Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Copper-catalyzed oxidative amidation of α, β-unsaturated ketone via selective C–H or C–C bond cleavage

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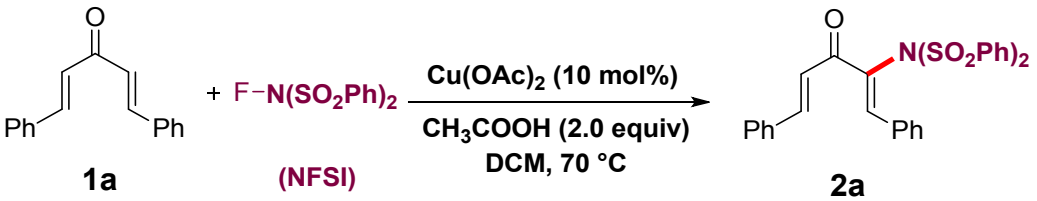
I. General Information

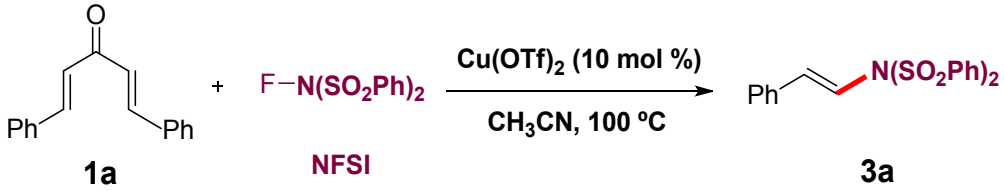
With the exception of Errors or omissions, all materials were purchased from commercial source and used as received. The melting points were obtained with a micro melting point XT4A Beijing Keyi electrooptic apparatus and are uncorrected. Ketones, namely α,β-unsaturated ketones **1** were prepared according to the previous literature.[1-4]^{1}H NMR Spectra were obtained at ambient temperature on a Varian 600 MHz, 500 MHz and 400 MHz, ^{13}C NMR spectra were recorded at ambient temperature on a Varian 125 MHz, 150 MHz and TMS as internal standard. The chemical shifts (**δ**) were reported in parts per million (ppm) relative to internal standard TMS (0 ppm for H^{1}) and $CDCl_3$ (77.0 ppm for ^{13}C). High resolution mass spectra were recorded on Bruck microtof. Coupling Constants (**J**) were then expressed in Hz. The signals have been described according to the following rule: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. All reactions were monitored by thin layer chromatography (TLC) using Machery-Nagel 0.20 mm silica gel 60 plates. Flash column chromatography was also realized on silica gel 60 (particle size 300-400 mesh ASTM, purchased from Taizhou, China) to increase the pressure.

II. General Procedure forOxidative Cleavage of C(vinyl)–H Bond of Ketone for the Synthesis of α-Amino Substituted Ketones 2



1,4-dien-3-ketone **1a** (58.5 mg, 0.25 mmol), NFSI (157.5 mg, 0.5 mmol) and $Cu(OAc)_2$ (4.5 mg, 0.025 mmol) were placed in a round-bottomed flask containing a magnetic stirrer under N_2 atmosphere. 2.5 mL of dichloromethane (DCM) was dissolved. The mixture was then stirred at room temperature for five minutes. To the mixture was added acetic acid glacial (30 μL, 0.5 mmol) and stirred for 24 hours at 70 ºC (monitored by TLC). After the reaction was quenched with water, the aqueous layer was extracted with CH_2Cl_2 (3 × 5.0 mL) and the organic layers were combined, washed with water and brine (1 × 5 mL), and dried over Na_2SO_4. The organic layer was filtered, concentrated by rotary evaporation and purified by flash colunm chromatography on silicate gel as solid phase and petroleum/ethyl acetate (25:1, v:v) as the eluent to give compound **2a** (103.1 mg, 78%) as a yellow solid.

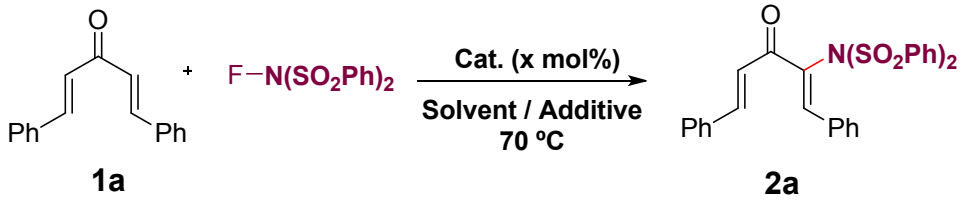
III. General Procedure for Oxidative Cleavage of C(CO)–C(vinyl) Bond of Ketone for the Synthesis of β-Amino Styrenes 3



1,4-dien-3-ketone **1a** (46.8 mg, 0.2 mmol), NFSI (189 mg, 0.6 mmol) and $Cu(OTf)_2$ (7.7 mg, 0.02 mmol) were placed in a Schlenk-tube containing a magnetic stirrer under N_2 atmosphere. The acetonitrile (2 mL) was added as solvent. The mixture was then stirred at 100°C for 0.5 hours and monitored gradually by TLC. The resulting mixture was extracted with dichloromethane (3 ×10 mL). Next, the organic layer was dried over anhydrous Na_2SO_4. After removing the solvent, the residue was purified by column chromatography using the silicate gel as the solid phase and petroleum/ethyl acetate (25:1, v:v) as eluent to afford **3a** (64.6 mg, 81%) as a white solid.

IV. Optimization of Oxidative Amidation of α, β-Unsaturated Ketone for the synthesis of enamides.

1. Optimization of Oxidative Cleavage of C(vinyl)–H Bond of Ketone for the Synthesis of α-Amino Substituted Ketones 2[a]

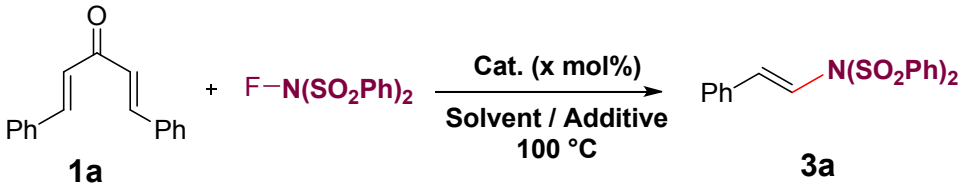


1a **2a**

Entry	Catalyst (mol%)	Solvent	Additive (equiv)	T (° C)	Yield (%)[b]
1	-	DCE	-	70	0
2	$Cu(OAc)_2$	DCE	-	70	10
3	$Cu(OAc)_2$	DMF	-	70	0
4	$Cu(OAc)_2$	PhCN	-	70	trace
5	$Cu(OAc)_2$	CH_3OH	-	70	trace
6	$Cu(OAc)_2$	Toluene	-	70	0
7	$Cu(OAc)_2$	DCM	-	70	43
8	$Cu(OAc)_2$	CH_3CN	-	70	27
9	$Cu(OAc)_2$	DCM	CH_3OH (1.0)	70	39
10[c]	$Cu(OAc)_2$	DCM	$Zn(OTf)_2$	70	42
11	$Cu(OAc)_2$	DCM	TFA (1.0)	70	51
12	$Cu(OAc)_2$	DCM	TFA (2.0)	70	56
13	$Cu(OAc)_2$	DCM	CH_3COOH (1.0)	70	60
14	**$Cu(OAc)_2$**	**DCM**	**CH_3COOH (2.0)**	**70**	**78**
15	CuBr	DCM	CH_3COOH (2.0)	70	14
16	CuCl	DCM	CH_3COOH (2.0)	70	16
17	CuI	DCM	CH_3COOH (2.0)	70	11
18	$CuCl_2$	DCM	CH_3COOH (2.0)	70	41
19	$Cu(OTf)_2$	DCM	CH_3COOH (2.0)	70	63
20	$Cu(NO_3)_2{\cdot}3H_2O$	DCM	CH_3COOH (2.0)	70	32
21[d]	$Cu(OAc)_2$	DCM	CH_3COOH (2.0)	70	47

[a]Reaction conditions: **1a** (0.25 mmol), NFSI (2.0 equiv), Cat (10 mol%), additives (x equiv), and solvent (2 mL) under N_2 atmosphere at 70 °C for 24 h. [b]Yield of the isolated product.[c]$Zn(OTf)_2$ (10 mol%).[d]The reaction was performed in the presence of 10% of Phen = 1,10-phenanthroline. TFA = trifluoroacetic acid, DMF = dimethylformamide, DCM = dichloromethane, DCE = 1,2-dichloroethene.

2. Optimization of Oxidative Cleavage of C(CO)–C(vinyl) Bond of Ketone for the Synthesis of β-Amino Styrenes 3[a]



1a 3a

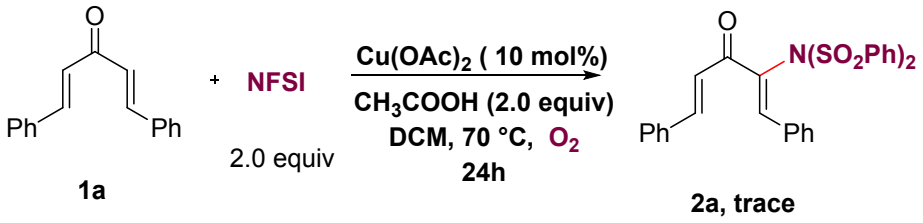
Entry	Catalyst (mol%)	Solvent	Additive (equiv)	T(°C)	times (h)	Yield(%)[b]
1	-	DCM	-	70	24	0
2	CuBr	DCM	-	70	24	12
3	CuCl	DCM	-	70	24	10
4	CuCN	DCM	-	70	24	9
5	$Cu(OAc)_2$	CH_3CN		70	24	33
6	$Cu(OTf)_2$	DCM	-	70	24	20
7	$Cu(OTf)_2$	CH_3CN	-	70	2	42
8	$Cu(OTf)_2$	CH_3CN	-	70	1	56
9	$Cu(OAc)_2$	CH_3CN	-	70	1	51
10	$CuCl_2$	CH_3CN	-	70	1	11
11	$CuBr_2$	CH_3CN	-	70	1	8
12	$Cu(OTf)_2$	CH_3CN	-	100	1	68
13	**$Cu(OTf)_2$**	**CH_3CN**	**-**	**100**	**0.5**	**81**
14	$Cu(OTf)_2$	DCE	-	100	0.5	13
15	$Cu(OTf)_2$	DMF	-	100	0.5	Trace
16	$Cu(OTf)_2$	THF	-	100	0.5	Trace
17	$Cu(OTf)_2$	PhCl	-	100	0.5	Trace
18	$Cu(OTf)_2$	DMSO	-	100	0.5	Trace
19	$Cu(OTf)_2$	Toluene	-	100	0.5	0
20	$Cu(OTf)_2$	CH_3NO_2	-	100	0.5	11
21	$Cu(OAc)_2$	CH_3CN	-	100	0.5	62
22	$Cu(OAc)_2$	CH_3CN	Phen (2.0)	100	0.5	11
23	$Cu(OAc)_2$	CH_3CN	Pyridine (2.0)	100	0.5	9
24	$Cu(OAc)_2$	CH_3CN	PPh_3 (2.0)	100	0.5	0
25	$Cu(OAc)_2$	CH_3CN	ddpe (2.0)	100	0.5	0

[a]Reaction conditions: **1a** (0.2 mmol), NFSI (0.6 mmol),$Cu(OTf)_2$ (10 mol%), solvent (2 mL) under N_2 atmosphere.[b]Isolated yield. DCM = dichloromethane, TFA = trifluoroacetic acid, DMF = dimethylformamide, THF = tetrahydrofuran, DMSO = dimethylsulfoxide, Phen = 1,10-phenanthroline, PPh_3 = triphenylphosphine, dppe = 1,2-Bis(diphenylphosphino)ethane.

V. Control Experiments for Oxidative Amidation of α, β-Unsaturated Ketone

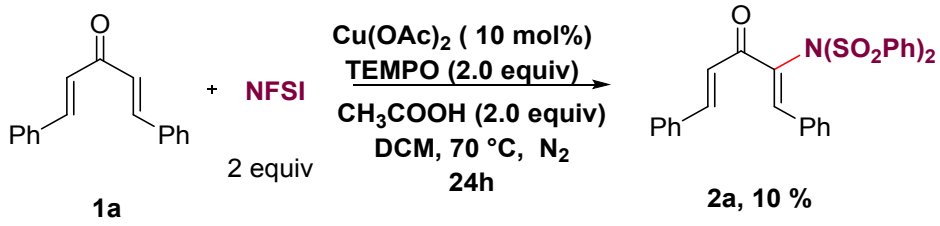
1. Control Experiments for Oxidative Cleavage of C(vinyl)–H Bond of Ketone

1.1 The reaction of 1a with NFSI under O_2 atmosphere



The reaction of 1,4-dien-3-ketone **1a** (58.5 mg, 0.25 mmol), $Cu(OAc)_2$ (4.5 mg, 0.025 mmol), *N*-Fluorobenzenesulfonimide (157.5 mg, 0.6 mmol), acetic acid (30 μL, 0.5 mmol), and dichloromethane (2.5 mL) at 70 ºC under O_2 atmosphere afforded a trace of compound **2a**.

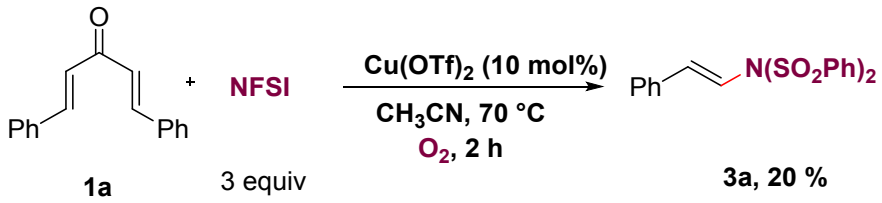
1.2 Experimental Procedure for Oxidative Cleavage C(vinyl)–H Bond of Ketone with TEMPO



1,4-dien-3-ketone **1a** (58.5 mg, 0.25 mmol), NFSI (157.5 mg, 0.5 mmol), 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (62.5 mg, 0.4 mmol) and $Cu(OAc)_2$ (4.4 mg, 0.025 mmol) were placed in a round-bottomed flask containing a magnetic stirrer under N_2 atmosphere. 2.5 mL of dichloromethane (DCM) was dissolved. The mixture was then stirred at room temperature for five minutes. To the mixture was added acetic acid glacial (30 μL, 0.5 mmol) and stirred for 24 hours at 70 ºC (monitored by TLC). Upon completion of the reaction (monitored by TLC), the amidation product **2a** was found and then purified by flash colunm chromatography on silicate gel as solid phase and petroleum/ethyl acetate (25:1, v:v) as the eluent with 10 % yield.

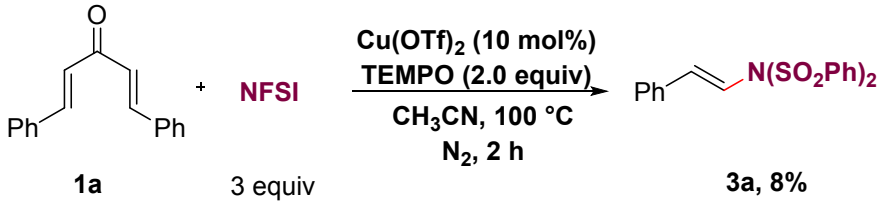
2. Control Experiments for Oxidative Cleavage of C(CO)–C(vinyl) Bond of Ketone

2.1 The reaction of 1a with NFSI under O_2 atmosphere



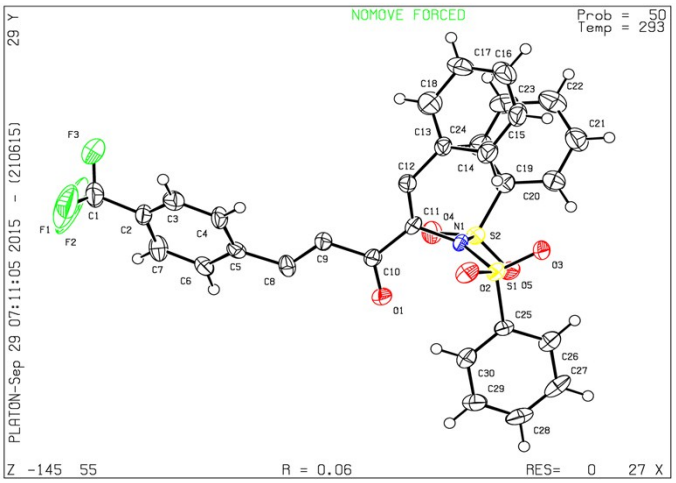
1,4-dien-3-ketone **1a** (46.8 mg, 0.2 mmol), $Cu(OTf)_2$ (7.7 mg, 0.02 mmol), *N*-Fluorobenzenesulfonimide (236 mg, 0.6 mmol), and acetonitrile (CH_3CN) (2.0 mL) were placed in 25 ml round-bottom flask. The flask was sealed with a rubber septum and degassed and refilled with O_2 (3 times). Then, the reaction flask was heated at 70 ˚C in a preheated oil bath for 2 hours. The resulting solution was extracted with dichloromethane (3 × 10 mL), and the combined organic layer was washed with brin solution (10 mL) and concentrated in *vacuo*. The crude residue was purified using silica gel column chromatography with petrolum ether / ethyl acetate (20:1) as the eluent to afford the corresponding amidation product **3a** at 20 % yield.

2.2 Experimental Procedure Oxidative Cleavage C(CO)–C(vinyl) Bond of Ketone with TEMPO



1,4-dien-3-ketone **1a** (46.8 mg, 0.2 mmol), NFSI (189 mg, 0.6 mmol), 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (62.5 mg, 0.4 mmol) and $Cu(OTf)_2$ (7.7 mg, 0.02 mmol) were placed in a Schlenk-tube containing a magnetic stirrer under N_2 atmosphere. The acetonitrile (2mL) was added as solvent. The mixture was then stirred at 100°C for 2 hours and monitored gradually by TLC. Upon completion of the reaction (monitored by TLC), the amidation product **3a** was found and then purified by flash colunm chromatography on silicate gel as solid phase and petroleum/ethyl acetate (25:1, v:v) as the eluent with 8 % yield.

VI. Stracture analysis X-ray crystallography of compound 2m (CCDC: 1518182)

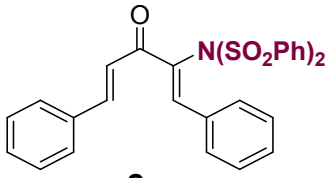


Crystal data and structure refinement for ***(E)*-2m**

Compound Empirical formula	(*E*)-2r $C_{30}H_{22}F_3NO_5S_2$
Formula weight Crystal system	597.62 Monoclinic
Space group Hall group	*P* 21 *P* 2ac 2ab
Temperature (K) Bond precision C-C (Å)	293 0.0082
Wavelength *a* (Å)	0.71069 8.086 (5)
b (Å) *c* (Å)	14.196 (5) 23.304 (5)
α (º) β (º)	90 90
γ (º) V (Å^3)	90 2675 (2)
Z D/g cm^{-3}	4 1.476
μ/mm^{-1} *F* (000)	0.263 1220.0
h, k, l_{max} Nref	9, 16, 27 4728 [2702]
Data completeness Theta (max)	1.75 / 1.00 25.000
R(reflections) *w*R_2(reflections)	0.0640 (3493) 0.1534 (4725)
S Npar	1.030 370

VII. Characterization data of New compounds

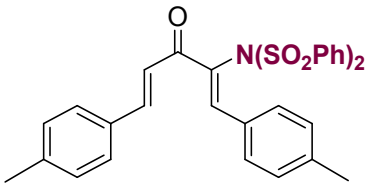
1. Characterization data of compound 2



2a

***N*-((*1Z,4E*)-3-oxo-1,5-diphenylpenta-1,4-dien-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (2a)**

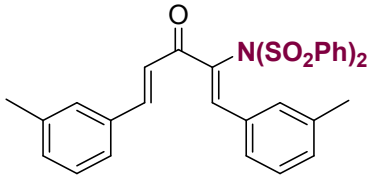
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (103.0 mg, 78%); mp: 209 – 210 ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.05 (d, J = 15.6 Hz, 1H), 7.13 (t, J = 8.0 Hz, 2H), 7.15 – 7.26 (m, 3H), 7.35 – 7.39 (m, 8H), 7.50 (t, J = 7.6 Hz, 2H), 7.62 (t, J = 6.4 Hz, 3H), 7.98 (d, J = 6.8 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 120.9, 128.5, 128. 6, 128.7, 128.8, 129.2, 129.6, 130.6, 131.2, 131.3, 131.6, 134.1, 134.5, 139.4, 144.7, 145.0, 186.7. HRMS (ESI-TOF) calcd for $C_{29}H_{23}NNaO_5S_2$, $[M+Na]^+$ 552.0915 Found 552.0917.



2b

***N*-((*1Z,4E*)-3-oxo-1,5-di-*p*-tolylpenta-1,4-dien-2-yl)-(phenylsulfonyl)benzenesulfonamide (2b)**

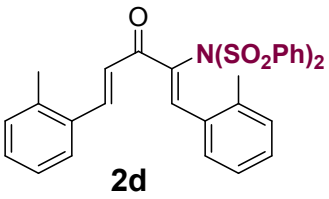
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (84.94 mg, 61%); mp: 209 – 210 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.30 (s, 3H), 2.38 (s, 3H), 6.93 (d, J = 8.0 Hz, 2H), 7.00 (d, J = 15.5 Hz, 1H), 7.14 (d, J = 8.0 Hz, 2H), 7.25 (d, J = 8.5 Hz, 3H), 7.37 (t, J = 7.5 Hz, 4H), 7.49 – 7.53 (m, 4H), 7.57 (d, J = 15.5 Hz, 1H), 7.97 (t, J = 7.5 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 21.5, 21.6, 120.0, 128.4, 128.5, 128.6, 128.8, 128.9, 129.2, 129.6, 131.4, 133.9, 134.1, 139.1, 141.1, 142.1, 144.6, 145.1, 186.8. HRMS (ESI-TOF) calcd for $C_{31}H_{27}NaNO_5S_2$, $[M+H]^+$ 558.1228 Found 558.1218.



2c

***N-((1Z,4E)*-3-oxo-1,5-di-*m*-tolylpenta-1,4-dien-2-yl)-*N*-(phenylsulfonyl)benzenesulfona mide (2c)**

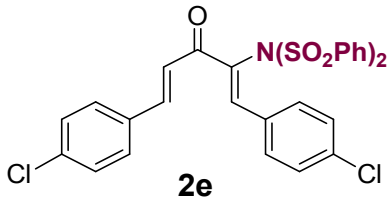
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (66.83 mg, 48%); mp: 210 – 211 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.15 (s, 3H), 2.40 (s, 3H), 7.06 – 7.10 (m, 3H), 7.19 – 7.29 (m, 4H), 7.41 (t, J = 15.5 Hz, 5H), 7.48 (s, 1H), 7.53 (t, J = 7.5 Hz, 2H), 7.63 (d, J = 15.5 Hz, 1H), 7.98 – 8.03 (m, 5H) ^{13}C NMR (125 MHz; $CDCl_3$): δ = 21.2, 21.3, 120.8, 125.9, 128.4, 128.5, 128.6, 128.8, 128.9, 129.0, 129.2, 129.6, 130.8, 131.4, 132.3, 134.0, 134.5, 138.2, 138.3, 139.5, 144.9, 145.1, 186.8. HRMS (ESI-TOF) calcd for $C_{31}H_{27}NNaO_5S_2$, $[M+Na]^+$ 580.1228 Found 580.1241.



2d

***N-((1Z,4E)*-3-oxo-1,5-di-o-tolylpenta-1,4-dien-2-yl)-*N*-(phenylsulfonyl)benzenesulfonami de (2d)**

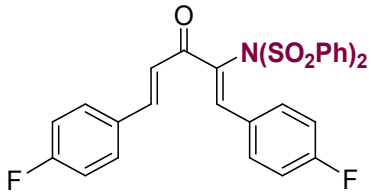
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (78.0 mg, 56%); mp: 206 – 207 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.41 (s, 3H), 2.45 (s, 3H), 6.94 (t, J = 5.0 Hz, 1H), 7.07 (d, J = 15.0 Hz, 1H), 7.20 – 7.24 (m, 4H), 7.28-7.39 (m, 6H), , 7.49 (t, J = 5.0 Hz, 2H), 7.81 (d, J = 5.0 Hz, 1H), 7.86 (d, J = 15.0 Hz, 4H), 7.94 (d, J = 15.0 Hz, 1H), 8.16 (s, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 19.8, 20.2, 121.8, 125.4, 125.9, 126.2, 126.3, 128.4, 129.1, 130.1, 131.3, 132.6, 133.3, 133.8, 134.0, 138.1, 138.3, 139.2, 140.3, 142.4, 143.4, 145.2, 186.8. HRMS (ESI-TOF) calcd for $C_{31}H_{28}NO_5S_2$, $[M+H]^+$ 558.1409 Found 558.2350.



2e

***N-((1Z,4E)*-1,5-bis(4-chlorophenyl)-3-oxopenta-1,4-dien-2-yl)-*N*-(phenylsulfonyl)benzenesu lfonamide (2e)**

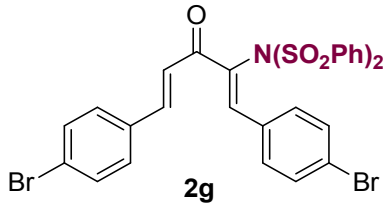
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (108.9 mg, 73%); mp: 219 – 220 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.95 (d, J = 15.5 Hz, 1H), 7.08 (t, J = 8.0Hz, 1H), 7.25 -7.30 (m, 5H), 7.35 (d, J = 8.0 Hz, 1H), 7.40 (t, J = 7.5 Hz, 3H), 7.45 (d, J = 7.5 Hz, 1H), 7.91 – 8.01 (m, 4H), 7.91 (s, 1H), 8.01 (d, J = 7.5 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ =121.7, 127.0, 127.5, 128.6, 129.3, 129.5, 129.9, 130.3, 130.4, 133.1, 134.3, 134.4, 136.0, 138.9, 143.1, 143.2, 186.1. HRMS (ESI-TOF) calcd for $C_{29}H_{21}Cl_2NO_5S_2$, $[M+H]^+$ 598.0316 Found 598.0465.



2f

***N*-((*1Z,4E*)-1,5-bis(4-fluorophenyl)-3-oxopenta-1,4-dien-2-yl)-*N*-(phenylsulfonyl)benzene sulfonamide (2f)**

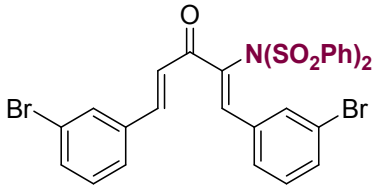
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (114.4 mg, 81%); mp: 220 – 222 ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.80 (t, J = 8.4 Hz, 2H), 6.91 (d, J = 15.6 Hz, 1H), 7.03 (t, J = 8.4 Hz, 2H), 7.26 – 731 (m, 2H), 7.39 (t, J = 8.4 Hz, 4H), 7.51 – 7.59 (m, 3H), 7.58 – 7.60 (m, 2H), 7.95 – 8.00 (m, 5H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 115.7, 115.8, 115.9, 120.5, 128.6, 128.8, 129.1, 129.5, , 130.4, 130.5, 133.5, 133.7, 134.2, 139.3, 143.4, 143.5, 186.5. HRMS (ESI-TOF) calcd for $C_{29}H_{22}F_2NO_5S_2$, $[M+H]^+$ 566.0907 Found 566.1031.



2g

***N*-((*1Z,4E*)-1,5-bis(4-bromophenyl)-3-oxopenta-1,4-dien-2-yl)-*N*-(phenylsulfonyl)benzene sulfonamide (2g)**

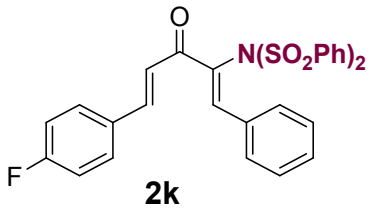
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (143.8 mg, 84%); mp: 210 – 213 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.95 (d, J = 15.5 Hz, 1H), 7.06 (d, J = 8.5 Hz, 2H), 7.23-7.25 (m, 3H), 7.31 (d, J = 8.5 Hz, 2H), 7.39 (t, J = 8.0 Hz, 4H), 7.49 (d, J = 8.5 Hz, 2H), 7.53 – 7.56 (m, 3H), 7.93 – 7.98 (m, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ=. 121.2, 128.7, 128.8, 128.9, 129.0, 129.1, 129.2, 129.5, 131.2, 132.3, 134.2, 136.6, 137.5, 139.2, 143.4, 143.5, 186.4. HRMS (ESI-TOF) calcd for $C_{29}H_{21}Br_2NNaO_5S_2$, $[M+Na]^+$ 707.9105 Found 707.9112.



2h

***N*-((*1Z,4E*)-1,5-bis(3-bromophenyl)-3-oxopenta-1,4-dien-2-yl)-*N*-(phenylsulfonyl)benzene sulfonamide (2h)**

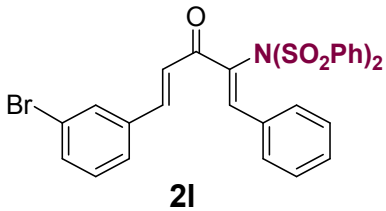
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (87.3 mg, 51%); mp: 210 – 212 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.81 (t, J = 8.5 Hz, 2H), 7.02 (d, J = 15.5 Hz, 1H), 7.33 – 7.40 (m, 6H), 7.52 (t, J = 7.5 Hz, 3H), 7.58 – 7.63 (m, 4H), 7.95 – 7.99 (m, 5H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 121.2, 124.2, 128.7, 128.8, 128.9, 129.0, 129.1, 129.5, 131.2, 132.3, 132.9, 134.2, 134.3, 136.6, 137.5, 139.2, 142.0, 143.4, 143.5, 144.2, 186.3. HRMS (ESI-TOF) calcd for $C_{29}H_{22}Br_2NNaO_5S_2$, $[M+Na]^+$ 707.9105 Found 707.9102.



2k

***N*-((*1Z,4E*)-5-(4-fluorophenyl)-3-oxo-1-phenylpenta-1,4-dien-2-yl)-*N*-(phenylsulfonyl)ben zenesulfonamide (2k)**

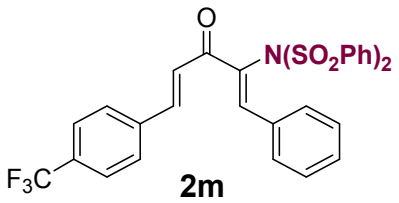
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (55.8 mg, 51%); mp: 204 – 205 ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.82 (t, J = 8.8 Hz, 2H), 7.01 (d, J = 15.6 Hz, 1H), 7.32 – 7.40 (m, 8H), 7.54 (t, J = 2.4 Hz, 3H), 7.58 – 7.63 (m, 4H), 7.95 – 7,99 (m, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 115.7, 115.9, 120.7, 120.8, 127.9, 128.6, 129.2, 129.6, 130.7, 133.6, 133.6, 134.2, 139.3, 139.4, 143.5, 144.8, 186.6. HRMS (ESI-TOF) calcd for $C_{29}H_{22}FNNaO_5S_2$, $[M+Na]^+$ 570.0821 Found 570.0804.



2l

***N*-((*1Z,4E*)-5-(3-bromophenyl)-3-oxo-1-phenylpenta-1,4-dien-2-yl)-*N*-(phenylsulfonyl)ben ze nesulfonamide (2l)**

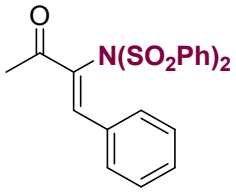
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (74.4 mg, 49%); mp: 207–208 ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.96 (d, J = 16.0 Hz, 1H), 7.13 (t, J = 8.0 Hz, 2H), 7.21 – 7.25 (m, 3H), 7.34 – 7.40 (m, 6H), 7.49 – 7.54 (m, 4H), 7.78 (d, J = 7.2 Hz, 2H), 7.98 – 8.00 (m, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 122.2, 122.8, 127.6, 128.5, 128.8, 129.5, 130.6, 131.4, 131.5, 133.3, 133.4, 134.0, 134.3, 136.6, 139.3, 142.8, 145.3, 186.5. HRMS (ESI-TOF) calcd for $C_{29}H_{22}BrNNaO_5S_2$, $[M+Na]^+$ 630.0020 Found 630.0104.



2m

***N-((1Z,4E)*-3-oxo-1-phenyl-5-(4-(trifluoromethyl)phenyl)penta-1,4-dien-2-yl)-*N*-(phenyl sulfonyl)benzenesulfonamide (2m)**

Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); yellow solid (89.6 mg, 60%); mp: 239 – 240 ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ =7.06 (s, 1H), 7.12 (t, J = 8.0 Hz, 3H), 7.25 (t, J = 4.8 Hz, 1H), 7.35 – 7.49 (m, 5H), 7.42 (2, J = 8.0 Hz, 2H), 7.47 – 7.51 (m, 2H), 7.57 – 7.60 (m, 4H), 7.97 – 7.99 (m, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 123.3, 125.6, 125.7, 128.2, 128.5, 128.6, 128.7, 129.6, 130.6, 131.4, 131.6, 132.0, 134.2, 137.8, 139.3, 142.5, 145.5, 186.6. HRMS (ESI-TOF) calcd for $C_{30}H_{23}F_3NO_5S_2$, $[M+H]^+$ 598.0970 Found 598.0256.

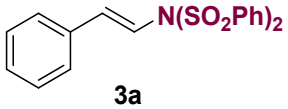


2n

(*Z*)-*N*-(3-oxo-1-phenylbut-1-en-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (2n)

Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:2); Yellow solid (29.9 mg, 34%); mp: 195 – 200 °C^{1}H NMR (600 MHz, $CDCl_3$): δ =2.39 (s, 3H), 7.16 (t, J = 7.8 Hz, 2H), 7.27 (t, J = 7.2 Hz, 1H), 7.40 (t, J = 7.8 Hz, 4H), 7.56 (t, J = 7.2 Hz, 2H), 7.61 (d, J = 7.8 Hz, 2H), 7.75 (s, 1H), 7.91 (d, J = 7.8 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 25.8, 128.4, 128.5, 129.5, 131.1, 131.2, 131.3, 131.5, 134.0, 139.5, 145.9, 194.3. HRMS (ESI-TOF) calcd for $C_{22}H_{19}NNaO_5S_2$, $[M+Na]^+$ 464.0602 Found 464.0600.

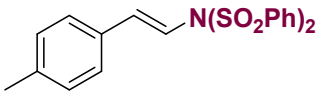
2. Characterization data of compound 3



3a

(*E*)-*N*-(phenylsulfonyl)-*N*-styrylbenzenesulfonamid (3a)

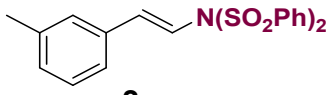
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (64.6 mg, 81%); mp: 171 – 172 °C; ^{1}H NMR (400 MHz; $CDCl_3$): δ = 6.53 (d, J = 13.6 Hz,1H), 6.69 (d, J = 13.6 Hz, 1H), 7.34 – 7.37 (m, 5H), 7.56 (t, J = 8.0 Hz, 4H), 7.68 (t, J = 7.6 Hz, 2H), 8.00 (dd, J_{12} = 1.2 Hz, J_{13} = 8.8 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 119.4, 127.2, 128.1, 128.8, 129.1, 129.4, 133.7, 134.0, 139.1, 139.4. HRMS (ESI-TOF) calcd for $C_{20}H_{17}NNaO_4S_2$, $[M+Na]^+$ 422.0497 Found 422.0502.



3b

(*E*)-*N*-(4-methylstyryl)-*N*-(phenylsufonyl)benzenesulfonamide (3b)

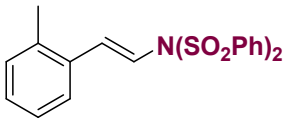
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (68.40 mg, 76%); mp: 134 – 135°C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.36 (s, 3H), 6.46 (d, J = 13.6 Hz, 1H), 6.64 (d, J = 14.0 Hz, 1H), 7.15 (d, J = 7.6 Hz, 2H), 7.25 (d, J = 7.2 Hz, 2H), 7.56 (t, J = 8.0 Hz, 4H), 7.67 (t, J = 7.2 Hz, 2H), 7.99 (d, J = 8.0 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 21.3, 118.3, 127.2, 128.2, 129.0, 129.5, 130.9, 133.9, 139.3, 139.5, 139.6. HRMS (ESI-TOF) calcd for $C_{21}H_{19}NNaO_4S_2$, $[M+Na]^+$ 436.0653 Found 436.0642.



3c

(*E*)-*N*-(4-methylstyryl)-*N*-(phenylsufonyl)benzenesulfonamide (3c)

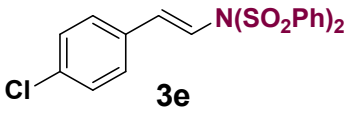
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (58.0 mg, 70%); mp: 132 – 134 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.35 (s, 3H), 6.51 (d, J = 13.6 Hz, 1H), 6.65 (d, J = 14.0 Hz, 1H), 7.15-7.19 (m, 3H), 7.24 (t, J = 6.4 Hz, 1H), 7.57 (t, J = 8.0 Hz, 4H), 7.65 – 7.69 (m, 2H), 8.00 (d, J = 7.2 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 21.3, 119.1, 124.4, 127.8, 128.0, 128.1, 128.7, 129.1, 133.6, 133.9, 136.0, 138.5, 139.3. HRMS (ESI-TOF) calcd for $C_{21}H_{19}NNaO_4S_2$, $[M+Na]^+$ 436.0653 Found 436.0651.



3d

(*E*)-*N*-(2-methylstyryl)-*N*-(phenylsufonyl)benzenesulfonamide (3d)

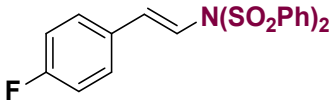
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (53.7 mg, 65%); mp: 130 – 132 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.50 (s, 3H), 6.4 (d, J = 13.6 Hz, 1H), 7.16 (d, J = 13.6 Hz, 1H), 7.44 – 7.52 (m, 3H), 7.66 (d, J = 6.8 Hz, 1H), 7.84 (t, J = 7.2 Hz, 4H), 7.95 (t, J = 7.2 Hz, 2H), 8.29 (d, J = 7.2 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 19.7, 120.3, 126.2, 126.3, 128.1, 129.1, 129.2, 130.5, 132.8, 133.9, 136.6, 137.9, 139.5. HRMS (ESI-TOF) calcd for $C_{21}H_{20}NO_4S_2$, $[M+H]^+$ 414.0828 Found 414.0837.



3e

(*E*)-*N*-(4-chlorostyryl)-*N*-(phenylsufonyl)benzenesulfonamide (3e)

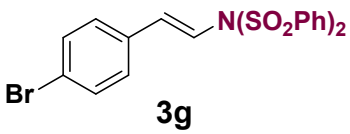
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (61.14 mg, 65%); mp: 154 – 155 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.51 (d, J = 13.6 Hz, 1H), 6.65 (d, J = 13.6 Hz, 1H), 7.56 – 7.33 (m, 4H), 7.67 (t, J = 8.0 Hz, 4H), 7.69 (t, J = 7.2 Hz, 2H), 7.99 (d, J = 7.6 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 120.0, 128.1, 128.4, 129.0, 129.1, 132.2, 134.1, 135.2, 137.4, 139.4. HRMS (ESI-TOF) calcd for $C_{20}H_{16}ClNNaO_4S_2$, $[M+Na]^+$ 456.0107 Found 456.0496.



3f

(*E*)-*N*-(4-fluorostyryl)-*N*-(phenylsufonyl)benzenesulfonamide (3f)

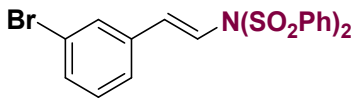
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (51.7 mg, 62%); mp: 156 – 158 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ= 6.45 (d, J = 13.6 Hz, 1H), 6.65 (d, J = 14.0 Hz, 1H), 7.04 (t, J = 8.4 Hz, 2H), 7.32 – 7.35 (m, 2H), 7.57 (t, J = 8.0 Hz, 4H), 7.69 (t, J = 7.2 Hz, 2H), 7.99 (d, J = 7.2 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 115.8, 116.0, 119.1, 128.2, 128.9, 129.1, 134.0, 137.9, 139.4, 141.9 HRMS (ESI-TOF) calcd for $C_{20}H_{16}FNNaO_4S_2$, $[M+Na]^+$ 440.0402 Found 440.0417.



3g

(*E*)-*N*-(4-bromostyryl)-*N*-(phenylsufonyl)benzenesulfonamide (3g)

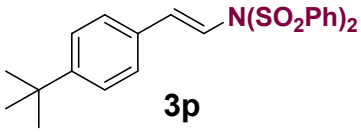
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (65.1 mg, 68%); mp: 153 – 154 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6. 53 (d, *J* = 13.6 Hz, 1H), 6.64 (d, *J* = 13.6 Hz, 1H), 7.22 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.4 Hz, 2H), 7.57 (t, *J* = 8.4 Hz, 4H), 7.67 (t, *J* = 7.2 Hz, 2H), 7.99 (d, *J* = 7.2 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): *δ* = 119.4, 127.2, 128.2, 128.8, 129.1, 129.4, 133.7, 134.0, 139.1, 139.5. HRNS (ESI-TOF) calcd for $C_{20}H_{17}BrNO_4S_2$, $[M+H]^+$ 477.9777; Found 477.9781



3h

(*E*)-*N*-(3-bromostyryl)-*N*-(phenylsufonyl)benzenesulfonamide (3h)

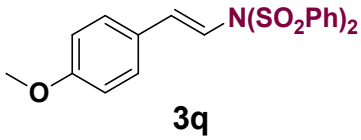
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (54.3 mg, 57%); mp: 150 – 152 °C; ^{1}H NMR (400 MHz, $CDCl_3$): *δ* = 6.56 (d, *J* = 14.0 Hz, 1H), 6.65 (d, *J* = 14.0 Hz, 1H), 7.22 – 7.27 (m, 2H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.51 (s, 1H), 7.58 (t, *J* = 8.0 Hz, 4H), 7.69 (t, *J* = 7.2 Hz, 2H), 8.00 (dd, J_{12} = 1.6 Hz, J_{13} = 8.0 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): *δ* = 120.8, 122.9, 125.8, 128.1, 129.1, 129.8, 130.3, 132.1, 134.1, 135.8, 136.8, 139.3. HRMS (ESI-TOF) calcd for $C_{20}H_{16}BrNNaO_4S_2$, $[M+Na]^+$ 499.9596 Found 499.9587.



3p

(*E*)-*N*-(4-(tert-butyl)styryl)-*N*-(phenylsufonyl)benzenesulfonamide (3p)

Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (66.4 mg, 73%); mp: 136 – 138 °C; ^{1}H NMR (400 MHz, $CDCl_3$): *δ* = 1.32 (s, 9H), 6.49 (d, *J* = 13.6 Hz, 1H), 6.66 (d, *J* = 13.6 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 2H), 7.57 (t, *J* = 8.0 Hz, 4H), 7.64 – 7.69 (m, 2H), 8.00 (dd, J_{12} = 1.6 Hz, J_{13} = 8.0 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): *δ* = 31.2, 34.8, 118.5, 125.7, 127.0, 128.1, 129.1, 130.9, 133.9, 139.1, 139.5, 152.8. HRMS (ESI-TOF) calcd for $C_{24}H_{26}NO_4S_2$, $[M+H]^+$ 456.1298 Found 456.1291.

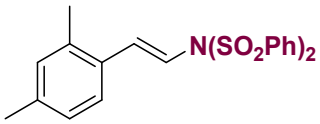


3q

***(E)*-*N*-(4-methoxystyryl)-*N*-(phenylsufonyl)benzenesulfonamide (3q)**

Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (26.6 mg, 31%); mp: 129 – 131°C; ^{1}H NMR (400 MHz, $CDCl_3$): *δ* = 3.82 (s, 3H), 6.37 (d, *J* = 13.6 Hz, 1H), 6.60 (d, *J* = 13.6 Hz, 1H), 6.87 (d, *J* = 8.8 Hz, 2H), 7.30 (d, *J* = 8.8 Hz, 2H), 7.56

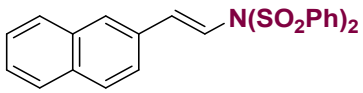
(t, J = 8.0 Hz, 4H), 7.67 (t, J = 7.2 Hz, 2H), 7.99 (d, J = 7.6 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 55.4, 114.2, 117.0, 126.3, 128.2, 128.7, 129.1, 133.9, 139.3, 139.5, 160.5. HRMS (ESI-TOF) calcd for $C_{21}H_{19}NNaO_5S_2$, $[M+Na]^+$ 452.0602 Found 452.0813.



3r

(*E*)-*N*-(2,4-dimethylstyryl)-*N*-(phenylsufonyl)benzenesulfonamide (3r)

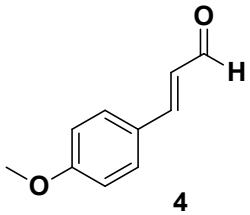
Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (66.6 mg, 78%); mp: 152–153°C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.18 (s, 3H), 2.31 (s, 3H), 6.33 (d, J = 13.6 Hz, 1H), 6.84 (d, J = 13.6 Hz, 1H), 6.99 (d, J = 5.6 Hz, 2H), 7.29 (d, J = 8.0 Hz, 1H), 7.53 – 7.57 (m, 4H), 7.64 – 7.69 (m, 2H), 8.00 (d, J = 7.2 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 19.6, 21.2, 119.3, 126.2, 126.9, 128.1, 129.1, 129.9, 131.3, 133.9, 136.5, 138.0, 139.4, 139.5. HRMS (ESI-TOF) calcd for $C_{22}H_{22}NO_4S_2$, $[M+H]^+$ 428.0985 Found 428.0977.



3s

(*E*)-*N*-(2-(naphthalen-2-yl)vinyl)-*N*-(phenylsufonyl)benzenesulfonamide (3s)

Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); white solid (37.7 mg, 42%); mp: 128–129°C; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.65 (d, J = 13.5 Hz, 1H), 6.85 (d, J = 14.0 Hz, 1H), 7.49 – 7.59 (m, 7H), 7.67 (t, J = 7.5 Hz, 2H), 7.74 (s, 1H), 7.81-7.83 (m, 3H), 8.02 (d, J = 7.0 Hz, 4H)^{13}C NMR (125 MHz; $CDCl_3$): δ = 119.5, 123.3, 126.7, 126.9, 127.7, 128.2, 128.6, 129.1, 131.1, 133.2, 133.6, 134.0, 139.1, 139.5. HRMS (ESI-TOF) calcd for $C_{24}H_{19}NNaO_4S_2$, $[M+Na]^+$ 472.0653 Found 472.1061.



4

***(E)*-3-(4-methoxyphenyl)acrylaldehyde**

Purified by flash column chromatography (eluent: petroleum ether / EtOAc = 25:1); Yellow liquid (7.8 mg, 24%); ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.79 (s, 3H), 6.89 - 6.91 (m, 1H), 7.00 – 7.05 (m, 2H), 7.09 – 7.17 (m, 2H), 7.31 (t, J = 8.5 Hz, 1H), 9.54 (s, 1H). ^{13}C NMR (150 MHz;

$CDCl_3$): δ = 55.0, 114.8, 115.2, 122.3, 129.4, 136.2, 149.4, 159.4, 195.1HRMS (ESI-TOF) calcd for $C_{10}H_{10}NaO_2$, $[M+Na]^+$ 185.0578 Found 185.0542.

References:

(1) (a) C. Conard, M. Dolliver. *Org. Synth. Coll*, **1943**, 2, 167-168.

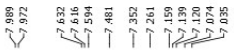
(2) L. A. Hull, *J.Chem. Ed*, **2001**,78 (2), 2264.

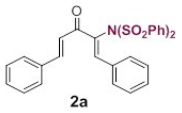
(3) R. V. Smerbeck, E. P. Pittz, (**1986**). U.S. Patent No. 4,587,260. Washington, DC: U.S. Patent and Trademark Office.2.

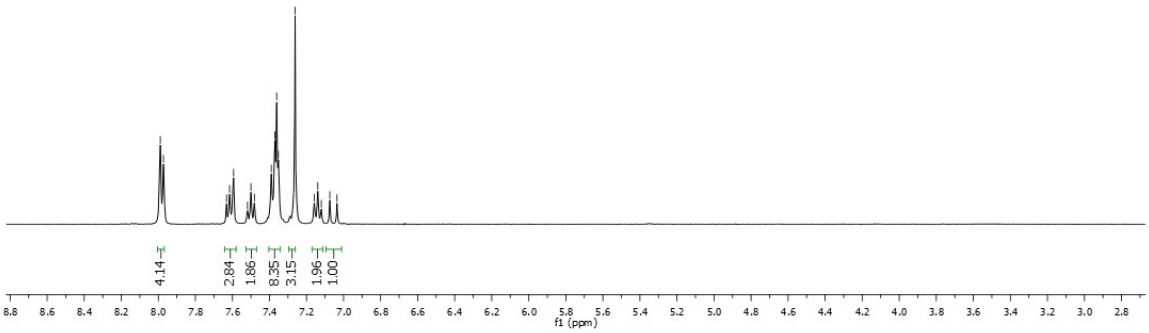
(4) Ajani O O, Ituen R I, Falomo A. *Pak. J. Sci. Ind. Res*, *Series A: Physical Sciences*, **2011**, 54(2), 59-67.

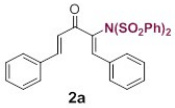
VIII. ^{1}H and ^{13}C Spectra of New Compound

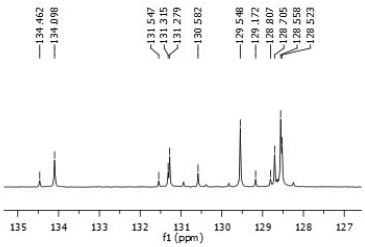
Compound 2a

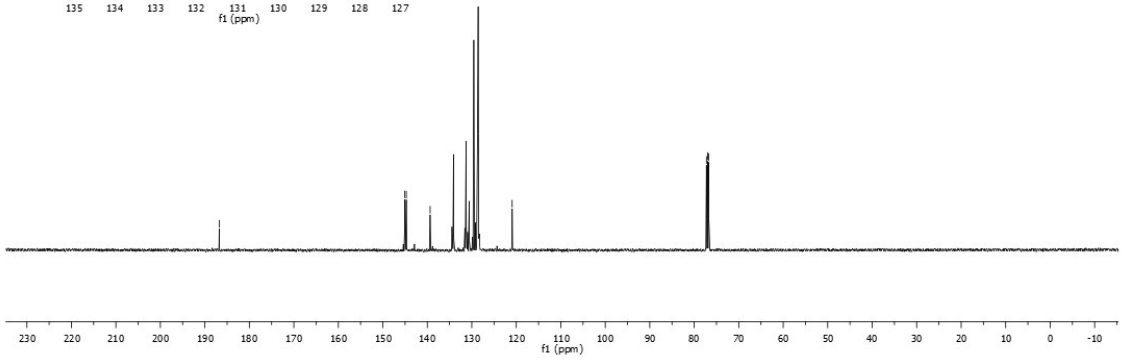




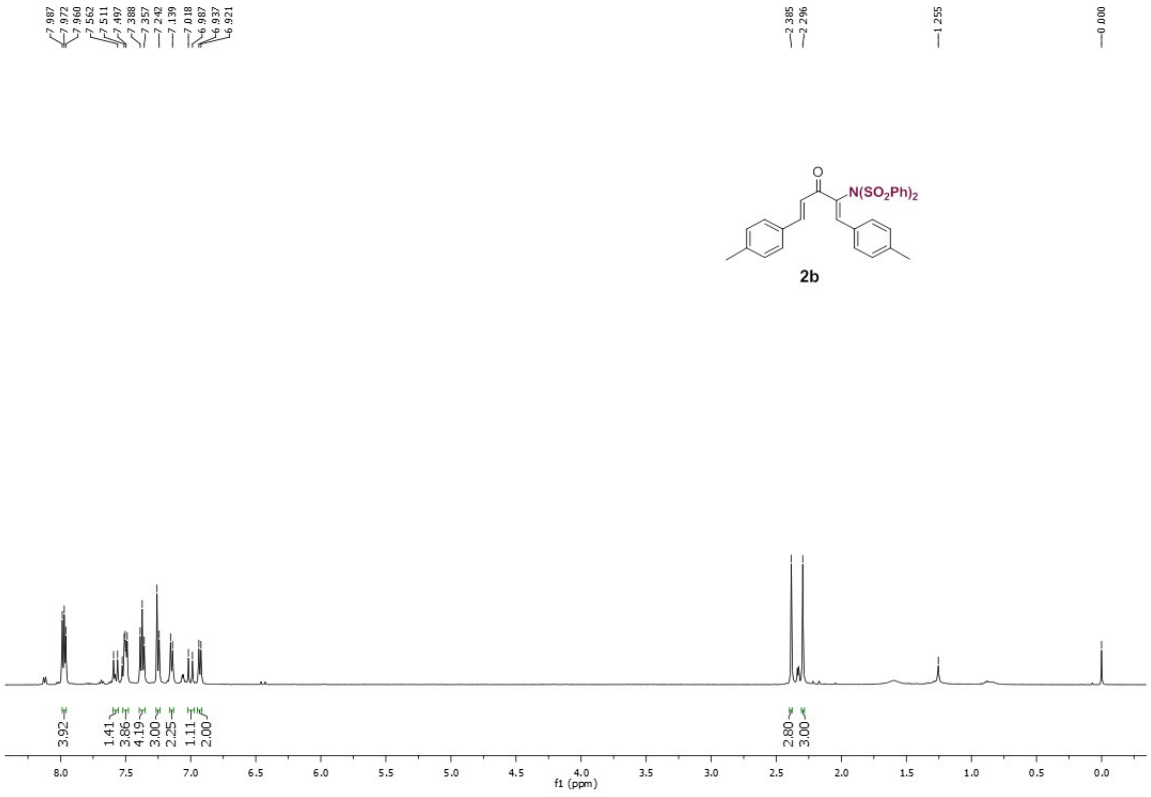


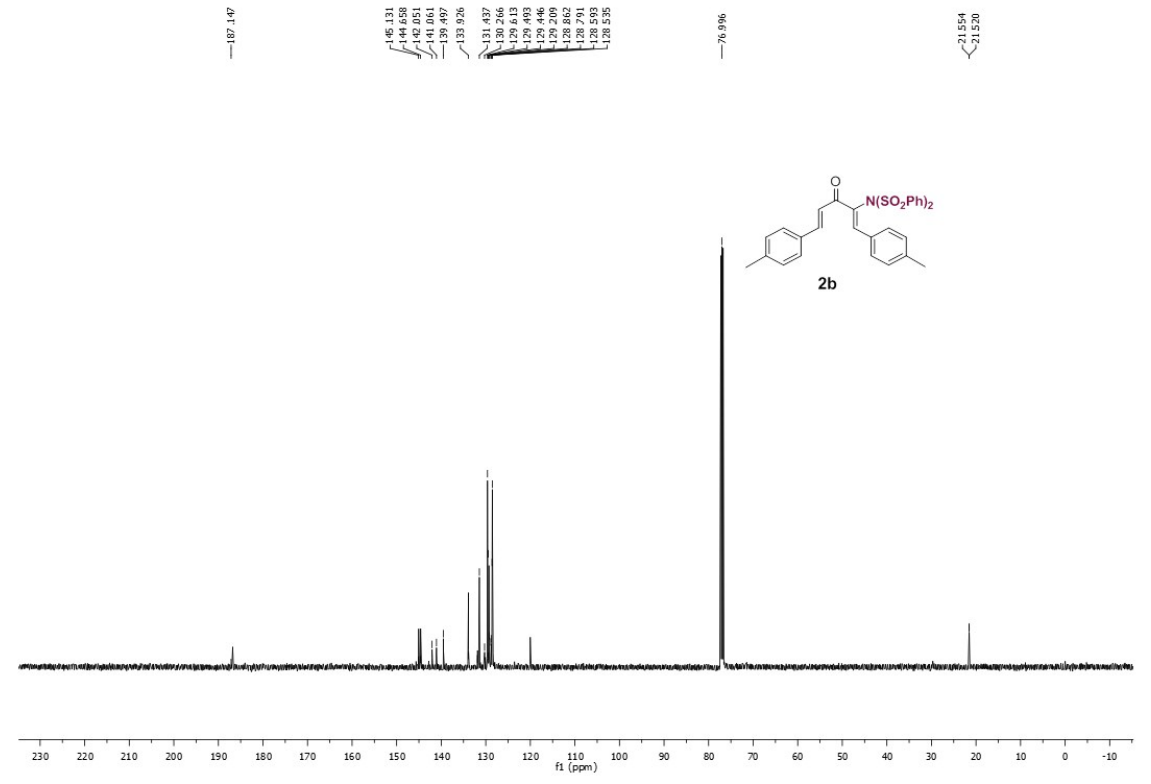




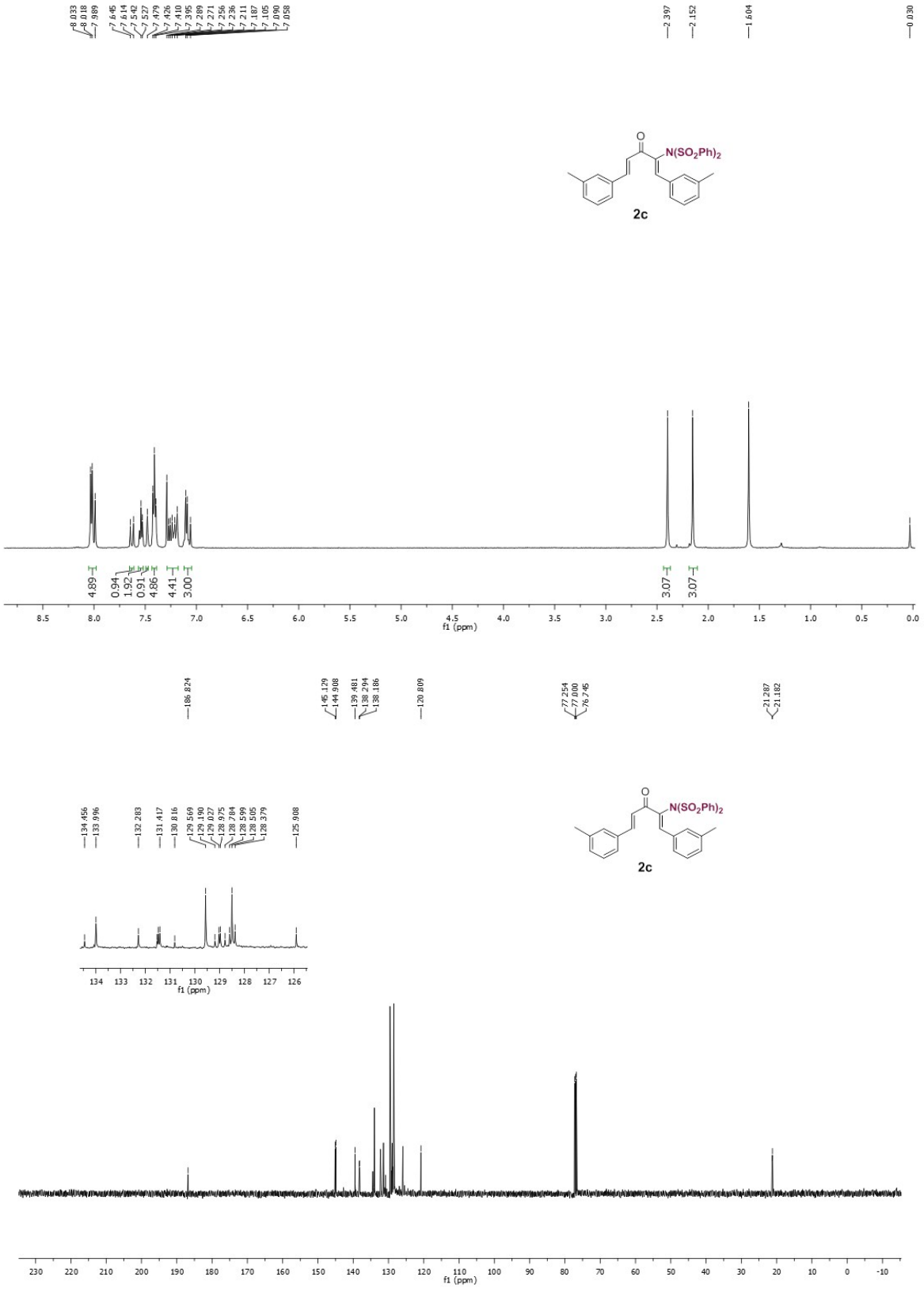


Compound 2b

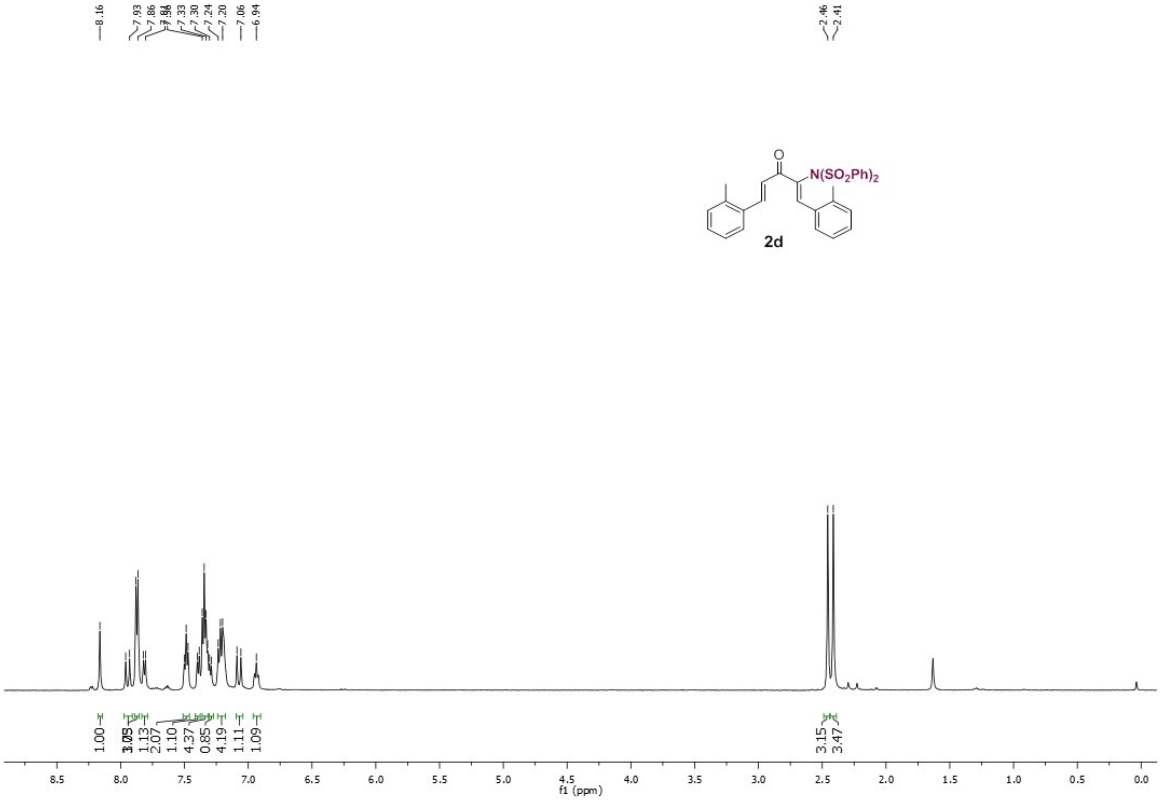

7.987
7.972
7.960
7.562
7.511
7.497
7.388
7.357
7.242
7.139
7.018
6.987
6.937
6.921
2.385
2.296
1.255
0.000
N(SO2Ph)2
2b
3.92
1.41
3.86
4.19
3.00
2.25
1.11
2.00
2.80
3.00
f1 (ppm)

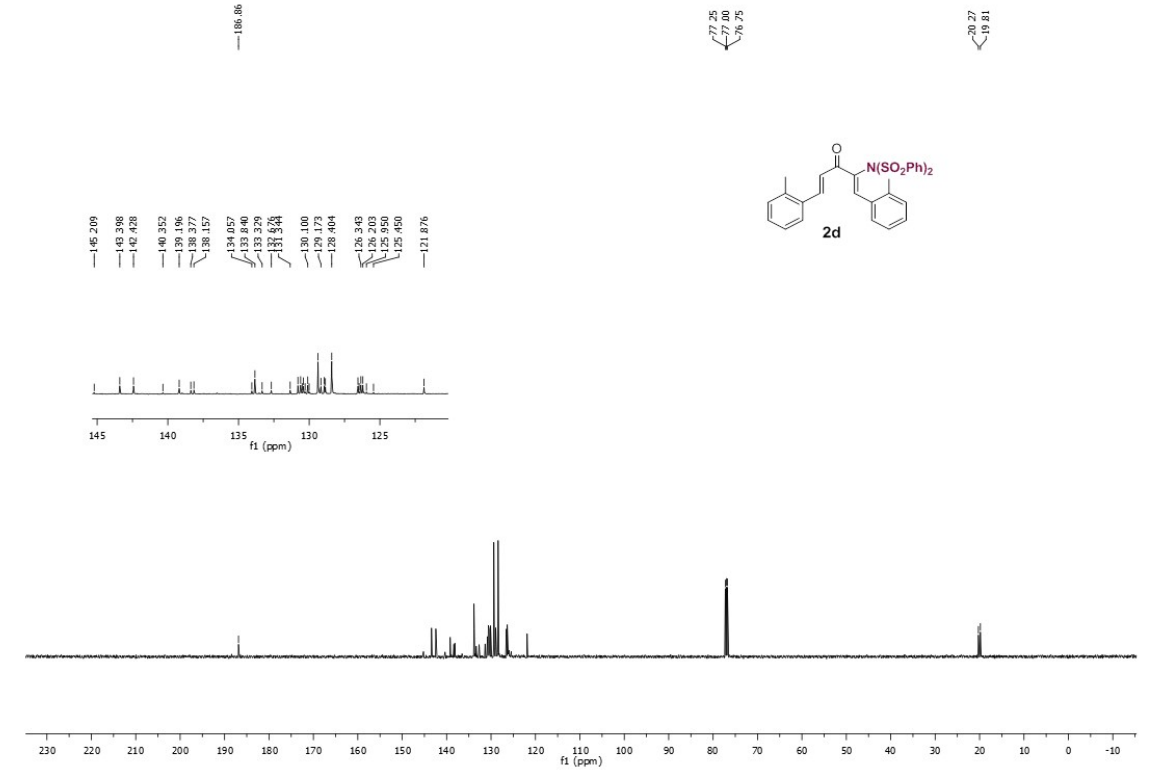

187.147
145.131
144.658
142.051
141.061
139.497
133.926
131.437
130.266
129.613
129.493
129.446
129.209
128.862
128.791
128.593
128.535
76.996
21.554
21.520
N(SO2Ph)2
2b
f1 (ppm)

Compound 2c

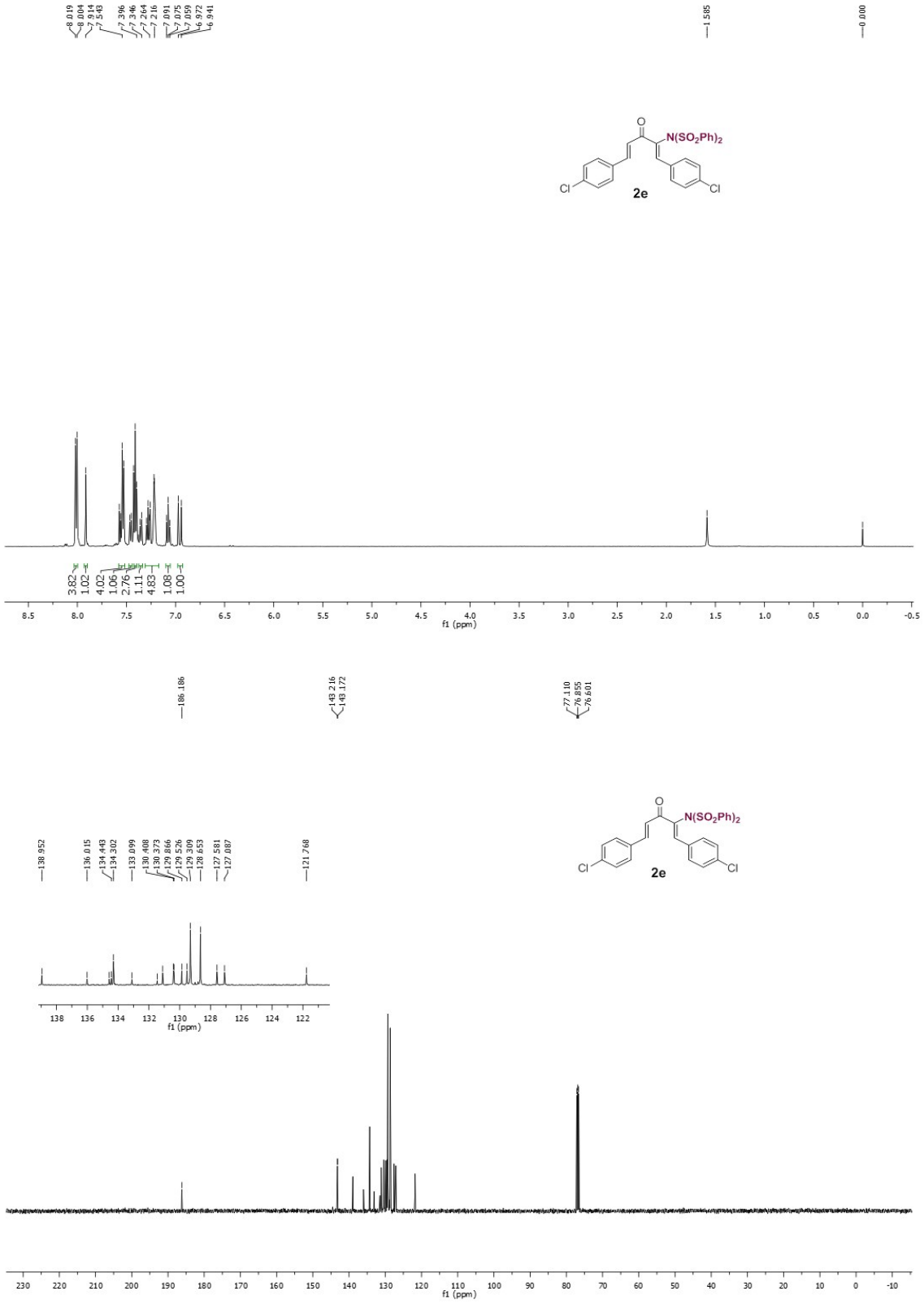

8.033
8.018
7.989
7.645
7.614
7.542
7.527
7.479
7.426
7.410
7.395
7.289
7.271
7.256
7.236
7.211
7.187
7.105
7.090
7.058
2.397
2.152
1.604
0.030
N(SO2Ph)2
2c
4.89
0.94
1.92
0.91
4.86
4.41
3.00
3.07
3.07
f1 (ppm)
186.824
145.129
144.908
139.481
138.294
138.186
120.809
77.254
77.000
76.745
21.287
21.182
134.456
133.996
132.283
131.417
130.816
129.569
129.190
129.027
128.975
128.784
128.599
128.505
128.379
125.908
N(SO2Ph)2
2c
f1 (ppm)
f1 (ppm)

Compound 2d

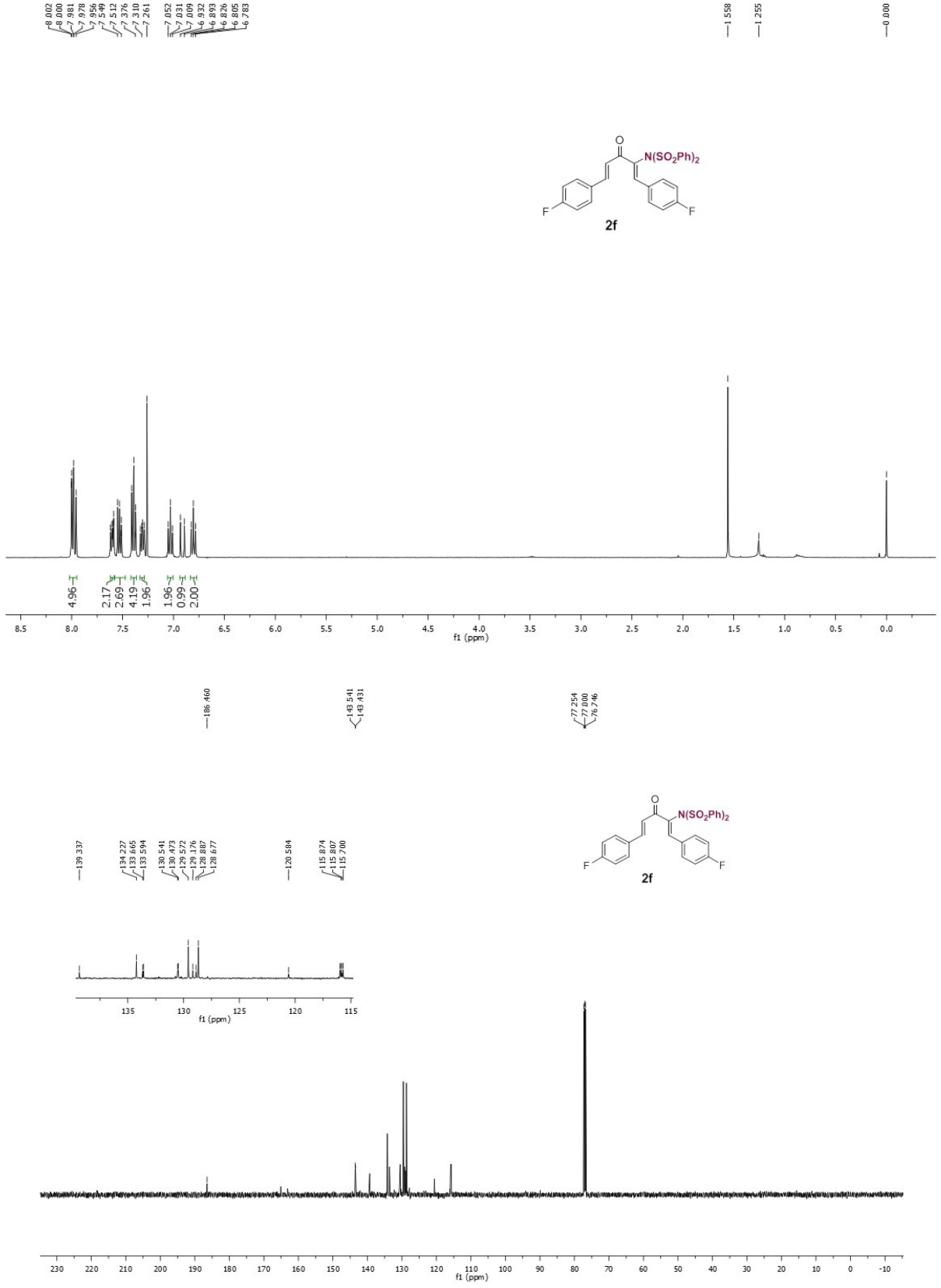

8.16
7.93
7.86
7.33
7.30
7.24
7.20
7.06
6.94
2.46
2.41
N(SO2Ph)2
2d
1.00
3.15
3.47
f1 (ppm)


186.86
77.25
77.00
76.75
20.27
19.81
N(SO2Ph)2
2d
145.209
143.398
142.428
140.352
139.196
138.377
138.157
134.057
133.840
133.329
130.100
129.173
128.404
126.343
126.203
125.950
125.450
121.876
f1 (ppm)

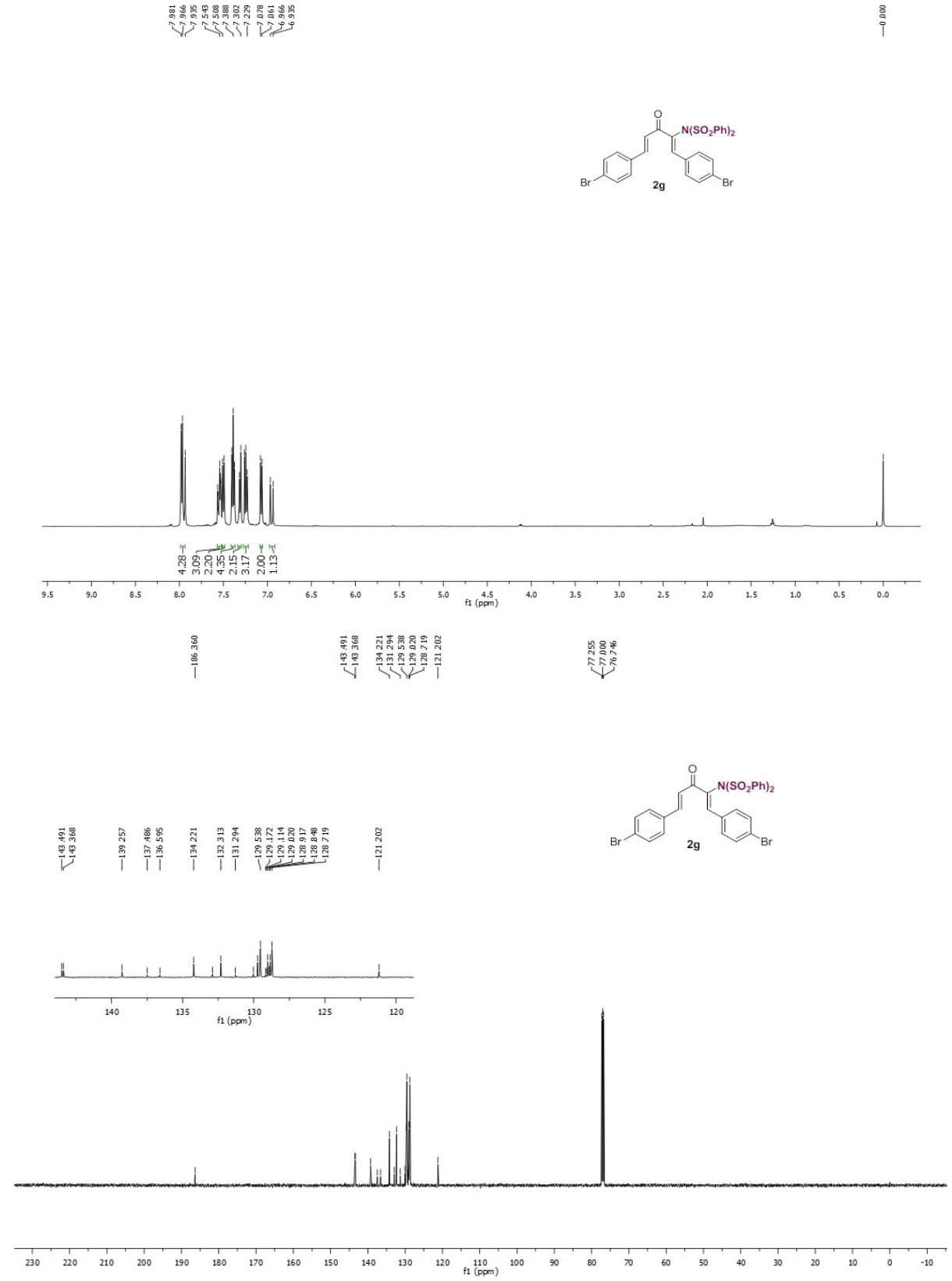
Compound 2e


8.019
8.004
7.914
7.543
7.396
7.346
7.264
7.216
7.091
7.075
7.059
6.972
6.941
1.585
0.000
N(SO2Ph)2
Cl
Cl
2e
3.82
1.02
4.02
1.06
2.76
1.11
4.83
1.08
1.00
f1 (ppm)
186.186
143.216
143.172
77.110
76.855
76.601
138.952
136.015
134.443
134.302
133.099
130.408
130.373
129.866
129.526
129.309
128.653
127.581
127.087
121.768
f1 (ppm)
f1 (ppm)

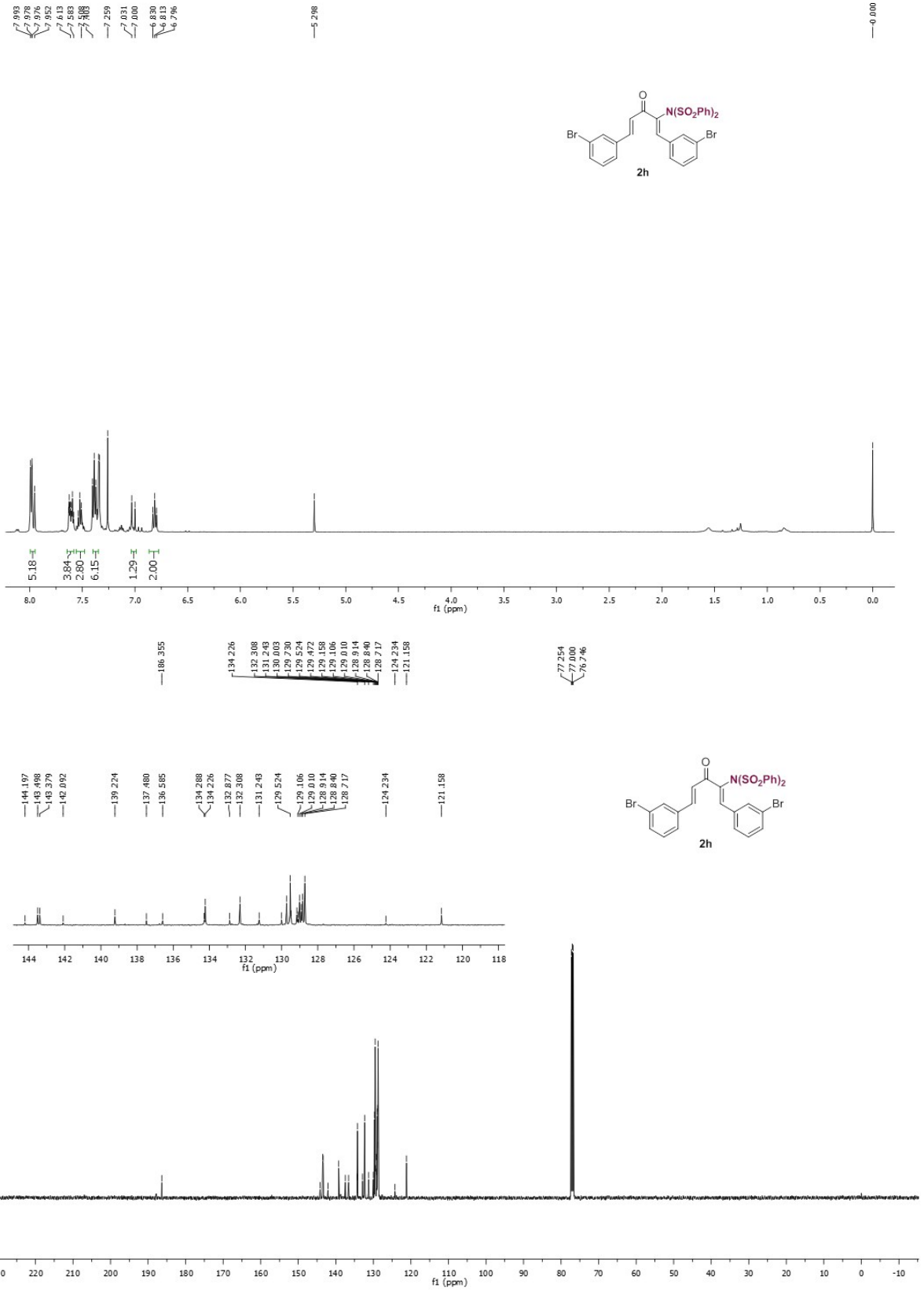
Compound 2f


8.002
8.000
7.981
7.978
7.956
7.549
7.512
7.376
7.310
7.261
7.052
7.031
7.009
6.932
6.893
6.826
6.805
6.783
1.558
1.255
0.000
N(SO2Ph)2
F
F
2f
4.96
2.17
2.69
4.19
1.96
1.96
0.99
2.00
f1 (ppm)
186.460
143.541
143.431
77.254
77.000
76.746
139.337
134.227
133.865
133.594
130.541
130.473
129.572
129.176
128.887
128.677
120.584
115.874
115.807
115.700
f1 (ppm)
f1 (ppm)

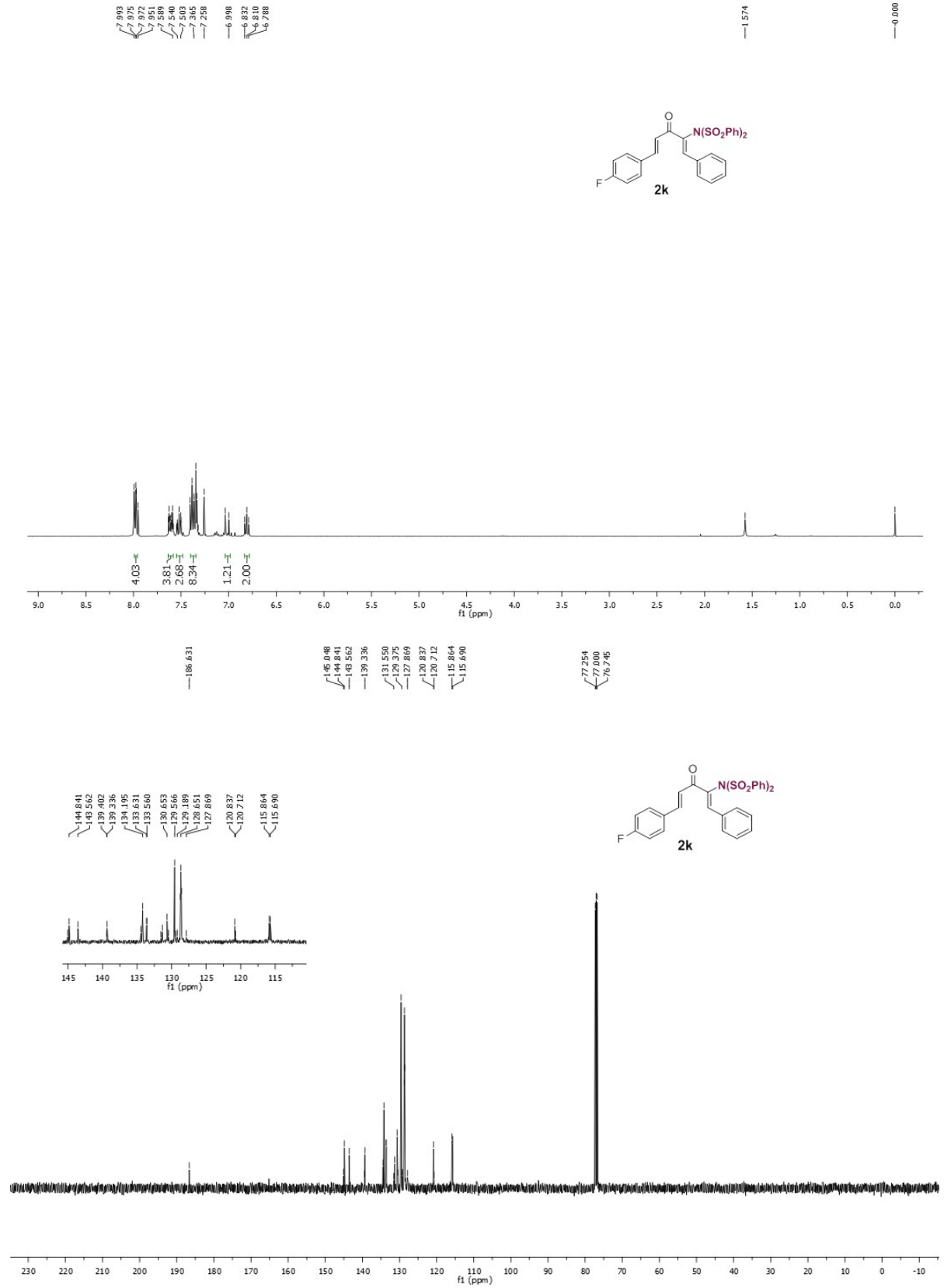
Compound 2g


7.981
7.966
7.935
7.548
7.508
7.388
7.302
7.299
7.078
7.061
6.966
6.935
0.000
N(SO2Ph)2
Br
Br
2g
4.28
3.09
2.20
4.35
2.15
3.17
2.00
1.13
f1 (ppm)
186.360
143.491
143.368
134.221
131.294
129.538
129.020
128.719
121.202
77.255
77.000
76.746
N(SO2Ph)2
Br
Br
2g
143.491
143.368
139.257
137.486
136.595
134.221
132.313
131.294
129.538
129.172
129.114
129.020
128.917
128.848
128.719
121.202
f1 (ppm)

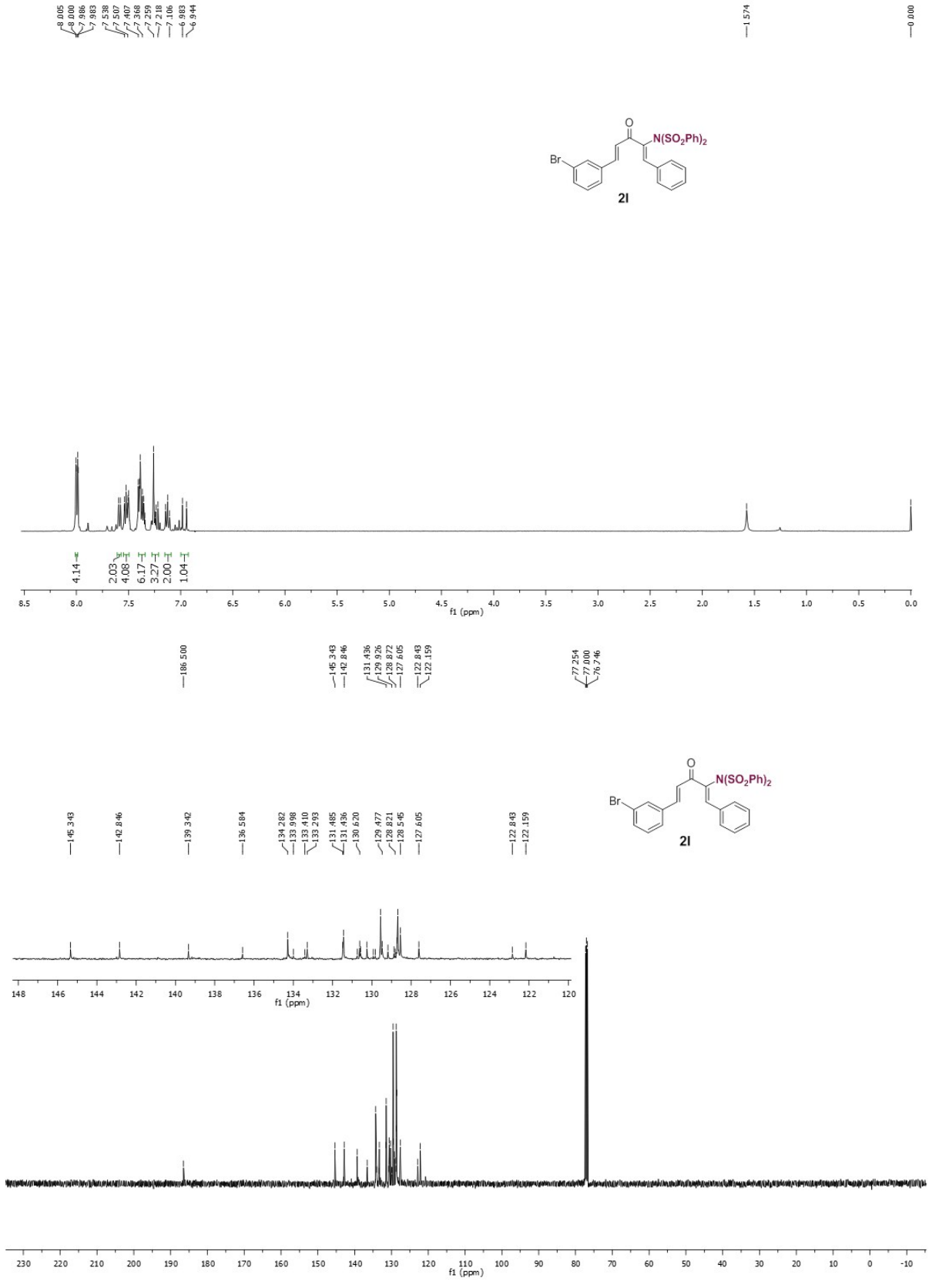
Compound 2h


7.993
7.978
7.976
7.952
7.613
7.583
7.508
7.403
7.259
7.031
7.000
6.830
6.813
6.796
5.298
0.000
O
N(SO2Ph)2
Br
Br
2h
5.18
3.84
2.80
6.15
1.29
2.00
f1 (ppm)
186.355
134.226
132.308
131.243
130.003
129.730
129.524
129.472
129.158
129.106
129.010
128.914
128.840
128.717
124.234
121.158
77.254
77.000
76.746
144.197
143.498
143.379
142.092
139.224
137.480
136.585
134.288
134.226
132.877
132.308
131.243
129.524
129.106
129.010
128.914
128.840
128.717
124.234
121.158
f1 (ppm)

Compound 2k

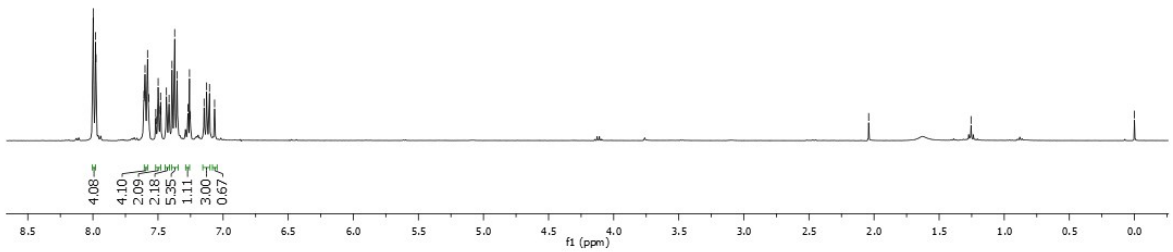

7.993
7.975
7.972
7.951
7.589
7.540
7.503
7.365
7.258
6.998
6.832
6.810
6.788
1.574
0.000
N(SO2Ph)2
F
2k
4.03
3.81
2.68
8.34
1.21
2.00
f1 (ppm)
186.631
145.048
144.841
143.562
139.336
131.550
129.375
127.869
120.837
120.712
115.864
115.690
77.254
77.000
76.745
144.841
143.562
139.402
139.336
134.195
133.631
133.560
130.653
129.566
129.189
128.651
127.869
120.837
120.712
115.864
115.690
N(SO2Ph)2
F
2k
f1 (ppm)
f1 (ppm)

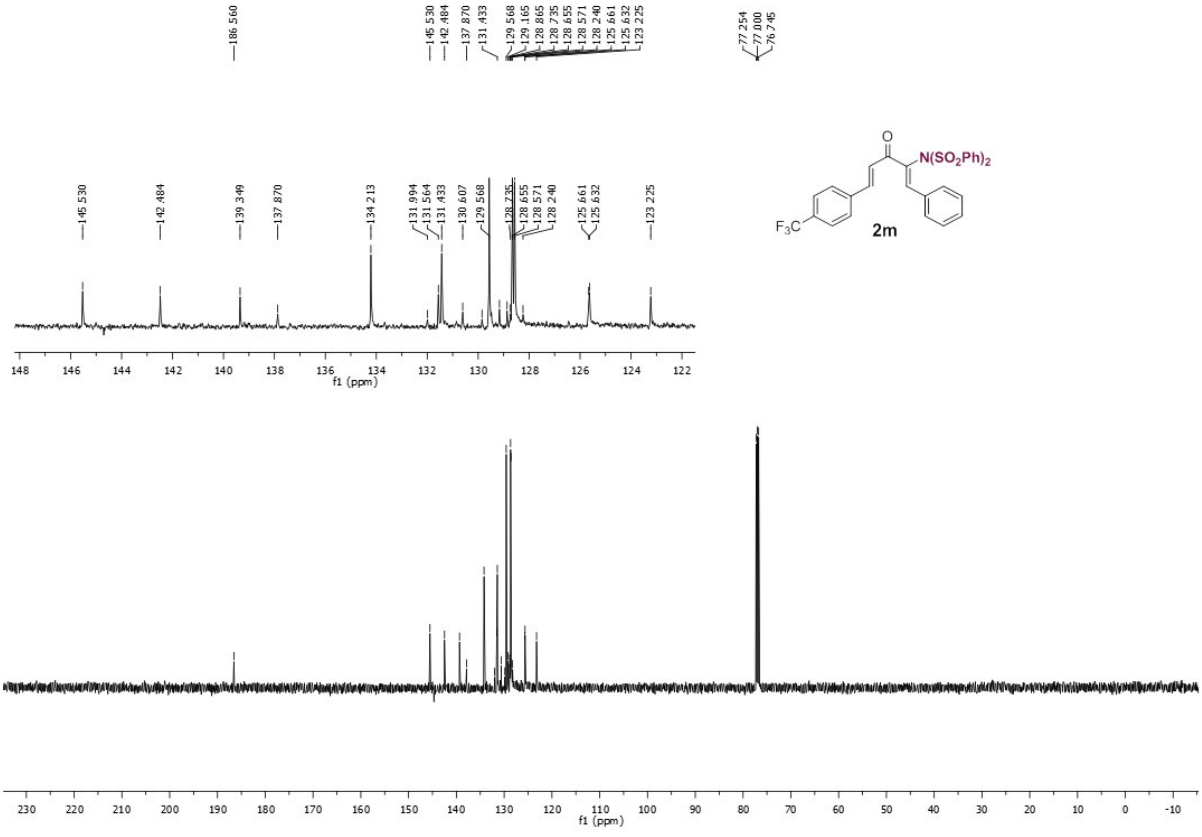
Compound 2l


8.005
8.000
7.986
7.983
7.538
7.507
7.407
7.368
7.259
7.218
7.106
6.983
6.944
1.574
0.000
O
N(SO2Ph)2
Br
2l
4.14
2.03
4.08
6.17
3.27
2.00
1.04
f1 (ppm)
186.500
145.343
142.846
131.436
129.926
128.872
127.605
122.843
122.159
77.254
77.000
76.746
145.343
142.846
139.342
136.584
134.282
133.998
133.410
133.293
131.485
131.436
130.620
129.477
128.821
128.545
127.605
122.843
122.159

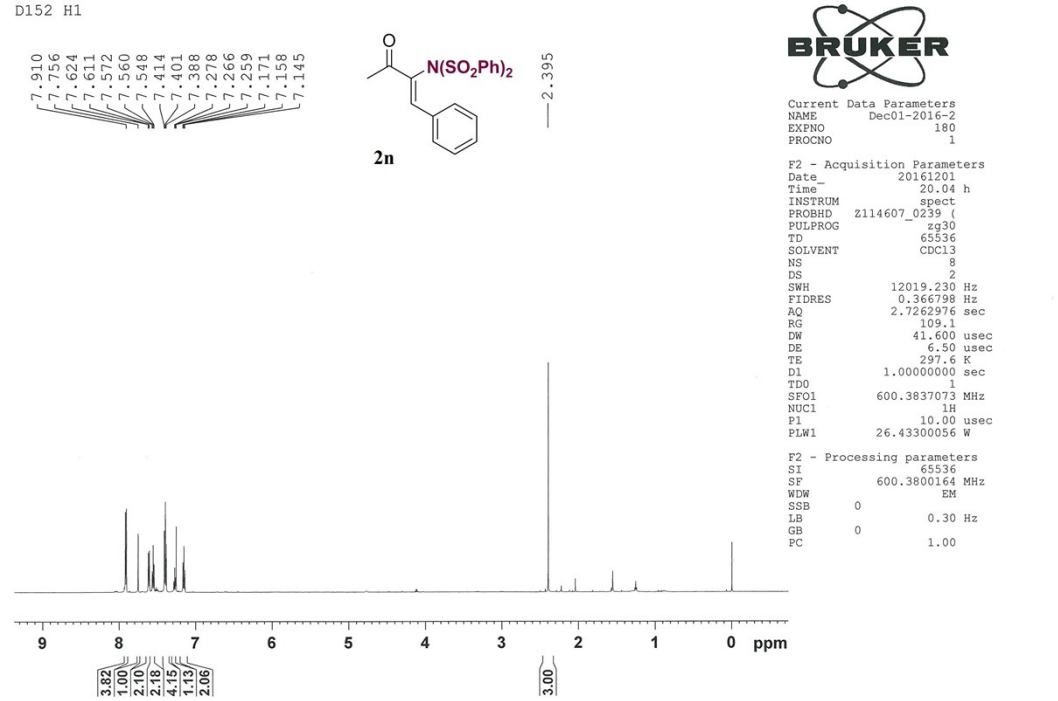
Compound 2m

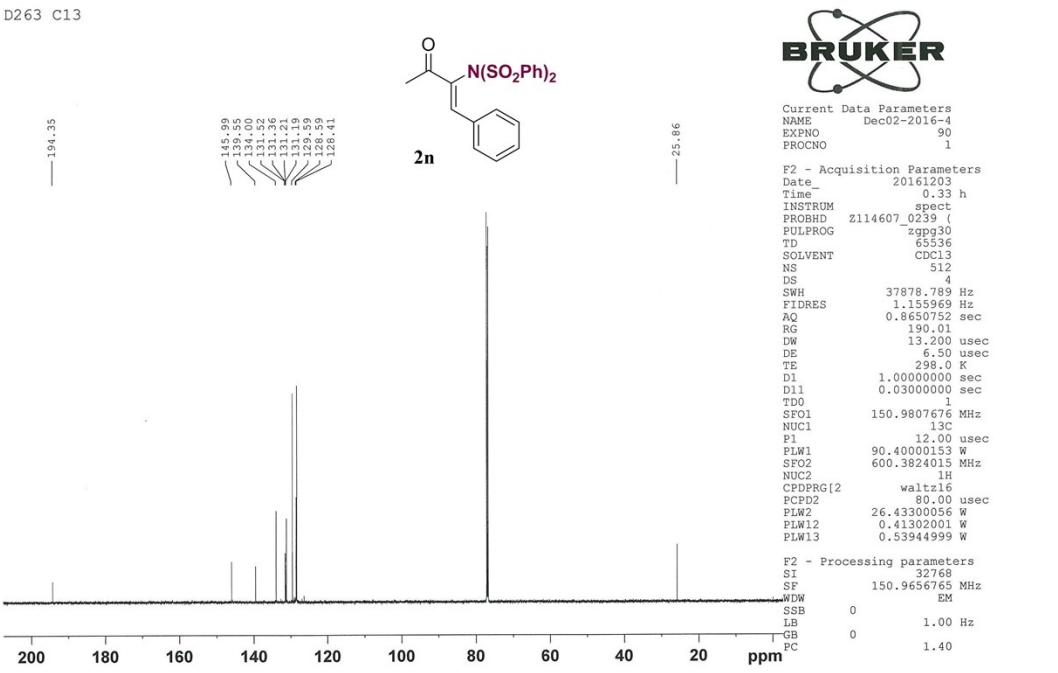

7.998
7.996
7.980
7.977
7.608
7.600
7.580
7.570
7.517
7.515
7.499
7.483
7.480
7.477
7.435
7.415
7.392
7.372
7.353
7.270
7.258
7.252
7.146
7.126
7.105
7.065
2.041
1.255
0.000
N(SO2Ph)2
F3C
2m


4.08
4.10
2.09
2.18
5.35
1.11
3.00
0.67
f1 (ppm)

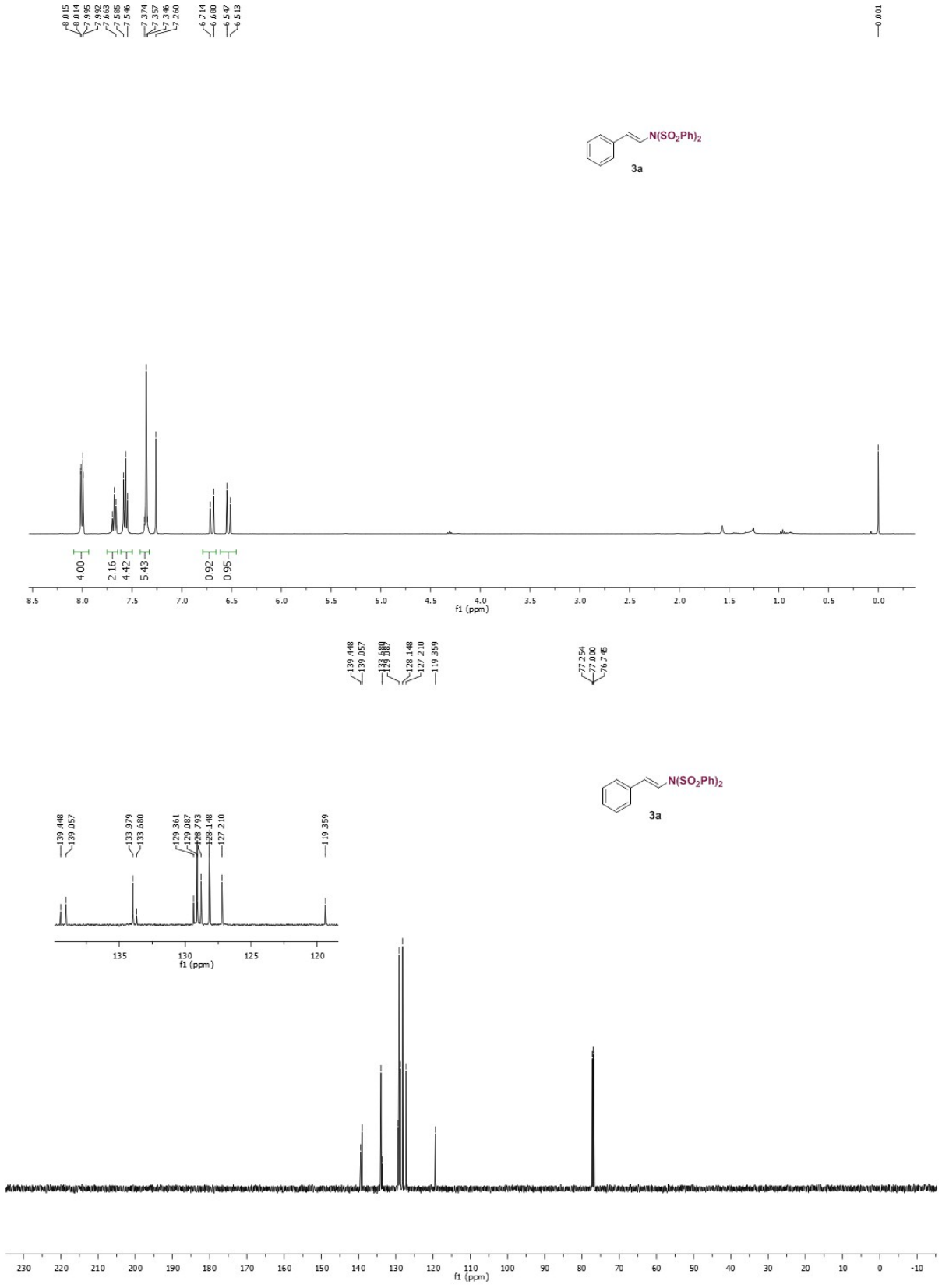

186.560
145.530
142.484
137.870
131.433
129.568
129.165
128.865
128.735
128.655
128.571
128.240
125.661
125.632
123.225
77.254
77.000
76.745
N(SO2Ph)2
F3C
2m
145.530
142.484
139.349
137.870
134.213
131.994
131.564
131.433
130.607
129.568
128.735
128.655
128.571
128.240
125.661
125.632
123.225
f1 (ppm)
f1 (ppm)

Compound 2n

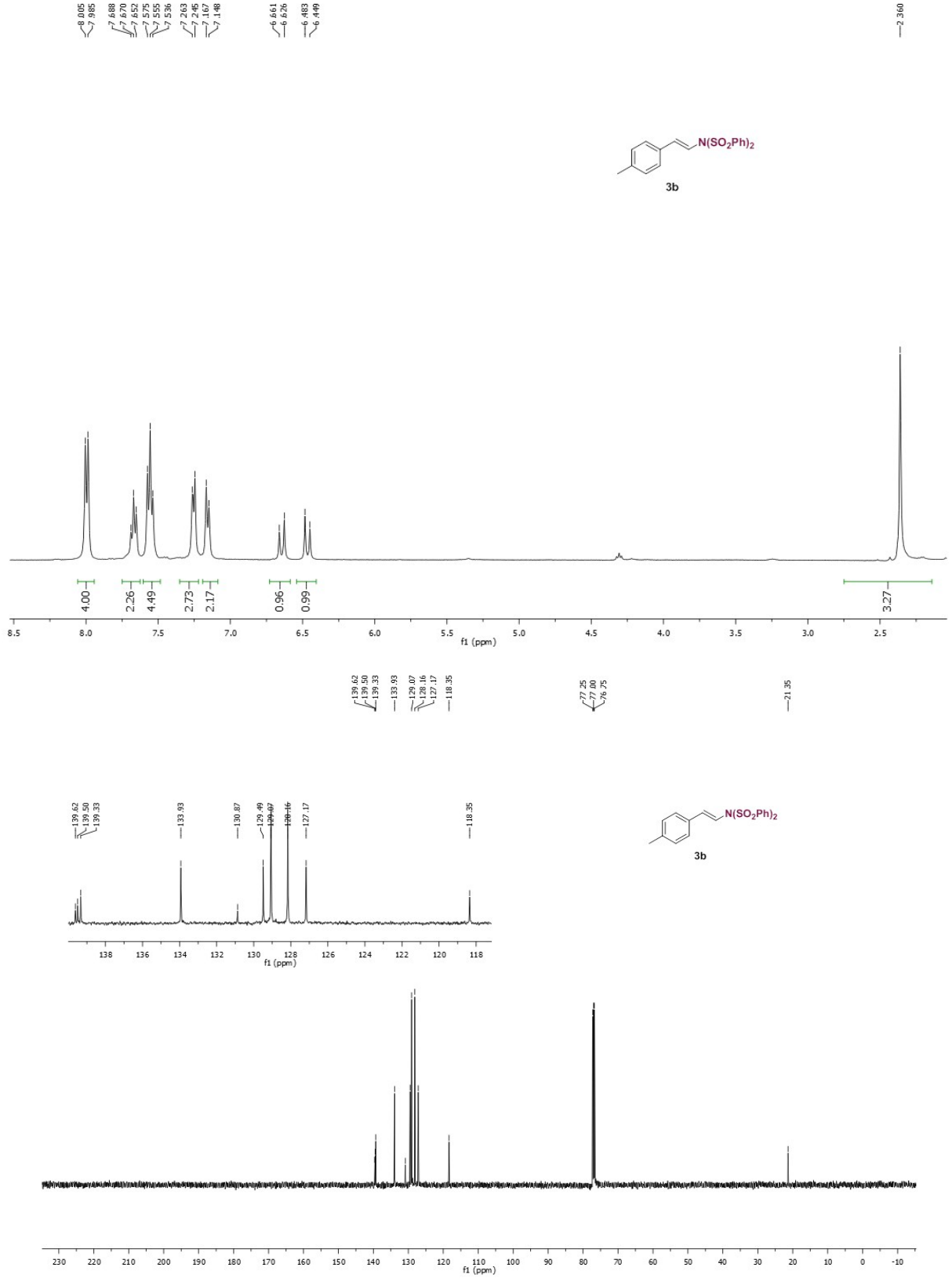

D152 H1
7.910
7.756
7.624
7.611
7.572
7.560
7.548
7.414
7.401
7.388
7.278
7.266
7.259
7.171
7.158
7.145
N(SO2Ph)2
2n
2.395
BRUKER
Current Data Parameters
NAME Dec01-2016-2
EXPNO 180
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161201
Time 20.04 h
INSTRUM spect
PROBHD Z114607_0239 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7262976 sec
RG 109.1
DW 41.600 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
SFO1 600.3837073 MHz
NUC1 1H
P1 10.00 usec
PLW1 26.43300056 W
F2 - Processing parameters
SI 65536
SF 600.3800164 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
3.82
1.00
2.10
2.18
4.15
1.13
2.06
3.00


D263 C13
194.35
145.99
139.55
134.00
131.52
131.36
131.21
131.19
129.59
128.59
128.41
N(SO2Ph)2
2n
25.86
BRUKER
Current Data Parameters
NAME Dec02-2016-4
EXPNO 90
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161203
Time 0.33 h
INSTRUM spect
PROBHD Z114607_0239 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 37878.789 Hz
FIDRES 1.155969 Hz
AQ 0.8650752 sec
RG 190.01
DW 13.200 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9807676 MHz
NUC1 13C
P1 12.00 usec
PLW1 90.40000153 W
SFO2 600.3824015 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 26.43300056 W
PLW12 0.41302001 W
PLW13 0.53944999 W
F2 - Processing parameters
SI 32768
SF 150.9656765 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm

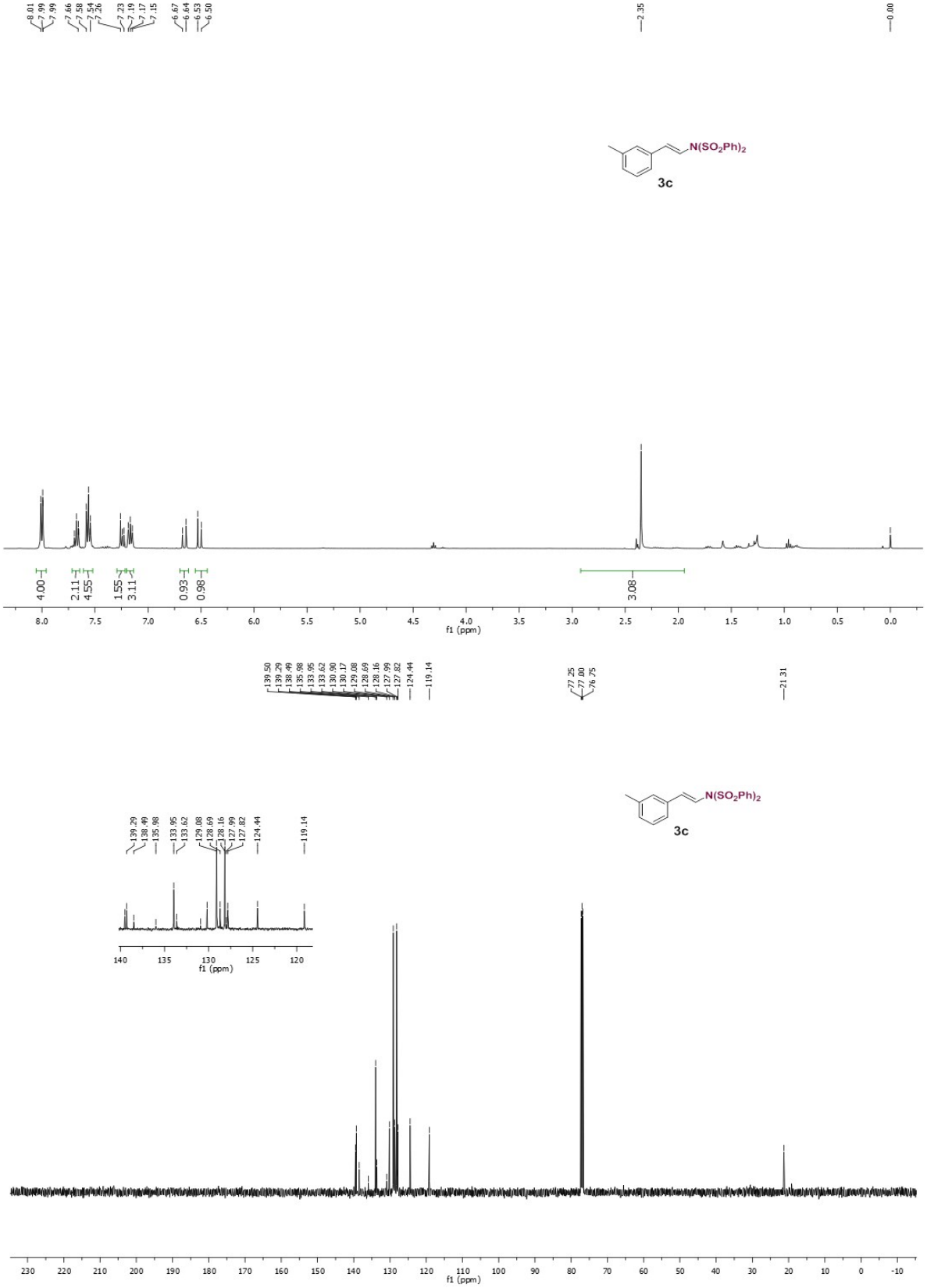
Compound 3a


8.015
8.014
7.995
7.992
7.663
7.585
7.546
7.374
7.357
7.346
7.260
6.714
6.680
6.547
6.513
0.001
N(SO2Ph)2
3a
4.00
2.16
4.42
5.43
0.92
0.95
f1 (ppm)
139.448
139.057
133.979
133.680
129.361
129.087
128.793
128.148
127.210
119.359
77.254
77.000
76.745
N(SO2Ph)2
3a
f1 (ppm)

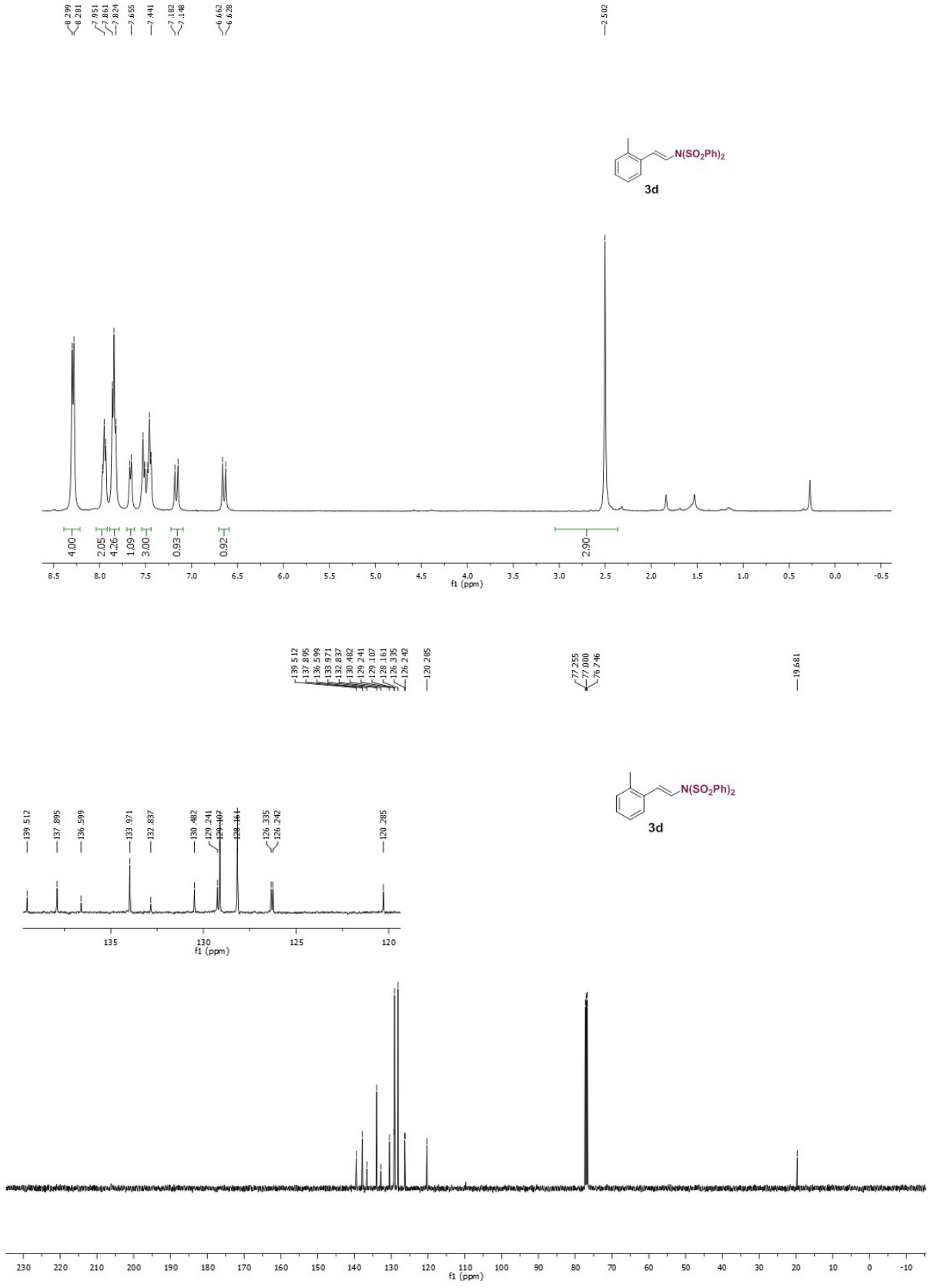
Compound 3b


8.005
7.985
7.688
7.670
7.652
7.575
7.555
7.536
7.263
7.245
7.167
7.148
6.661
6.626
6.483
6.449
2.360
N(SO2Ph)2
3b
4.00
2.26
4.49
2.73
2.17
0.96
0.99
3.27
f1 (ppm)
139.62
139.50
139.33
133.93
129.07
128.16
127.17
118.35
77.25
77.00
76.75
21.35
130.87
129.49
120.46
f1 (ppm)

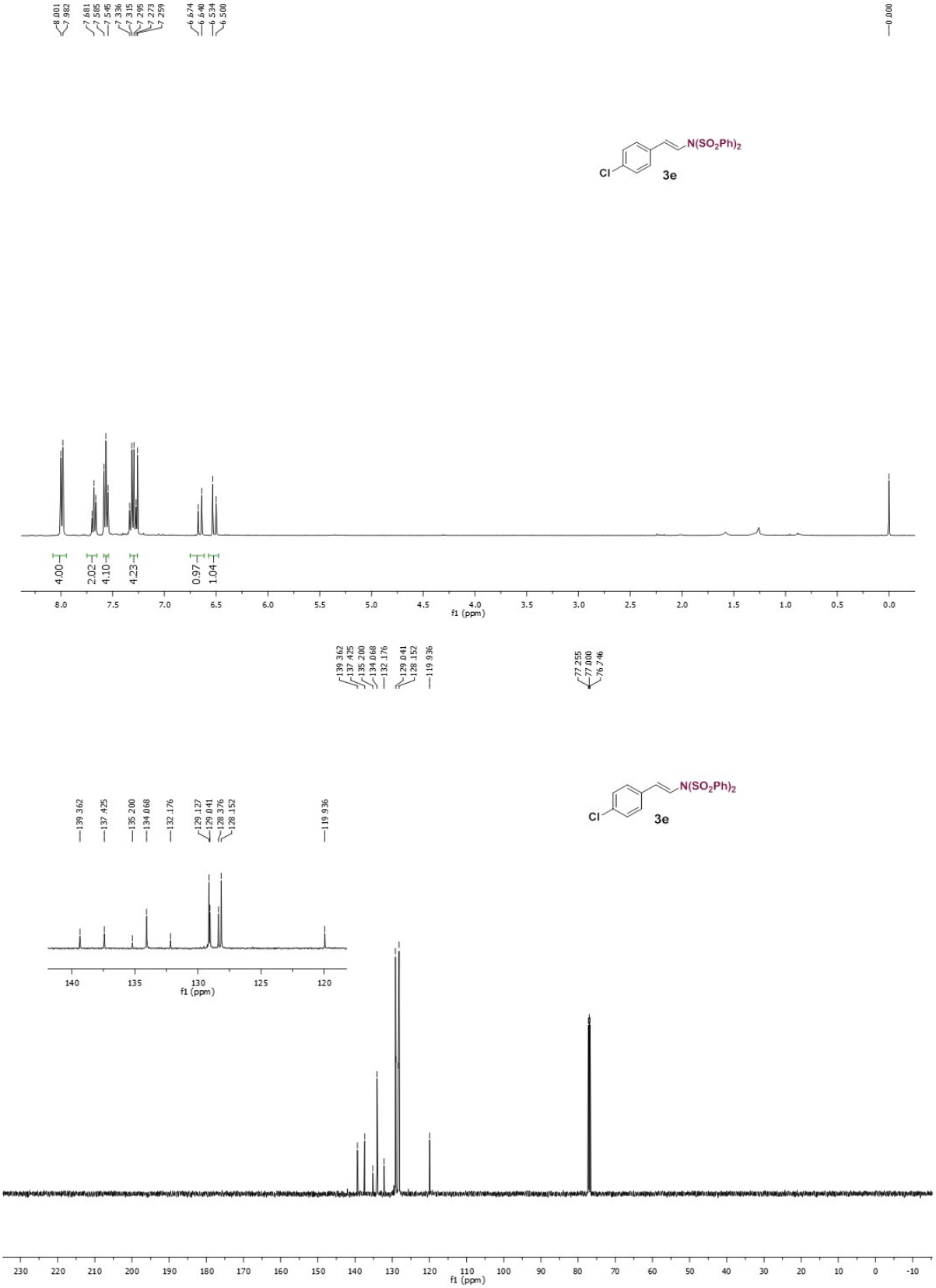
Compound 3c


8.01
7.99
7.99
7.66
7.58
7.54
7.26
7.23
7.19
7.17
7.15
6.67
6.64
6.53
6.50
2.35
0.00
N(SO2Ph)2
3c
4.00
2.11
4.55
1.55
3.11
0.93
0.98
3.08
f1 (ppm)
139.50
139.29
138.49
135.98
133.95
133.62
130.90
130.17
129.08
128.69
128.16
127.99
127.82
124.44
119.14
77.25
77.00
76.75
21.31
N(SO2Ph)2
3c
f1 (ppm)

Compound 3d

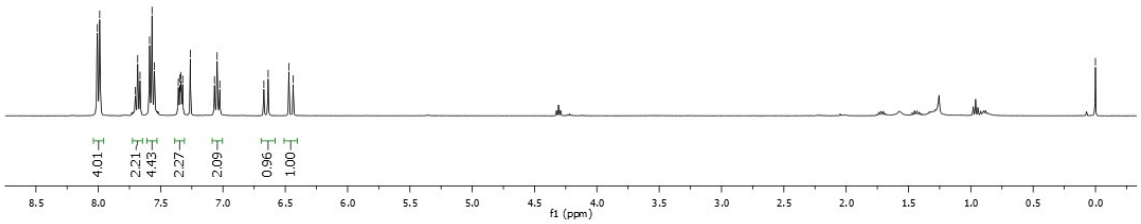

8.299
8.281
7.951
7.861
7.824
7.655
7.441
7.182
7.148
6.662
6.628
2.502
N(SO2Ph)2
3d
4.00
2.05
4.26
1.09
3.00
0.93
0.92
2.90
f1 (ppm)
139.512
137.895
136.599
133.971
132.837
130.482
129.241
129.107
128.161
126.335
126.242
120.285
77.255
77.000
76.746
19.681
N(SO2Ph)2
3d
f1 (ppm)

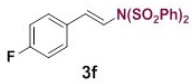
Compound 3e


8.001
7.982
7.681
7.585
7.546
7.336
7.315
7.295
7.273
7.259
6.674
6.640
6.534
6.500
0.000
N(SO2Ph)2
Cl
3e
4.00
2.02
4.10
4.23
0.97
1.04
f1 (ppm)
139.362
137.425
135.200
134.068
132.176
129.041
128.152
119.936
77.255
77.000
76.746
f1 (ppm)

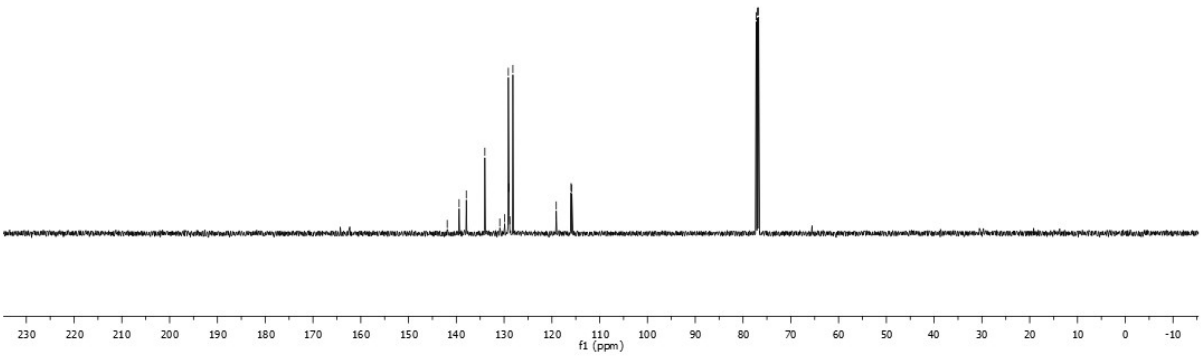
Compound 3f

3f

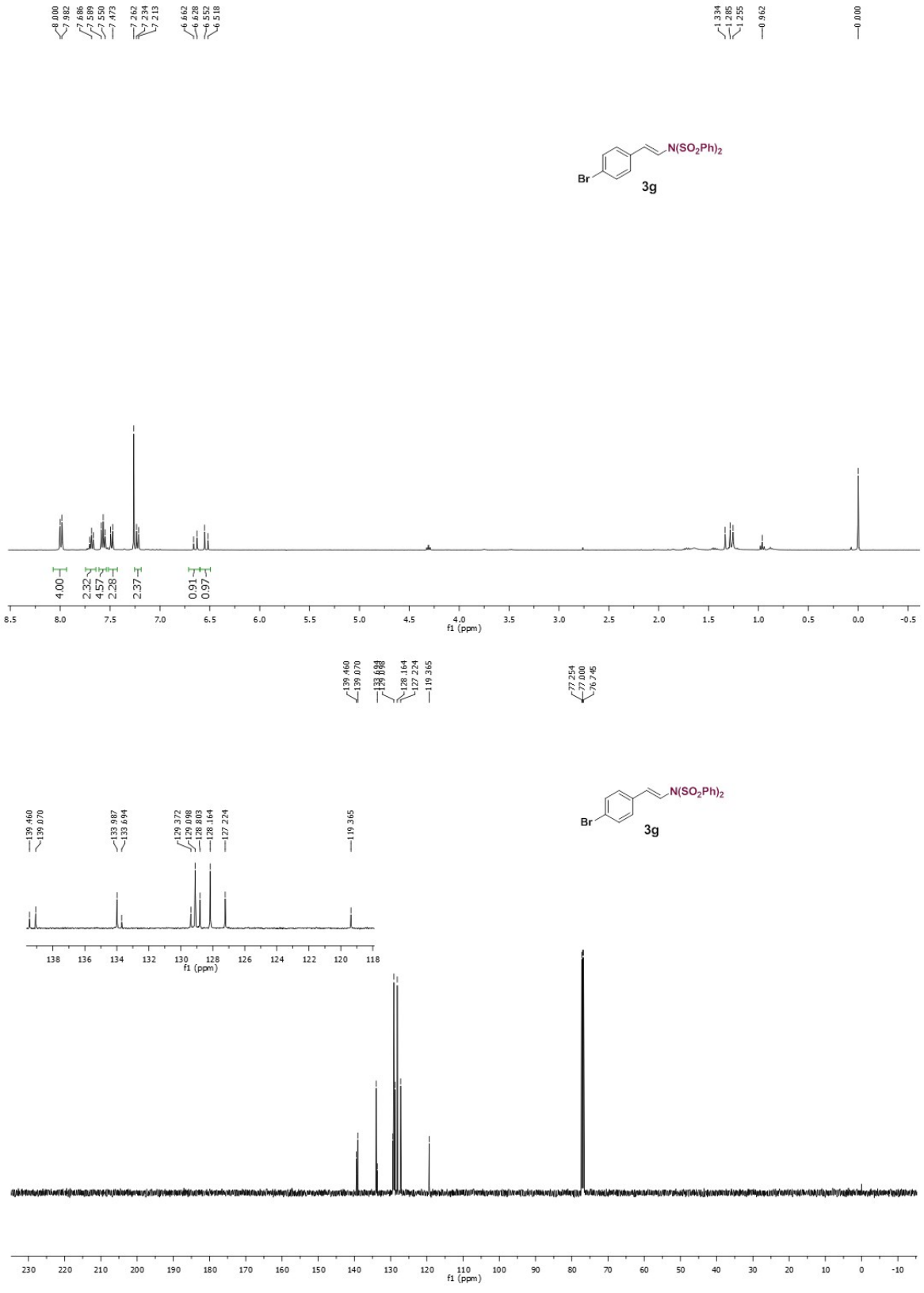




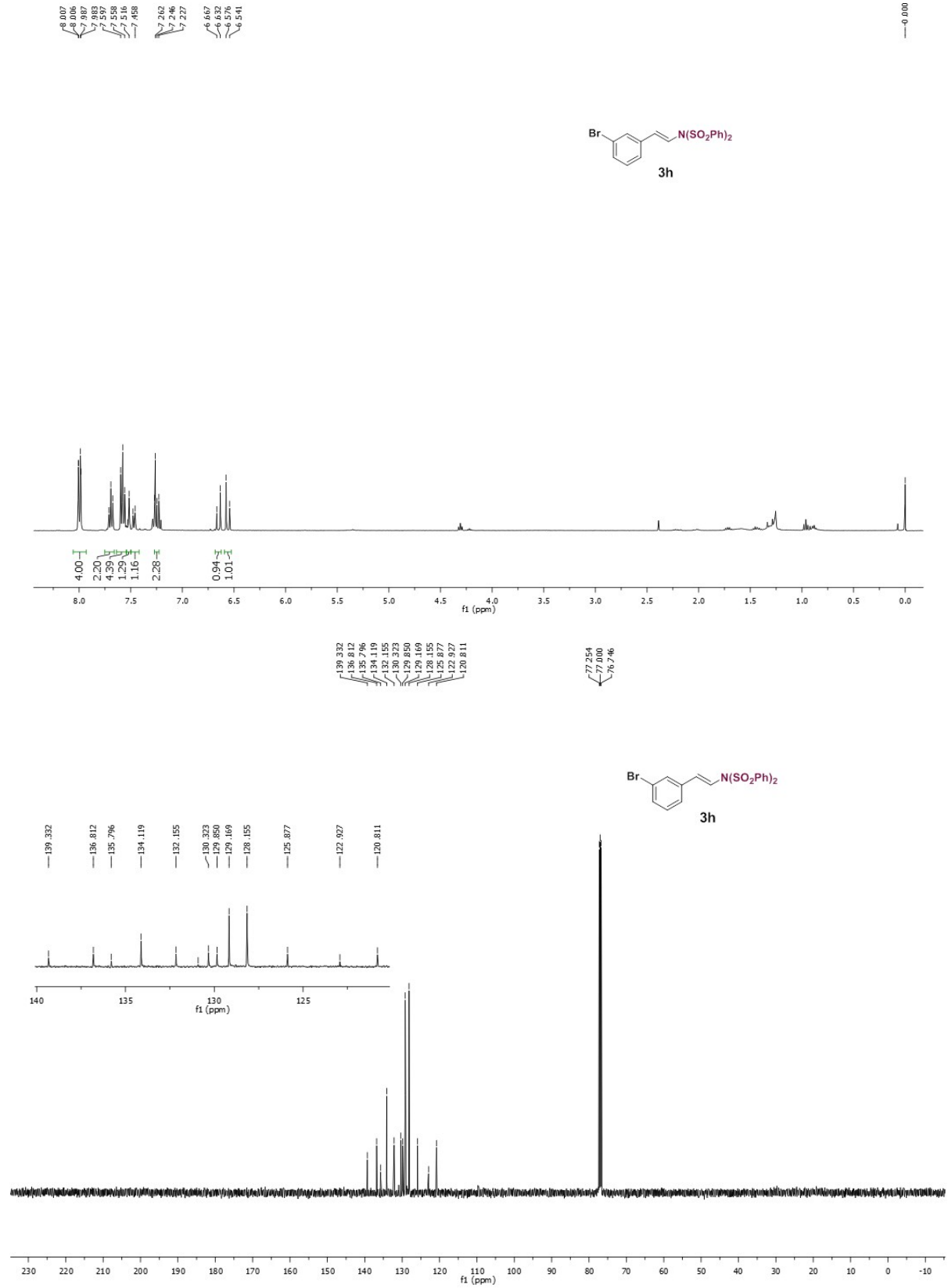
3f



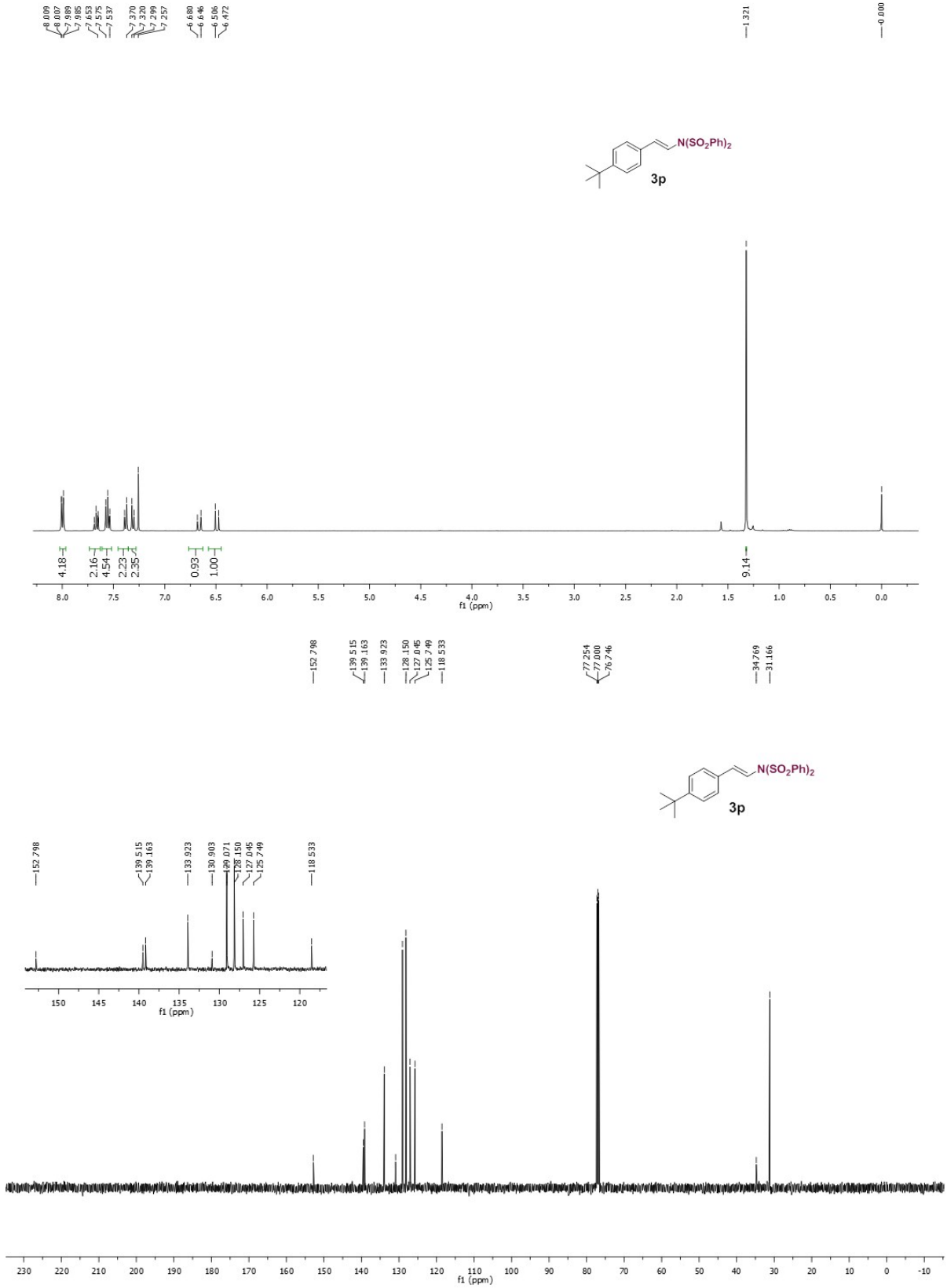
Compound 3g


8.000
7.982
7.686
7.589
7.550
7.473
7.262
7.234
7.213
6.662
6.628
6.552
6.518
1.334
1.285
1.255
0.962
0.000
N(SO2Ph)2
Br
3g
4.00
2.32
4.57
2.28
2.37
0.91
0.97
f1 (ppm)
139.460
139.070
133.987
133.694
129.372
129.098
128.803
128.164
127.224
119.365
77.254
77.000
76.745

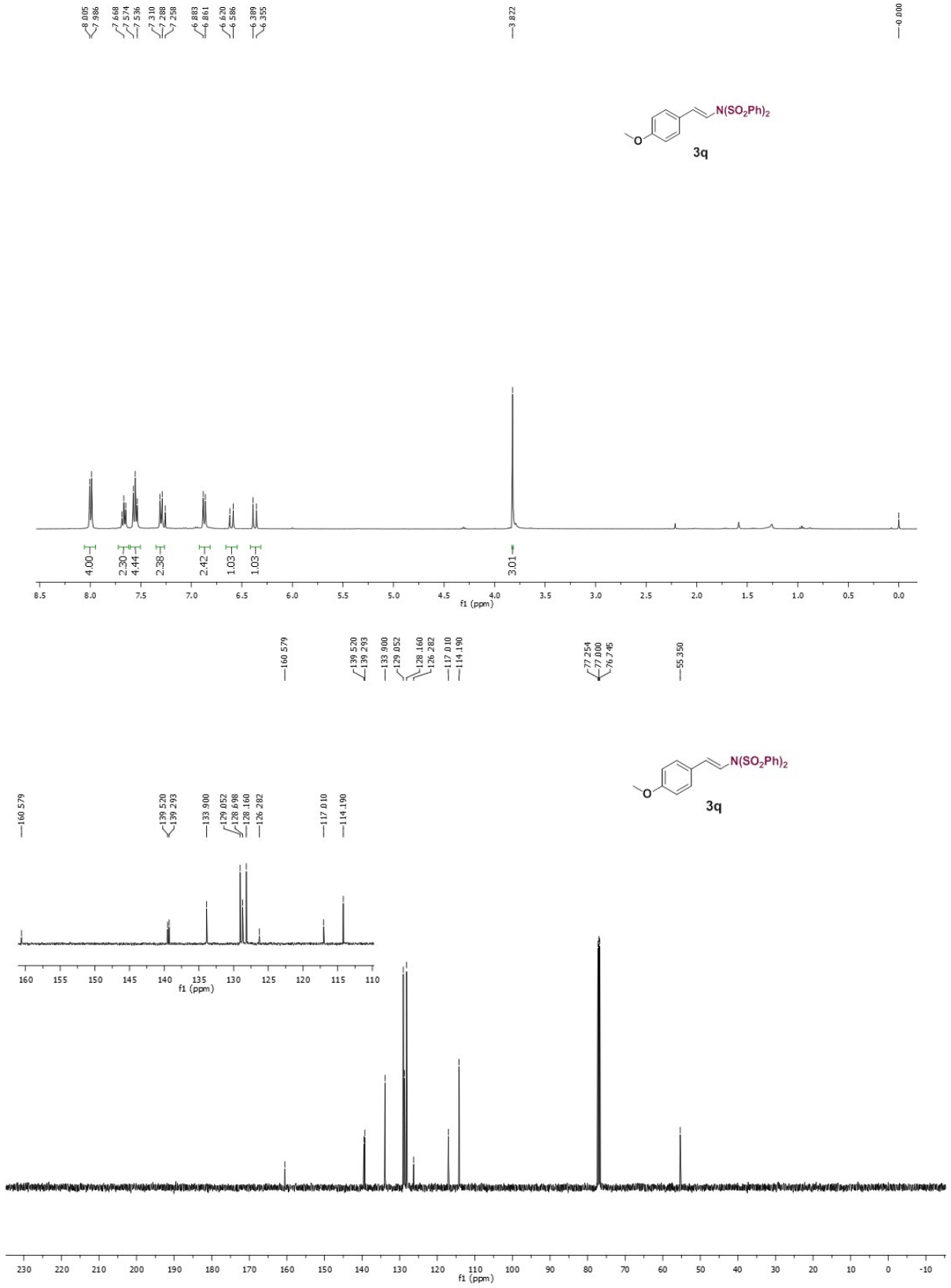
Compound 3h


8.007
8.006
7.987
7.983
7.597
7.558
7.516
7.458
7.262
7.246
7.227
6.667
6.632
6.576
6.541
0.000
Br
N(SO2Ph)2
3h
4.00
2.20
4.39
1.29
1.16
2.28
0.94
1.01
f1 (ppm)
139.332
136.812
135.796
134.119
132.155
130.323
129.850
129.169
128.155
125.877
122.927
120.811
77.254
77.000
76.746
f1 (ppm)

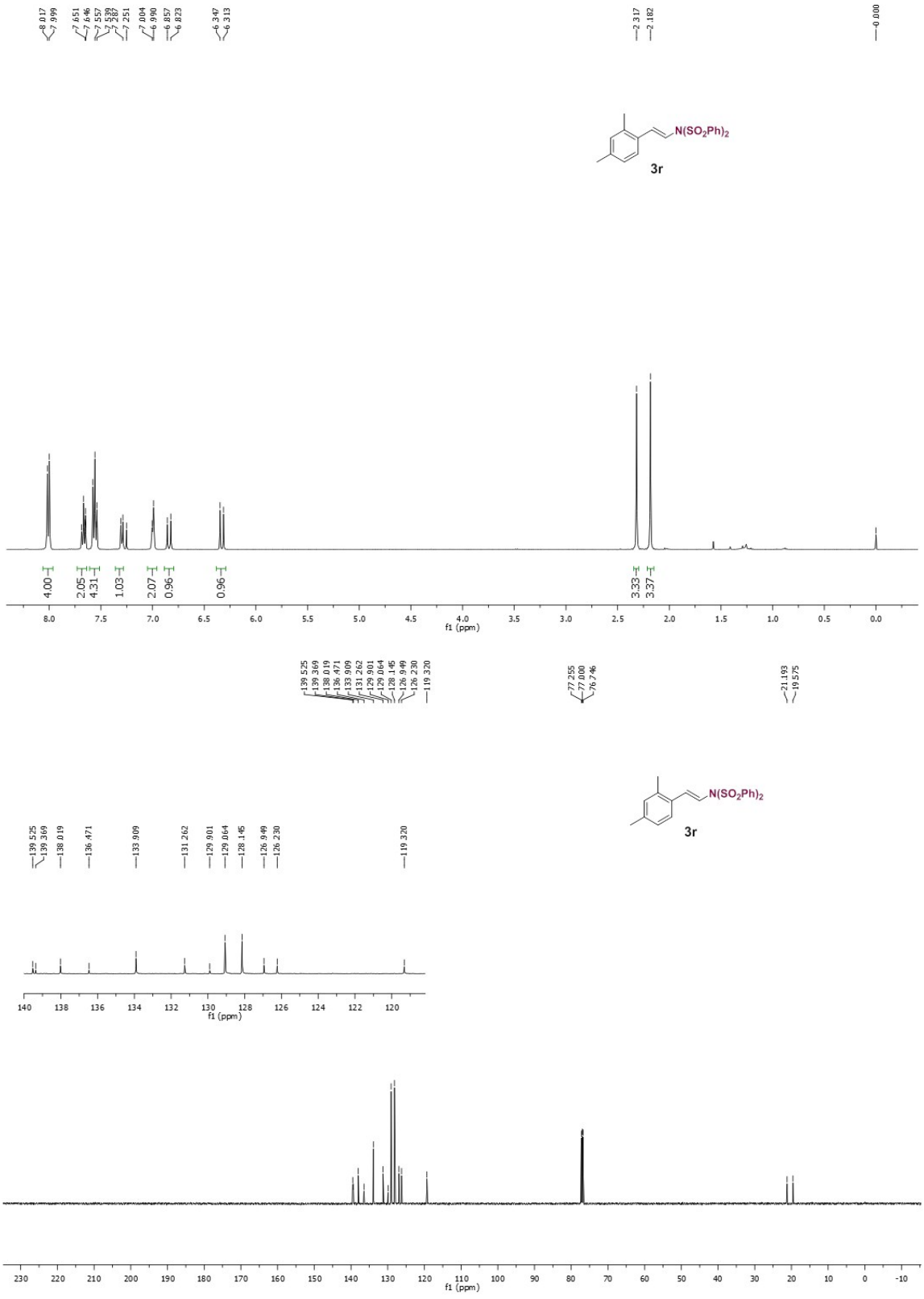
Compound 3p


8.009
8.007
7.989
7.985
7.553
7.535
7.537
7.370
7.320
7.299
7.257
6.680
6.646
6.506
6.472
1.321
0.000
N(SO2Ph)2
3p
4.18
2.16
4.54
2.23
2.35
0.93
1.00
9.14
f1 (ppm)
152.798
139.515
139.163
133.923
130.903
129.071
128.150
127.045
125.749
118.533
77.254
77.000
76.746
34.769
31.166

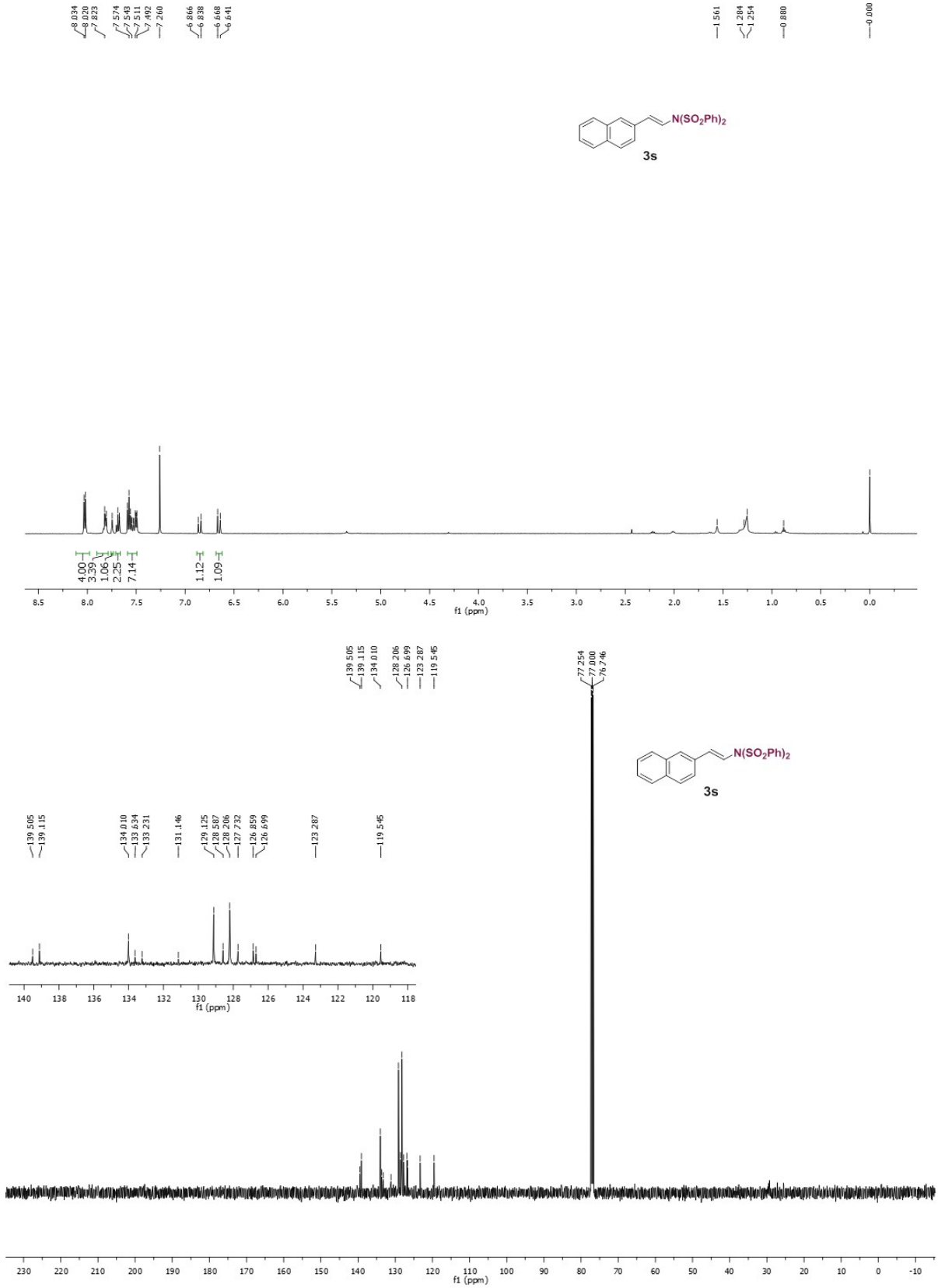
Compound 3q


8.005
7.986
7.668
7.574
7.536
7.310
7.288
7.258
6.883
6.861
6.620
6.586
6.389
6.355
3.822
0.000
N(SO2Ph)2
O
3q
4.00
2.30
4.44
2.38
2.42
1.03
1.03
3.01
f1 (ppm)
160.579
139.520
139.293
133.900
129.052
128.160
126.282
117.010
114.190
77.254
77.000
76.745
55.350
f1 (ppm)

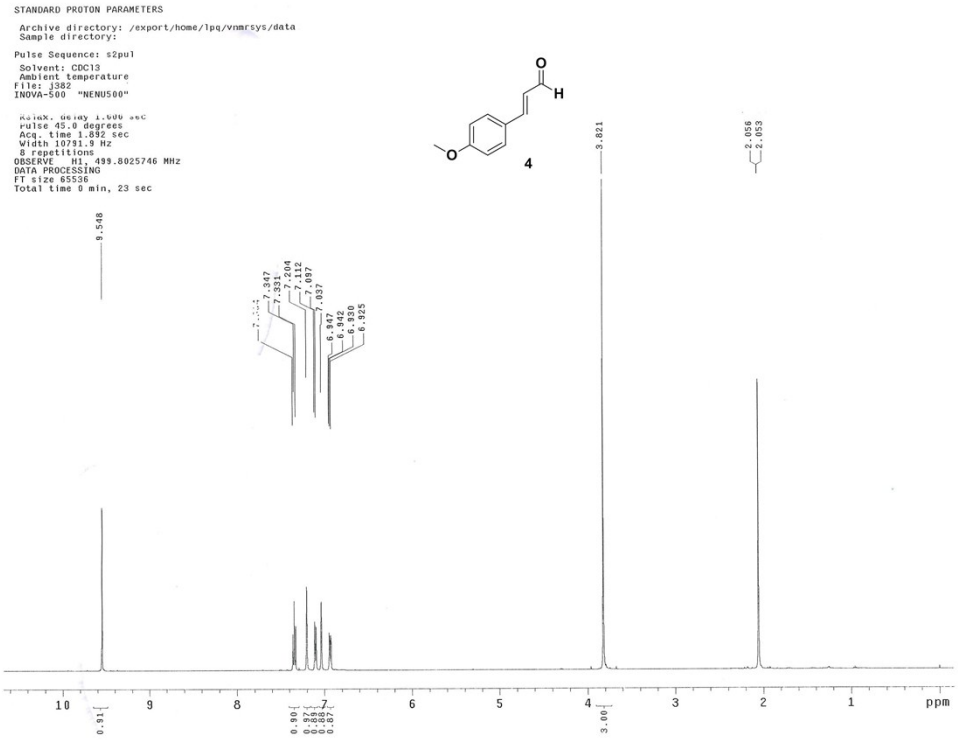
Compound 3r

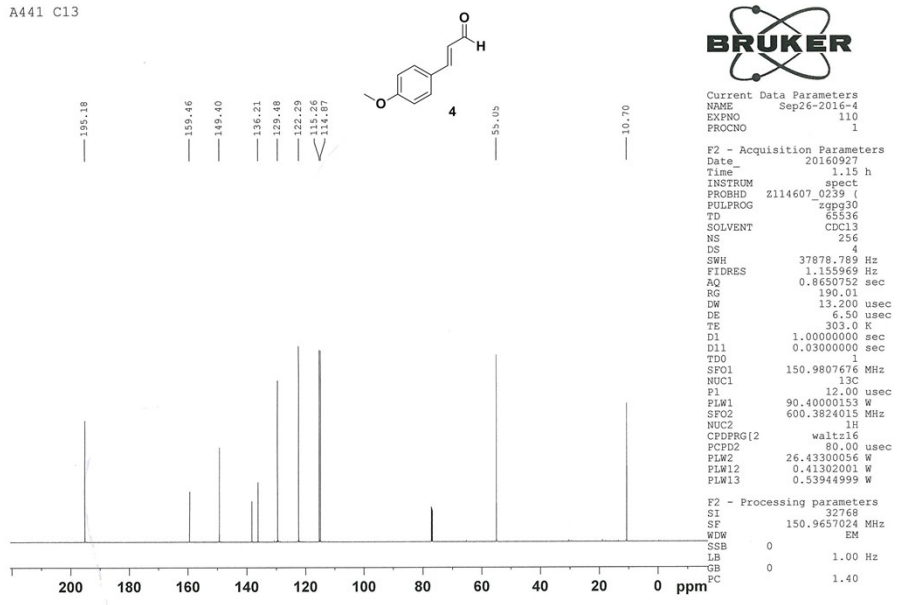

8.017
7.999
7.651
7.646
7.557
7.539
7.287
7.251
7.004
6.990
6.857
6.823
6.347
6.313
2.317
2.182
0.000
N(SO2Ph)2
3r
4.00
2.05
4.31
1.03
2.07
0.96
0.96
3.33
3.37
f1 (ppm)
139.525
139.369
138.019
136.471
133.909
131.262
129.901
129.064
128.145
126.949
126.230
119.320
77.255
77.000
76.746
21.193
19.575

Compound 3s


8.034
8.020
7.823
7.574
7.543
7.511
7.492
7.260
6.866
6.838
6.668
6.641
1.561
1.284
1.254
0.880
0.000
N(SO2Ph)2
3s
4.00
3.39
1.06
2.25
7.14
1.12
1.09
f1 (ppm)
139.505
139.115
134.010
128.206
126.699
123.287
119.545
77.254
77.000
76.746
131.146
129.125
128.587
127.732
126.859
f1 (ppm)

The byproduct: (*E*)-3-(4-methoxyphenyl)acrylaldehyde






```
Current Data Parameters
NAME          Sep26-2016-4
EXPNO                  110
PROCNO                   1

F2 - Acquisition Parameters
Date_              20160927
Time                   1.15 h
INSTRUM               spect
PROBHD    Z114607_0239 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     256
DS                       4
SWH              37878.789 Hz
FIDRES            1.155969 Hz
AQ               0.8650752 sec
RG                  190.01
DW                 13.200 usec
DE                  6.50 usec
TE                 303.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                      1
SFO1           150.9807676 MHz
NUC1                   13C
P1                  12.00 usec
PLW1           90.40000153 W
SFO2           600.3824015 MHz
NUC2                   1H
CPDPRG[2            waltz16
PCPD2               80.00 usec
PLW2           26.43300056 W
PLW12           0.41302001 W
PLW13           0.53944999 W

F2 - Processing parameters
SI                     32768
SF           150.9657024 MHz
WDW                    EM
SSB          0
LB                   1.00 Hz
GB           0
PC                   1.40
```