.17 V, no obvious oxidation peaks for benzotriazole **2a** (Figure S4) and 1-(2-iodo-1-(4-methoxyphenyl)ethyl)-1*H*-benzo[*d*][1,2,3]triazole (**8a**) is around +1.52 V (Figure S5). For **8a**, the reduction potential range studied was 0.0 V to -3.0 V vs. Ag/AgCl (3 M KCl). The onset potential for the reduction of **8a** is around -2.25 V (Figure S6).

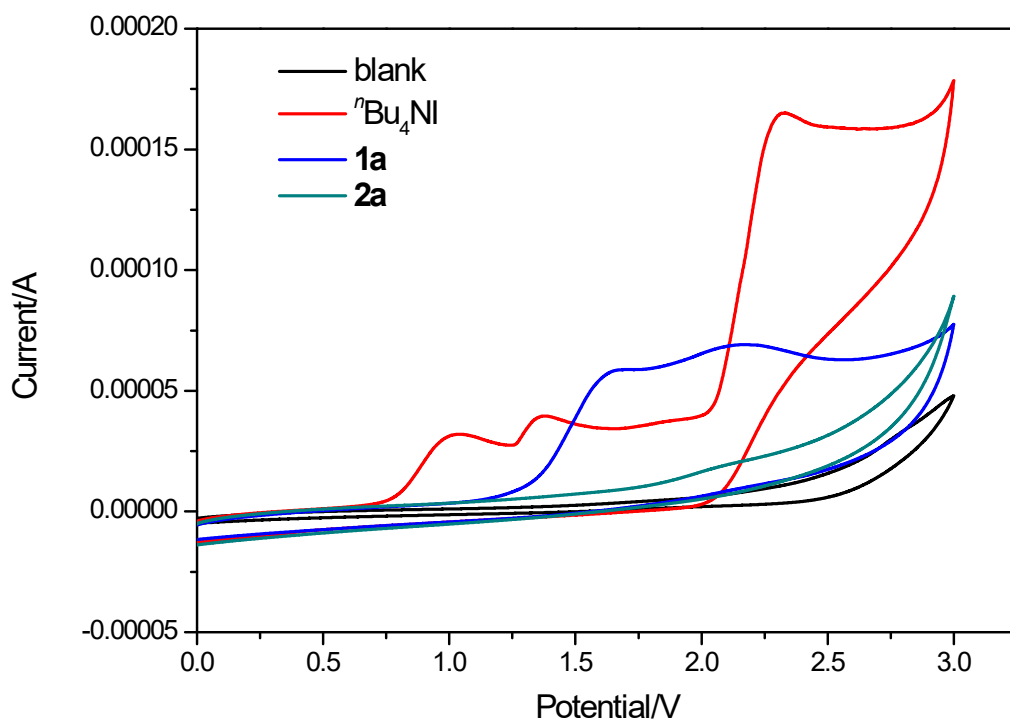


Figure S4 Cyclic voltammogram oxidation potential of $n\text{Bu}_4\text{NI}$, **1a** and **2a**

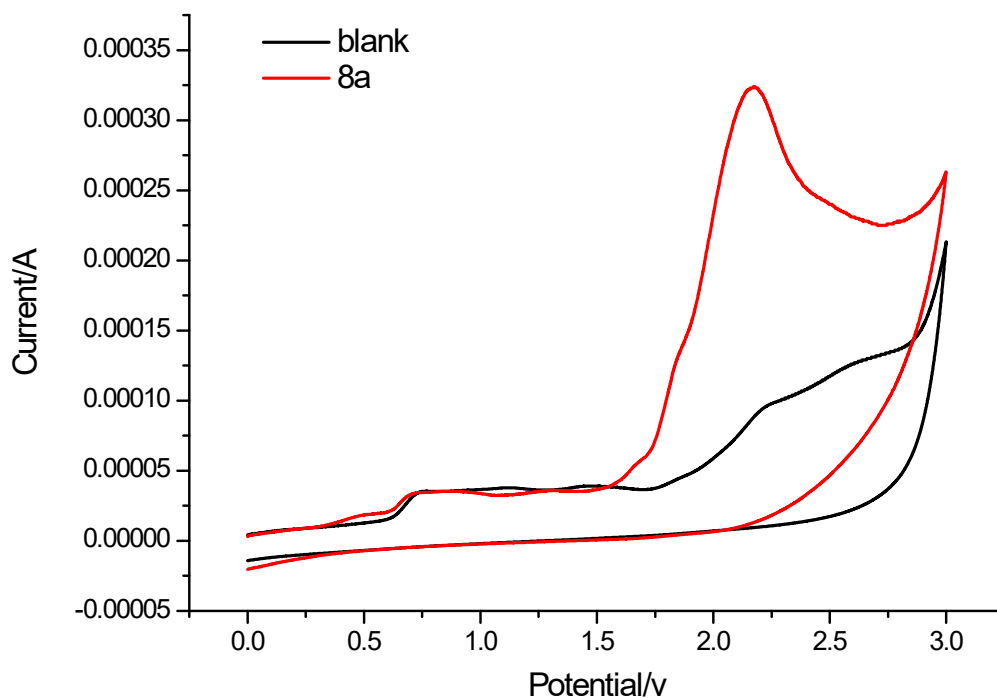


Figure S5 Cyclic voltammogram oxidation potential of **8a**

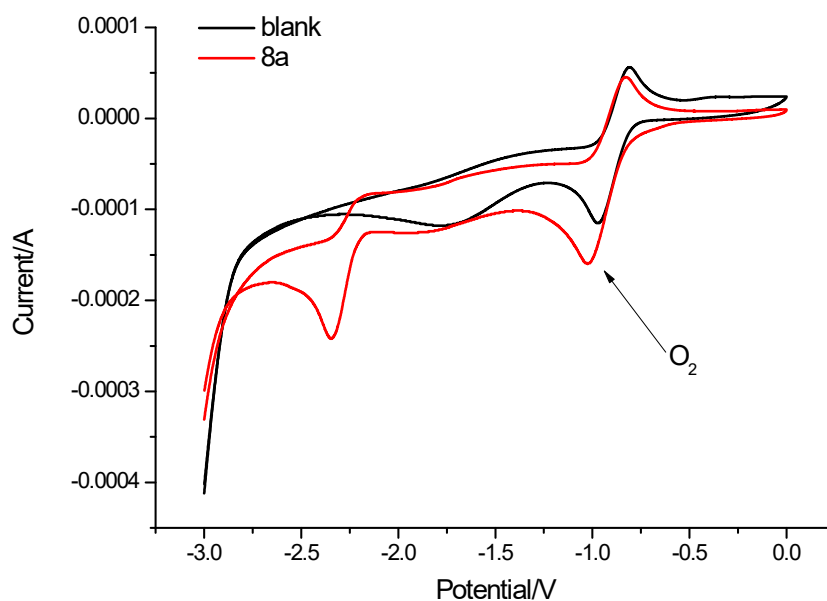


Figure S6 Cyclic voltammogram reduction potential of **8a**

5.3. The measurement of pH values

We monitored the pH values at the onset of the model reaction and after 3 h of electrolysis. The initial pH was around 6 (Figure S7 a), while after 3 h of electrolysis, the pH increased to approximately 8 (Figure S7 b).

Additionally, we assessed the pH of 0.1 mmol of $n\text{Bu}_4\text{NI}$ dissolved in 5 mL of non-dried MeCN (analytical grade). In the absence of electrical current, the pH was approximately 6 (Figure S7 c). After 3 h of exposure to a 10 mA electrical current, the pH was approximately 8 (Figure S7 d).

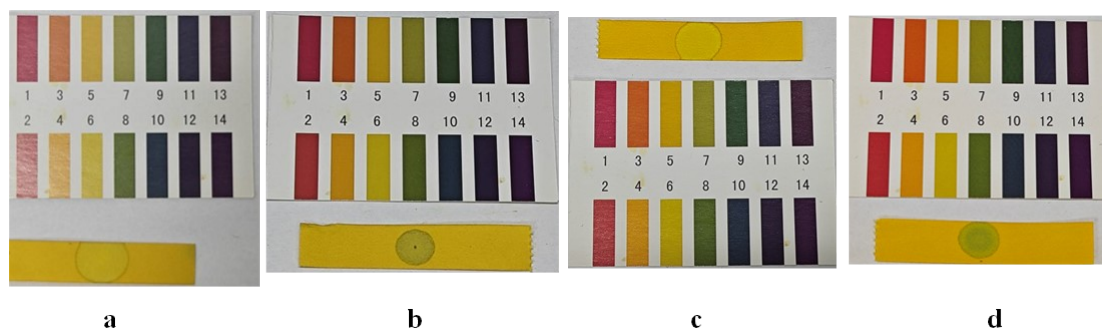
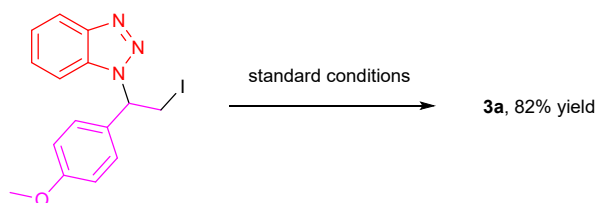


Figure S7 The measurement of pH values. a) The model reaction system (before electrolysis); b) The model reaction system (after electrolysis under a 10 mA current for 3 h); c) 0.1 mmol of $n\text{Bu}_4\text{NI}$ in a non-dried 5 mL MeCN (analytical grade) (before electrolysis); d) 0.1 mmol of $n\text{Bu}_4\text{NI}$ in a non-dried 5 mL MeCN (analytical grade) (after electrolysis under a 10 mA current

for 3 h).

5.4. Possible explanations for the elimination of HI from **8a** to yield **3a**



Based on the aforementioned cyclic voltammetry measurements and pH determinations, we put forward possible explanations for the elimination of HI from **8a** to yield **3a**. The observational results suggest that, under our reaction conditions, **8a** is neither oxidized nor reduced. We speculate that I^- from nBu_4NI undergoes anodic oxidation, while H^+ ions, derived from the dissociation of water in the solvent, undergo cathodic reduction, resulting in the release of H_2 . This leads to an increase in OH^- concentration within the system, consequently causing the reaction system to become basic, which is conducive to the elimination of HI. Notably, this reaction was conducted under elevated temperature at $50^\circ C$, and heating also promotes the elimination of HI.

6. The possible mechanism of nBu_4NBr participation

The possible mechanism when nBu_4NBr is used in place of nBu_4NI (Figure S8).

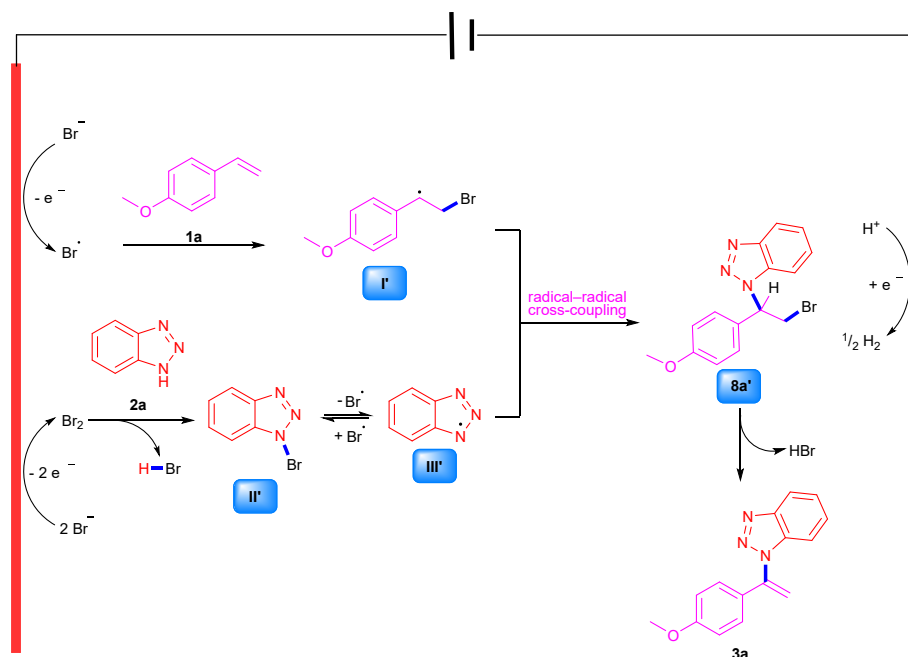
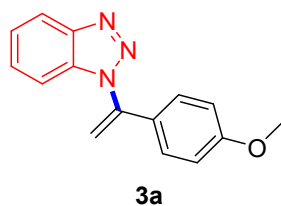
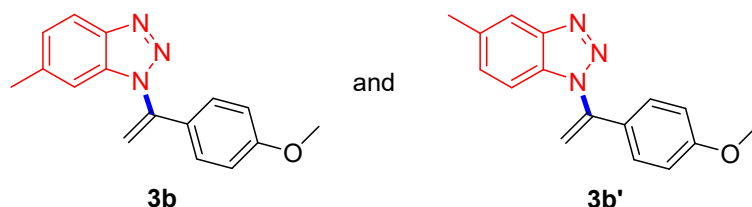


Figure S8 Proposed mechanism.

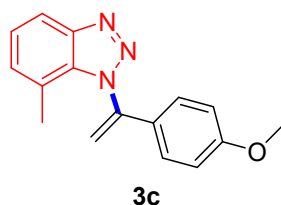
7. Characterization data of the products



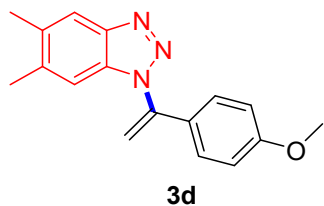
1-(1-(4-Methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3a)^[3]: $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). 34.2 mg, 68% yield. Yellow oil. $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 8.15-8.07 (m, 1H), 7.40-7.33 (m, 2H), 7.25-7.16 (m, 2H), 7.13-7.06 (m, 1H), 6.93-6.86 (m, 2H), 5.69 (d, $J = 1.1$ Hz, 1H), 5.65 (d, $J = 1.1$ Hz, 1H), 3.83 (s, 3H). $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 160.9, 146.2, 142.3, 133.0, 128.3, 127.7, 127.2, 124.1, 120.1, 114.2, 111.3, 109.3, 55.4.



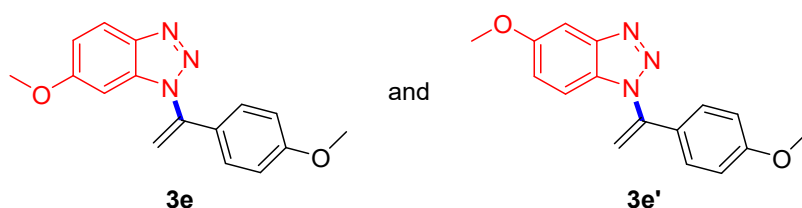
1-(1-(4-Methoxyphenyl)vinyl)-6-methyl-1H-benzo[d][1,2,3]triazole (3b) and **1-(1-(4-Methoxyphenyl)vinyl)-5-methyl-1H-benzo[d][1,2,3]triazole (3b')**: $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). Isomer (1.3:1 based on NMR), 29.7 mg, 56% yield. Yellow oil. $^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.97 (d, $J = 8.6$ Hz, 0.8H), 7.86 (s, 1H), 7.25-7.19 (m, 4.6H), 7.18 (d, $J = 1.4$ Hz, 0.8H), 6.96 (d, $J = 8.5$ Hz, 1H), 6.93-6.86 (m, 4.4H), 5.70 (s, 0.8H), 5.65 (s, 1H), 5.64 (s, 1H), 5.60 (s, 0.8H), 3.83 (d, $J = 1.4$ Hz, 5.4H), 2.50 (s, 3H), 2.42 (s, 2.4H). $^{13}\text{C NMR}$ (101 MHz, Chloroform-*d*) δ 160.8, 146.8, 144.7, 142.4, 142.3, 138.4, 134.2, 131.5, 129.8, 128.4, 128.2, 127.3, 127.2, 126.4, 119.5, 119.0, 114.2, 110.9, 110.4, 109.3, 109.0, 55.4, 22.0, 21.4. HRMS (ESI): m/z : calcd for $\text{C}_{16}\text{H}_{15}\text{N}_3\text{ONa}$ ($\text{M}+\text{Na}$)⁺ 288.1107; found 288.1110. Calcd for $\text{C}_{16}\text{H}_{15}\text{N}_3\text{OK}$ ($\text{M}+\text{K}$)⁺ 304.0847; found 304.0860.



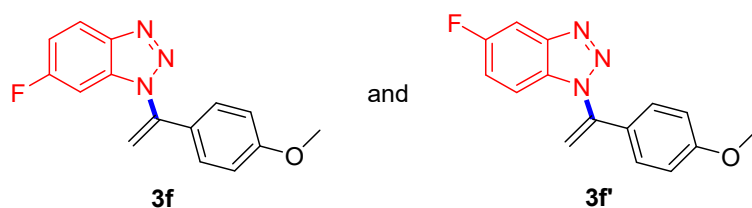
1-(1-(4-Methoxyphenyl)vinyl)-7-methyl-1H-benzo[d][1,2,3]triazole (3c): $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). 27.6 mg, 52% yield. Yellow oil. $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.26-7.19 (m, 3H), 7.15-7.10 (m, 1H), 6.93-6.85 (m, 3H), 5.68 (d, $J = 1.0$ Hz, 1H), 5.63 (d, $J = 0.9$ Hz, 1H), 3.83 (s, 3H), 2.84 (s, 3H). $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 160.8, 146.1, 142.5, 133.0, 131.0, 128.3, 127.6, 127.3, 123.9, 114.2, 109.3, 108.6, 55.4, 16.7. HRMS (ESI): m/z : calcd for $\text{C}_{16}\text{H}_{15}\text{N}_3\text{ONa}$ ($\text{M}+\text{Na}$)⁺ 288.1107; found 288.1107.



1-(1-(4-Methoxyphenyl)vinyl)-5,6-dimethyl-1H-benzo[d][1,2,3]triazole (3d): $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). 34.0 mg, 61% yield. Colorless oil. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.83 (s, 1H), 7.25-7.16 (m, 2H), 6.92-6.84 (m, 3H), 5.67 (s, 1H), 5.60 (s, 1H), 3.83 (s, 3H), 2.39 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.8, 145.3, 142.4, 138.1, 133.9, 132.1, 128.3, 127.4, 119.1, 114.2, 110.7, 109.0, 55.4, 20.9, 20.3. HRMS (ESI): m/z : calcd for $\text{C}_{17}\text{H}_{17}\text{N}_3\text{ONa}$ ($\text{M}+\text{Na}$) $^+$ 302.1264; found 302.1265.

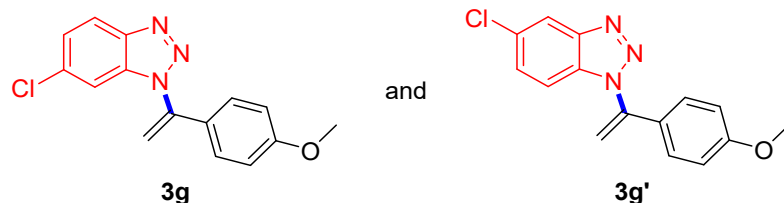


6-Methoxy-1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3e) and 5-Methoxy-1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3e'): $R_f = 0.25$ (Petroleum ether/EtOAc, 10:1). Isomer (2.5:1 based on NMR), 24.2 mg, 43% yield. Yellow oil. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, $J = 9.1$ Hz, 0.4H), 7.42 (d, $J = 2.3$ Hz, 1H), 7.25-7.20 (m, 2.8H), 7.04 (d, $J = 2.2$ Hz, 0.4H), 7.02-7.01 (m, 0.8H), 6.95-6.87 (m, 4H), 6.40 (d, $J = 2.2$ Hz, 0.4H), 5.70 (d, $J = 0.9$ Hz, 0.4H), 5.66-5.64 (m, 2H), 5.63 (d, $J = 0.9$ Hz, 0.4H), 3.89 (s, 3H), 3.83 (d, $J = 0.9$ Hz, 4.2H), 3.70 (s, 1.2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.8 (160.84), 160.8 (160.80), 160.1, 157.3, 147.2, 142.3, 128.5, 128.4, 128.3, 127.1, 120.7, 120.3, 116.2, 114.2 (114.20), 114.2 (114.18), 111.9, 109.2, 109.0, 98.9, 91.8, 55.7, 55.6, 55.4 (55.40), 55.4 (55.38). HRMS (ESI): m/z : calcd for $\text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 304.1056; found 304.1052. Calcd for $\text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_2\text{K}$ ($\text{M}+\text{K}$) $^+$ 320.0796; found 320.0792.

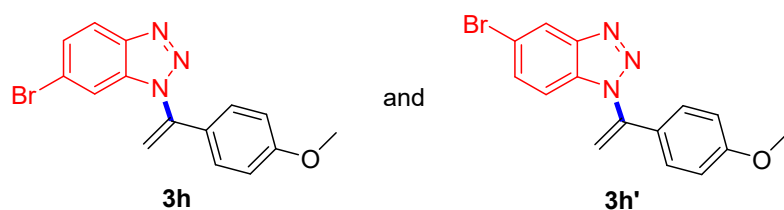


6-Fluoro-1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3f) and 5-Fluoro-1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3f'): $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). Isomer (1:1 based on NMR), 20.4 mg, 38% yield. Orange oil. ^1H NMR (600 MHz, Chloroform-*d*) δ 8.10-8.01 (m, 1H), 7.76-7.69 (m, 1H), 7.26-7.18 (m, 4H), 7.18-7.09 (m, 2H), 7.04-7.00 (m, 1H), 6.93-6.86 (m, 4H), 6.76-6.63 (m, 1H), 5.69 (s, 1H), 5.68 (s, 1H), 5.66 (s, 1H), 5.65 (s, 1H), 3.84 (d, $J = 2.8$ Hz, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 162.3 (d, $J = 248.7$ Hz), 161.0, 159.7 (d, $J = 243.7$ Hz), 146.5, 146.4, 143.0, 142.2, 142.1, 133.5, 133.4, 130.0, 128.3 (128.33),

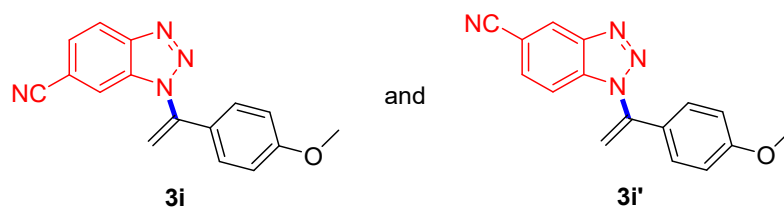
128.3 (128.29), 126.9, 126.7, 121.6, 121.5, 117.7, 117.5, 114.4, 114.3, 114.2, 114.0, 112.3 (112.32), 112.3 (112.26), 109.5, 109.4, 104.7, 104.5, 97.3, 97.1, 55.4. ^{19}F NMR (565 MHz, Chloroform-*d*) δ -111.00, -117.60. HRMS (ESI): m/z : calcd for $\text{C}_{15}\text{H}_{12}\text{FN}_3\text{ONa}$ ($\text{M}+\text{Na}$) $^+$ 292.0857; found 292.0857. Calcd for $\text{C}_{15}\text{H}_{12}\text{FN}_3\text{OK}$ ($\text{M}+\text{K}$) $^+$ 308.0596; found 308.0597.



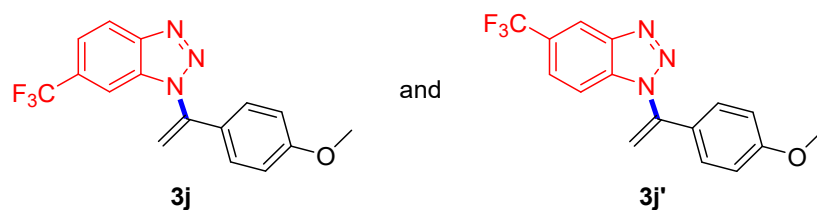
6-Chloro-1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3g) and 5-Chloro-1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3g'): R_f = 0.25 (Petroleum ether/EtOAc, 15:1). Isomer (1:1 based on NMR), 23.4 mg, 41% yield. Orange oil. ^1H NMR (600 MHz, Chloroform-*d*) δ 8.09 (d, J = 1.9 Hz, 1H), 8.03 (d, J = 8.8 Hz, 1H), 7.35-7.31 (m, 2H), 7.23-7.18 (m, 4H), 7.12 (d, J = 1.9 Hz, 1H), 6.99 (d, J = 8.8 Hz, 1H), 6.92-6.89 (m, 4H), 5.72 (s, 1H), 5.69 (s, 1H), 5.66 (s, 1H), 5.63 (s, 1H), 3.84 (m, 6H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 161.0 (161.04), 161.0 (161.02), 146.8, 144.7, 142.1, 142.0, 134.2, 133.6, 131.7, 130.1, 128.6, 128.3, 128.2, 126.8, 126.7, 125.4, 121.0, 119.5, 114.4, 114.3, 112.2, 111.0, 109.8, 109.6, 55.4. HRMS (ESI): m/z : calcd for $\text{C}_{15}\text{H}_{12}\text{ClN}_3\text{ONa}$ ($\text{M}+\text{Na}$) $^+$ 308.0561; found 308.0559.



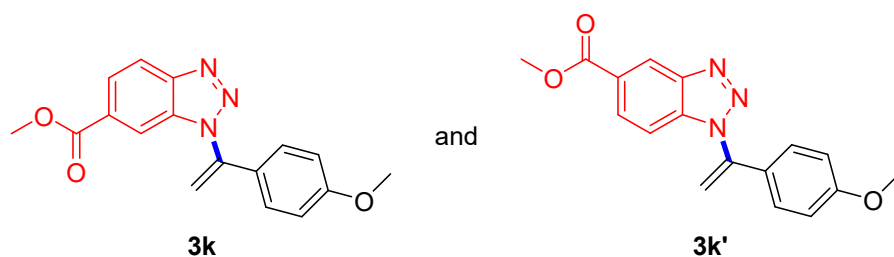
6-Bromo-1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3h) and 5-Bromo-1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3h'): R_f = 0.25 (Petroleum ether/EtOAc, 15:1). Isomer (1.3:1 based on NMR), 28.6 mg, 43% yield. Orange oil. ^1H NMR (600 MHz, Chloroform-*d*) δ 8.26 (d, J = 1.7 Hz, 0.8H), 7.97 (d, J = 8.8 Hz, 1H), 7.46 (m, 2H), 7.32 (d, J = 1.7 Hz, 1H), 7.20 (m, 3.4H), 6.95 (d, J = 8.8 Hz, 1H), 6.91-6.88 (m, 3.4H), 5.72 (d, J = 1.2 Hz, 1H), 5.70 (d, J = 1.1 Hz, 0.8H), 5.66 (d, J = 1.1 Hz, 0.8H), 5.62 (d, J = 1.2 Hz, 1H), 3.84 (d, J = 4.5 Hz, 5.4H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 161.0, 161.0(160.99), 147.3, 145.0, 142.0, 142.0(141.97), 134.0, 131.9, 131.1, 128.3, 128.2, 128.0, 126.7, 126.7(126.67), 122.7, 122.2, 121.2, 117.4, 114.4, 114.3, 114.1, 112.6, 109.9, 109.7, 55.4. HRMS (ESI): m/z : calcd for $\text{C}_{15}\text{H}_{12}\text{BrN}_3\text{ONa}$ ($\text{M}+\text{Na}$) $^+$ 352.0056; found 352.0048.



1-(1-(4-Methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole-6-carbonitrile (3i) and **1-(1-(4-Methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole-5-carbonitrile (3i')**: $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). Isomer (1.3:1 based on NMR), 33.1 mg, 60% yield. Yellow oil. ^1H NMR (600 MHz, Chloroform-*d*) δ 8.50 (s, 0.8H), 8.22 (d, $J = 8.6$ Hz, 1H), 7.61-7.57 (m, 1.8H), 7.44 (s, 1H), 7.21-7.18 (m, 3.6H), 7.17-7.15 (m, 0.8H), 6.94-6.90 (m, 3.6H), 5.77-5.75 (m, 1.8H), 5.71-5.69 (m, 1.8H), 3.86 (s, 3H), 3.85 (s, 2.4H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 161.3, 161.2, 147.4, 145.4, 141.8 (141.81), 141.8 (141.77), 134.5, 132.3, 129.9, 128.2 (128.24), 128.2 (128.23), 126.5, 126.4, 126.2 (126.21), 126.2 (126.15), 121.6, 118.3, 118.2, 117.1, 114.6, 114.5, 112.8, 111.3, 110.4 (110.41), 110.4 (110.39), 108.1, 55.4. HRMS (ESI): m/z : calcd for $\text{C}_{16}\text{H}_{12}\text{N}_4\text{ONa}$ ($\text{M}+\text{Na}$) $^+$ 299.0903; found 299.0897. Calcd for $\text{C}_{16}\text{H}_{12}\text{N}_4\text{OK}$ ($\text{M}+\text{K}$) $^+$ 315.0643; found 315.0647.

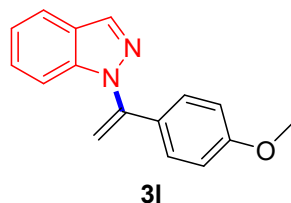


1-(1-(4-Methoxyphenyl)vinyl)-6-(trifluoromethyl)-1H-benzo[d][1,2,3]triazole (3i) and **1-(1-(4-Methoxyphenyl)vinyl)-5-(trifluoromethyl)-1H-benzo[d][1,2,3]triazole (3i')**: $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). Isomer (1.4:1 based on NMR), 40.2 mg, 63% yield. Yellow oil. ^1H NMR (600 MHz, Chloroform-*d*) δ 8.43 (s, 0.7H), 8.23 (d, $J = 8.7$ Hz, 1H), 7.64-7.57 (m, 1.7H), 7.46 (s, 1H), 7.24-7.18 (m, 3.4H), 7.18 (d, $J = 8.7$ Hz, 0.7H), 6.94-6.87 (m, 3.4H), 5.78 (d, $J = 1.4$ Hz, 1H), 5.74 (d, $J = 1.3$ Hz, 0.7H), 5.70 (d, $J = 1.3$ Hz, 0.7H), 5.67 (d, $J = 1.3$ Hz, 1H), 3.84 (s, 5.1H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 161.2, 161.1, 147.3, 145.4, 142.0 (142.00), 142.0 (141.98), 134.3, 132.5, 130.0 (q, $J = 32.6$ Hz), 128.3, 128.2, 126.9 (q, $J = 33.0$ Hz) 126.7, 126.6, 124.3 (q, $J = 2.7$ Hz), 123.8 (q, $J = 272.6$ Hz), 123.1, 121.2, 120.9 (q, $J = 3.0$ Hz), 118.4 (q, $J = 4.5$ Hz), 114.4 (114.43), 114.4 (114.41), 112.3, 110.1, 110.0, 109.4 (q, $J = 4.5$ Hz), 55.4 (55.40), 55.4 (55.39). ^{19}F NMR (565 MHz, Chloroform-*d*) δ -61.47, -61.78. HRMS (ESI): m/z : calcd for $\text{C}_{16}\text{H}_{13}\text{F}_3\text{N}_3\text{O}$ ($\text{M}+\text{H}$) $^+$ 320.1005; found 320.1007.

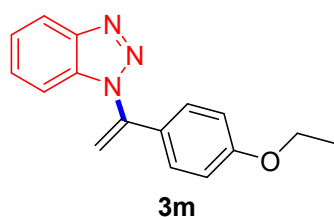


Methyl 1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole-6-carboxylate (3k) and **Methyl 1-(1-(4-methoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole-5-carboxylate (3k')**: $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). Isomer (1.6:1 based on NMR), 35.2 mg, 57% yield. Yellow oil. ^1H NMR (400 MHz, Chloroform-*d*) δ 8.87-8.78 (m, 0.6H), 8.16-8.13 (m, 1H), 8.09-8.05 (m, 1H), 8.05-8.04 (m, 0.6H), 7.98-7.94 (m, 1H), 7.23-7.19 (m, 3.2H), 7.13-7.09 (m, 0.6H), 6.92-6.87 (m, 3.2H), 5.80 (d, $J = 1.2$ Hz, 1H), 5.73 (d, $J = 1.1$ Hz, 0.6H), 5.69 (d, $J = 1.2$ Hz, 0.6H), 5.65 (d, $J = 1.2$ Hz, 1H), 3.97 (s, 1.8H), 3.92 (s, 3H), 3.83 (d, $J = 1.7$ Hz, 4.8H). ^{13}C NMR (101 MHz,

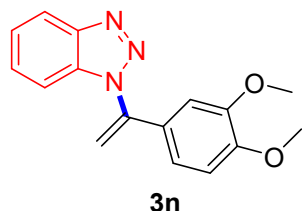
Chloroform-*d*) δ 166.4, 166.3, 161.0, 148.0, 145.9, 142.0, 135.2, 132.9, 129.7, 128.5, 128.3, 128.1, 126.9, 126.7, 126.6, 124.9, 122.9, 120.0, 114.3 (114.33), 114.3 (114.31), 113.7, 111.2, 110.1, 109.8, 55.4, 52.5, 52.4. HRMS (ESI): m/z : calcd for $C_{17}H_{15}N_3O_3Na$ ($M+Na$)⁺ 332.1006; found 332.1006. Calcd for $C_{17}H_{15}N_3O_3K$ ($M+K$)⁺ 348.0745; found 348.0746.



1-(1-(4-Methoxyphenyl)vinyl)-1H-indazole (3l): R_f = 0.25 (Petroleum ether/EtOAc, 15:1). 12.5 mg, 25% yield. Yellow oil. ¹H NMR (600 MHz, Chloroform-*d*) δ 7.99 (s, 1H), 7.76 (d, J = 8.8 Hz, 1H), 7.66-7.60 (m, 1H), 7.36-7.29 (m, 3H), 7.13-7.06 (m, 1H), 6.96-6.90 (m, 2H), 5.80 (s, 1H), 5.40 (s, 1H), 3.85 (d, J = 1.3 Hz, 3H). ¹³C NMR (151 MHz, Chloroform-*d*) δ 160.7, 149.4, 146.2, 129.4, 128.1, 126.9, 123.5, 122.3, 121.8, 120.4, 117.9, 114.1, 107.4, 55.4. HRMS (ESI): m/z : calcd for $C_{16}H_{14}N_2ONa$ ($M+Na$)⁺ 273.0998; found 273.0998.

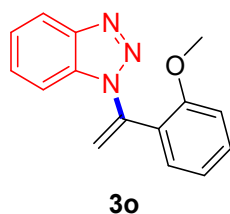


1-(1-(4-Ethoxyphenyl)vinyl)-1H-benzo[*d*][1,2,3]triazole (3m): R_f = 0.25 (Petroleum ether/EtOAc, 15:1). 32.9 mg, 62% yield. Orange oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.14-8.05 (m, 1H), 7.39-7.33 (m, 2H), 7.24-7.18 (m, 2H), 7.12-7.07 (m, 1H), 6.90-6.85 (m, 2H), 5.69 (d, J = 0.9 Hz, 1H), 5.65 (d, J = 0.9 Hz, 1H), 4.05 (q, J = 7.0 Hz, 2H), 1.42 (t, J = 7.0 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 160.2, 146.1, 142.3, 133.0, 128.3, 127.7, 126.9, 124.1, 120.1, 114.7, 111.4, 109.2, 63.6, 14.7. HRMS (ESI): m/z : calcd for $C_{16}H_{15}N_3ONa$ ($M+Na$)⁺ 288.1107; found 288.1093. Calcd for $C_{16}H_{15}N_3OK$ ($M+K$)⁺ 304.0847; found 304.0834.

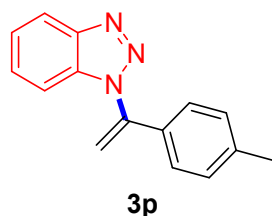


1-(1-(3,4-Dimethoxyphenyl)vinyl)-1H-benzo[*d*][1,2,3]triazole (3n): R_f = 0.25 (Petroleum ether/EtOAc, 15:1). 39.4 mg, 70% yield. Colorless oil. ¹H NMR (600 MHz, Chloroform-*d*) δ 8.17-8.05 (m, 1H), 7.41-7.34 (m, 2H), 7.13-7.05 (m, 1H), 6.86 (s, 2H), 6.82 (s, 1H), 5.70 (s, 1H), 5.69 (s, 1H), 3.91 (s, 3H), 3.78 (s, 3H). ¹³C NMR (151 MHz, Chloroform-*d*) δ 150.6, 149.3, 146.2, 142.5, 133.0, 127.7, 127.5, 124.1, 120.2, 120.1, 111.4, 111.3, 110.2, 109.4, 56.0 (56.03), 56.0 (56.00).

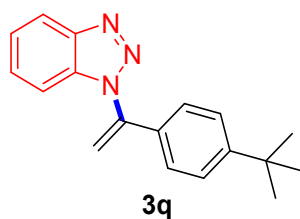
HRMS (ESI): m/z : calcd for $C_{16}H_{15}N_3O_2Na$ ($M+Na$)⁺ 304.1056; found 304.1059. Calcd for $C_{16}H_{15}N_3O_2K$ ($M+K$)⁺ 320.0796; found 320.0799.



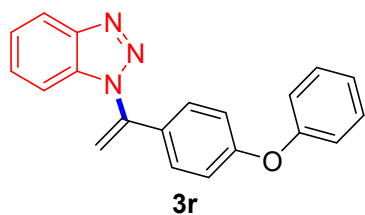
1-(1-(2-Methoxyphenyl)vinyl)-1H-benzo[*d*][1,2,3]triazole (3o): R_f = 0.25 (Petroleum ether/EtOAc, 15:1). 12.1 mg, 24% yield. Yellow oil. ¹H NMR (600 MHz, Chloroform-*d*) δ 8.10-8.04 (m, 1H), 7.44-7.37 (m, 2H), 7.34-7.29 (m, 2H), 7.07-7.00 (m, 2H), 6.87 (d, J = 8.3 Hz, 1H), 5.95 (s, 1H), 5.63 (s, 1H), 3.44 (s, 3H). ¹³C NMR (151 MHz, Chloroform-*d*) δ 157.5, 146.1, 140.7, 132.6, 131.2, 130.7, 127.4, 124.2, 123.8, 120.9, 119.9, 111.4, 111.3, 111.0, 55.5. HRMS (ESI): m/z : calcd for $C_{15}H_{13}N_3ONa$ ($M+Na$)⁺ 274.0951; found 274.0945.



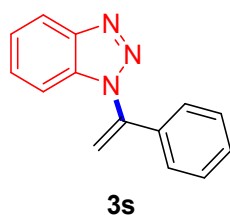
1-(1-(*p*-tolyl)vinyl)-1H-benzo[*d*][1,2,3]triazole (3p)^[4]: R_f = 0.25 (Petroleum ether/EtOAc, 15:1). 7.1 mg, 15% yield. Yellow oil. ¹H NMR (600 MHz, Chloroform-*d*) δ 8.13-8.09 (m, 1H), 7.40-7.34 (m, 2H), 7.18 (s, 4H), 7.10-7.05 (m, 1H), 5.75 (d, J = 1.0 Hz, 1H), 5.72 (d, J = 1.1 Hz, 1H), 2.39 (s, 3H). ¹³C NMR (151 MHz, Chloroform-*d*) δ 146.2, 142.7, 140.0, 133.0, 131.9, 129.5, 127.7, 126.8, 124.1, 120.1, 111.3, 110.2, 21.3.



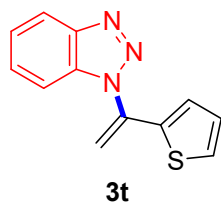
1-(1-(4-(Tert-butyl)phenyl)vinyl)-1H-benzo[*d*][1,2,3]triazole (3q)^[4]: R_f = 0.25 (Petroleum ether/EtOAc, 15:1). 15.5 mg, 28% yield. Yellow oil. ¹H NMR (600 MHz, Chloroform-*d*) δ 8.16-8.07 (m, 1H), 7.42-7.36 (m, 4H), 7.25-7.19 (m, 2H), 7.17-7.11 (m, 1H), 5.79 (s, 1H), 5.71 (s, 1H), 1.33 (s, 9H). ¹³C NMR (151 MHz, Chloroform-*d*) δ 153.2, 146.1, 142.7, 133.1, 131.8, 127.7, 126.6, 125.7, 125.6, 125.5, 124.1, 120.1, 111.2, 110.3, 34.8, 31.3, 31.2, 31.0.



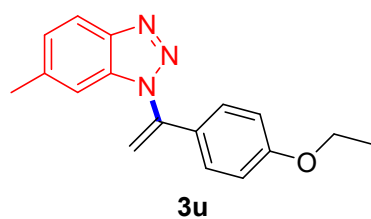
1-(1-(4-Phenoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3r): $R_f = 0.25$ (Petroleum ether/EtOAc, 10:1). 25.0 mg, 40% yield. Yellow oil. $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 8.12 (d, $J = 8.1$ Hz, 1H), 7.44-7.33 (m, 4H), 7.28-7.22 (m, 2H), 7.20-7.11 (m, 2H), 7.10-7.03 (m, 2H), 7.02-6.95 (m, 2H), 5.75 (s, 1H), 5.70 (s, 1H). $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 159.0, 156.3, 146.2, 142.2, 133.0, 129.9, 129.4, 128.5, 127.8, 124.2, 124.0, 120.2, 119.6, 118.4, 111.2, 110.1. HRMS (ESI): m/z : calcd for $\text{C}_{20}\text{H}_{15}\text{N}_3\text{ONa}$ ($\text{M}+\text{Na}$) $^+$ 336.1107; found 336.1113.



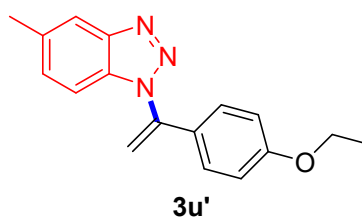
1-(1-phenylvinyl)-1H-benzo[d][1,2,3]triazole (3s)⁴¹: $R_f = 0.25$ (Petroleum ether/EtOAc, 20:1). 4.4 mg, 10% yield. Yellow oil. $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 8.21-8.04 (m, 1H), 7.43-7.34 (m, 5H), 7.32-7.25 (m, 2H), 7.09-6.98 (m, 1H), 5.79 (s, 1H), 5.77 (s, 1H). $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 146.2, 142.7, 134.6, 132.9, 129.8, 128.9, 127.9, 126.9, 124.2, 120.1, 111.3, 111.1.



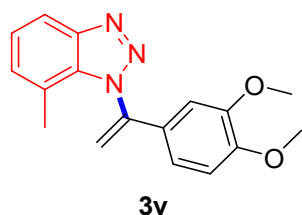
1-(1-(Thiophen-2-yl)vinyl)-1H-benzo[d][1,2,3]triazole (3t): $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). 20.0 mg, 44% yield. Yellow oil. $^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 8.16-8.07 (m, 1H), 7.47-7.29 (m, 4H), 7.00 (q, $J = 5.1, 3.7$ Hz, 1H), 6.91 (q, $J = 3.7, 1.2$ Hz, 1H), 5.85 (d, $J = 1.4$ Hz, 1H), 5.60 (d, $J = 1.4$ Hz, 1H). $^{13}\text{C NMR}$ (101 MHz, Chloroform-*d*) δ 146.0, 137.7, 136.8, 133.0, 128.0, 127.8, 127.4, 127.2, 124.3, 120.2, 111.0, 110.4. HRMS (ESI): m/z : calcd for $\text{C}_{12}\text{H}_9\text{N}_3\text{SNa}$ ($\text{M}+\text{Na}$) $^+$ 250.0409; found 250.0409. Calcd for $\text{C}_{12}\text{H}_9\text{N}_3\text{SK}$ ($\text{M}+\text{K}$) $^+$ 266.0149; found 266.0149.



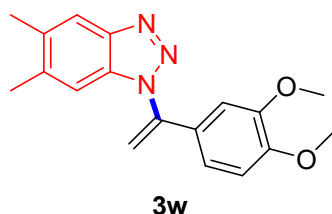
and



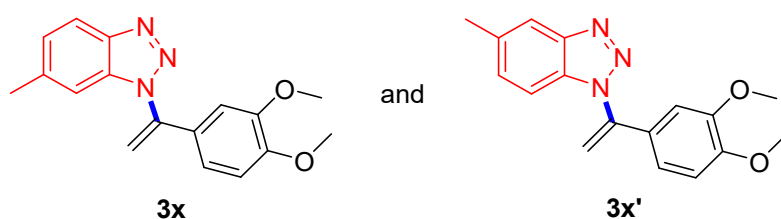
1-(1-(4-Ethoxyphenyl)vinyl)-6-methyl-1H-benzo[d][1,2,3]triazole (3u) and **1-(1-(4-Ethoxyphenyl)vinyl)-5-methyl-1H-benzo[d][1,2,3]triazole (3u')**: $R_f = 0.25$ (Petroleum ether/EtOAc, 10:1). Isomer (1.9:1 based on NMR), 25.1 mg, 45% yield. Orange oil. $^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.97 (d, $J = 8.5$ Hz, 0.5H), 7.86 (s, 1H), 7.23-7.17 (m, 4.5H), 6.97-6.94 (m, 1H), 6.91-6.85 (m, 3.5H), 5.69 (s, 0.5H), 5.65 (s, 1H), 5.63 (s, 1H), 5.60 (s, 0.5H), 4.08-4.02 (m, 3H), 2.50 (s, 3H), 2.42 (s, 1.5H), 1.45-1.40 (m, 4.5H). $^{13}\text{C NMR}$ (101 MHz, Chloroform-*d*) δ 160.2 (160.19), 160.2 (160.18), 146.7, 144.7, 142.4 (142.39), 142.4 (142.35), 138.4, 134.2, 133.5, 131.5, 129.8, 128.4, 128.2, 127.1, 127.0, 126.4, 119.5, 119.0, 114.7 (114.67), 114.7 (114.66), 110.9, 110.4, 109.2, 108.9, 63.6, 22.0, 21.4, 14.7. HRMS (ESI): m/z : calcd for $\text{C}_{17}\text{H}_{17}\text{N}_3\text{ONa}$ ($\text{M}+\text{Na}$) $^+$ 302.1264; found 302.1265. Calcd for $\text{C}_{17}\text{H}_{17}\text{N}_3\text{OK}$ ($\text{M}+\text{K}$) $^+$ 318.1003; found 318.1005.



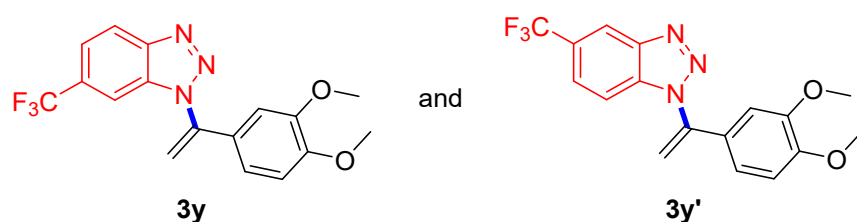
1-(1-(3,4-Dimethoxyphenyl)vinyl)-7-methyl-1H-benzo[d][1,2,3]triazole (3v): $R_f = 0.25$ (Petroleum ether/EtOAc, 10:1). 31.9 mg, 54% yield. Yellow oil. $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.25 (t, 1H), 7.15-7.11 (m, 1H), 6.89 (d, $J = 8.4$ Hz, 1H), 6.85 (d, $J = 1.2$ Hz, 2H), 6.81 (t, $J = 1.2$ Hz, 1H), 5.69 (d, 1H), 5.66 (d, 1H), 3.91 (s, 3H), 3.78 (s, 3H), 2.84 (s, 3H). $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 150.5, 149.2, 146.0, 142.6, 132.9, 131.0, 127.7, 127.6, 124.0, 120.1, 111.2, 110.2, 109.4, 108.6, 56.0 (56.03), 56.0 (55.99), 16.7. HRMS (ESI): m/z : calcd for $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 318.1213; found 318.1213.



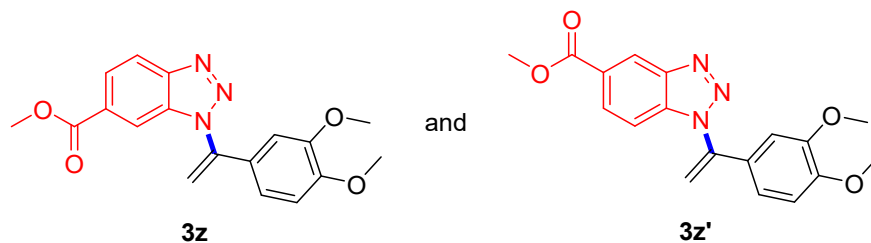
1-(1-(3,4-Dimethoxyphenyl)vinyl)-5,6-dimethyl-1H-benzo[d][1,2,3]triazole (3w): $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). 30.9 mg, 50% yield. Yellow oil. $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.83 (s, 1H), 6.90 (s, 1H), 6.84 (q, 3H), 5.67 (s, 1H), 5.62 (s, 1H), 3.91 (d, $J = 1.5$ Hz, 3H), 3.79 (d, $J = 1.4$ Hz, 3H), 2.40 (s, 3H), 2.31 (s, 3H). $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 150.5, 149.2, 145.3, 142.6, 138.1, 133.9, 132.1, 127.7, 120.1, 119.1, 111.3, 110.7, 110.2, 109.1, 56.0 (56.03), 56.0 (55.99), 20.8, 20.3. HRMS (ESI): m/z : calcd for $\text{C}_{18}\text{H}_{19}\text{N}_3\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 332.1369; found 332.1366. Calcd for $\text{C}_{18}\text{H}_{19}\text{N}_3\text{O}_2\text{K}$ ($\text{M}+\text{K}$) $^+$ 348.1109; found 348.1110.



1-(1-(3,4-Dimethoxyphenyl)vinyl)-6-methyl-1H-benzo[d][1,2,3]triazole (3x) and **1-(1-(3,4-Dimethoxyphenyl)vinyl)-5-methyl-1H-benzo[d][1,2,3]triazole (3x')**: $R_f = 0.25$ (Petroleum ether/EtOAc, 10:1). Isomer (1.7:1 based on NMR), 29.5 mg, 50% yield. Yellow oil. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.97 (d, $J = 8.5$ Hz, 0.6H), 7.85 (q, $J = 1.2$ Hz, 1H), 7.21-7.18 (m, 1.6H), 6.97 (d, $J = 8.5$ Hz, 1H), 6.92 (q, $J = 1.1$ Hz, 0.6H), 6.86-6.81 (m, 4.8H), 5.71 (d, $J = 1.0$ Hz, 0.6H), 5.67-5.65 (m, 2H), 5.63 (d, $J = 0.9$ Hz, 0.6H), 3.92-3.90 (m, 4.8H), 3.81-3.78 (m, 4.8H), 2.51-2.49 (m, 3H), 2.44-2.41 (m, 1.8H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 150.4, 149.2, 146.7, 144.7, 142.5, 142.4, 138.5, 134.2, 133.5, 131.5, 129.8, 127.6, 127.5, 126.4, 120.1, 120.0, 119.5, 119.0, 111.2, 111.1, 110.9, 110.4, 110.1, 109.9, 109.5, 109.1, 56.0 (55.99), 56.0 (55.97), 22.0, 21.4. HRMS (ESI): m/z : calcd for $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 318.1213; found 318.1213. Calcd for $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}_2\text{K}$ ($\text{M}+\text{K}$) $^+$ 334.0952; found 334.0952.

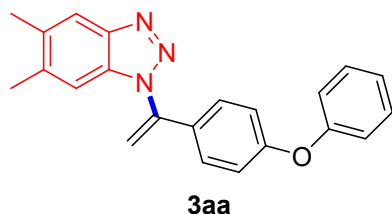


1-(1-(3,4-Dimethoxyphenyl)vinyl)-6-(trifluoromethyl)-1H-benzo[d][1,2,3]triazole (3y) and **1-(1-(3,4-Dimethoxyphenyl)vinyl)-5-(trifluoromethyl)-1H-benzo[d][1,2,3]triazole (3y')**: $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). Isomer (1.8:1 based on NMR), 38.4 mg, 55% yield. Yellow oil. ^1H NMR (400 MHz, Chloroform-*d*) δ 8.46-8.40 (m, 0.6H), 8.26-8.22 (m, 1H), 7.66-7.58 (m, 1.8H), 7.52-7.49 (m, 1H), 7.21 (d, $J = 8.7$ Hz, 0.6H), 6.88-6.76 (m, 4.6H), 5.81 (d, $J = 1.2$ Hz, 1H), 5.76 (d, $J = 1.2$ Hz, 0.6H), 5.73 (d, $J = 1.1$ Hz, 0.6H), 5.70 (d, $J = 1.3$ Hz, 1H), 3.94-3.91 (m, 4.8H), 3.84-3.81 (m, 4.8H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 150.8, 150.7, 149.4 (149.37), 149.4 (149.35), 147.3, 145.3, 142.1, 142.0, 134.3, 132.4, 130.0 (q, $J = 32.6$ Hz), 127.0, 126.8 (126.84), 126.8 (126.79), 126.7, 124.4 (q, $J = 3.2$ Hz), 123.7 (q, $J = 272.8$ Hz), 121.2, 121.0 (q, $J = 3.1$ Hz), 120.1, 120.0, 118.3 (q, $J = 4.4$ Hz), 112.4, 111.2 (111.23), 111.2 (111.21), 110.3, 110.2, 109.8 (109.84), 109.8 (109.77), 109.4 (q, $J = 4.6$ Hz), 56.0 (56.01), 56.0 (55.98), 56.0 (55.97). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -61.47, -61.75. HRMS (ESI): m/z : calcd for $\text{C}_{17}\text{H}_{15}\text{F}_3\text{N}_3\text{O}_2$ ($\text{M}+\text{H}$) $^+$ 350.1111; found 350.1116.

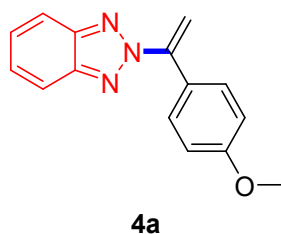


Methyl 1-(1-(3,4-dimethoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole-6-carboxylate (3z) and **Methyl 1-(1-(3,4-dimethoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole-5-carboxylate (3z')**: $R_f = 0.25$ (Petroleum ether/EtOAc, 10:1). Isomer (1.5:1 based on NMR), 49.5 mg, 73% yield. Yellow oil. ^1H NMR (600 MHz, Chloroform-*d*) δ 8.87-8.81 (m, 0.7H), 8.15 (d, $J = 8.7$ Hz, 1H), 8.08-8.04 (m, 1.7H), 8.00-7.93 (m, 1H), 7.10 (d, $J = 8.8$ Hz, 0.7H), 6.87-6.79 (m, 5.1H), 5.81 (d, $J = 1.3$ Hz,

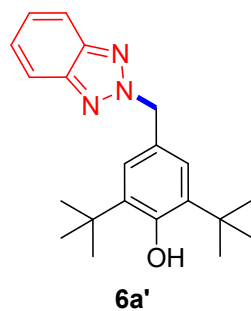
1H), 5.75-5.73 (m, 0.7H), 5.73-5.71 (m, 0.7H), 5.67 (d, $J = 1.3$ Hz, 1H), 3.98 (s, 2.1H), 3.94-3.90 (m, 8.1H), 3.81 (s, 3H), 3.79 (s, 2.1H). ^{13}C NMR (151 MHz, Chloroform- d) δ 166.4, 166.3, 150.8 (150.80), 150.8 (150.76), 149.4, 148.0, 145.9, 142.2, 135.2, 133.0, 129.8, 128.5, 127.2, 127.1, 126.7, 125.0, 122.9, 120.1, 120.0, 113.6, 111.3, 111.2, 110.4, 110.1, 110.0, 109.9, 56.1, 56.0, 52.5, 52.4. HRMS (ESI): m/z : calcd for $\text{C}_{18}\text{H}_{17}\text{N}_3\text{O}_4\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 362.1111; found 362.1111. Calcd for $\text{C}_{18}\text{H}_{17}\text{N}_3\text{O}_4\text{K}$ ($\text{M}+\text{K}$) $^+$ 378.0851; found 378.0855.



5,6-Dimethyl-1-(1-(4-phenoxyphenyl)vinyl)-1H-benzo[d][1,2,3]triazole (3aa): $R_f = 0.25$ (Petroleum ether/EtOAc, 10:1). 25.2 mg, 37% yield. Yellow oil. ^1H NMR (400 MHz, Chloroform- d) δ 7.83 (s, 1H), 7.35 (t, $J = 7.7$ Hz, 2H), 7.24 (q, $J = 9.3, 2.7$ Hz, 2H), 7.15-7.11 (m, 1H), 7.06-7.02 (m, 2H), 6.99-6.94 (m, 3H), 5.72 (s, 1H), 5.64 (s, 1H), 2.39 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, Chloroform- d) δ 158.8, 156.3, 145.3, 142.2, 138.3, 134.1, 132.1, 129.9, 129.6, 128.5, 124.0, 119.5, 119.2, 118.5, 110.6, 109.8, 21.0, 20.4. HRMS (ESI): m/z : calcd for $\text{C}_{22}\text{H}_{19}\text{N}_3\text{ONa}$ ($\text{M}+\text{Na}$) $^+$ 364.1420; found 364.1420. Calcd for $\text{C}_{22}\text{H}_{19}\text{N}_3\text{OK}$ ($\text{M}+\text{K}$) $^+$ 380.1160; found 380.1153.

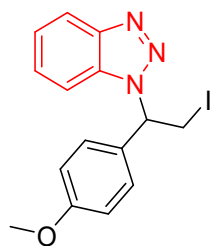


2-(1-(4-Methoxyphenyl)vinyl)-2H-benzo[d][1,2,3]triazole (4a): $R_f = 0.25$ (Petroleum ether/EtOAc, 30:1). 5.1 mg, 10% yield. Yellow oil. ^1H NMR (600 MHz, Chloroform- d) δ 7.93-7.88 (m, 2H), 7.43-7.36 (m, 4H), 6.97-6.93 (m, 2H), 6.10 (s, 1H), 5.58 (s, 1H), 3.85 (s, 3H). ^{13}C NMR (151 MHz, Chloroform- d) δ 160.7, 144.7, 129.7, 127.2, 127.1, 118.5, 113.9, 109.6, 55.4. HRMS (ESI): m/z : calcd for $\text{C}_{15}\text{H}_{13}\text{N}_3\text{ONa}$ ($\text{M}+\text{Na}$) $^+$ 274.0951; found 274.0961. Calcd for $\text{C}_{15}\text{H}_{13}\text{N}_3\text{OK}$ ($\text{M}+\text{K}$) $^+$ 290.0690; found 290.0709.



4-((2H-benzo[d][1,2,3]triazol-2-yl)methyl)-2,6-di-tert-butylphenol (6a')¹⁵: $R_f = 0.25$ (Petroleum ether/EtOAc, 20:1). 27.0 mg, 10% yield. Yellow oil. ^1H NMR (600 MHz, Chloroform- d) δ 7.87 (dd,

$J = 6.5, 3.1$ Hz, 2H), 7.36 (dd, $J = 6.6, 3.0$ Hz, 2H), 7.29 (s, 2H), 5.78 (s, 2H), 5.25 (s, 1H), 1.41 (s, 18H). ^{13}C NMR (151 MHz, Chloroform- d) δ 154.2, 144.6, 136.3, 126.2, 125.5 (125.54), 125.5 (125.45), 118.1, 60.8, 34.3, 30.2.



8a

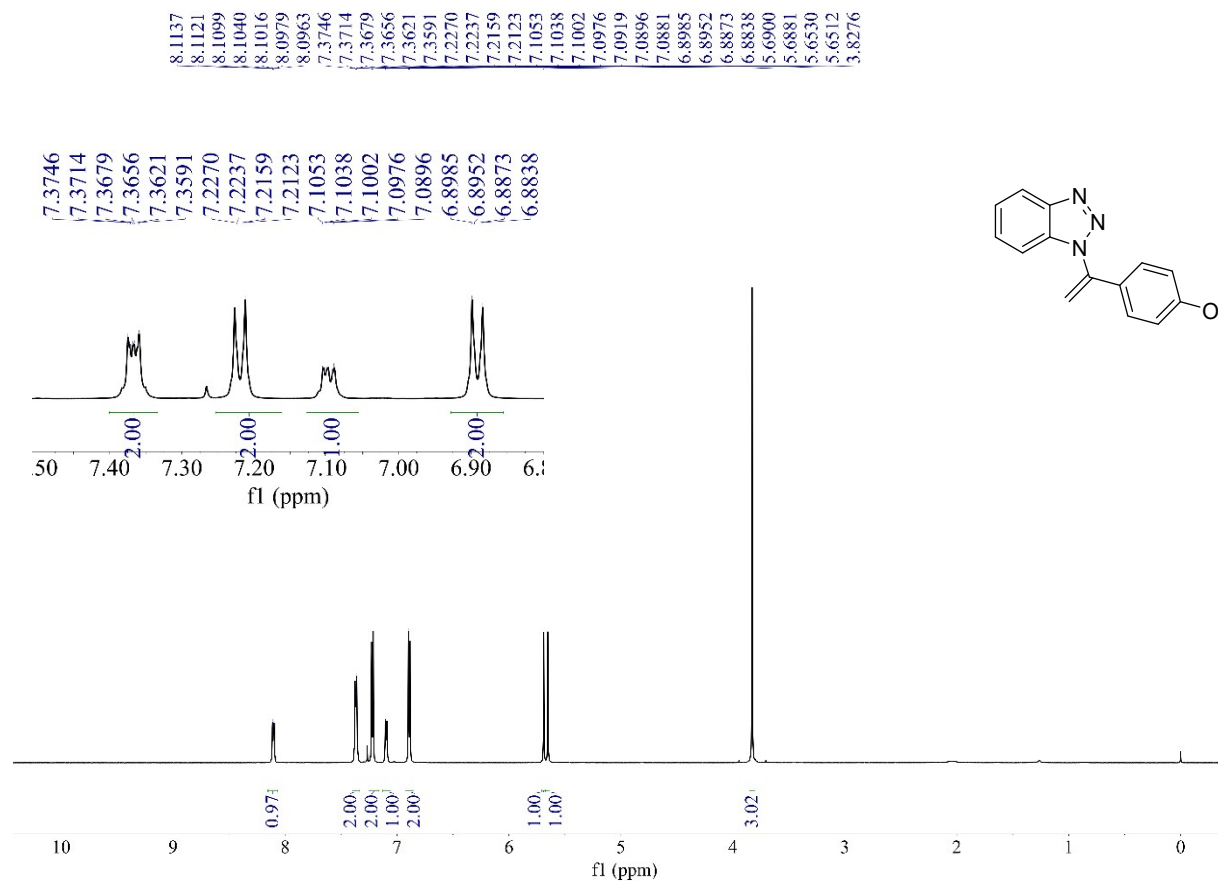
1-(2-Iodo-1-(4-methoxyphenyl)ethyl)-1H-benzo[d][1,2,3]triazole (8a)^[2]: $R_f = 0.25$ (Petroleum ether/EtOAc, 15:1). 64.4 mg, 85% yield. Yellow oil. ^1H NMR (400 MHz, Chloroform- d) δ 8.13-8.04 (m, 1H), 7.45-7.31 (m, 5H), 6.86 (dd, $J = 8.8, 1.9$ Hz, 2H), 5.96-5.88 (m, 1H), 4.48-4.38 (m, 1H), 4.03-3.95 (m, 1H), 3.76 (d, $J = 1.7$ Hz, 3H). ^{13}C NMR (101 MHz, Chloroform- d) δ 160.1, 146.0, 133.0, 129.6, 128.1, 127.6, 124.1, 120.2, 114.5, 109.4, 65.0, 55.3, 5.6.

8. References

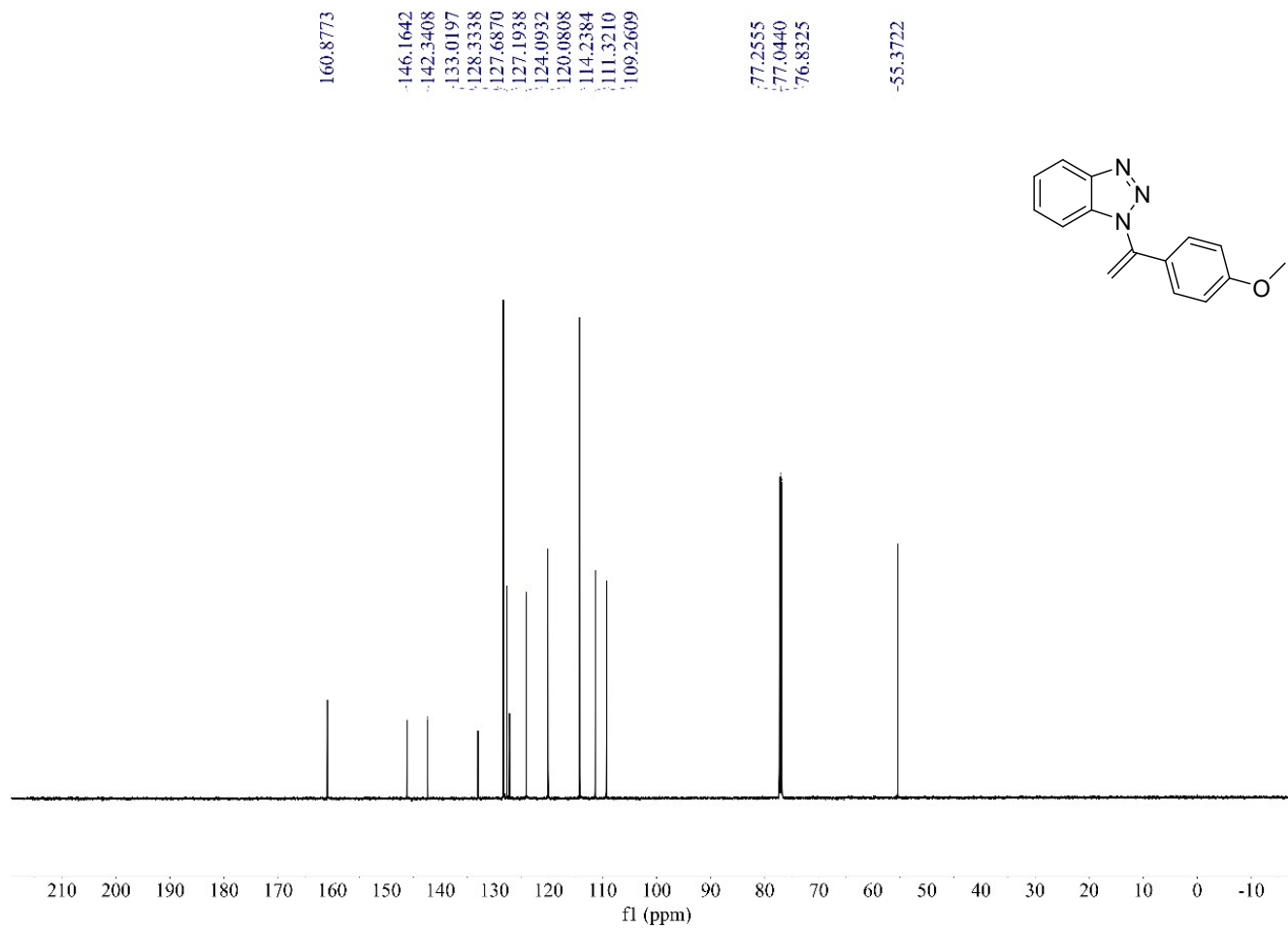
- [1] R. Damschroder and W. Peterson, *Organic Syntheses*, **1940**, 16-16.
- [2] K. Sun, F. Ma, L. Liu, J. Sun, X. Liu, Y. Wang, Z. Zhang, G. Zhang, *RSC Adv.* **2015**, *5*, 82492–82495.
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- [4] X. Wang, Q. Wang, Y. Xue, K. Sun, L. Wu, B. Zhang, *Chem. Commun.* **2020**, *56*, 4436–4439.
- [5] Y. Li, Y. Li, Y. Li, C. Chen, F. Ying, Y. Dong, D. Liang, *Synth. Commun.* **2019**, *49*, 2053–2065.

9. NMR spectra of products

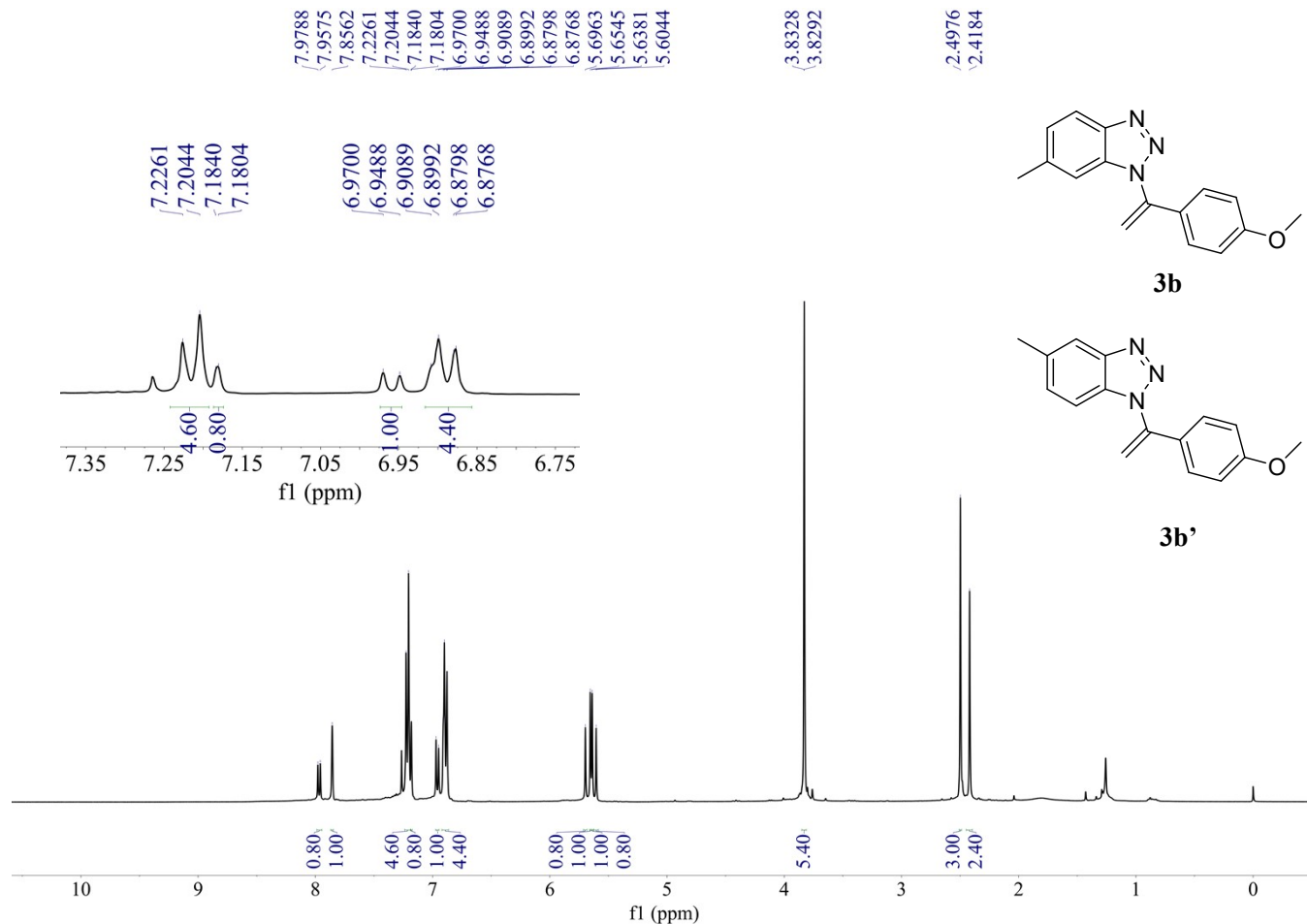
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3a



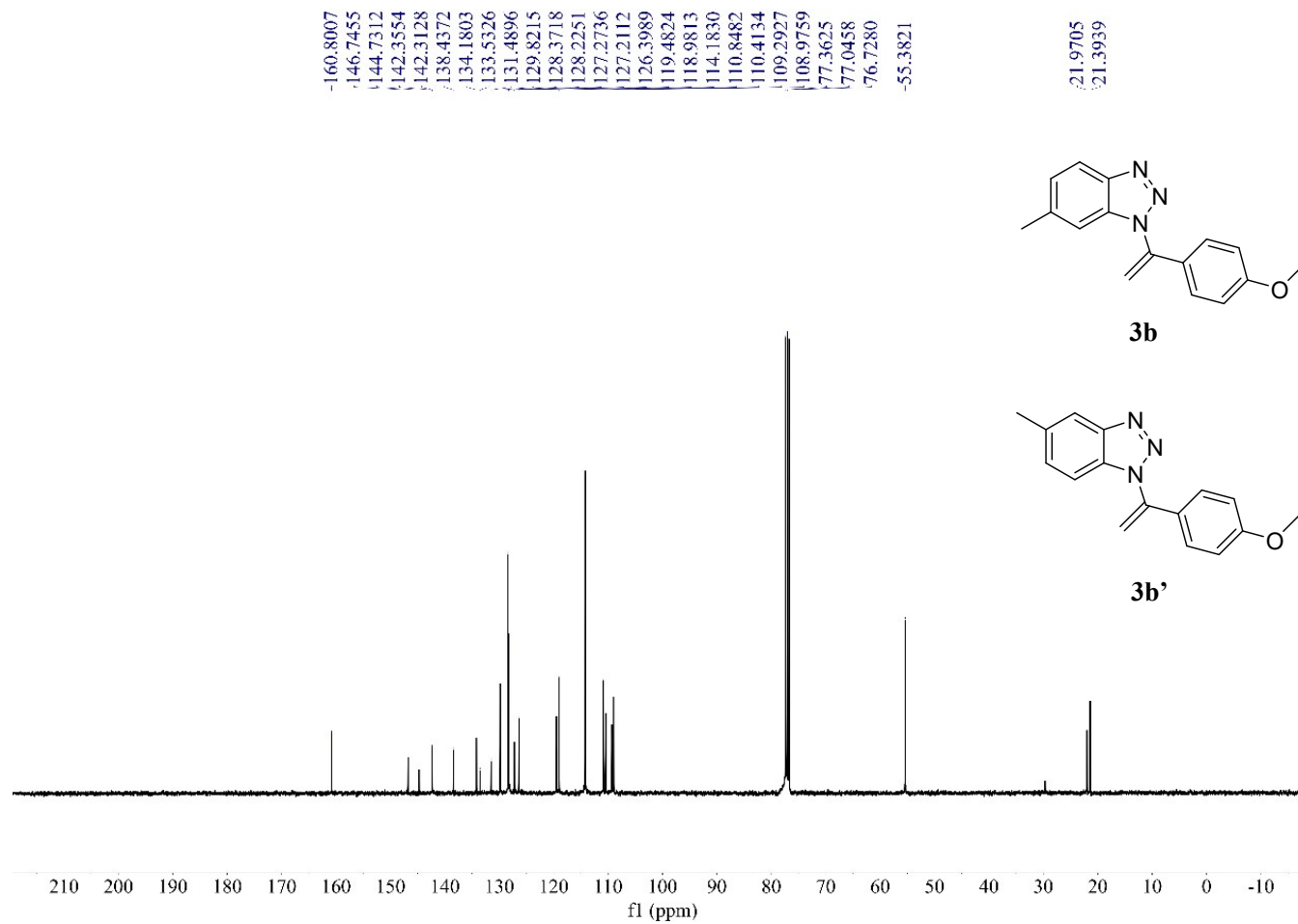
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3a



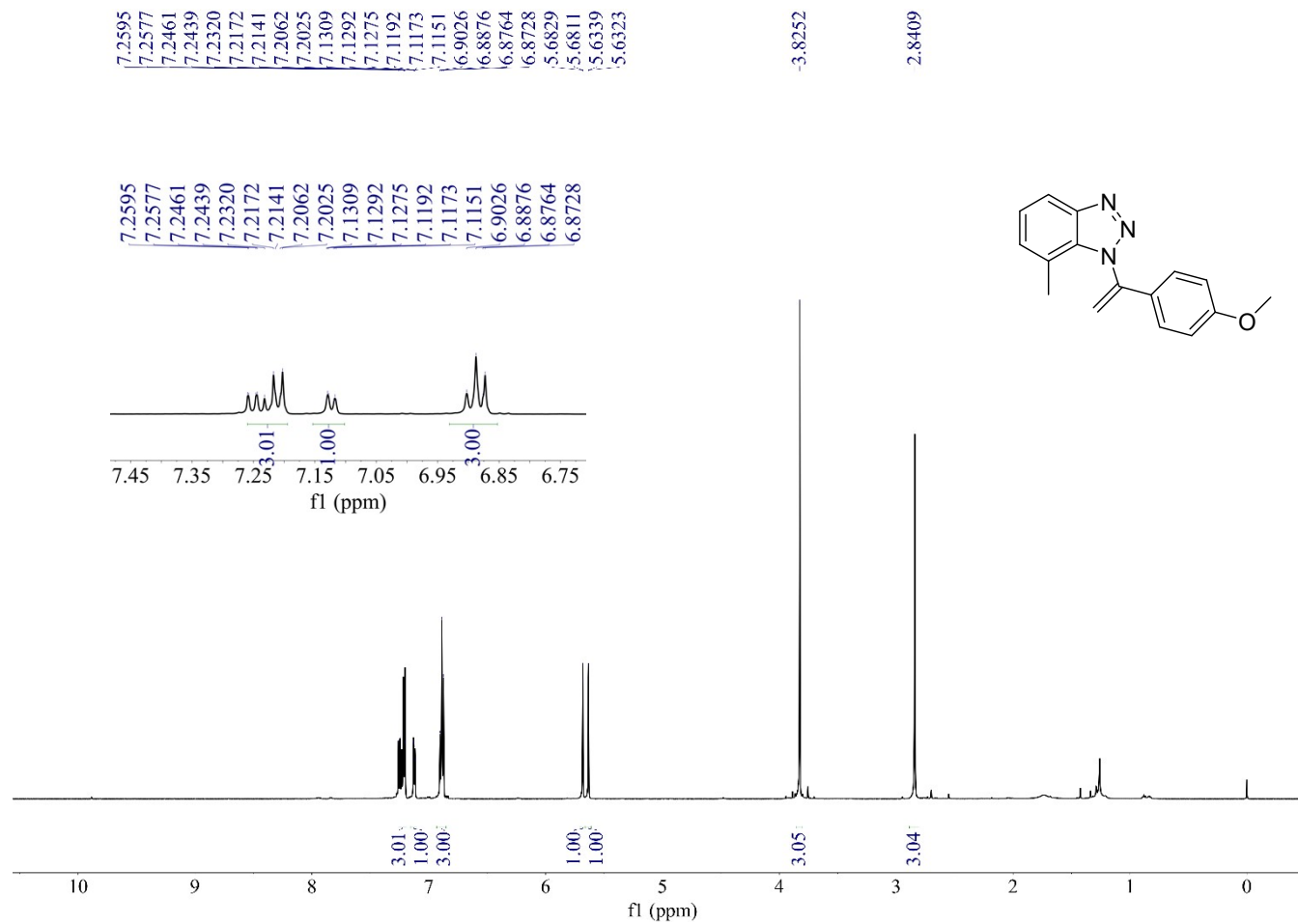
¹H-NMR Spectrum (400 MHz, CDCl₃) of 3b and 3b'



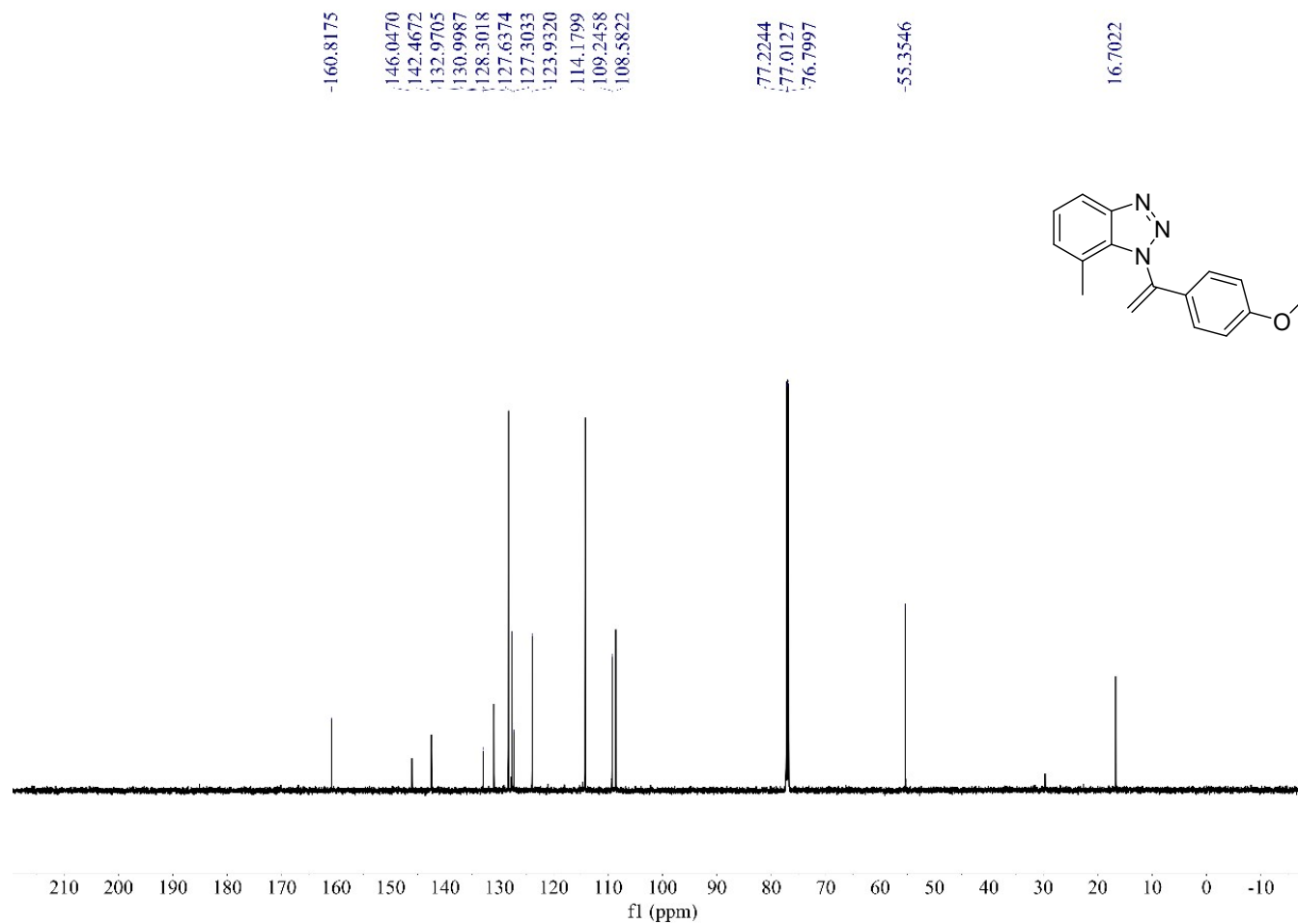
¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3b and 3b'



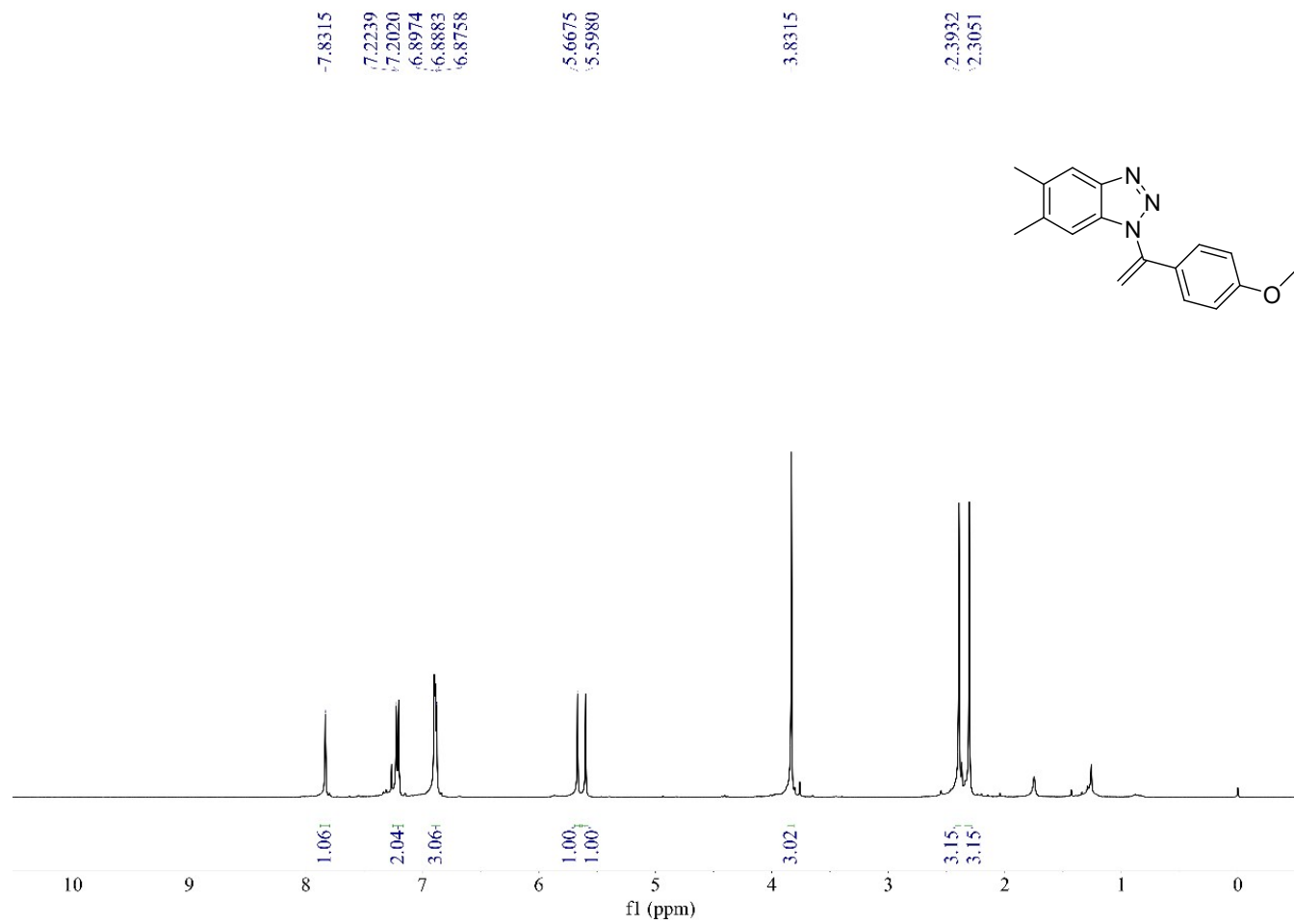
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3c



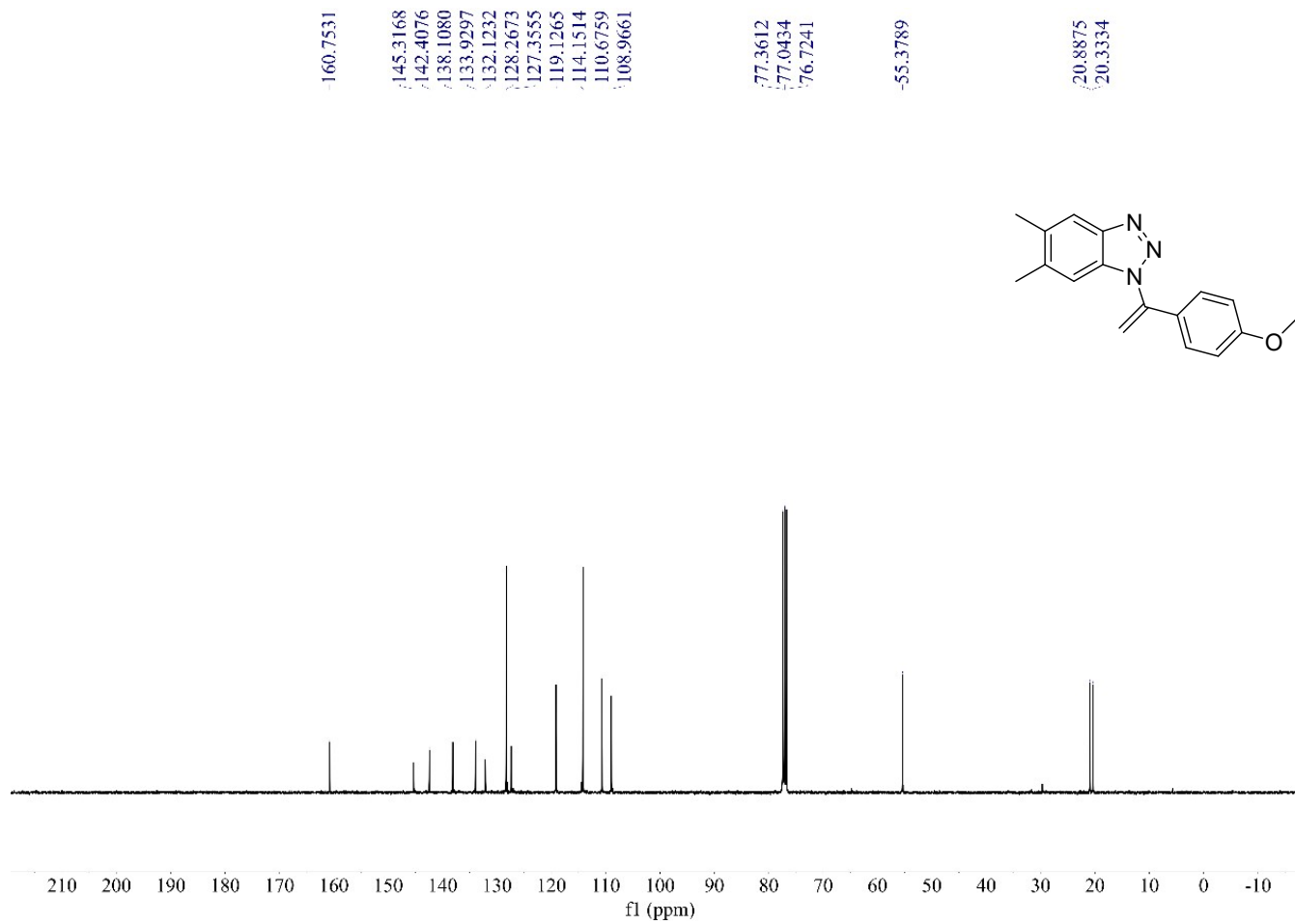
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3c



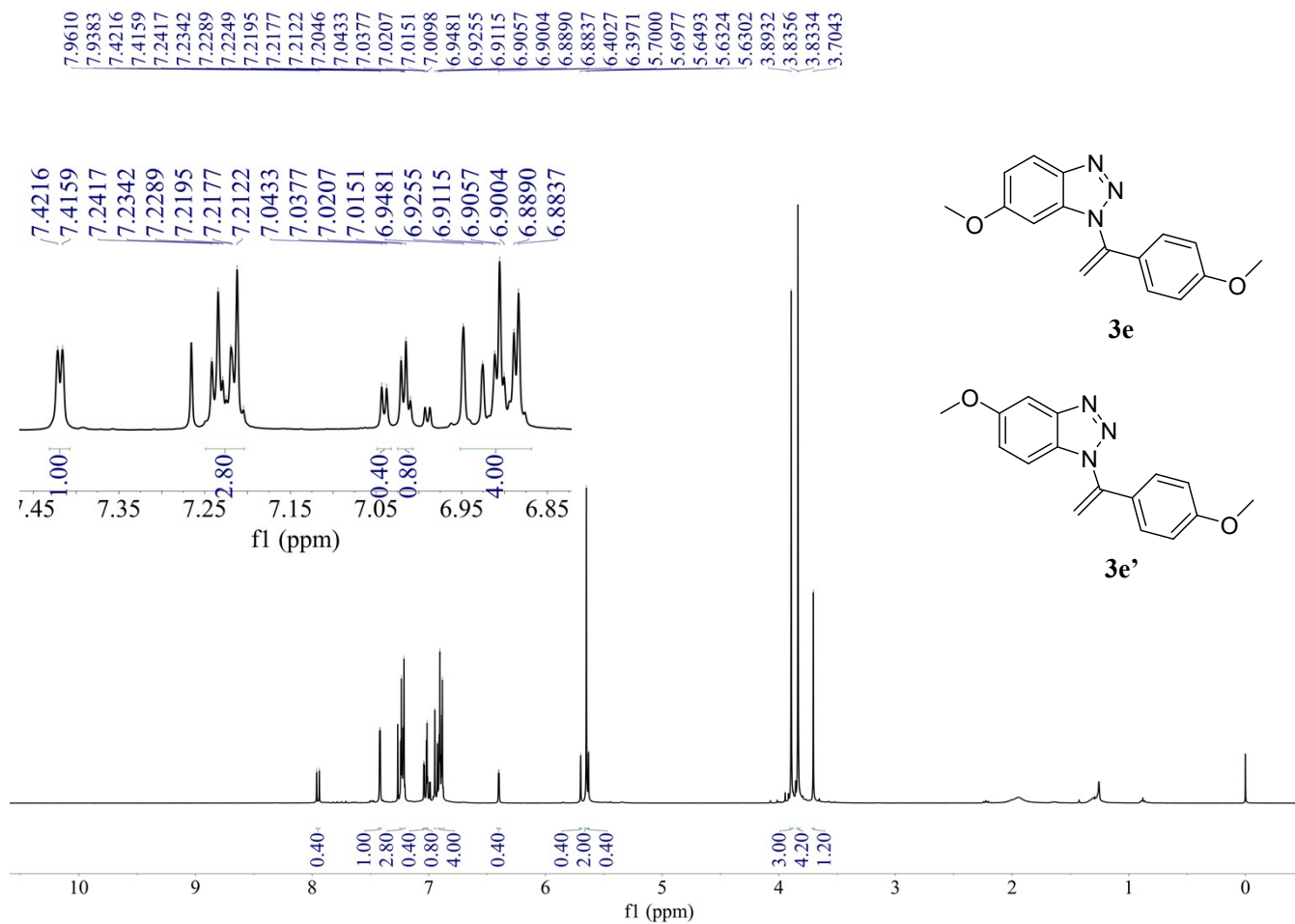
¹H-NMR Spectrum (400 MHz, CDCl₃) of 3d



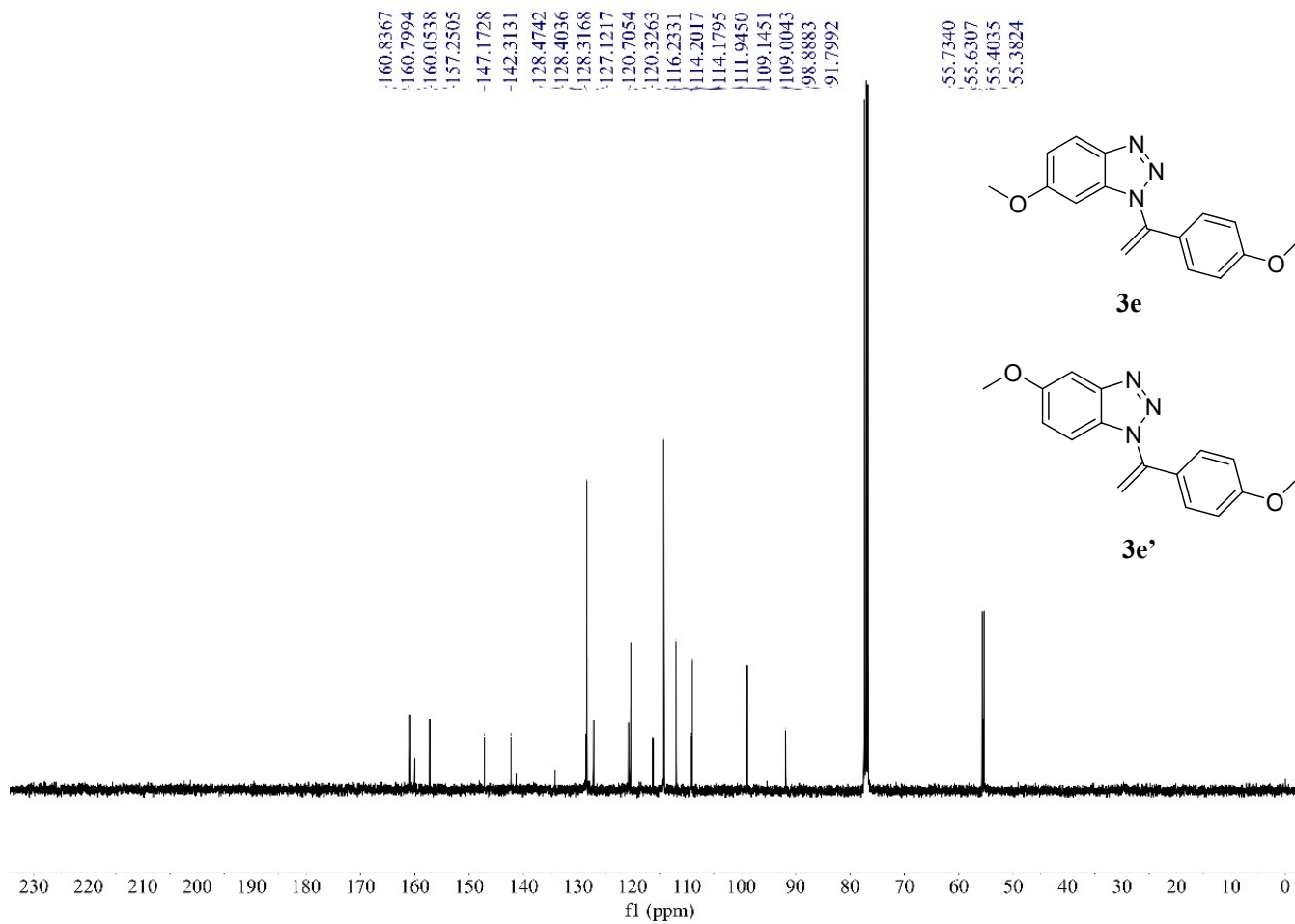
¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3d



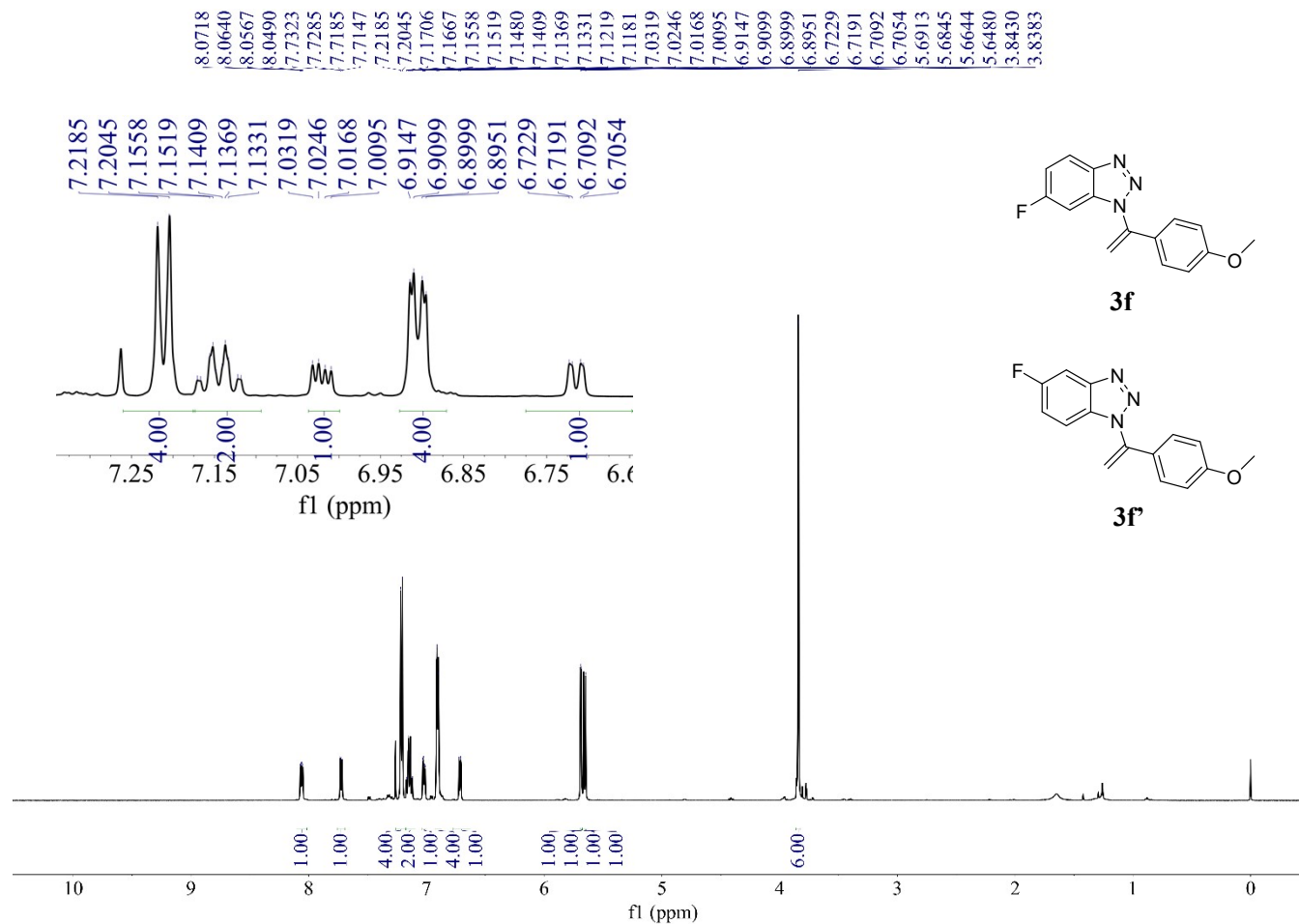
¹H-NMR Spectrum (400 MHz, CDCl₃) of 3e and 3e'



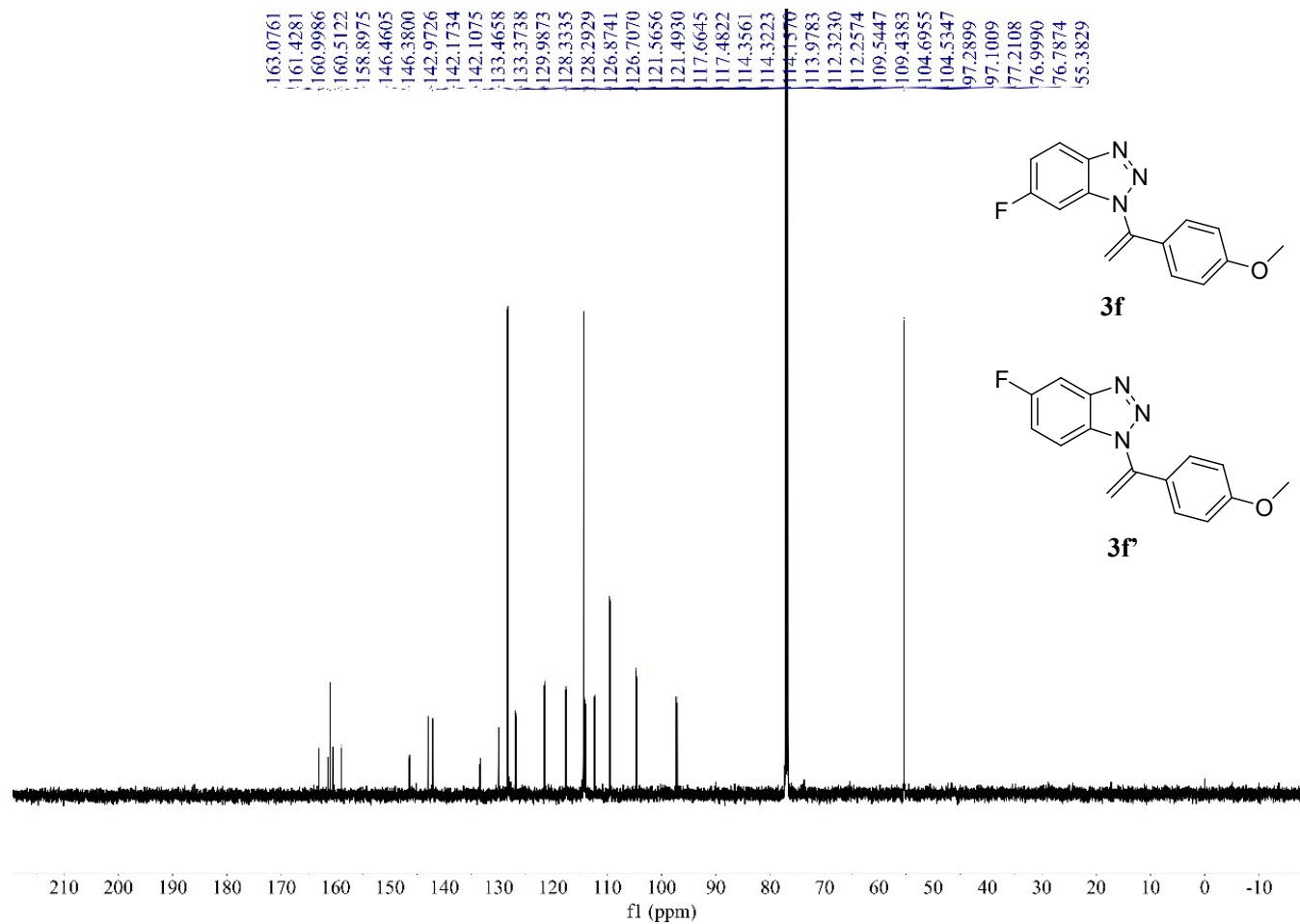
¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3e and 3e'



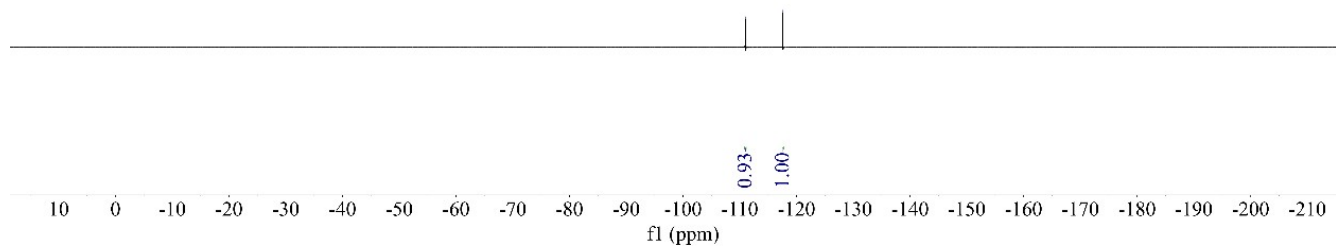
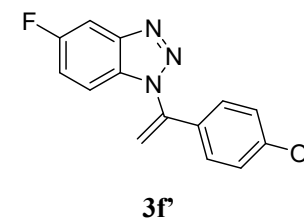
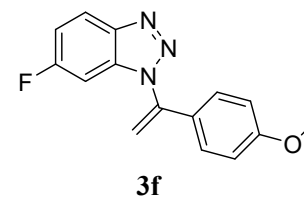
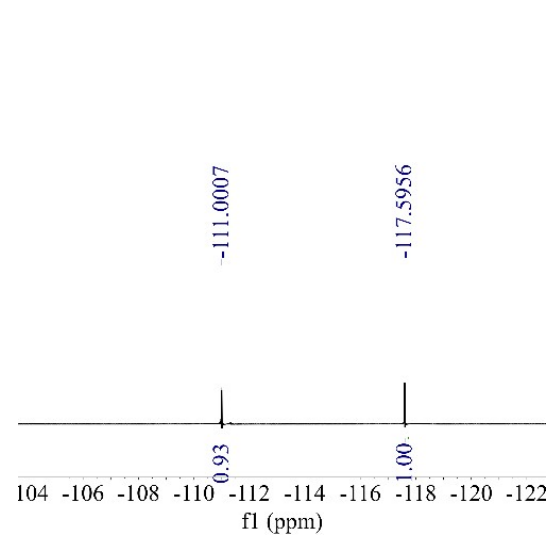
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3f and 3f'



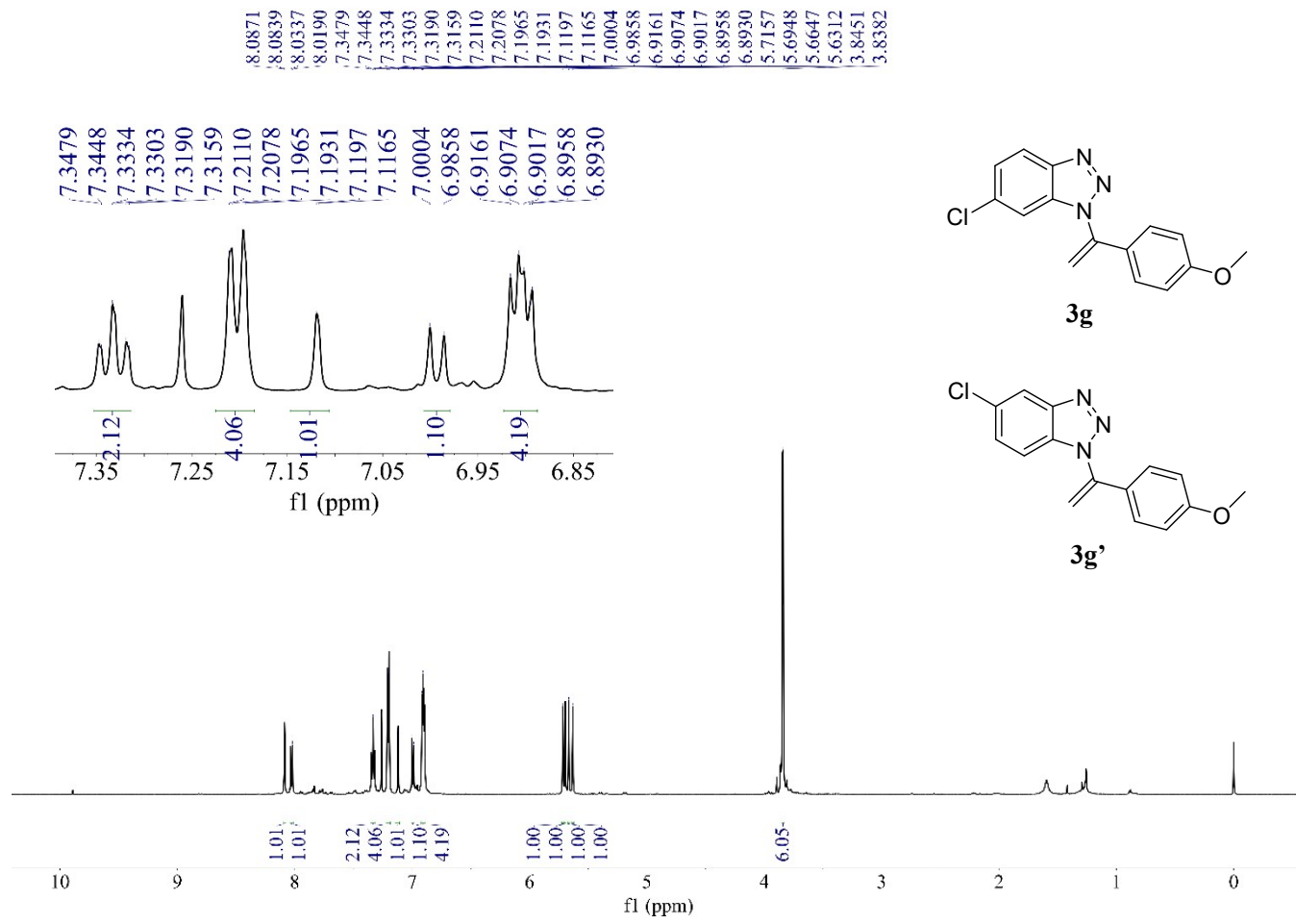
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3f and 3f'



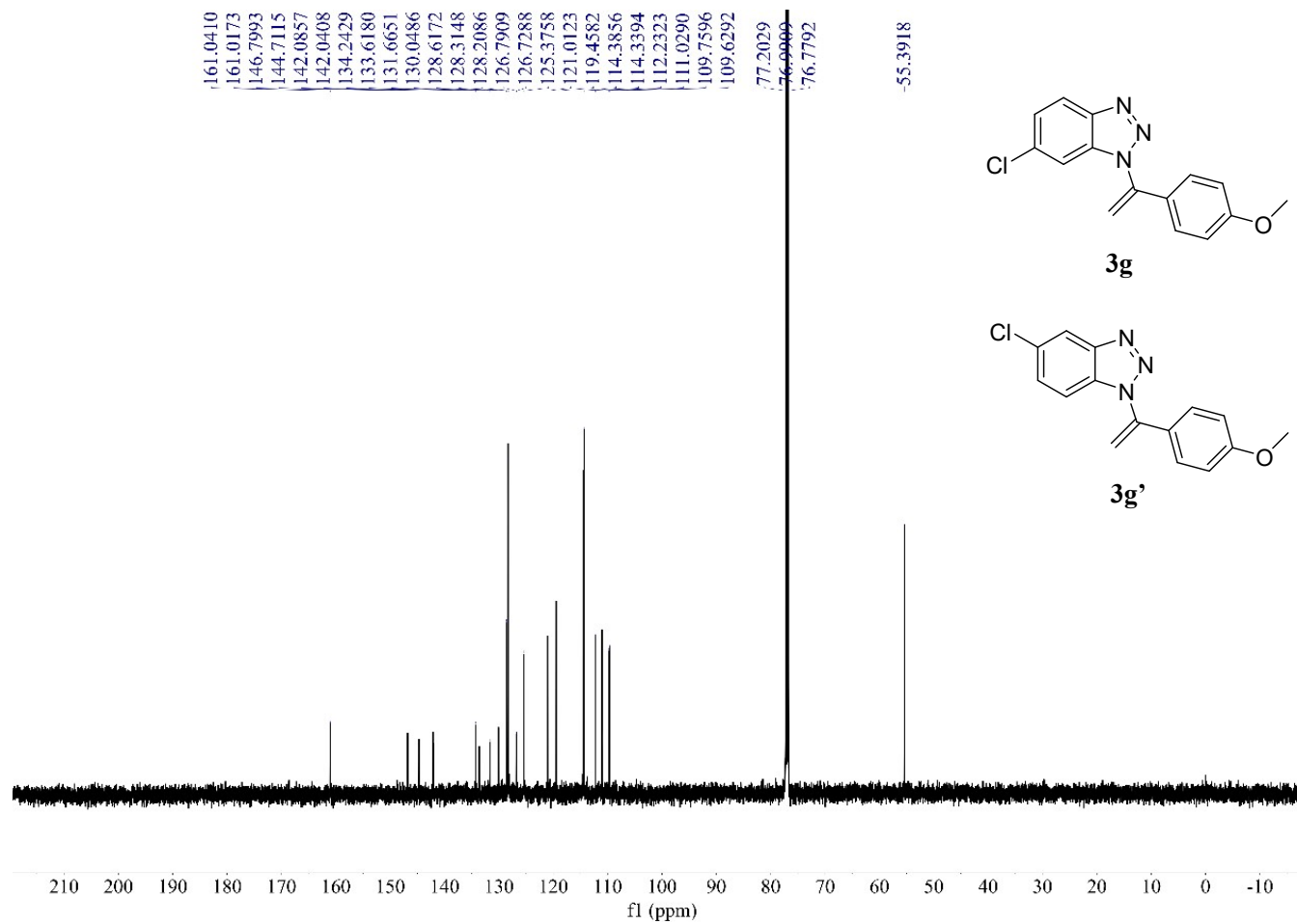
¹⁹F-NMR Spectrum (565 MHz, CDCl₃) of 3f and 3f'



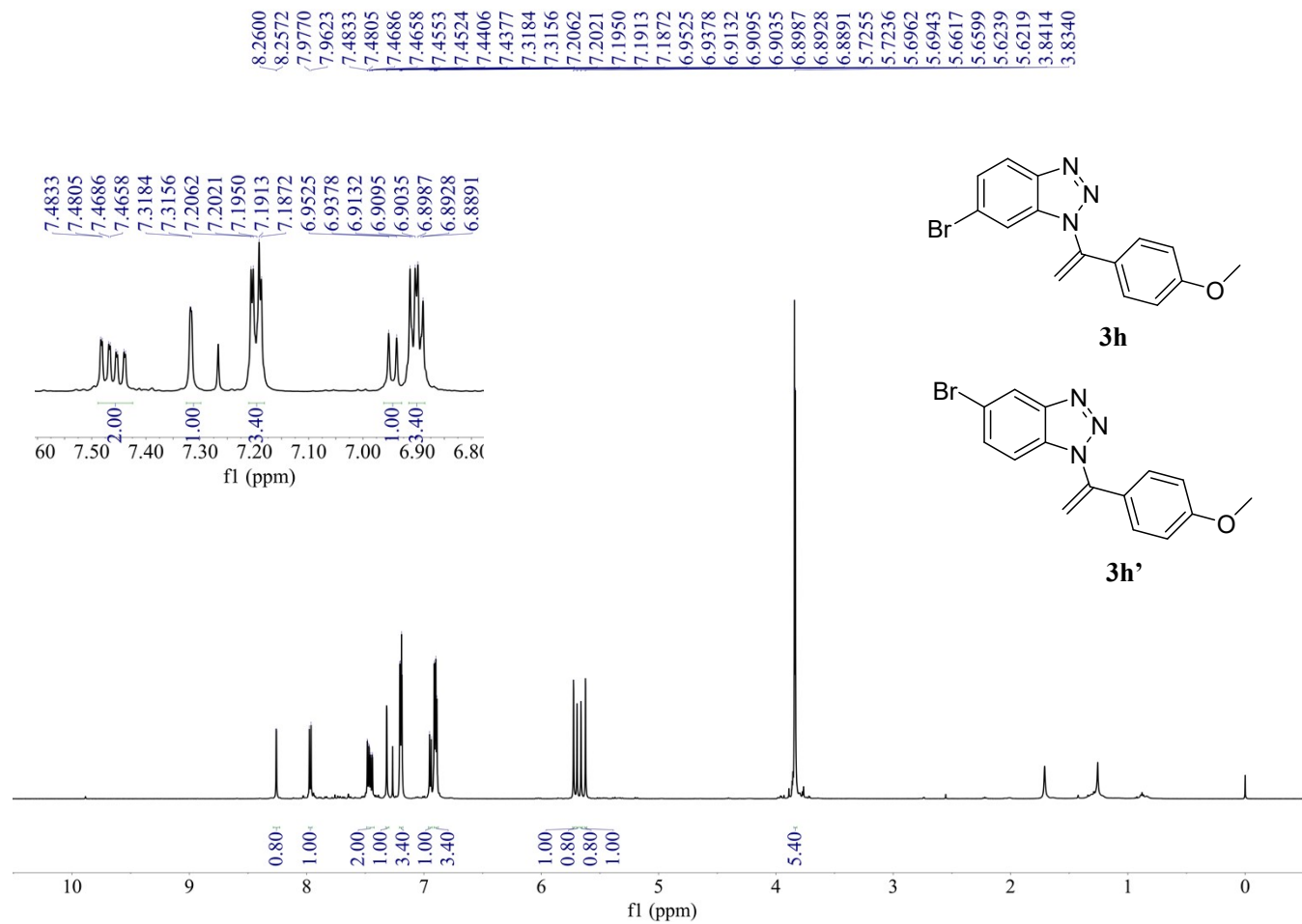
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3g and 3g'



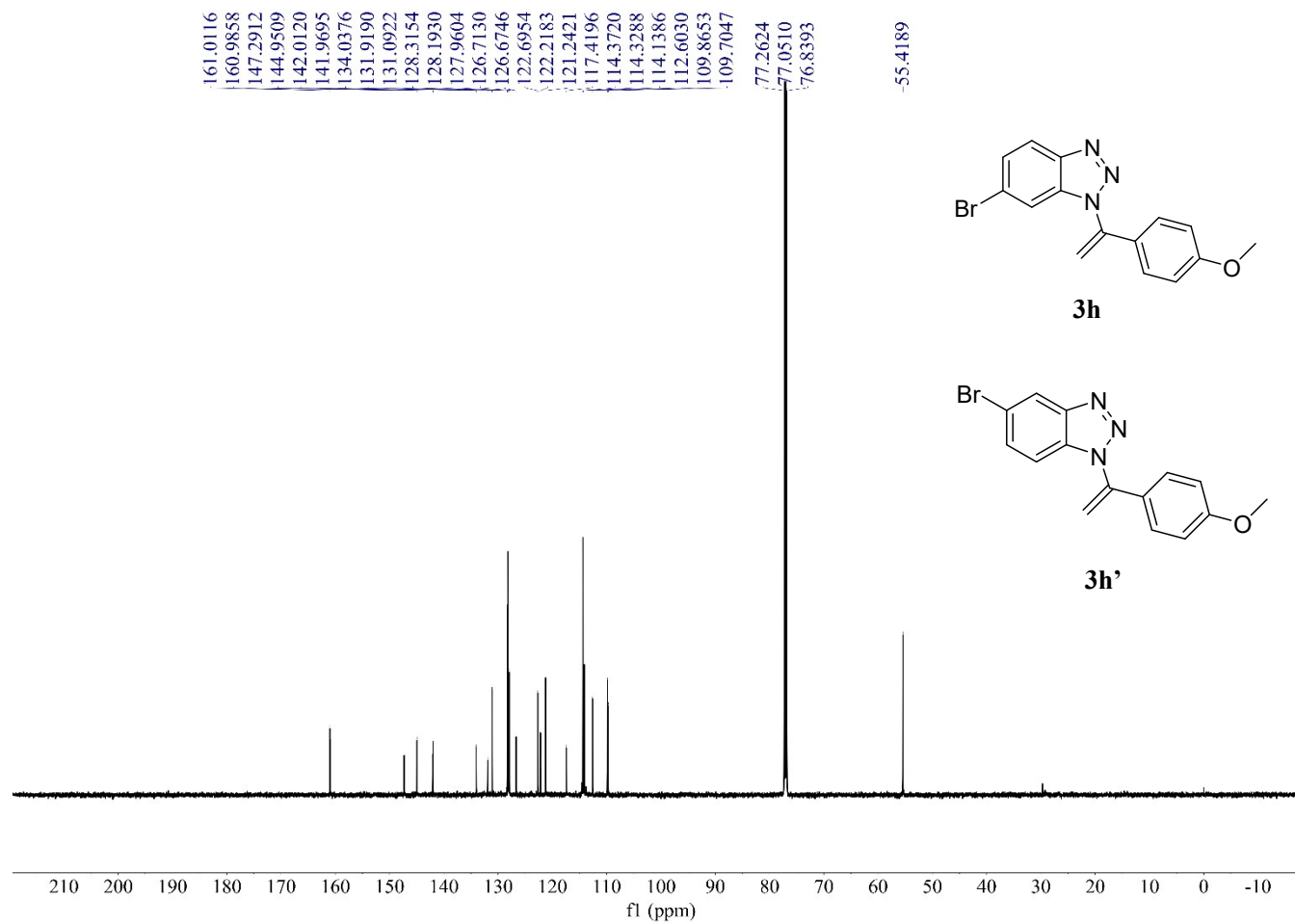
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3g and 3g'



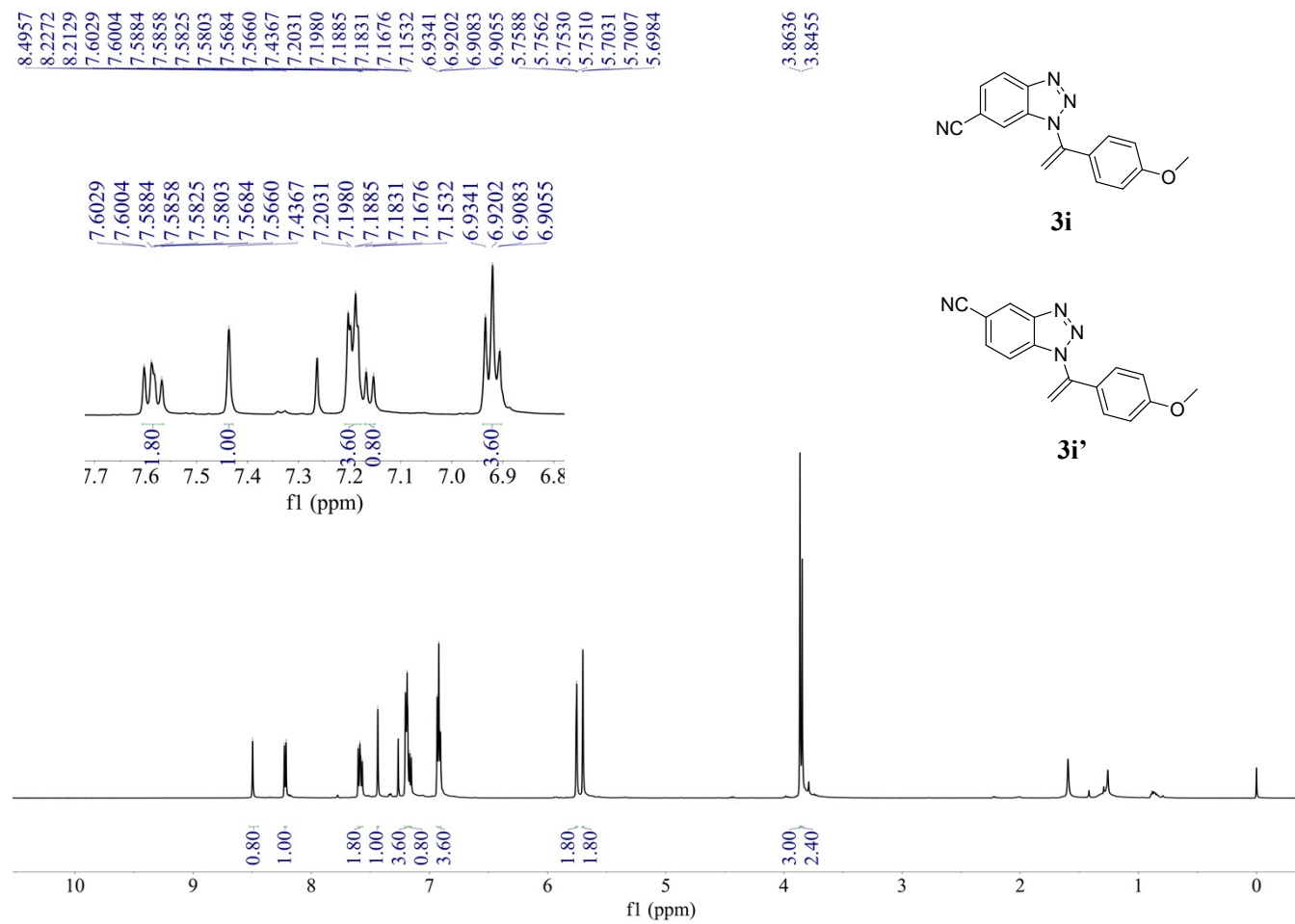
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3h and 3h'



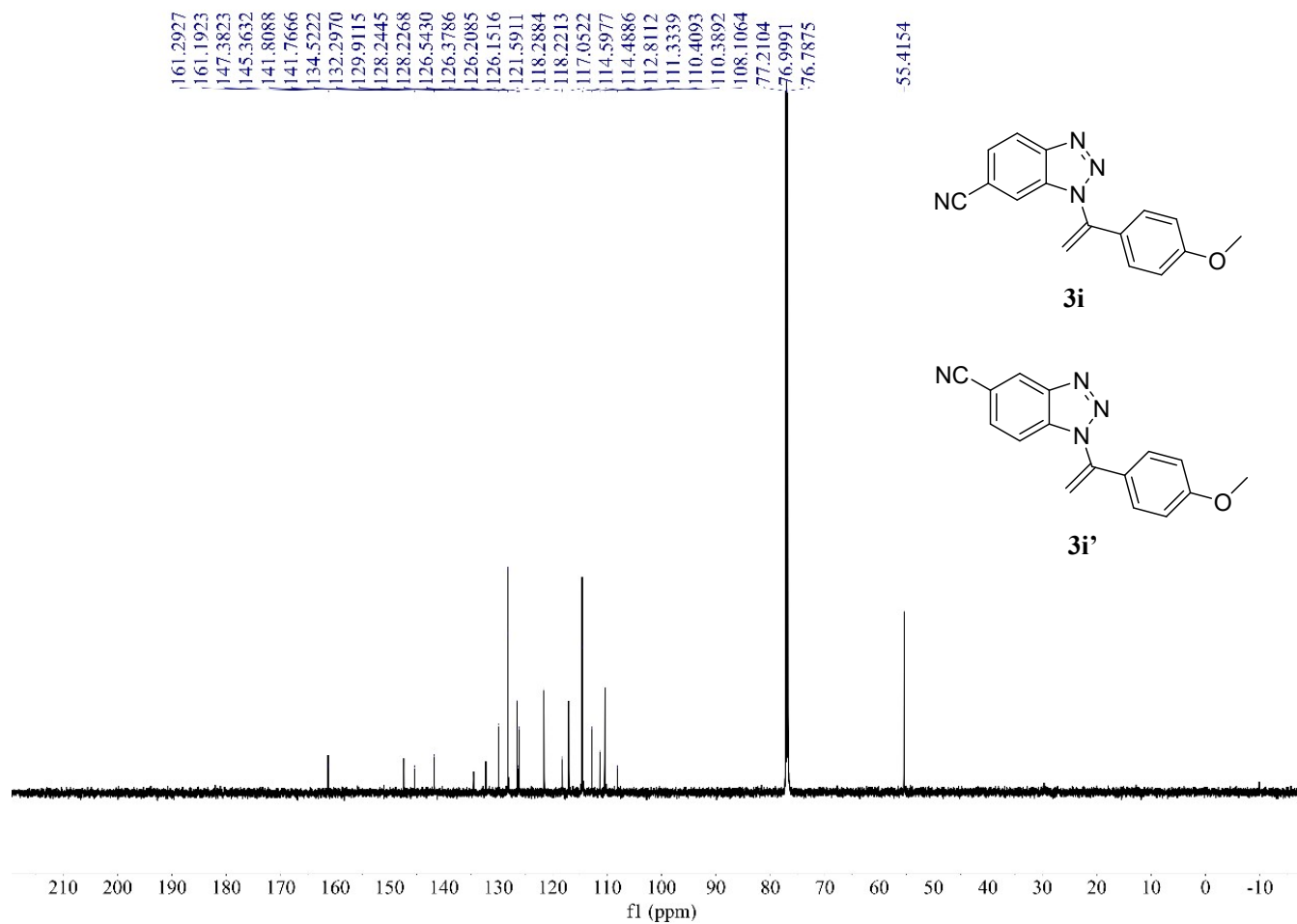
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3h and 3h'



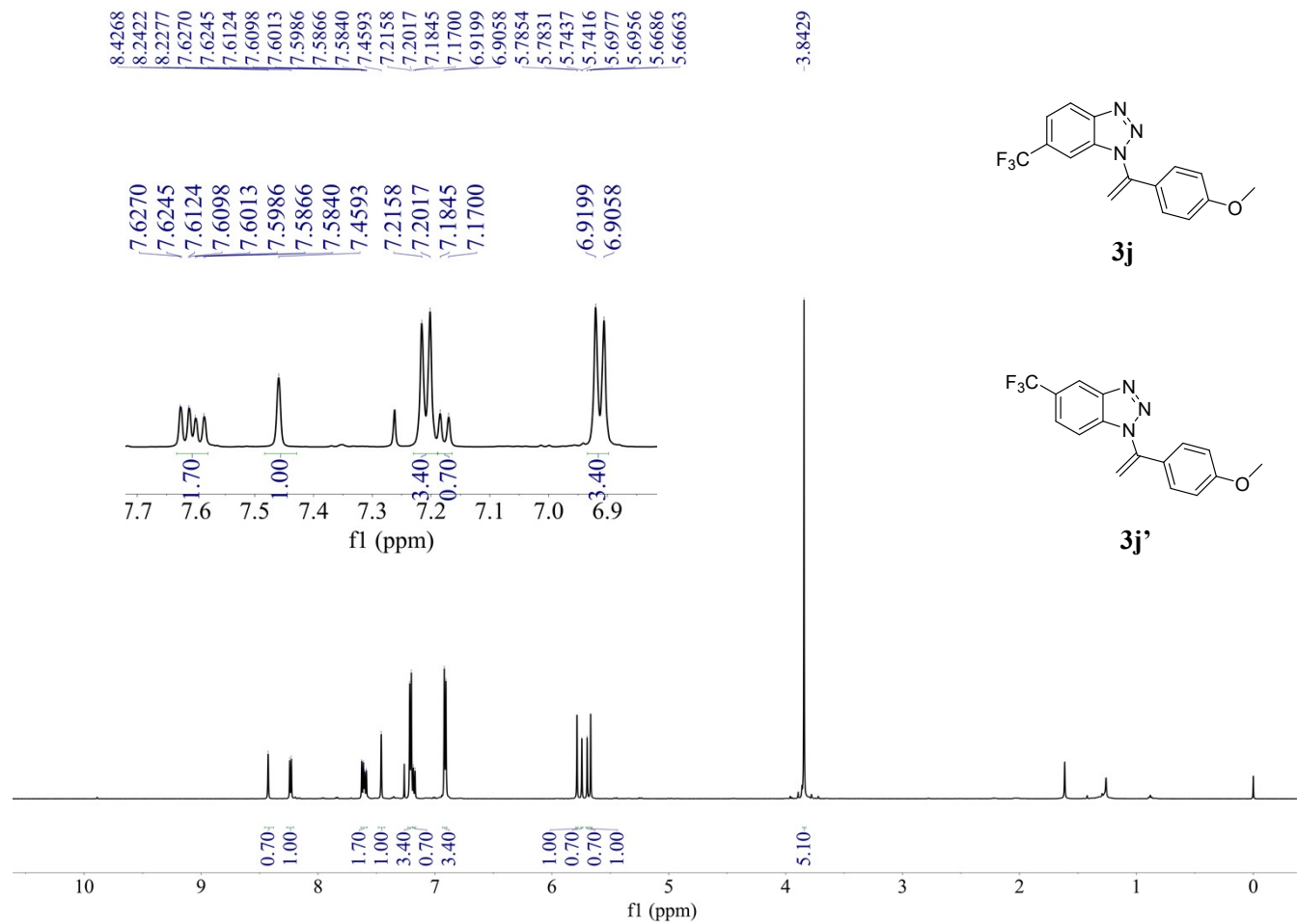
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3i and 3i'



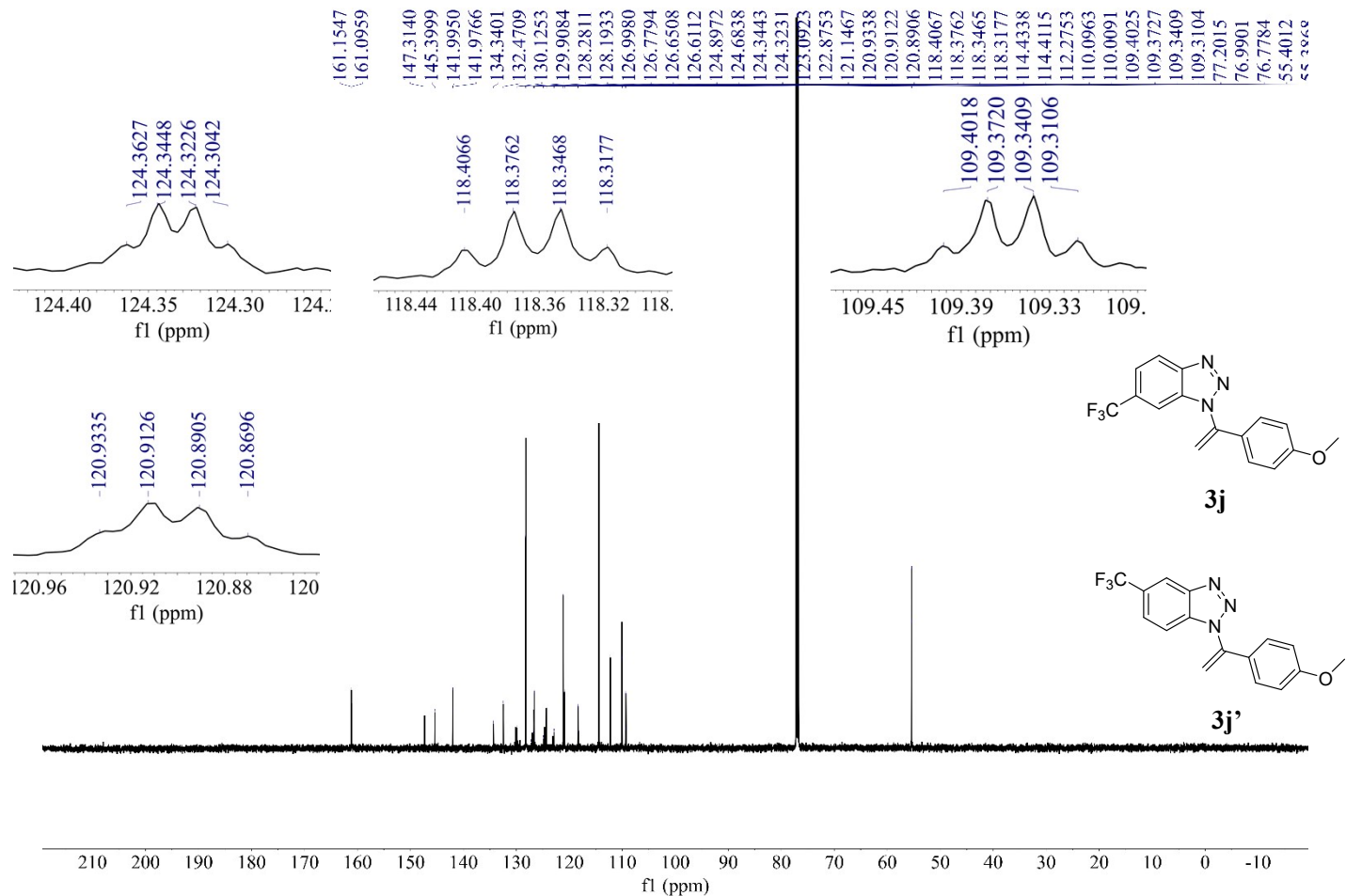
¹³C-NMR Spectrum (151 MHz, CDCl₃) of **3i** and **3i'**



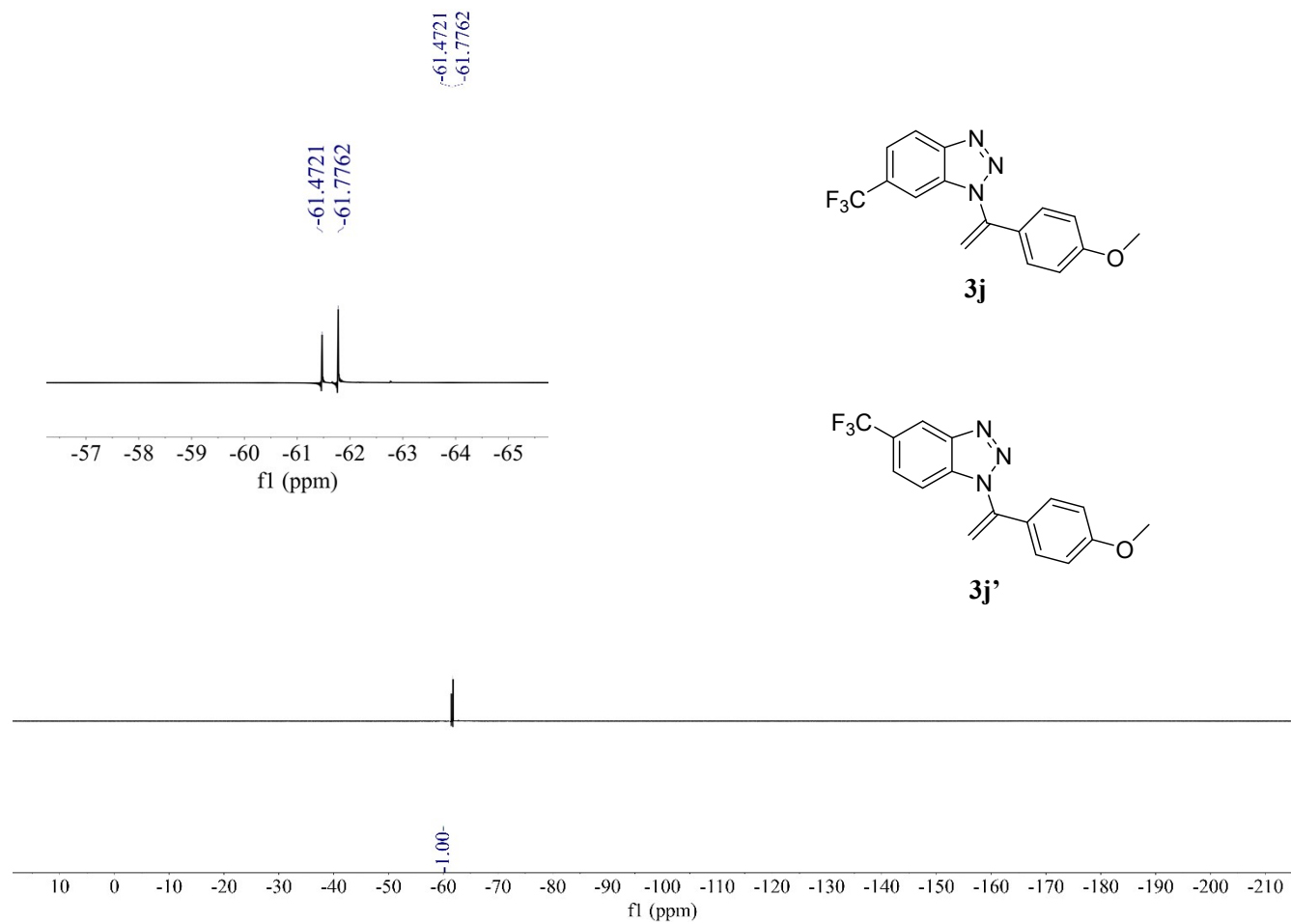
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3j and 3j'



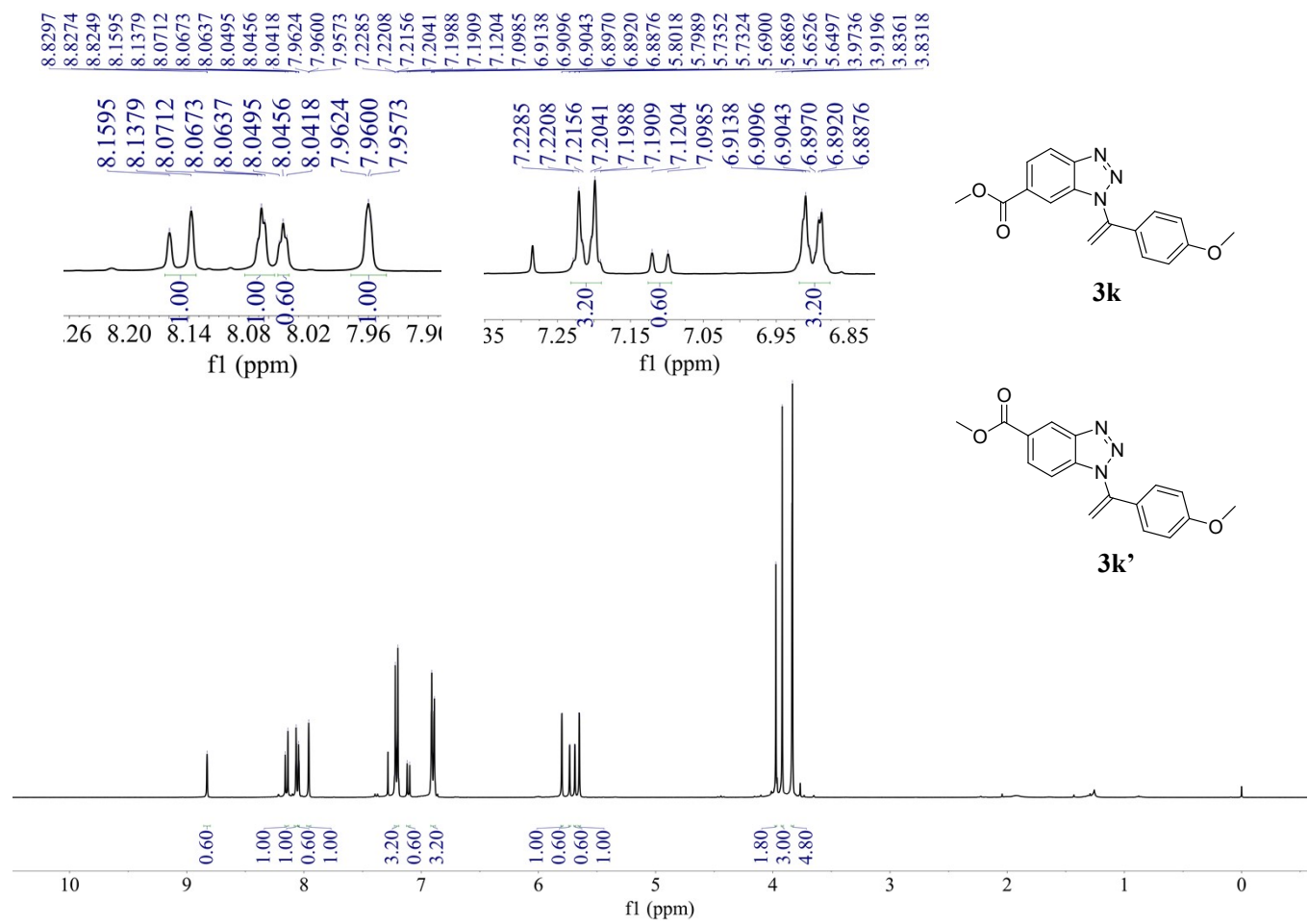
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3j and 3j'



¹⁹F-NMR Spectrum (565 MHz, CDCl₃) of 3j and 3j'

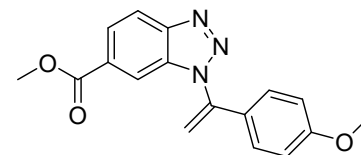


¹H-NMR Spectrum (400 MHz, CDCl₃) of 3k and 3k'

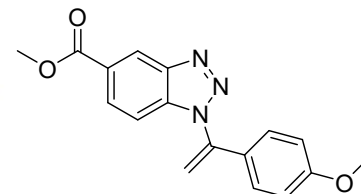


¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3k and 3k'

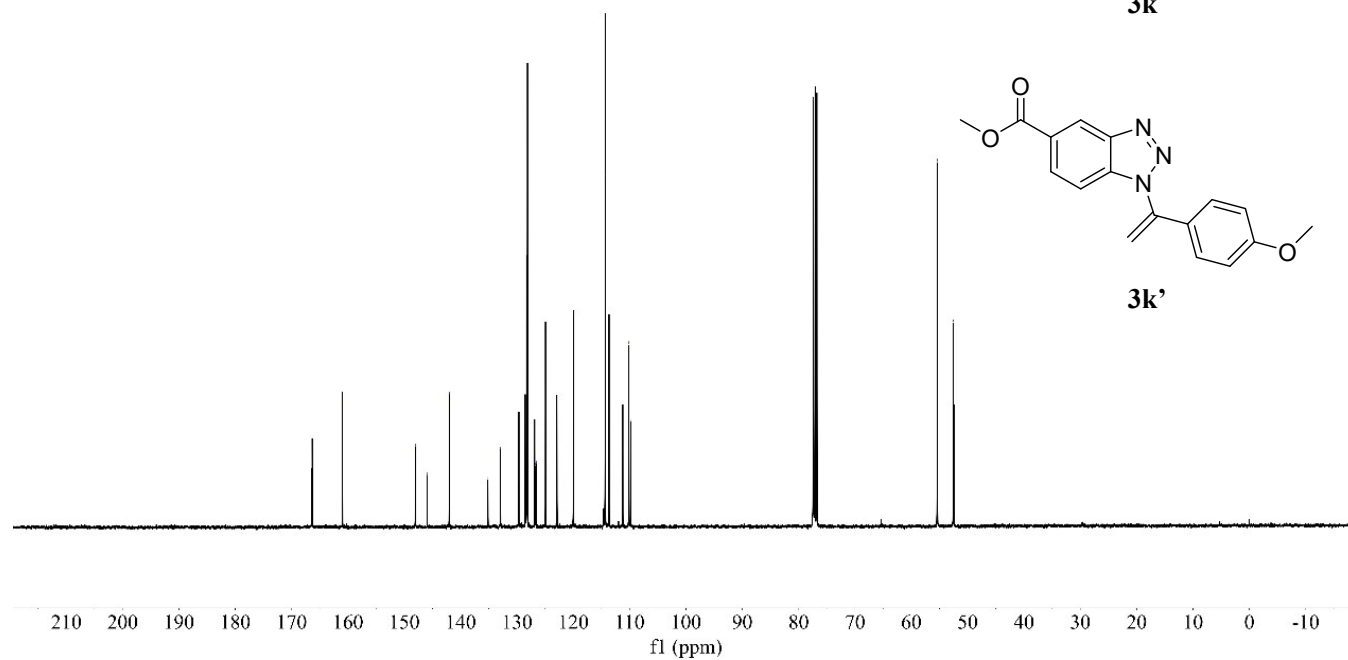
166.3927
166.3298
160.9756
148.0257
145.9104
141.9945
135.1457
132.9209
129.6622
128.5218
128.2776
128.1239
126.8474
126.7008
126.5614
124.9155
122.8998
119.9698
114.3292
114.3145
113.6570
111.2119
110.1327
109.8271
77.3977
77.0792
76.7613
55.3898
52.5353
52.4334



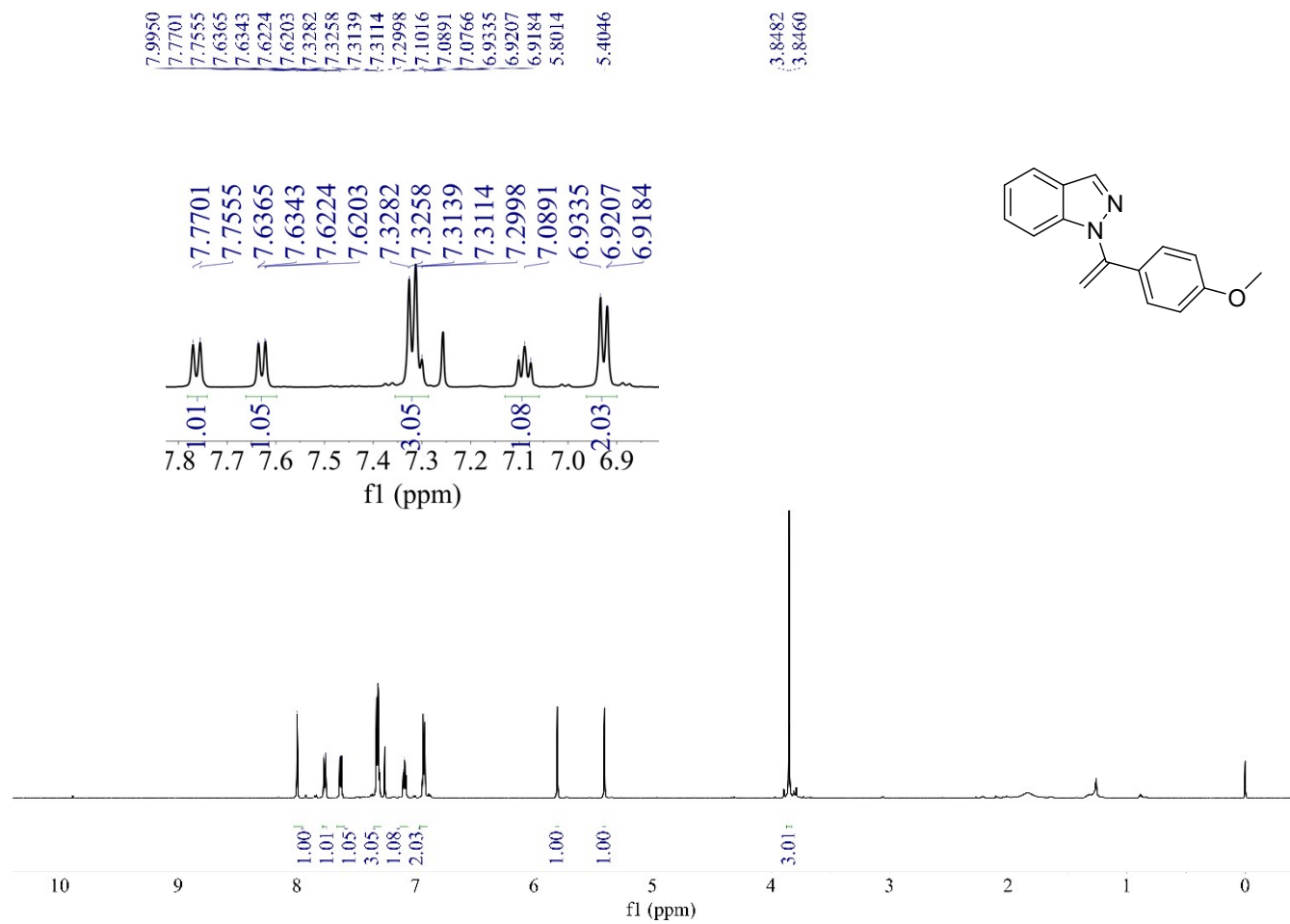
3k



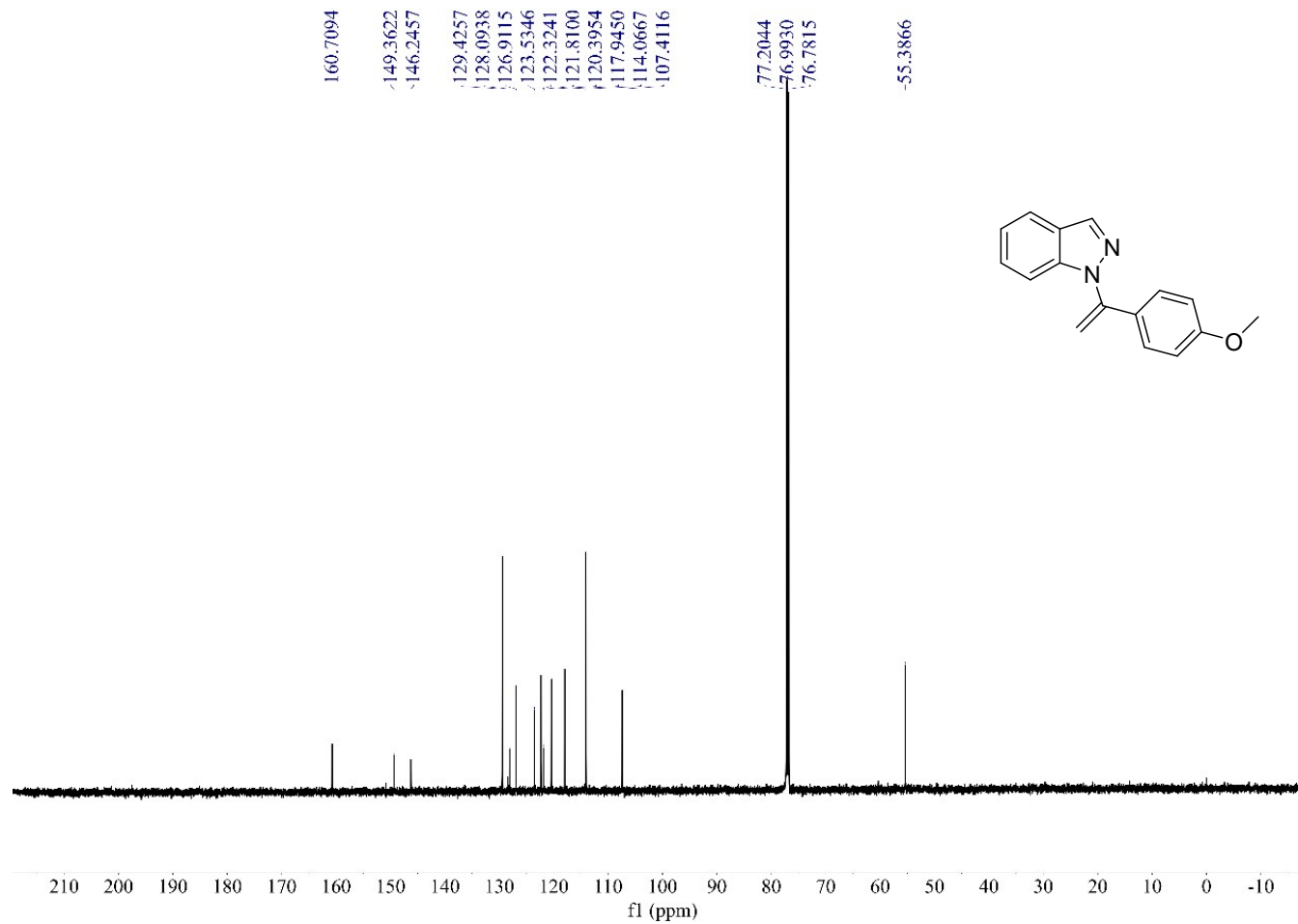
3k'



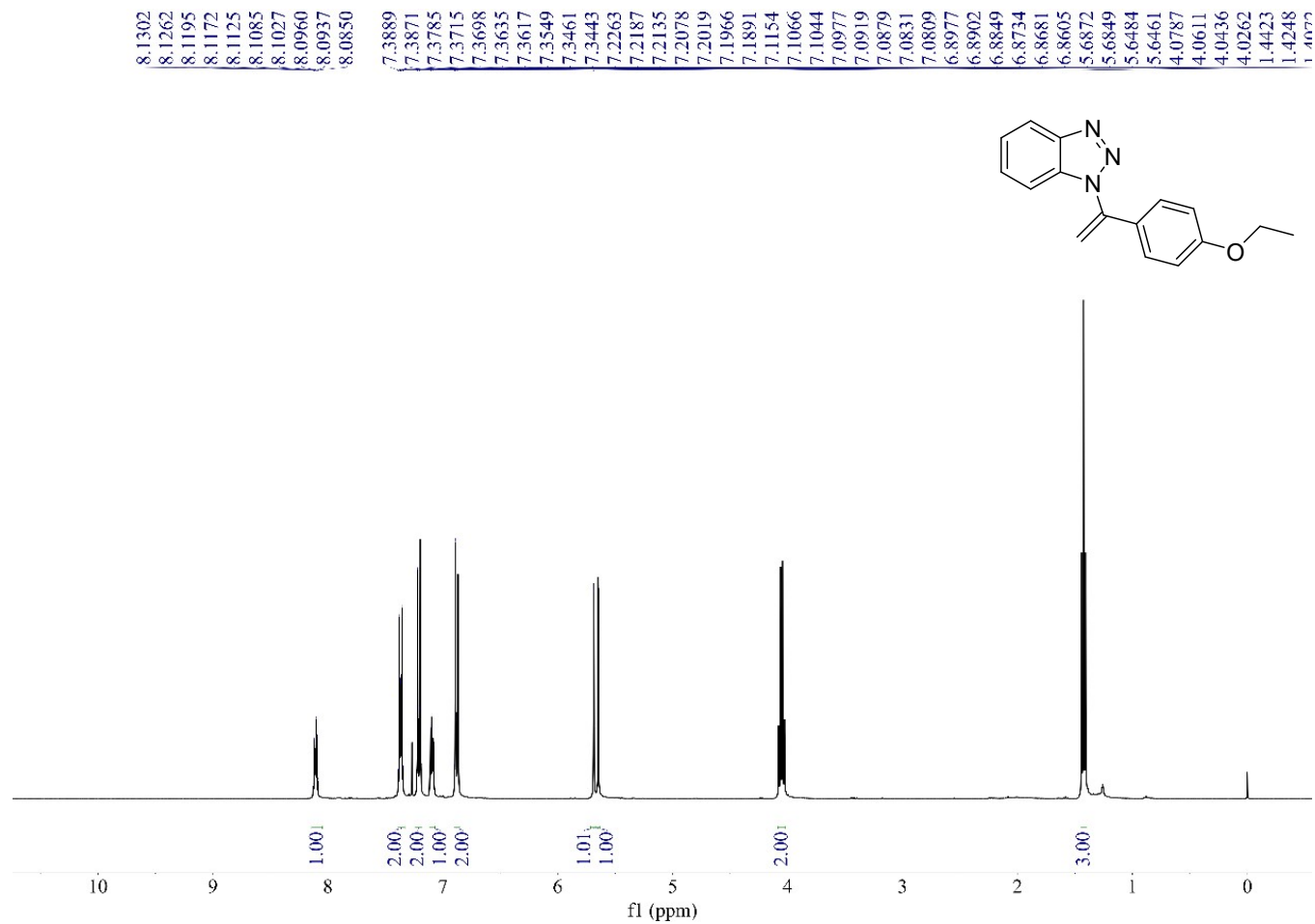
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3l



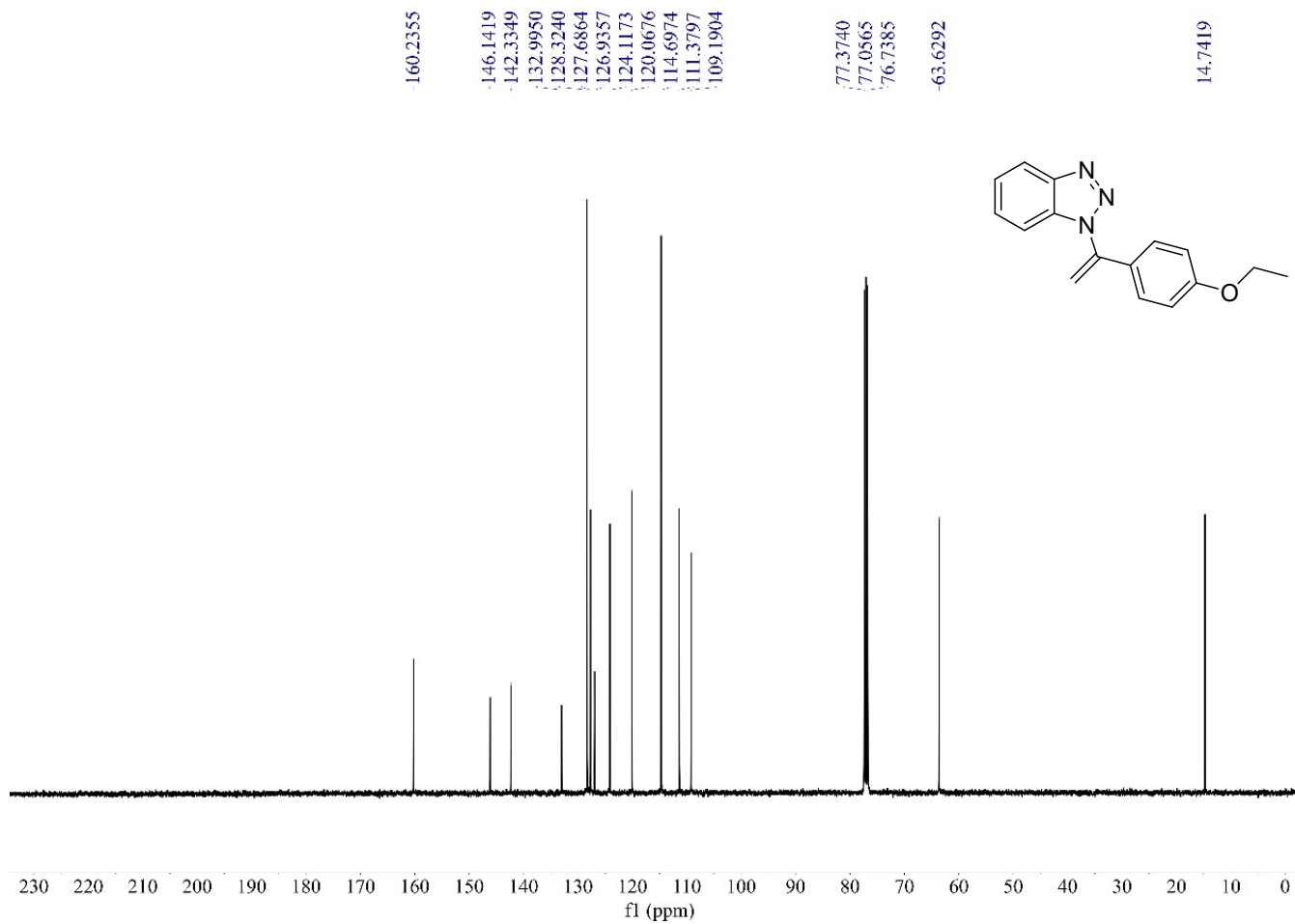
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3l



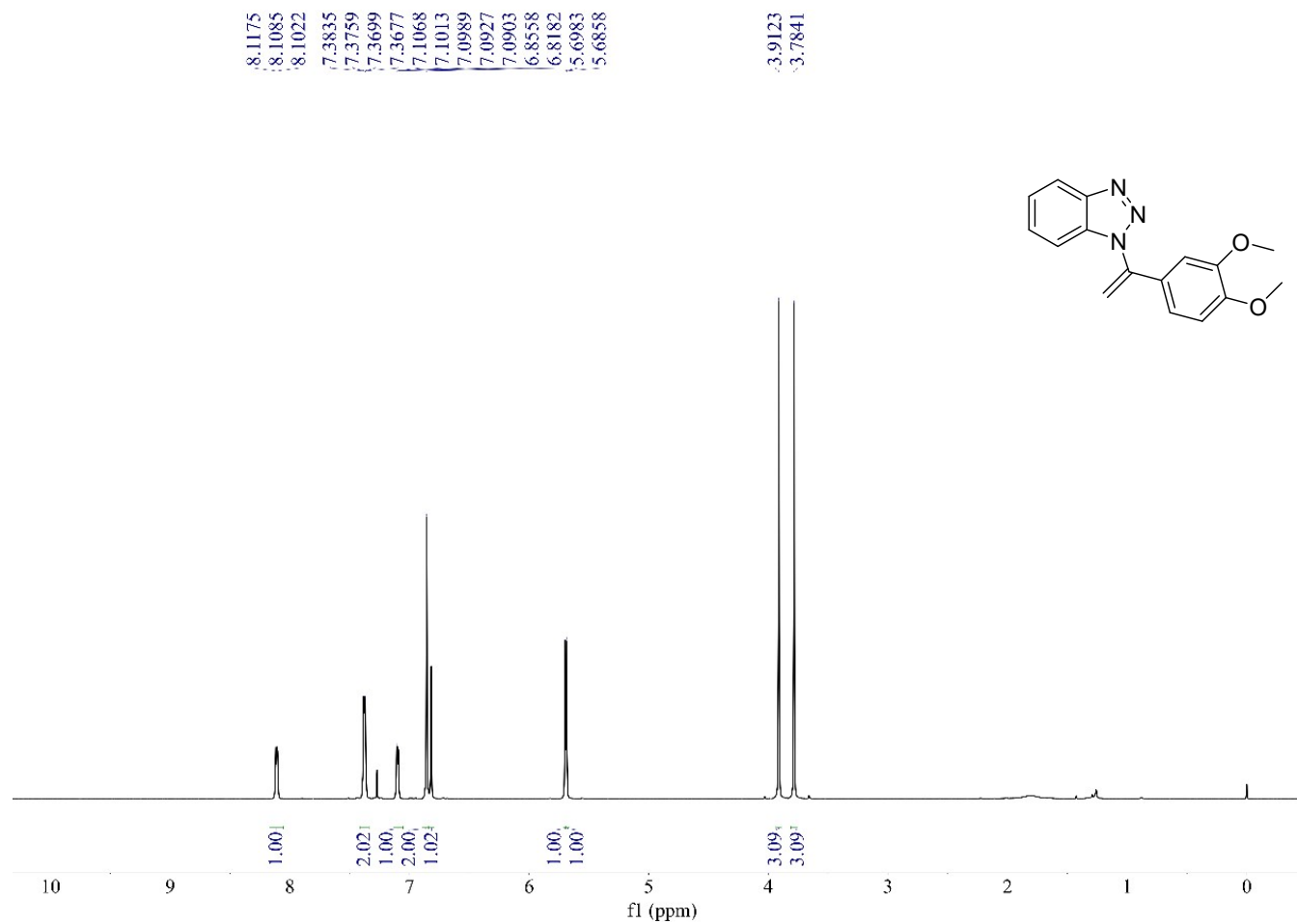
¹H-NMR Spectrum (400 MHz, CDCl₃) of 3m



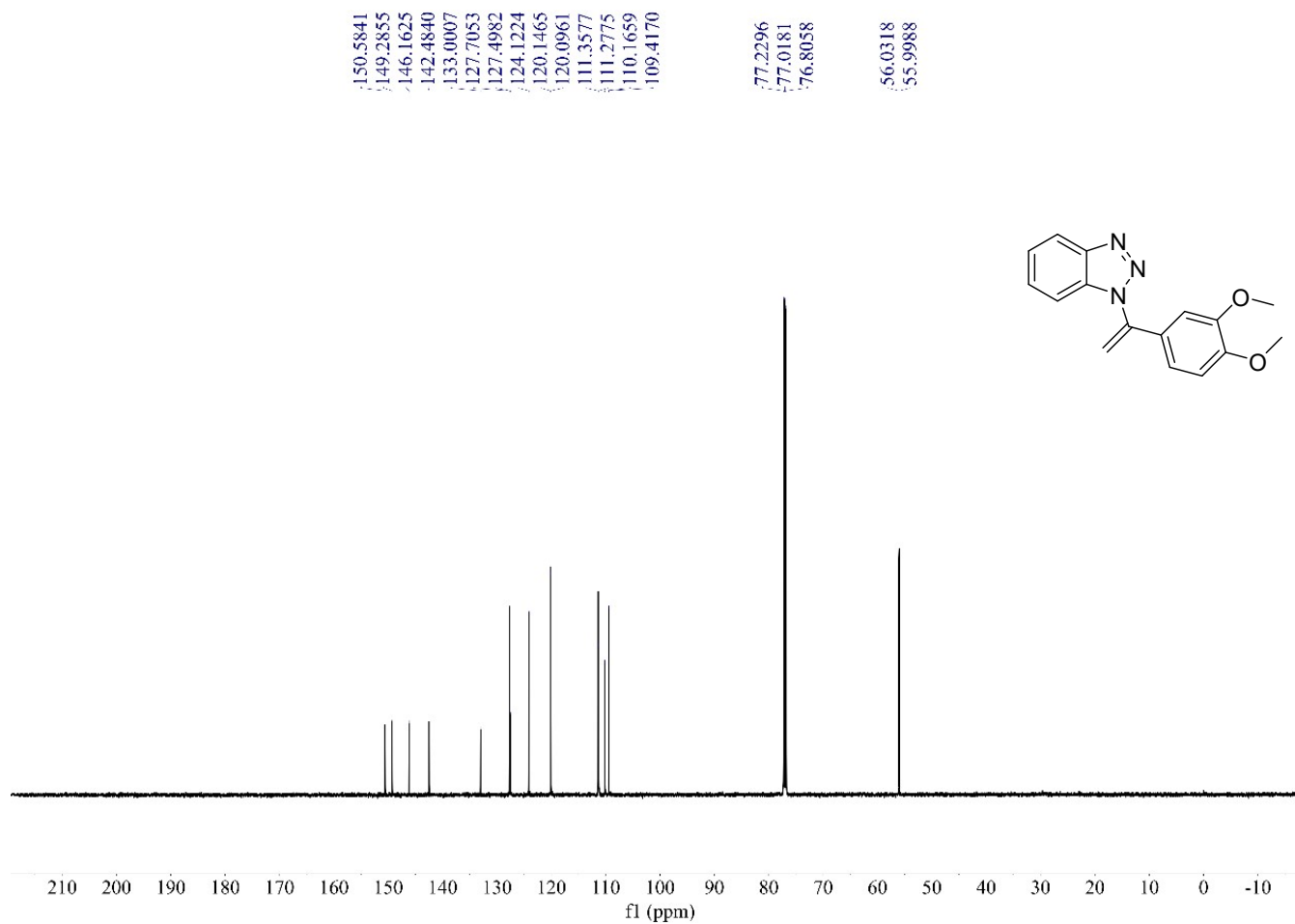
¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3m



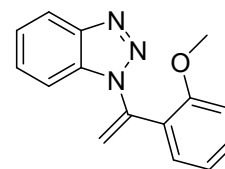
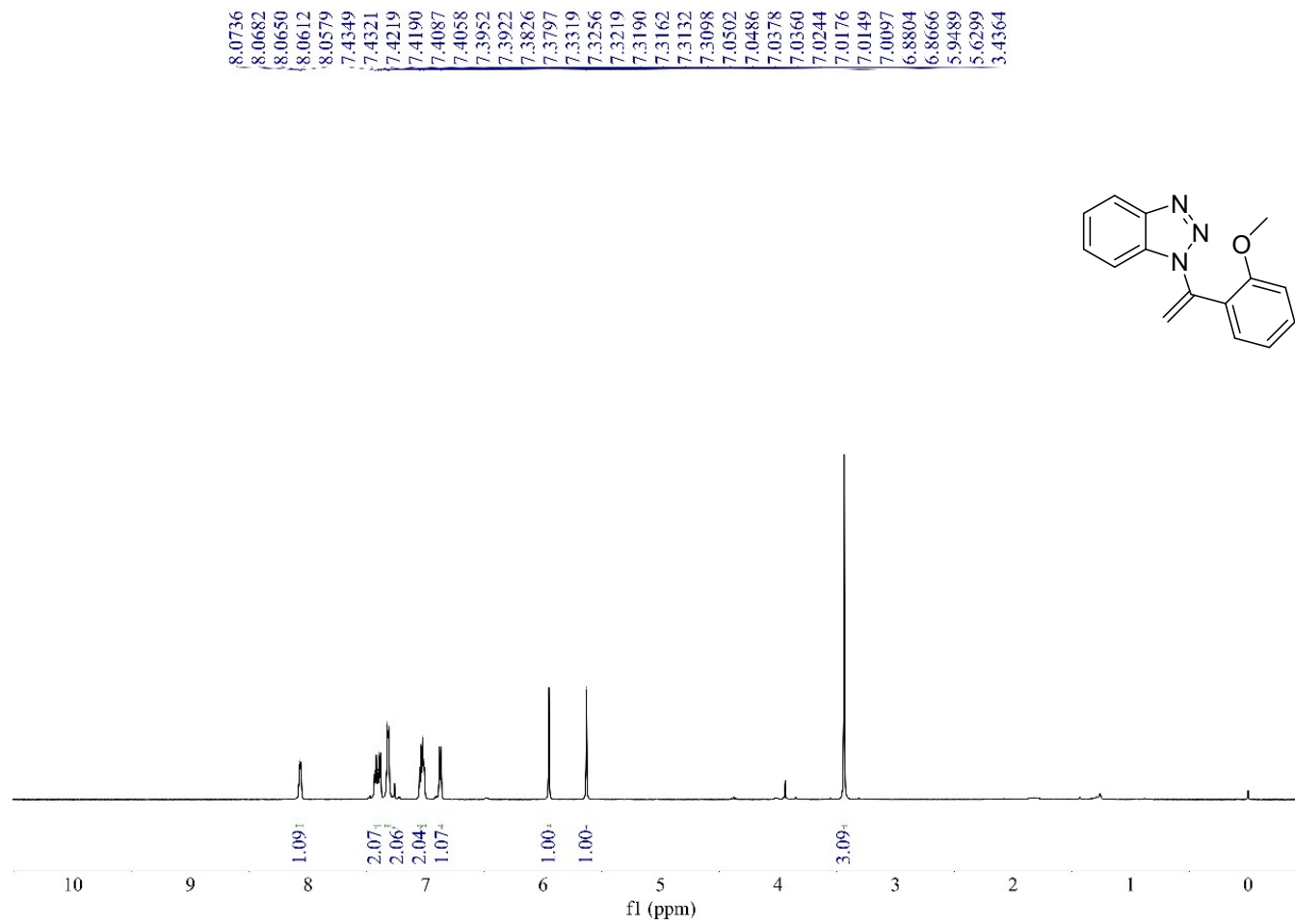
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3n



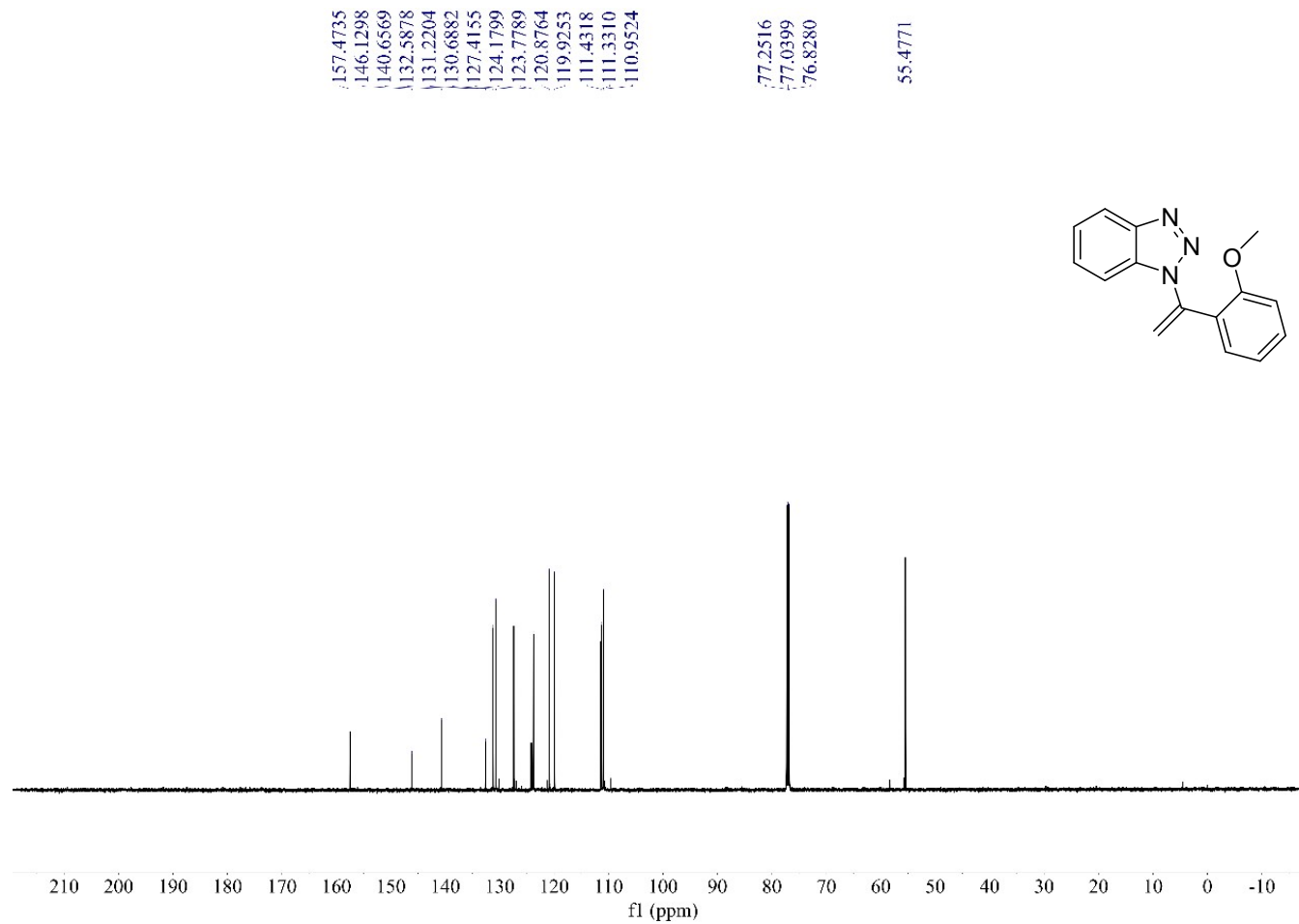
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3n



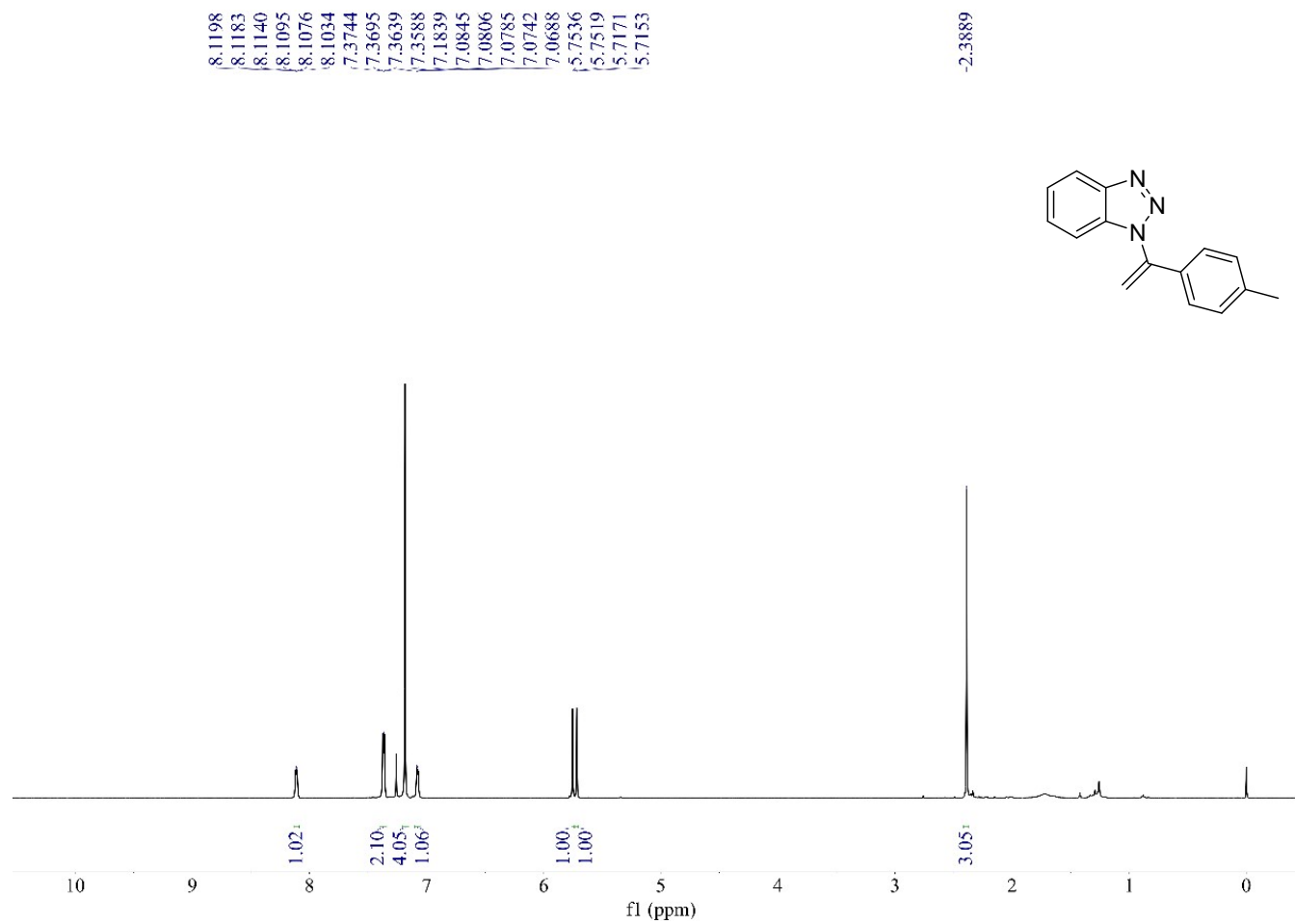
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3o



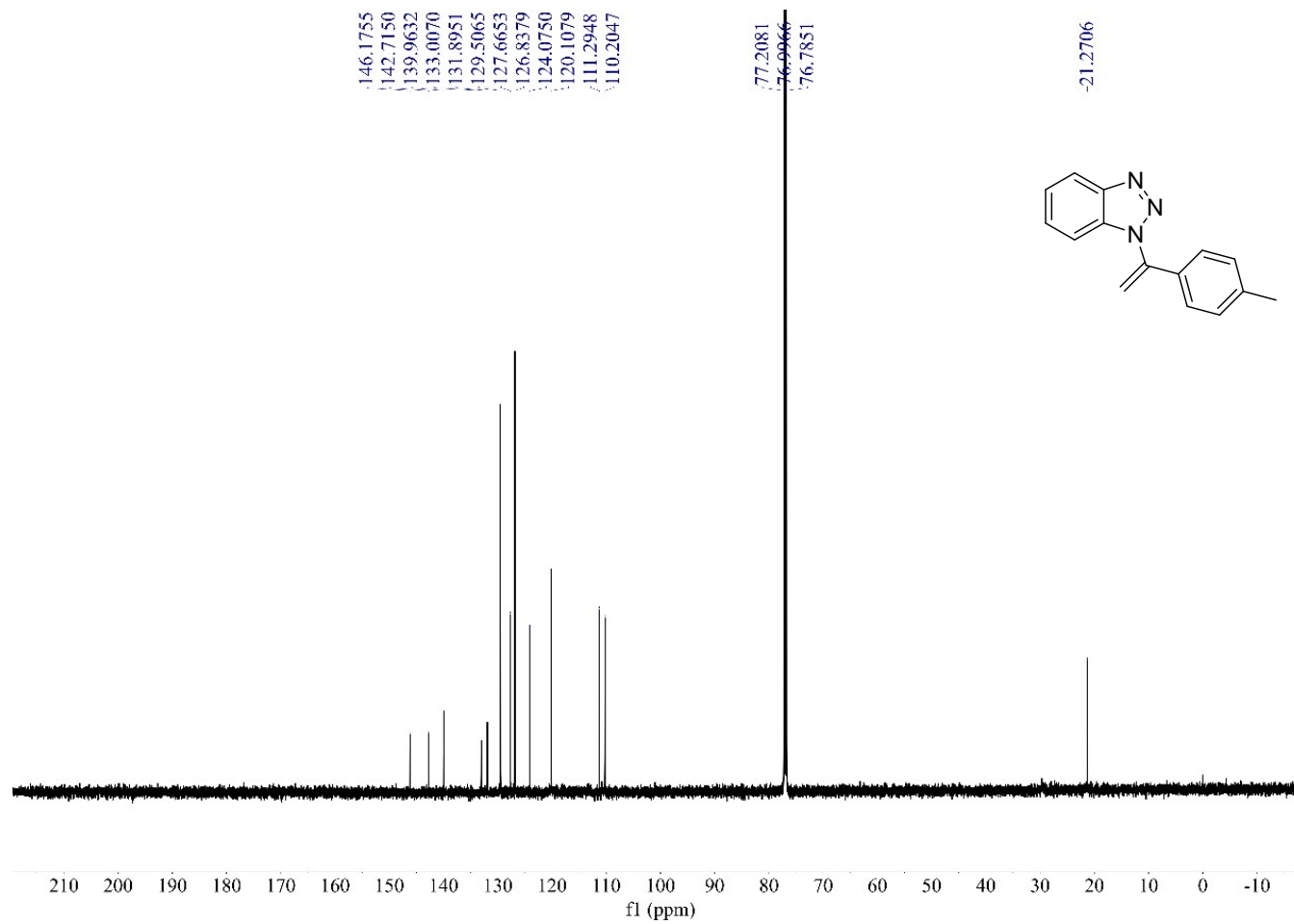
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3o



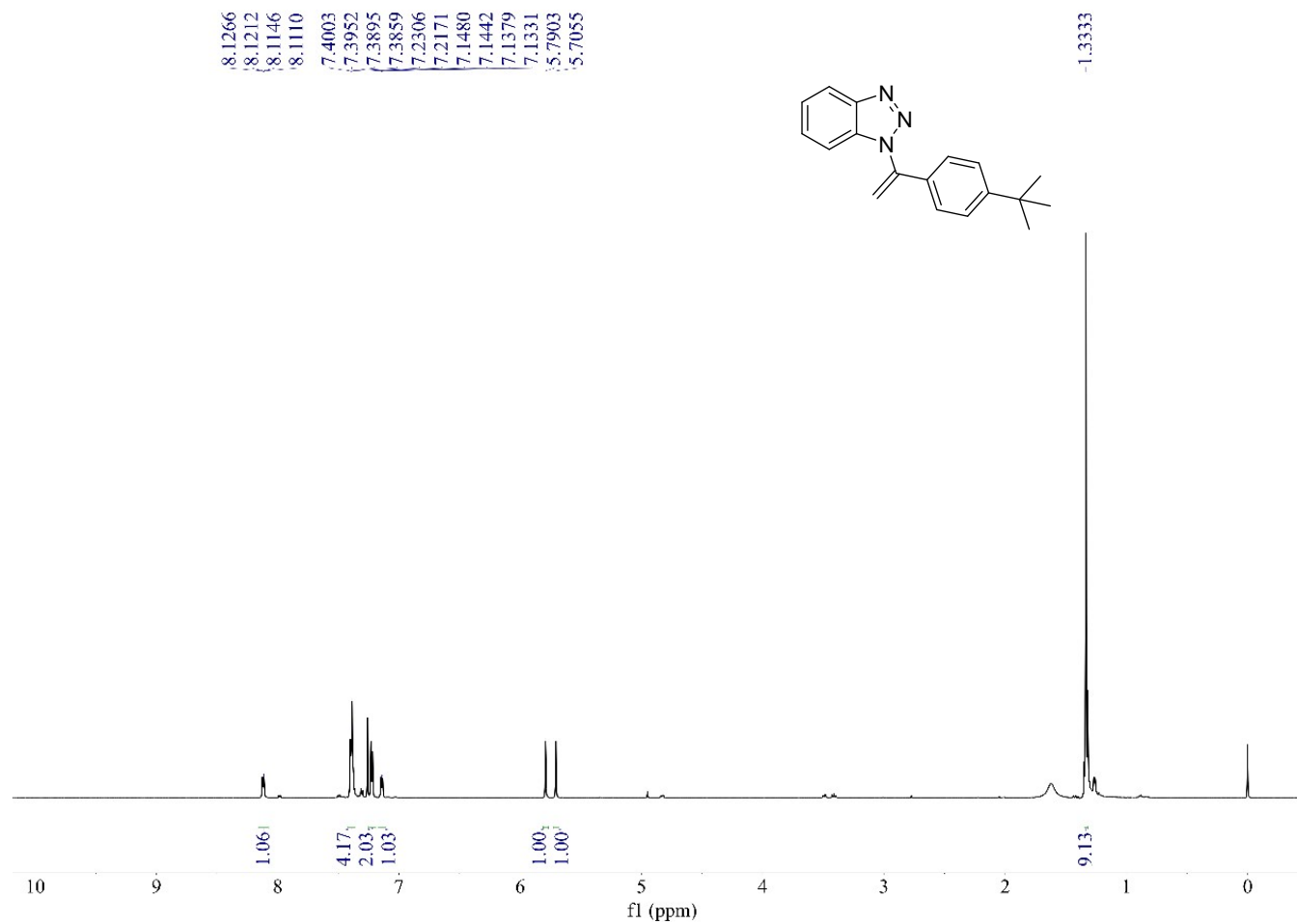
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3p



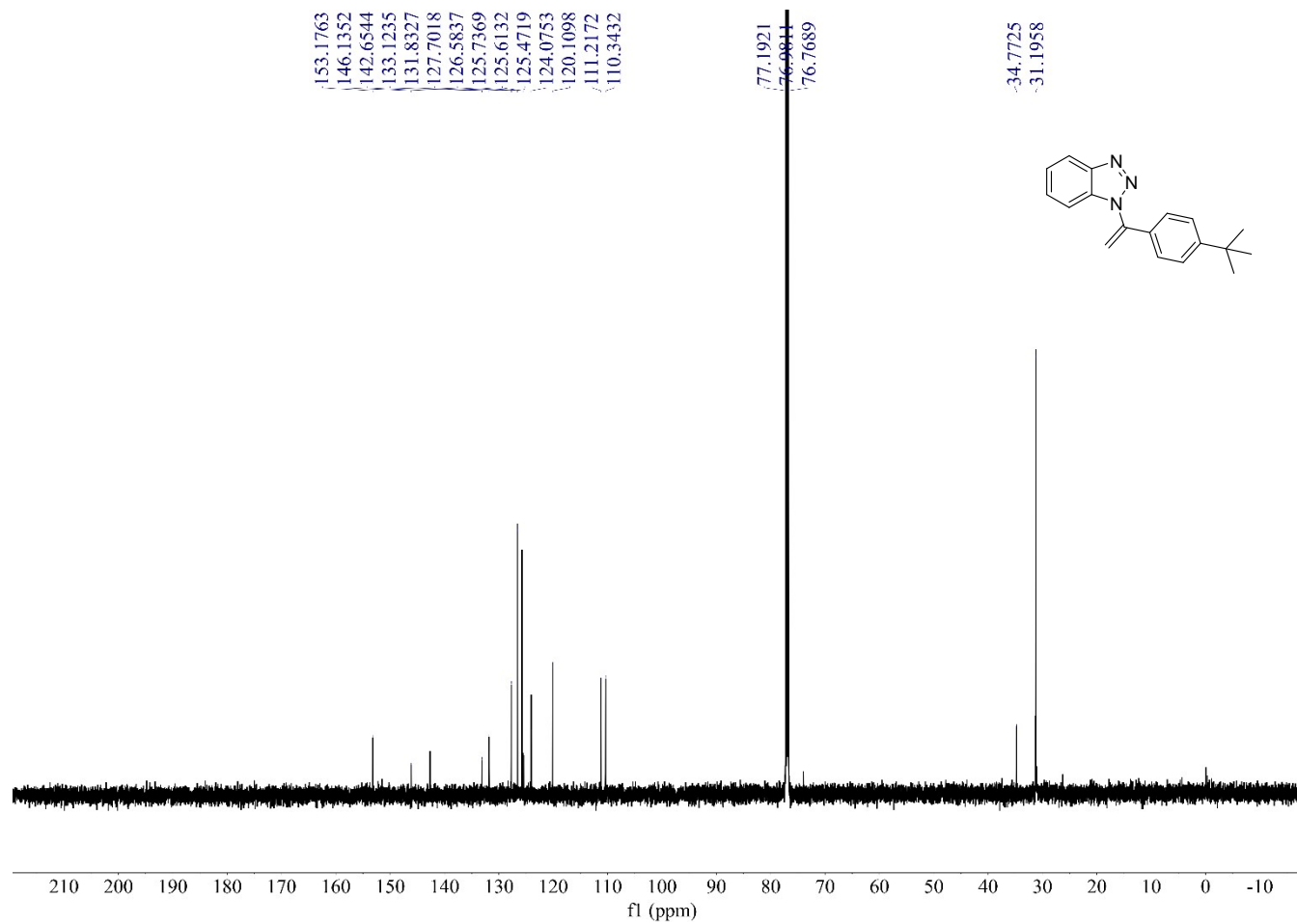
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3p



¹H-NMR Spectrum (600 MHz, CDCl₃) of 3q

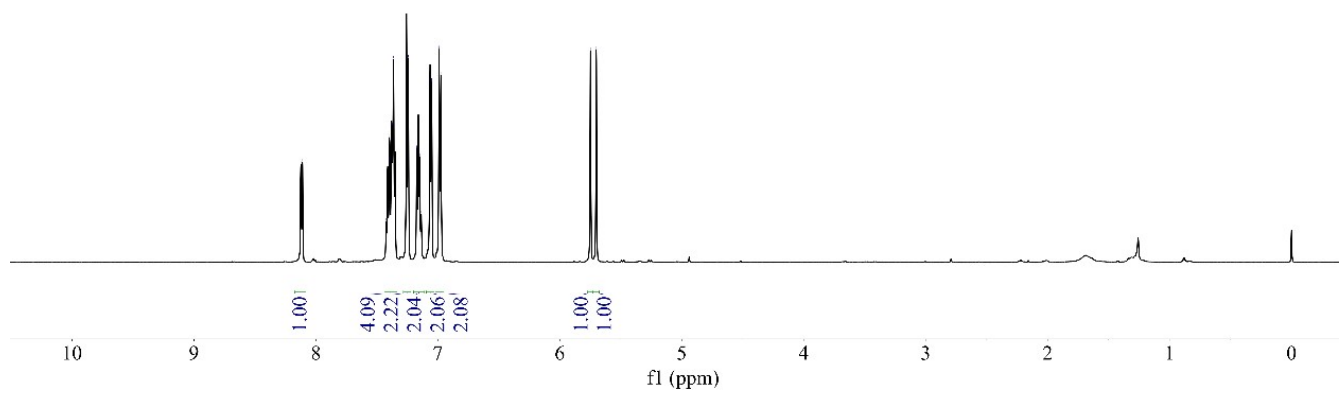
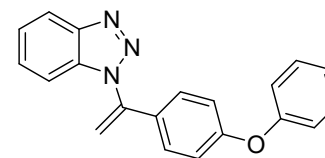


¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3q

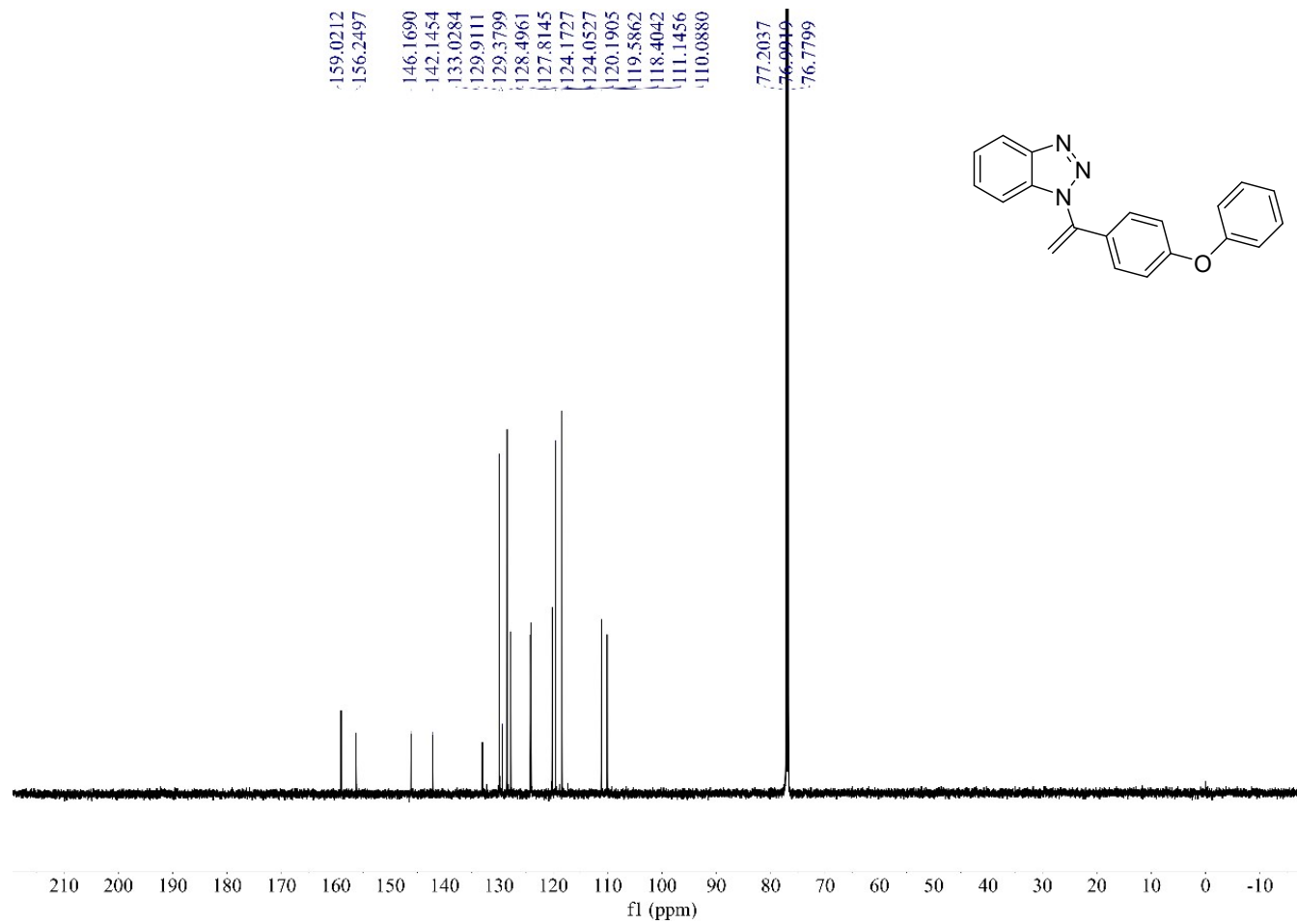


¹H-NMR Spectrum (600 MHz, CDCl₃) of 3r

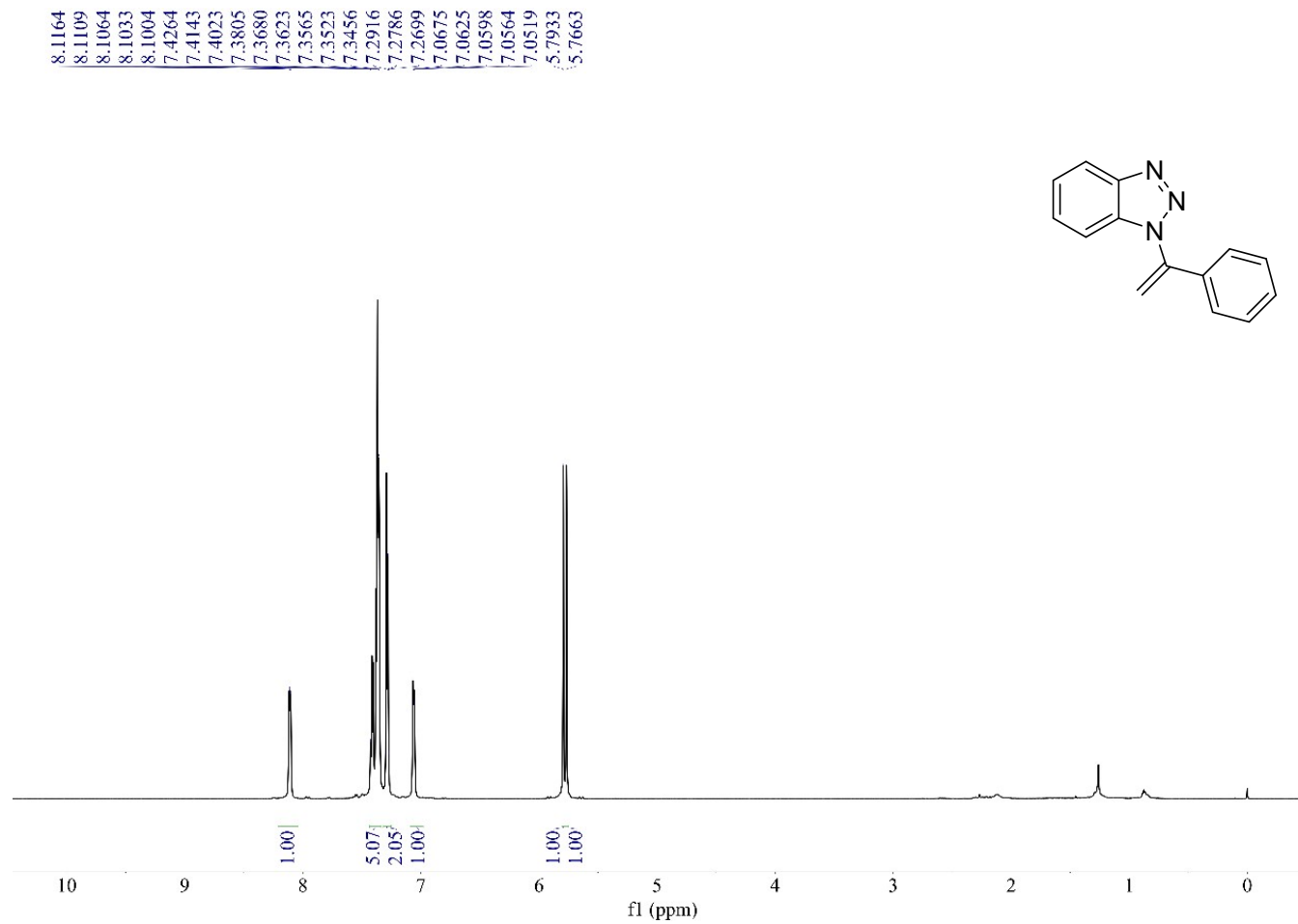
8.1233
8.1099
7.4236
7.4116
7.3999
7.3969
7.3934
7.3808
7.3761
7.3636
7.3502
7.2567
7.2427
7.1724
7.1596
7.1484
7.1360
7.0642
7.0508
6.9889
6.9749
5.7484
5.7007



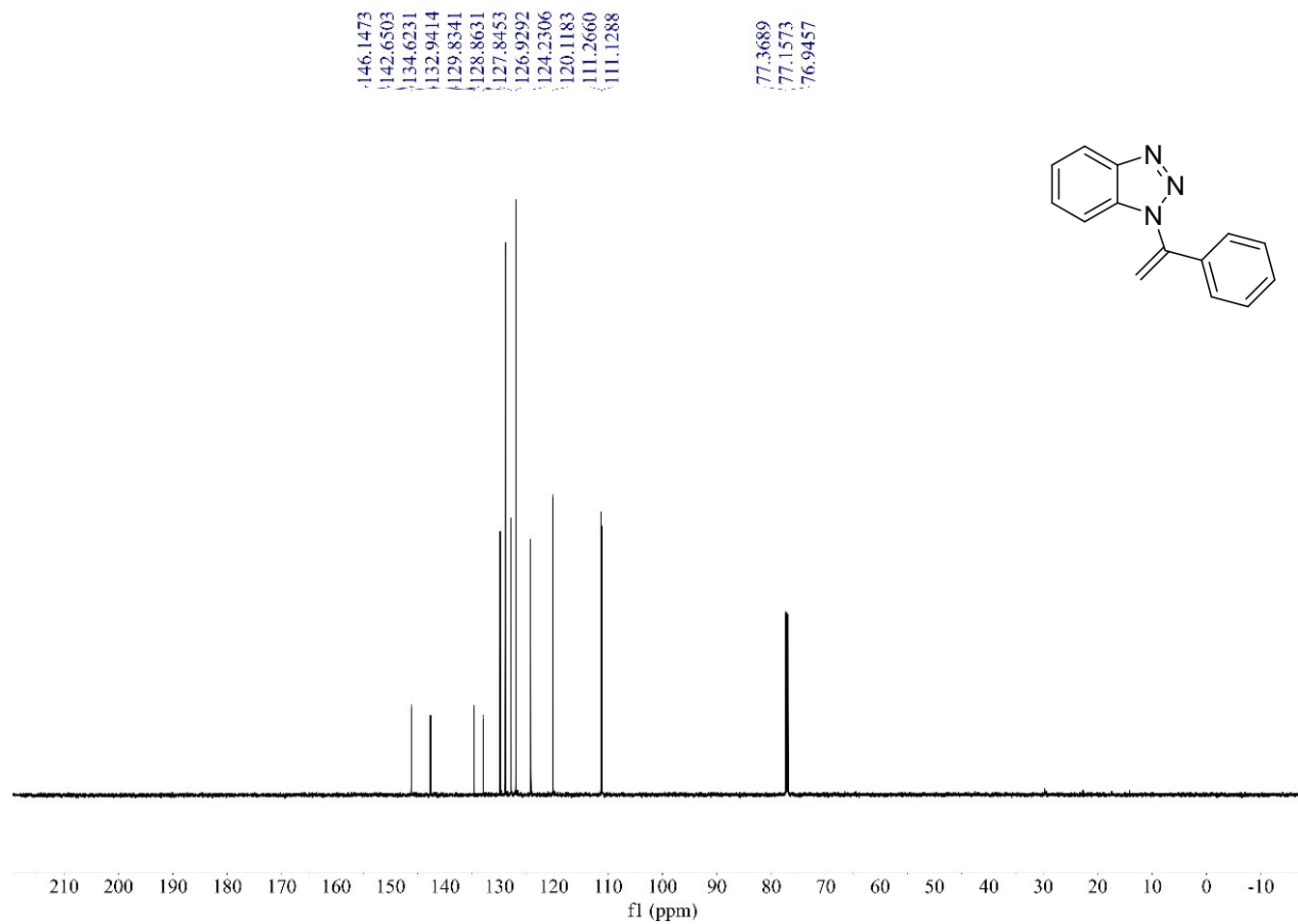
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3r



¹H-NMR Spectrum (600 MHz, CDCl₃) of 3s

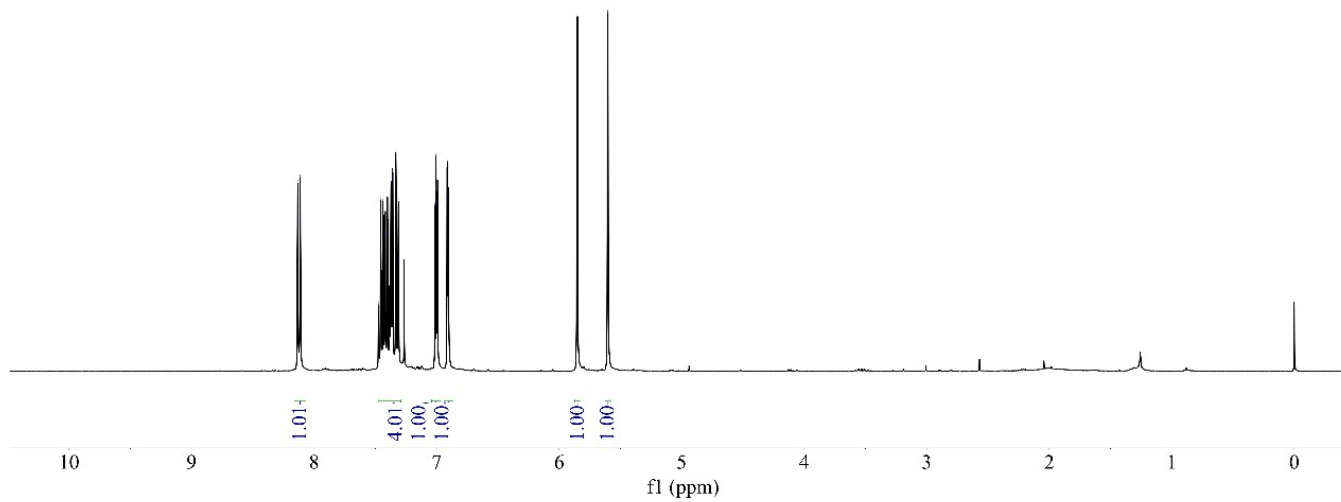
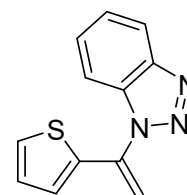


¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3s

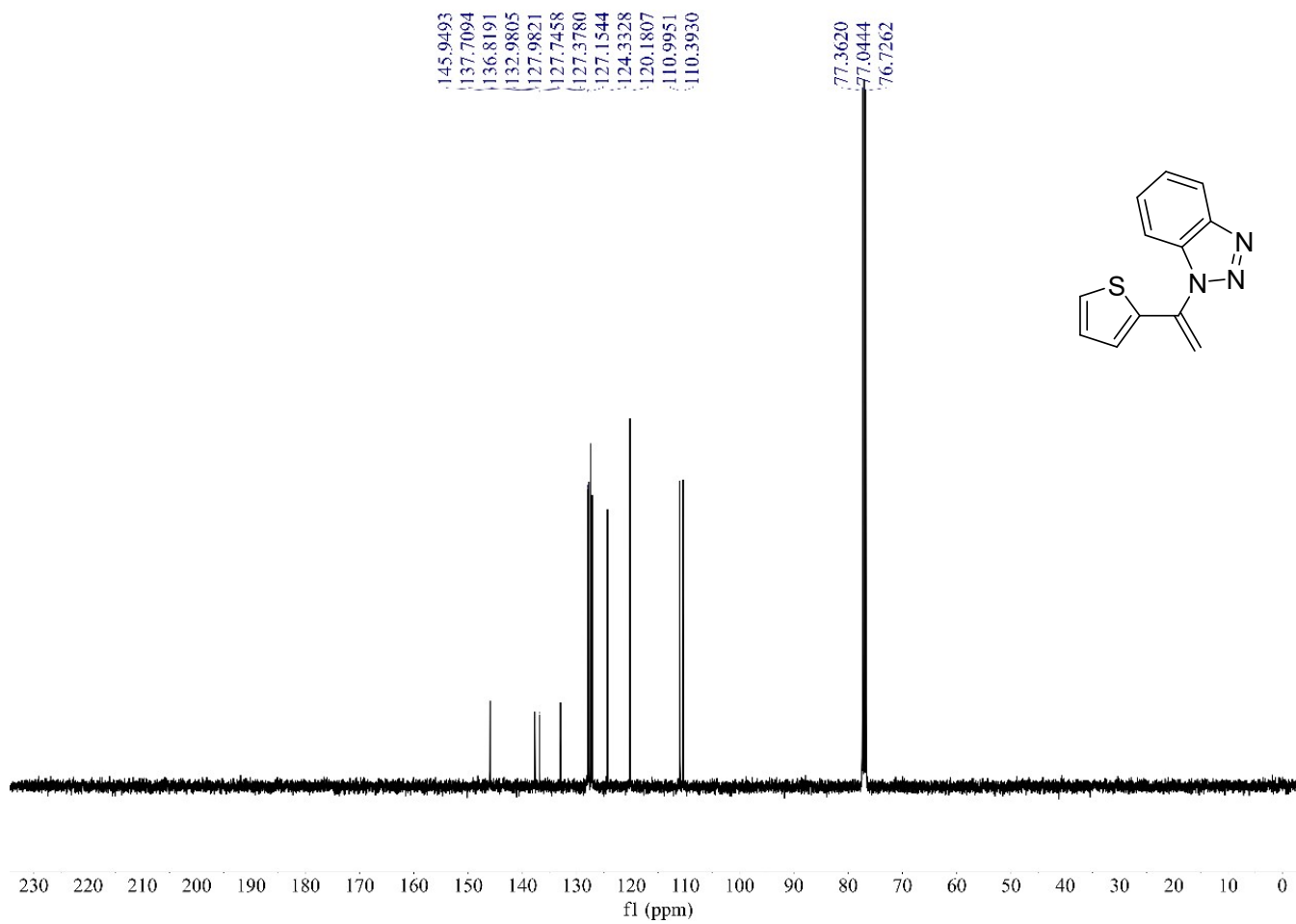


¹H-NMR Spectrum (400 MHz, CDCl₃) of 3t

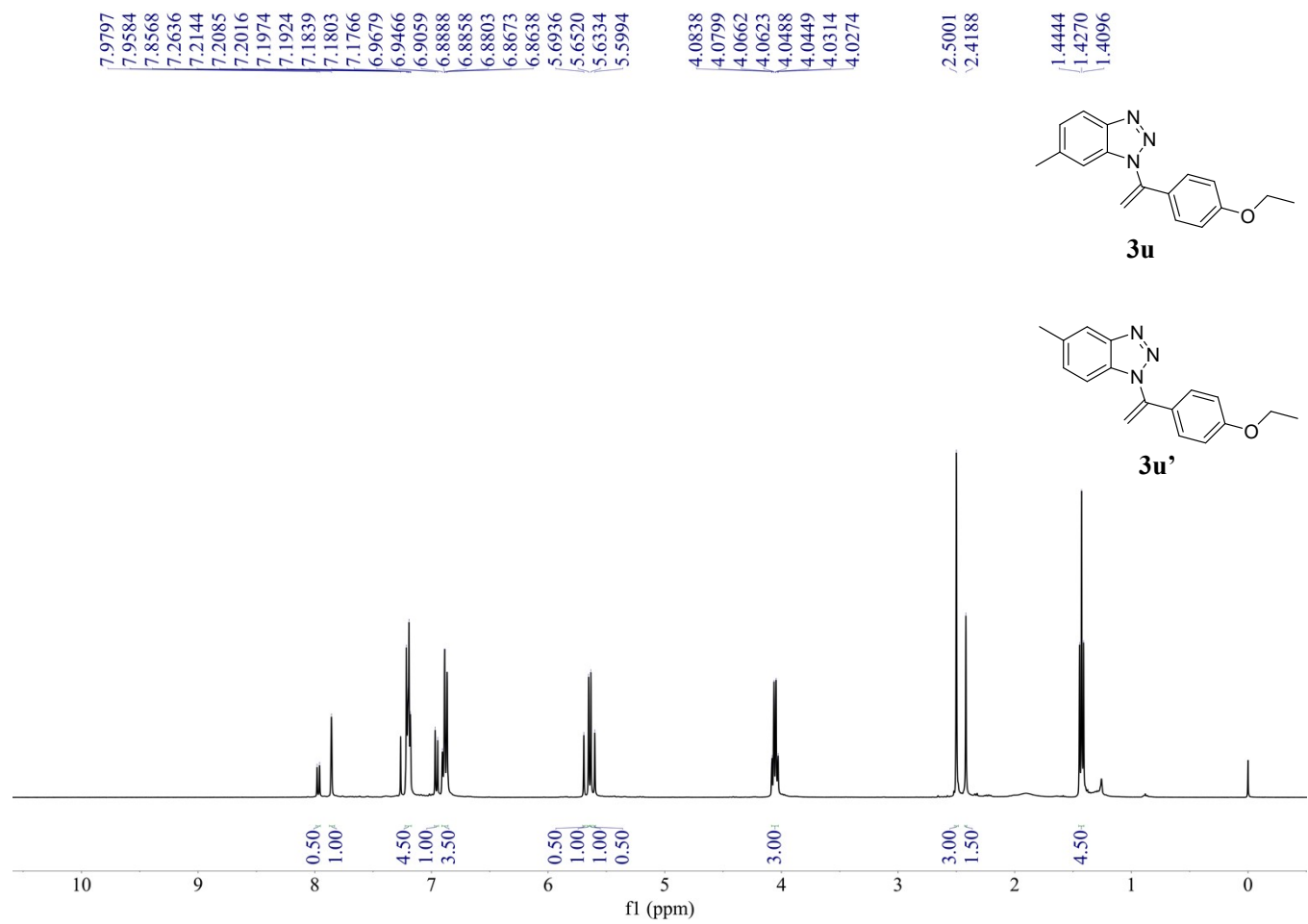
8.1363
8.1337
8.1307
8.1155
8.1132
8.1105
7.4751
7.4722
7.4577
7.4548
7.4516
7.4376
7.4343
7.4223
7.4190
7.4051
7.4018
7.3988
7.3844
7.3813
7.3731
7.3700
7.3605
7.3574
7.3351
7.3324
7.3300
7.3150
7.3120
7.3094
7.0138
7.0046
7.0011
6.9919
6.9144
6.9113
6.9052
6.9021
5.8543
5.8508
5.6048
5.6014



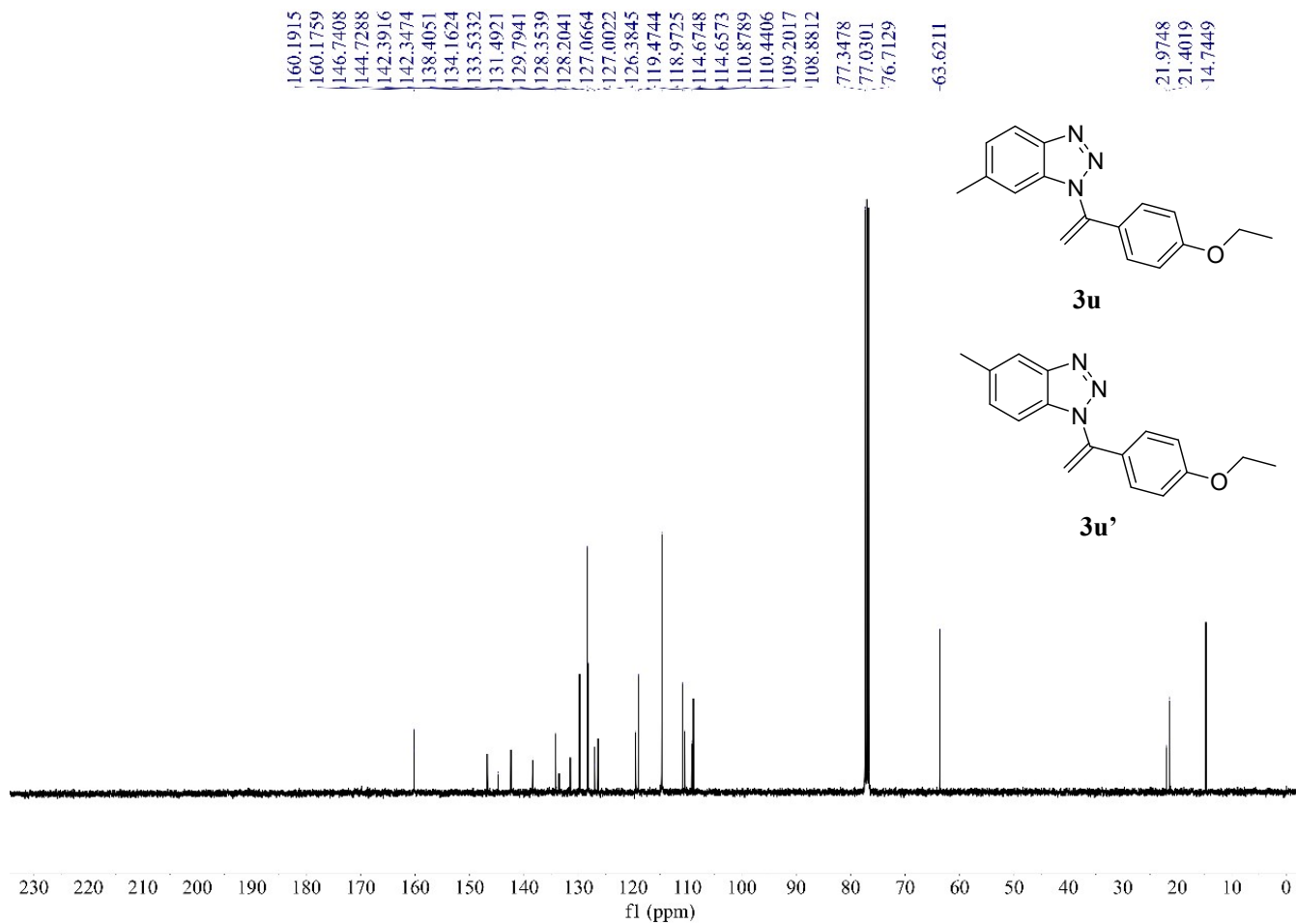
¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3t



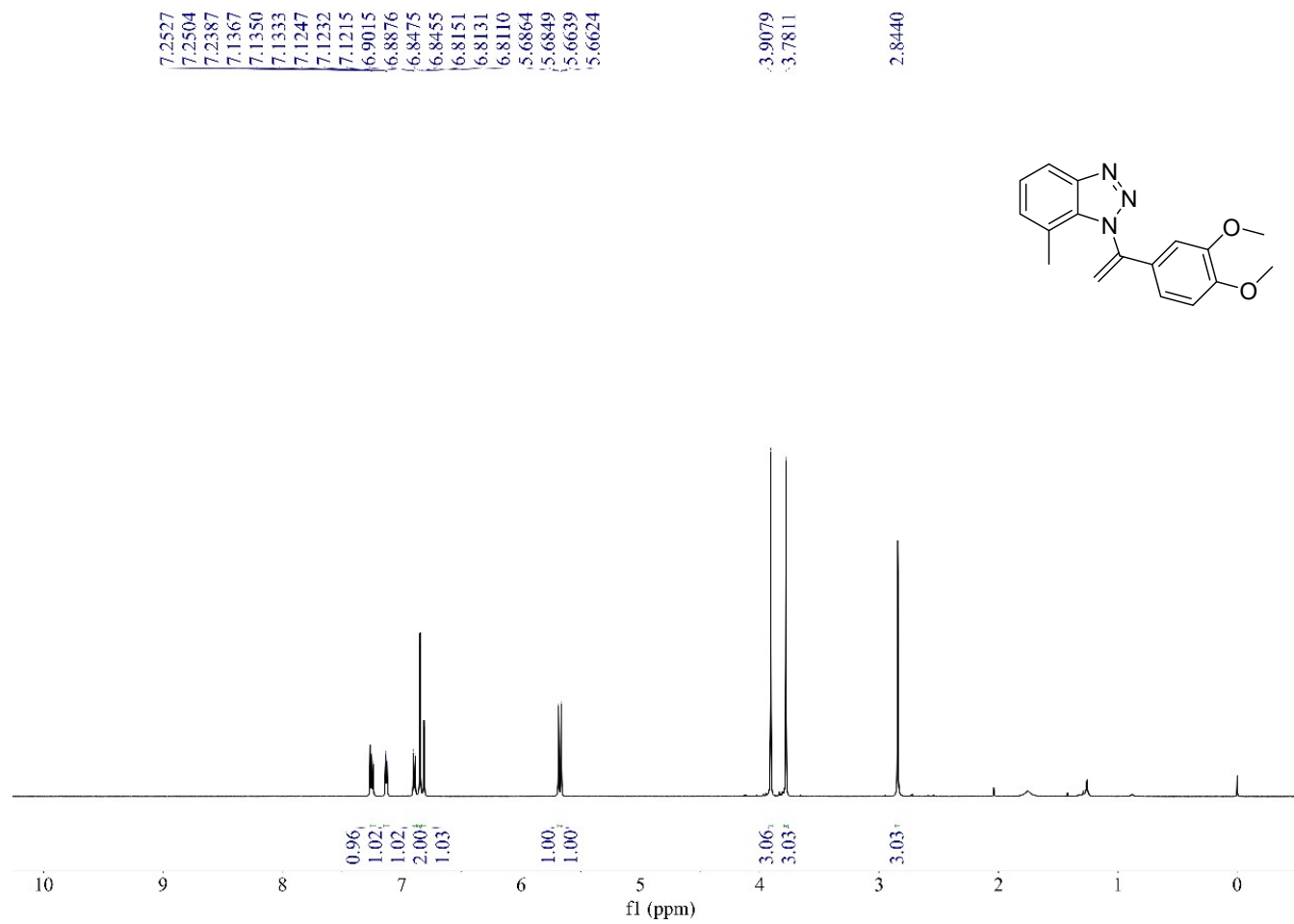
¹H-NMR Spectrum (400 MHz, CDCl₃) of 3u and 3u'



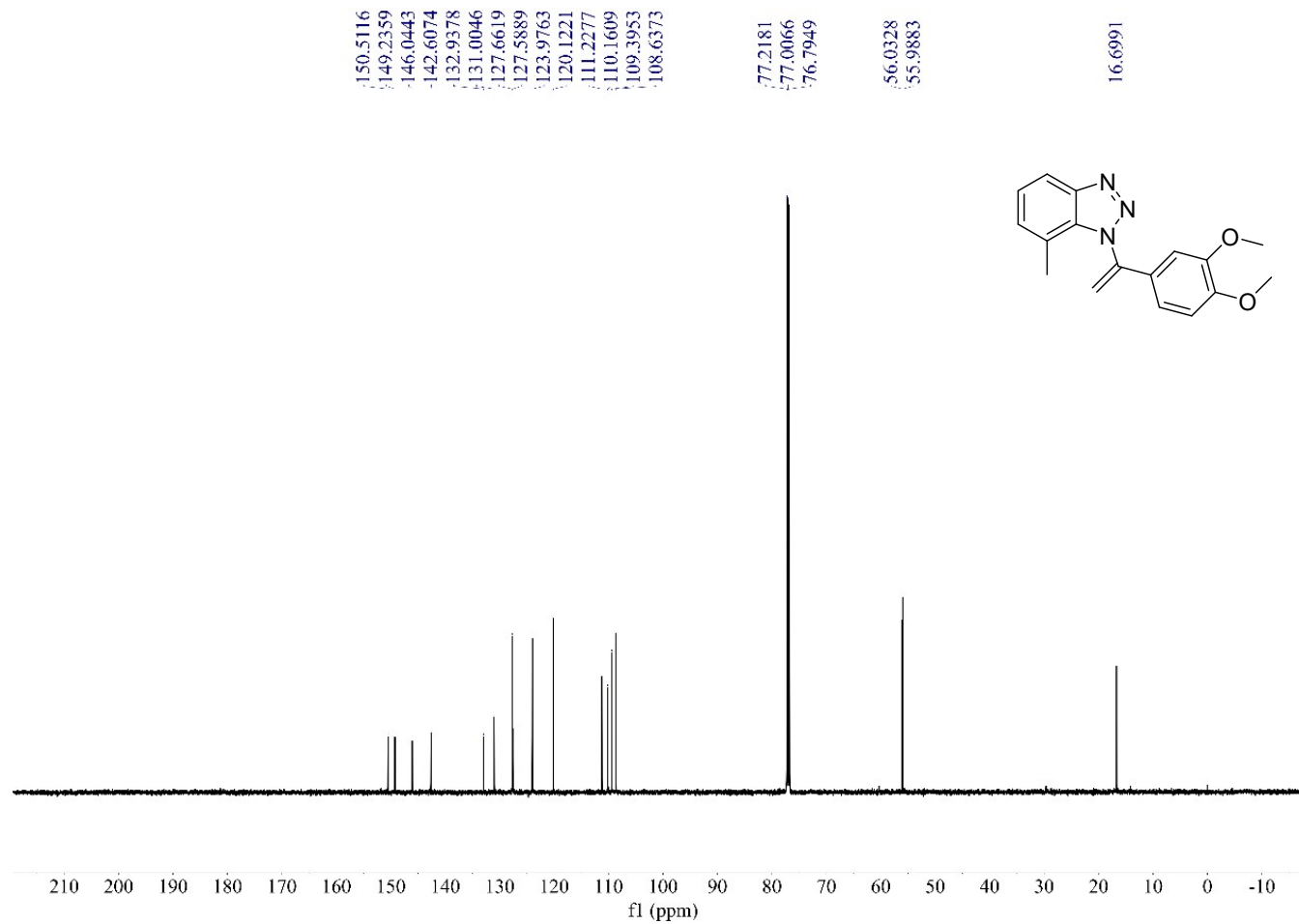
¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3u and 3u'



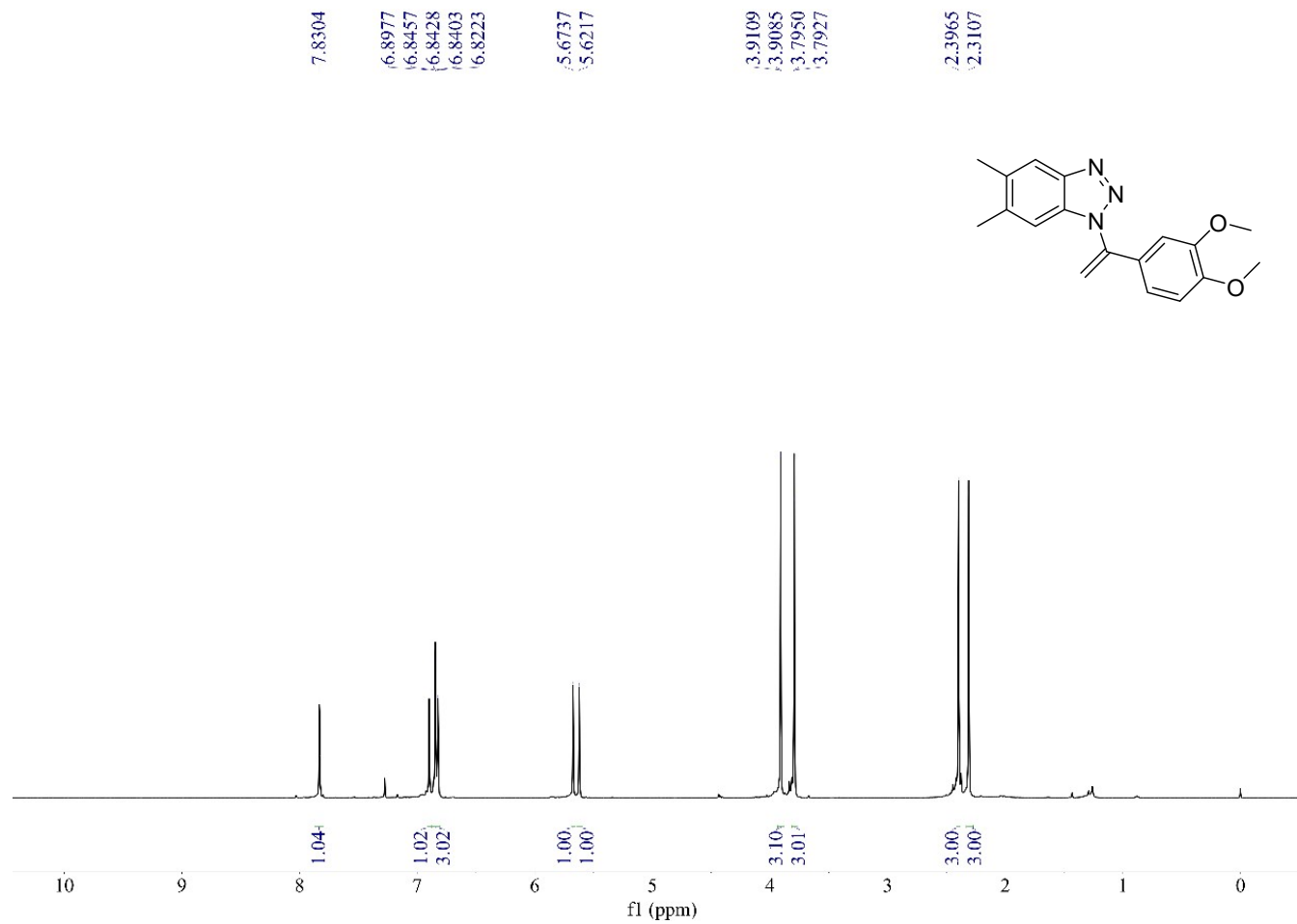
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3v



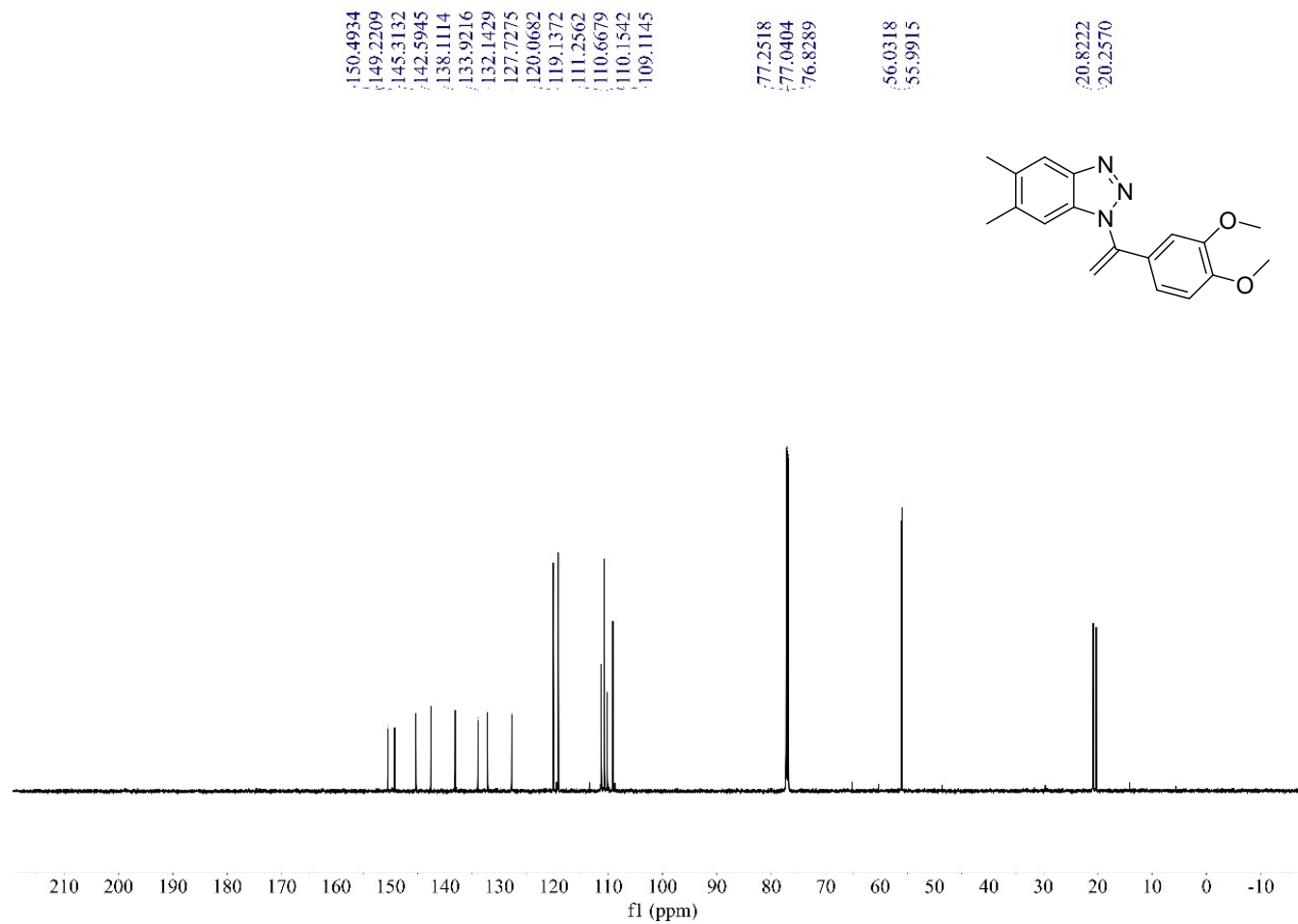
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3v



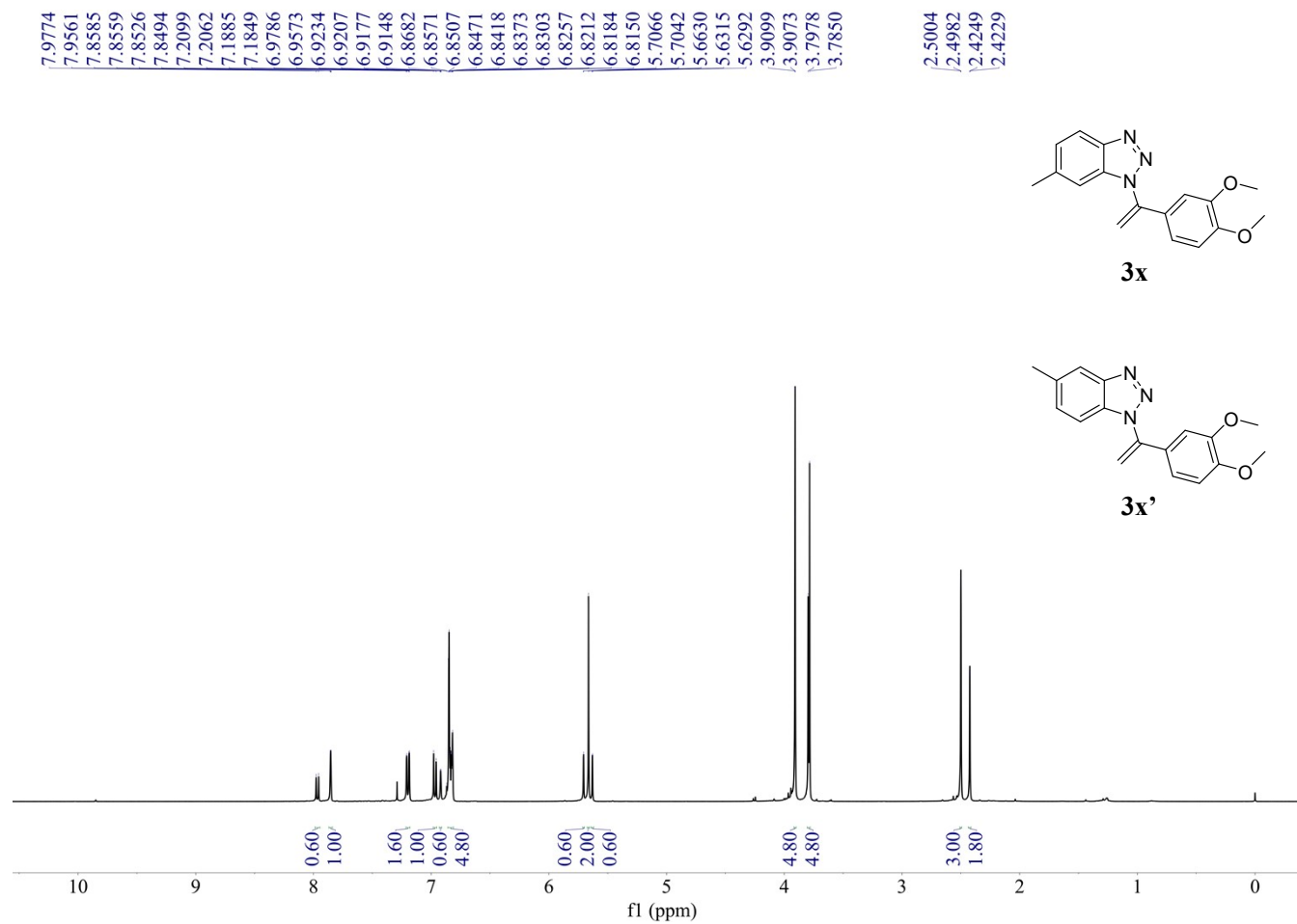
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3w



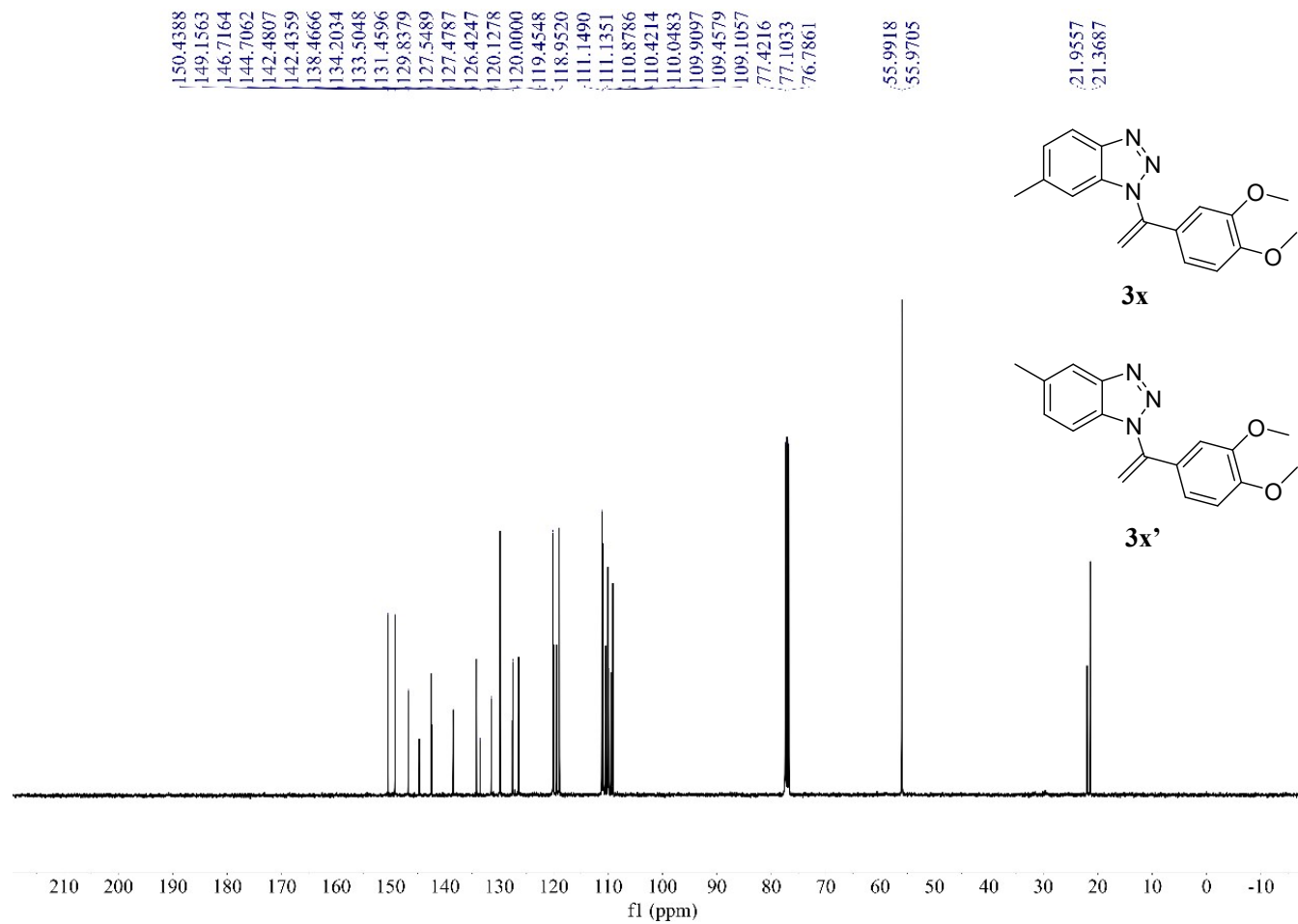
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3w



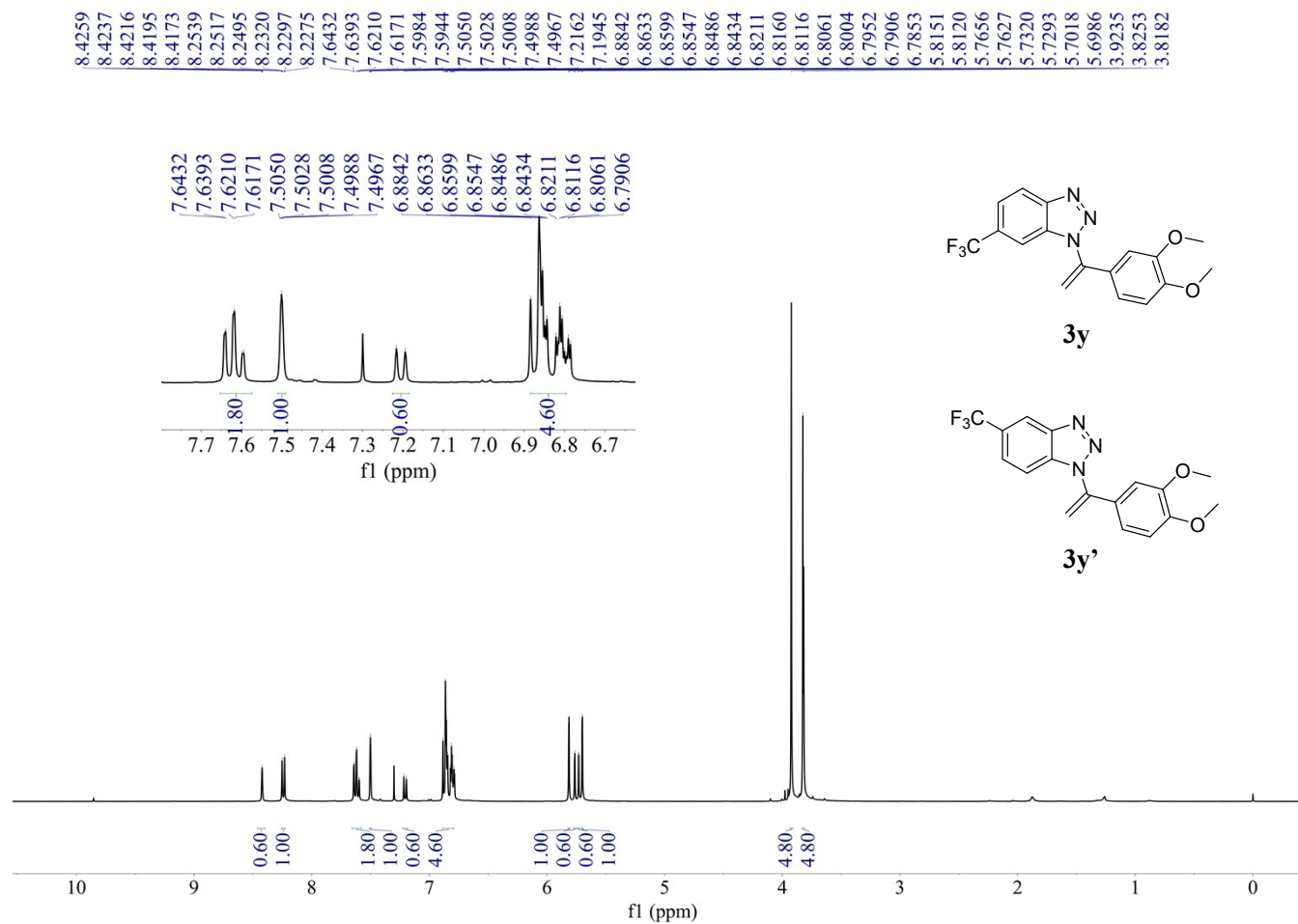
¹H-NMR Spectrum (400 MHz, CDCl₃) of 3x and 3x'



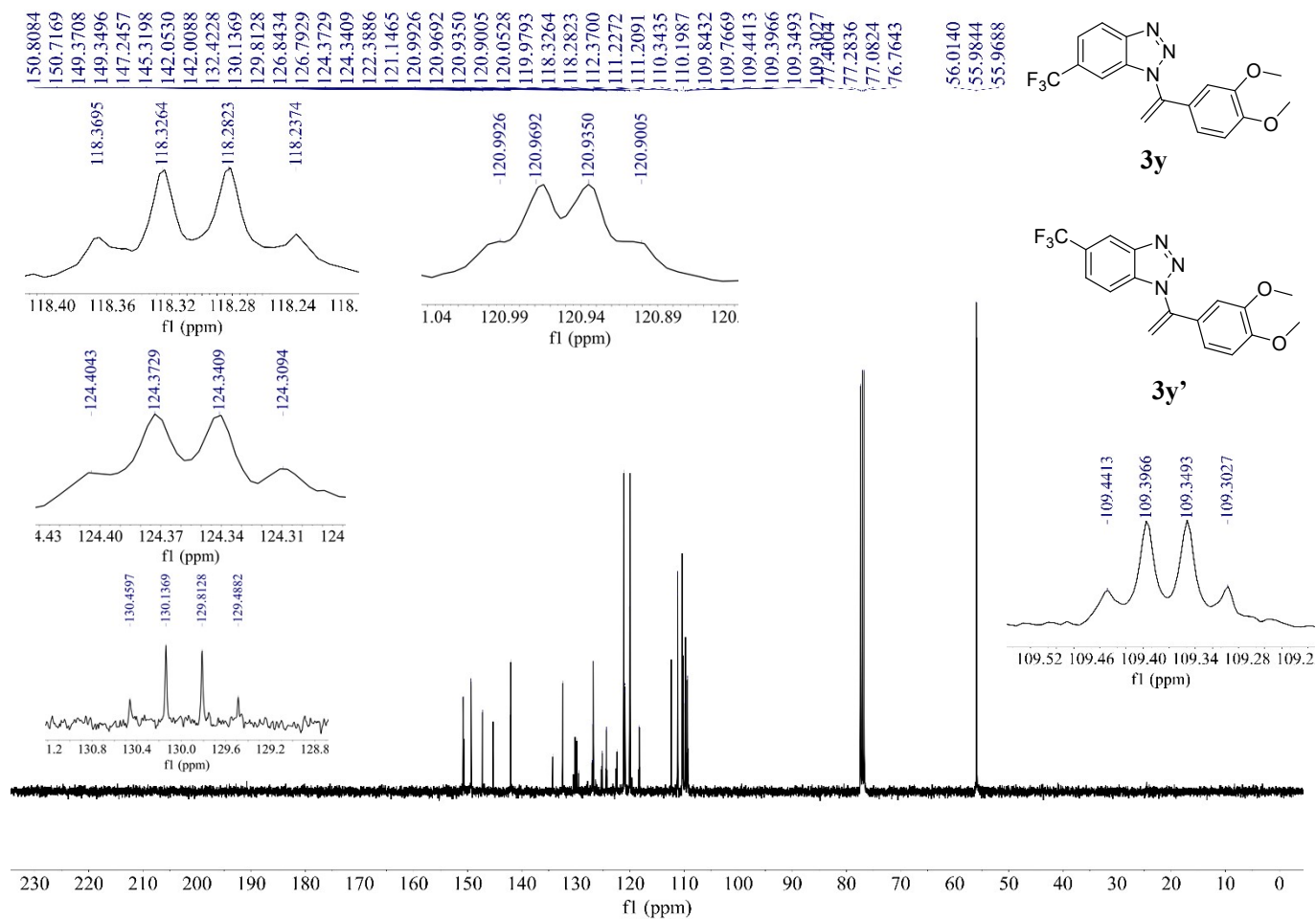
¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3x and 3x'



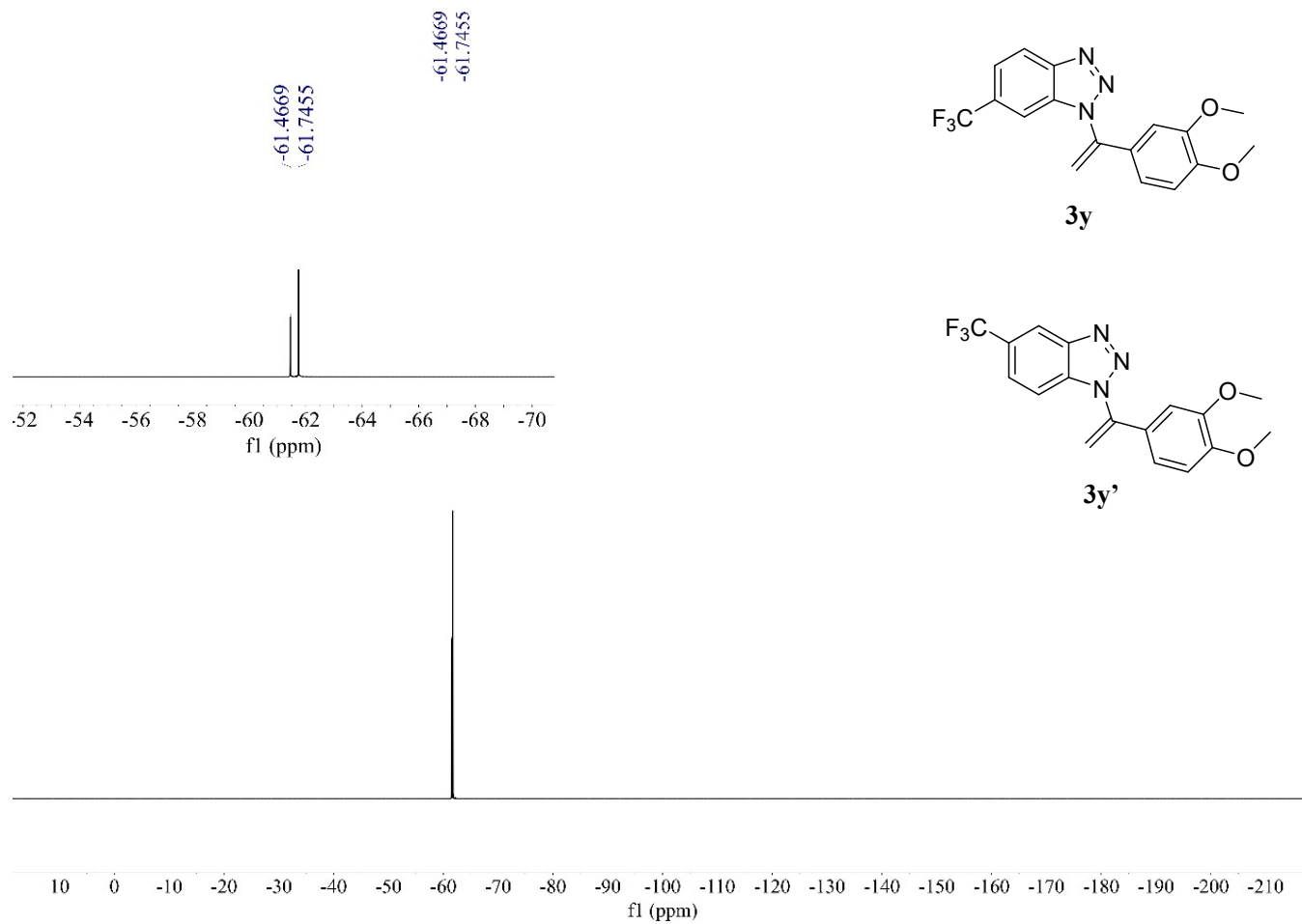
¹H-NMR Spectrum (400 MHz, CDCl₃) of 3y and 3y'



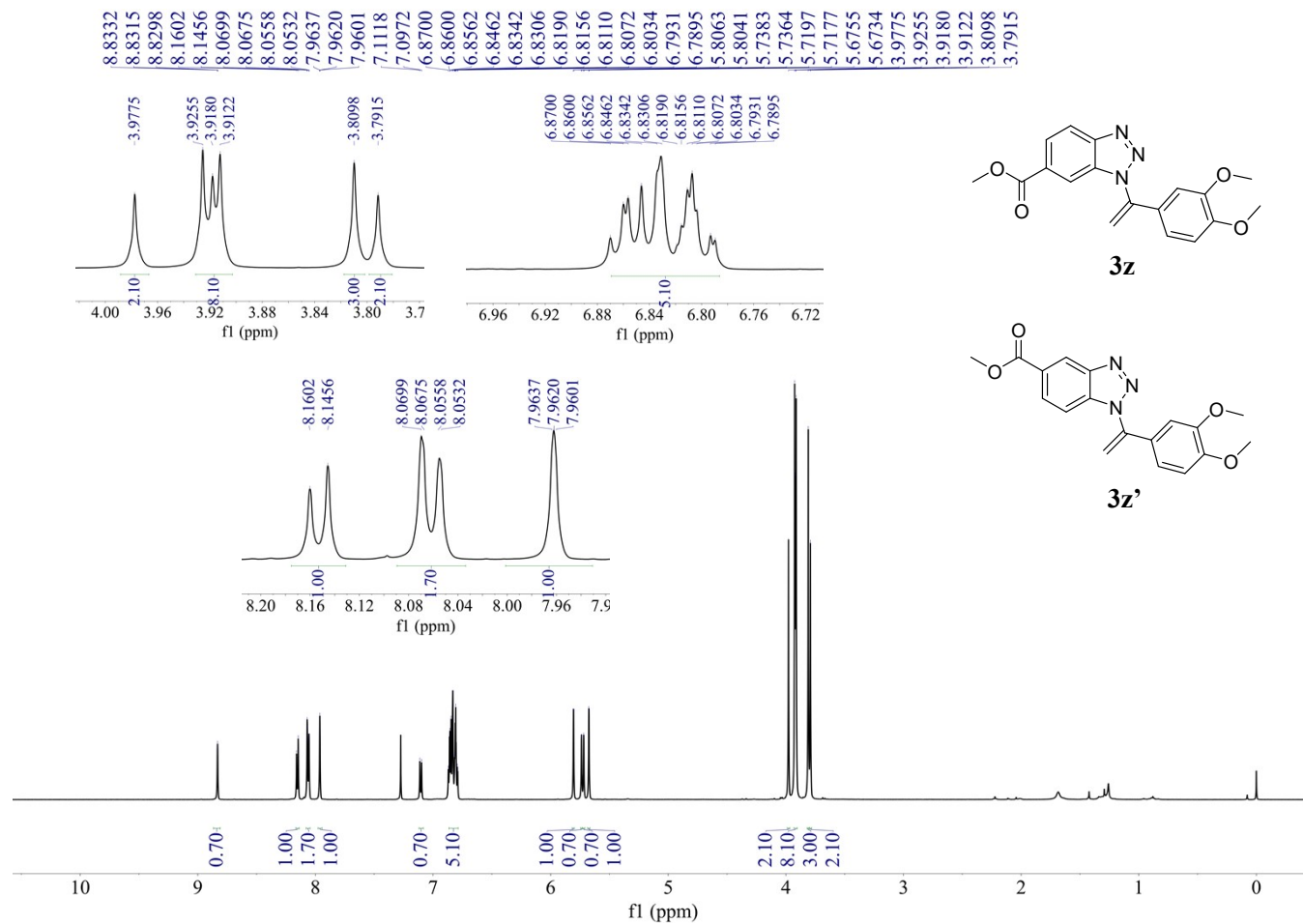
¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3y and 3y'



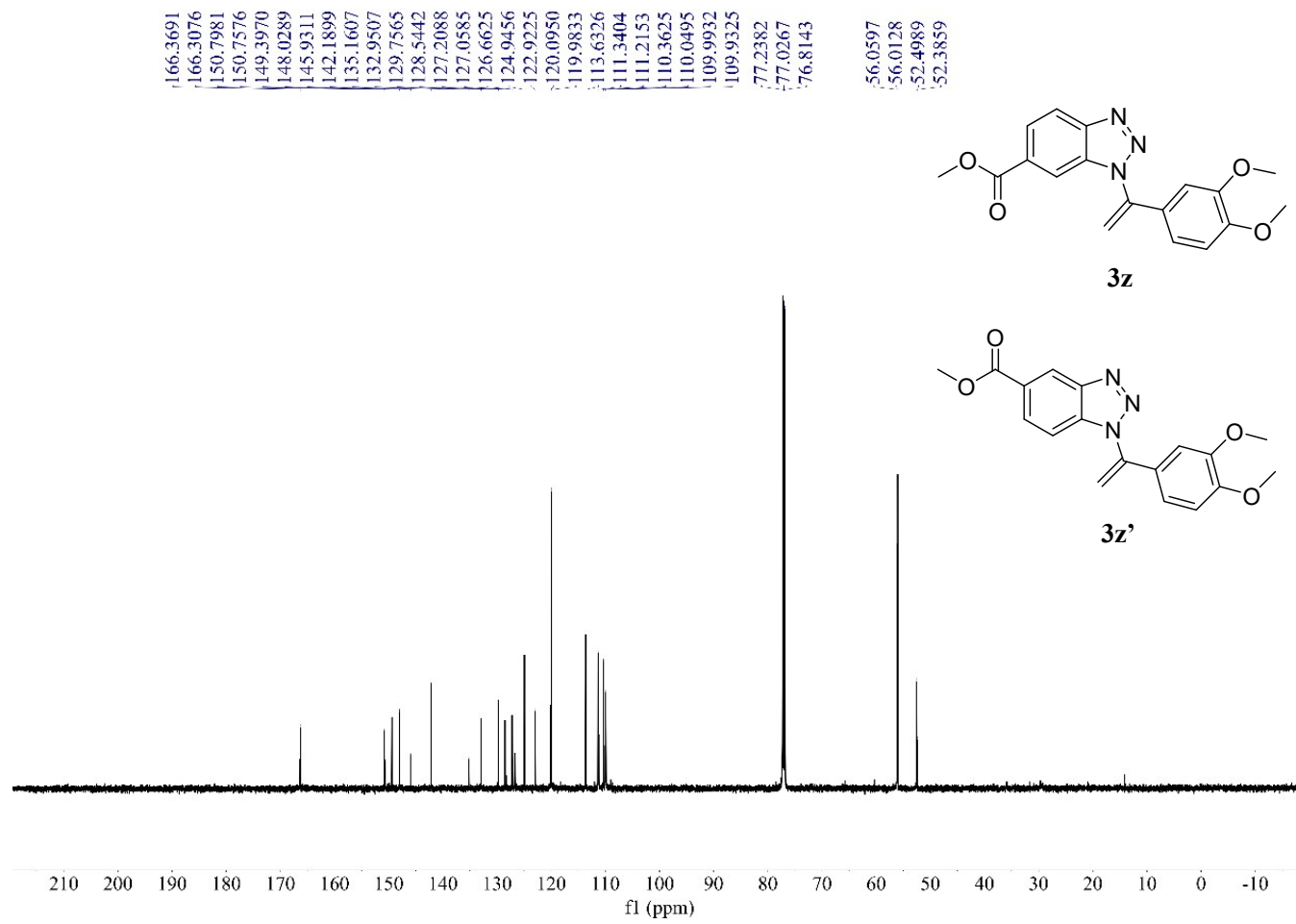
¹⁹F-NMR Spectrum (376 MHz, CDCl₃) of 3y and 3y'



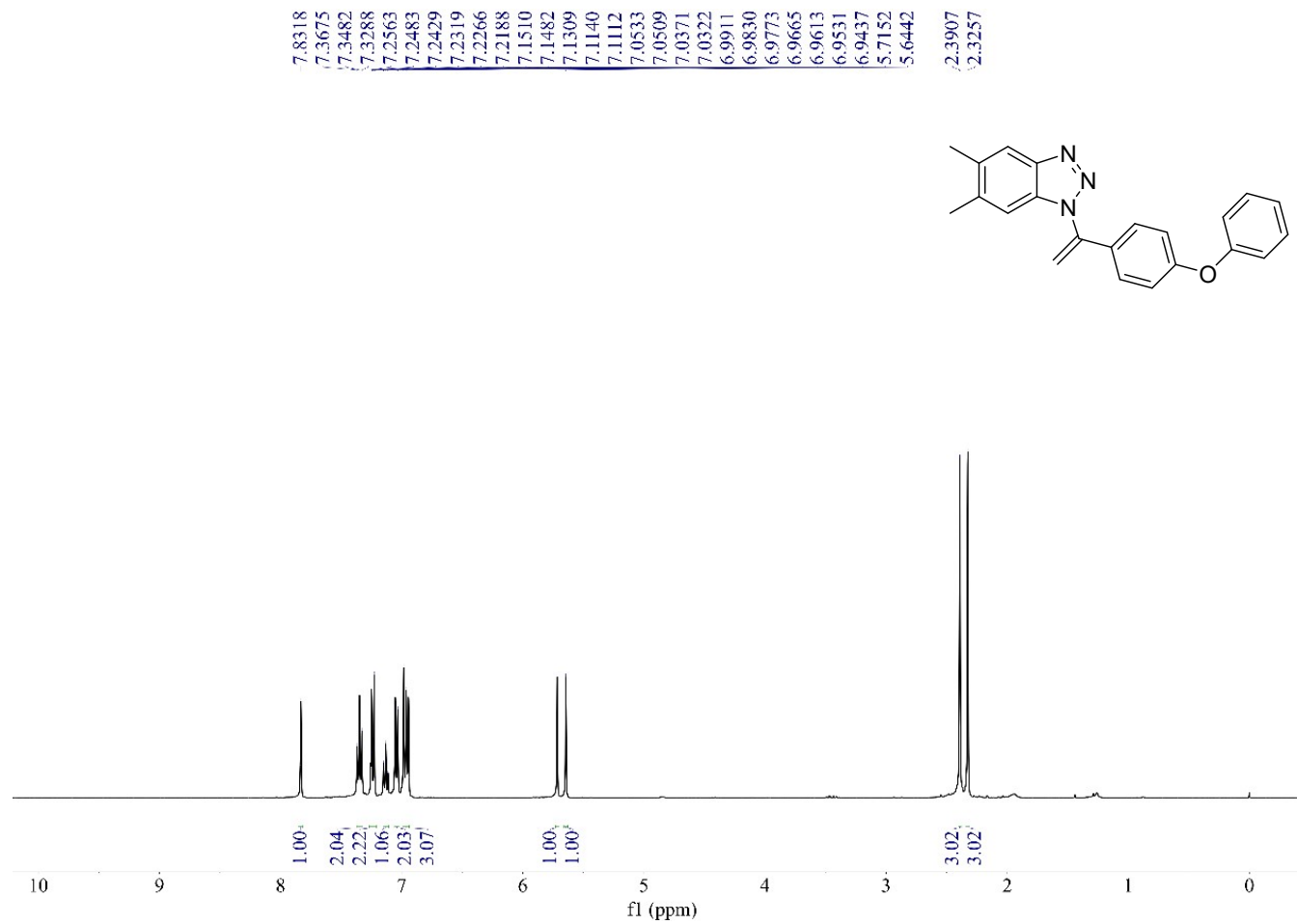
¹H-NMR Spectrum (600 MHz, CDCl₃) of 3z and 3z'



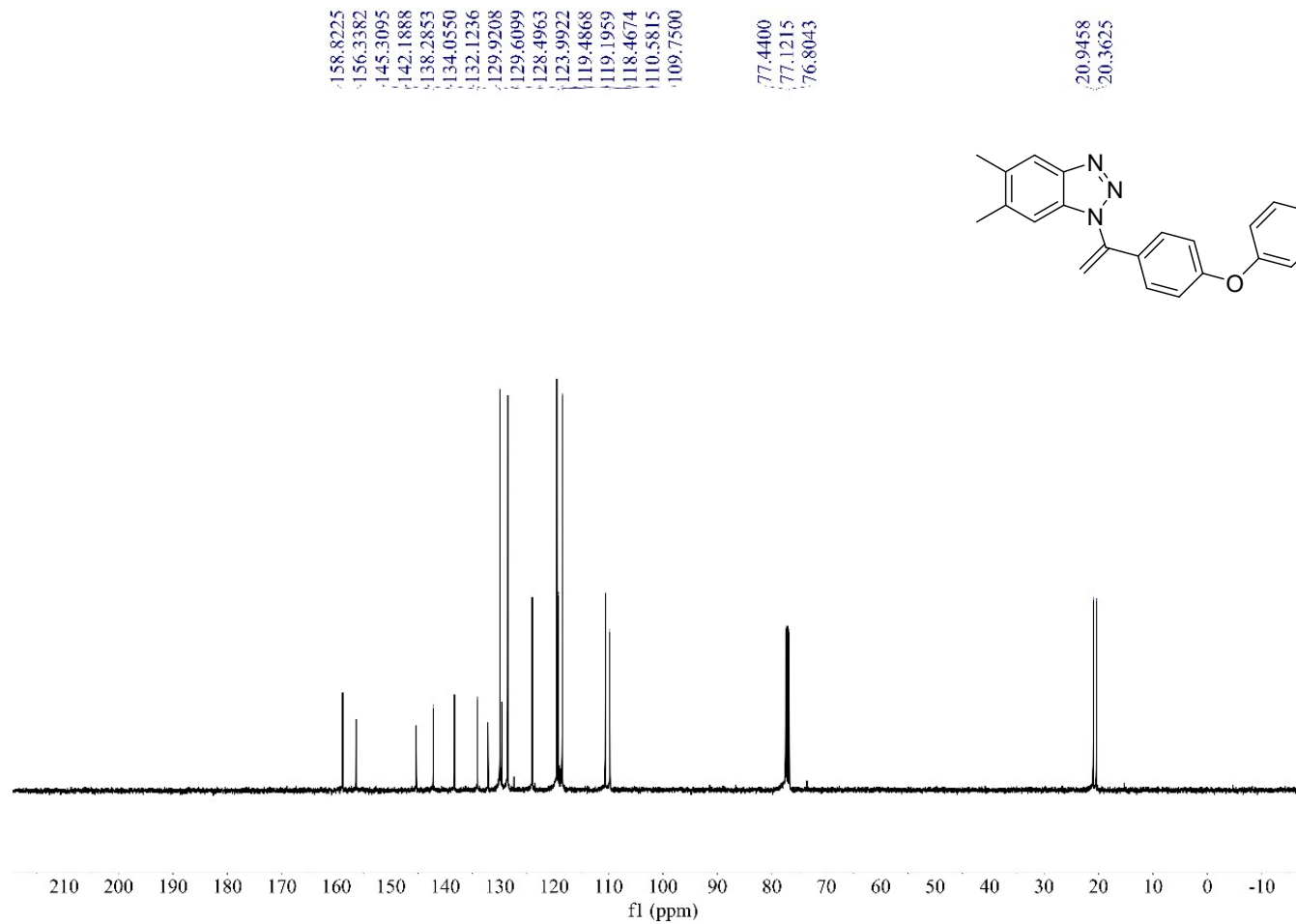
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 3z and 3z'



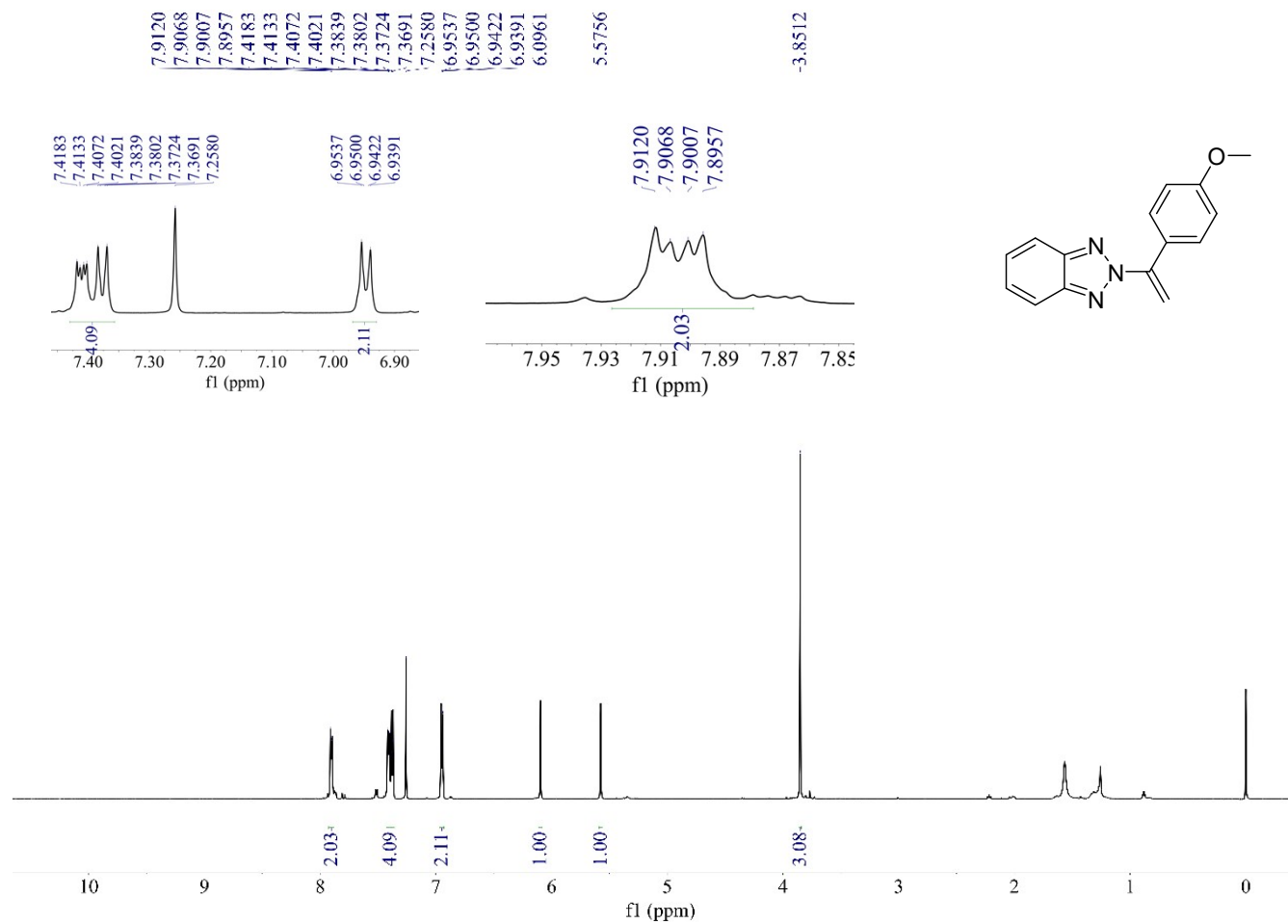
¹H-NMR Spectrum (400 MHz, CDCl₃) of 3aa



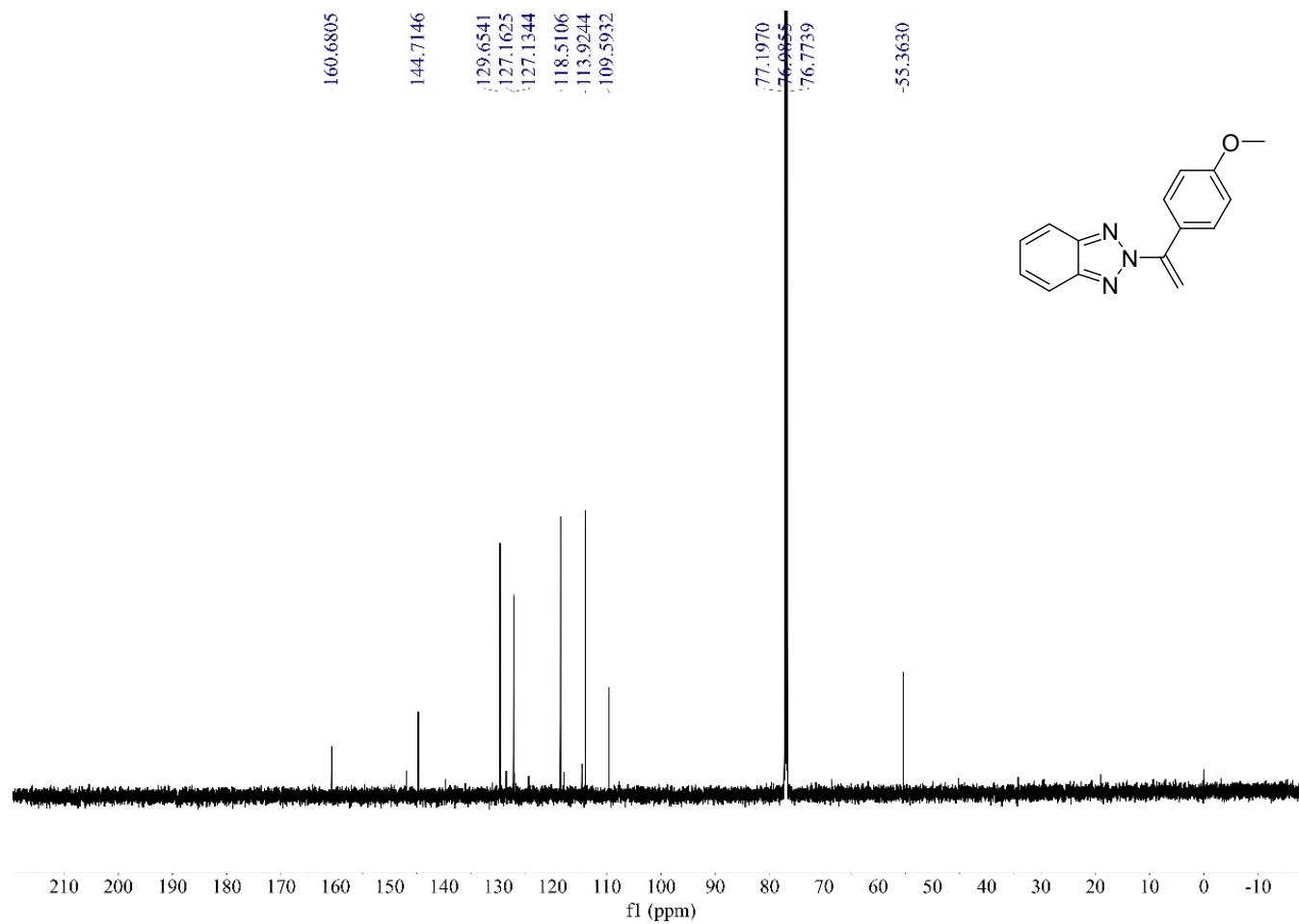
¹³C-NMR Spectrum (101 MHz, CDCl₃) of 3aa



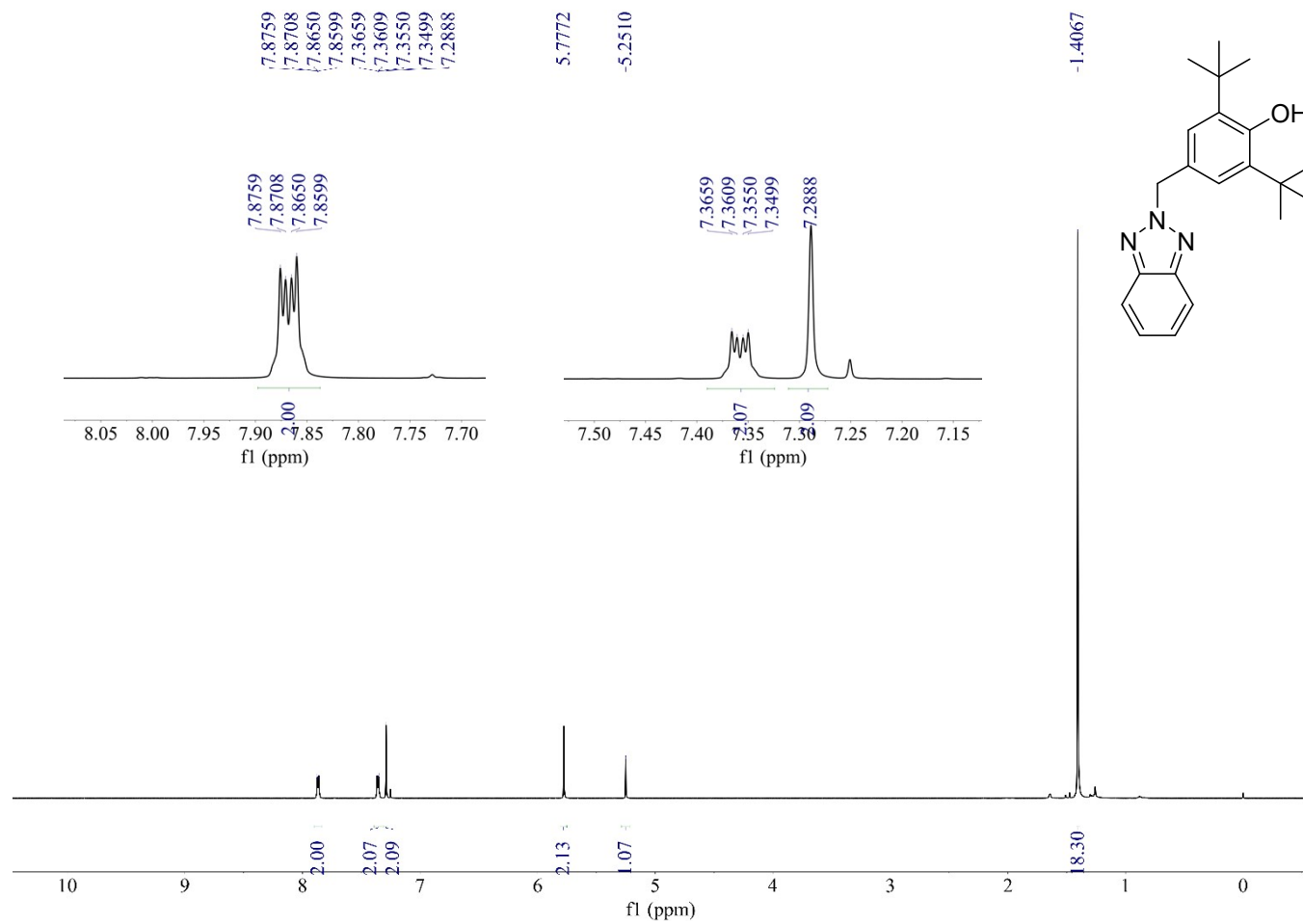
¹H-NMR Spectrum (600 MHz, CDCl₃) of 4a



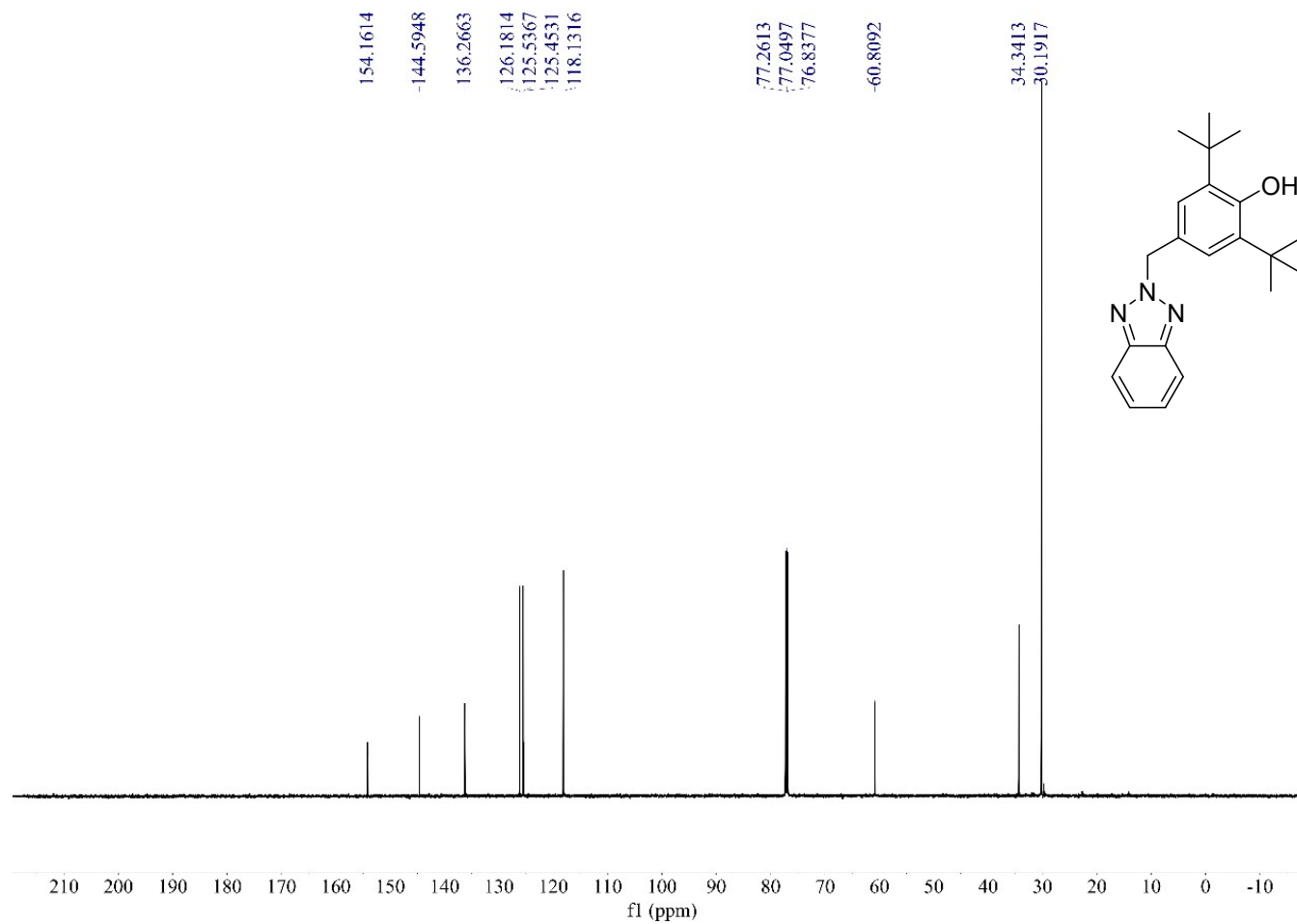
¹³C-NMR Spectrum (151 MHz, CDCl₃) of 4a



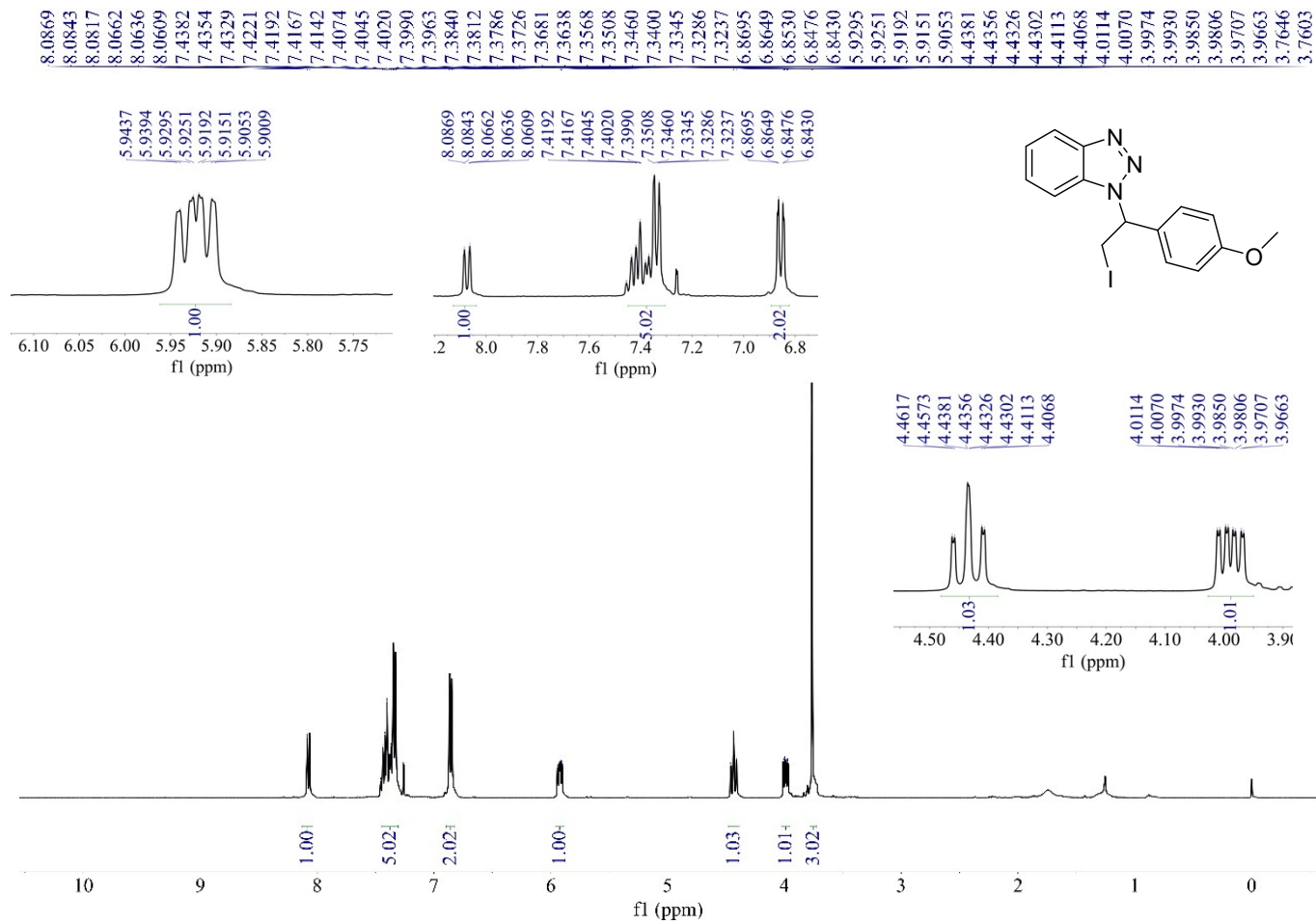
¹H-NMR Spectrum (600 MHz, CDCl₃) of 6a'



¹³C-NMR Spectrum (151 MHz, CDCl₃) of 6a'



¹H-NMR Spectrum (400 MHz, CDCl₃) of 8a



¹³C-NMR Spectrum (101 MHz, CDCl₃) of 8a

