Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Oxidant-Directed Chemoselective Sulfonylation and Sulfonyloximation of Alkenes via Cleaving C-S Bond in TosMIC

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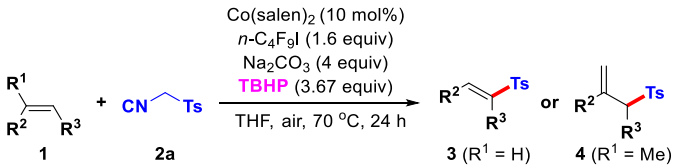
1. General information

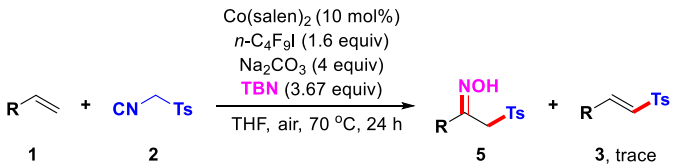
Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. Unless otherwise noted, all reactions were carried out under air using undistilled solvent, without the need of precautions to exclude air and moisture. Melting points were recorded on an Electrothermal digital melting point apparatus. IR spectra were recorded on a FT-IR spectrophotometer using KBr optics. ^{1}H, ^{19}F, and ^{13}C NMR spectra were recorded in $CDCl_3$ or DMSO-d_6 on Bruker Avance or Joel 400 MHz spectrometers. The chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. High resolution mass spectra (HRMS) were obtained using a commercial apparatus (ESI or EI Source). Column chromatography was generally performed on silica gel (300-400 mesh) or alkali alumina (200-300 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light to visualize the course of the reactions.

2. General procedures for the synthesis of vinyl sulfones 3 or allyl sulfones 4



A solution of alkene **1** (0.6 mmol, 2 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 59 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), perfluorobutyl iodide (166 mg, 0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), and *tert*-butyl hydroperoxide (142 mg, 1.1 mmol, 3.67 equiv, TBHP, 70% solution in H_2O) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. The reaction was then quenched by saturated $NaHSO_3$ solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was separated and washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) using petroleum ether/ethyl acetate (20/1~6/1) as eluent to afford the pure product vinyl sulfone **3** or allyl sulfone **4**.

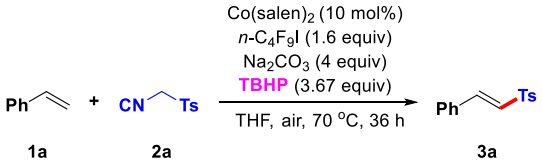
3. General procedures for the synthesis of α-sulfonylethanone oximes 5



A solution of alkene **1** (0.6 mmol, 2 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 59 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), perfluorobutyl iodide (166 mg, 0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), and *tert*-butyl nitrite (114 mg, 1.1 mmol, 3.67 equiv, TBN) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. The reaction was then quenched by saturated $NaHSO_3$ solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was separated and washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) using petroleum

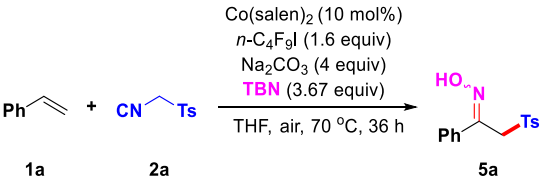
ether/ethyl acetate (20/1~3/1) as eluent to afford the pure product α-sulfonylethanone oxime **5**.

4. 10 mmol scale synthesis of (*E*)-1-methyl-4-(styrylsulfonyl)benzene (3a)



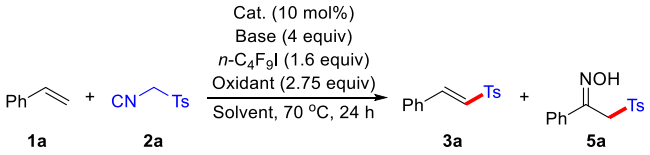
A solution of styrene (**1a**; 2.08 g, 20 mmol, 2 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 1.95 g, 10 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (0.33 g, 1 mmol, 0.1 equiv), perfluorobutyl iodide (5.53 g, 16 mmol, 1.6 equiv), Na_2CO_3 (4.23 g, 40 mmol, 4 equiv), and *tert*-butyl hydroperoxide (4.73 g, 36.7 mmol, 3.67 equiv, TBHP, 70% solution in H_2O) in THF (50 mL) was stirred under air atmosphere at 70 °C (oil bath) for 36 h. The reaction was then quenched by saturated $NaHSO_3$ solution (200 mL) and diluted with EtOAc (200 mL). The organic layer was separated and washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) using petroleum ether/ethyl acetate (20/1~6/1) as eluent to afford the pure product (*E*)-1-methyl-4-(styrylsulfonyl)benzene (**3a**) in 46% yield (1.19 g).

5. 10 mmol scale synthesis of 1-phenyl-2-tosylethan-1-one oxime (5a)



A solution of styrene (**1a**; 2.08 g, 20 mmol, 2 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 1.95 g, 10 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (0.33 g, 1 mmol, 0.1 equiv), perfluorobutyl iodide (5.53 g, 16 mmol, 1.6 equiv), Na_2CO_3 (4.23 g, 40 mmol, 4 equiv), and *tert*-butyl nitrite (3.79 g, 36.7 mmol, 3.67 equiv, TBN) in THF (50 mL) was stirred under air atmosphere at 70 °C (oil bath) for 36 h. The reaction was then quenched by saturated $NaHSO_3$ solution (200 mL) and diluted with EtOAc (200 mL). The organic layer was separated and washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) using petroleum ether/ethyl acetate (20/1~3/1) as eluent to afford the pure product 1-phenyl-2-tosylethan-1-one oxime (**5a**) in 51% yield (1.47 g).

6. Table S1. Optimization of the reaction conditions[a]



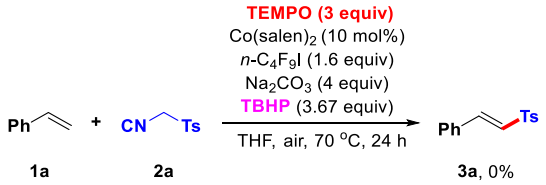
Entry	Catalyst	Base	Oxidant	Solvent	Time (h)	Yield of 3a (%)[b]	Yield of 5a (%)[b]
1	$Co(salen)_2$	DABCO	TBHP	THF	12	36	0
2	$Co(salen)_2$	DABCO	TBHP	MeCN	12	trace	0

3	$Co(salen)_2$	DABCO	TBHP	DCE	12	trace	0
4	$Co(salen)_2$	DABCO	TBHP	DMF	12	<5	0
5	$Co(salen)_2$	DABCO	TBHP	DMSO	12	<5	0
6	$Co(salen)_2$	DABCO	TBHP	DME	12	<10	0
7	$Co(salen)_2$	DABCO	TBHP	toluene	12	0	0
8	$Co(salen)_2$	DABCO	TBHP	1,4-dioxane	12	<5	0
9	$Co(salen)_2$	DABCO	TBHP	EtOAc	12	trace	0
10	$Co(salen)_2$	K_2CO_3	TBHP	THF	24	52	0
11	$Co(salen)_2$	Cs_2CO_3	TBHP	THF	24	<5	0
12	$Co(salen)_2$	DBU	TBHP	THF	24	20	0
13	$Co(salen)_2$	PMEDA	TBHP	THF	24	42	0
14	$Co(salen)_2$	Et_3N	TBHP	THF	24	55 (54)[c]	0
15	$Co(salen)_2$	NaOH	TBHP	THF	24	47	0
16	$Co(salen)_2$	Na_2CO_3	TBHP	THF	24	60 (59)[c]	0
17	**$Co(salen)_2$**	**Na_2CO_3**	**TBHP**	**THF**	**24**	**86[d](75)[c]**	**0**
18	$Co(acac)_2$	Na_2CO_3	TBHP	THF	24	85[d]	0
19	$CoBr_2$	Na_2CO_3	TBHP	THF	24	82[d]	0
20	$Co(acac)_3$	Na_2CO_3	TBHP	THF	24	79[d]	0
21	Co	Na_2CO_3	TBHP	THF	24	80[d]	0
22	--	Na_2CO_3	TBHP	THF	24	55[d]	0
23	$Co(salen)_2$	Na_2CO_3	TBPB	THF	24	83[d]	0
24	$Co(salen)_2$	Na_2CO_3	AIBN	THF	24	<10[d]	0
25	$Co(salen)_2$	Na_2CO_3	DHP	THF	24	78[d]	0
26	$Co(salen)_2$	Na_2CO_3	DTBP	THF	24	<10[d]	0
27	$Co(salen)_2$	Na_2CO_3	TBHP	THF	24	38[d,e]	0[d,e]
28	$Co(salen)_2$	Na_2CO_3	TBN	THF	24	trace[d,e]	28[d,e]
29	$Co(salen)_2$	Na_2CO_3	$K_2S_2O_8$	THF	24	trace[d]	0
30	$Co(salen)_2$	K_2CO_3	TBN	THF	24	trace[d]	<20[d]
31	$Co(salen)_2$	DABCO	TBN	THF	24	10[d,e]	30[d,e]
32	**$Co(salen)_2$**	**Na_2CO_3**	**TBN**	**THF**	**24**	**trace[d]**	**(73)[c,d]**
33	$Co(salen)_2$	Na_2CO_3	--	THF	24	<10[d]	0[d]

[a] Reaction conditions: **1a** (0.45 mmol), **2a** (0.3 mmol), catalyst (0.03 mmol), base (1.2 mmol), *n*-C_4F_9I (0.48 mmol), and oxidant (0.825 mmol) in solvent (2 mL) at 70 ℃ under air; (TBHP = *tert*-butyl hydroperoxide; TBPB = *tert*-butyl peroxybenzoate; AIBN = 2,2'-azobis(2-methylpropionitrile); DHP = cumyl hydroperoxide; TBN = *tert*-butyl nitrite; PMEDA = 1,1,4,7,7-pentamethyl-diethylenetriamine). [b] Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard. [c] Isolated yields. [d] **1a** (0.6 mmol), *n*-C_4F_9I (0.48 mmol), Na_2CO_3 (1.2 mmol), and oxidant (3.67 equiv) were employed. [e] Without *n*-C_4F_9I.

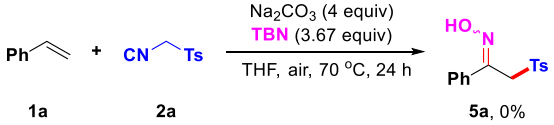
7. Mechanistic studies

1) Trapping experiment with 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO)



1a **2a** **3a**, 0%

A solution of styrene (**1a**; 63 mg, 0.6 mmol, 2 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 59 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), perfluorobutyl iodide (166 mg, 0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), 2,2,6,6-tetramethylpiperidin-1-oxyl (141 mg, 0.9 mmol, 3 equiv, TEMPO), and *tert*-butyl hydroperoxide (142 mg, 1.1 mmol, 3.67 equiv, TBHP, 70% solution in H_2O) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. No (*E*)-1-methyl-4-(styrylsulfonyl)benzene (**3a**) was detected.



1a **2a** **5a**, 0%

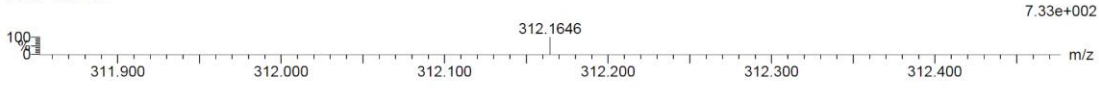
A solution of styrene (**1a**; 63 mg, 0.6 mmol, 2 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 59 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), perfluorobutyl iodide (166 mg, 0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), 2,2,6,6-tetramethylpiperidin-1-oxyl (141 mg, 0.9 mmol, 3 equiv, TEMPO), and *tert*-butyl nitrite (114 mg, 1.1 mmol, 3.67 equiv, TBN) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. No 1-phenyl-2-tosylethan-1-one oxime (**5a**) was detected.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

calcd for $C_{16}H_{26}NO_3S^+$ [M+H]$^+$: 312.1628
found: 312.1646

Monoisotopic Mass, Even Electron Ions
93 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 15-25 H: 25-28 N: 0-3 O: 1-3 S: 1-1 Fe: 0-3 Se: 0-2
CXQ-27 836 (3.126)
1: TOF MS ES+



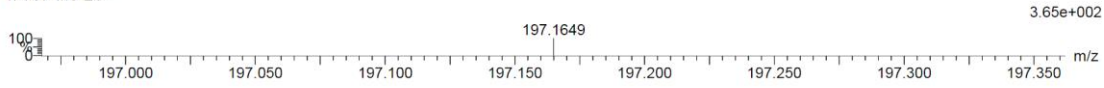
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
312.1646	312.1633	1.3	4.2	4.5	23.2	n/a	n/a	C16 H26 N O3 S

Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

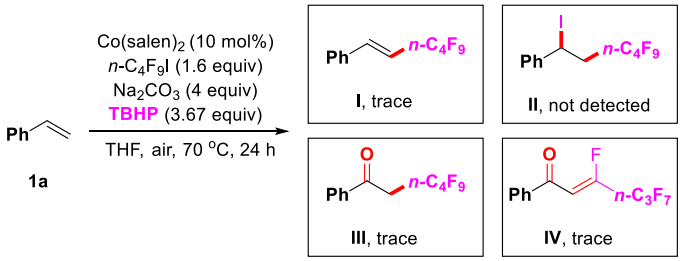
calcd for $C_{11}H_{21}N_2O^+$ [M+H]$^+$: 197.1648
found: 197.1649

Monoisotopic Mass, Even Electron Ions
51 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 10-15 H: 20-25 N: 0-3 O: 1-3 Fe: 0-3 Se: 0-2
CXQ-27 443 (1.664)
1: TOF MS ES+



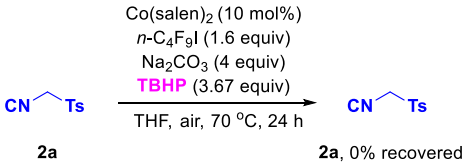
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
197.1649	197.1654	-0.5	-2.5	2.5	20.1	n/a	n/a	C11 H21 N2 O

2) Control experiment without 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (2a)



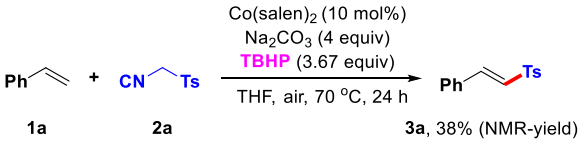
A solution of styrene (**1a**; 63 mg, 0.6 mmol, 2 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), perfluorobutyl iodide (166 mg, 0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), and *tert*-butyl hydroperoxide (142 mg, 1.1 mmol, 3.67 equiv, TBHP, 70% solution in H_2O) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. No products **I**-**IV** were obtained under the optimized conditions.

3) Control experiment without styrene (1a)



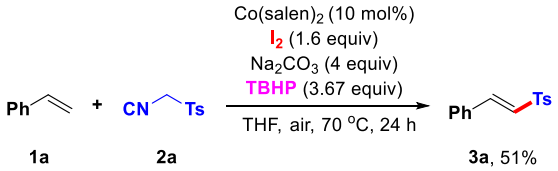
A solution of 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 59 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), perfluorobutyl iodide (166 mg, 0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), and *tert*-butyl hydroperoxide (142 mg, 1.1 mmol, 3.67 equiv, TBHP, 70% solution in H_2O) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. No 1-((isocyanomethyl)sulfonyl)-4-methylbenzene **2a** was recovered under the optimized conditions. The reaction is efficient for the cleavage of C-S bond in TosMIC.

4) Control experiment without perfluorobutyl iodide



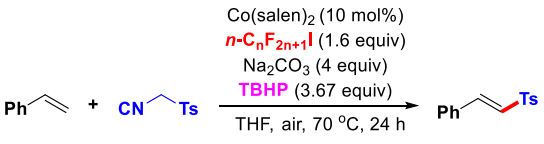
A solution of styrene (**1a**; 63 mg, 0.6 mmol, 2 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 59 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), and *tert*-butyl hydroperoxide (142 mg, 1.1 mmol, 3.67 equiv, TBHP, 70% solution in H_2O) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard.

5) Control experiment using iodine instead of perfluorobutyl iodide



A solution of styrene (**1a**; 63 mg, 0.6 mmol, 2 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 59 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), iodine (122 mg, 0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), and *tert*-butyl hydroperoxide (142 mg, 1.1 mmol, 3.67 equiv, TBHP, 70% solution in H_2O) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. The reaction was then quenched by saturated $NaHSO_3$ solution (20 mL) and diluted with EtOAc (20 mL). The organic layer was separated and washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (300-400 mesh) using petroleum ether/ethyl acetate (20/1~6/1) as eluent to afford the pure product (*E*)-1-methyl-4-(styrylsulfonyl)benzene (**3a**) in 51% yield (40 mg).

6) Control experiments using other perfluoroalkyl halides instead of perfluorobutyl iodide

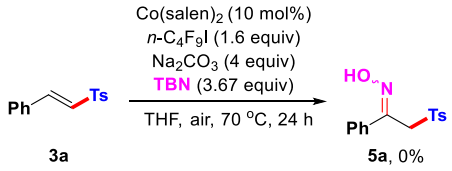


n-$C_nF_{2n+1}I$	NMR yield of **3a**
n-C_3F_7I	77%
n-$C_8F_{17}I$	83%
n-$C_{10}F_{21}I$	85%

A solution of styrene (**1a**; 63 mg, 0.6 mmol, 2 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 59 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), *n*-$C_nF_{2n+1}I$ (0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), and *tert*-butyl hydroperoxide (TBHP; 142 mg, 1.1 mmol, 3.67 equiv, 70% solution in H_2O) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. The reaction was then quenched by saturated $NaHSO_3$ solution (20 mL) and diluted with EtOAc (20 mL). The organic

layer was separated and washed with saturated brine twice, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Yields were determined by NMR analysis with 1,4-dimethoxybenzene as an internal standard.

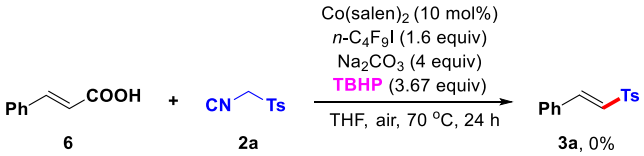
7) Control experiment by using (*E*)-1-methyl-4-(styrylsulfonyl)benzene (3a)



3a **5a**, 0%

A solution of (*E*)-1-methyl-4-(styrylsulfonyl)benzene (**3a**; 78 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), perfluorobutyl iodide (166 mg, 0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), and *tert*-butyl nitrite (114 mg, 1.1 mmol, 3.67 equiv, TBN) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. No 1-phenyl-2-tosylethan-1-one oxime (**5a**) was detected.

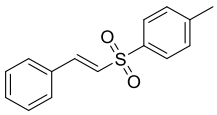
8) Control experiment by using cinnamic acid (6)



6 **2a** **3a**, 0%

A solution of cinnamic acid (**6**; 89 mg, 0.3 mmol, 1 equiv), 1-((isocyanomethyl)sulfonyl)-4-methylbenzene (**2a**; 59 mg, 0.3 mmol, 1 equiv), *N*,*N*'-bis(salicylidene)ethylenediamine cobalt(II) (10 mg, 0.03 mmol, 0.1 equiv), perfluorobutyl iodide (166 mg, 0.48 mmol, 1.6 equiv), Na_2CO_3 (127 mg, 1.2 mmol, 4 equiv), and *tert*-butyl hydroperoxide (TBHP; 142 mg, 1.1 mmol, 3.67 equiv, 70% solution in H_2O) in THF (1.5 mL) was stirred under air atmosphere at 70 °C (oil bath) for 24 h. However, no desired product of (*E*)-1-methyl-4-(styrylsulfonyl)benzene (**3a**) was detected.

8. Characterization data for products



(*E*)-1-Methyl-4-(styrylsulfonyl)benzene (3a):

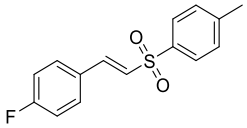
Yield = 75% (58 mg). White solid. M.p. = 173.4–173.8 °C.

IR (KBr): ν = 3045, 2923, 1595, 1449, 1304, 1143, 973, 810 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 – 7.81 (m, 2H), 7.66 (d, J = 15.4 Hz, 1H), 7.49 (s, 2H), 7.42 – 7.36 (m, 3H), 7.34 (d, J = 8.0 Hz, 2H), 6.85 (d, J = 15.4 Hz, 1H), 2.43 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.4, 141.9, 137.6, 132.4, 131.1, 129.9, 129.0, 128.5, 127.7, 127.5, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{15}O_2S$ $[M+H]^+$ 259.0787, found: 259.0793.



(*E*)-1-Fluoro-4-(2-tosylvinyl)benzene (3b):

Yield = 49% (41 mg). White solid. M.p. = 177.1–178.5 ºC.

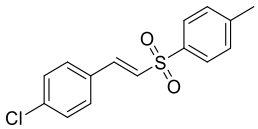
IR (KBr): ν = 3269, 2927, 1582, 1392, 1151, 945, 816 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (dd, J = 8.4, 1.9 Hz, 2H), 7.62 (d, J = 15.4 Hz, 1H), 7.51 – 7.44 (m, 2H), 7.35 (d, J = 8.4 Hz, 2H), 7.12 – 7.04 (m, 2H), 6.79 (d, J = 15.4 Hz, 1H), 2.43 (s, 3H) ppm.

^{19}F NMR (376 MHz, $CDCl_3$): δ = -107.8 (s) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 164.2 (d, $J_{C\text{-}F}$ = 251.0 Hz), 144.4, 140.6, 137.6, 130.5 (d, $J_{C\text{-}F}$ = 8.7 Hz), 130.0, 128.7 (d, $J_{C\text{-}F}$ = 3.4 Hz), 127.6, 127.4 (d, $J_{C\text{-}F}$ = 2.6 Hz), 116.2 (d, $J_{C\text{-}F}$ = 21.9 Hz), 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{14}FO_2S$ $[M+H]^+$ 277.0693, found: 277.0699.



(*E*)-1-Chloro-4-(2-tosylvinyl)benzene (3c):

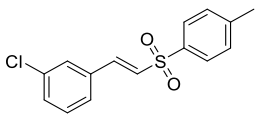
Yield = 57% (50 mg). Light yellow solid. M.p. = 127.1–129.2 ºC.

IR (KBr): ν = 3053, 2922, 1613, 1489, 1304, 1011, 787 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (d, J = 8.3 Hz, 2H), 7.60 (d, J = 15.4 Hz, 1H), 7.43 – 7.32 (m, 6H), 6.84 (d, J = 15.4 Hz, 1H), 2.43 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.5, 140.3, 137.4, 137.0, 130.8, 130.0, 129.6, 129.3, 128.1, 127.7, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{14}ClO_2S$ $[M+H]^+$ 293.0398, found: 293.0403.



(*E*)-1-Chloro-3-(2-tosylvinyl)benzene (3d):

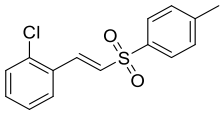
Yield = 47% (41 mg). Brown oil.

IR (KBr): ν = 3051, 2926, 1594, 1301, 1085, 810, 778 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.85 – 7.80 (m, 2H), 7.59 (d, J = 15.4 Hz, 1H), 7.45 (t, J = 1.7 Hz, 1H), 7.39 – 7.30 (m, 5H), 6.87 (d, J = 15.4 Hz, 1H), 2.44 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.6, 140.1, 137.2, 135.0, 134.2, 130.9, 130.3, 130.0, 129.1, 128.1, 127.8, 126.7, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{14}ClO_2S$ $[M+H]^+$ 293.0398, found: 293.0406.



(*E*)-1-Chloro-2-(2-tosylvinyl)benzene (3e):

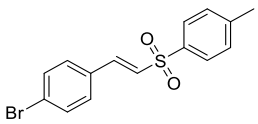
Yield = 42% (37 mg). Yellow solid. M.p. = 159.8–160.4 °C.

IR (KBr): ν = 3055, 2922, 1706, 1439, 1320, 1145, 964, 808 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.05 (d, J = 15.5 Hz, 1H), 7.87 – 7.81 (m, 2H), 7.50 (dd, J = 7.8, 1.7 Hz, 1H), 7.41 (dd, J = 8.0, 1.3 Hz, 1H), 7.38 – 7.33 (m, 2H), 7.32 (dd, J = 8.0, 1.7 Hz, 1H), 7.28 – 7.25 (m, 1H), 6.90 (d, J = 15.4 Hz, 1H), 2.44 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.6, 137.7, 137.2, 135.1, 131.8, 130.6, 130.3, 130.2, 130.0, 128.1, 127.8, 127.1, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{14}ClO_2S$ $[M+H]^+$ 293.0398, found: 293.0403.



(*E*)-1-Bromo-4-(2-tosylvinyl)benzene (3f):

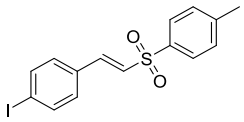
Yield = 53% (54 mg). Yellow solid. M.p. = 145.5–146.9 °C.

IR (KBr): ν = 3043, 2925, 1619, 1487, 1303, 1142, 813 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 – 7.79 (m, 2H), 7.58 (d, J = 15.4 Hz, 1H), 7.54 – 7.48 (m, 2H), 7.34 (td, J = 6.7, 1.2 Hz, 4H), 6.86 (d, J = 15.4 Hz, 1H), 2.43 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.5, 140.4, 137.3, 132.3, 131.3, 130.0, 129.8, 128.2, 127.7, 125.4, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{14}{}^{79}BrO_2S$ $[M+H]^+$ 336.9892, found: 336.9898.



(*E*)-1-Iodo-4-(2-tosylvinyl)benzene (3g):

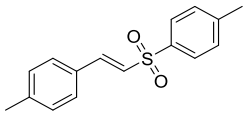
Yield = 55% (64 mg). White solid. M.p. = 165.2–167.2 °C.

IR (KBr): ν = 3041, 1617, 1580, 1481, 1303, 1142, 972, 857 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (d, J = 8.2 Hz, 2H), 7.75 – 7.70 (m, 2H), 7.56 (d, J = 15.4 Hz, 1H), 7.37 – 7.32 (m, 2H), 7.21 – 7.17 (m, 2H), 6.86 (d, J = 15.4 Hz, 1H), 2.43 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.5, 140.6, 138.2, 137.3, 131.8, 130.0, 129.8, 128.3, 127.7, 97.6, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{14}IO_2S$ $[M+H]^+$ 384.9754, found: 384.9759.



(*E*)-1-Methyl-4-((4-methylstyryl)sulfonyl)benzene (3h):

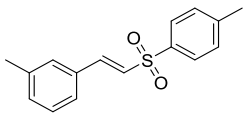
Yield = 56% (46 mg). White solid. M.p. = 133.4–135.0 °C.

IR (KBr): ν = 3045, 2921, 1607, 1314, 1141, 795, 659 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (d, J = 8.2 Hz, 2H), 7.63 (d, J = 15.4 Hz, 1H), 7.35 (dd, J = 12.4, 8.1 Hz, 4H), 7.18 (d, J = 7.9 Hz, 2H), 6.80 (d, J = 15.4 Hz, 1H), 2.43 (s, 3H), 2.36 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.2, 141.9, 141.7, 137.8, 129.9, 129.7, 129.6, 128.5, 127.6, 126.3, 21.6, 21.5 ppm. 144.2, 141.9, 141.7, 137.8, 129.9, 129.8, 129.7, 129.6, 128.5, 127.6, 126.3,

HRMS (m/z): calcd for $C_{16}H_{17}O_2S$ $[M+H]^+$ 273.0944, found: 273.0939.



(*E*)-1-Methyl-3-(2-tosylvinyl)benzene (3i):

Yield = 50% (41 mg). Light yellow oil.

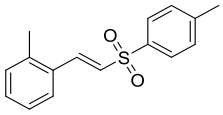
IR (KBr): ν = 3047, 2922, 1614, 1452, 1301, 1144, 1085, 844, 663 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.84 – 7.80 (m, 2H), 7.62 (d, J = 15.4 Hz, 1H), 7.36 – 7.31 (m,

2H), 7.30 – 7.25 (m, 3H), 7.23 – 7.19 (m, 1H), 6.83 (d, *J* = 15.4 Hz, 1H), 2.43 (s, 3H), 2.34 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.3, 142.1, 138.7, 137.7, 132.3, 131.9, 129.9, 129.0, 128.9, 127.6, 127.2, 125.7, 21.6, 21.2 ppm.

HRMS (m/z): calcd for $C_{16}H_{17}O_2S$ $[M+H]^+$ 273.0944, found: 273.0949.



(*E*)-1-Methyl-2-(2-tosylvinyl)benzene (3j):

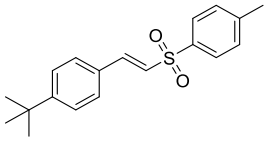
Yield = 50% (41 mg). Brown solid. M.p. = 174.4–176.0 °C.

IR (KBr): ν = 3058, 2966, 1614, 1596, 1302, 1143, 1086, 961, 760 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94 (d, *J* = 15.3 Hz, 1H), 7.83 (d, *J* = 8.3 Hz, 2H), 7.42 (d, *J* = 7.7 Hz, 1H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.31 – 7.25 (m, 1H), 7.21 – 7.15 (m, 2H), 6.78 (d, *J* = 15.3 Hz, 1H), 2.44 (s, 3H), 2.43 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.3, 139.5, 138.0, 137.6, 131.2, 130.9, 130.7, 129.9, 128.4, 127.6, 126.7, 126.4, 21.5, 19.7 ppm.

HRMS (m/z): calcd for $C_{16}H_{17}O_2S$ $[M+H]^+$ 273.0944, found: 273.0949.



(*E*)-1-(*Tert*-butyl)-4-(2-tosylvinyl)benzene (3k):

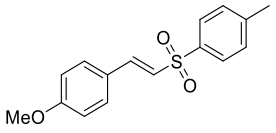
Yield = 53% (50 mg). White solid. M.p. = 149.4–150.3 °C.

IR (KBr): ν = 3051, 2964, 1615, 1316, 1084, 975, 800 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (d, *J* = 8.3 Hz, 2H), 7.64 (d, *J* = 15.4 Hz, 1H), 7.44 – 7.38 (m, 4H), 7.33 (d, *J* = 8.1 Hz, 2H), 6.81 (d, *J* = 15.4 Hz, 1H), 2.42 (s, 3H), 1.30 (s, 9H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 154.8, 144.2, 141.9, 137.9, 129.9, 129.6, 128.3, 127.6, 126.5, 126.0, 34.9, 31.0, 21.5 ppm.

HRMS (m/z): calcd for $C_{19}H_{23}O_2S$ $[M+H]^+$ 315.1413, found: 315.1415.



(*E*)- 1-Methoxy-4-(2-tosylvinyl)benzene (3l):

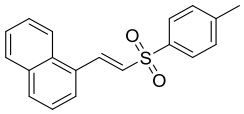
Yield = 34% (29 mg). Yellow solid. M.p. = 79.8–80.3 °C.

IR (KBr): ν = 2923, 1603, 1513, 1316, 1141, 1024, 974, 757 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82 (d, J = 8.3 Hz, 2H), 7.61 (d, J = 15.3 Hz, 1H), 7.42 (d, J = 8.7 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 6.89 (d, J = 8.8 Hz, 2H), 6.70 (d, J = 15.3 Hz, 1H), 3.83 (s, 3H), 2.43 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 161.9, 144.1, 141.7, 138.1, 130.3, 129.8, 127.5, 125.0, 124.7, 114.4, 55.4, 21.6 ppm.

HRMS (m/z): calcd for $C_{16}H_{17}O_3S$ $[M+H]^+$ 289.0893, found: 289.0898.



(*E*)-1-(2-Tosylvinyl)naphthalene (3m):

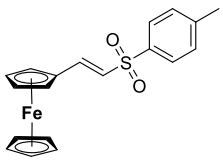
Yield = 58% (54 mg). Yellow oil.

IR (KBr): ν = 3045, 2929, 1595, 1301, 1144, 1084, 792 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.49 (d, J = 15.2 Hz, 1H), 8.15 (d, J = 8.3 Hz, 1H), 7.92 – 7.84 (m, 4H), 7.65 – 7.52 (m, 3H), 7.43 (t, J = 7.7 Hz, 1H), 7.35 (d, J = 8.0 Hz, 2H), 6.95 (d, J = 15.2 Hz, 1H), 2.43 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.4, 138.9, 137.5, 133.6, 131.3, 131.2, 130.0, 129.8, 129.5, 128.8, 127.7, 127.2, 126.4, 125.6, 125.2, 123.0, 21.6 ppm.

HRMS (m/z): calcd for $C_{19}H_{17}O_2S$ $[M+H]^+$ 309.0944, found: 309.0949.



(*E*)-1-Methyl-4-(styrylsulfonyl)ferrocene (3n):

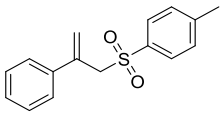
Yield = 50% (55 mg). Brown solid. M.p. = 131.0–132.3 °C.

IR (KBr): ν = 3047, 2918, 1609, 1311, 1084, 963, 722 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.86 – 7.72 (m, 2H), 7.57 (d, J = 15.2 Hz, 1H), 7.32 (d, J = 8.7 Hz, 2H), 6.40 (d, J = 15.1 Hz, 1H), 4.47 – 4.44 (m, 2H), 4.44 – 4.42 (m, 2H), 4.14 (s, 5H), 2.42 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.9, 143.6, 138.4, 129.8, 127.3, 123.0, 76.3, 71.4, 69.7, 68.9, 21.6 ppm.

HRMS (m/z): calcd for $C_{19}H_{19}FeO_2S$ $[M+H]^+$ 367.0450, found: 367.0457.



1-Methyl-4-((2-phenylallyl)sulfonyl)benzene (4a):

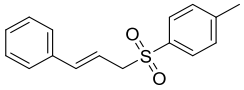
Yield = 25% (20 mg). White solid. M.p. = 93.1–94.7 °C.

IR (KBr): ν = 3058, 2976, 1624, 1446, 1313, 709 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.66 (d, J = 8.3 Hz, 2H), 7.29 – 7.18 (m, 7H), 5.59 (s, 1H), 5.21 (s, 1H), 4.25 (s, 2H), 2.39 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.6, 138.8, 136.5, 135.3, 129.5, 128.6, 128.3, 127.9, 126.2, 121.7, 62.1, 21.6 ppm.

HRMS (m/z): calcd for $C_{16}H_{17}O_2S$ $[M+H]^+$ 273.0944, found: 273.0949.



1-(Cinnamylsulfonyl)-4-methylbenzene (4b):

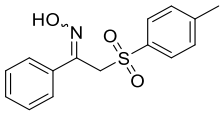
Yield = 22% (18 mg). White solid. M.p. = 113.4–114.8 °C.

IR (KBr): ν = 3022, 2920, 1592, 1489, 1318, 1151, 964, 816 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.78 – 7.74 (m, 2H), 7.35 – 7.27 (m, 7H), 6.39 (d, J = 15.9 Hz, 1H), 6.16 – 6.05 (m, 1H), 3.94 (d, J = 8.6 Hz, 2H), 2.44 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.7, 139.0, 135.8, 135.4, 129.7, 128.6, 128.5, 128.4, 126.6, 115.3, 60.5, 21.6 ppm.

HRMS (m/z): calcd for $C_{16}H_{17}O_2S$ $[M+H]^+$ 273.0944, found: 273.0949.



1-Phenyl-2-tosylethan-1-one oxime (5a):

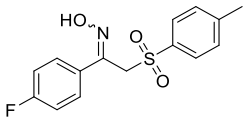
Yield = 73% (63 mg). Light yellow solid. M.p. = 170.8–171.9 °C.

IR (KBr): ν = 3061, 2920, 1593, 1464, 1318, 1151, 739 cm^{-1}.

^{1}H NMR (400 MHz, DMSO-D_6): δ = 11.75 (s, 1H), 7.62 – 7.57 (m, 4H), 7.34 – 7.30 (m, 5H), 4.87 (s, 2H), 2.34 (s, 3H) ppm.

^{13}C NMR (100 MHz, DMSO-D_6): δ = 145.7, 144.4, 136.9, 134.6, 129.5, 129.0, 128.2, 127.9, 126.4, 51.4, 21.1 ppm.

HRMS (m/z): calcd for $C_{15}H_{16}NO_3S$ $[M+H]^+$ 290.0845, found: 290.0851.



1-(4-Fluorophenyl)-2-tosylethan-1-one oxime (5b):

Yield = 61% (56 mg). White solid. M.p. = 194.0–195.6 °C.

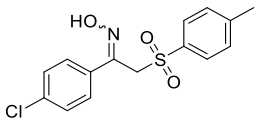
IR (KBr): ν = 3241, 3082, 2960, 2383, 1594, 1317, 1150, 843, 671 cm^{-1}.

^{1}H NMR (400 MHz, MeOH-D_4): δ = 7.68 – 7.61 (m, 4H), 7.31 (d, J = 8.5 Hz, 2H), 7.07 – 7.01 (m, 2H), 4.84 (s, 2H), 2.41 (s, 3H) ppm.

^{19}F NMR (376 MHz, MeOH-D_4): δ = -114.5 (s) ppm.

^{13}C NMR (100 MHz, MeOH-D_4): δ = 164.7 (d, $J_{C\text{-}F}$ = 245.8 Hz), 146.4 (d, $J_{C\text{-}F}$ = 10.7 Hz), 138.0, 132.4 (d, $J_{C\text{-}F}$ = 3.3 Hz), 130.5, 129.8, 129.8, 129.5, 116.1 (d, $J_{C\text{-}F}$ = 21.9 Hz), 52.9, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{15}FNO_3S$ $[M+H]^+$ 308.0751, found: 308.0757.



1-(4-Chlorophenyl)-2-tosylethan-1-one oxime (5c):

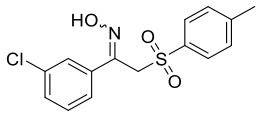
Yield = 49% (48 mg). White solid. M.p. = 167.4–169.0 °C.

IR (KBr): ν = 3289, 2928, 2405, 1593, 1317, 953, 767 cm^{-1}.

^{1}H NMR (400 MHz, MeOH-D_4): δ = 7.64 (d, J = 8.3 Hz, 2H), 7.61 – 7.57 (m, 2H), 7.32 – 7.27 (m, 4H), 4.83 (s, 2H), 2.40 (s, 3H) ppm.

^{13}C NMR (100 MHz, MeOH-D_4): δ = 157.0, 146.4, 137.9, 136.0, 134.7, 130.6, 129.5, 129.4, 129.2, 52.7, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{15}ClNO_3S$ $[M+H]^+$ 324.0456, found: 324.0461.



1-(3-Chlorophenyl)-2-tosylethan-1-one oxime (5d):

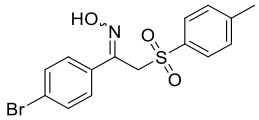
Yield = 38% (37 mg). Light yellow solid. M.p. = 161.1–162.6 °C.

IR (KBr): ν = 3045, 2923, 2409, 1595, 1449, 1304, 1143, 973, 810, 747 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.56 (s, 1H, NOH), 7.74 – 7.68 (m, 2H), 7.55 – 7.50 (m, 2H), 7.35 – 7.33 (m, 1H), 7.30 – 7.28 (m, 1H), 7.26 – 7.23 (m, 2H), 4.70 (s, 2H), 2.40 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.7, 145.1, 136.2, 135.4, 134.6, 129.8, 129.7, 129.6, 128.4, 126.5, 124.9, 52.5, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{15}ClNO_3S$ $[M+H]^+$ 324.0456, found: 324.0461.



1-(4-Bromophenyl)-2-tosylethan-1-one oxime (5f):

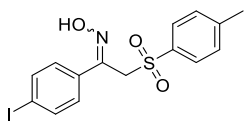
Yield = 61% (67 mg). Light yellow solid. M.p. = 172.3–174.1 °C.

IR (KBr): ν = 3292, 2929, 2413, 1587, 1409, 1317, 954, 831 cm^{-1}.

^{1}H NMR (400 MHz, MeOH-D_4): δ = 7.67 – 7.62 (m, 2H), 7.55 – 7.50 (m, 2H), 7.48 – 7.43 (m, 2H), 7.31 (d, J = 8.4 Hz, 2H), 4.84 (s, 2H), 2.42 (s, 3H) ppm.

^{13}C NMR (100 MHz, MeOH-D_4): δ = 146.5, 146.5, 137.9, 135.1, 132.4, 130.6, 129.5, 129.4, 124.2, 52.6, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{15}{}^{79}BrNO_3S$ $[M+H]^+$ 367.9951, found: 367.9956.



1-(4-Iodophenyl)-2-tosylethan-1-one oxime (5g):

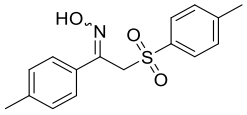
Yield = 59% (74 mg). Light yellow solid. M.p. = 186.4–187.1 °C.

IR (KBr): ν = 3045, 2921, 1917, 1607, 1314, 1141, 975, 795 cm^{-1}.

^{1}H NMR (400 MHz, MeOH-D_4): δ = 7.66 – 7.61 (m, 4H), 7.38 – 7.34 (m, 2H), 7.31 – 7.27 (m, 2H), 4.82 (s, 2H), 2.42 (s, 3H) ppm.

^{13}C NMR (100 MHz, MeOH-D_4): δ = 146.7, 146.5, 138.5, 137.8, 135.6, 130.6, 129.5, 129.4, 95.8, 52.5, 21.6 ppm.

HRMS (m/z): calcd for $C_{15}H_{15}INO_3S$ $[M+H]^+$ 415.9812, found: 415.9817.



1-(*p*-Tolyl)-2-tosylethan-1-one oxime (5h):

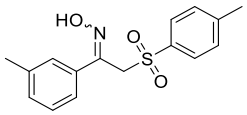
Yield = 64% (58 mg). Light yellow solid. M.p. = 151.3–152.0 ºC.

IR (KBr): ν = 3265, 3005, 2919, 2404, 1595, 1405, 1320, 1163, 1053, 947, 772 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.92 (s, 1H, NOH), 7.72 – 7.67 (m, 2H), 7.51 – 7.45 (m, 2H), 7.24 – 7.19 (m, 2H), 7.17 – 7.12 (m, 2H), 4.71 (s, 2H), 2.36 (s, 6H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 147.6, 144.8, 140.0, 136.4, 130.7, 129.3, 129.2, 128.4, 126.4, 52.7, 21.5, 21.2 ppm.

HRMS (m/z): calcd for $C_{16}H_{18}NO_3S$ $[M+H]^+$ 304.1002, found: 304.1007.



1-(*m*-Tolyl)-2-tosylethan-1-one oxime (5i):

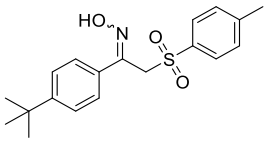
Yield = 66% (60 mg). Light yellow solid. M.p. = 131.8–132.6 ºC.

IR (KBr): ν = 3045, 2389, 1599, 1407, 1068, 955 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.85 (s, 1H, NOH), 7.69 (d, J = 8.2 Hz, 2H), 7.39 (d, J = 8.0 Hz, 1H), 7.33 (s, 1H), 7.24 – 7.18 (m, 4H), 4.72 (s, 2H), 2.36 (s, 3H), 2.32 (s, 3H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 144.8, 138.1, 136.4, 133.5, 130.5, 130.0, 129.4, 128.4, 128.4, 127.1, 123.7, 52.8, 21.5, 21.3 ppm.

HRMS (m/z): calcd for $C_{16}H_{18}NO_3S$ $[M+H]^+$ 304.1002, found: 304.1007.



1-(4-(*Tert*-butyl)phenyl)-2-tosylethan-1-one oxime (5j):

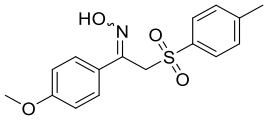
Yield = 48% (50 mg). Light yellow solid. M.p. = 181.7–183.6 °C.

IR (KBr): ν = 3056, 2962, 2389, 1681, 1568, 1506, 1093, 798 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.67 (s, 1H, NOH), 7.72 – 7.67 (m, 2H), 7.55 – 7.49 (m, 2H), 7.37 – 7.33 (m, 2H), 7.23 – 7.18 (m, 2H), 4.72 (s, 2H), 2.36 (s, 3H), 1.32 (s, 9H) ppm.

^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.1, 147.6, 144.7, 136.5, 130.7, 129.4, 128.4, 126.3, 125.5, 52.6, 34.7, 31.1, 21.6 ppm.

HRMS (m/z): calcd for $C_{19}H_{24}NO_3S$ $[M+H]^+$ 346.1471, found: 346.1477.



1-(4-Methoxyphenyl)-2-tosylethan-1-one oxime (5k):

Yield = 32% (31 mg). Yellow solid. M.p. = 155.9–157.9 °C.

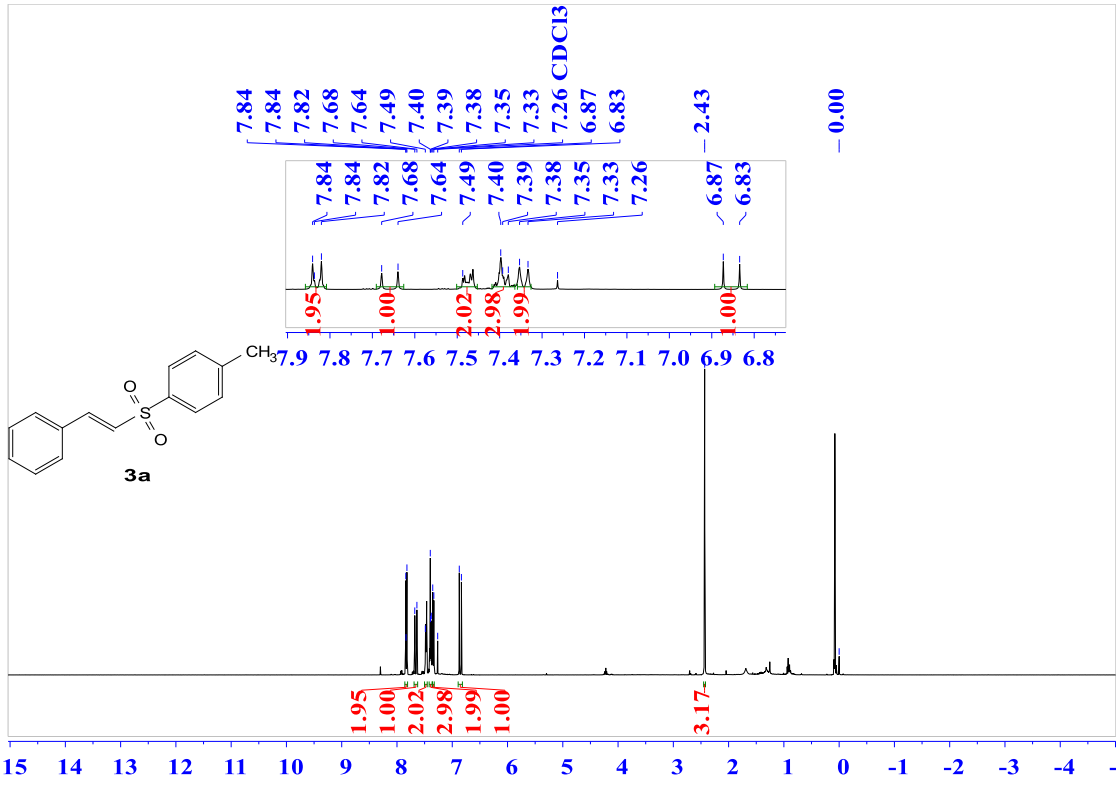
IR (KBr): ν = 3319, 2989, 2845, 1605, 1514, 1316, 1250, 945, 813 cm^{-1}.

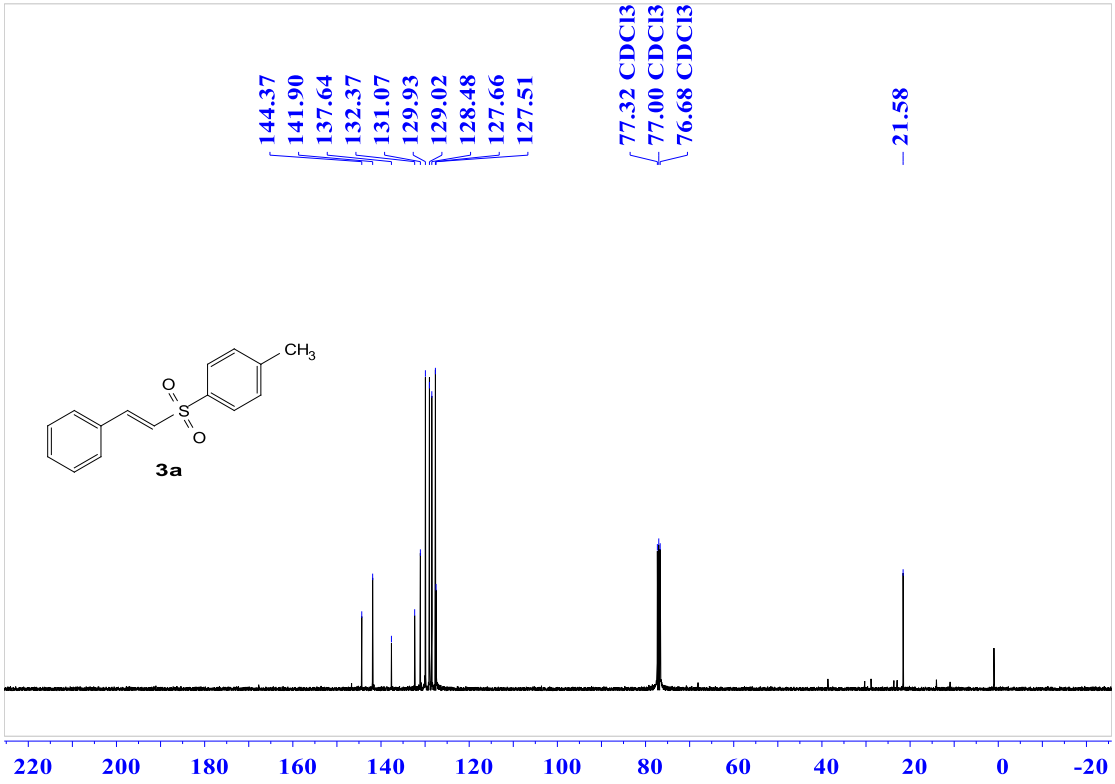
^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.62 (s, 1H, NOH), 7.70 (d, J = 8.3 Hz, 2H), 7.56 (d, J = 8.7 Hz, 2H), 7.23 (d, J = 8.0 Hz, 2H), 6.87 (d, J = 8.9 Hz, 2H), 4.70 (s, 2H), 3.83 (s, 3H), 2.38 (s, 3H) ppm.

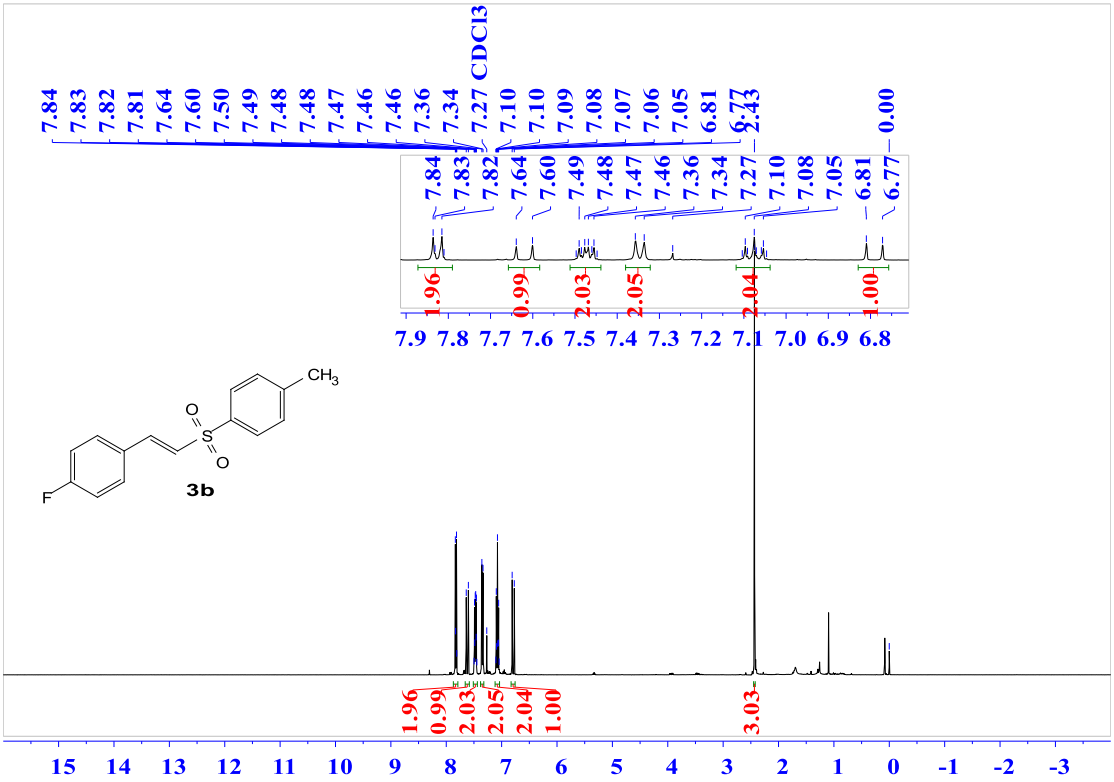
^{13}C NMR (100 MHz, $CDCl_3$): δ = 160.9, 147.3, 144.8, 136.4, 129.4, 128.4, 128.0, 126.1, 113.9, 55.3, 52.6, 21.6 ppm.

HRMS (m/z): calcd for $C_{16}H_{18}NO_4S$ $[M+H]^+$ 320.0951, found: 320.0955.

9. The ^{1}H , ^{19}F, ^{13}C spectra of products



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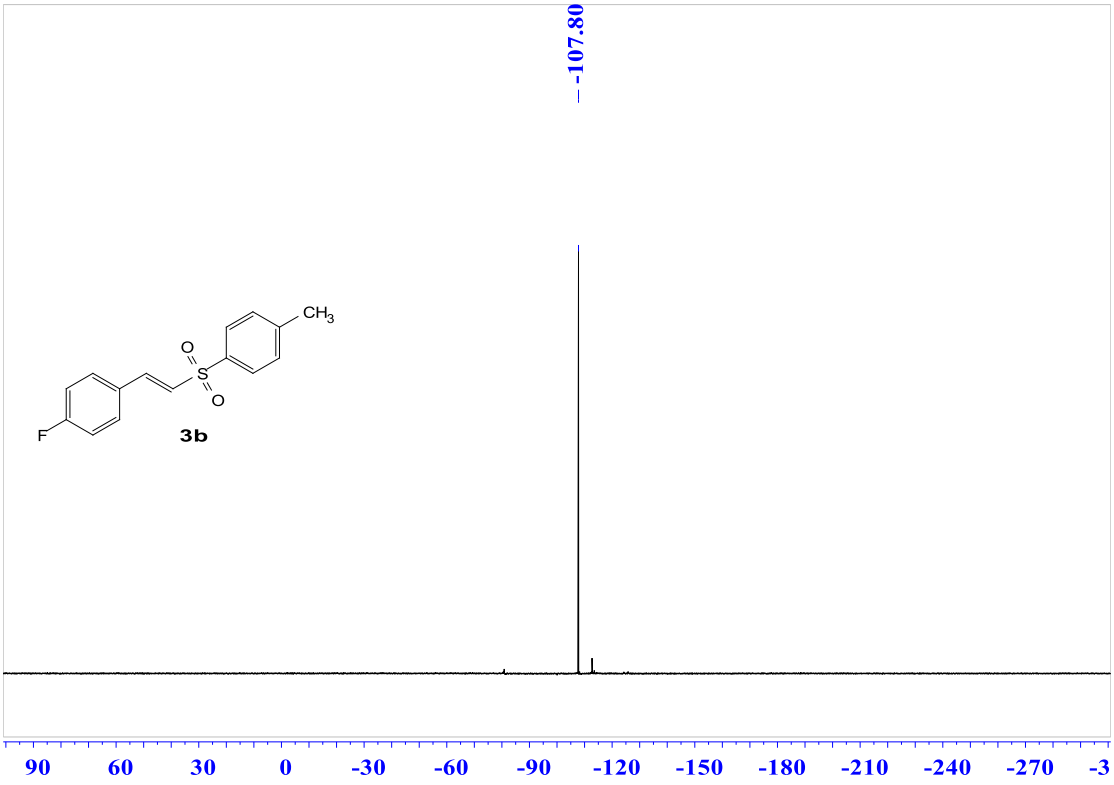
1.96 0.99 2.03 2.05 2.04 1.00
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8
CH3
O
S
O
F
3b
1.96 0.99 2.03 2.05 2.04 1.00 3.03
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3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O
F
3b
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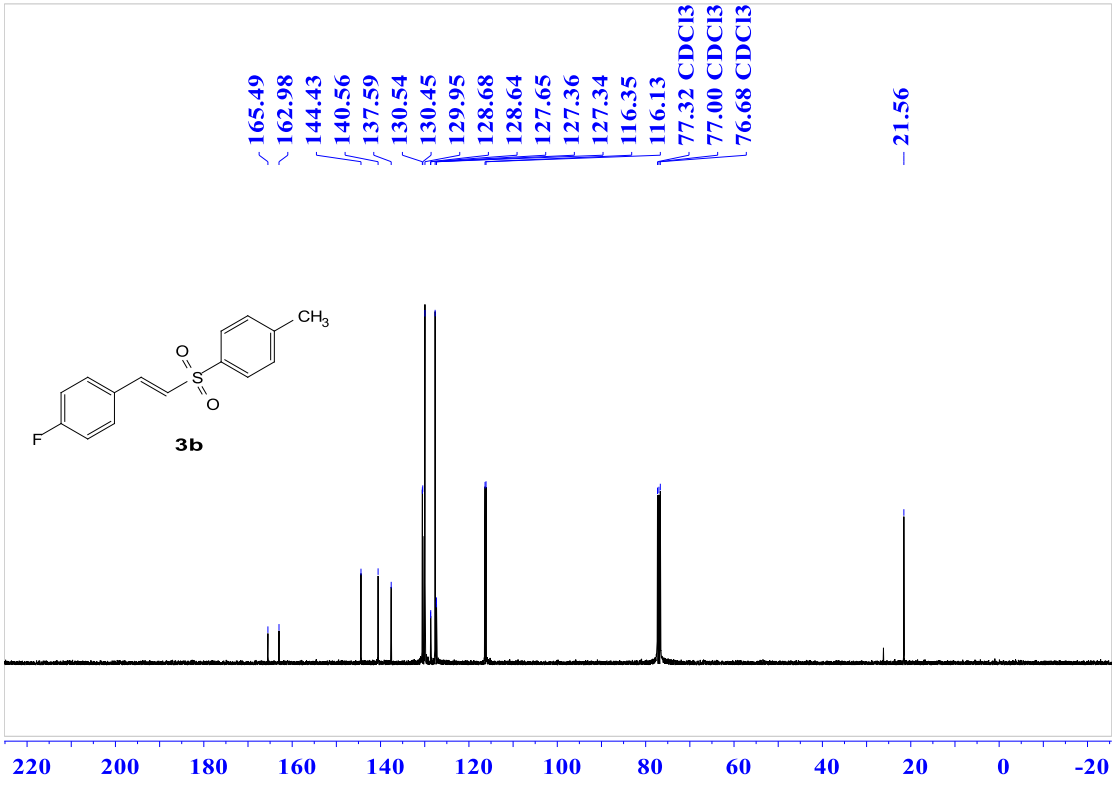
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129.95
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128.64
127.65
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127.34
116.35
116.13
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
21.56
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O
F
3b
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200
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120
100
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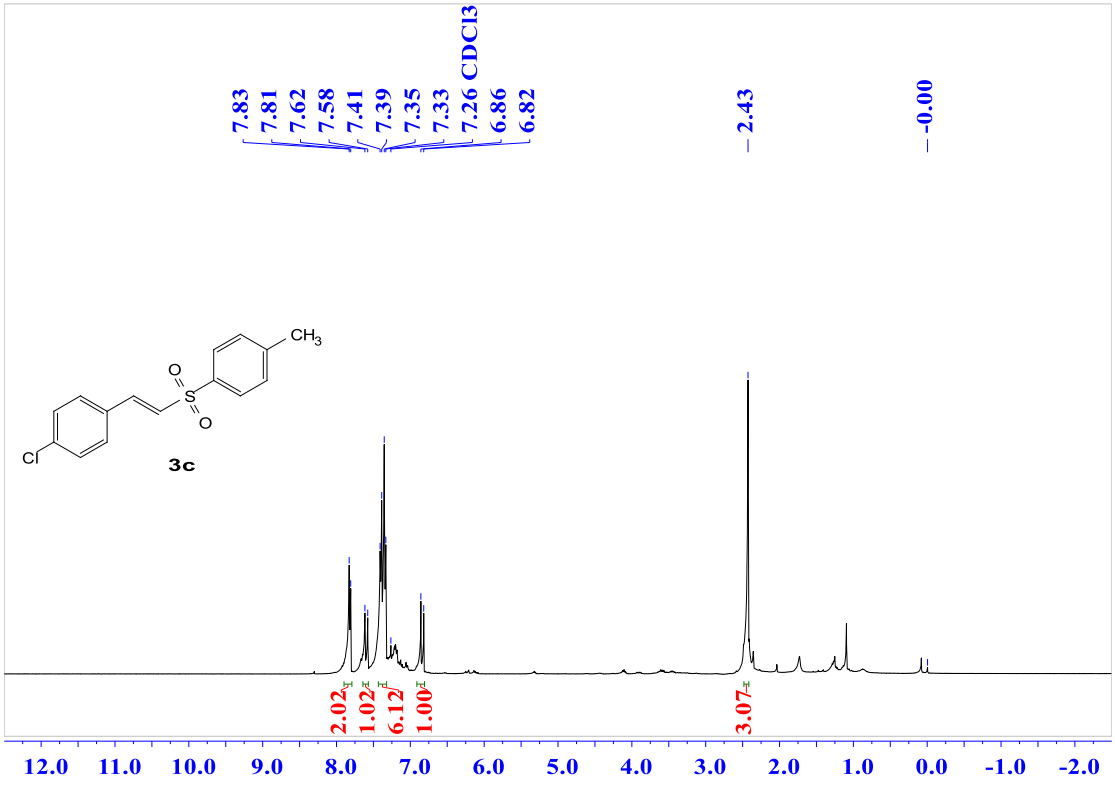
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7.33
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O
S
O
Cl
3c
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1.02
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1.00
3.07
12.0
11.0
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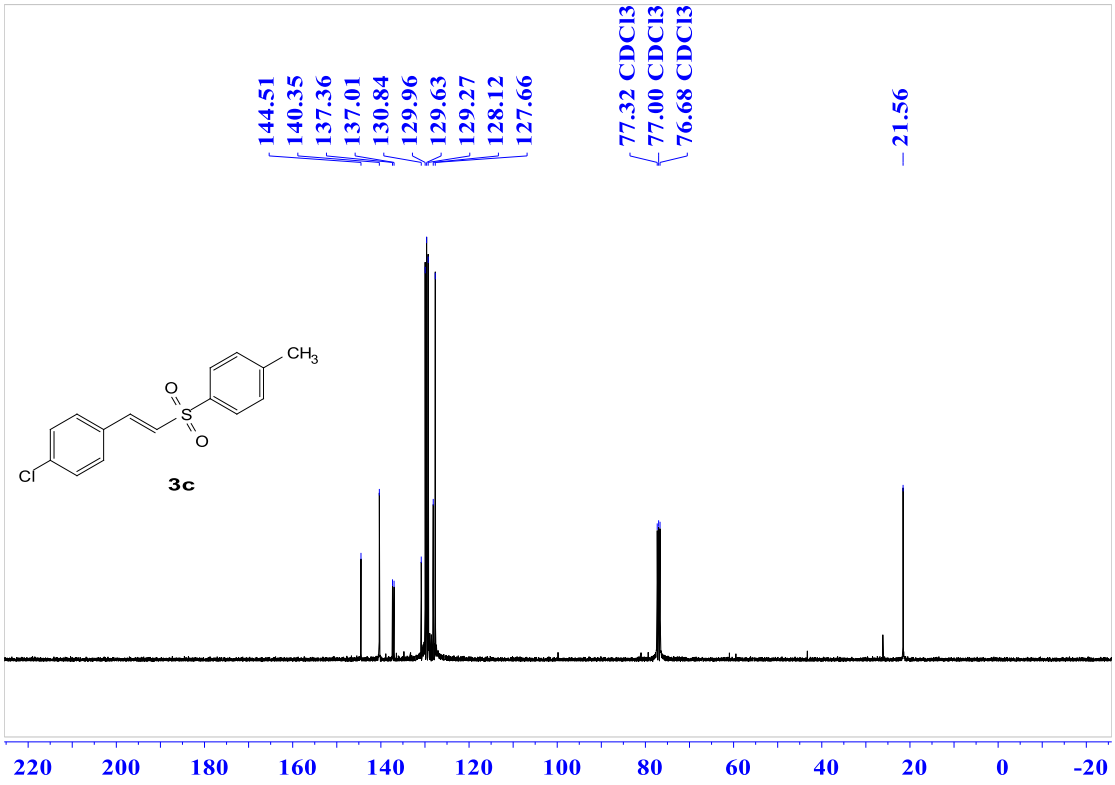
-2.0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129.96
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128.12
127.66
77.32 CDCl3
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76.68 CDCl3
21.56
CH3
O
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O
Cl
3c
220
200
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40
20
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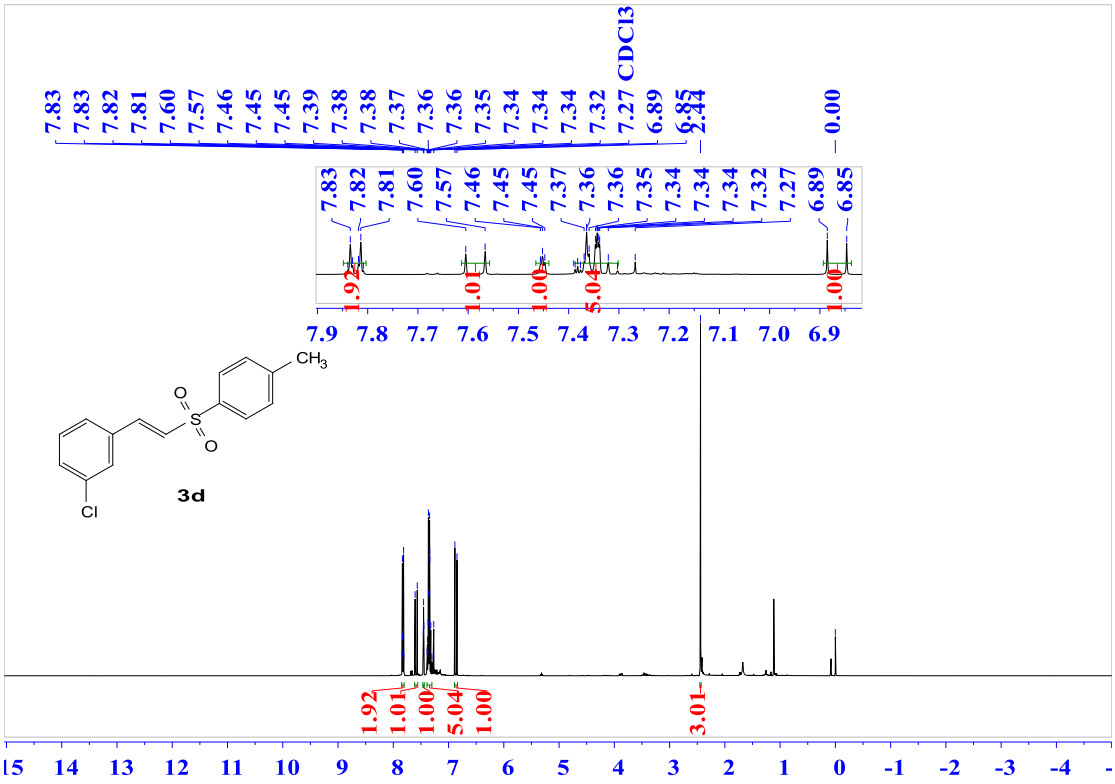
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3.01
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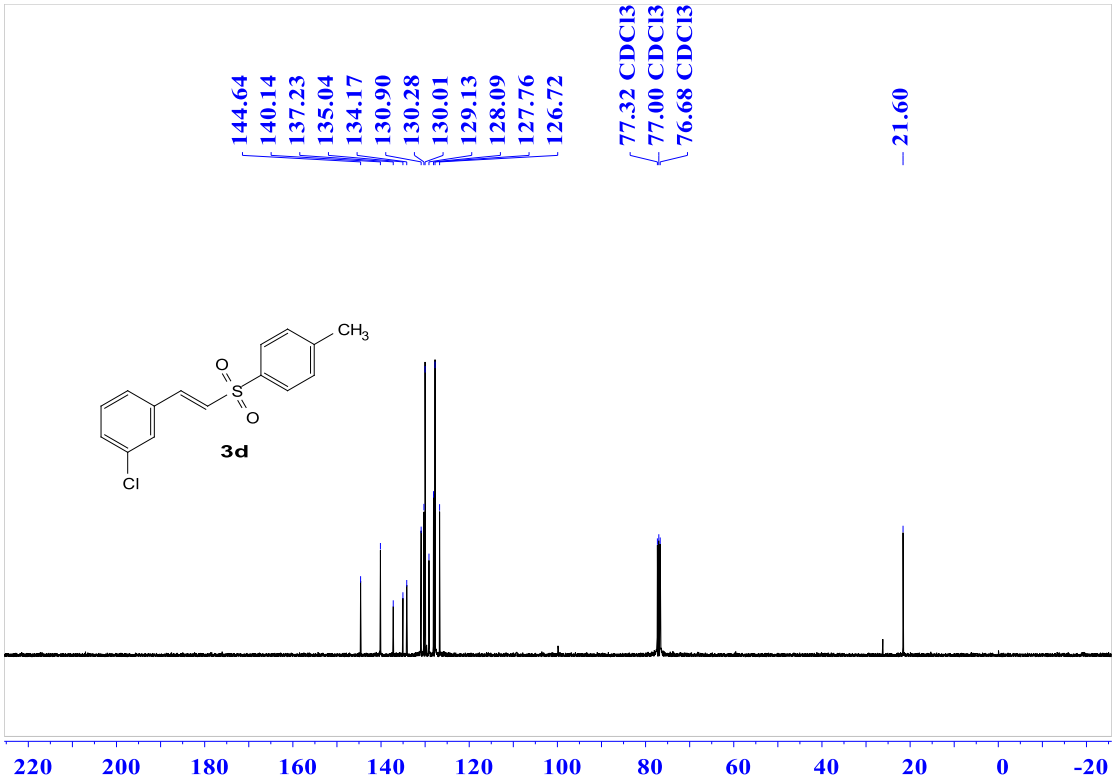
S
O
Cl
3d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129.13
128.09
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77.00 CDCl3
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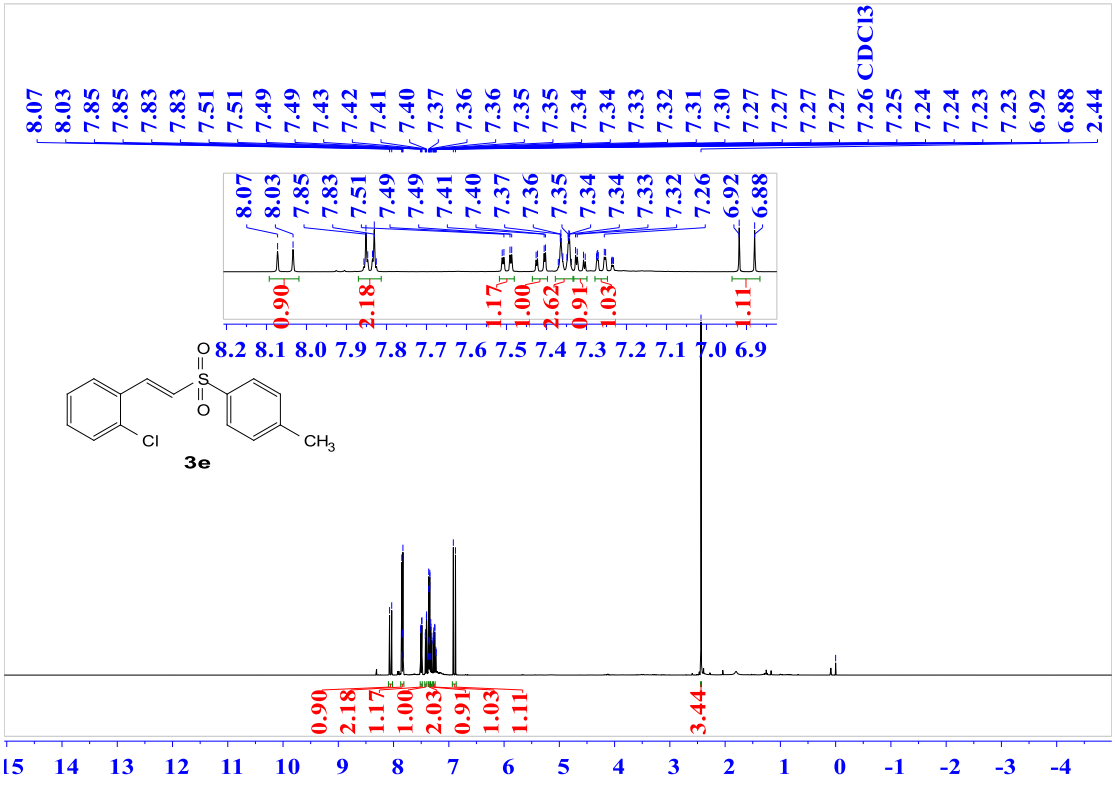
21.60
CH3
O
S
O
Cl
3d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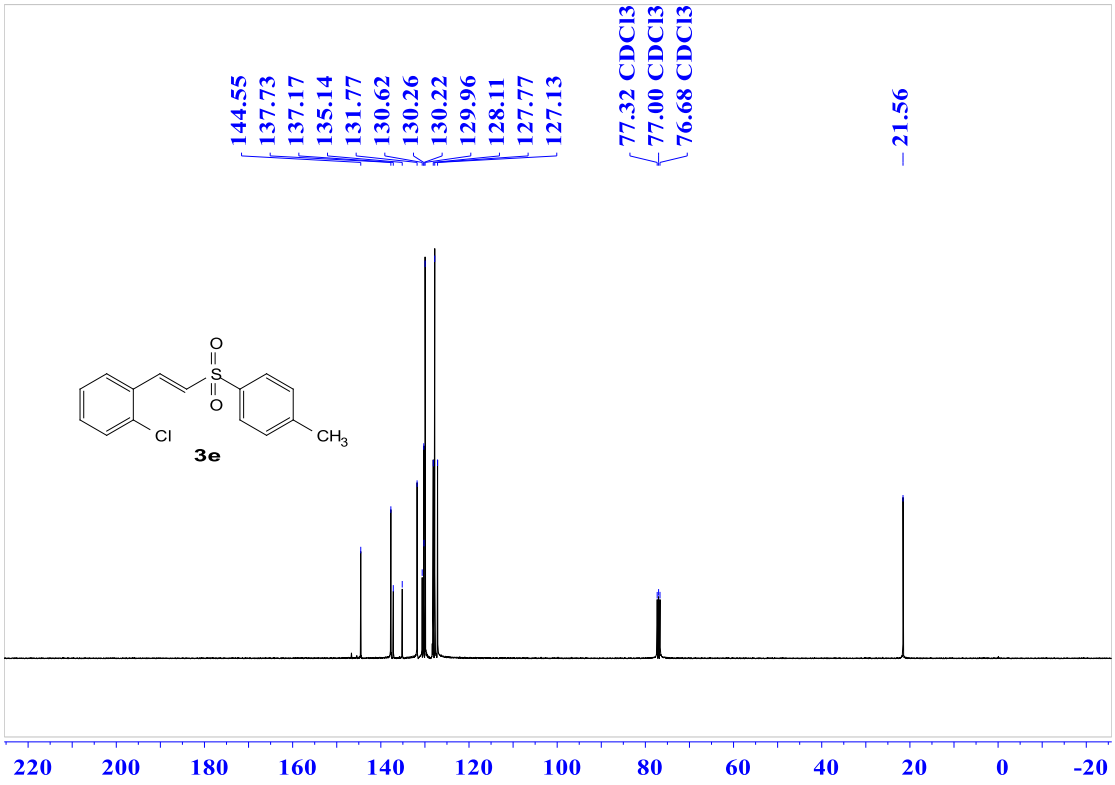
0.90 2.18 1.17 1.00 2.62 0.91 1.03 1.11
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
O
S
O
Cl
CH3
3e
0.90 2.18 1.17 1.00 2.03 0.91 1.03 1.11 3.44
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4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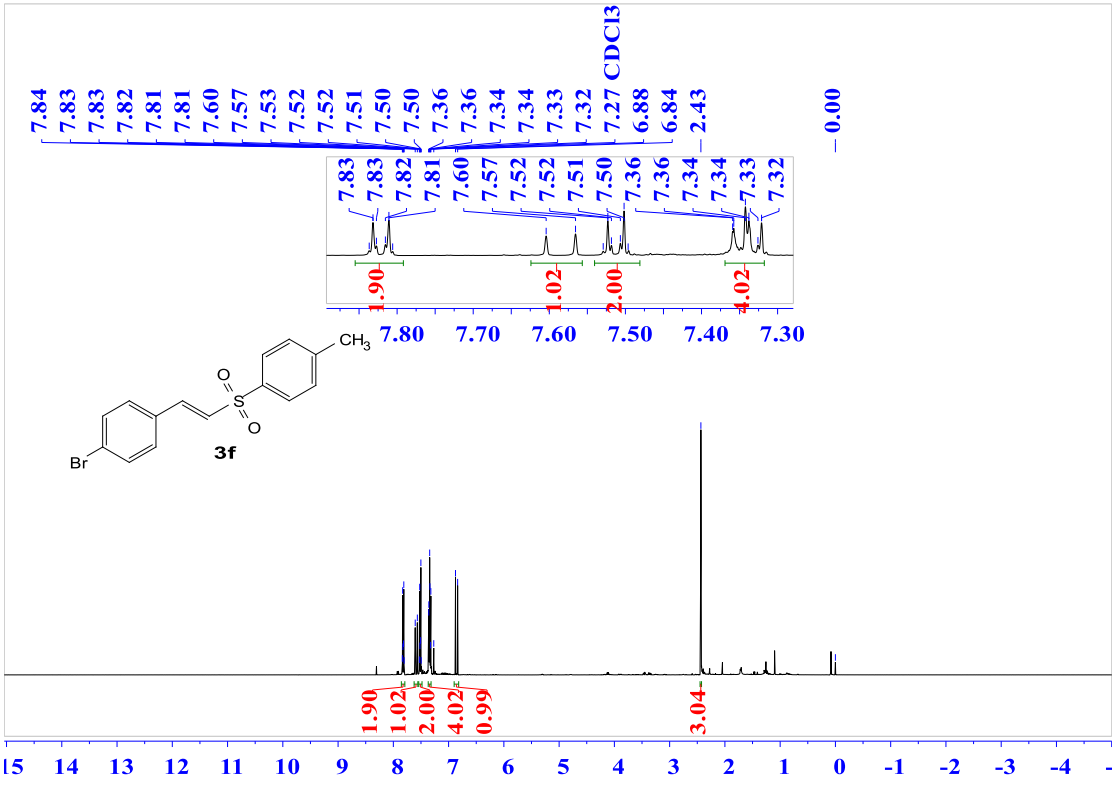
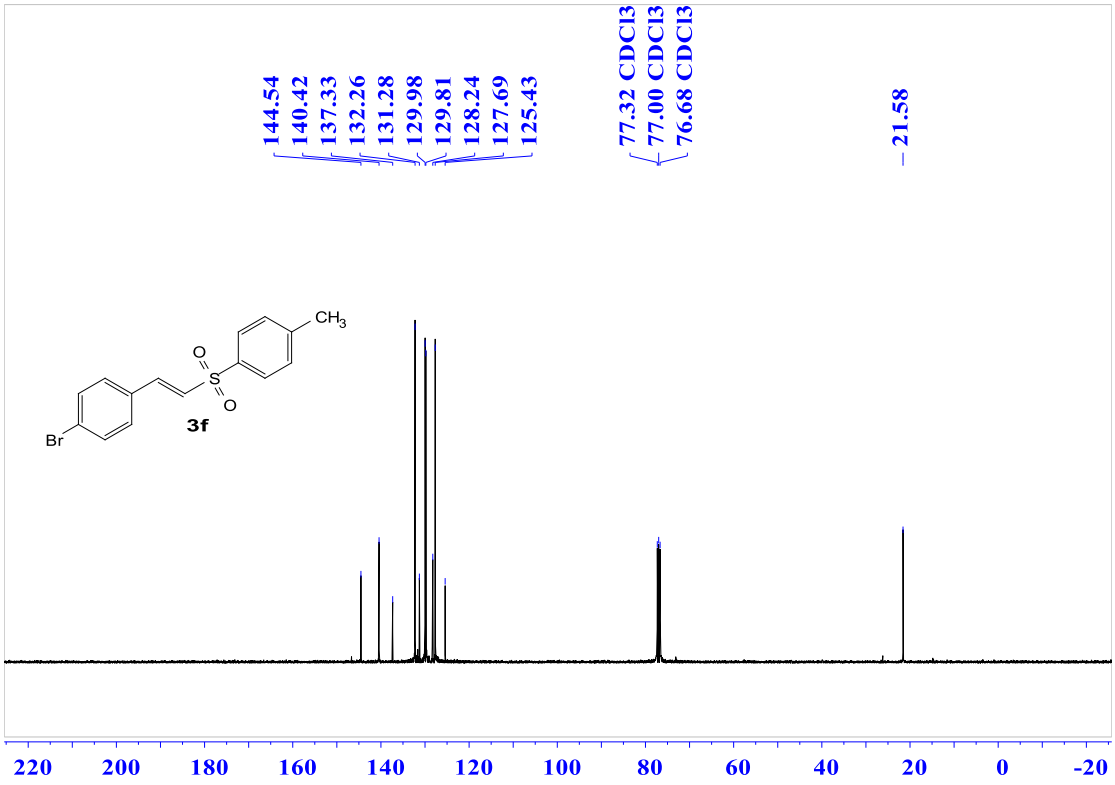
77.32 CDCl3 77.00 CDCl3 76.68 CDCl3
21.56
O
S
O
Cl
CH3
3e
220 200 180 160 140 120 100 80 60 40 20 0 -20


7.84 7.83 7.83 7.82 7.81 7.81 7.60 7.57 7.53 7.52 7.52 7.51 7.50 7.50 7.36 7.36 7.34 7.34 7.33 7.32 7.27 CDCl3 6.88 6.84 2.43 0.00
1.90 1.02 2.00 4.02
7.80 7.70 7.60 7.50 7.40 7.30
CH3
O
S
O
Br
3f
1.90 1.02 2.00 4.02 0.99 3.04
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4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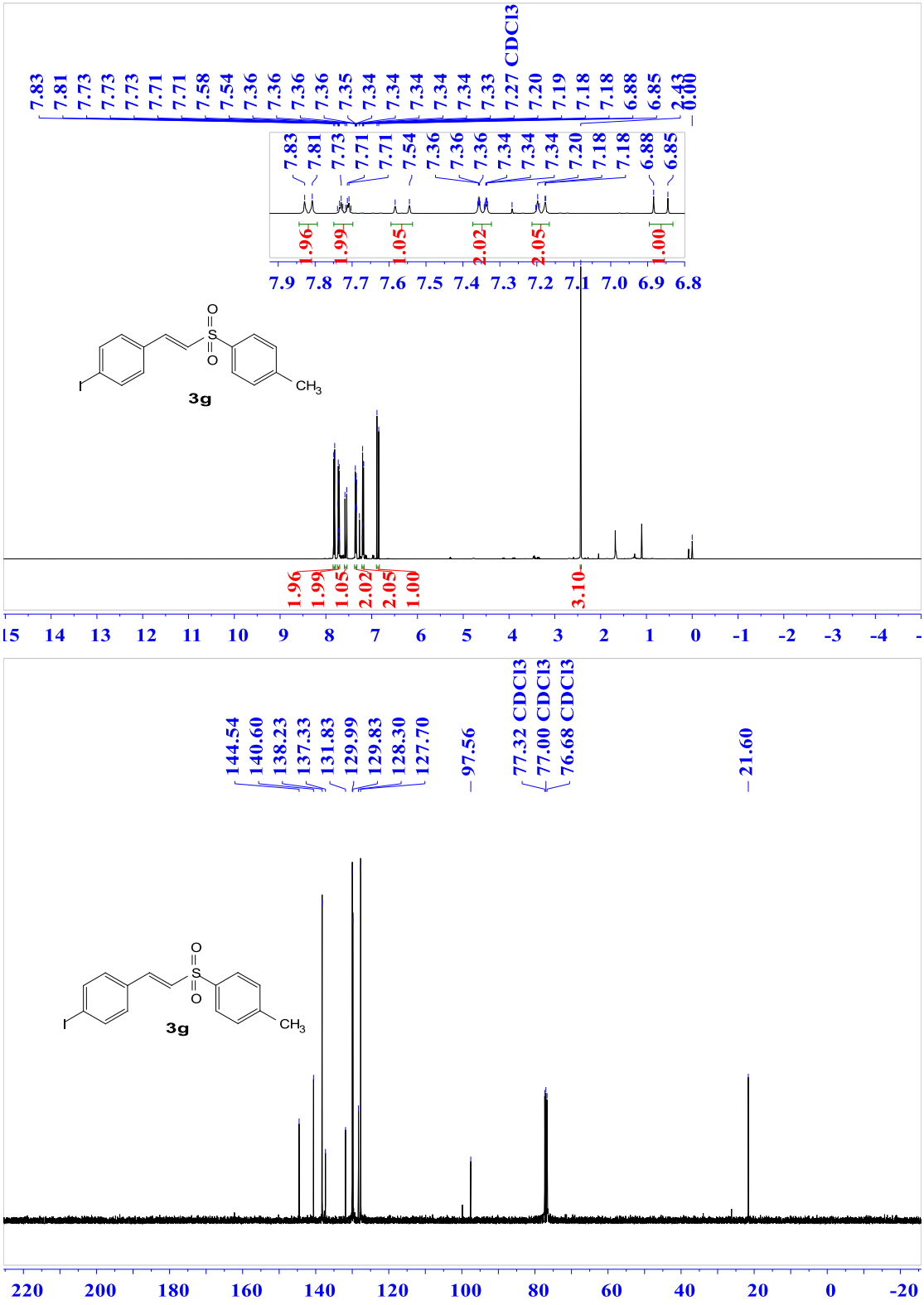
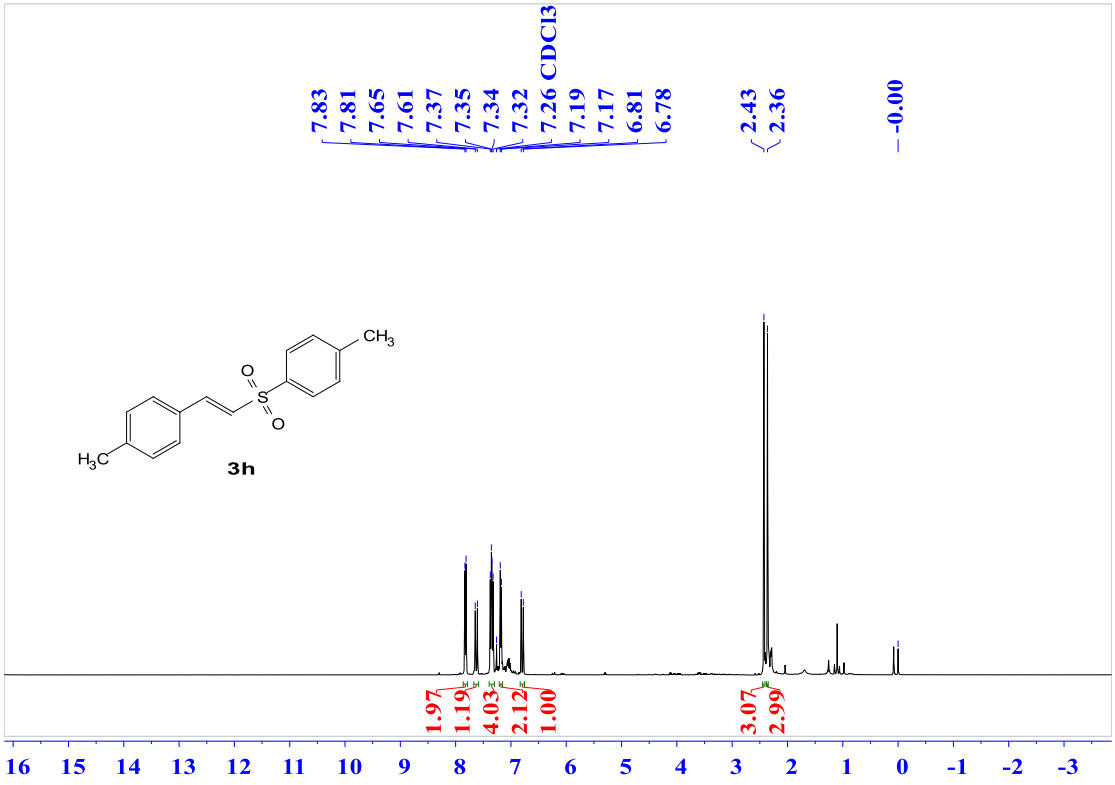
21.58
CH3
O
S
O
Br
3f
220 200 180 160 140 120 100 80 60 40 20 0 -20


7.83 7.81 7.73 7.73 7.73 7.71 7.71 7.58 7.54 7.36 7.36 7.36 7.36 7.35 7.34 7.34 7.34 7.34 7.34 7.33 7.27 CDCl3 7.20 7.19 7.18 7.18 6.88 6.85 2.43 0.00
1.96 1.99 1.05 2.02 2.05 1.00
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8
3g
1.96 1.99 1.05 2.02 2.05 1.00 3.10
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
144.54 140.60 138.23 137.33 131.83 129.99 129.83 128.30 127.70 97.56 77.32 CDCl3 77.00 CDCl3 76.68 CDCl3 21.60
3g
220 200 180 160 140 120 100 80 60 40 20 0 -20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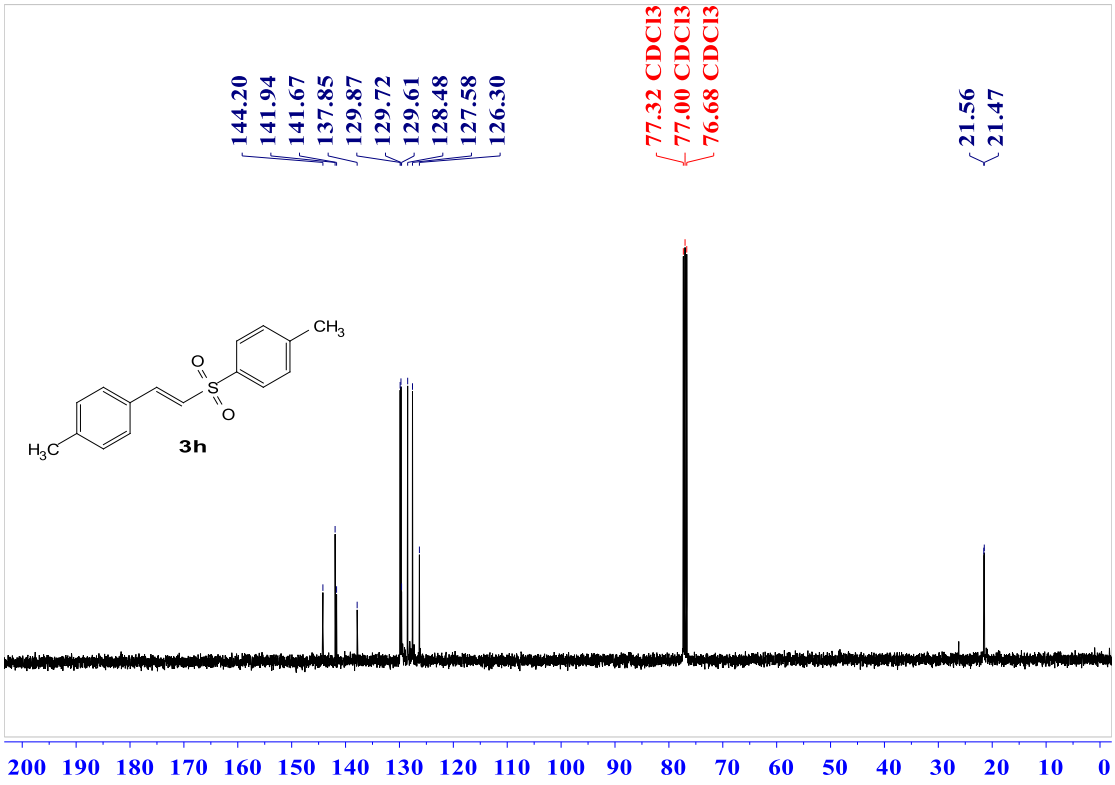
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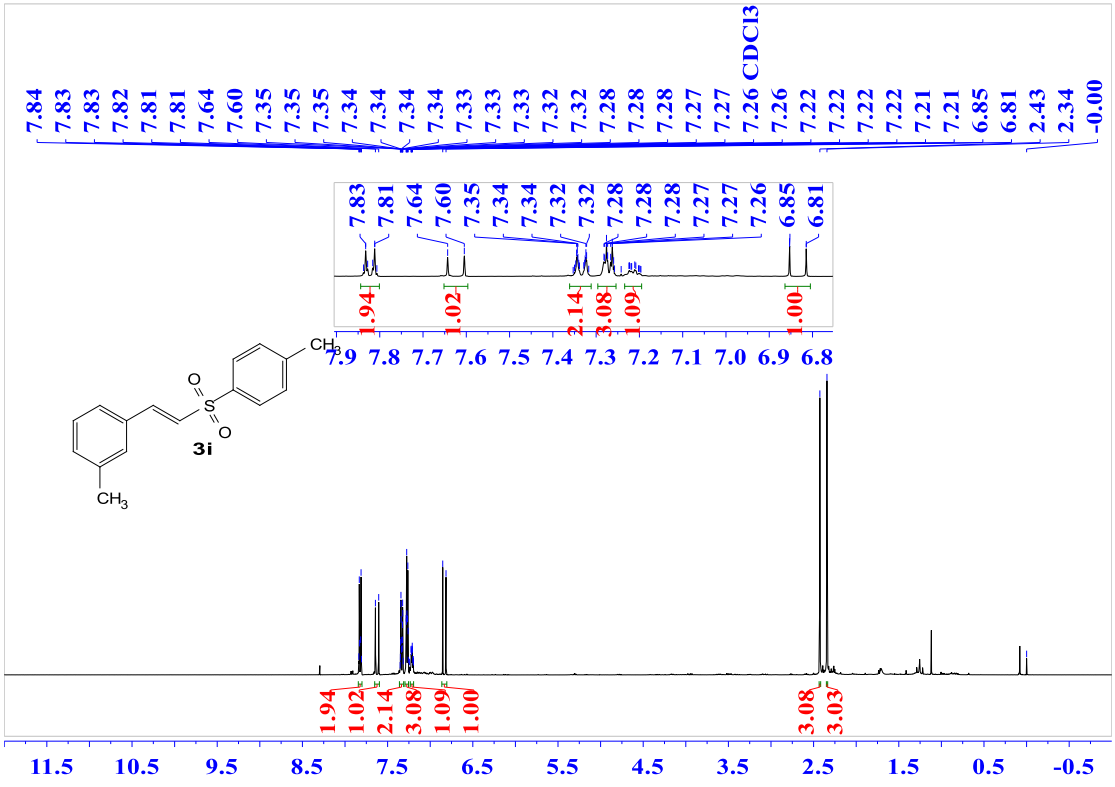
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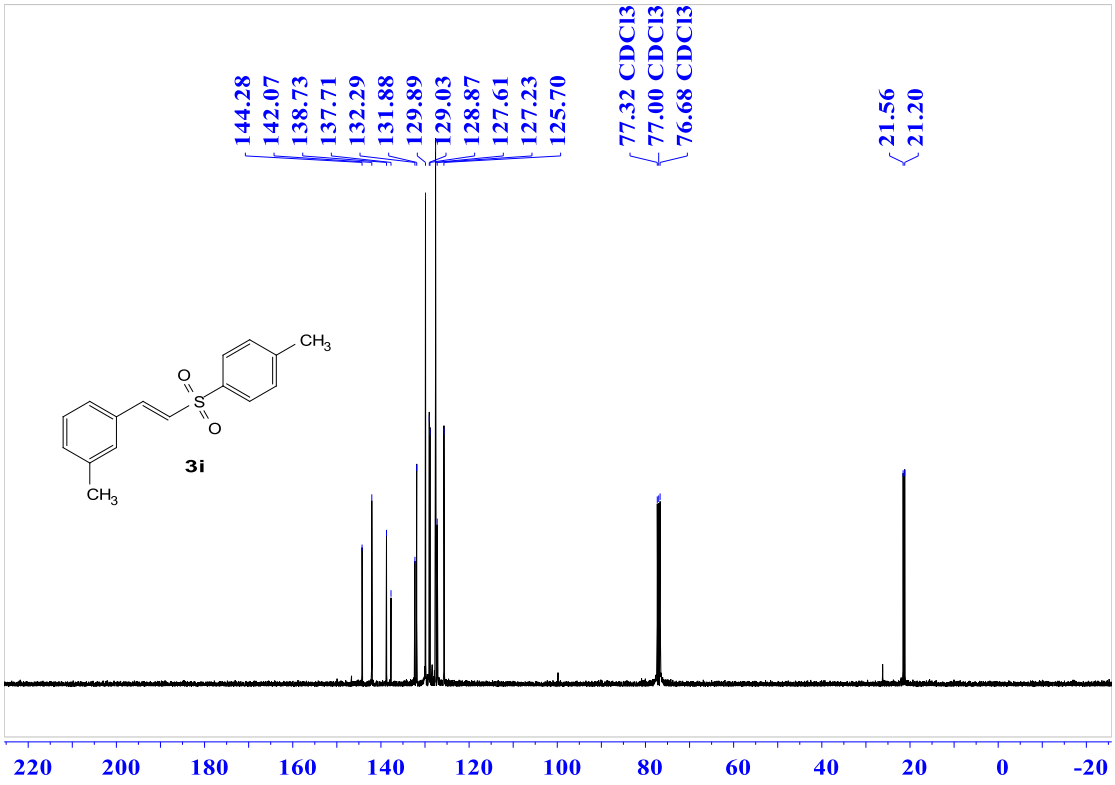
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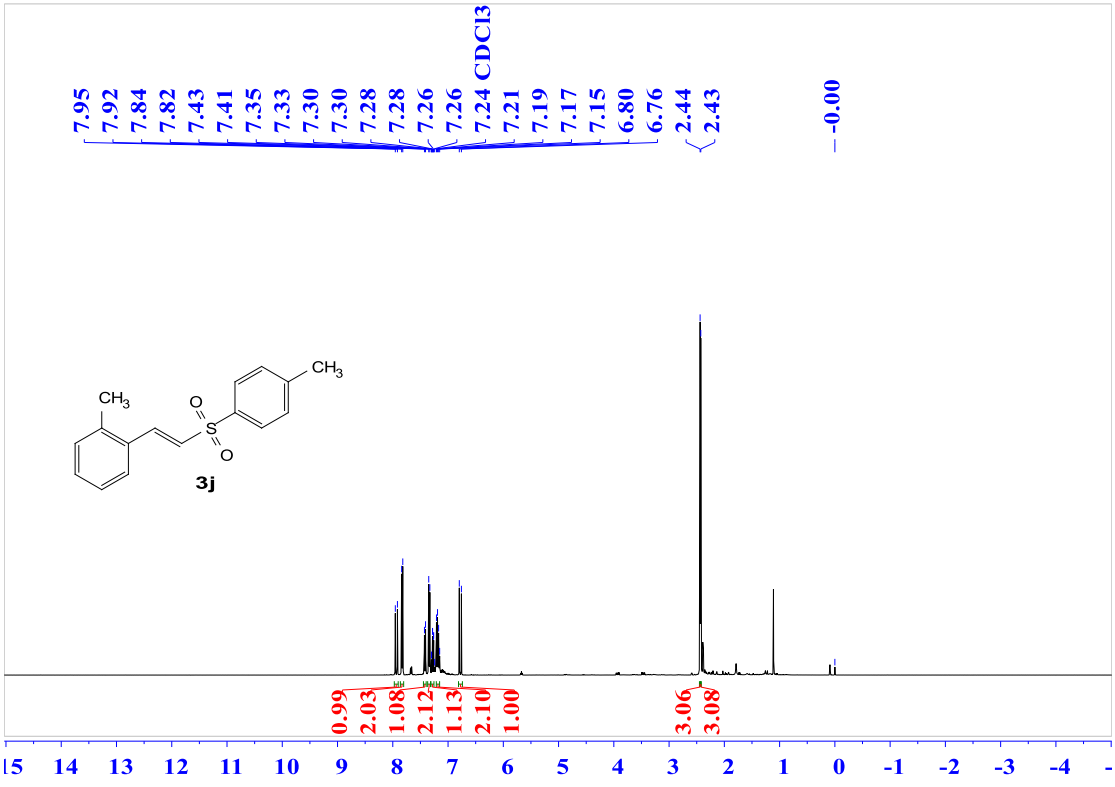
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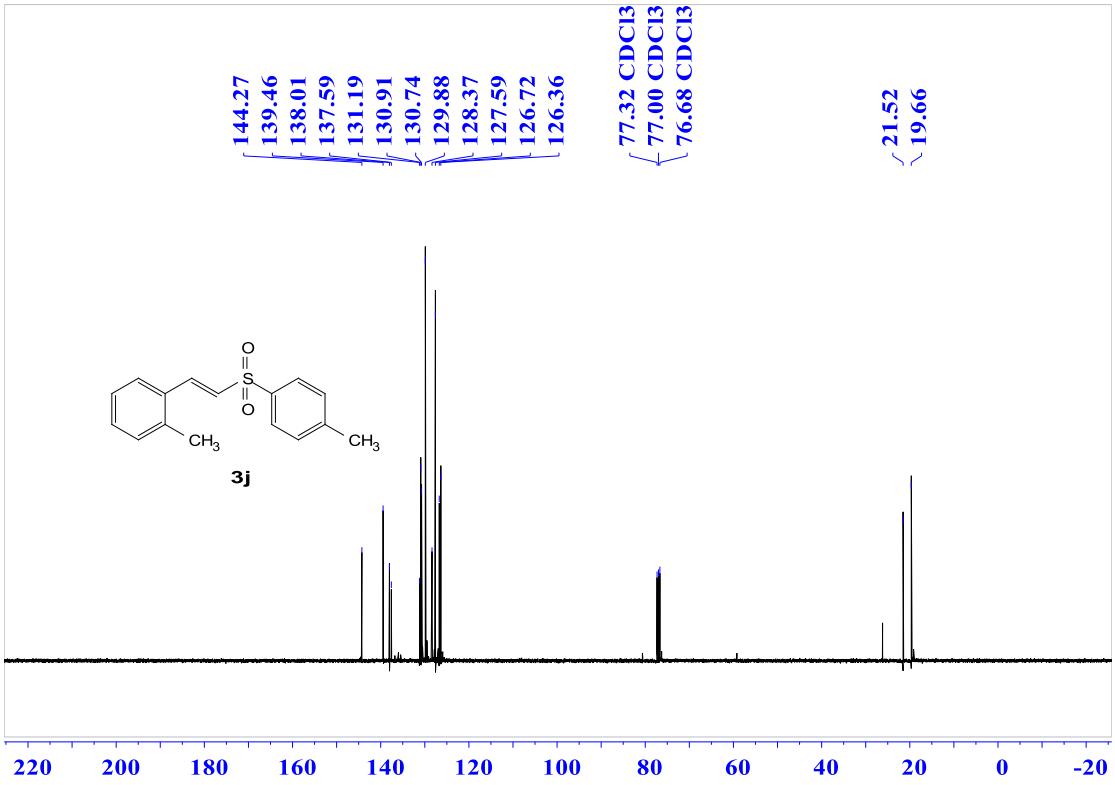
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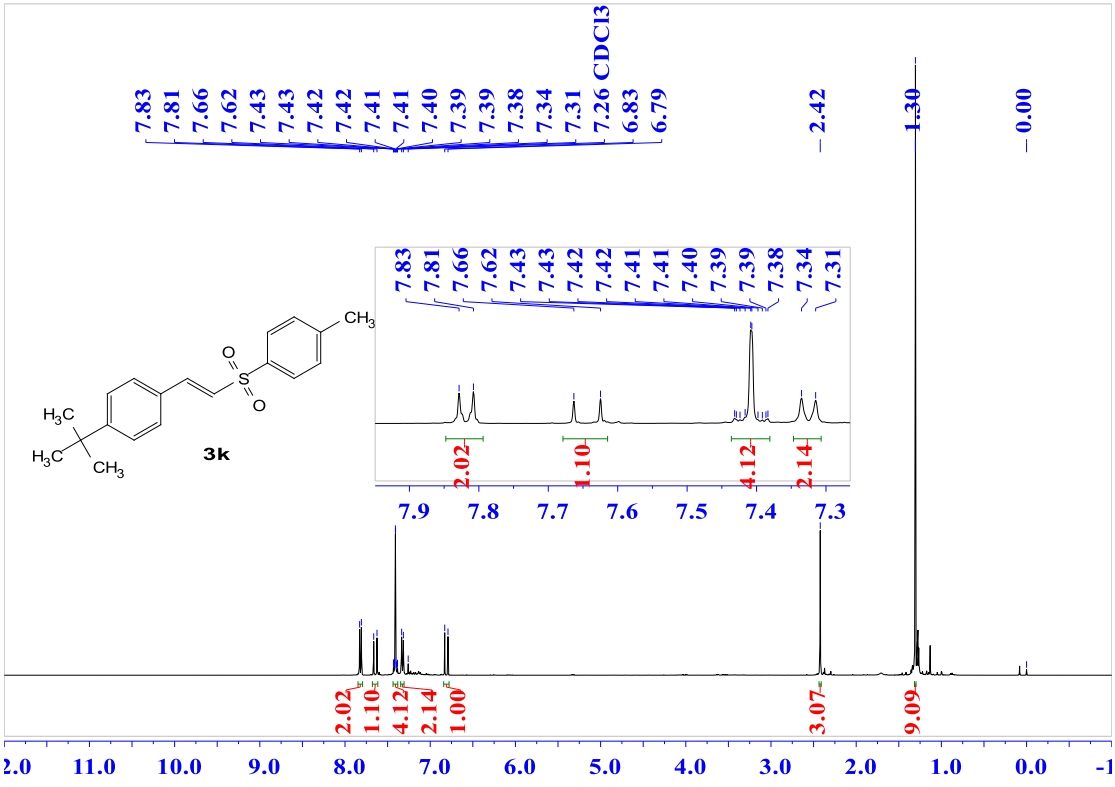
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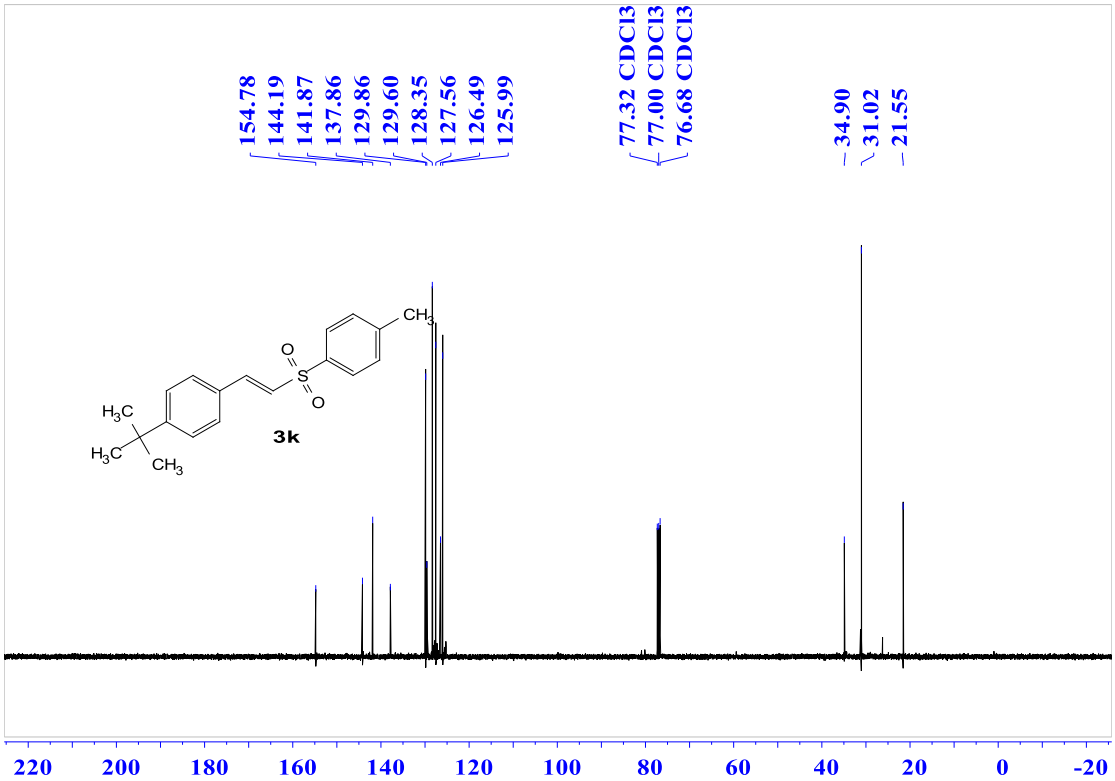
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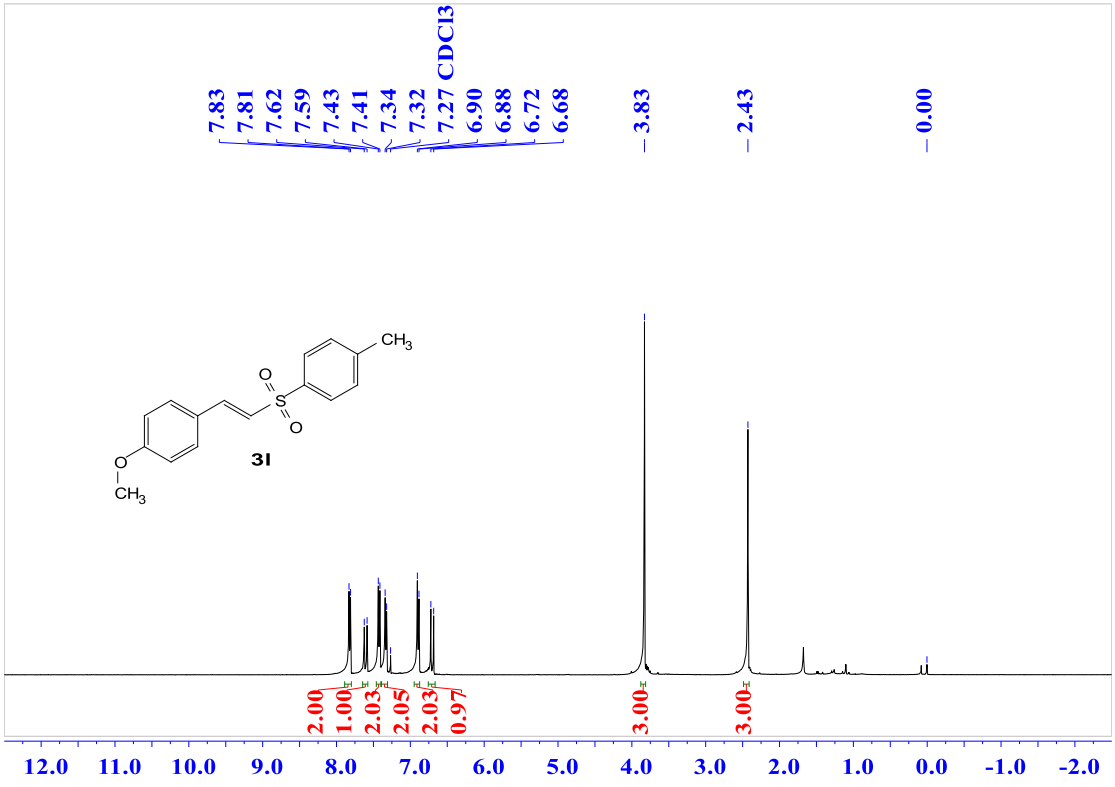
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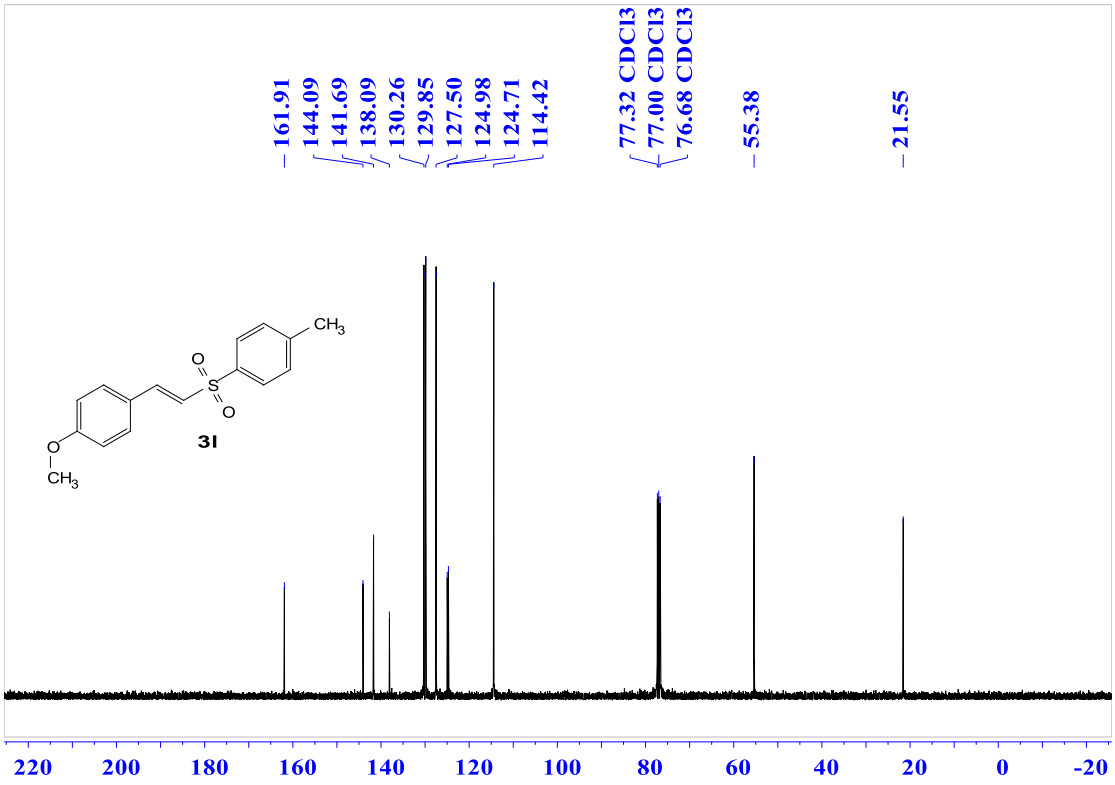
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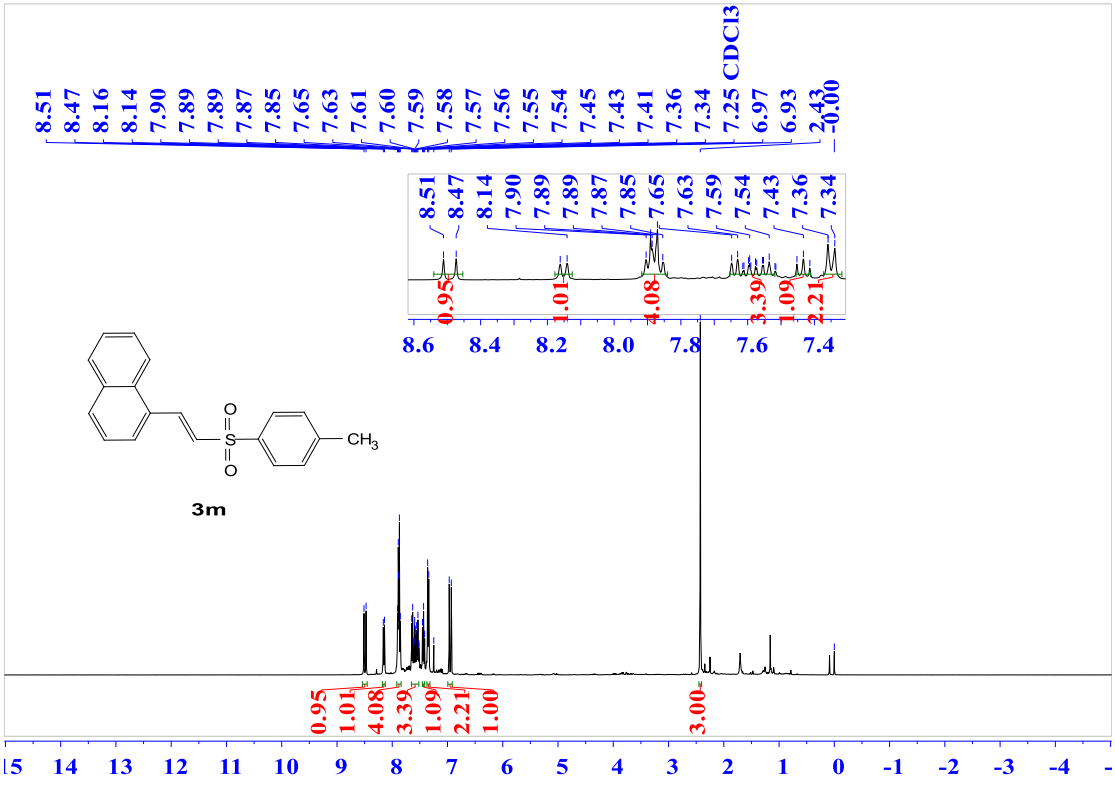
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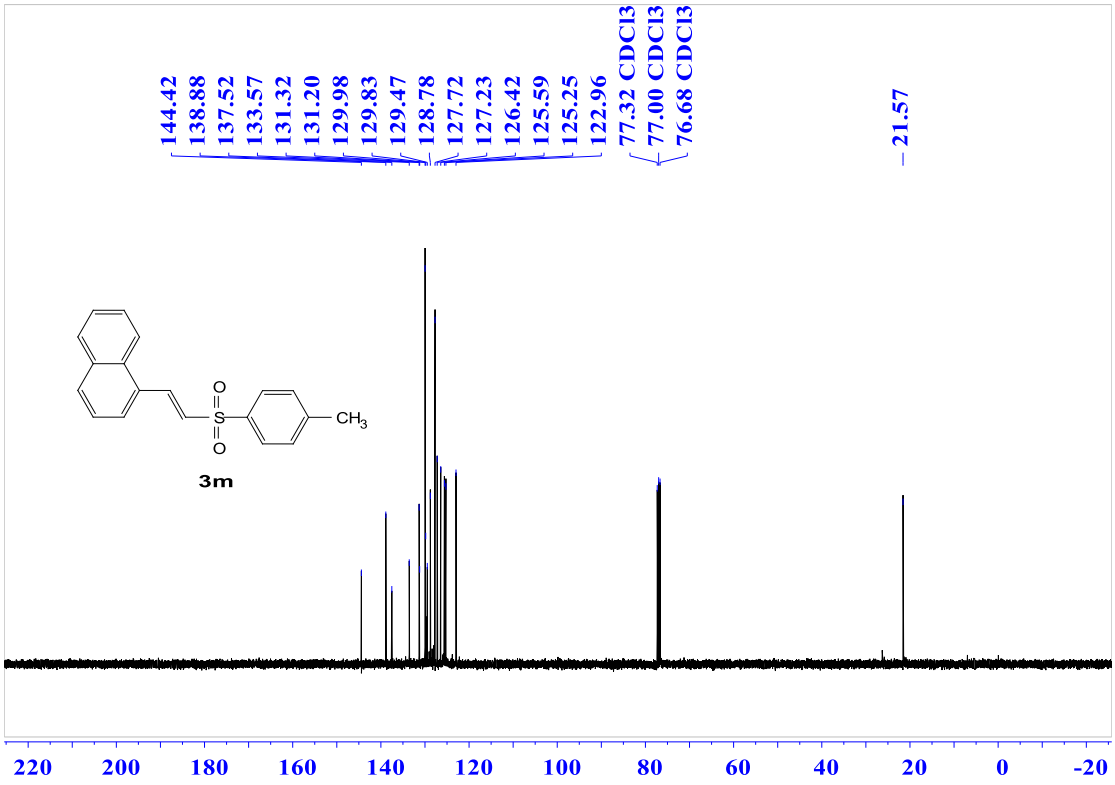
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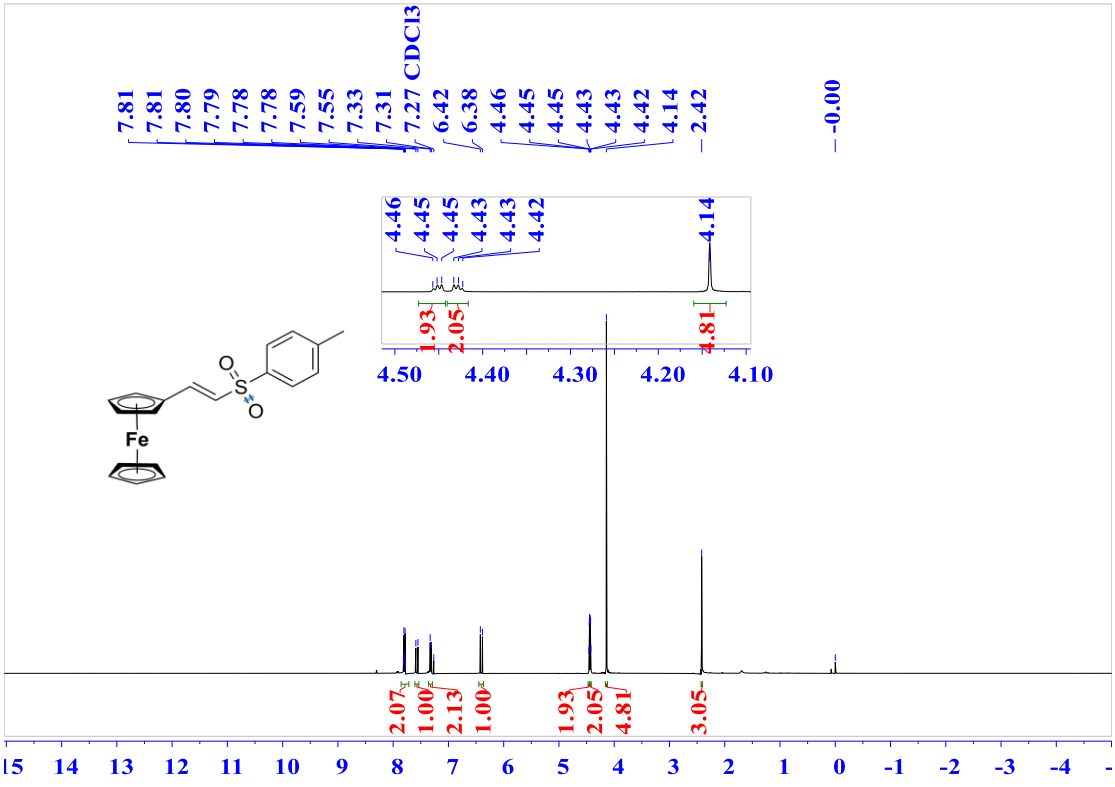
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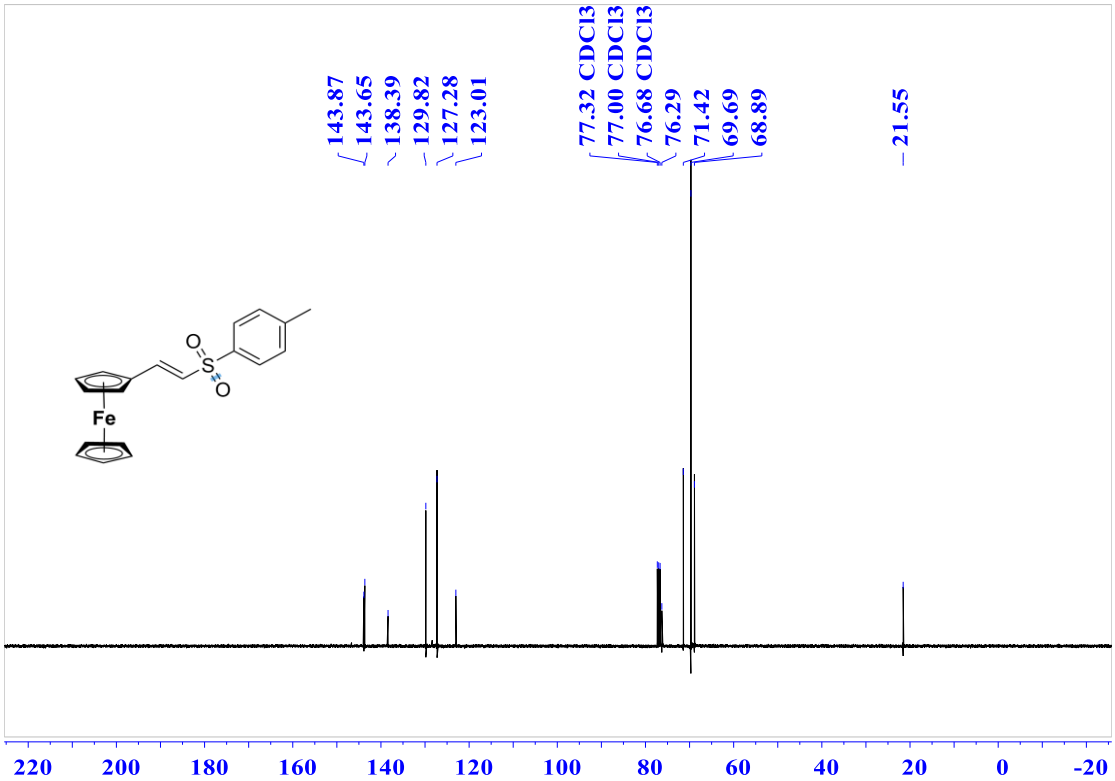
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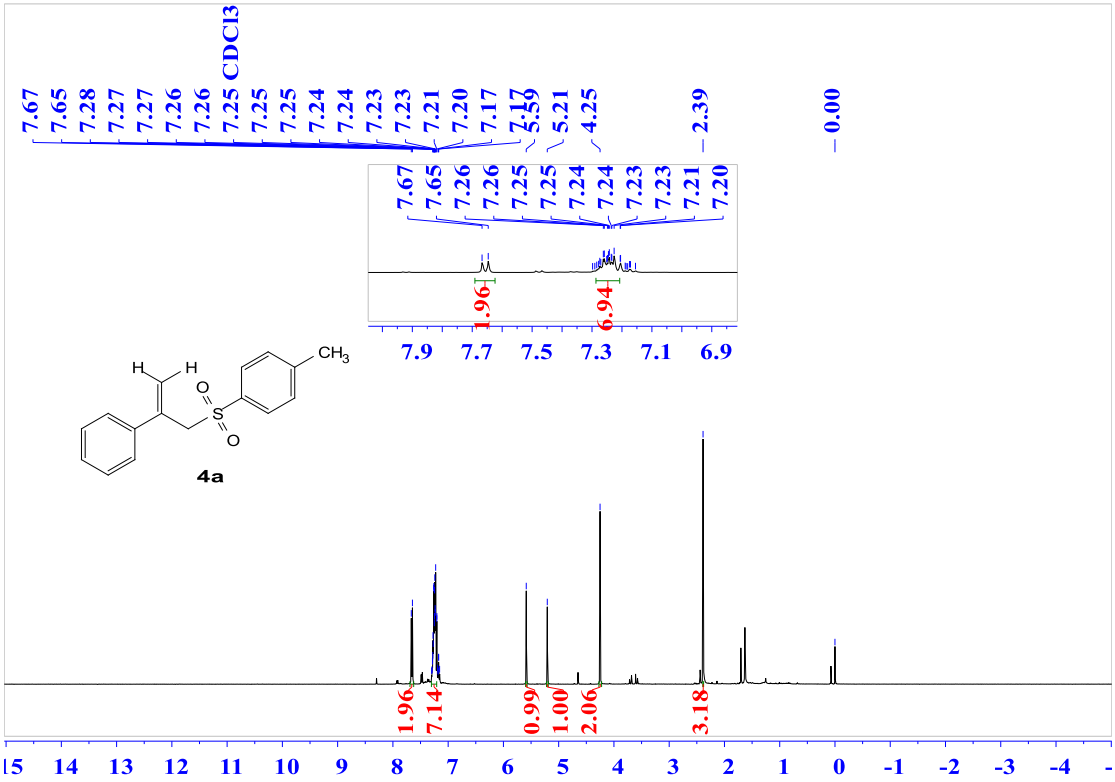
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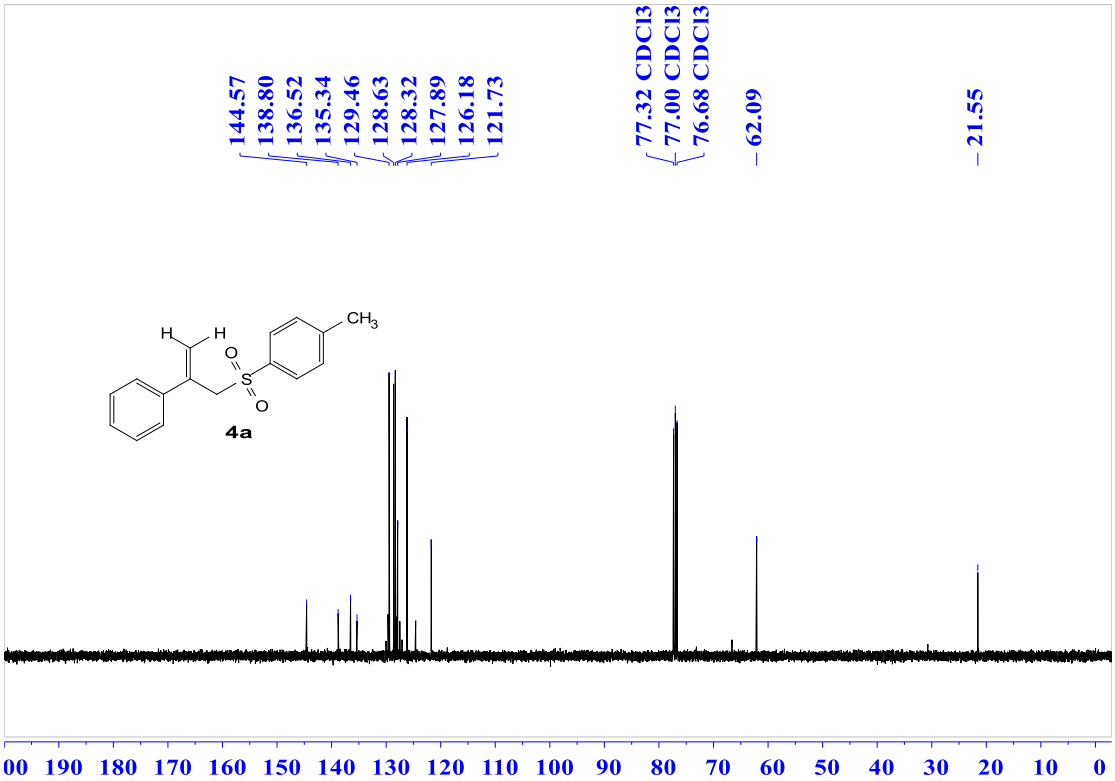
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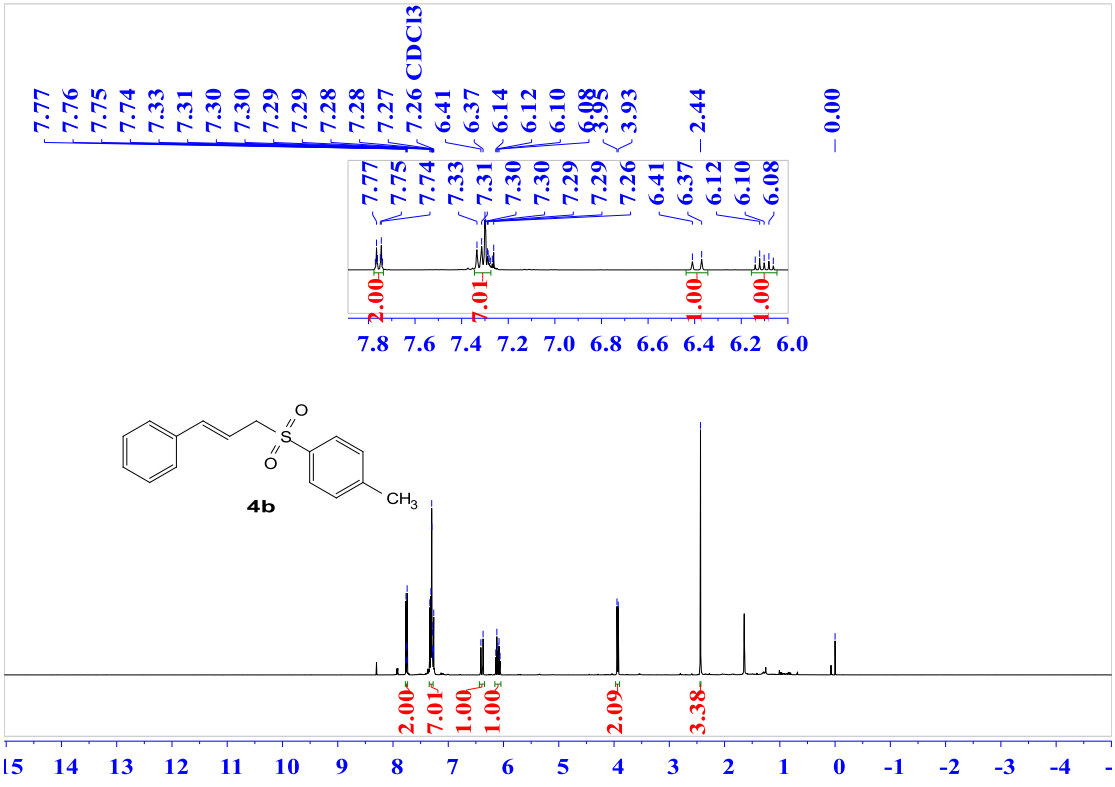
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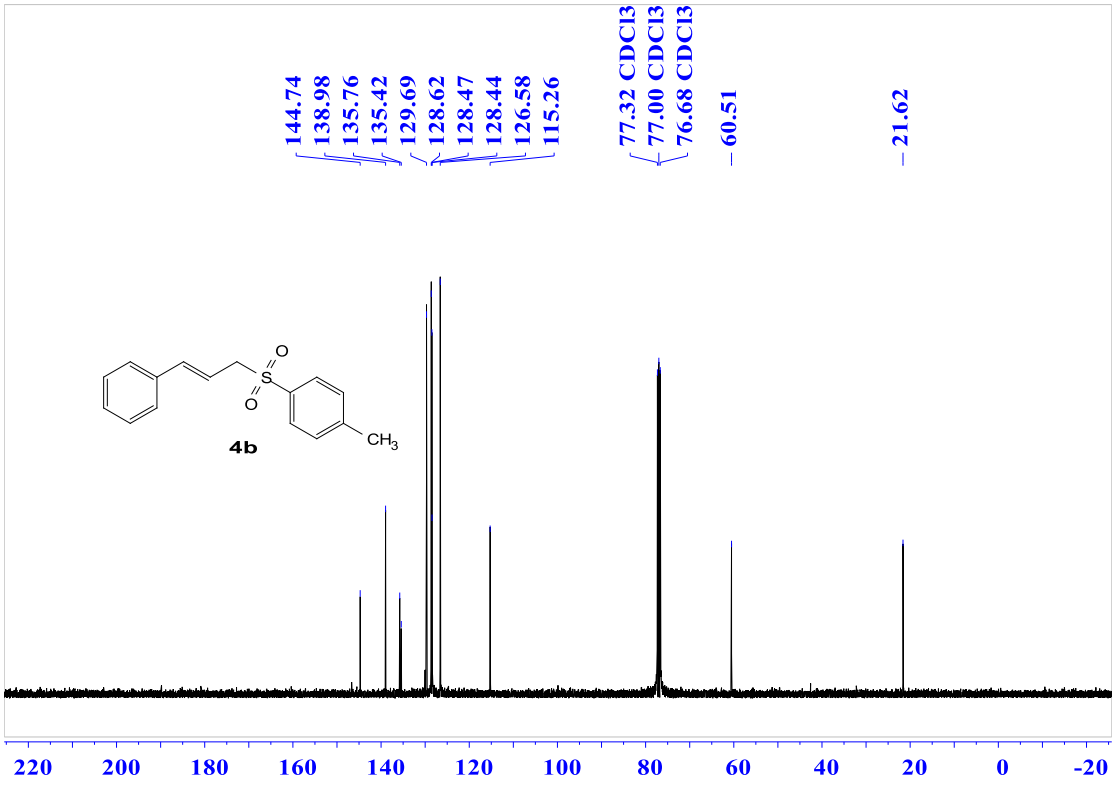
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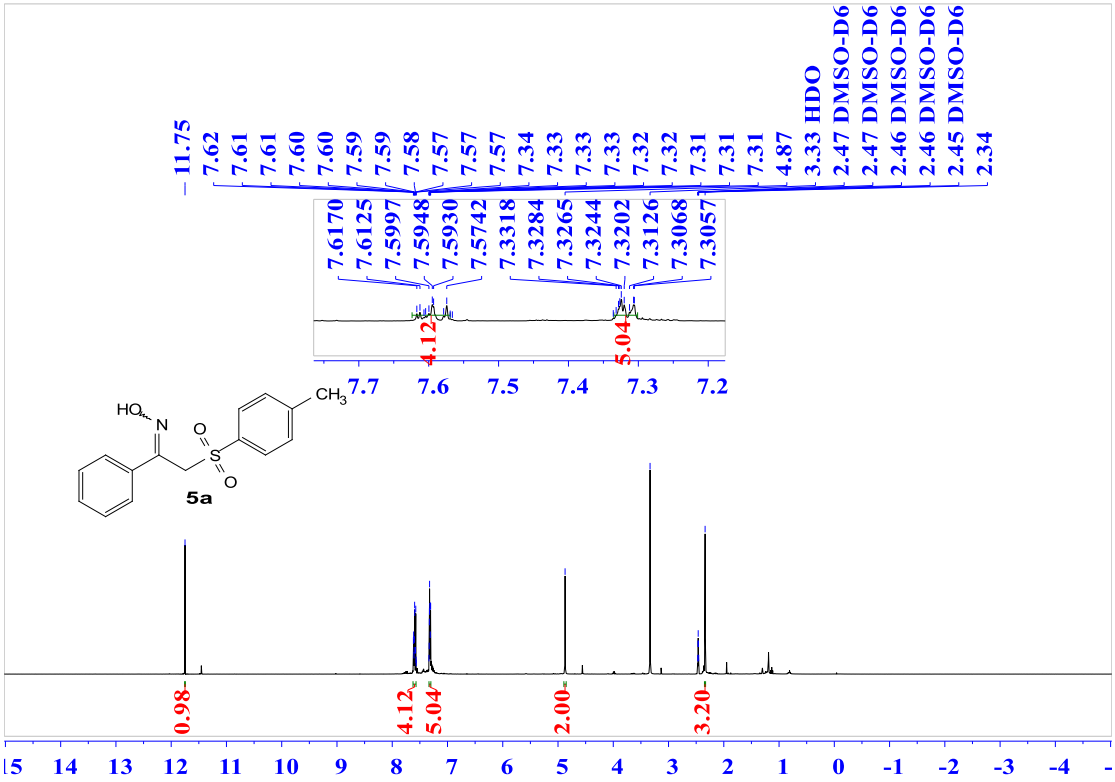
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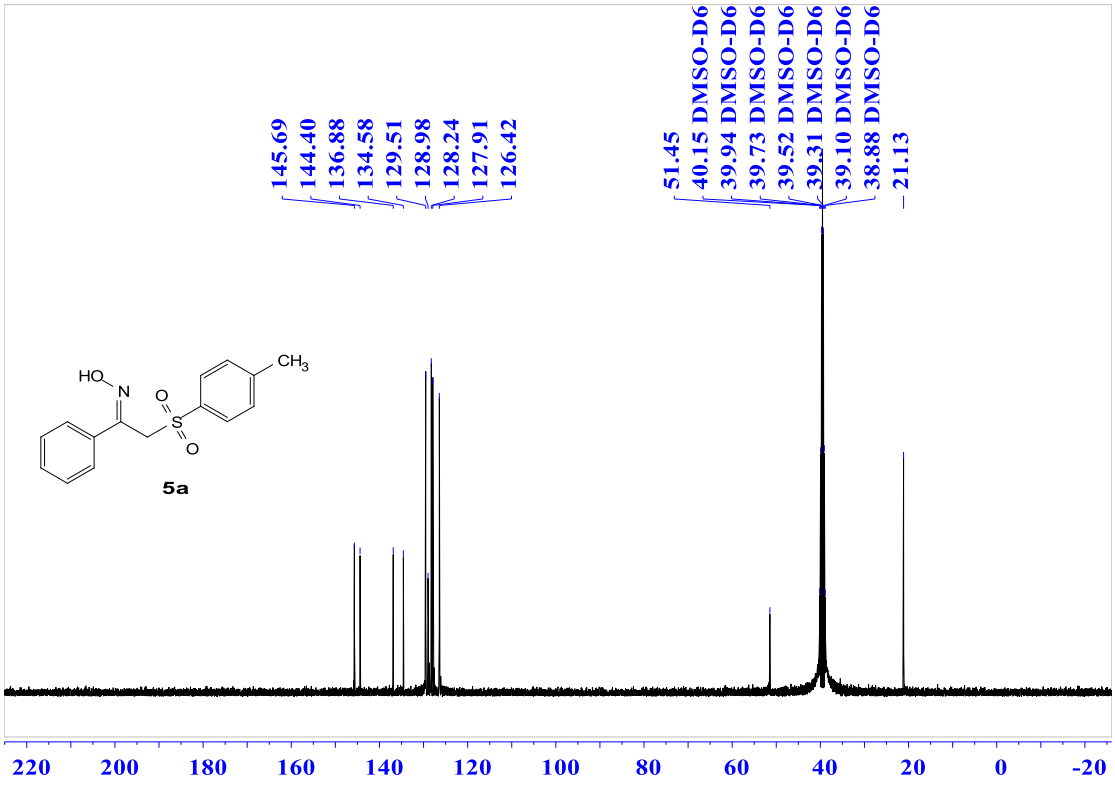
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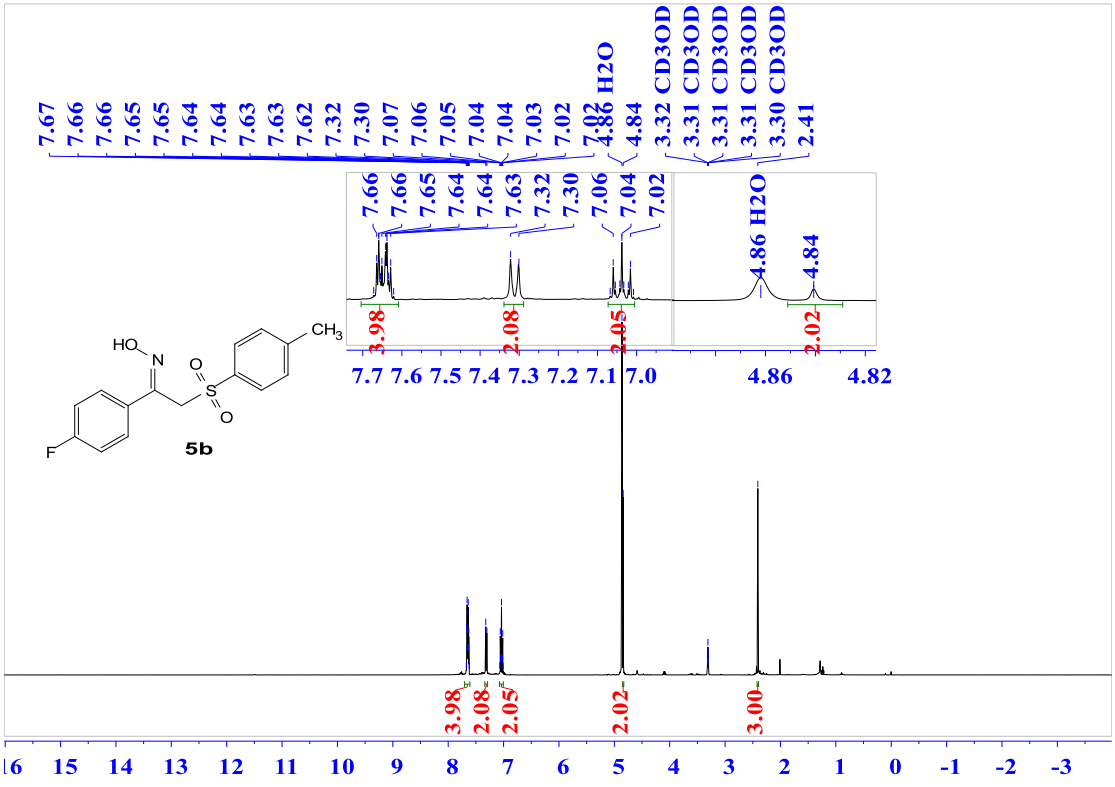
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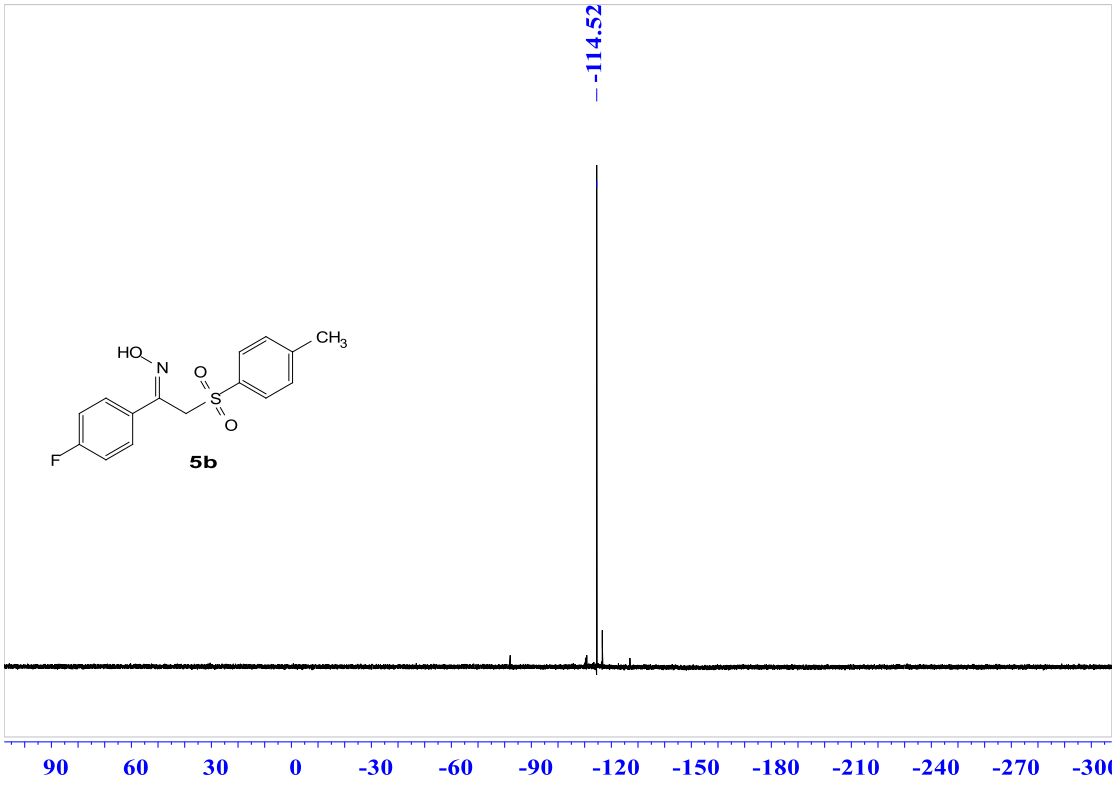
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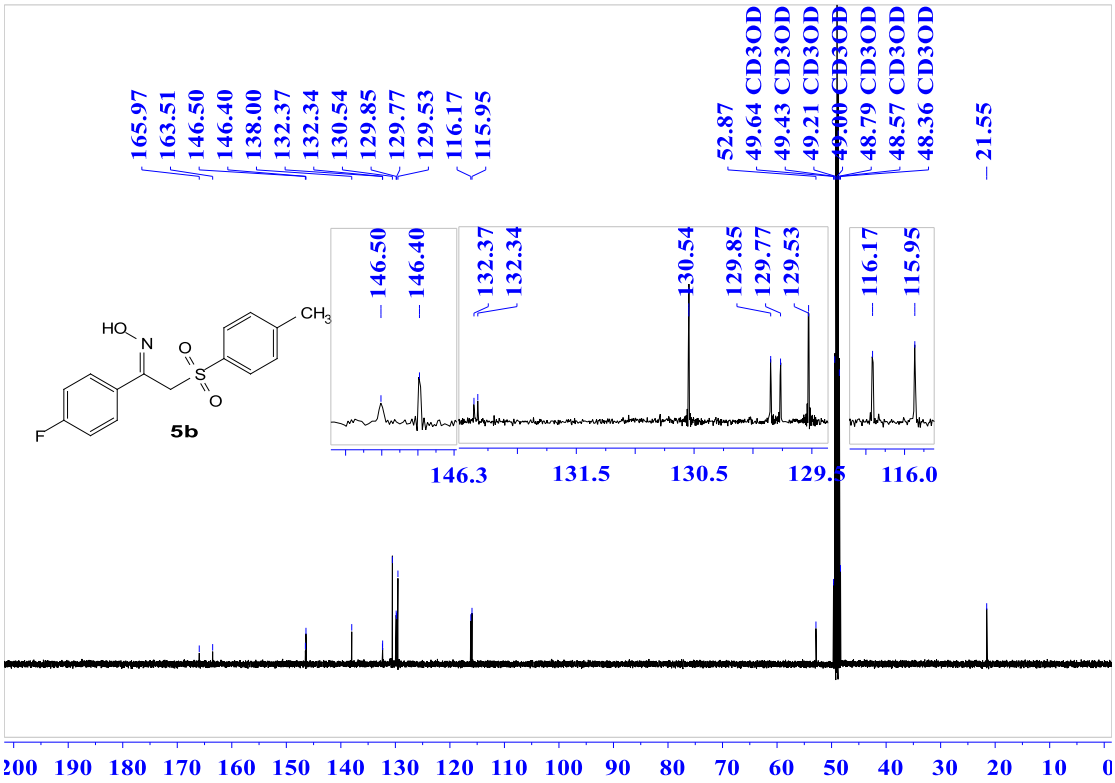
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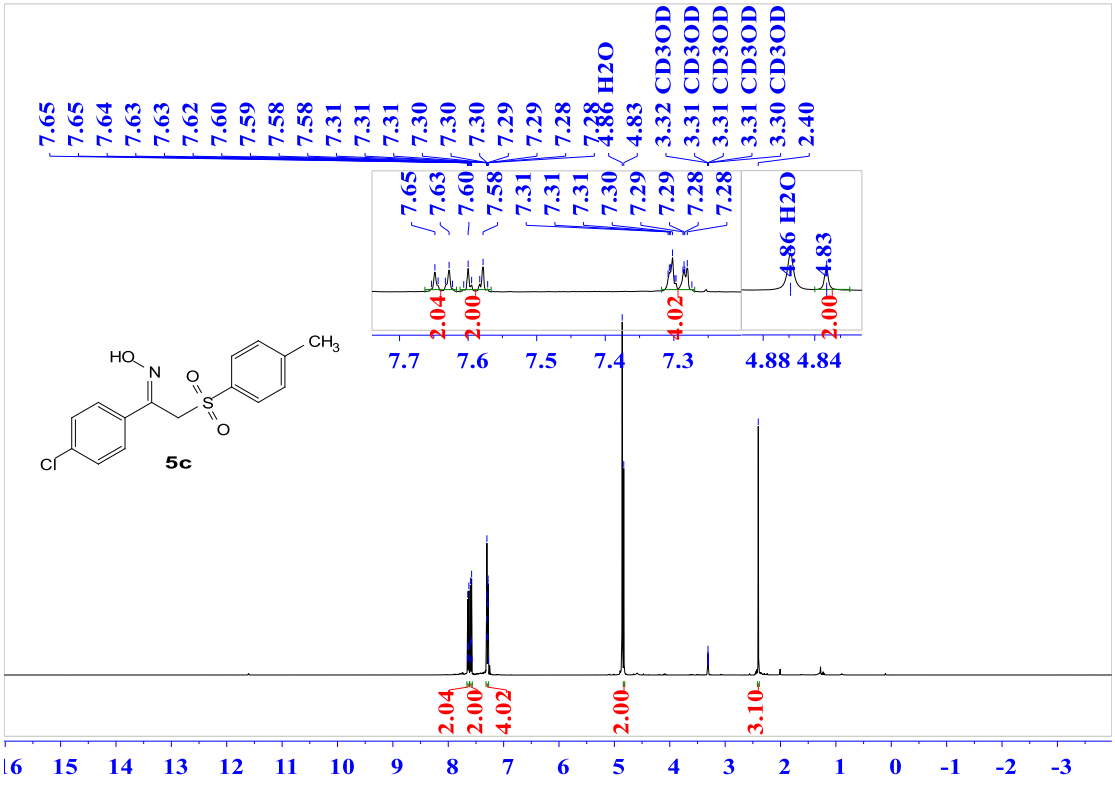
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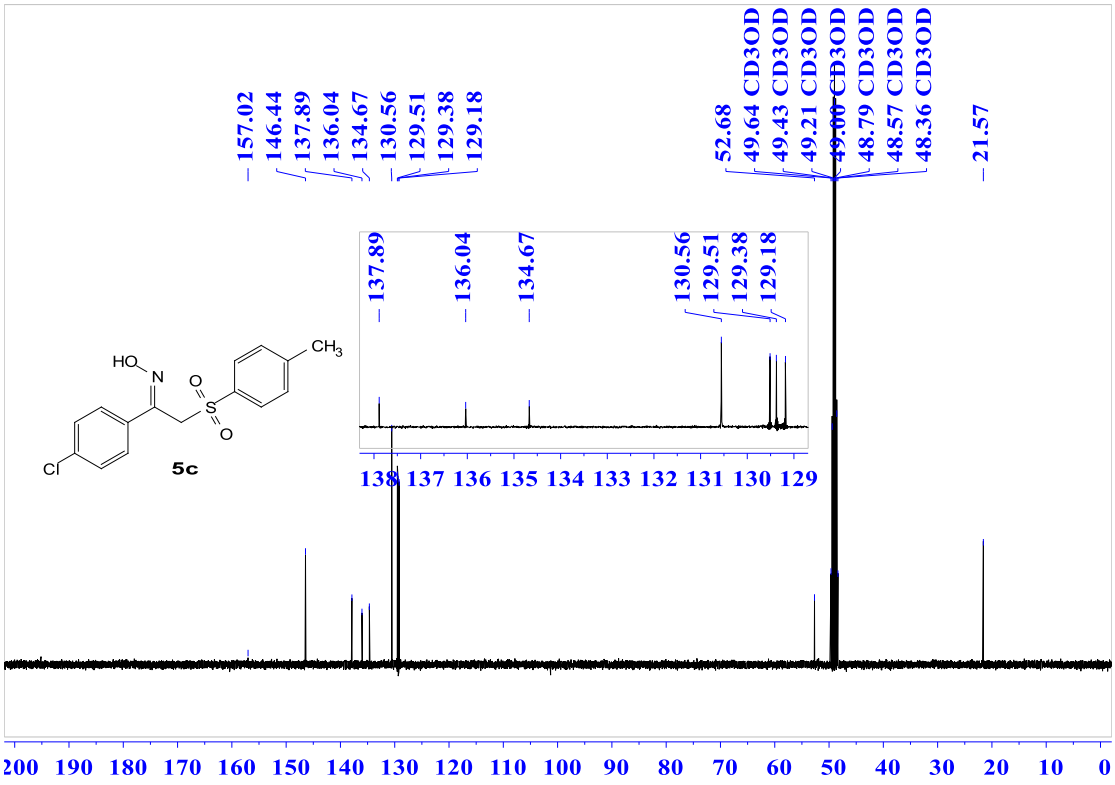
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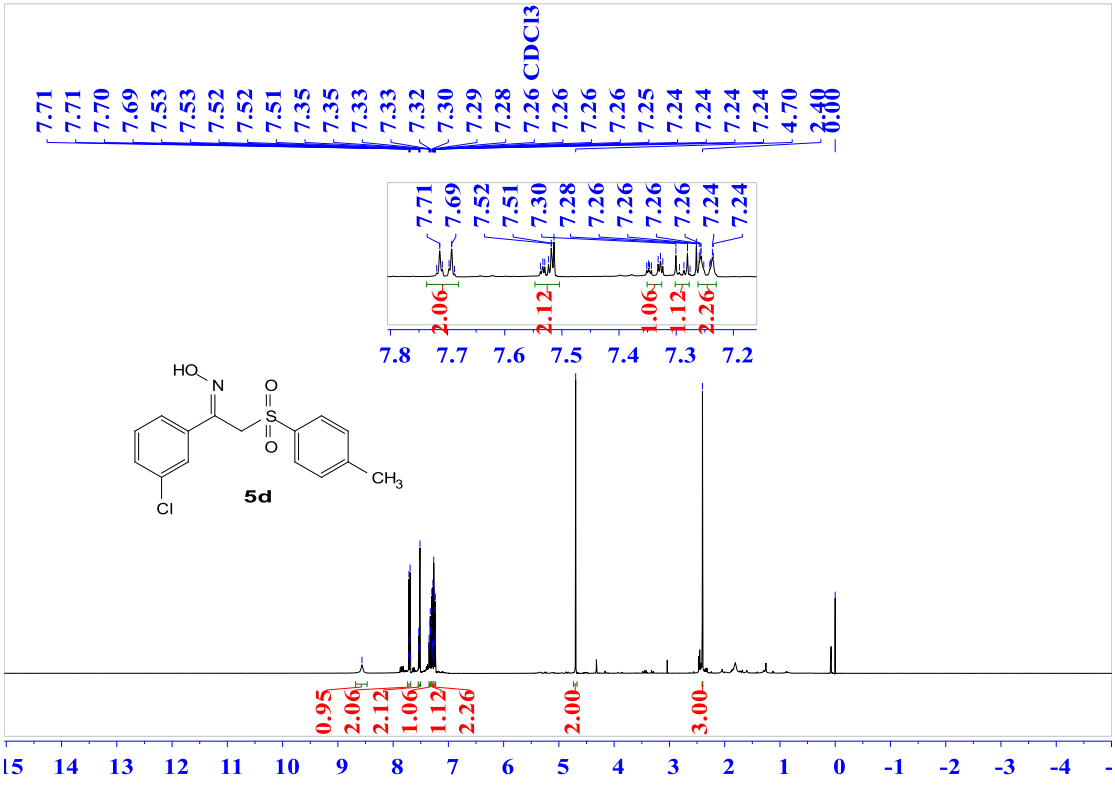
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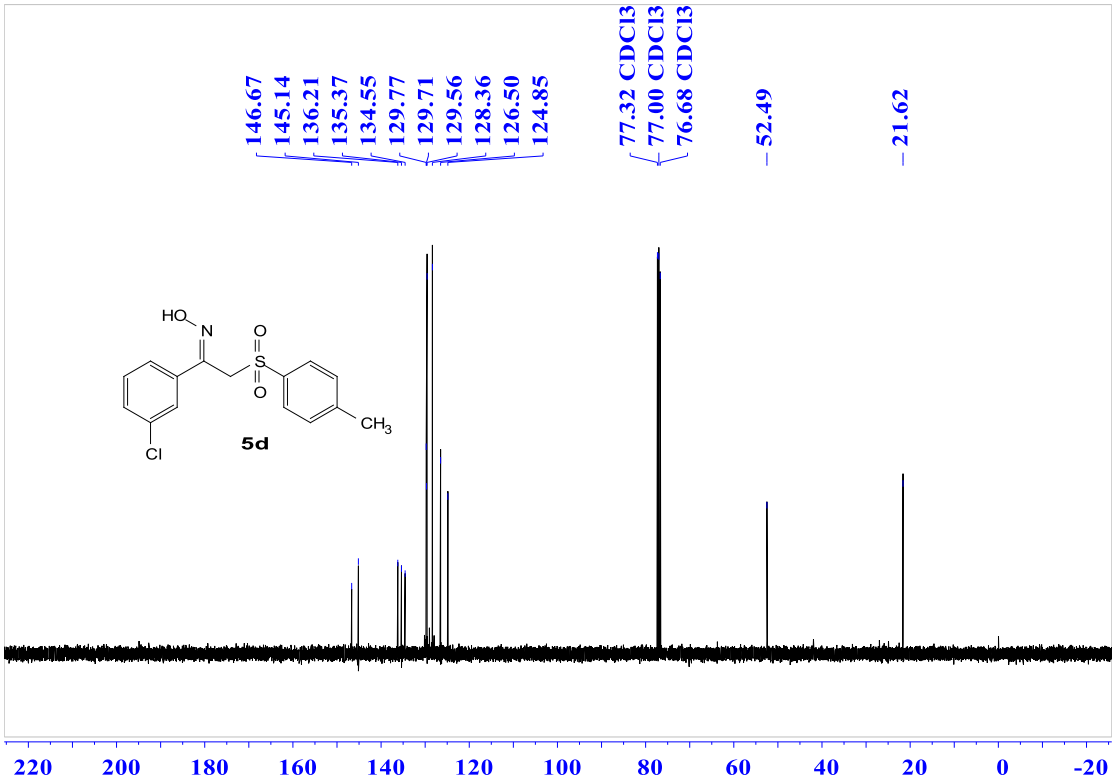
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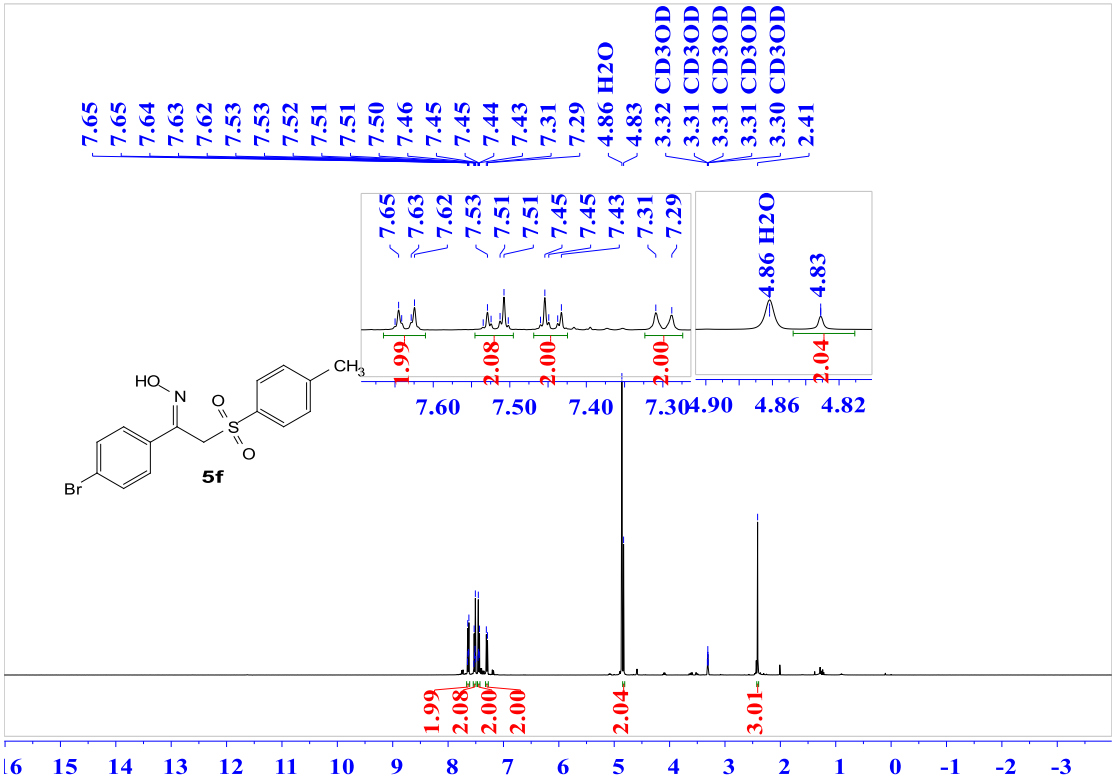
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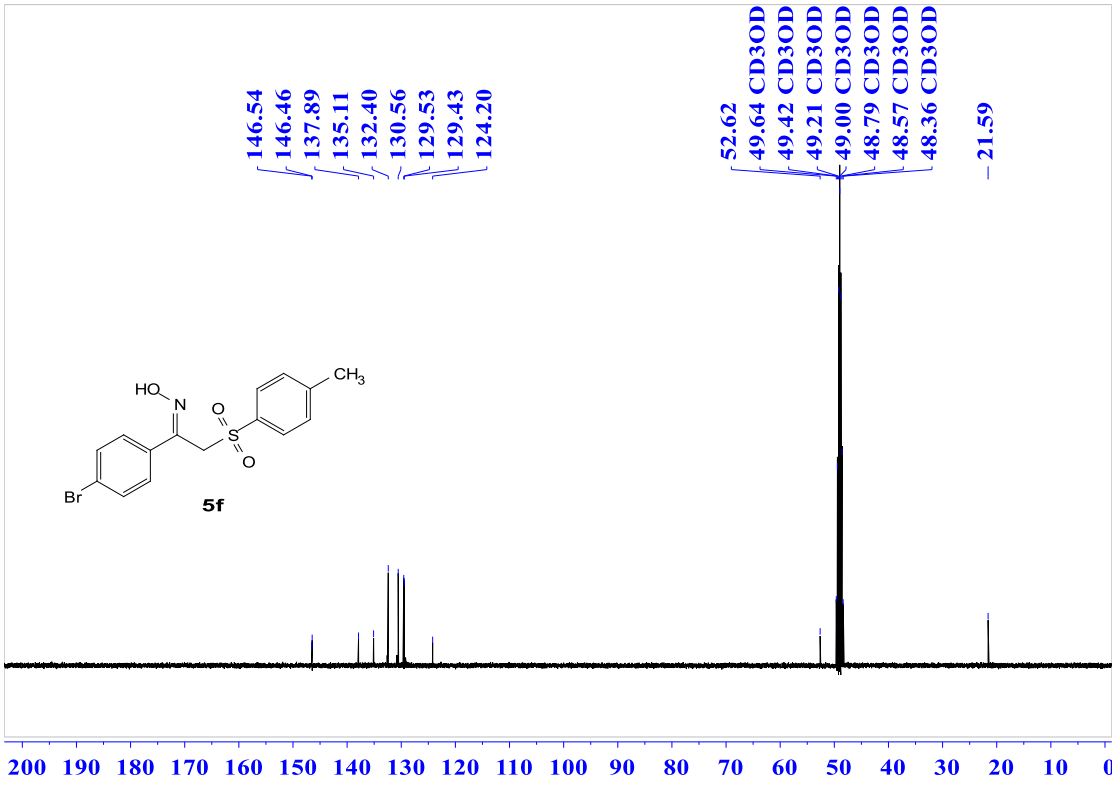
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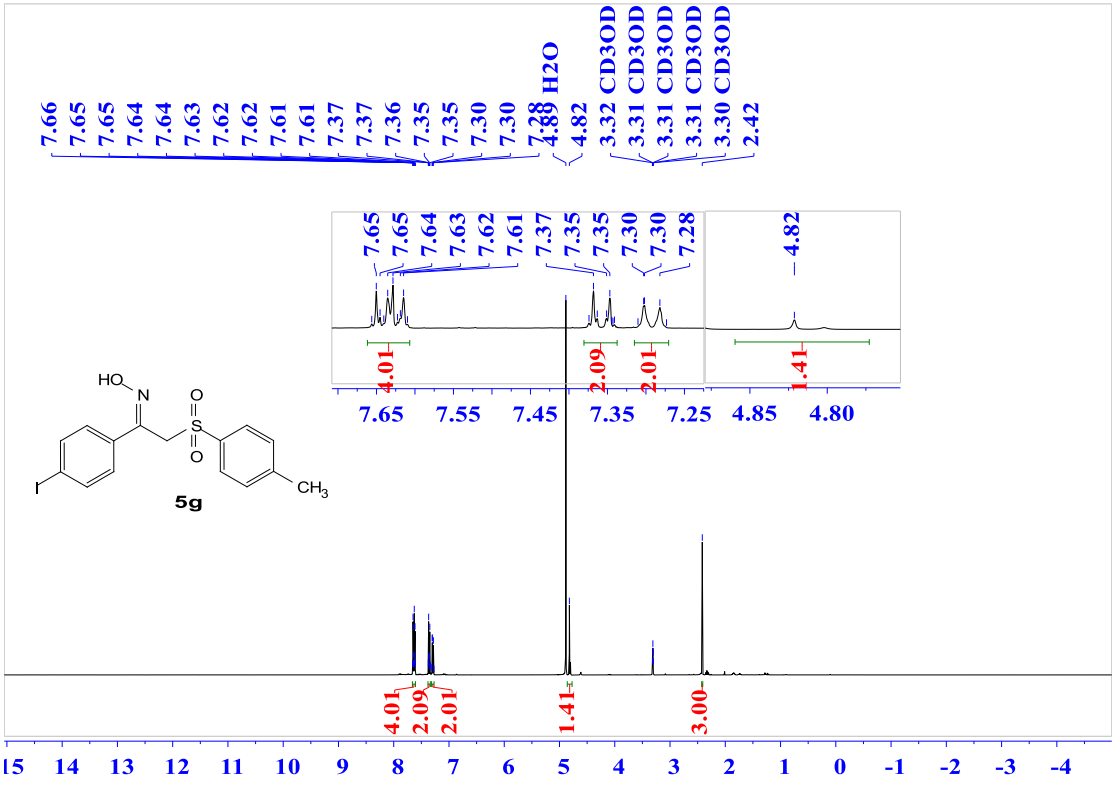
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7.65 7.55 7.45 7.35 7.25 4.85 4.80
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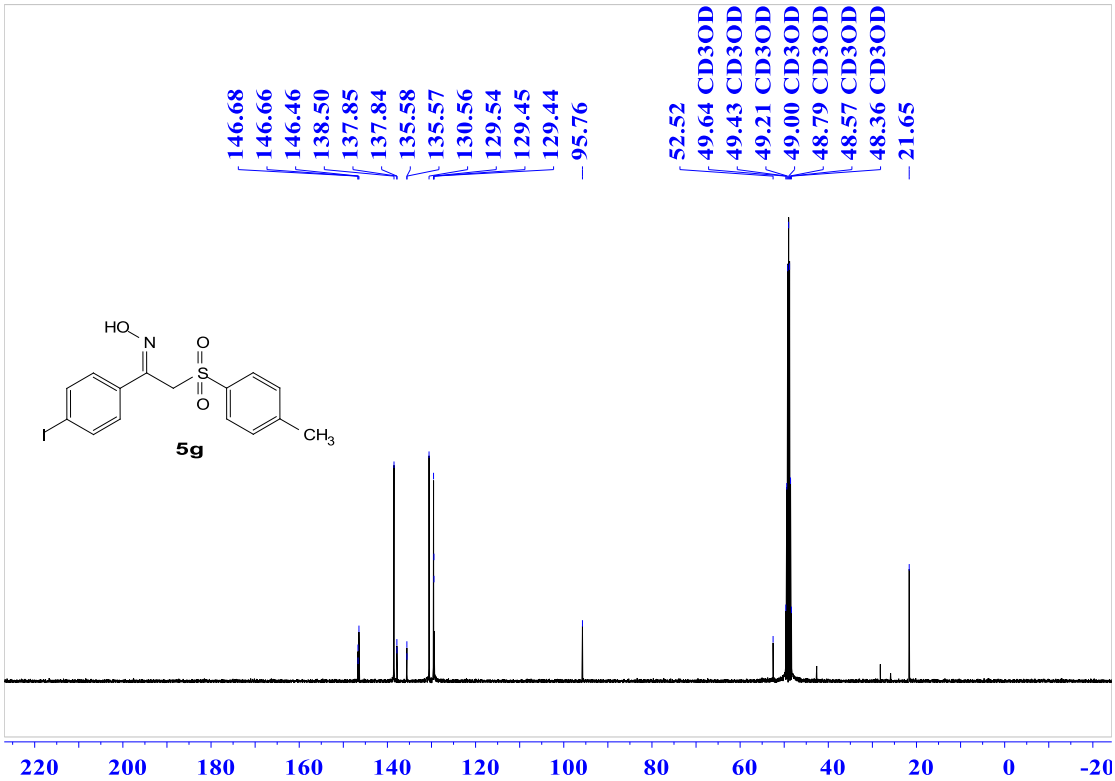
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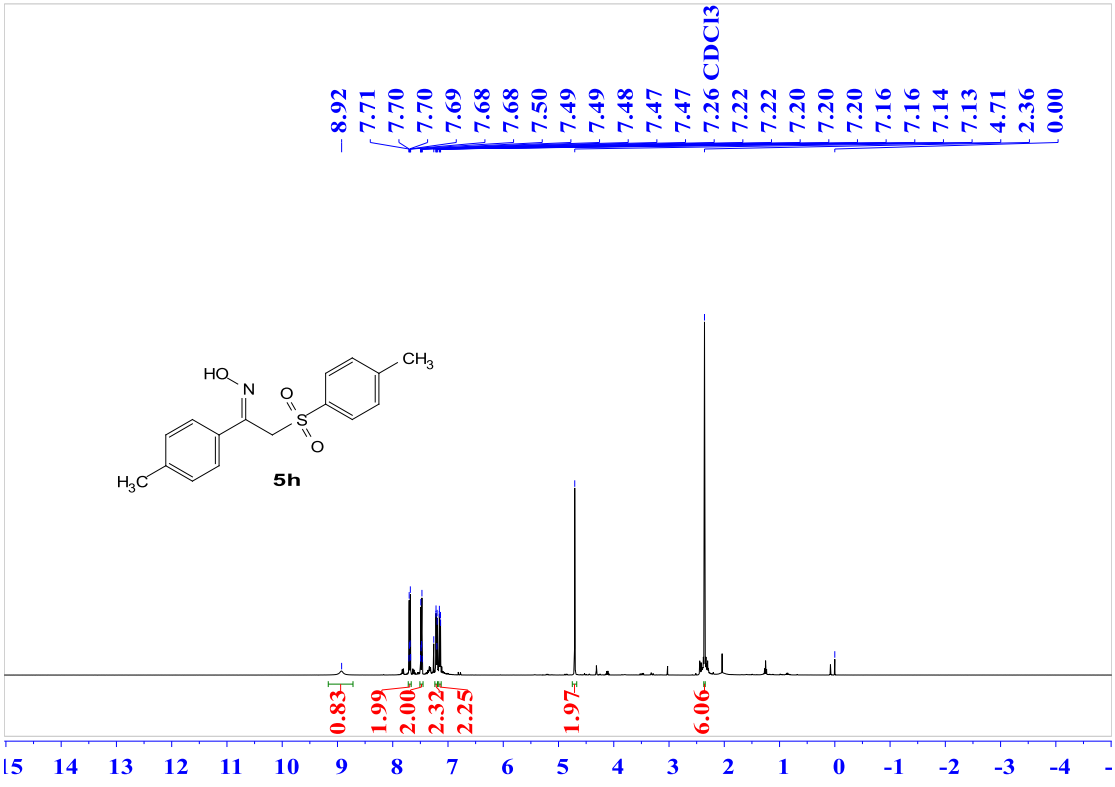
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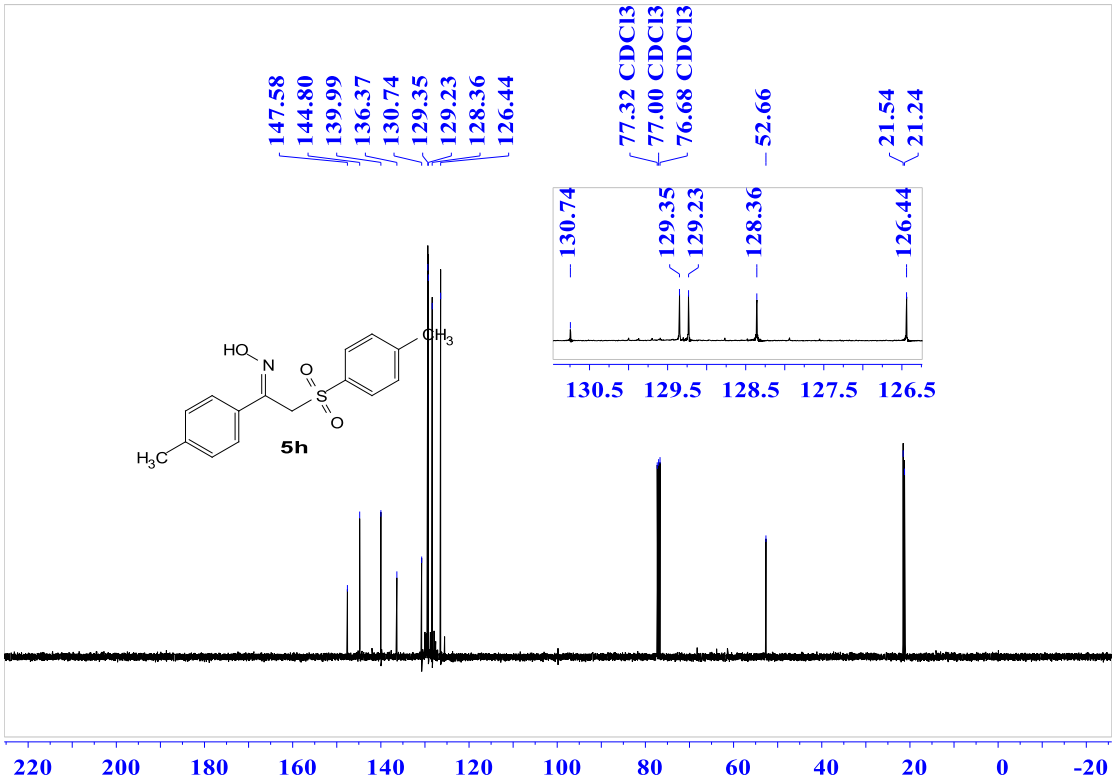
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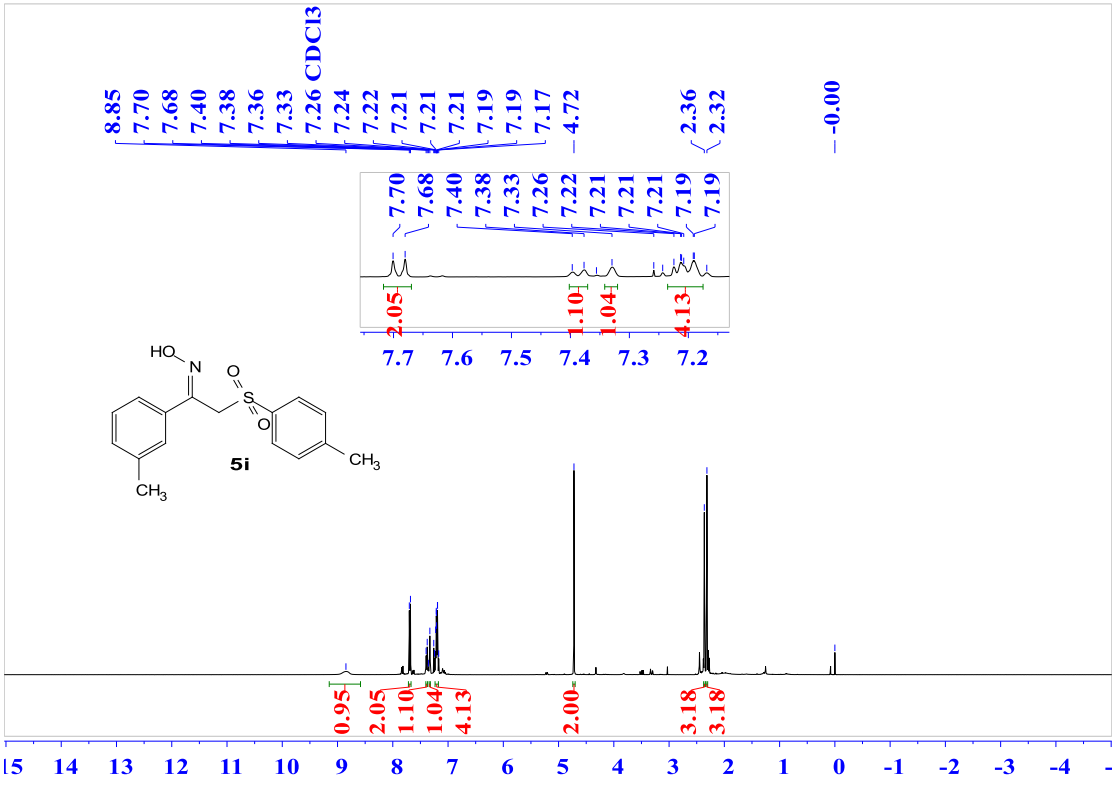
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1.04
4.13
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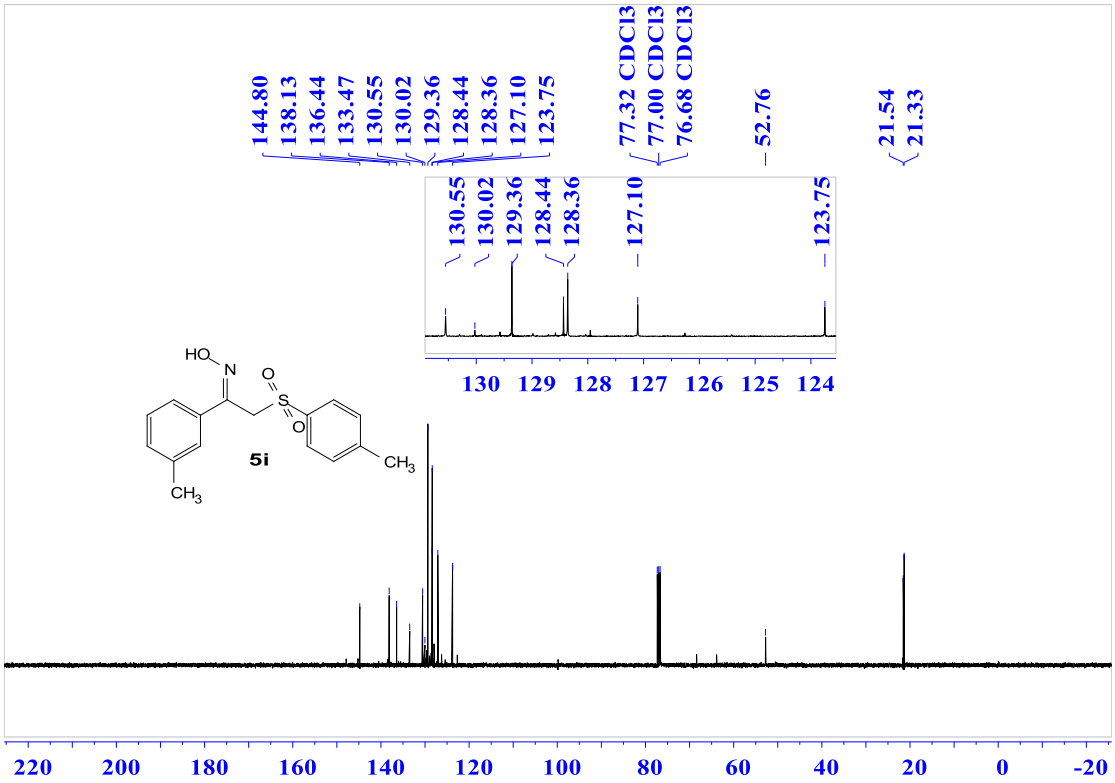
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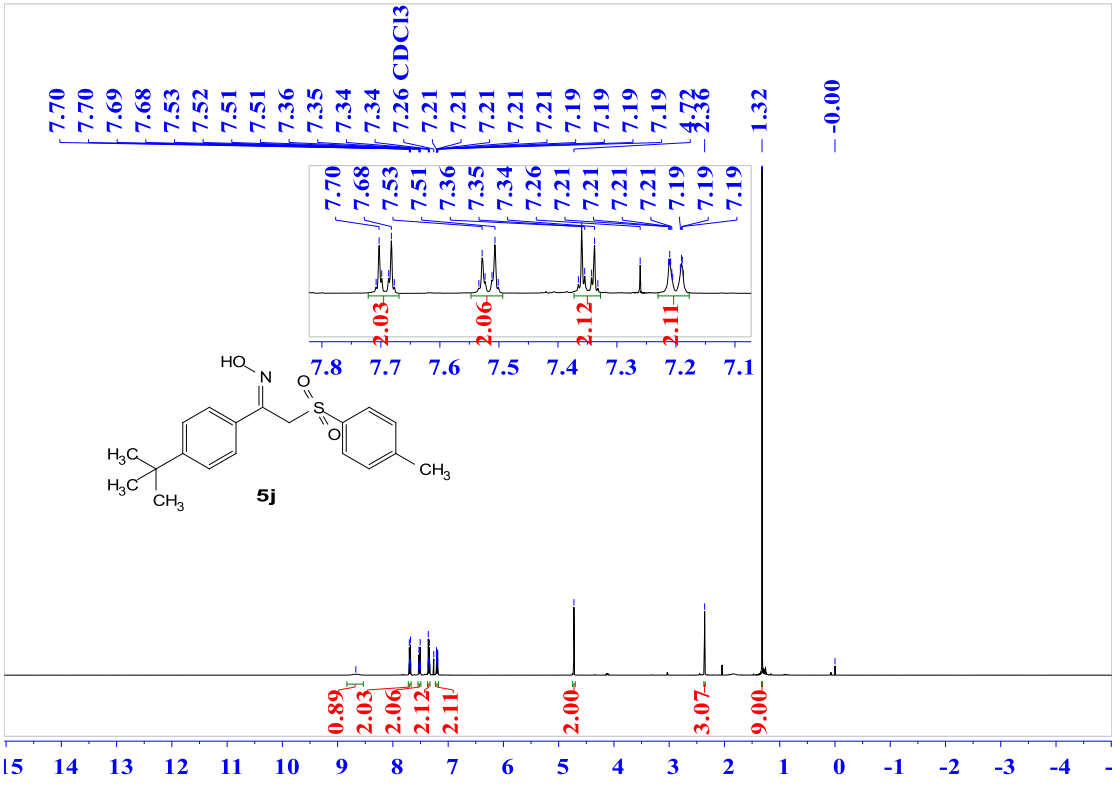
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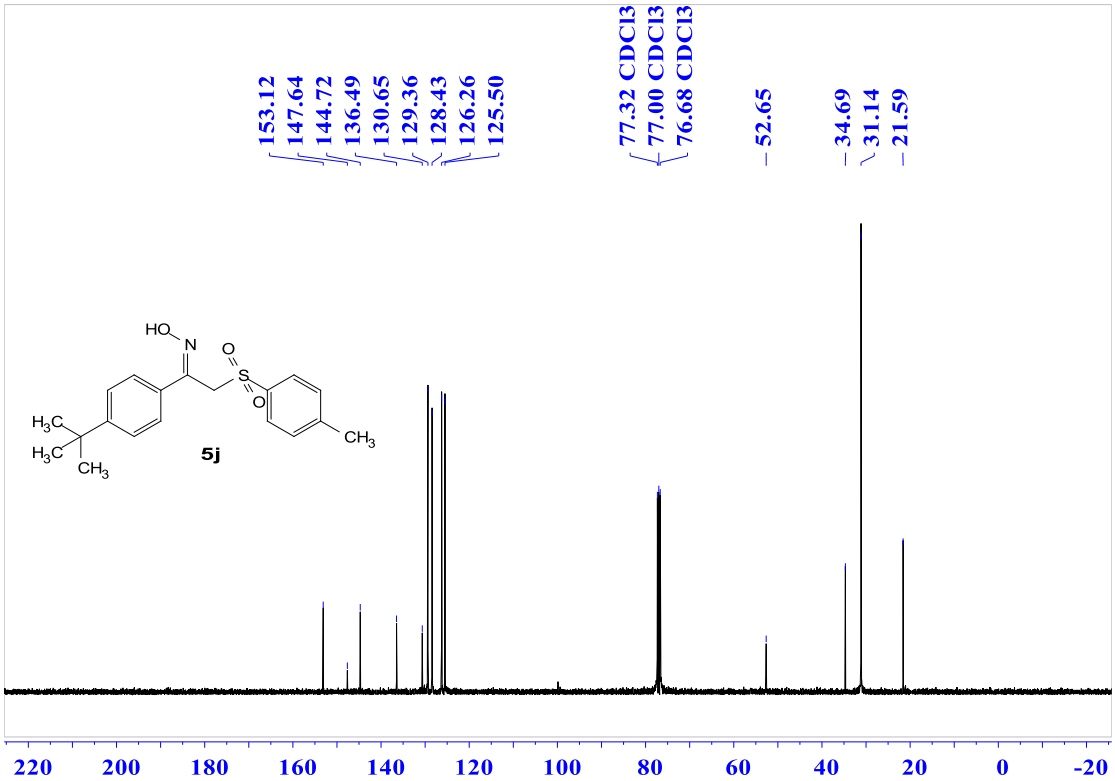
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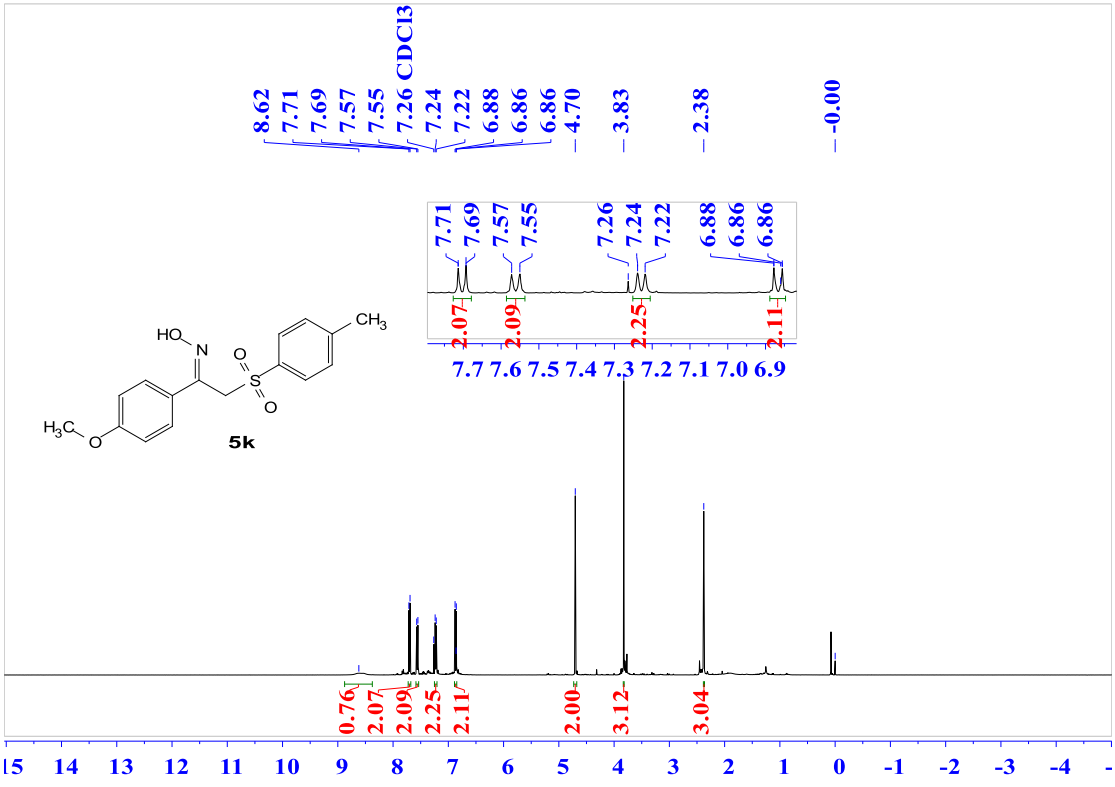
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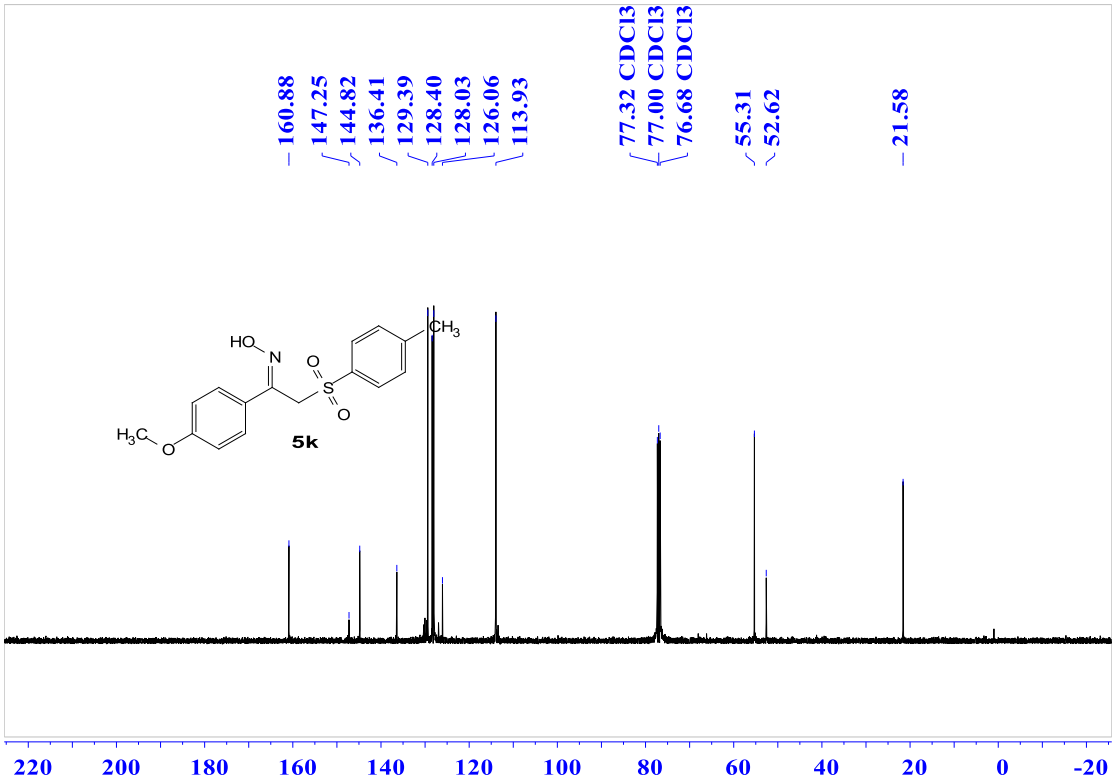
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HO
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