Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

TBN-mediated regio- and stereoselective sulfonylation & oximation (oximosulfonylation) of alkynes with sulfonyl hydrazines in EtOH/H_2O

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1. General Remarks

All substrates were purchased commercially without further purification. The yields were determined based on sulfonyl hydrazides. GC-MS spectra were measured with Bruker GC-MS 456-Scion. All new compounds were characterized by ^{1}H NMR, ^{13}C NMR and HRMS. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AC-300 FT spectrometer at 400 MHz and100 MHz, respectively, and tetramethylsilane (TMS) was used as an internal standard. Chemical shifts (δ) and coupling constants (*J*) were expressed in ppm and Hz, respectively. All chemical shifts were reported relative to tetramethylsilane (0 ppm for ^{1}H), $CDCl_3$ (7.26 ppm for ^{1}H, 77.16 ppm for ^{13}C) and DMSO (2.50 ppm for ^{1}H, 39.52 ppm for ^{13}C), respectively. High resolution mass spectra (HRMS) were recorded on a Waters Xevo G2-XS QTOF spectrometer (Tolerance = 10.0 ppm). All reagents were obtained from commercial sources (purity >97%) and used without further purification except for special instructions. Silica gel for column chromatography was purchased from Qingdao Haiyang Chemical Co., Ltd.

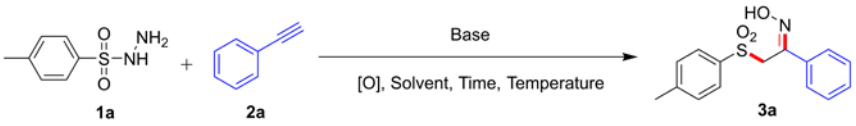
2. General Procedures for the Synthesis of Arylsulfonyl Hydrazides

Arylsulfonyl hydrazides **2c**-**2u** were prepared according to the literature procedure.[1] To a solution of an arylsulfonyl chloride (3.0 mmol) in tetrahyrdofuran (15 mL), was added hydrazine monohydrate (375 mg, 7.5 mmol) dropwise under nitrogen at 0 °C. After vigorous stirring for 30 min at 0 °C, the reaction mixture was added ethyl acetate (60 mL), and washed with saturated brine (3 × 10 mL). The organic layer was dried over sodium sulfate, filtered, concentrated and added to hexane (12 mL) over 5 min. The mixture was filtered, and the collected solid was dried in vacuum. But, it is worth noting that Arylsulfonyl hydrazides **2b** were prepared according to the next procedure. To a solution of a hydrazine monohydrate (375 mg, 7.5 mmol) in tetrahyrdofuran (5 mL), was added arylsulfonyl chloride (3.0 mmol) in tetrahyrdofuran (10 mL) dropwise under nitrogen at 0 °C. After vigorous stirring for 30 min at 0 °C, the reaction mixture was added ethyl acetate (60 mL), and washed with saturated brine (3 × 10 mL). The organic layer was dried over sodium sulfate, filtered, concentrated and added to hexane (12 mL) over 5 min. The mixture was filtered, and the collected solid was dried in vacuum.

3. Typical Procedure for the Synthesis of α-Sulfonylethanone Oximes

A mixture of sulfonyl hydrazides (0.25 mmol), phenylacetylene (0.75 mmol), TBN (0.50 mmol), imidazole (0.375 mmol) and $N_2H_4{\cdot}H_2O$ (0.125 mmol), in 4.1 mL CH_3CH_2OH/water (v:v = 40:1) was put into a Schlenk tube at 100 °C under magnetic stirring for 1.5 h under N_2 protection. After the reaction was complete, the sealed tube was allowed to cool to room temperature. Then, the mixture was extracted with EtOAc (3 × 5 mL) and then the combined organic extracts were washed with brine (10 mL), dried over sodium sulfate, and filtered. The solvent was removed under reduced pressure and the residue was purified by flash column chromatography (SiO_2, 200-300 mesh, dichlormethane/ethyl acetate = 30 : 1) to give desired product **3aa**.

4. Optimization of the reaction conditions



1a 2a 3a

Entry	Solvent	[O]	Base	T [oC]	Yield[%]
1	**DMSO**	**TBN**	**Imidazole**	**80**	**18**
2	NMP	TBN	Imidazole	80	8
3	THF	TBN	Imidazole	80	9
4	$(CH_3)_2CHOH$	TBN	Imidazole	80	19
5	**PhCl**	**TBN**	**Imidazole**	**80**	**10**
6	DMF	TBN	Imidazole	80	10
7	$CHCl_3$	TBN	Imidazole	80	3
8	**CH_3CH_2OH**	**TBN**	**Imidazole**	**80**	**21**
9	1,4-dioxane	TBN	Imidazole	80	4
10	$(CF_3)_2CH_2OH$	TBN	Imidazole	80	1
11	1,2-Dichloroethane	TBN	Imidazole	80	3
12	CH_3CN	TBN	Imidazole	80	4
13	CH_3COCH_3	TBN	Imidazole	80	Trace
14	H_2O	TBN	Imidazole	80	5
15	$CH_3CH_2NO_2$	TBN	Imidazole	80	7

[a] Reaction conditions:**1a** (0.25 mmol), **2a** (0.5 mmol), **TBN** (0.5 mmol),Base (0.375 mmol), solvent(1.0 mL), 3 h, air.
[b] Yields were calculated by UPLC.

Fig.ESI-1 Optimization of the reaction conditions by **the single solvents**

Entry	Solvent	[O]	**Base**	Yield[%]
1	$CH_3CH_2OH/H_2O(1:1)$	TBN	**-**	10
2	$CH_3CH_2OH/H_2O(1:1)$	TBN	**KO^tBu**	n.d
3	$CH_3CH_2OH/H_2O(1:1)$	TBN	**DMAP**	13
4	$CH_3CH_2OH/H_2O(1:1)$	TBN	**K_2CO_3**	trace
5	$CH_3CH_2OH/H_2O(1:1)$	TBN	**CH_3COONa**	20
6	$CH_3CH_2OH/H_2O(1:1)$	TBN	**NaOH**	trace
7	$CH_3CH_2OH/H_2O(1:1)$	TBN	**$NaHCO_3$**	28
8	$CH_3CH_2OH/H_2O(1:1)$	TBN	**piperazine**	12
9	$CH_3CH_2OH/H_2O(1:1)$	TBN	**CH_3ONa**	trace
10	$CH_3CH_2OH/H_2O(1:1)$	TBN	**Cs_2CO_3**	trace
11	**$CH_3CH_2OH/H_2O(1:1)$**	**TBN**	**Imidazole**	**38**
12	$CH_3CH_2OH/H_2O(1:1)$	TBN	**$(CH_3CH_2)_3N$**	20
13	$CH_3CH_2OH/H_2O(1:1)$	TBN	**DBU**	trace
14	$CH_3CH_2OH/H_2O(1:1)$	TBN	**DIPEA**	24
15	$CH_3CH_2OH/H_2O(1:1)$	TBN	**TEMED**	13
16	$CH_3CH_2OH/H_2O(1:1)$	TBN	**pyridine**	8
17	$CH_3CH_2OH/H_2O(1:1)$	TBN	**1,2-Dimethylimidazole**	29
18	$CH_3CH_2OH/H_2O(1:1)$	TBN	**N-Methylimidazole**	31

[a] Reaction conditions:**1a** (0.25 mmol), **2a** (0.5 mmol), **TBN** (0.5 mmol),Base (0.375 mmol), Solvent(1.5 mL), 3 h, 80 °C, air.
[b] Yields were calculated by UPLC.

Fig.ESI-2 Optimization of the reaction conditions by **the type of base**

Entry	Solvent	[O]	Base	T [°C]	Yield[%]
1	CH_3CH_2OH/H_2O(1:1)	TBN	Imidazole	85	34
2	CH_3CH_2OH/H_2O(1:1)	TBN	Imidazole	90	35
3	CH_3CH_2OH/H_2O(1:1)	TBN	Imidazole	95	36
4	**CH_3CH_2OH/H_2O(1:1)**	**TBN**	**Imidazole**	**100**	**44**
5	CH_3CH_2OH/H_2O(1:1)	TBN	Imidazole	105	44
6	CH_3CH_2OH/H_2O(1:1)	TBN	Imidazole	110	42
7	CH_3CH_2OH/H_2O(1:1)	TBN	Imidazole	120	33
8	CH_3CH_2OH/H_2O(1:1)	TBN	Imidazole	130	30
9	CH_3CH_2OH/H_2O(1:1)	TBN	Imidazole	140	30

[a] Reaction conditions:**1a** (0.25 mmol), **2a** (0.5 mmol), **TBN** (0.5 mmol),Base (0.375 mmol), solvent(1.5 mL), 6h, air.

[b] Yields were calculated by UPLC.

Fig.ESI-3 Optimization of the reaction conditions by **the temperature**

Entry	Solvent	[NO] sources	Base	Yield[%]
1	**CH_3CH_2OH/H_2O(1:1)**	**TBN**	**Imidazole**	**44**
2	CH_3CH_2OH/H_2O(1:1)	**$CH_3(CH_2)ONO$**	Imidazole	42
3	CH_3CH_2OH/H_2O(1:1)	**$CH_3CH_2CH_2(CH_3)ONO$**	Imidazole	41
4	CH_3CH_2OH/H_2O(1:1)	**$CH_3(CH_2)_2CH_2(CH_3)ONO$**	Imidazole	32

[a] Reaction conditions:**1a** (0.25 mmol), **2a** (0.5 mmol), **[NO] sources** (0.5 mmol),Base (0.375 mmol), solvent(1.5 mL), 3 h, 80 °C, air.

[b] Yields were calculated by UPLC.

Fig.ESI-4 Optimization of the reaction conditions by **the [NO] sources**

Entry	Solvent	[Additive]	Base	Yield[%]
1	CH_3CH_2OH/H_2O(1:1)	**$Cu(OAc)_2$**	Imidazole	n.d
2	CH_3CH_2OH/H_2O(1:1)	**$Mn(OAc)_2$**	Imidazole	6
3	CH_3CH_2OH/H_2O(1:1)	**$Co(OAc)_2$**	Imidazole	trace
4	CH_3CH_2OH/H_2O(1:1)	**$FeCl_3$**	Imidazole	trace
5	CH_3CH_2OH/H_2O(1:1)	**CuCl**	Imidazole	n.d
6	CH_3CH_2OH/H_2O(1:1)	**$FeSO_4$**	Imidazole	4
7	**CH_3CH_2OH/H_2O(1:1)**	**$NH_2NH_2{\cdot}H_2O$(0.5 eq)**	**Imidazole**	**49**
8	CH_3CH_2OH/H_2O(1:1)	**$NH_2NH_2{\cdot}H_2O$(1.0 eq)**	Imidazole	44
9	CH_3CH_2OH/H_2O(1:1)	**$NH_2NH_2{\cdot}H_2O$(2.0 eq)**	Imidazole	5
10	CH_3CH_2OH/H_2O(1:1)	**$NH_2NH_2{\cdot}H_2O$(3.0 eq)**	Imidazole	trace

[a] Reaction conditions:**1a** (0.25 mmol), **2a** (0.5 mmol), **TBN** (0.5 mmol),Base (0.375 mmol), Solvent(1.5 mL), 3 h, 80 °C, air.

[b] Yields were calculated by UPLC.

Fig.ESI-5 Optimization of the reaction conditions by **the additives**

Entry	1a	2a	[O]	solvent	Yield[%]
1	**1.0 eq**	**1.0 eq**	**TBN (2.0eq)**	CH_3CH_2OH/H_2O(1:1)	30
2	**1.0 eq**	**2.0 eq**	**TBN (2.0eq)**	CH_3CH_2OH/H_2O(1:1)	47
3	**2.0 eq**	**1.0 eq**	**TBN (2.0eq)**	CH_3CH_2OH/H_2O(1:1)	45
4	**1.0 eq**	**3.0 eq**	**TBN (2.0eq)**	**CH_3CH_2OH/H_2O(1:1)**	**55**
5	**1.0 eq**	**3.0 eq**	**TBN (2.5eq)**	CH_3CH_2OH/H_2O(1:1)	50
6	**1.0 eq**	**4.0 eq**	**TBN (2.0eq)**	CH_3CH_2OH/H_2O(1:1)	45
7	**1.0 eq**	**2.5eq**	**TBN (2.0eq)**	CH_3CH_2OH/H_2O(1:1)	50

[a] Reaction conditions: Solvent(2.0 mL), **Imidazole** (0.375 mmol), 3 h, 100 °C, air.

[b] Yields were calculated by UPLC.

Fig.ESI-6 Optimization of the reaction conditions by **the dosage of substrates**

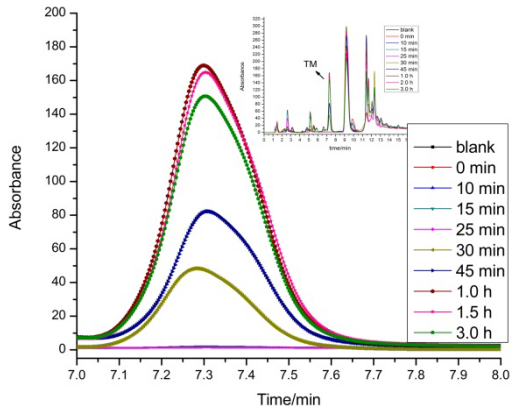


Fig.ESI-7 The effects of the reaction time on product yield of **3aa**

Entry	**Solvent**	Base	Yield[%]
1	**CH_3CH_2OH(1.0 mL)+H_2O(100 μL)**	Imidazole	48
2	**CH_3CH_2OH(2.0 mL)+H_2O(100 μL)**	Imidazole	54
3	**CH_3CH_2OH(3.0 mL)+H_2O(100 μL)**	Imidazole	60
4	**CH_3CH_2OH(4.0 mL)+H_2O(100 μL)**	**Imidazole**	**62**
5	**CH_3CH_2OH(5.0 mL)+H_2O(100 μL)**	Imidazole	59
6	**CH_3CH_2OH(4.0 mL)+H_2O(200 μL)**	Imidazole	56
7	**CH_3CH_2OH(4.0 mL)+H_2O(500 μL)**	Imidazole	55
8	**CH_3CH_2OH(4.0 mL)+H_2O(1.0 mL)**	Imidazole	49

[a] Reaction conditions:**1a** (0.25 mmol), **2a** (0.75 mmol), **TBN** (0.5 mmol),Base (0.375 mmol), 4 h, 100°C, air.

[b] Yields were calculated by UPLC.

Fig.ESI-8 Optimization of the reaction conditions by **the ratio of CH_3CH_2OH and H_2O**

Entry	Solvent	**Time**	Base	Yield[%]
1	CH_3CH_2OH/H_2O	**0 min**	Imidazole	n.d
2	CH_3CH_2OH/H_2O	**5 min**	Imidazole	n.d
3	CH_3CH_2OH/H_2O	**10 min**	Imidazole	n.d
4	CH_3CH_2OH/H_2O	**15 min**	Imidazole	n.d
5	CH_3CH_2OH/H_2O	**25 min**	Imidazole	Trace
6	CH_3CH_2OH/H_2O	**30 min**	Imidazole	18
7	CH_3CH_2OH/H_2O	**45 min**	Imidazole	34
8	CH_3CH_2OH/H_2O	**1.0 h**	Imidazole	60
9	**CH_3CH_2OH/H_2O**	**1.5 h**	**Imidazole**	**65**
10	CH_3CH_2OH/H_2O	**2.0 h**	Imidazole	65
11	CH_3CH_2OH/H_2O	**3.0 h**	Imidazole	61
12	CH_3CH_2OH/H_2O	**6.0 h**	Imidazole	58
13	CH_3CH_2OH/H_2O	**12 h**	Imidazole	52
14	CH_3CH_2OH/H_2O	**24 h**	Imidazole	50

[a] Reaction conditions:**1a** (0.25 mmol), **2a** (0.5 mmol), **TBN** (0.5 mmol), Base (0.375 mmol), Solvent (1.5 mL), 100 °C, air.

[b] Yields were calculated by UPLC.

Fig.ESI-9 Optimization of the reaction conditions by **the reaction time**

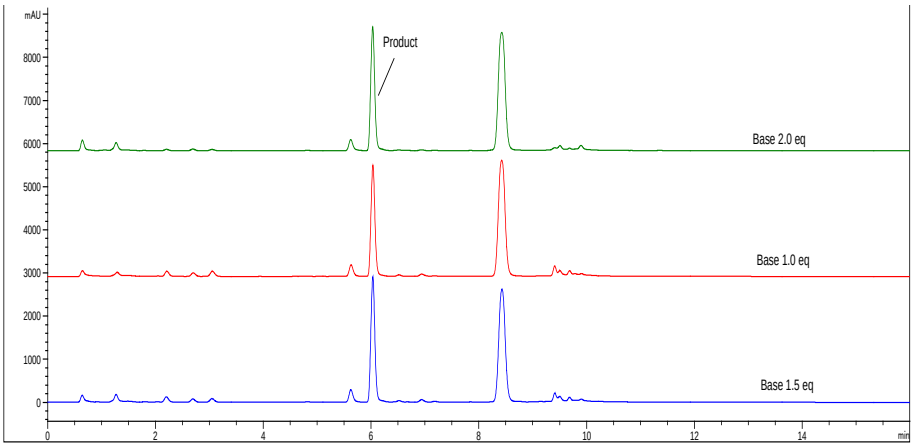


Fig.ESI-10 The UPLC of optimization of the reaction conditions by **the dosage of base**

Entry	1a	2a	**[Imidazole]**	solvent	Yield[%]
1	1.0 eq	2.0 eq	**1.0 eq**	CH_3CH_2OH/H_2O(1:1)	30
2	1.0 eq	2.0 eq	**1.5 eq**	CH_3CH_2OH/H_2O(1:1)	38
3	1.0 eq	2.0 eq	**2.0 eq**	CH_3CH_2OH/H_2O(1:1)	36

[a] Reaction conditions: Solvent(2.0 ml), **TBN**(0.5 mmol), 6h, 80 ^oC, air.

[b] Yields were calculated by UPLC.

Fig.ESI-11 The result of optimization of the reaction conditions by **the dosage of base**

5. The analysis of byproducts

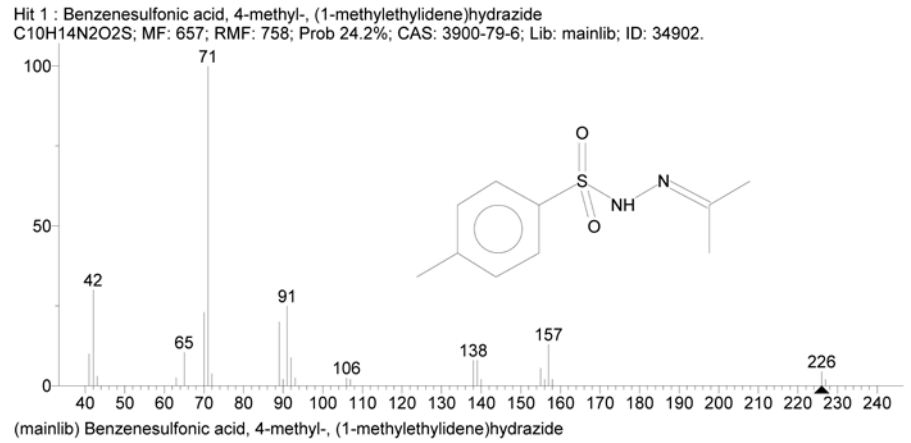


Fig.ESI-12 The analysis of byproduct **4a** by GC-MS when the solvent is CH_3COCH_3

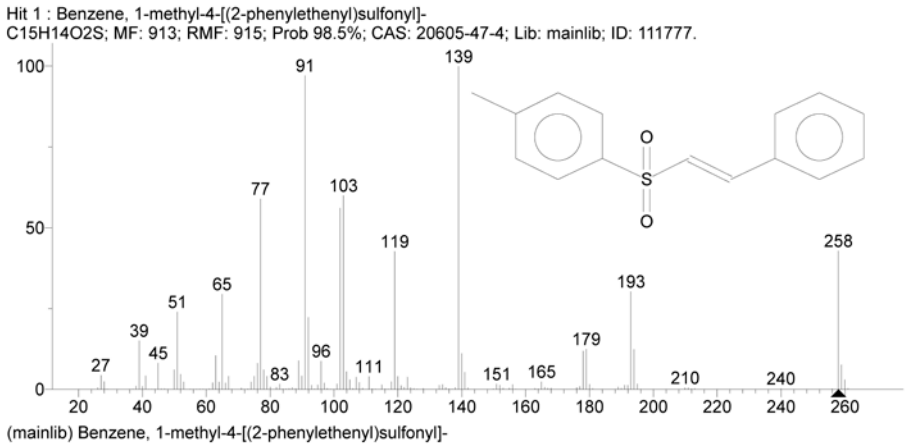


Fig.ESI-13 The analysis of byproduct **4b** by GC-MS

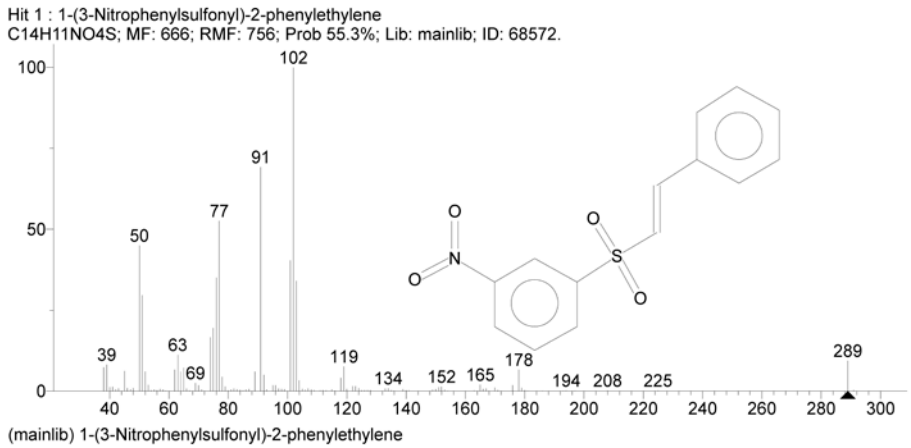


Fig.ESI-14 The analysis of vinyl sulfone **4c** by GC-MS

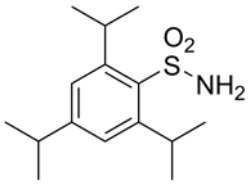


Fig.ESI-15 See characterization data and NMR spectra of products for the details data of **4d**

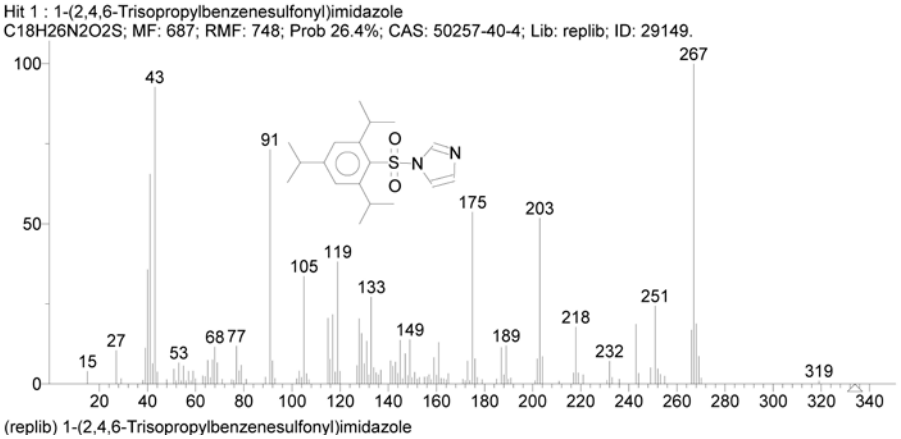


Fig.ESI-16 The analysis of byproduct **4e** by GC-MS when the substrate is **1o**

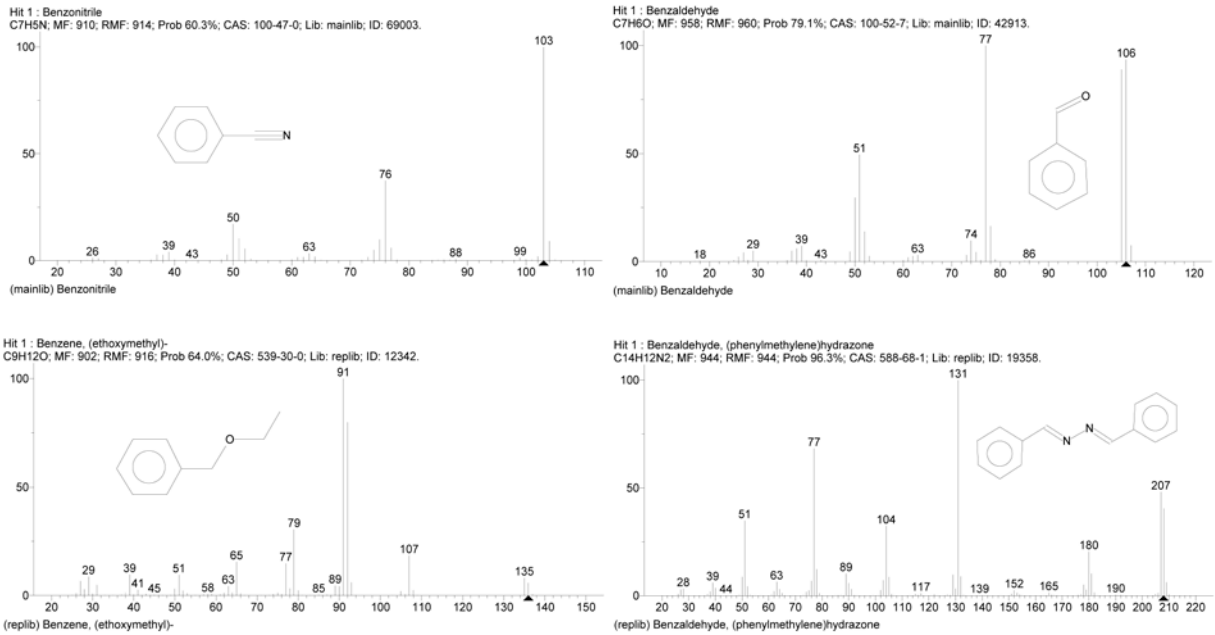


Fig.ESI-17 The analysis of byproduct by GC-MS when the substrate is **1t**

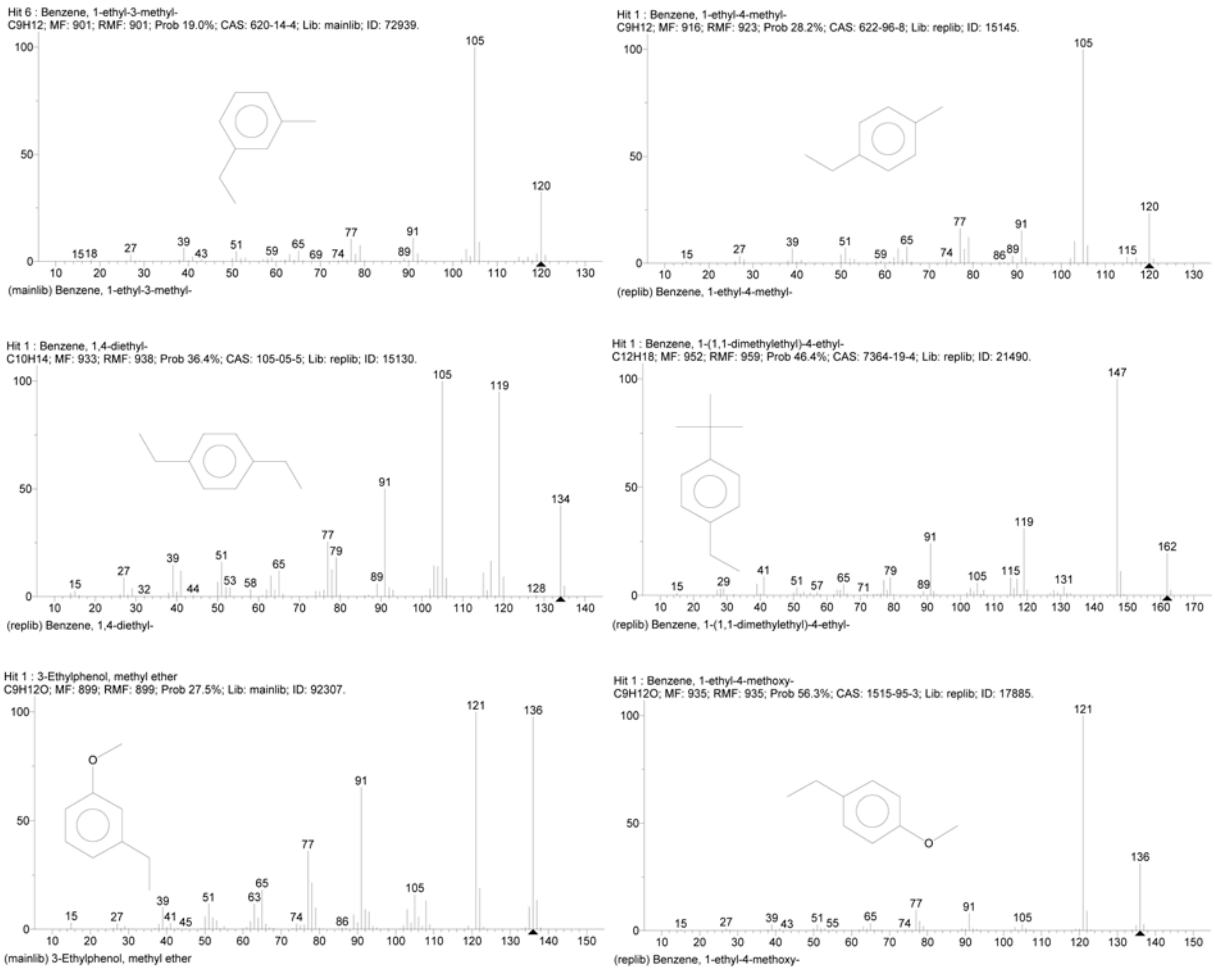


Fig.ESI-18 The analysis of crude product by GC-MS when the alkyne bearing electron-donating groups (R = 3-Me, 4-Me,4-Et, 4-C(CH_3)$_3$, 3-OMe, 4-OMe,) as substrate

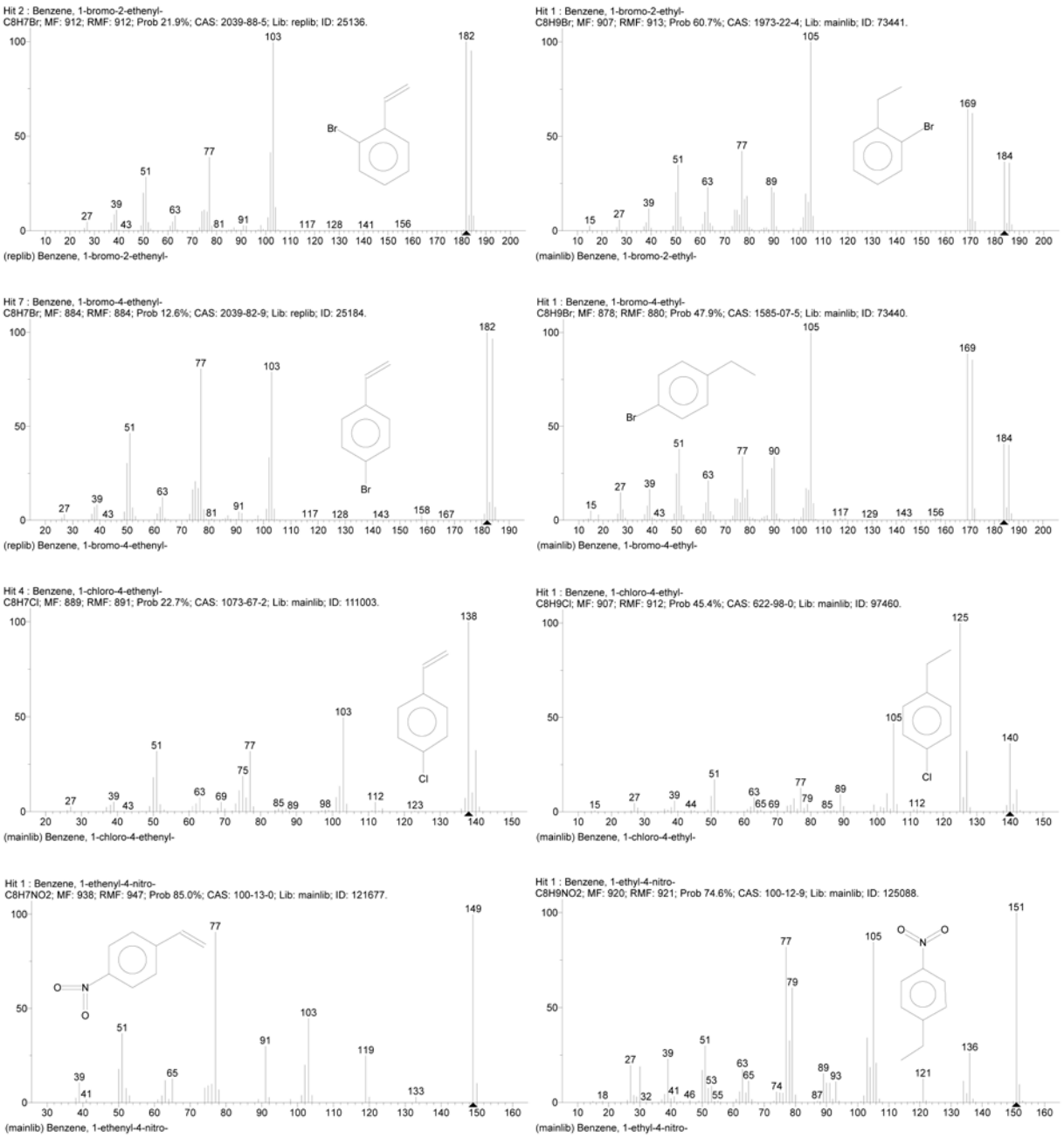


Fig.ESI-19 The analysis of crude product by GC-MS when the alkyne bearing electron-withdrawing groups (R = 2-Br, 4-Br, 4-Cl, 4-NO_2) as substrate

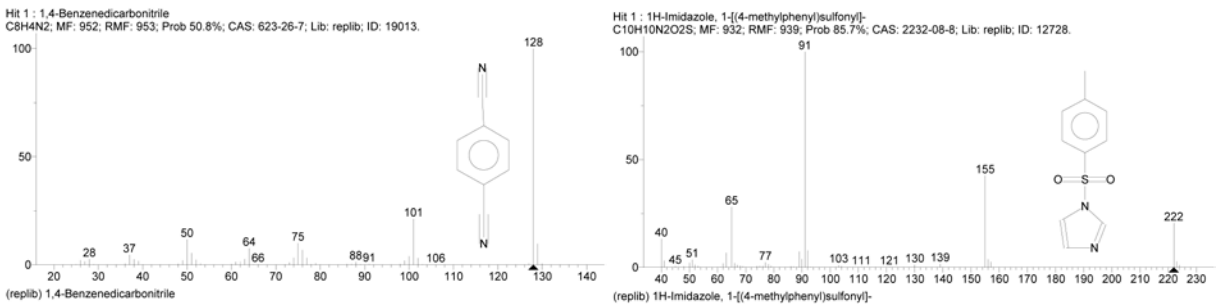
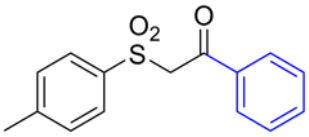


Fig.ESI-20 The analysis of byproduct **4f** and **4g** by GC-MS when the substrate is 4-Ethynylbenzonitrile



1-phenyl-2-tosylethan-1-one **(4h).**

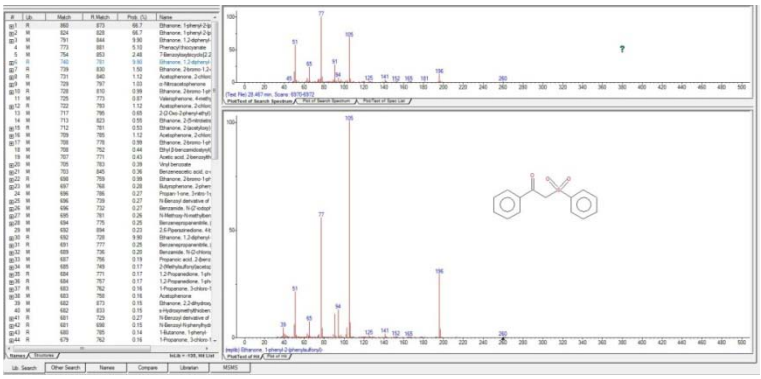


Fig.ESI-21 The analysis of sulfonyl ketone **4h** by GC-MS

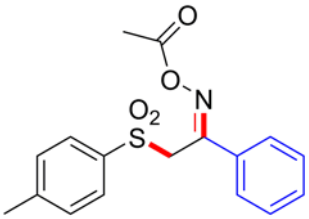


Fig.ESI-22 See characterization data and NMR spectra of products for the details data of **4i**

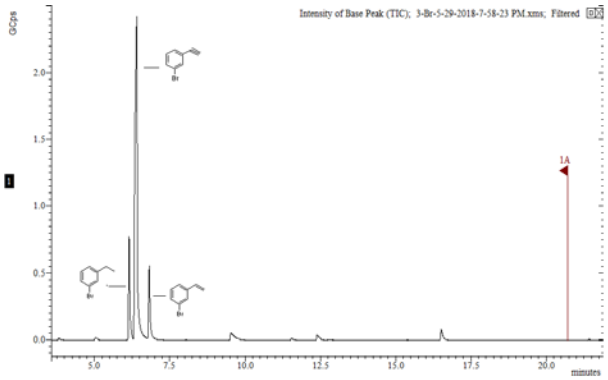


Fig.ESI-23 The analysis of crude product about m-bromobenzene acetylene system

6. Mechanistic Studies

6.1 Radial Trapping Experiments and High Resolution Quality Spectrum Data

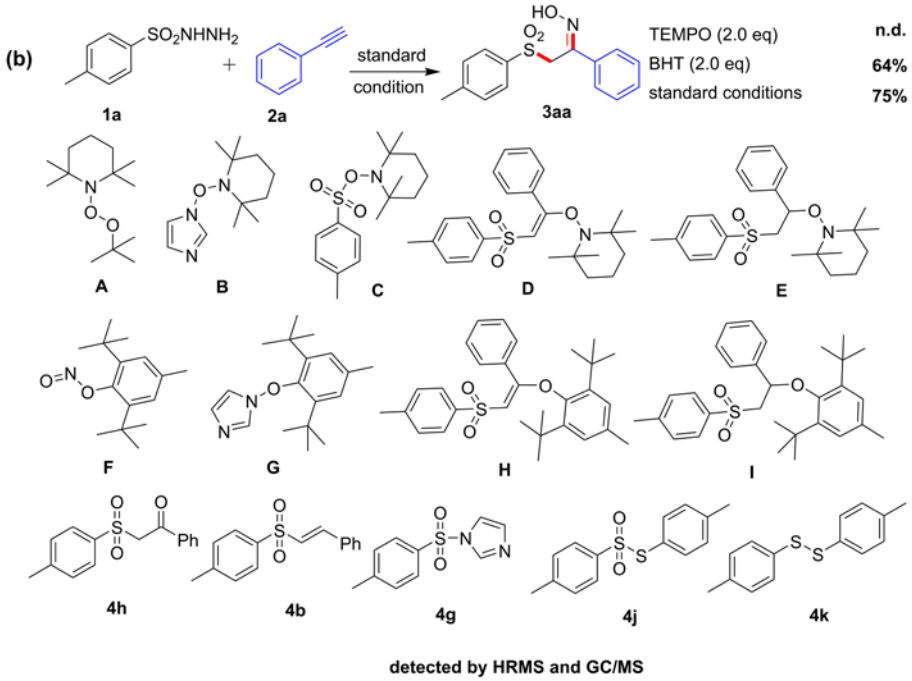


Table ESI-1 The HRMS data about the adducts of TEMPO, the adducts of BHT and some byproduct or intermediates

名称	Molecular Formula	$[M+H]^+$		$[M+Na]^+$	
		Calculate	found	Calculate	found
A	$C_{13}H_{27}NO_2$	230.2115	230.2123	252.1934	/
B	$C_{12}H_{21}N_3O$	224.1757	/	246.1577	246.1600
C	$C_{16}H_{25}NO_3S$	312.1628	/	334.1447	334.1447
D	$C_{24}H_{31}NO_3S$	414.2097	414.2082	436.1917	436.1922
E	$C_{24}H_{33}NO_3S$	416.2254	416.2249	438.2073	/
F	$C_{15}H_{23}NO_2$	250.1802	250.1794	272.1621	/
G	$C_{18}H_{26}N_2O$	287.2118	287.2116	309.1937	/
H	$C_{30}H_{36}O_3S$	477.2458	477.2457	499.2277	/
I	$C_{30}H_{38}O_3S$	479.2614	479.2637	501.2434	501.2437
4h	$C_{15}H_{14}O_3S$	275.0736	275.0743	297.0556	297.0557
4b	$C_{15}H_{14}O_2S$	259.0787	259.0793	281.0607	/
4g	$C_{10}H_{10}N_2O_2S$	223.0536	223.0540	245.0355	/
4j	$C_{14}H_{14}O_2S_2$	279.0508	279.0932	301.0327	301.1428
4k	$C_{14}H_{14}S_2$	247.0610	247.0790	269.0429	/

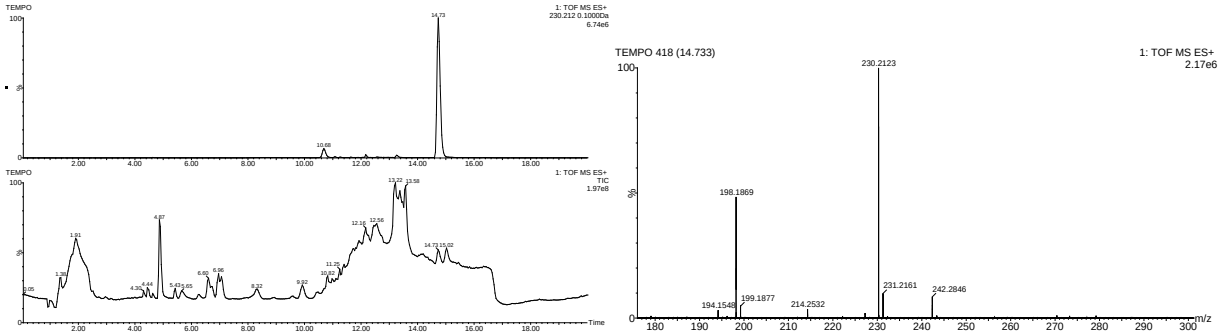


Fig.ESI-24 The analysis result of intermediate **A** by UPLC-TOF/MS (**[A+H]$^+$ Cal: 230.2115, Found: 230.2123**)

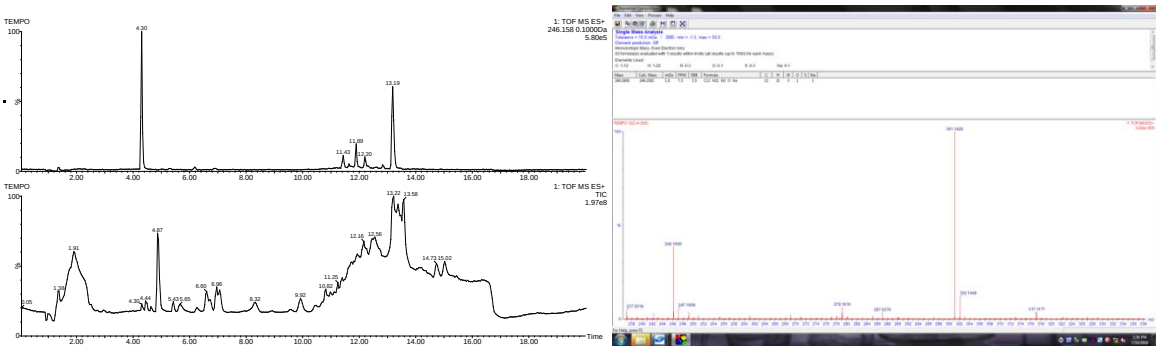


Fig.ESI-25 The analysis result of intermediate **B** by UPLC-TOF/MS and GC-MS (**[B+Na]$^+$ Cal: 246.1577, Found: 246.1600**)

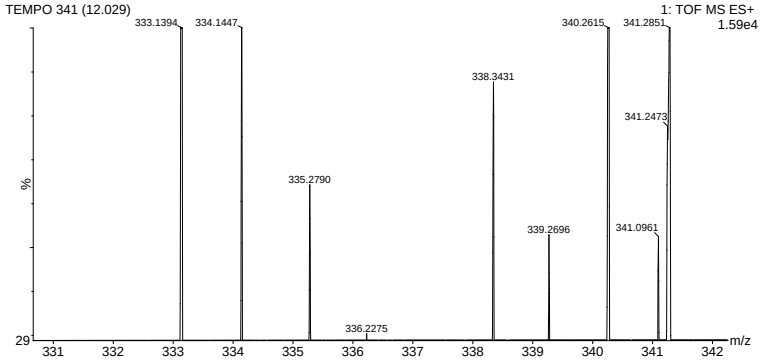


Fig.ESI-26 The analysis result of intermediate **C** by UPLC-TOF/MS (**[C+Na]$^+$ Cal: 334.1447, Found: 334.1447**)

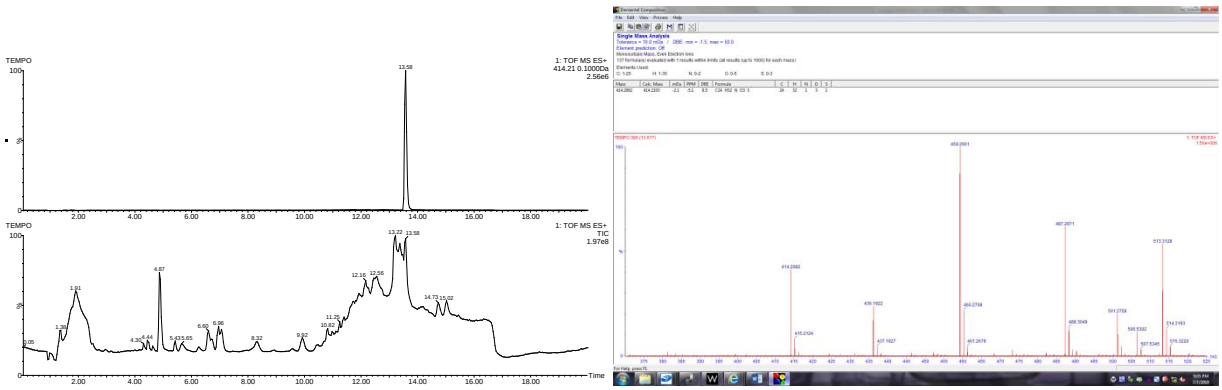


Fig.ESI-27 The analysis result of intermediate **D** by UPLC-TOF/MS and GC-MS (**[D+H]$^+$ Cal: 414.2097, Found: 414.2082**)

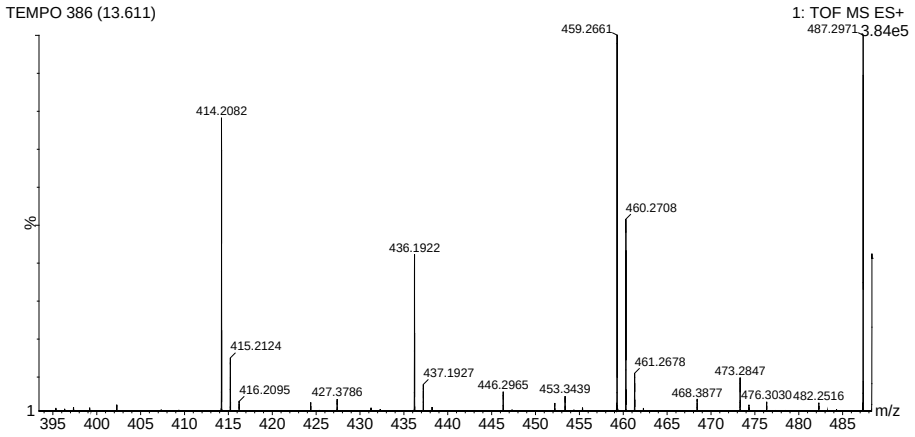


Fig.ESI-28 The analysis result of intermediate **D** by UPLC-TOF/MS (**[D+Na]$^+$ Cal: 436.1917, Found: 436.1922**)

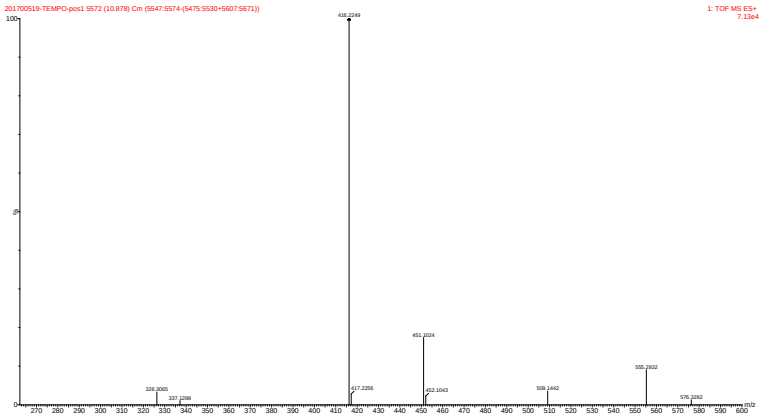


Fig.ESI-29 The analysis result of intermediate **E** by UPLC-TOF/MS (**[E+H]$^+$ Cal: 416.2254, Found: 416.2249**)

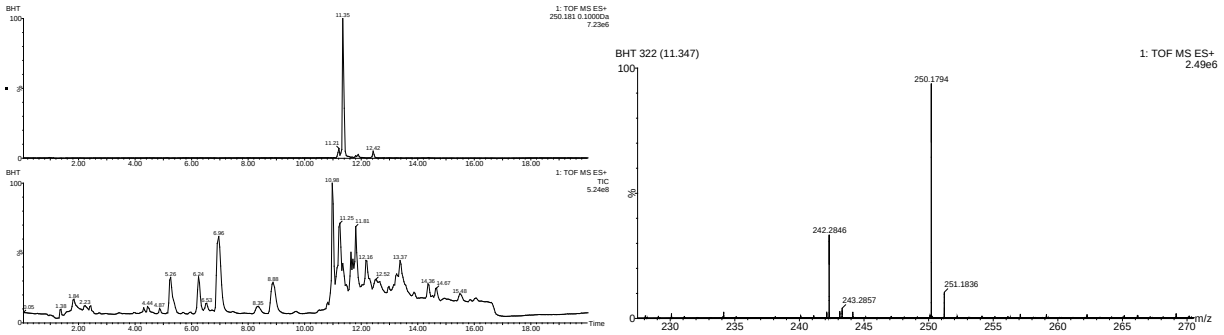


Fig.ESI-30 The analysis result of intermediate **F** by UPLC-TOF/MS (**[F+H]$^+$ Cal: 250.1802, Found: 250.1794**)

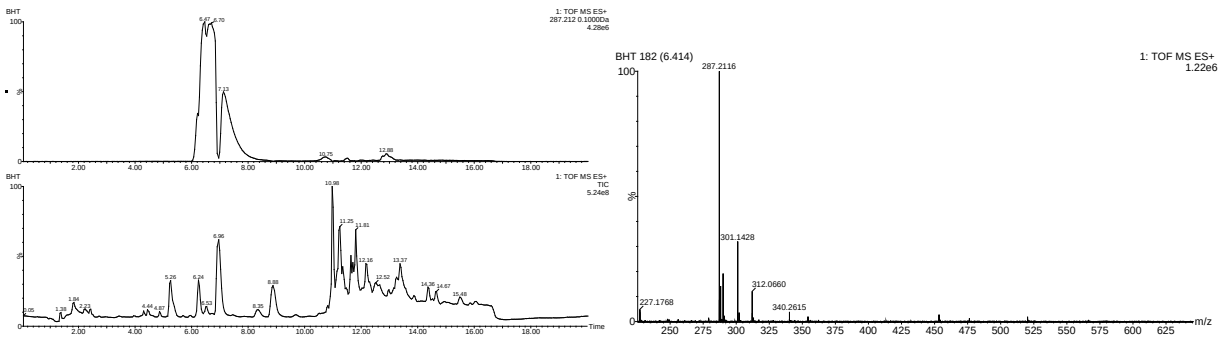


Fig.ESI-31 The analysis result of intermediate **G** by UPLC-TOF/MS (**[G+H]$^+$ Cal: 287.2118, Found: 287.2116**)

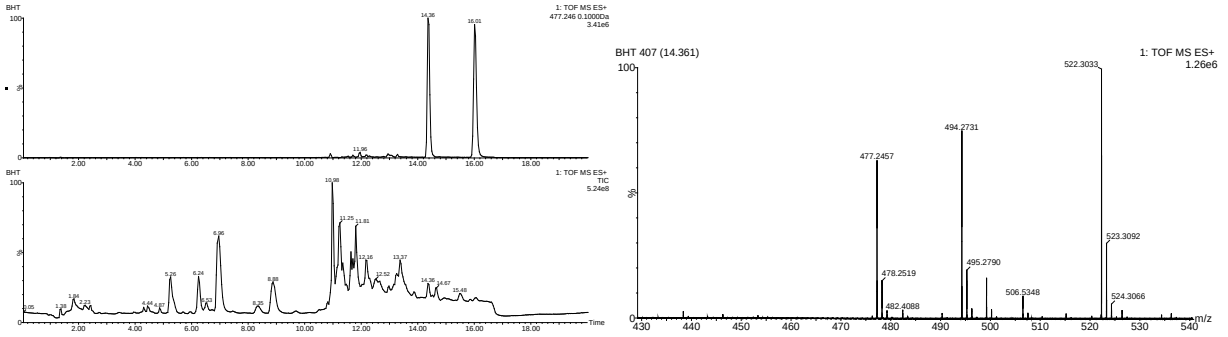


Fig.ESI-32 The analysis result of intermediate **H** by UPLC-TOF/MS (**[H+H]$^+$ Cal: 477.2458, Found: 477.2457**)

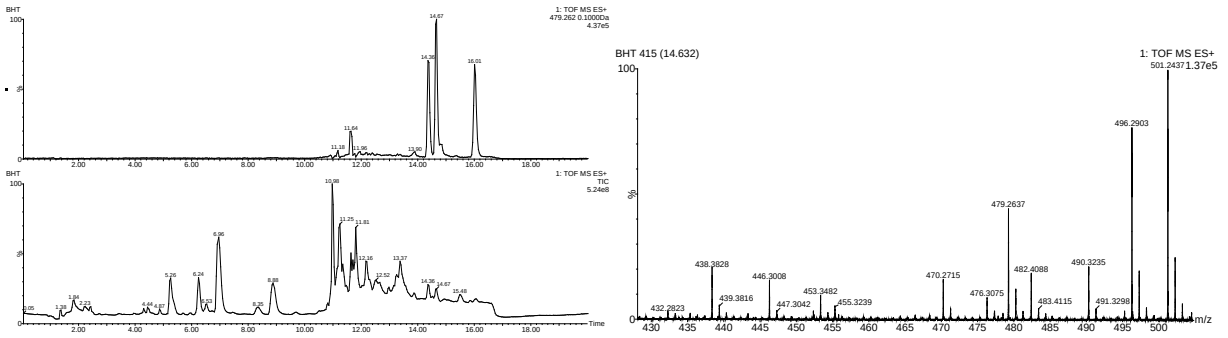


Fig.ESI-33 The analysis result of intermediate **I** by UPLC-TOF/MS (**[I+H]$^+$ Cal: 479.2614, Found: 479.2637**)

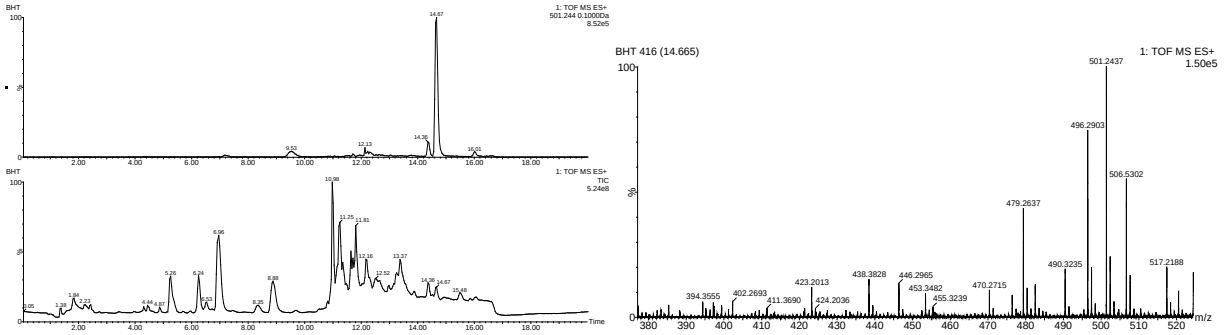


Fig.ESI-34 The analysis result of intermediate **I** by UPLC-TOF/MS (**[I+Na]$^+$ Cal: 501.2434, Found: 201.2437**)

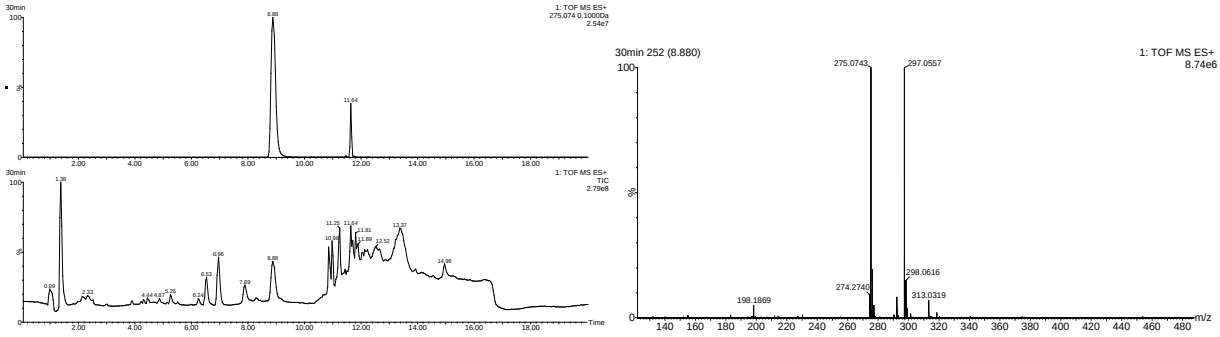


Fig.ESI-35 The analysis result of intermediate or byproduct **4h** by UPLC-TOF/MS (**[4h+H]$^+$ Cal: 275.0736, Found:275.0743**)

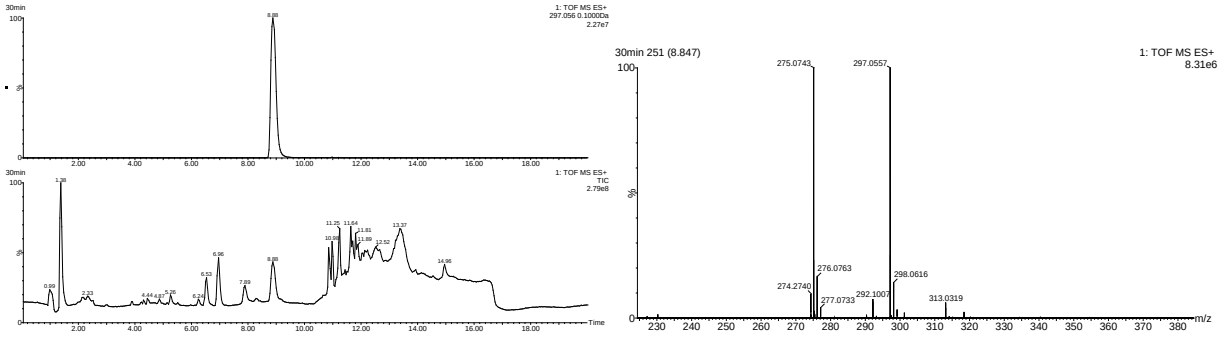


Fig.ESI-36 The analysis result of intermediate or byproduct **4h** by UPLC-TOF/MS (**[4h+Na]$^+$ Cal: 297.0556, Found: 297.0557**)

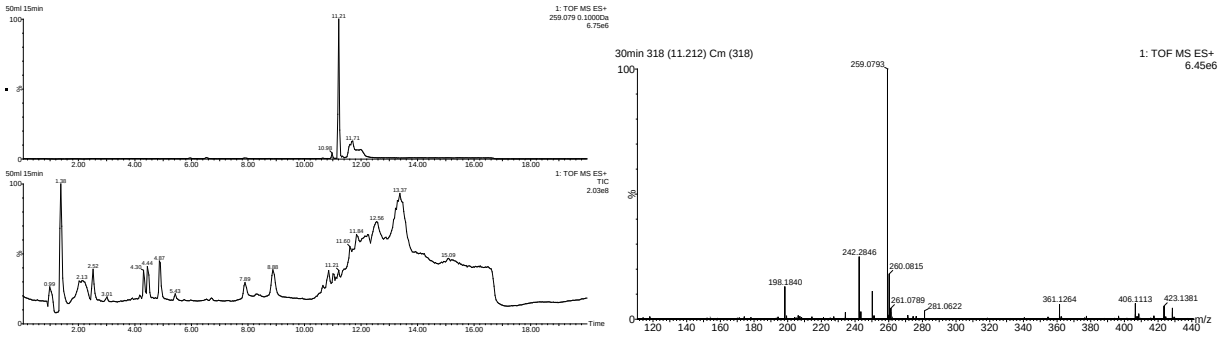


Fig.ESI-37 The analysis result of intermediate or byproduct **4b** by UPLC-TOF/MS (**[4b+H]$^+$ Cal: 259.0787, Found: 259.0793**)

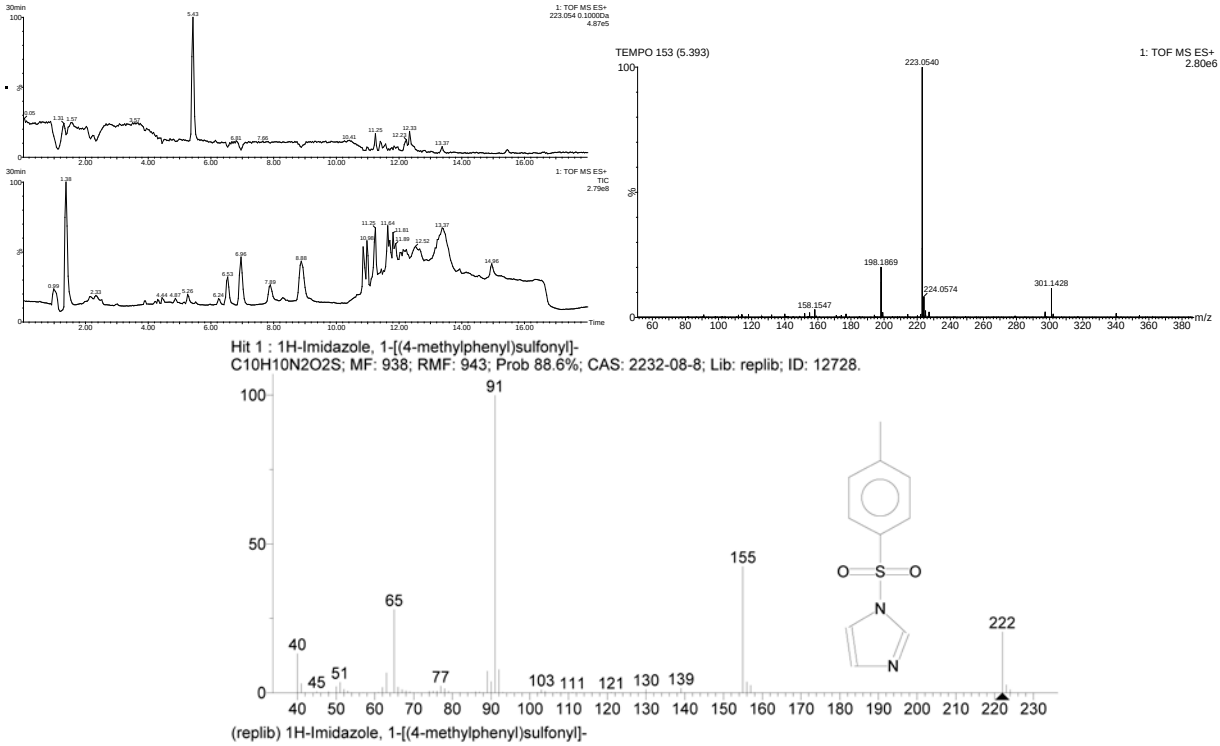


Fig.ESI-38 The analysis result of intermediate or byproduct **4g** by UPLC-TOF/MS and GC-MS (**[4g+H]$^+$ Cal: 223.0541, Found: 223.0540**)

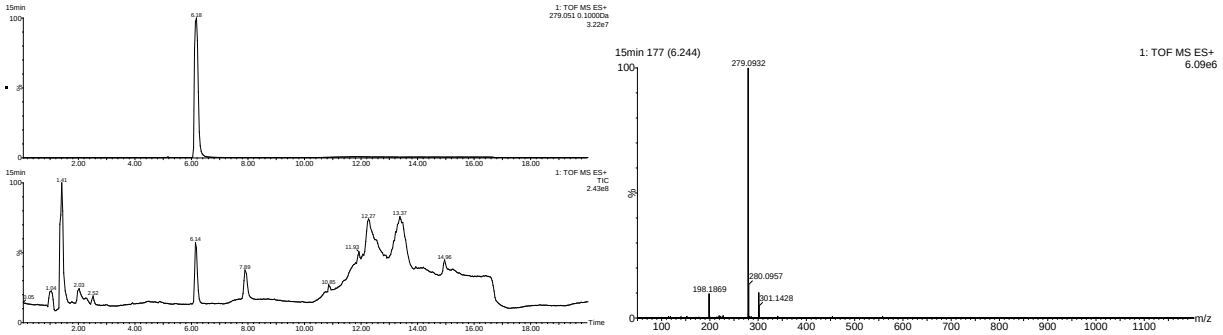


Fig.ESI-39 The analysis result of intermediate or byproduct **4j** by UPLC-TOF/MS (**[4j+H]$^+$ Cal: 279.0508, Found: 279.0932**)

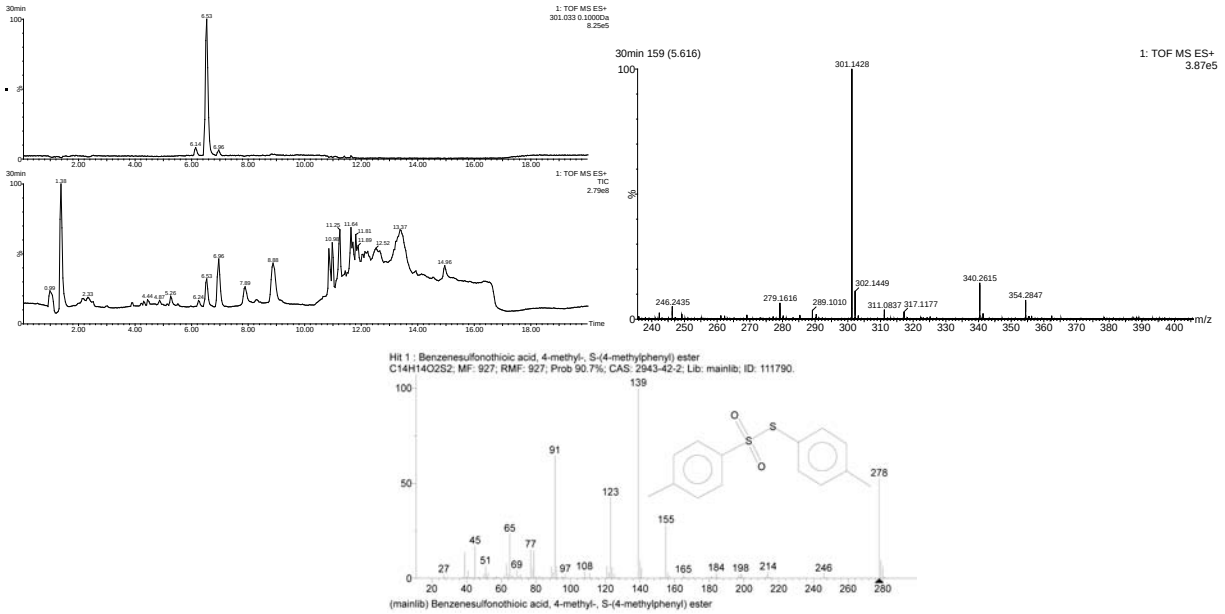


Fig.ESI-40 The analysis result of intermediate or byproduct **4j** by UPLC-TOF/MS and GC-MS (**[4j+Na]$^+$ Cal: 301.0327, Found: 301.1428**)

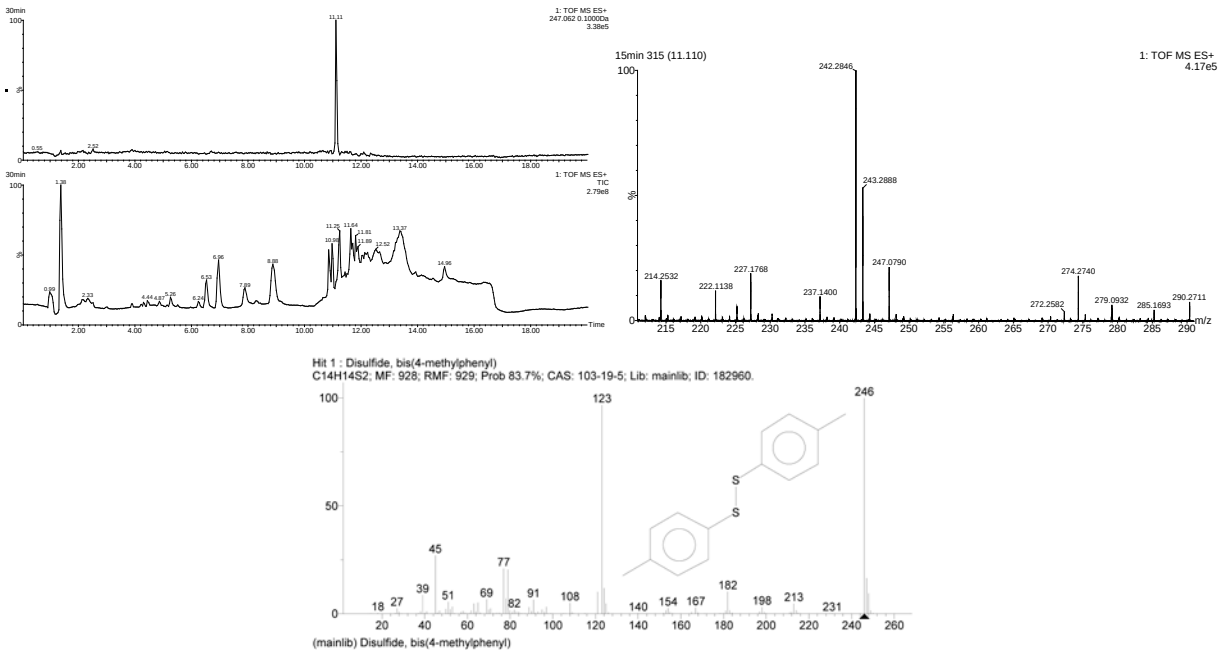


Fig.ESI-41 The analysis result of intermediate or byproduct **4k** by UPLC-TOF/MS and GC-MS (**[4k+H]$^+$ Cal: 247.0610, Found: 247.0790**)

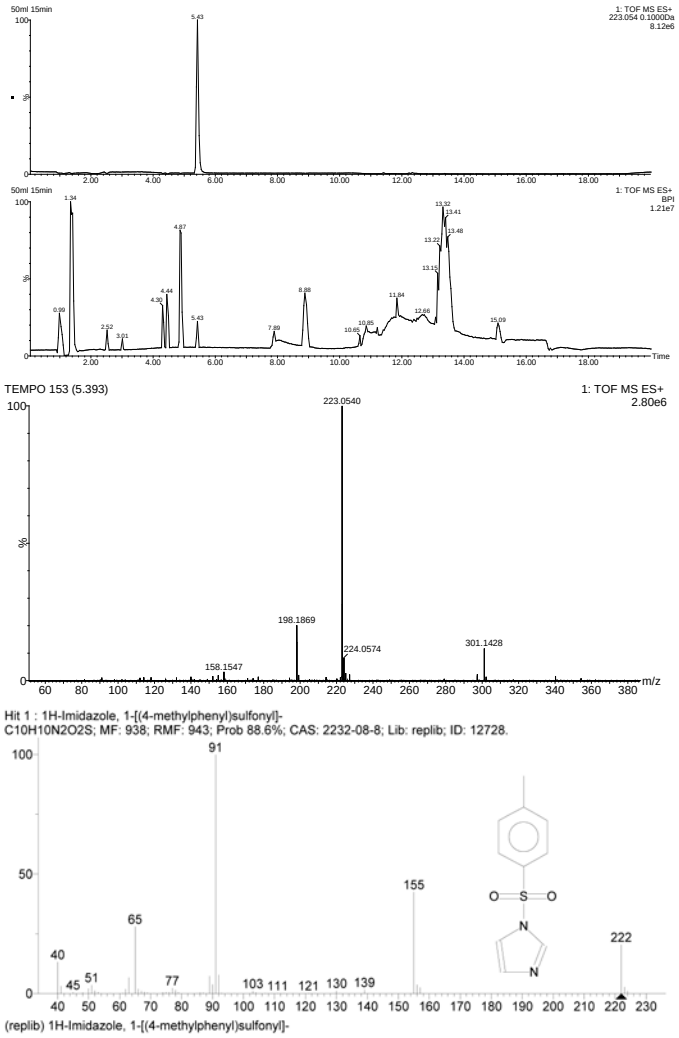
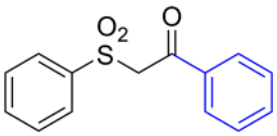


Fig.ESI-42 The analysis result of intermediate **4g** (t_R = 5.43min) by UPLC-TOF/MS and GC-MS ([**4g**+H]$^+$ Cal: 223.0541, Found: 223.0540)

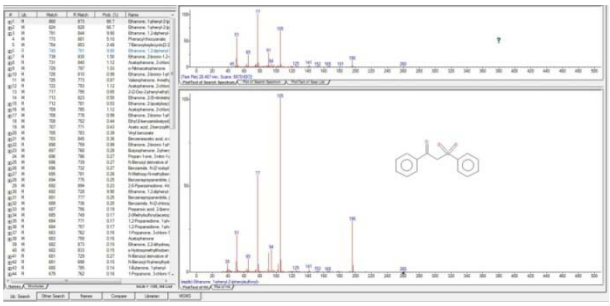
6.2 Intermediate Experiments

a. General procedures for the synthesis of 1-phenyl-2-(phenylsulfonyl)ethanone **4h'**

1-phenyl-2-(phenylsulfonyl)ethanone **4h'** was prepared according to the literature procedure.[2] At ambient temperature, 1 mmol of ketones in the presence of (121 mg, 1.2 mmol) Et_3N was stirred for 30 min in 5 mL of MeOH, then the mixture was treated with 1.2 mmol of benzene sulfinic acid sodium, (254 mg, 1 mmol) of molecular iodine and stirred in dark (aluminium foil wrapped around vessel) at room temperature. After completion of the reaction (monitored by TLC) solvent was removed in vacuum, diluted with ethyl acetate (15 mL) and washed sequentially with sat. sodium thiosulfate soln, water then brine. The organic layer was dried over Na_2SO_4. Removal of the solvent in vacuum and purification of the residue by silica gel chromatography with n-hexane-acetone as eluent gave the desired products **4h'**.

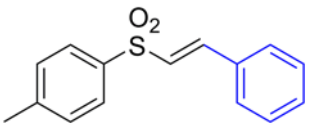


1-phenyl-2-(phenylsulfonyl)ethan-1-one (4h').



b. General procedures for the synthesis of (E)-1-methyl-4-(styrylsulfonyl)benzene **4b**

(E)-1-methyl-4-(styrylsulfonyl)benzene **4b** was prepared according to the literature procedure.[3] A 15mL Schlenk tube equipped with a magnetic stirring bar was charged with I_2 (1.5 equiv., 380 mg), alkenes (1.0 mmol), substituted various Sodium methylbenzene sulfonate (1.5 mmol) and H_2O (2 mL). The mixture was then stirred at room temperature for 2 h under air atmosphere, and then a saturated aqueous $Na_2S_2O_3$ solution (2 mL) was added. Following that, the solution was extracted with ethyl acetate (15～20 mL). Finally, the combined organic phases were concentrated and the remaining residue was purified by column chromatography on silica gel to provide the desired product **4b**.



(E)-1-methyl-4-(styrylsulfonyl)benzene (4b).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 15.4 Hz, 1H), 7.52 – 7.45 (m, 2H), 7.43 – 7.37 (m, 3H), 7.35 (d, *J* = 8.0 Hz, 2H), 6.85 (d, *J* = 15.4 Hz, 1H), 2.44 (s, 3H). This compound was known.[4]

c. Deuterium labeling experiments

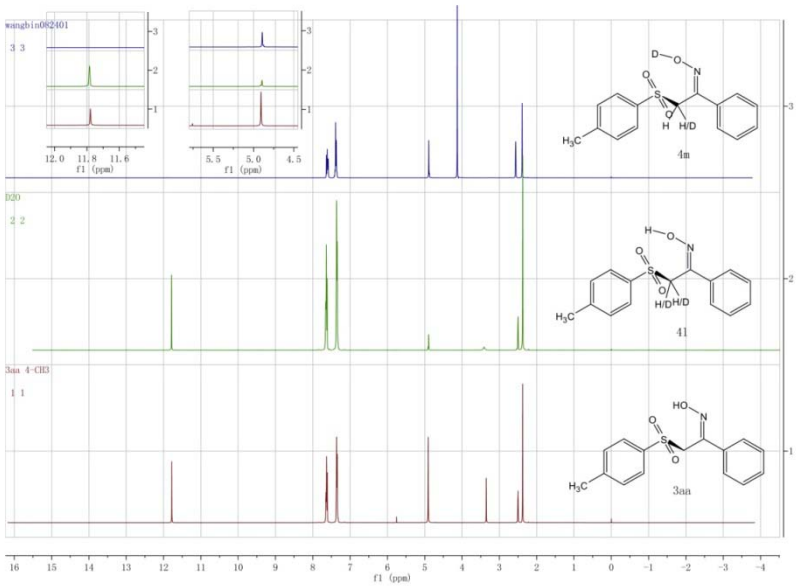


Fig.ESI-43 The ^{1}H NMR analysis of **3aa** through deuterium labeling experiments.

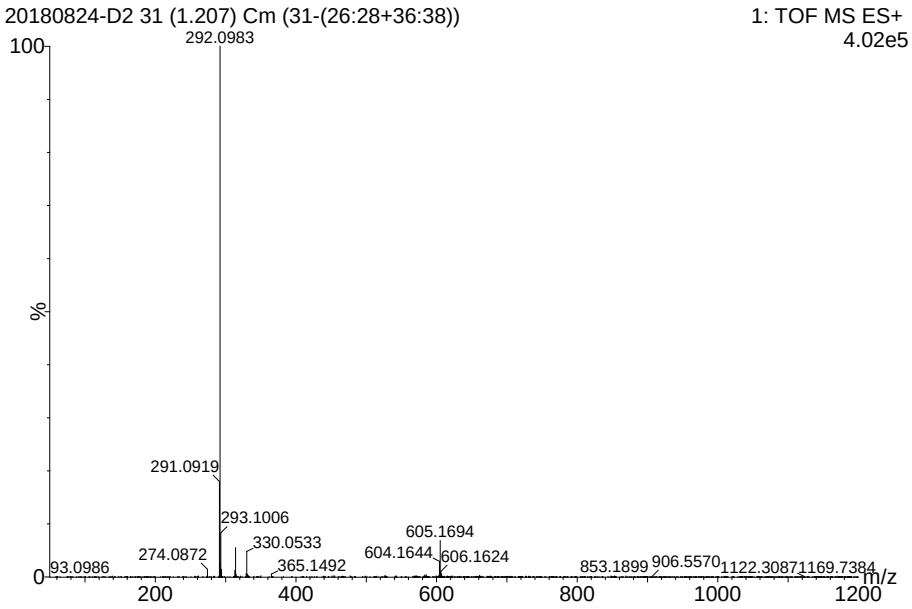


Fig.ESI-44 The analysis result of **4l** or **4m** by UPLC-TOF/MS (**[4m+H]$^{+}$ Cal: 292.0976, Found: 292.0983**)

7. General Procedure for UPLC

7.1 UPLC Analysis Methods

HPLC grade methanol was procured from Merck (Darmstadt, Germany). Water for UPLC analysis was purified by a Milli Q water purification system (Millipore Corporation, MA, USA). PTFE membrane filter (0.22 μm) was purchased from Waters Co. (Milford, MA). The sample was weighed in electronic balance (BP211D, Sartorius, Germany). The UPLC system (Agilent technologies co., USA) was equipped with a dual pump, an autosampler including a column oven controller, which was connected in ultravioletdetector (UV) quantitative analyzing and UV spectra acquisition. The chromatographic separation was performed on a EclipsePlus C_{18} column (1.8 μm, ø 2.1 mm × 100 mm, agilent, USA). The mobile phase was methanol (A) and water (B). A gradient program was as follows: 0-3 min, 30% A; 5-8 min, 45 % A; 9-12 min, 90 % A. The beginning gradient was held for 5 min. The flow rate was 0.4 mL/min. The injection volume was 1 μL and the column temperature was maintained at 25 °C. The detection wavelength was set at 250 nm.

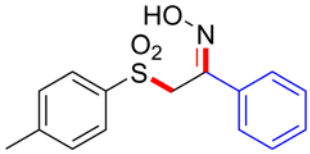
7.2 Preparation of Standard Solutions and Calibration Curves

The reference standard was dissolved in methanol to final concentration of 1.009 $mg \cdot mL^{-1}$ for product **3aa**. Calibration standard working solutions were prepared by diluting the above standard solutions in appropriate proportions with methanol, to gain the desired concentrations. Each calibration curve was constructed by running samples at six different concentrations in triplicate. The calibration curve was obtained by plotting the peak area of the compound at each level prepared versus the concentration of the sample. All standard solutions were stored in the refrigerator at 4 °C and were filtered through a 0.22 μm syringe filter before use.

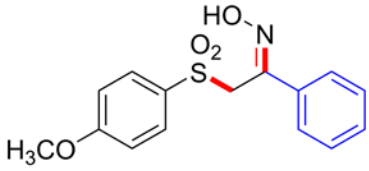
7.3 Preparation of Samples

After the end of model reaction of different optimization conditions, thereaction solution including product 3aa was diluted to the same volume. The obtained sample solutions was then diluted to desired concentrations with methanol and passed through a 0.22 μm Millipore membrane filte prior to analysis.

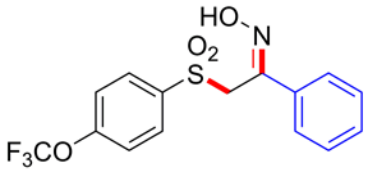
8. Characterization Data of Products



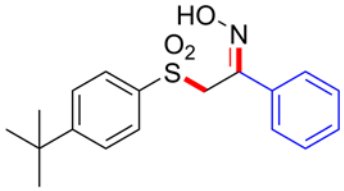
1-phenyl-2-tosylethan-1-one oxime (3aa). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 77.8% yield; mp = 201 - 202 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.78 (s, 1H), 7.65 – 7.61 (m, 4H), 7.38 – 7.32 (m, 5H), 4.91 (s, 2H), 2.38 (s, 3H).; ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.67, 144.37, 136.88, 134.57, 129.49, 128.96, 128.21, 127.88, 126.40, 51.44, 21.11.; HRMS[M+H]$^{+}$ calcd for $C_{15}H_{16}NO_3S$: 290.0851, found 290.0857. This compound was known.[5]



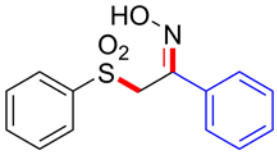
2-((4-methoxyphenyl)sulfonyl)-1-phenylethan-1-one oxime (3ab). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow solid: 39.8% yield; mp= 153-154 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.75 (s, 1H), 7.66 – 7.63 (m, 4H), 7.36 – 7.33 (m, 3H), 7.07 – 7.04 (m, 2H), 4.88 (s, 2H), 3.83 (s, 3H).; ^{13}C NMR (100 MHz, DMSO-d_6) δ 163.31, 145.81, 134.59, 131.22, 130.17, 128.95, 128.21, 126.40, 114.20, 55.77, 51.60; HRMS[M+H]$^{+}$ calcd for $C_{15}H_{16}NO_4S$: 306.0800, found 306.0808. This compound was known.[5]



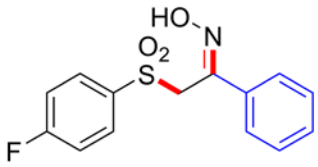
1-phenyl-2-((4-(trifluoromethoxy)phenyl)sulfonyl)ethan-1-one oxime (3ac). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 54.2% yield; mp= 137-139 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.86 (s, 1H), 7.86 (d, J=7.2Hz, 2H), 7.61(d, J=7.2Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.37 – 7.31 (m, 3H), 5.01 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 151.93, 145.45, 138.32, 134.35, 130.79, 129.03, 128.23, 126.39, 123.68, 121.08, 118.54, 51.30; ^{19}F NMR (376 MHz, DMSO-d_6): δ [ppm] = -56.7. HRMS[M+H]$^{+}$ calcd for $C_{15}H_{13}F_3NO_4S$: 360.0517, found 360.0514. This compound was known.[5]



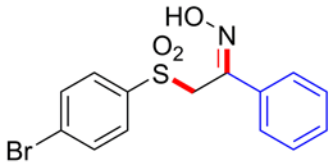
2-((4-(tert-butyl)phenyl)sulfonyl)-1-phenylethan-1-one oxime(3ad). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 60.3% yield; mp= 174-175 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.86 (s, 1H), 7.66 – 7.64 (m, 2H), 7.58 (dd, 7.6, 1.6Hz, 2H),7.54-7.52(m, 2H),7.34-7.27(m,3H), 4.92 (s, 2H), 1.28 (s, 9H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 156.92, 145.67, 136.93, 134.51, 128.89, 128.16, 127.71, 126.33, 125.90, 51.37, 34.94, 30.76; HRMS[M+H]$^{+}$ calcd for $C_{18}H_{21}NO_3S$: 332.1320, found 332.1322. This compound was known.[5]



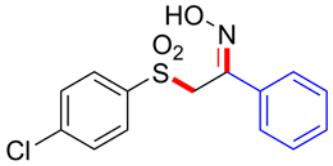
1-phenyl-2-(phenylsulfonyl)ethan-1-one oxime(3ae). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 58.8% yield; mp= 137-138 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.81 (s, 1H), 7.76– 7.74 (d, J = 7.6 Hz ,2H), 7.71-7.63 (m, 3H), 7.56 (t, J = 7.6 Hz, 2H), 7.36 – 7.32 (m, 3H), 4.95 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.58, 139.69, 134.53, 133.91, 129.07, 129.03, 128.25, 127.84, 126.39, 51.35; HRMS[M+H]$^{+}$ calcd for $C_{14}H_{13}NO_3S$: 276.0694, found 276.0700. This compound was known.[5]



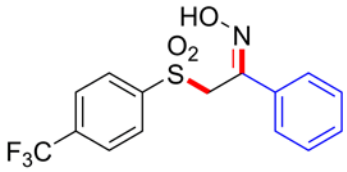
2-((4-fluorophenyl)sulfonyl)-1-phenylethan-1-one oxime(3af). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 71.4% yield; mp= 120-121 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.79 (s, 1H), 7.81 – 7.78 (m, 2H), 7.65 – 7.63 (m, 2H), 7.41 – 7.32 (m, 5H), 4.96 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.41, 163.90, 145.56, 135.84(d, J_{CF} = 2.5 Hz), 134.44, 131.24(d, J_{CF} = 10.0 Hz), 129.06, 128.26, 126.40, 116.24(d, J_{CF} = 229Hz), 51.41; ^{19}F NMR (376 MHz, DMSO-d_6): δ [ppm] = -104.7. HRMS[M+H]$^{+}$ calcd for $C_{14}H_{13}FNO_3S$: 294.0600, found 294.0599. This compound was known.[5]



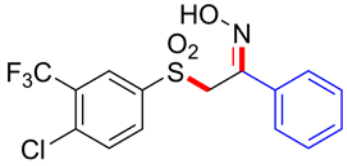
2-((4-bromophenyl)sulfonyl)-1-phenylethan-1-one oxime(3ag). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 66.4% yield; mp= 157-158 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.80 (s, 1H), 7.78 – 7.76 (m, 2H), 7.66–7.64 (m, 4H), 7.37-7.35 (m, 3H), 4.97 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.44, 138.73, 134.39, 132.10, 129.98, 129.03, 128.25, 128.14, 126.39, 51.33; HRMS[M+H]$^{+}$ calcd for $C_{14}H_{13}BrNO_3S$: 353.9800, found 353.9797. This compound was known.[5]



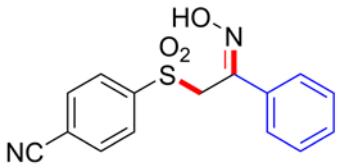
2-((4-chlorophenyl)sulfonyl)-1-phenylethan-1-one oxime(3ah). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 65.4% yield; mp= 135-136 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.81 (s, 1H), 7.75 – 7.73 (d, J = 8.4 Hz, 2H), 7.65 – 7.62 (m, 4H), 7.39 – 7.33 (m, 3H), 4.98 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.47, 139.03, 138.30, 134.41, 129.98, 129.18, 129.07, 128.28, 126.42, 51.35; HRMS[M+Na]$^{+}$ calcd for $C_{14}H_{12}ClNNaO_3S$: 332.0124, found 332.0127. This compound was known.[5]



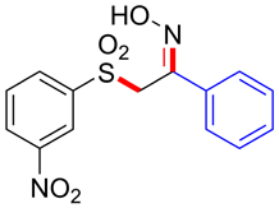
1-phenyl-2-((4-(trifluoromethyl)phenyl)sulfonyl)ethan-1-one oxime(3ai). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 55.2% yield; mp= 146-147 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.84 (s, 1H), 7.97 – 7.92 (m, 4H), 7.64-7.62 (m, J = 7.4, 2H), 7.38 – 7.31 (m, 3H), 5.05 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.32, 143.27, 134.32, 133.47(q, J_{CF} = 32 Hz), 129.15, 129.10, 128.28, 127.48, 126.23(q, J_{CF} = 3.7 Hz), 123.41(q, J_{CF} = 272 Hz), 51.21. ^{19}F NMR (376 MHz, DMSO-d_6): δ [ppm] = -61.7. HRMS[M+H]$^{+}$ calcd for $C_{15}H_{13}F_3NO_3S$: 344.0568, found 344.0574. This compound was known.[5]



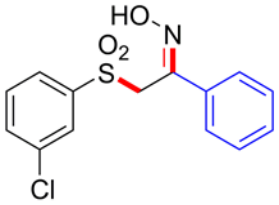
2-((4-chloro-3-(trifluoromethyl)phenyl)sulfonyl)-1-phenylethan-1-one oxime(3aj). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 61.4% yield; mp= 162-163 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.92 (s, 1H), 8.03 (dd, J = 8.4, 1.6 Hz, 1H), 7.97 – 7.91 (m, 2H), 7.58 (d, J = 7.2 Hz, 2H), 7.37-7.29 (m, 3H), 5.11 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.40, 138.75, 136.81, 134.13, 133.71, 132.78, 129.16, 128.29, 127.44(q, J_{CF} = 29Hz), 126.69 (q, J_{CF} = 57Hz), 126.07, 121.99 (q, J_{CF} = 272Hz), 51.22.; ^{19}F NMR (376 MHz, DMSO-d_6): δ [ppm] = -61.9. HRMS[M+H]$^{+}$ calcd for $C_{15}H_{12}ClF_3NO_3S$: 378.0179, found 378.0187. This compound was known.[5]



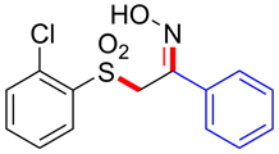
4-((2-(hydroxyimino)-2-phenylethyl)sulfonyl)benzonitrile(3ak). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 46.2% yield; mp= 160-161 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.80 (s, 1H), 8.07 – 8.04 (m, 2H), 7.93 – 7.91 (m, 2H), 7.65 – 7.63 (m, 2H), 7.40 – 7.33 (m, 3H), 5.04 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.25, 143.41, 134.30, 133.13, 129.17, 128.90, 128.33, 126.44, 117.64, 116.27, 51.19; HRMS[M+H]$^+$ calcd for $C_{15}H_{13}N_2O_3S$: 301.0647, found 301.0654. This compound was known.[5]



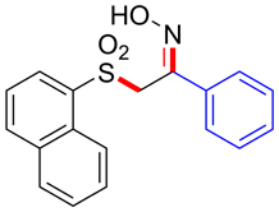
2-((3-nitrophenyl)sulfonyl)-1-phenylethan-1-one oxime(3al). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white crystal: 34.7% yield; mp= 168-169 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.87 (s, 1H), 8.53-8.51 (m, 1H), 8.37 (s, 1H),8.20-8.18 (m, 1H), 7.86 (t, *J* = 8.0 Hz, 1H), 7.62 – 7.60 (m, 2H), 7.36 – 7.30 (m, 3H), 5.10 (s, 2H);^{13}C NMR (100 MHz, DMSO-d_6) δ 147.45, 145.43, 140.92, 134.21, 134.14, 131.20, 129.16, 128.53, 128.33, 126.43, 123.11, 51.29; HRMS[M+H]$^+$ calcd for $C_{14}H_{13}N_2O_5S$: 321.0545, found 321.0553. This compound was known.[5]



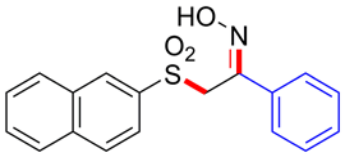
2-((3-chlorophenyl)sulfonyl)-1-phenylethan-1-one oxime(3am). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 44.2% yield; mp = 112-113 °C; ^{1}H NMR (400 MHz, DMSO-d6) δ 11.88 (s, 1H), 7.78-7.75(ddd, J = 8.0, 2.1, 1.1 Hz, 1H), 7.72-7.70 (m, 2H), 7.64 – 7.56 (m, 3H), 7.37 – 7.33 (m, 3H), 5.02 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) 145.43, 141.35, 134.34, 133.88, 133.61, 131.10, 129.13, 128.26, 127.63, 126.64, 126.38, 51.26; HRMS[M+Na]$^+$ calcd for $C_{14}H_{12}ClNNaO_3S$: 332.0124, found 332.0124. This compound was known.[5]



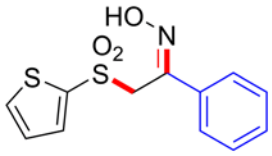
2-((2-chlorophenyl)sulfonyl)-1-phenylethan-1-one oxime(3an). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow solid: 31.0% yield; mp= 103-104 °C; ^{1}H NMR (400 MHz, Chloroform-d) δ 9.13 (s, 1H), 7.87 (d, J= 8.4Hz,1H), 7.53 – 7.51 (m, 2H), 7.44 – 7.41 (m, 2H), 7.36 – 7.26 (m, 4H), 5.01 (s, 2H);^{13}C NMR (100 MHz, Chloroform-d) δ 147.62, 137.49, 134.92, 133.67, 133.10, 131.83, 131.70, 129.99, 128.65, 127.21, 126.69, 50.95; HRMS[M+H]$^+$ calcd for $C_{14}H_{12}ClNNaO_3S$: 332.0124, found 332.0127. This compound was known.[5]



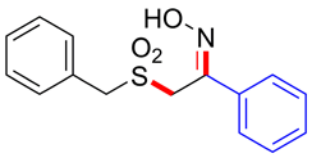
2-(naphthalen-1-ylsulfonyl)-1-phenylethan-1-one oxime(3aq). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 75.8% yield; mp= 168-169 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.74 (s, 1H), 8.76 (dd, *J* = 8.6, 1.2 Hz, 1H), 8.29 (d, *J* = 8.2 Hz, 1H), 8.13 – 8.10 (m, 1H), 8.06-8.04 (m, 1H), 7.78-7.68 (m, 2H), 7.65-7.60 (m, 3H), 7.36 – 7.28 (m, 3H), 5.10 (s, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.38, 135.41, 135.20, 134.63, 133.76, 130.30, 129.22, 129.01, 128.51, 128.43, 128.15, 127.02, 126.33, 124.57, 124.01, 51.32. HRMS[M+H]$^{+}$ calcd for $C_{18}H_{15}NO_3S$: 326.0851, found 326.0850.



2-(naphthalen-2-ylsulfonyl)-1-phenylethan-1-one oxime(3ar). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 71.3% yield; mp= 174-175 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.73 (s, 1H), 8.41 (d, *J* = 1.2 Hz, 1H), 8.10 (d, *J* = 8.8 Hz, 1H), 8.05 (d, *J* = 8 Hz, 1H), 7.80(dd, *J* = 8.6,1.8Hz, 1H), 7.75 – 7.71 (m, 1H), 7.69-7.65 (m, 3H), 7.35-7.30 (m, 3H), 5.03 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.62, 136.80, 134.86, 134.57, 131.51, 129.55, 129.54, 129.36, 129.07, 128.99, 128.20, 127.87, 127.59, 126.42, 122.93, 51.43; HRMS[M+H]$^{+}$ calcd for $C_{18}H_{15}NO_3S$: 326.0851, found 326.0859. This compound was known.[5]

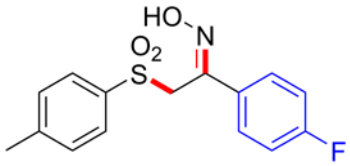


1-phenyl-2-(thiophen-2-ylsulfonyl)ethan-1-one oxime(3as). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow solid: 35.3% yield; mp= 100-101 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.89 (s, 1H), 8.04 (dd, *J* = 4.8, 1.2 Hz, 1H), 7.68 – 7.65 (m, 2H), 7.61 (dd, *J* = 3.6, 1.2 Hz, 1H),7.37-7.35(m, 3H), 7.17 (dd, *J* = 4.8, 4.0 Hz, 1H), 5.04 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.52, 140.17, 135.69, 134.73, 134.51, 129.06, 128.26, 128.13, 126.37, 52.51; HRMS[M+H]$^{+}$ calcd for $C_{12}H_{12}NO_3S_2$: 282.0259, found 282.0262. This compound was known.[5]

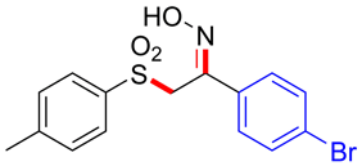


2-(benzylsulfonyl)-1-phenylethan-1-one oxime(3at). This title compound was prepared according to the general

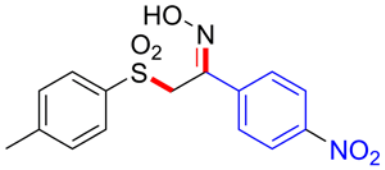
working procedure and purified by column chromatography to give the product as a light yellow oil: 19.7% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.24 (s, 1H), 7.74 – 7.71 (m, 2H), 7.40 (s, 8H), 4.77 (s, 2H), 4.54 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.94, 134.96, 131.30, 129.14, 128.54, 128.48, 128.33, 128.02, 126.36, 60.22, 48.37; HRMS[M+H]$^{+}$ calcd for $C_{15}H_{16}NO_3S$: 290.0851, found 290.0858. This compound was known.[5]



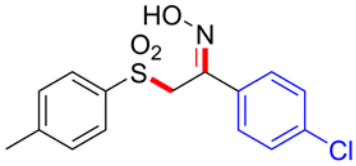
1-(4-fluorophenyl)-2-tosylethan-1-one oxime(3ba). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 55.4% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.79 (s, 1H), 7.70 – 7.65 (m, 2H), 7.62 – 7.60 (m, 2H), 7.37 – 7.35 (m, 2H), 7.21 – 7.15 (m, 2H), 4.92 (s, 2H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 163.84, 161.39, 144.86, 144.46, 136.74, 131.10, 131.07, 129.52, 128.68, 128.59, 127.92, 115.25, 115.04, 51.38, 21.11; ^{19}F NMR (376 MHz, DMSO-d_6): δ [ppm] = -112.7. HRMS[M+H]$^{+}$ calcd for $C_{15}H_{15}FNO_3S$: 308.0757, found 308.0765. This compound was known.[6]



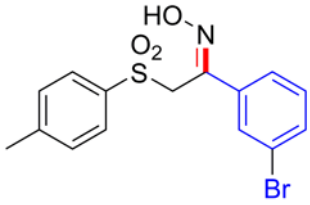
1-(4-bromophenyl)-2-tosylethan-1-one oxime(3ca). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 57.1% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.92 (s, 1H), 7.61 – 7.53 (m, 6H), 7.35 (d, J = 8.0 Hz, 2H), 4.91 (s, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 144.99, 144.53, 136.63, 133.82, 131.18, 129.55, 128.46, 127.94, 122.47, 51.18, 21.13. HRMS[M+H]$^{+}$ calcd for $C_{15}H_{15}BrNO_3S$: 367.9956, found 367.9962. This compound was known.[6]



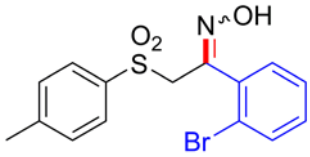
1-(4-nitrophenyl)-2-tosylethan-1-one oxime(3da). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow solid: 60.2% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.34 (s, 1H), 8.21 – 8.18 (m, 2H), 7.92 – 7.89 (m, 2H), 7.62 (d, J = 8.4 Hz, 2H), 7.36 (d, J = 8.0 Hz, 2H), 5.01 (s, 2H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 147.41, 144.66, 144.63, 140.80, 136.49, 129.57, 127.94, 127.56, 123.33, 51.12, 21.05. HRMS[M+H]$^{+}$ calcd for $C_{15}H_{15}N2O_5S$: 335.0702, found 335.0694. This compound was known.[6]



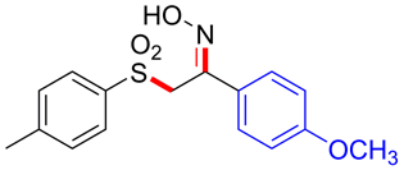
1-(4-chlorophenyl)-2-tosylethan-1-one oxime(3ea). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 53.8 % yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.89 (s, 1H), 7.65 – 7.60 (m, 4H), 7.41 (d, *J* = 8.8 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 4.91 (s, 1H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 144.87, 144.51, 136.65, 133.74, 133.45, 129.53, 128.26, 128.19, 127.94, 51.23, 21.11. HRMS[M+H]$^{+}$ calcd for $C_{15}H_{15}ClNO_3S$: 324.0461, found 324.0449. This compound was known.[6]



1-(3-bromophenyl)-2-tosylethan-1-one oxime(3fa). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow solid: 54.6% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.74 (s, 1H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.63 (t, *J* = 1.9 Hz, 1H), 7.57-7.54 (m, 1H), 7.49-7.46 (m, 1H), 7.25 - 7.19 (m, 3H), 4.69 (s, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 146.69, 145.34, 136.36, 135.78, 132.75, 130.17, 129.75, 129.53, 128.49, 125.48, 122.80, 52.69, 21.77. HRMS[M+H]$^{+}$ calcd for $C_{15}H_{15}BrNO_3S$: 367.9956, found 367.9962. This compound was known.[7]

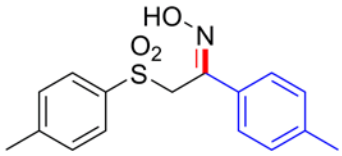


1-(2-bromophenyl)-2-tosylethan-1-one oxime (3ha). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow oil: 24.6% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) 8.66-8.42 (m, 1H), 7.75-7.69 (m, 2H), 7.55-7.48 (m, 1H), 7.38-7.28 (m, 3H), 7.25-7.23 (m, 2H),4.82-4.36 (m, 2H), 2.42-2.40 (m, 3H);^{13}C NMR (101 MHz, Chloroform-*d*) δ 149.13, 148.21, 145.18, 144.95, 137.11, 136.07, 135.41, 133.51, 133.02, 132.70, 132.32, 131.06, 130.97, 130.85, 130.66, 129.98, 129.75, 129.65, 128.42, 128.19, 127.65, 127.23, 121.87, 120.12, 60.58, 54.52, 53.56, 21.78, 21.76. HRMS[M+H]$^{+}$ calcd for $C_{15}H_{15}BrNO_3S$: 367.9956, found 367.9962.

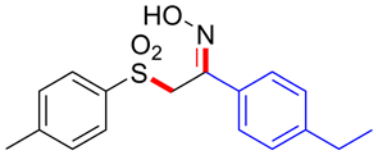


1-(4-methoxyphenyl)-2-tosylethan-1-one oxime(3ia). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 78.2% yield; ^{1}H NMR

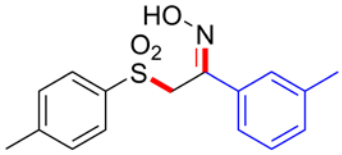
(400 MHz, DMSO-d_6) δ 11.54 (s, 1H), 7.62 – 7.56 (m, 4H), 7.35 (d, *J* = 8.0 Hz, 2H), 6.90 (d, *J* = 8.08 Hz, 2H), 4.87 (s, 2H), 3.78 (s, 3H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 159.95, 145.20, 144.31, 136.90, 129.47, 127.90, 127.82, 126.99, 113.64, 55.21, 51.41, 21.10. HRMS[M+H]$^+$ calcd for $C_{16}H_{18}NO_4S$: 320.0957, found 320.0973. This compound was known.[6]



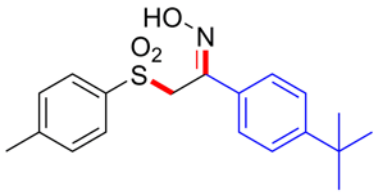
1-(p-tolyl)-2-tosylethan-1-one oxime(3ja). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 71.7% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.65 (s, 1H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.52 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 4.87 (s, 2H), 2.38 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.56, 144.35, 138.54, 136.88, 131.81, 129.47, 128.81, 127.89, 126.34, 51.43, 21.11, 20.82. HRMS[M+Na]$^+$ calcd for $C_{16}H_{17}NNaO_3S$: 326.0827, found 326.0813. This compound was known.[8]



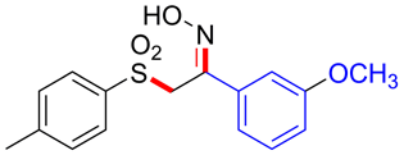
1-(4-ethylphenyl)-2-tosylethan-1-one oxime(3ka). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 74.2 % yield; mp= 162-163 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.65 (s, 1H), 7.61 – 7.59 (m, 2H), 7.54 – 7.52 (m, 2H), 7.35 – 7.33 (m, 2H), 7.17 ((d, *J* = 7.6 Hz, 2H), 4.87 (s, 2H), 2.61 (q, *J* = 7.6 Hz, 2H), 2.38 (s, 3H), 1.19 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.59, 144.76, 144.29, 136.89, 132.03, 129.45, 127.90, 127.61, 126.40, 51.44, 27.92, 21.11, 15.48. HRMS[M+Na]$^+$ calcd for $C_{17}H_{19}NO_3SNa$: 340.0983, found 340.0996.



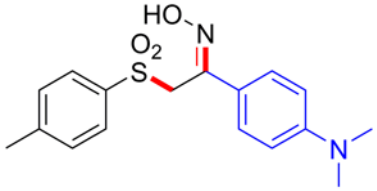
1-(m-tolyl)-2-tosylethan-1-one oxime(3la). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow oil: 74.9% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.49 (s, 1H), 7.72 – 7.69 (m, 2H), 7.41 – 7.38 (m, 1H), 7.35 – 7.34 (m, 1H), 7.26 – 7.19 (m, 4H), 4.72 (s, 2H), 2.37 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 148.16, 144.96, 138.32, 136.71, 133.67, 130.75, 129.54, 128.62, 128.56, 127.30, 123.95, 52.96, 21.71, 21.51. HRMS[M+Na]$^+$ calcd for $C_{16}H_{17}NNaO_3S$: 326.0827, found 326.0813.



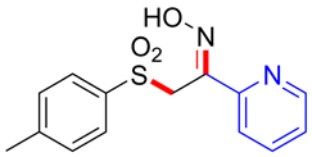
1-(4-(tert-butyl)phenyl)-2-tosylethan-1-one oxime(3ma). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 78.4% yield; mp= 175-176 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.69 (s, 1H), 7.59 (d, *J* = 7.6 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.34-7.32 (m, 4H), 4.89 (s, 2H), 2.36 (s, 3H), 1.28 (s, 9H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 151.48, 145.51, 144.21, 136.91, 131.73, 129.43, 127.89, 126.11, 124.95, 51.41, 34.36, 31.00, 21.11. HRMS[M+Na]$^+$ calcd for $C_{19}H_{23}NNaO_3S$: 368.1296, found 368.1297.



1-(3-methoxyphenyl)-2-tosylethan-1-one oxime(3na). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow oil: 80.1% yield; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.80 (s, 1H), 7.63 – 7.60 (m, 2H), 7.36 – 7.33 (m, 2H), 7.26 – 7.20 (m, 2H), 7.15-7.14 (m, 1H), 6.95-6.92 (m, 1H), 4.90 (s, 2H), 3.74 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 159.05, 145.60, 144.34, 136.88, 135.91, 129.44, 129.26, 127.87, 118.85, 114.58, 111.87, 55.05, 51.59, 21.07. HRMS[M+H]$^+$ calcd for $C_{16}H_{18}NO_4S$: 320.0957, found 320.0973. This compound was known.[7]

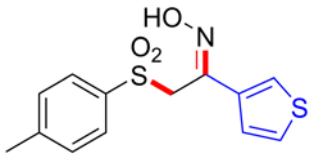


1-(4-(dimethylamino)phenyl)-2-tosylethan-1-one oxime(3oa). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow solid: 46.3% yield; mp= 194-196 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.24 (s, 1H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 9.2 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 6.65 (d, *J* = 8.8 Hz, 2H), 4.81 (s, 1H), 2.93 (s, 6H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 150.69, 145.38, 144.12, 137.06, 129.37, 127.85, 127.23, 121.98, 111.49, 51.39, 21.07. HRMS[M+H]$^+$ calcd for $C_{17}H_{21}N_2O_3S$: 333.1273, found 333.1283.

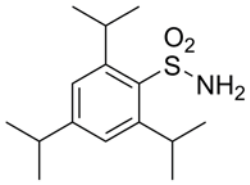


1-(pyridin-2-yl)-2-tosylethan-1-one oxime(3sa). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 45.9% yield; mp= 183-184 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.12 (s, 1H), 8.41-8.39 (m, 1H), 7.81 – 7.74 (m, 2H), 7.63 – 7.60 (m, 2H), 7.35 – 7.31

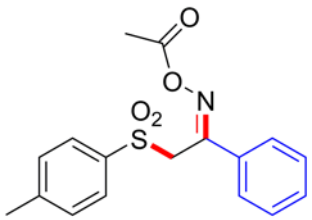
(m, 3H), 4.97 (s, 2H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 152.12, 148.40, 146.76, 144.10, 137.56, 136.74, 129.38, 127.75, 123.72, 120.07, 50.59, 21.04. HRMS[M+H]$^+$ calcd for $C_{14}H_{15}N_2O_3S$: 291.0803, found 291.0820.



1-(thiophen-3-yl)-2-tosylethan-1-one oxime(3ta). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow solid: 72.4% yield; mp= 208-210 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.52 (s, 1H), 7.74 (dd, *J* = 2.9, 1.3 Hz, 1H), 7.67 – 7.64 (m, 2H), 7.51 (dd, *J* = 5.2, 2.9 Hz, 1H), 7.38 – 7.36 (m, 2H), 7.33 (dd, *J* = 5.2, 1.3 Hz, 1H), 4.82 (s, 2H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 144.34, 142.21, 136.91, 136.87, 129.47, 127.91, 126.44, 125.19, 125.05, 52.39, 21.10. HRMS[M+H]$^+$ calcd for $C_{13}H_{14}NO_3S_2$: 296.0415, found 296.0427.



2,4,6-triisopropylbenzenesulfonamide (4d). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a light yellow solid: 48.1% yield; ^{1}H NMR (400 MHz, DMSO-d_6) 6.96 (s, 2H), 4.61-4.51(m, 2H), 3.46-3.39 (s, 2H). 2.85-2.75 (s, 1H). 1.17-1.16 (d, *J* =17 Hz, 6H). 1.12-1.10 (d, *J* =17 Hz, 12H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 147.30, 146.80, 141.74, 121.40, 33.31, 28.07, 24.85, 23.87; HRMS[M+H]$^+$ calcd for $C_{23}H_{32}NO_3S$: 402.2103, found 402.2096. This compound was known.[9]



(Z)-1-phenyl-2-tosylethan-1-one O-acetyl oxime (4i). This title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid: 96% yield; mp= 153-154 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.79-7.77 (m, 2H), 7.69-7.67 (m, 2H), 7.54 – 7.50 (m, 1H), 7.46 – 7.39 (m, 4H), 5.12 (s, 2H), 2.38 (s, 3H), 1.96 (s, 3H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 166.92, 154.56, 145.00, 136.12, 132.51, 131.05, 129.74, 128.57, 128.08, 127.80, 53.52, 21.07, 19.08. HRMS[M+Na]$^+$ calcd for $C_{17}H_{17}NNaO_4S$: 354.0776, found 354.0776.

9. Reference

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[5] B. Wang, L. Tang, L. Liu, Y. Li, Y. Yang and Z. Wang, *Green Chem.*,2017, **19**, 5794;

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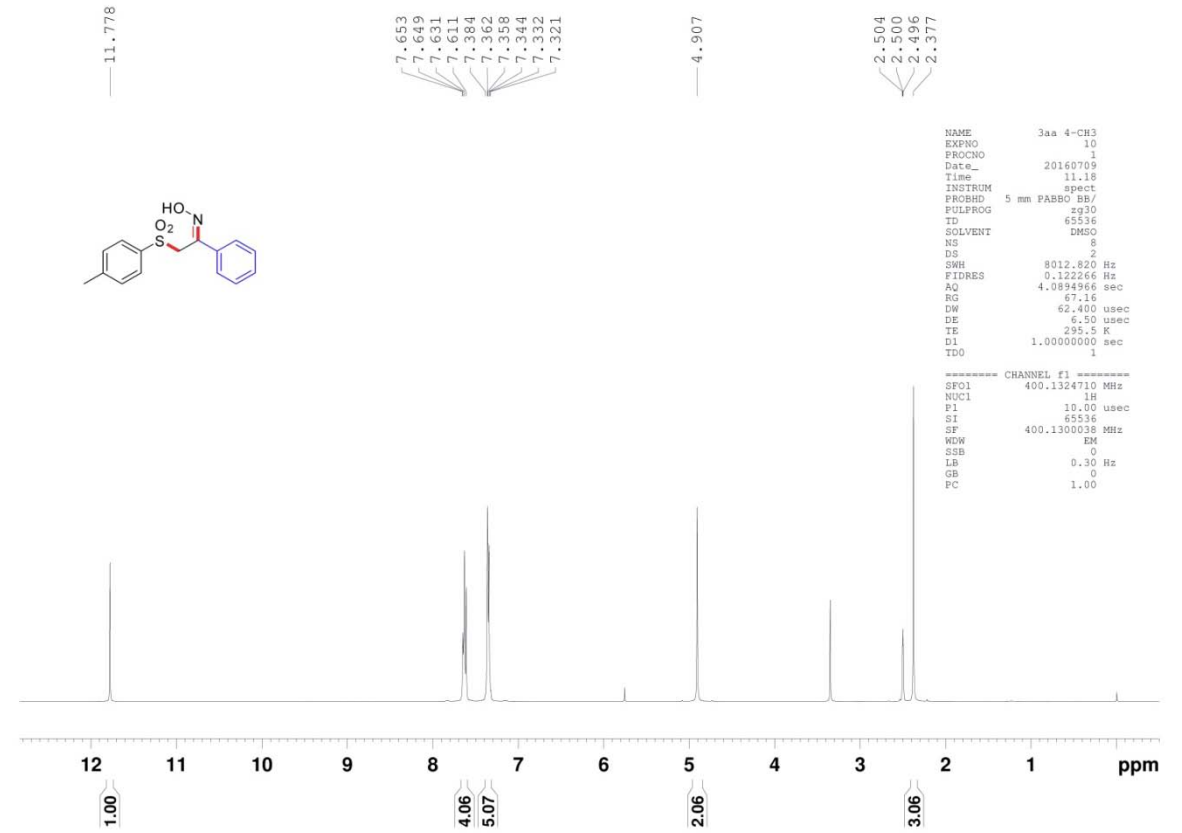
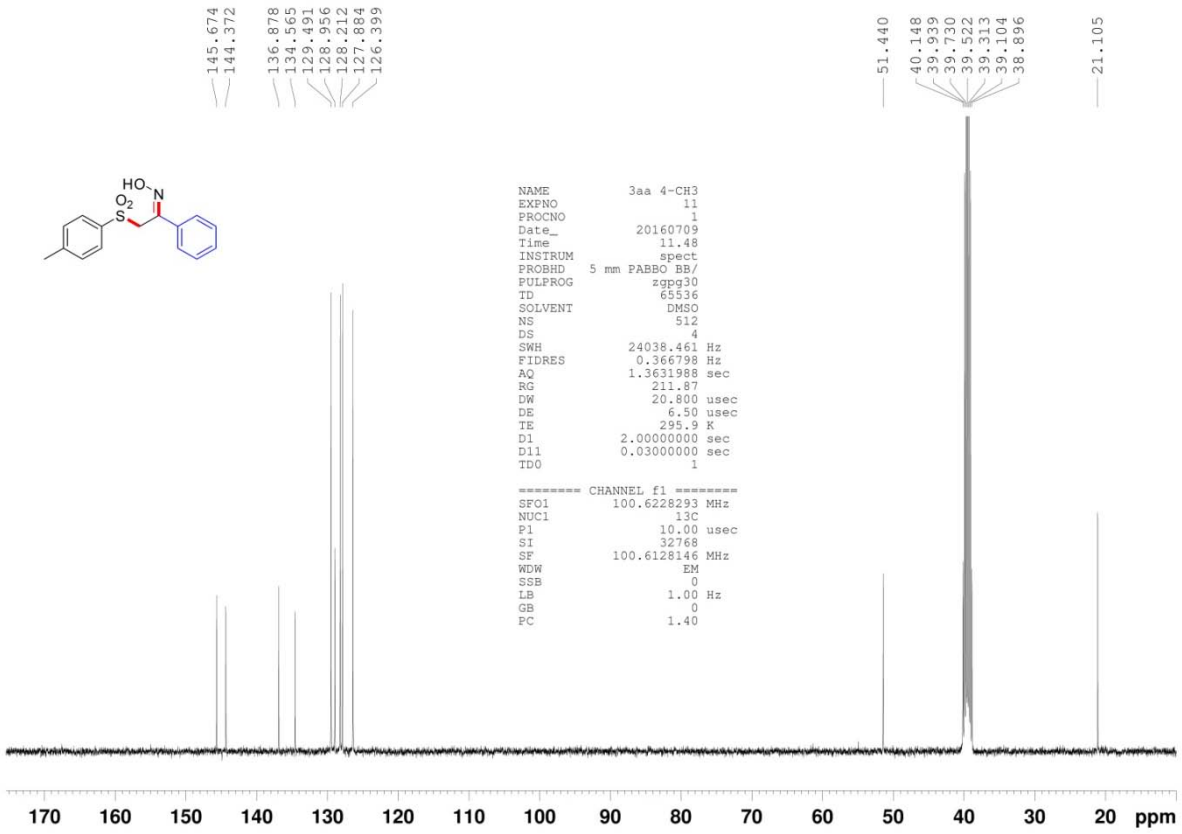
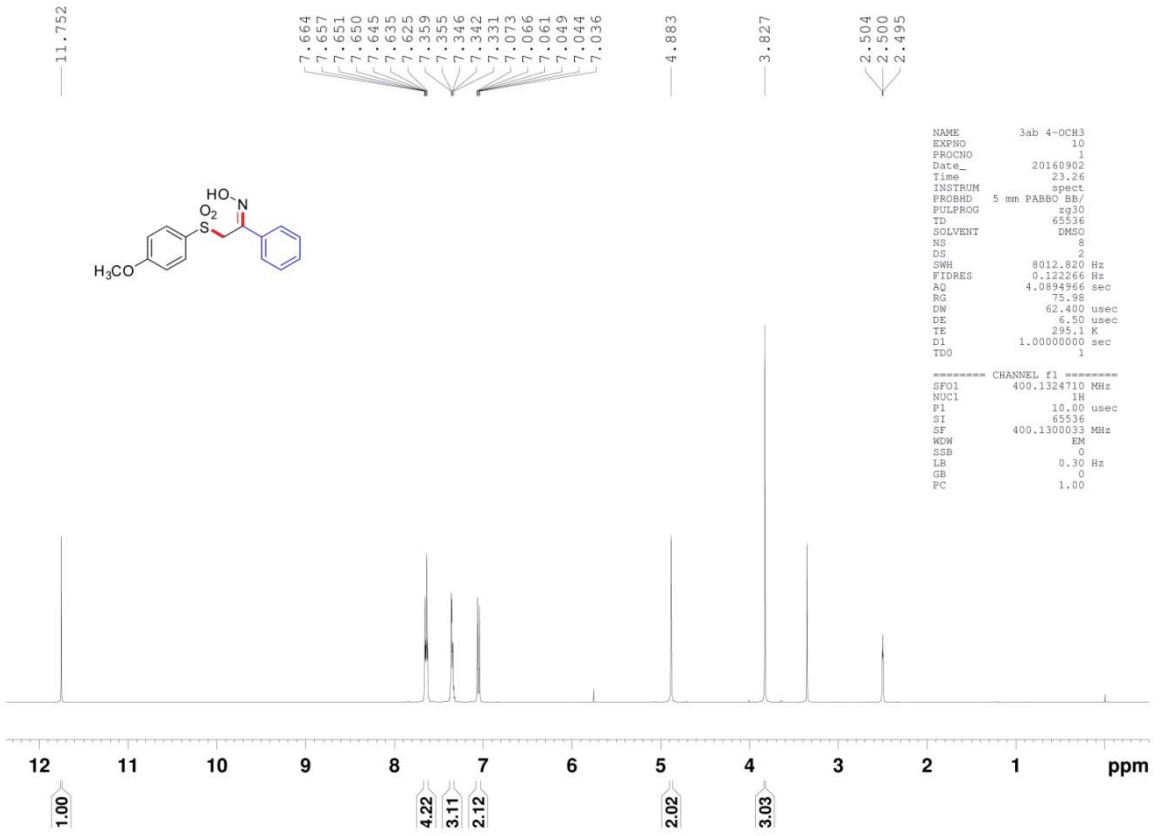
10. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra of products

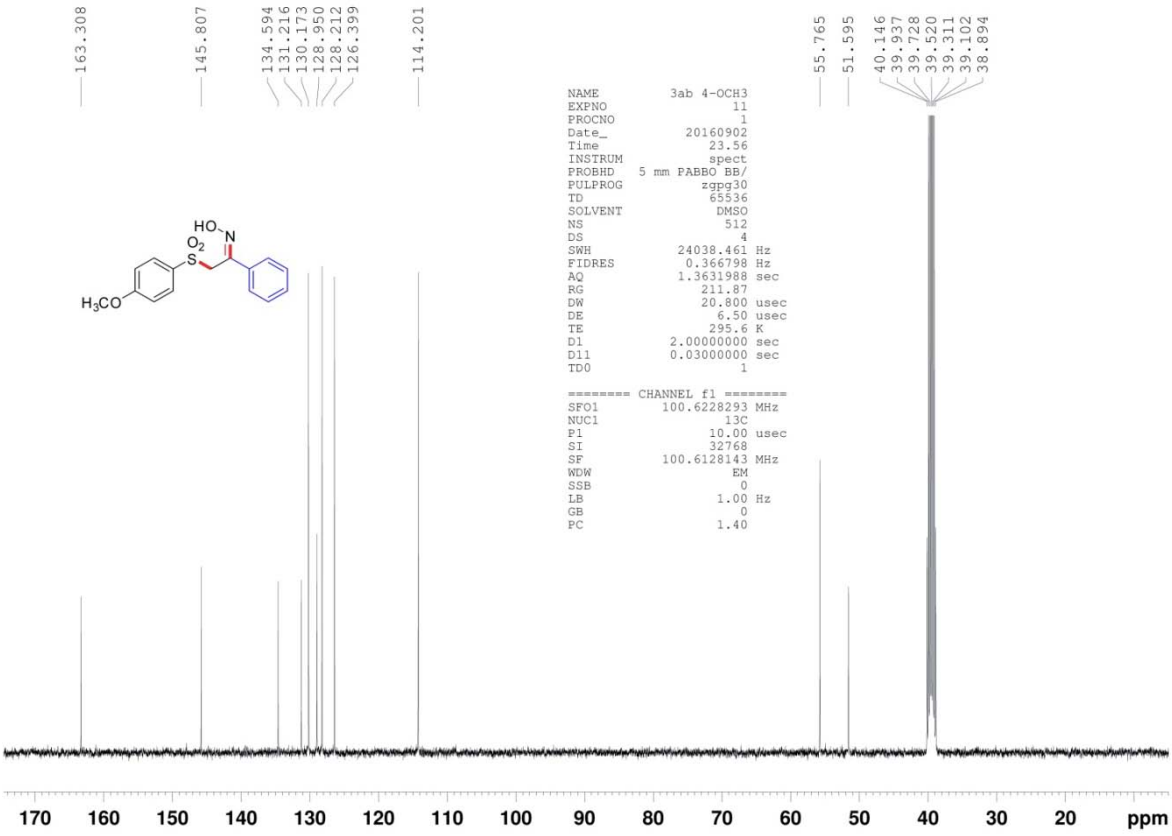
1-phenyl-2-tosylethan-1-one oxime (3aa).



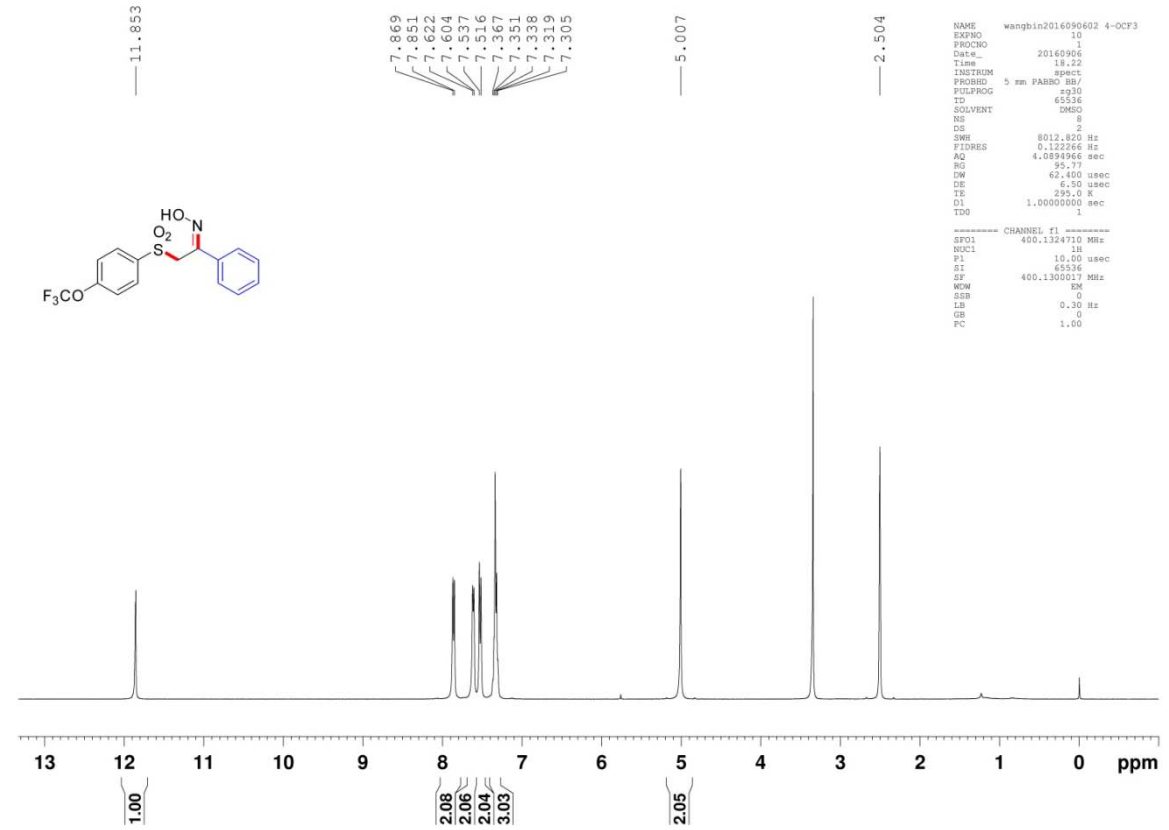


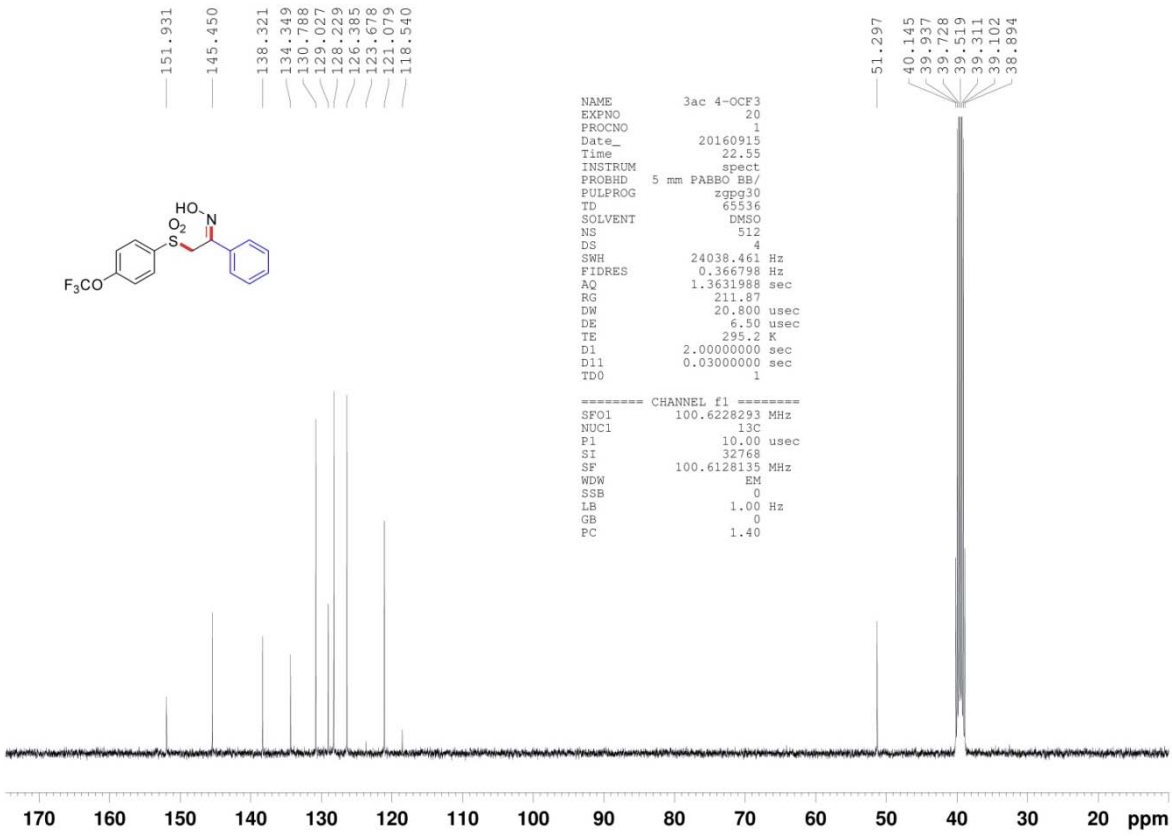
2-((4-methoxyphenyl)sulfonyl)-1-phenylethan-1-one oxime (3ab).

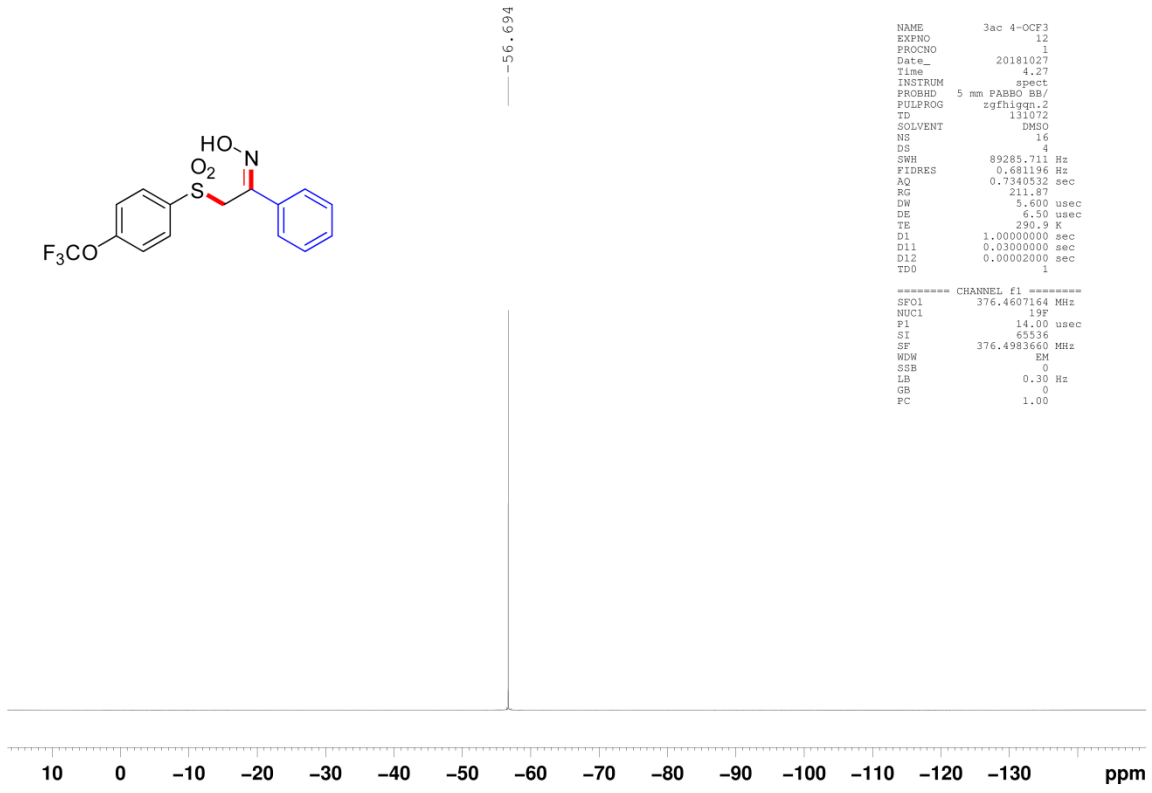




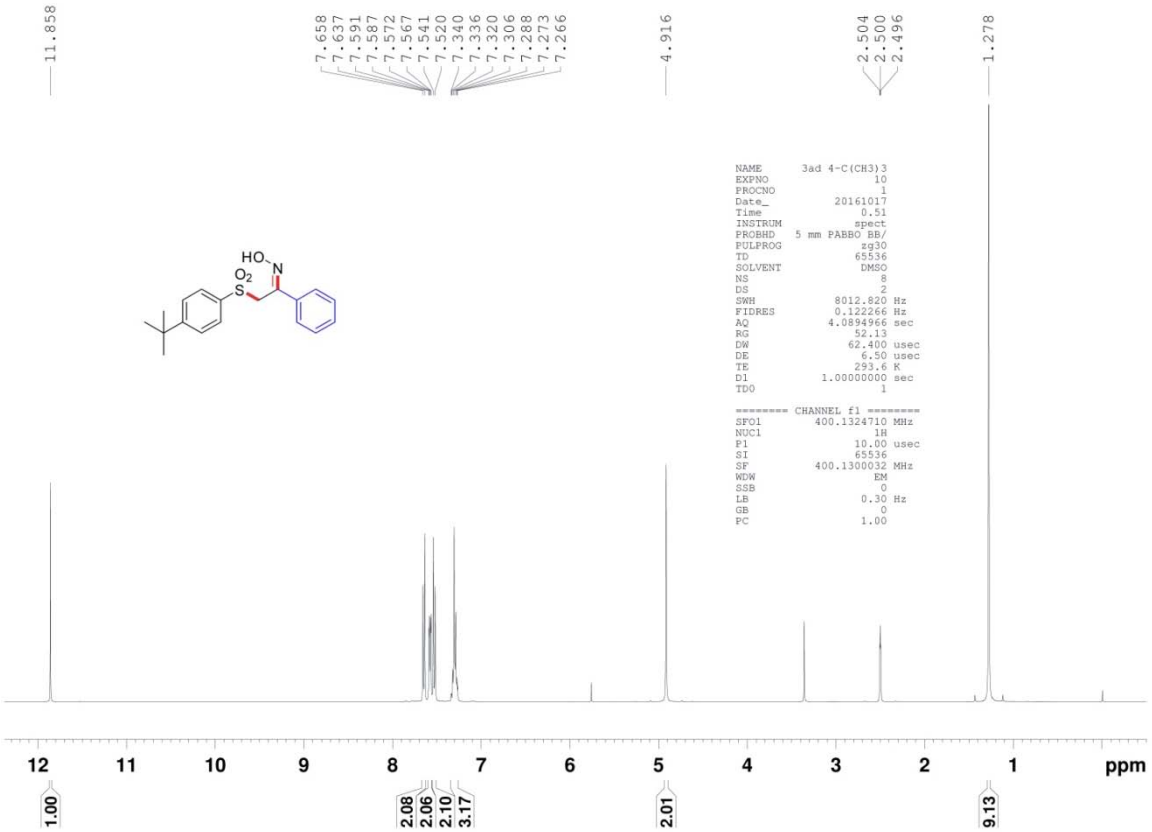
1-phenyl-2-((4-(trifluoromethoxy)phenyl)sulfonyl)ethan-1-one oxime (3ac).

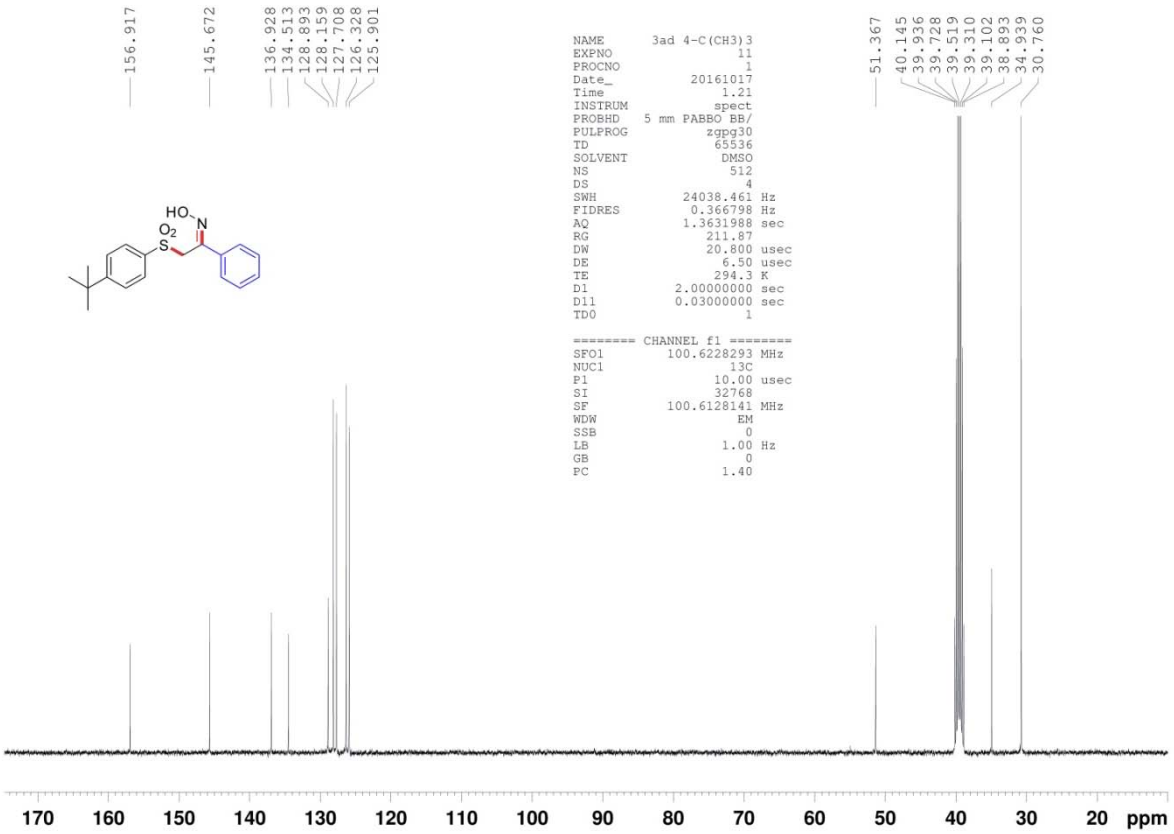




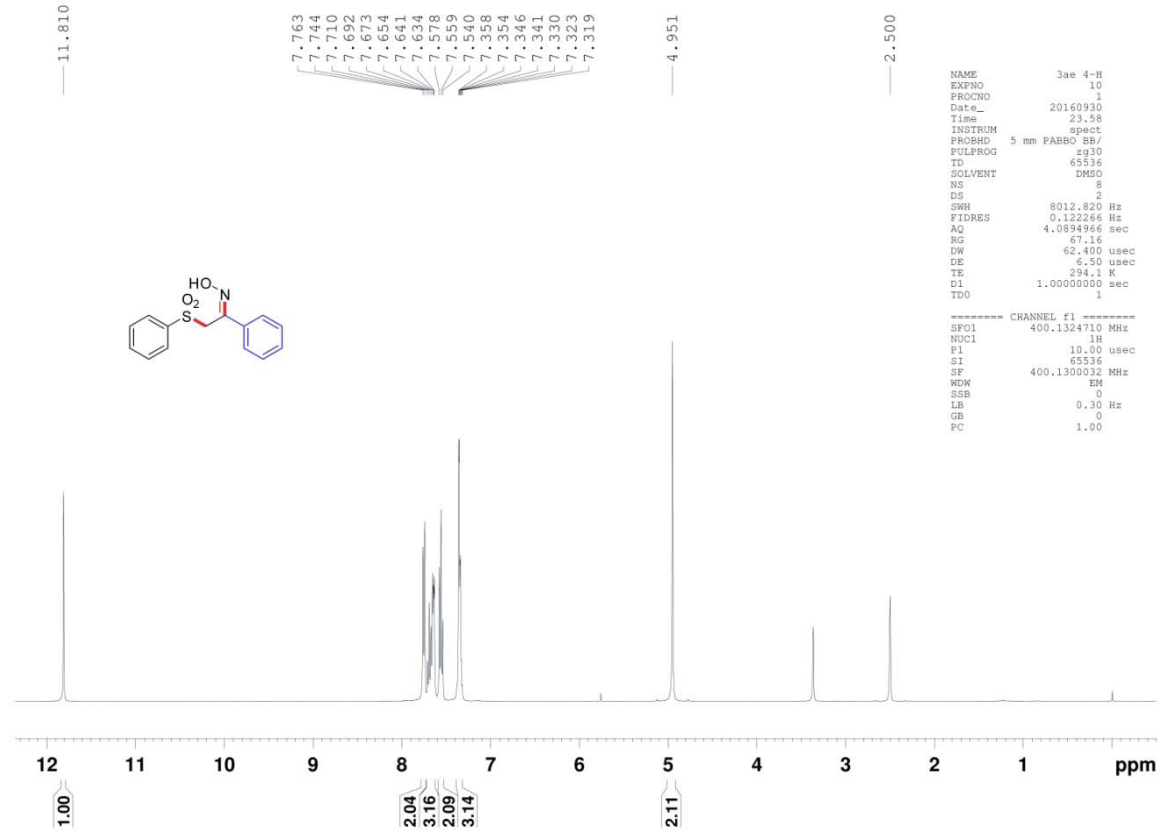


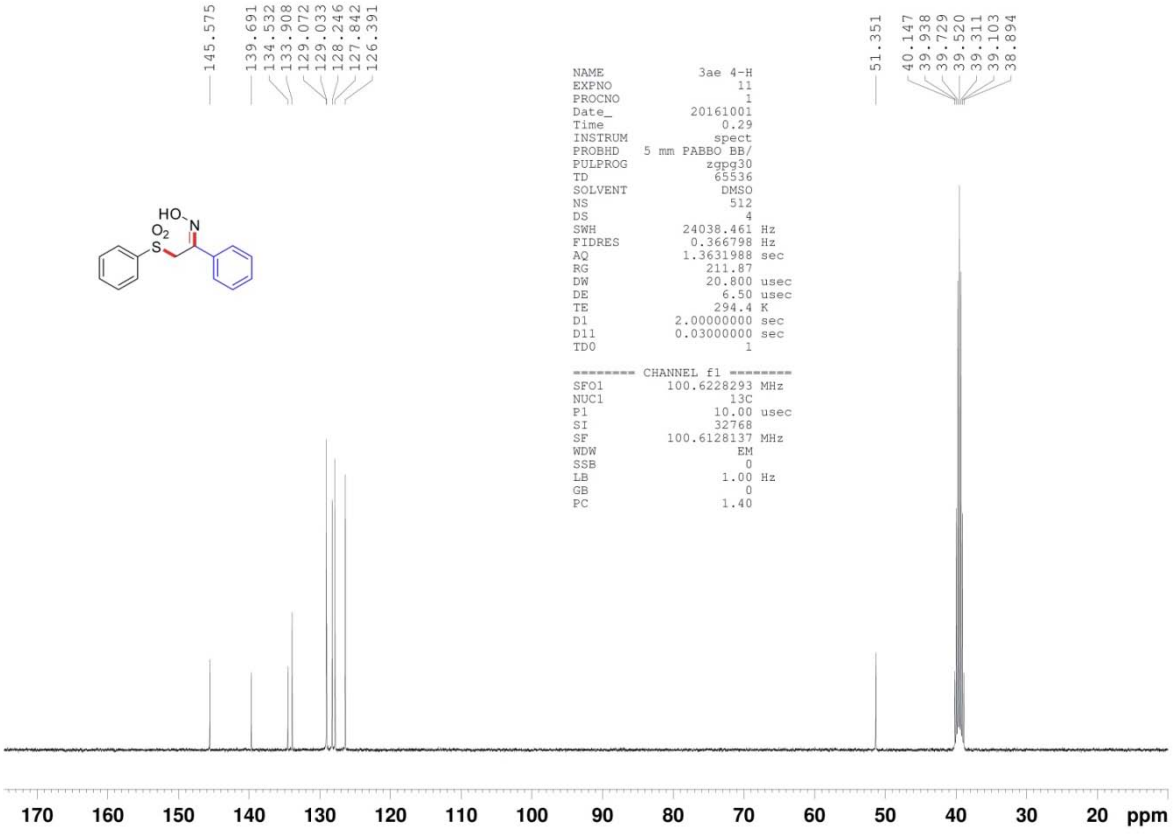
2-((4-(tert-butyl)phenyl)sulfonyl)-1-phenylethan-1-one oxime(3ad).



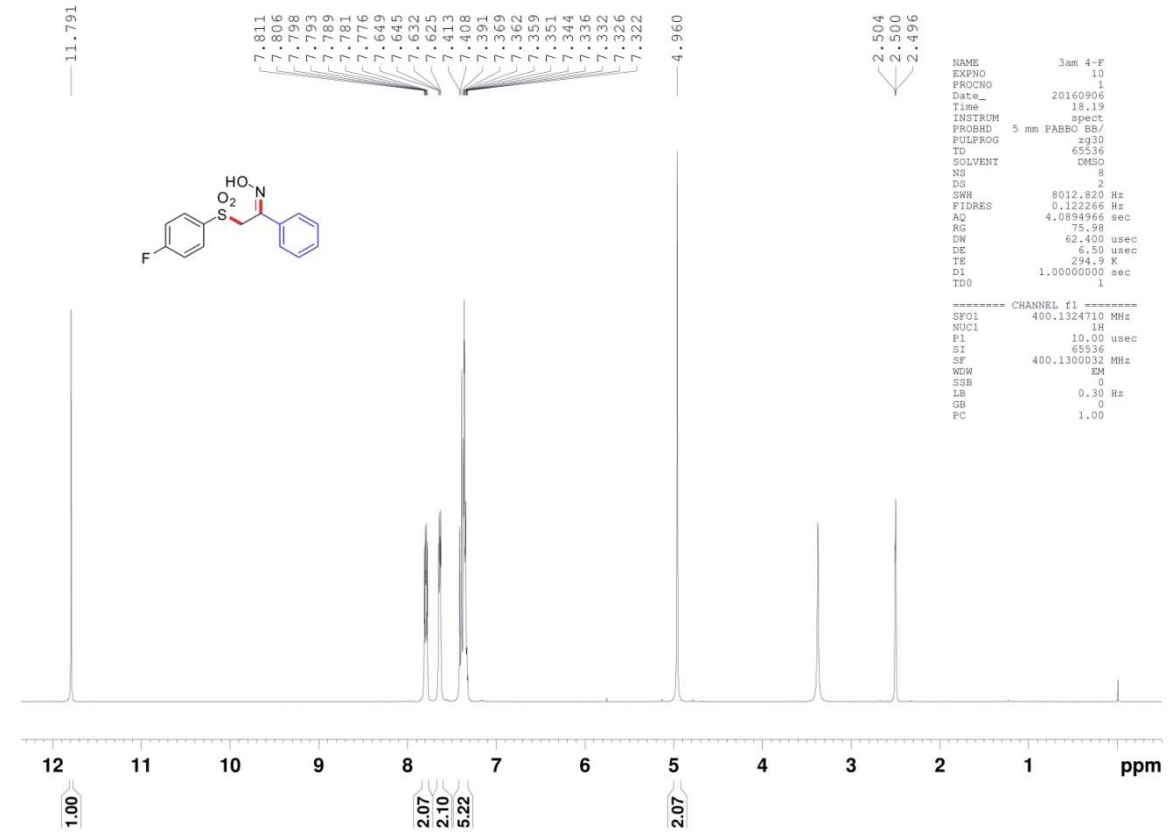


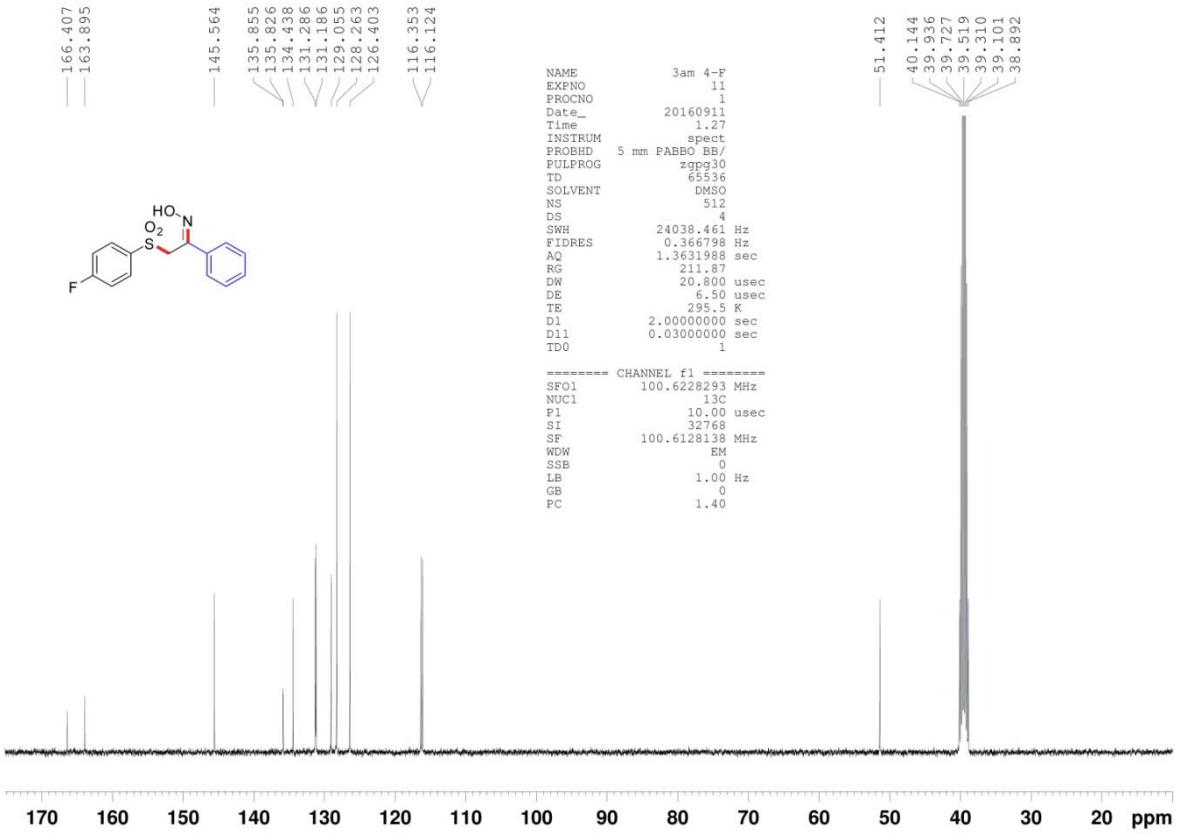
1-phenyl-2-(phenylsulfonyl)ethan-1-one oxime(3ae).





2-((4-fluorophenyl)sulfonyl)-1-phenylethan-1-one oxime(3af).

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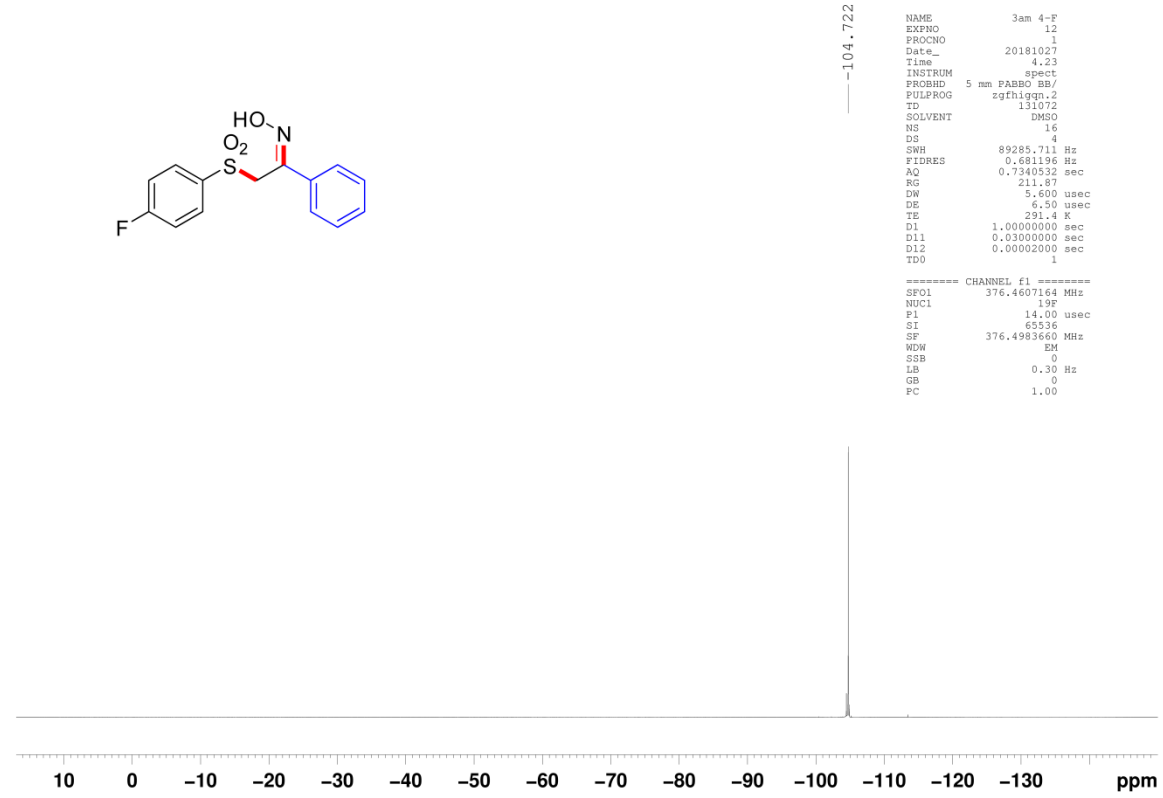
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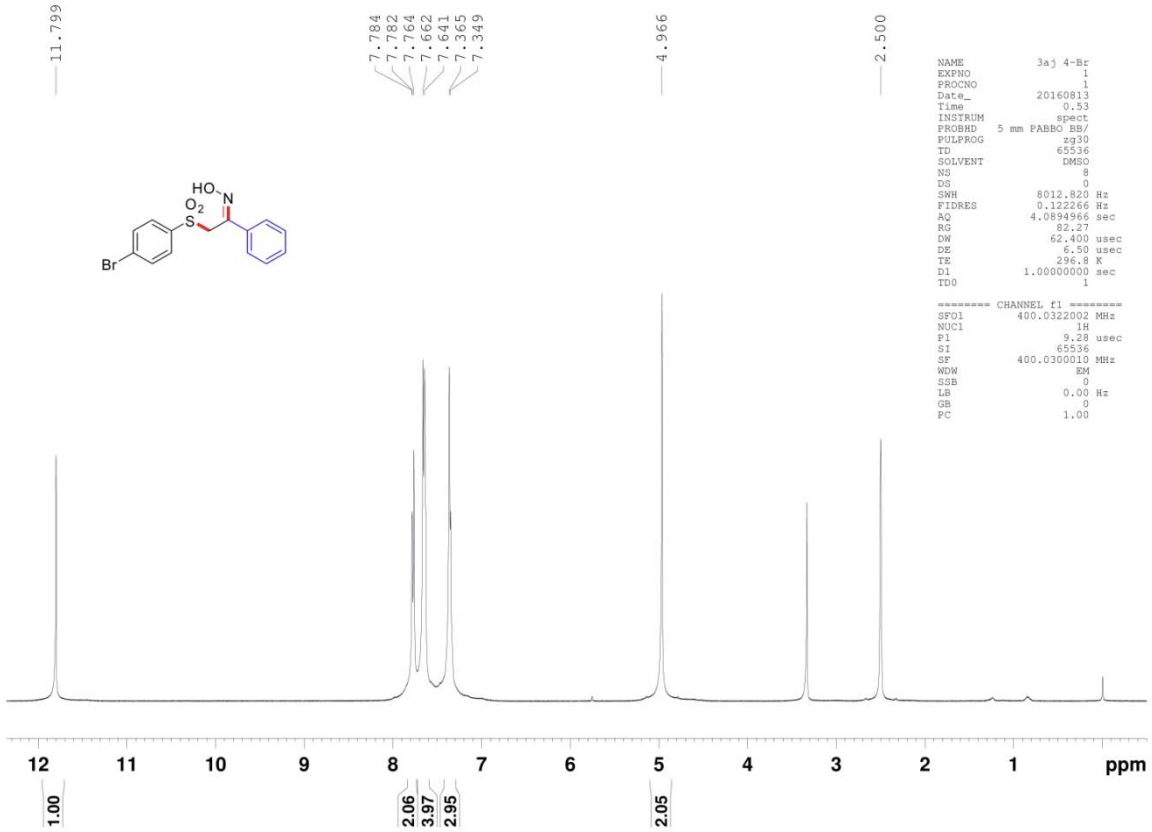
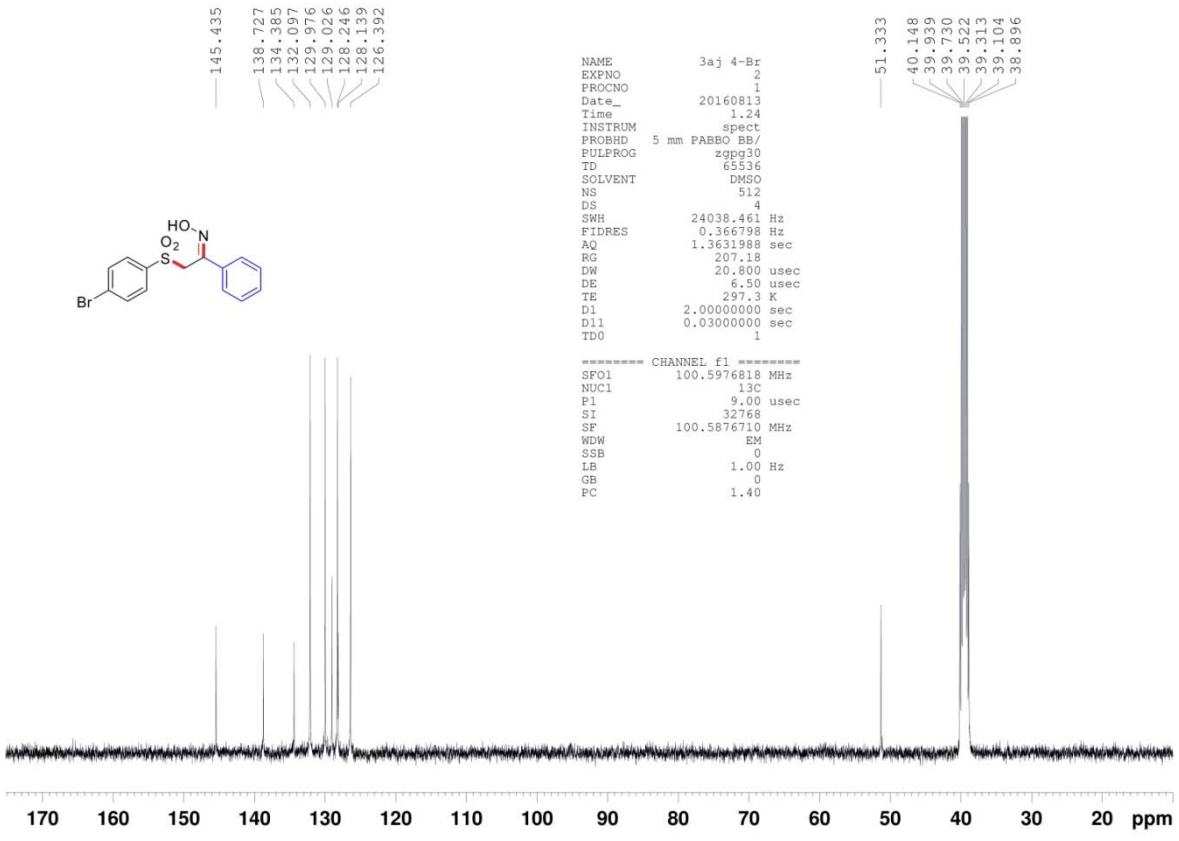
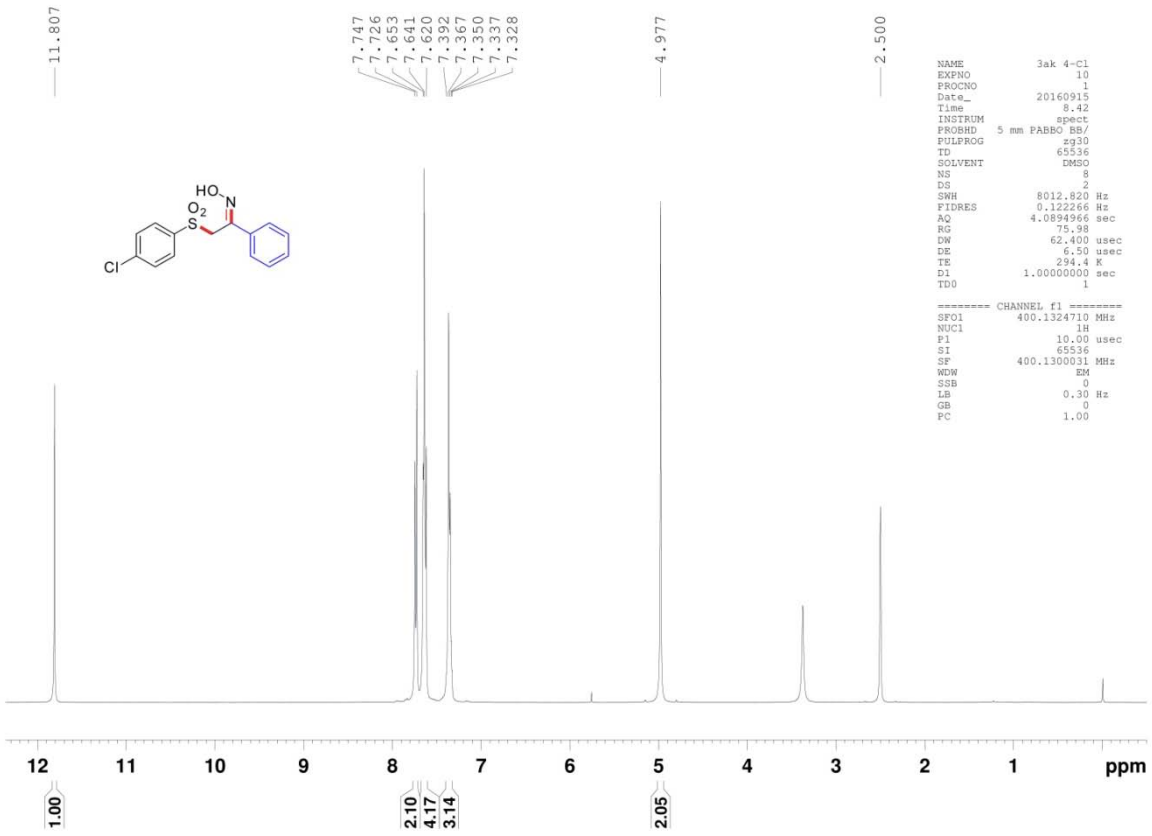
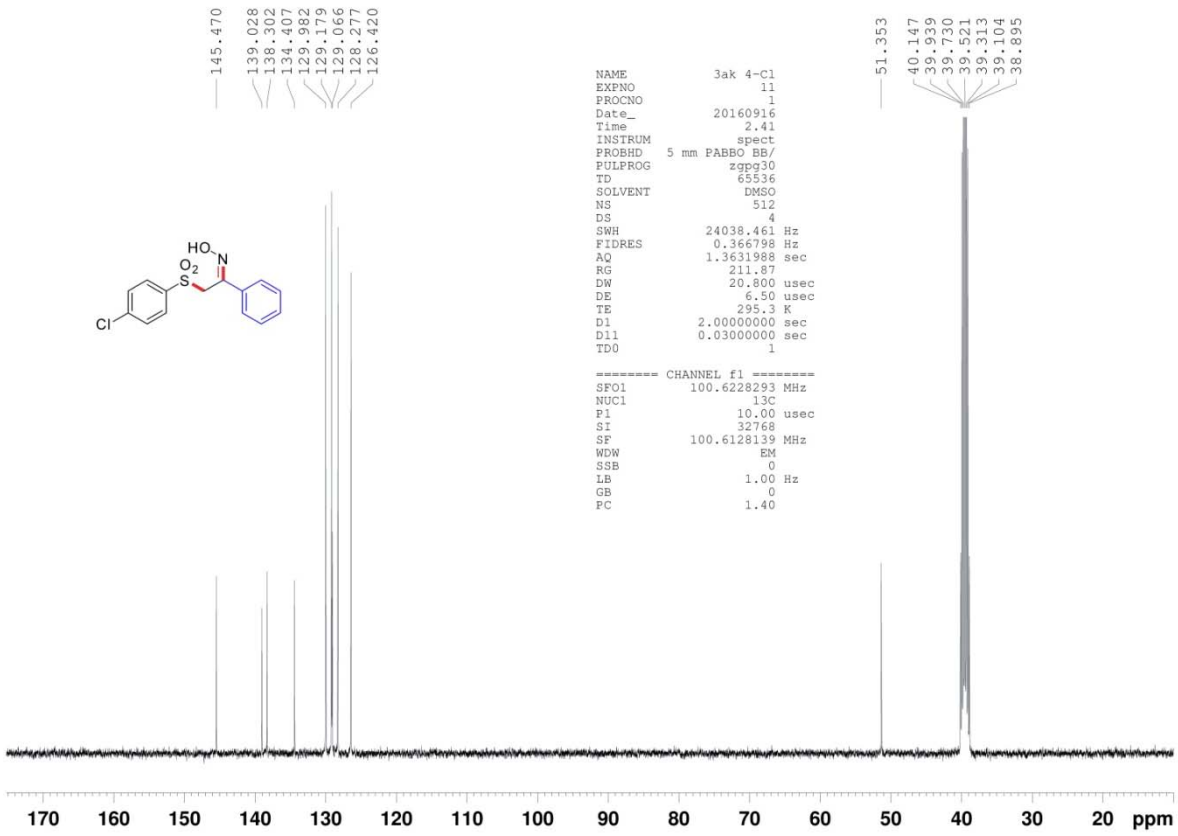
2-((4-bromophenyl)sulfonyl)-1-phenylethan-1-one oxime(3ag).



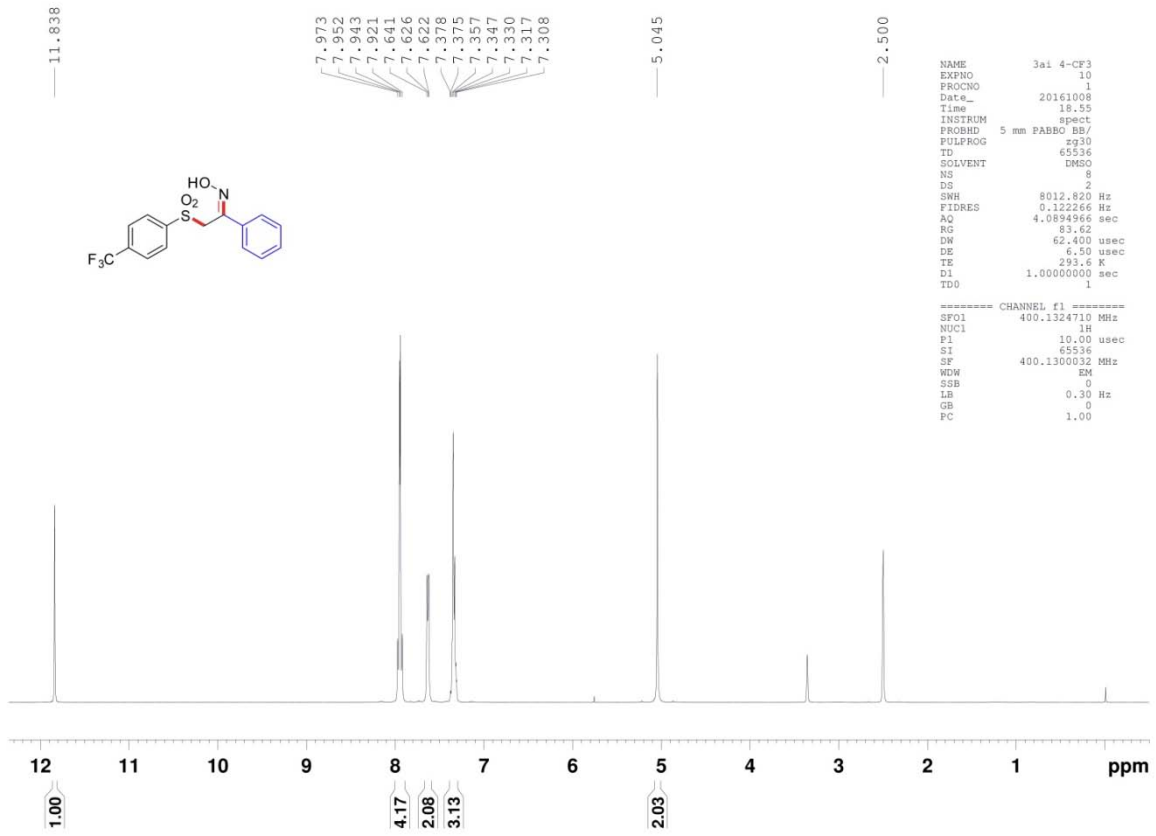


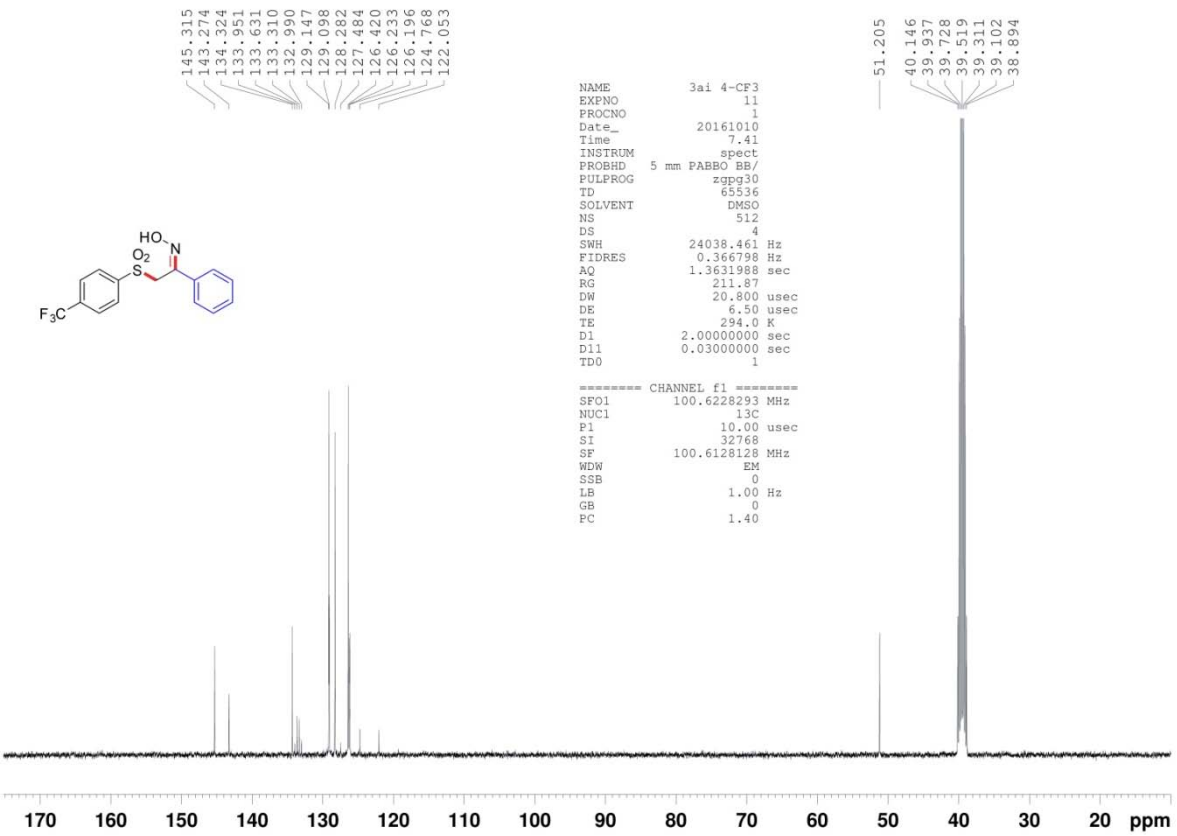
2-((4-chlorophenyl)sulfonyl)-1-phenylethan-1-one oxime(3ah).

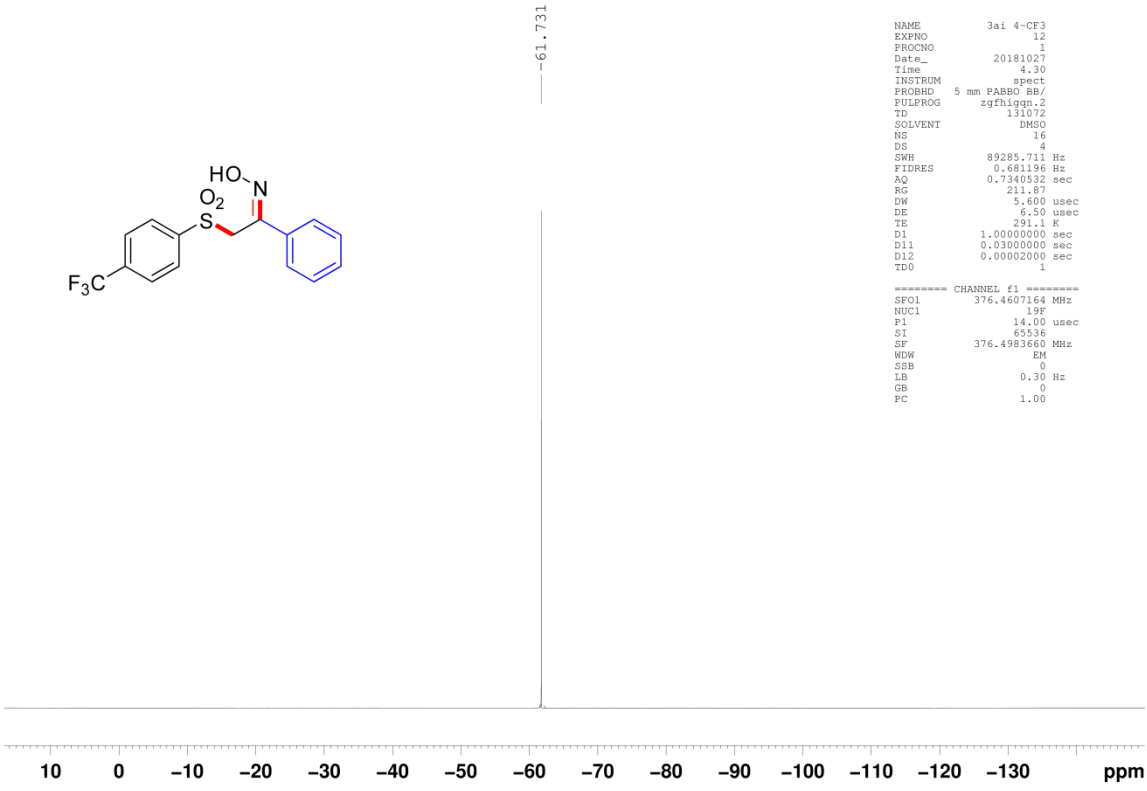




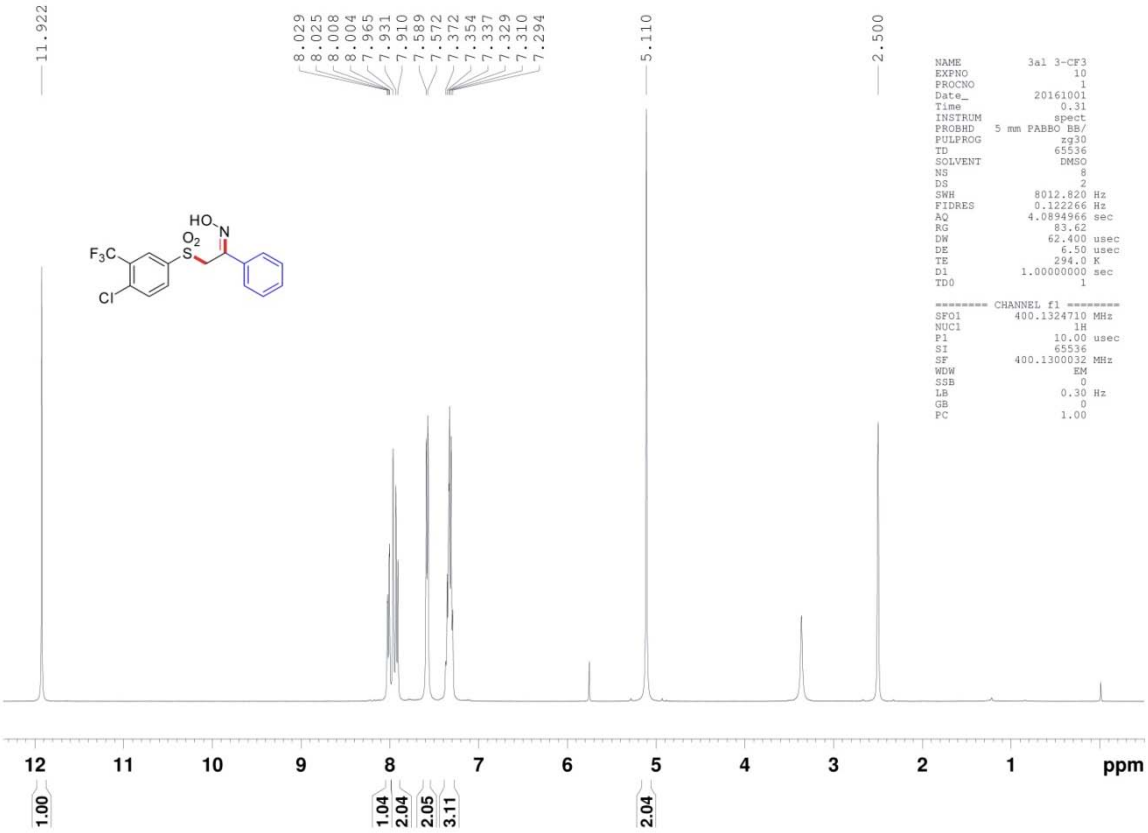
1-phenyl-2-((4-(trifluoromethyl)phenyl)sulfonyl)ethan-1-one oxime(3ai).

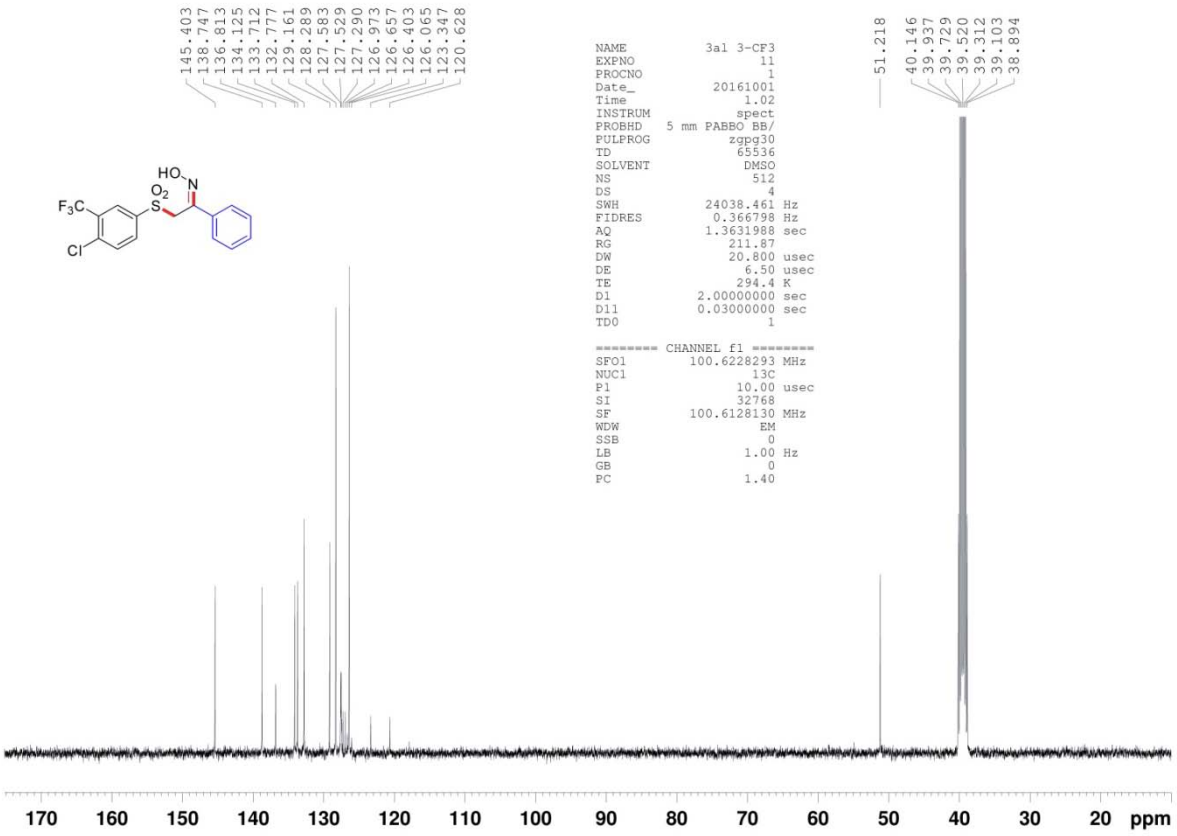






2-((4-chloro-3-(trifluoromethyl)phenyl)sulfonyl)-1-phenylethan-1-one oxime(3aj).

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DW 20.800 usec
DE 6.50 usec
TE 294.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
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NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6128130 MHz
WDW EM
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N
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S
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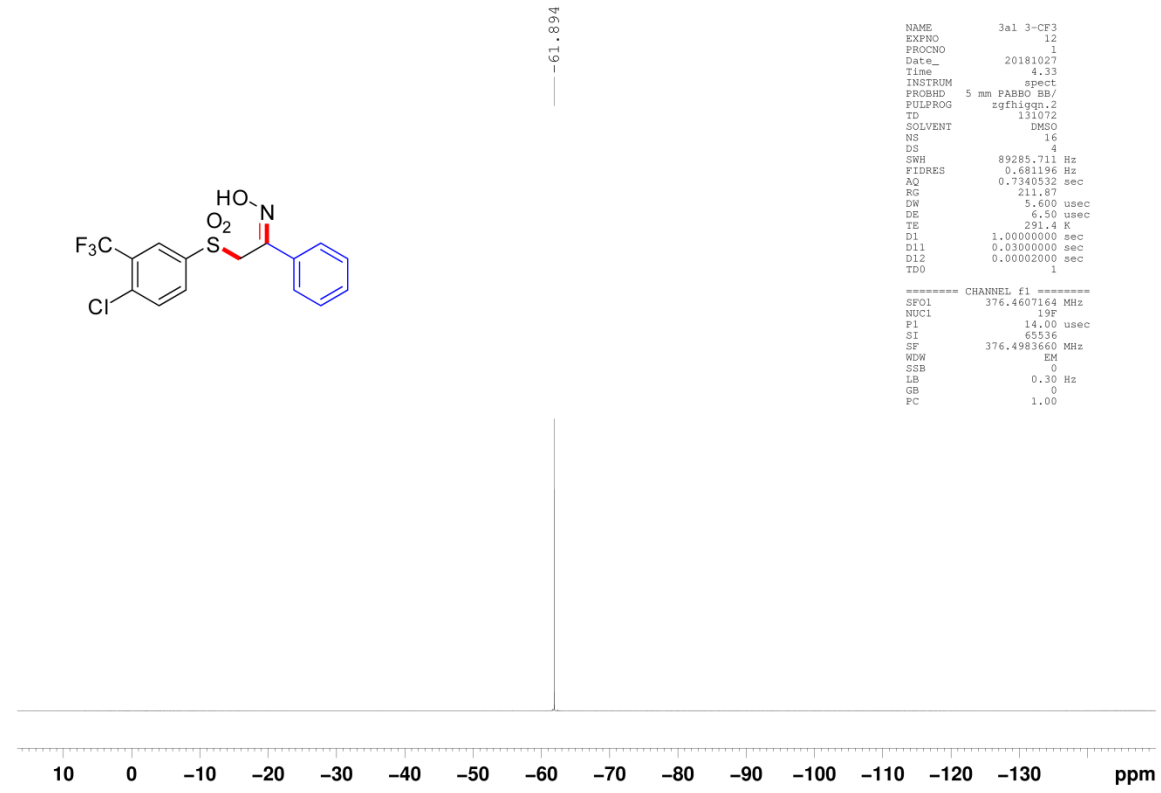
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LB 0.30 Hz
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PC 1.00
HO
N
O2
S
F3C
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10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 ppm

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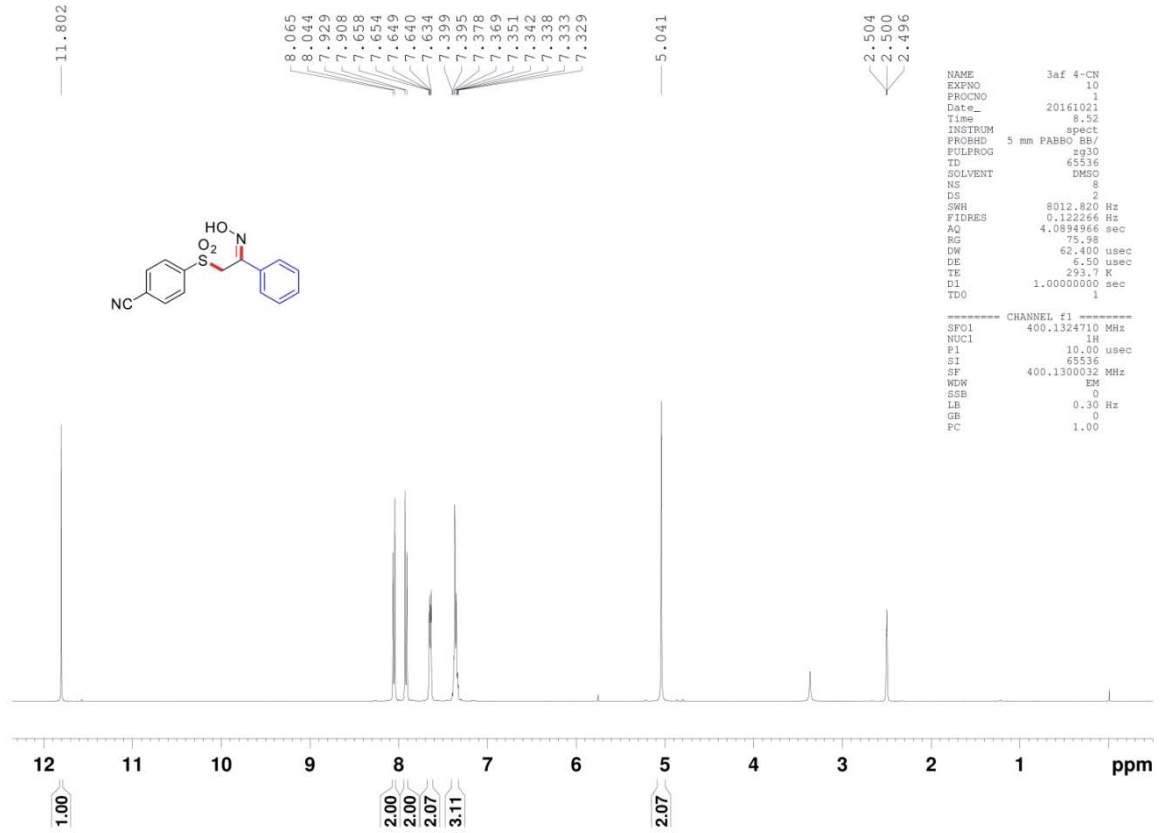
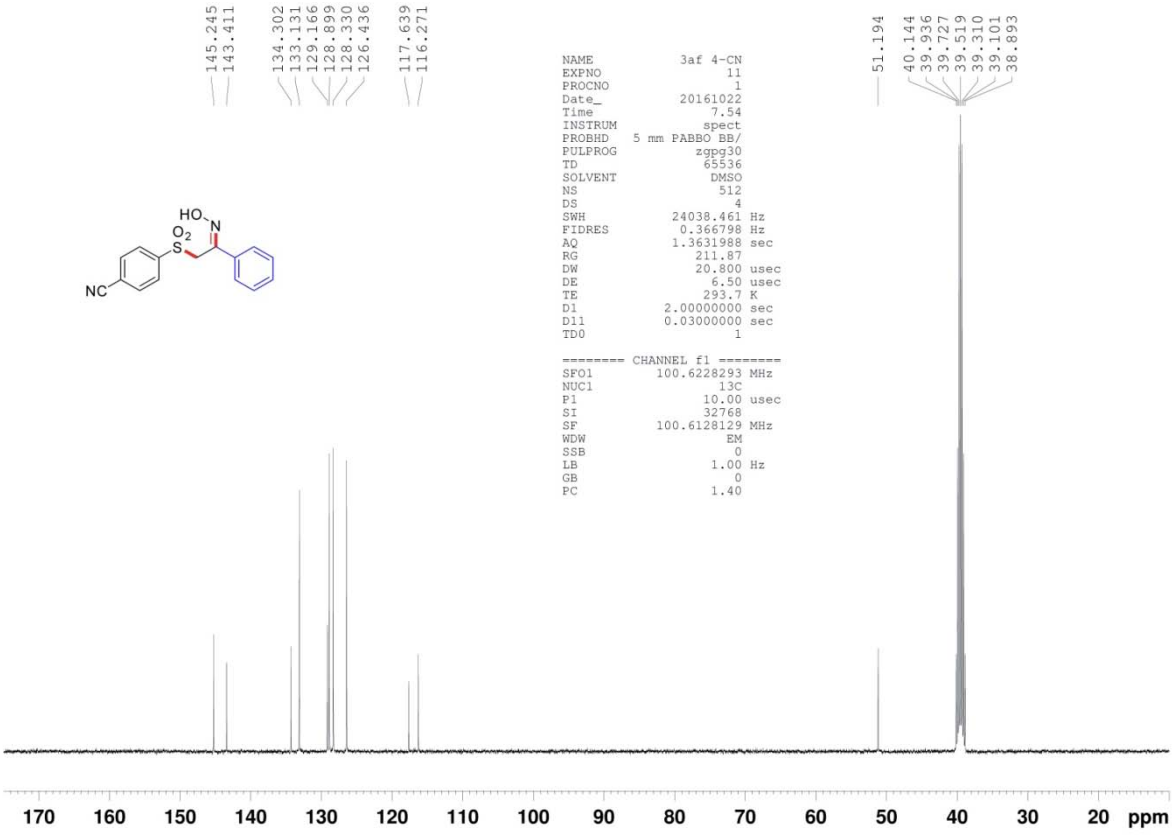
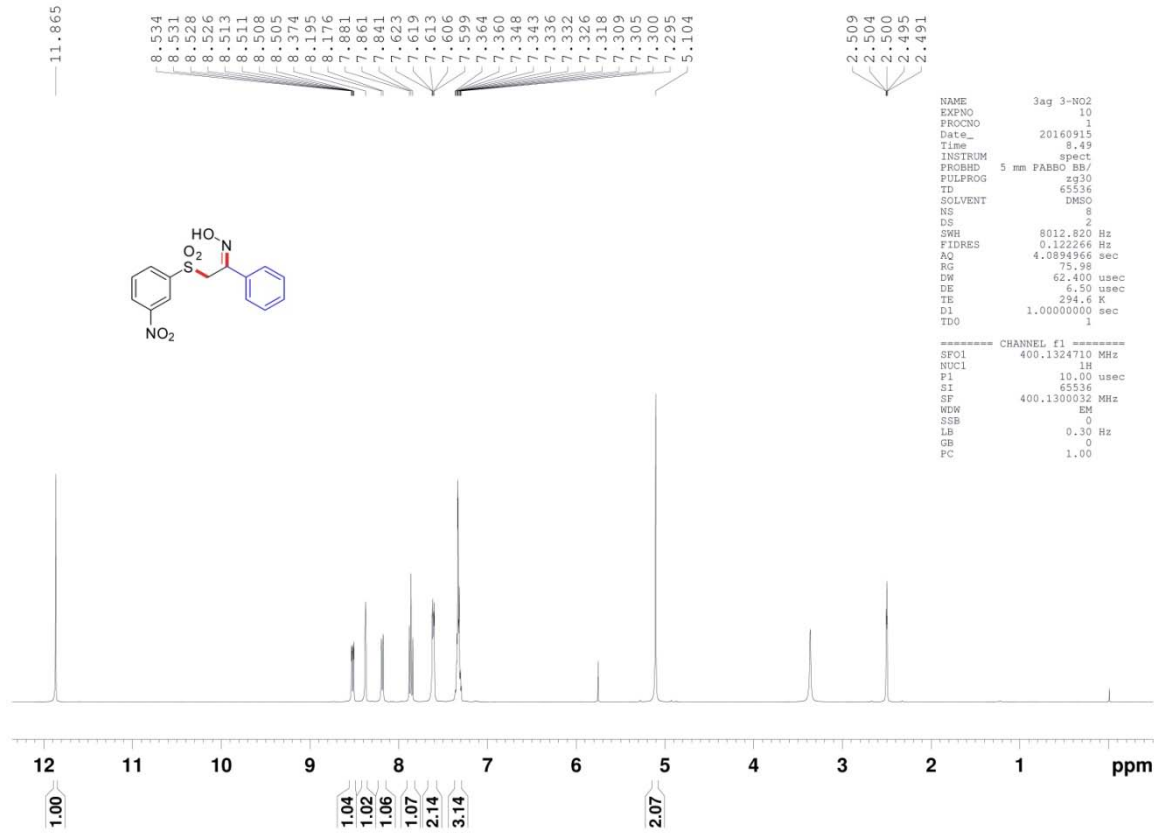
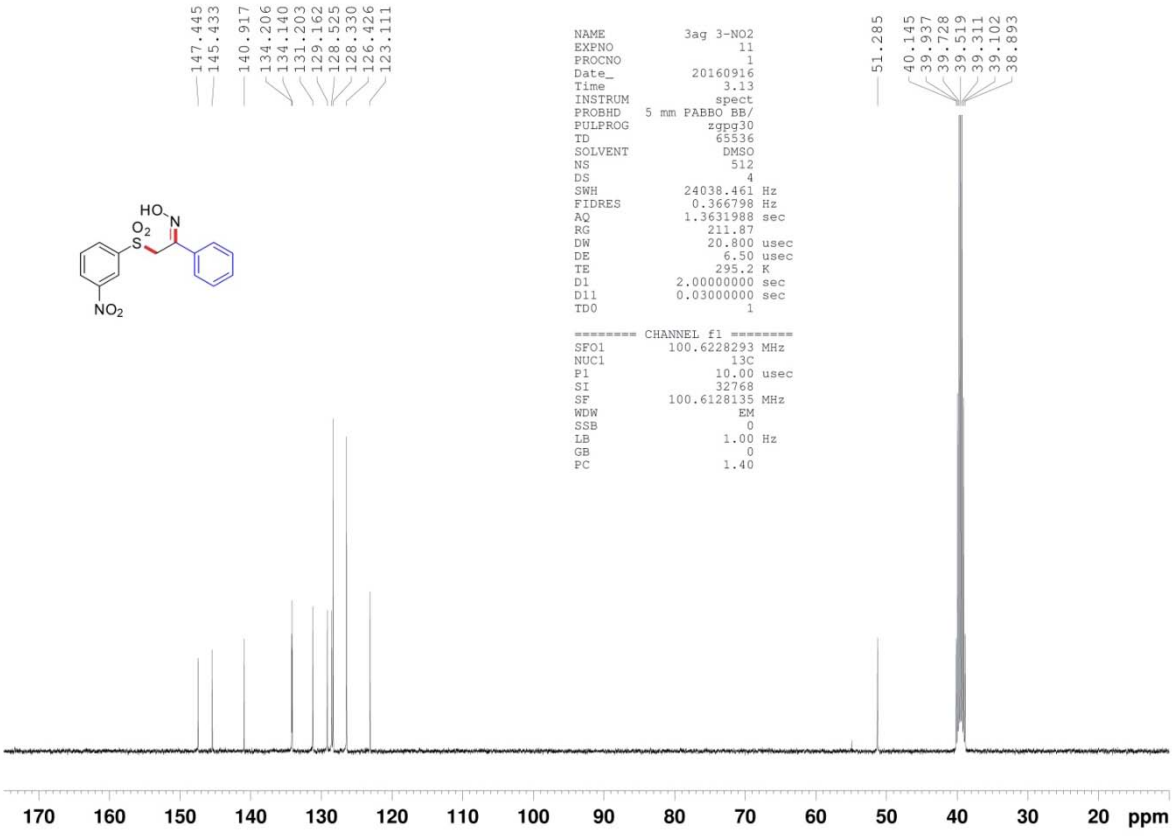


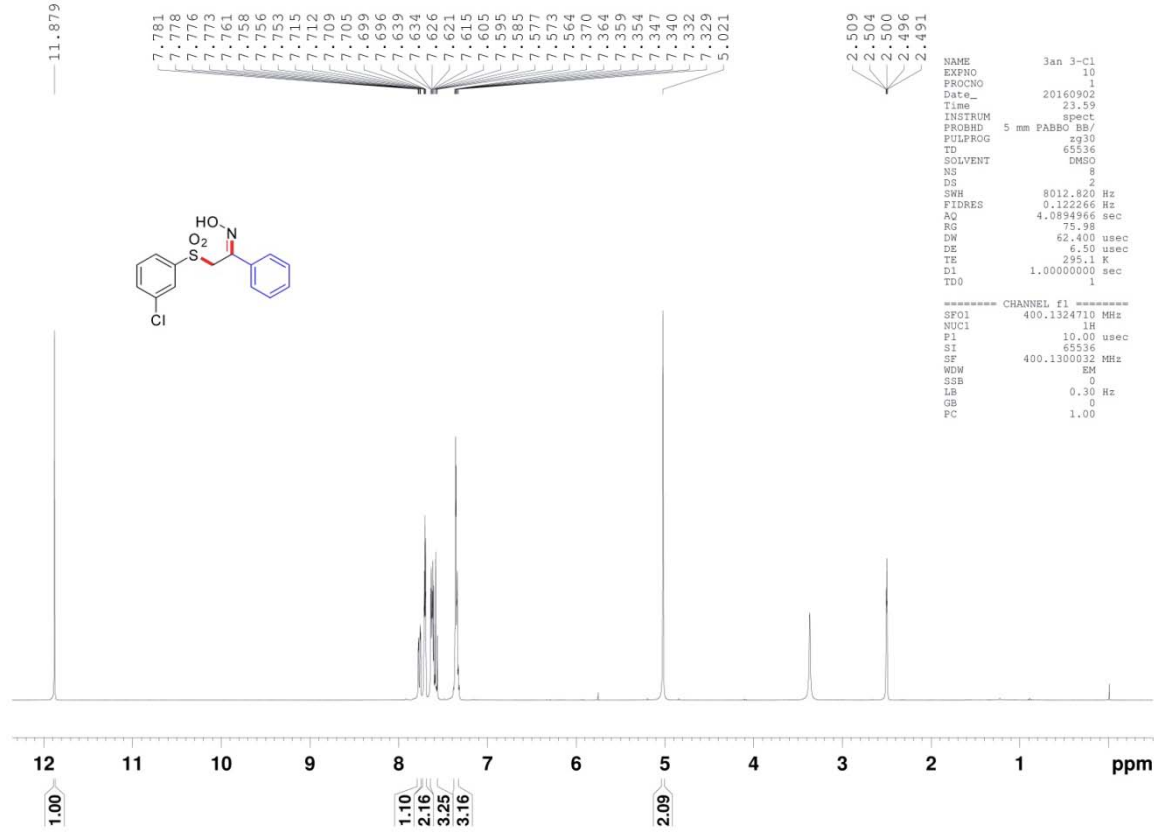


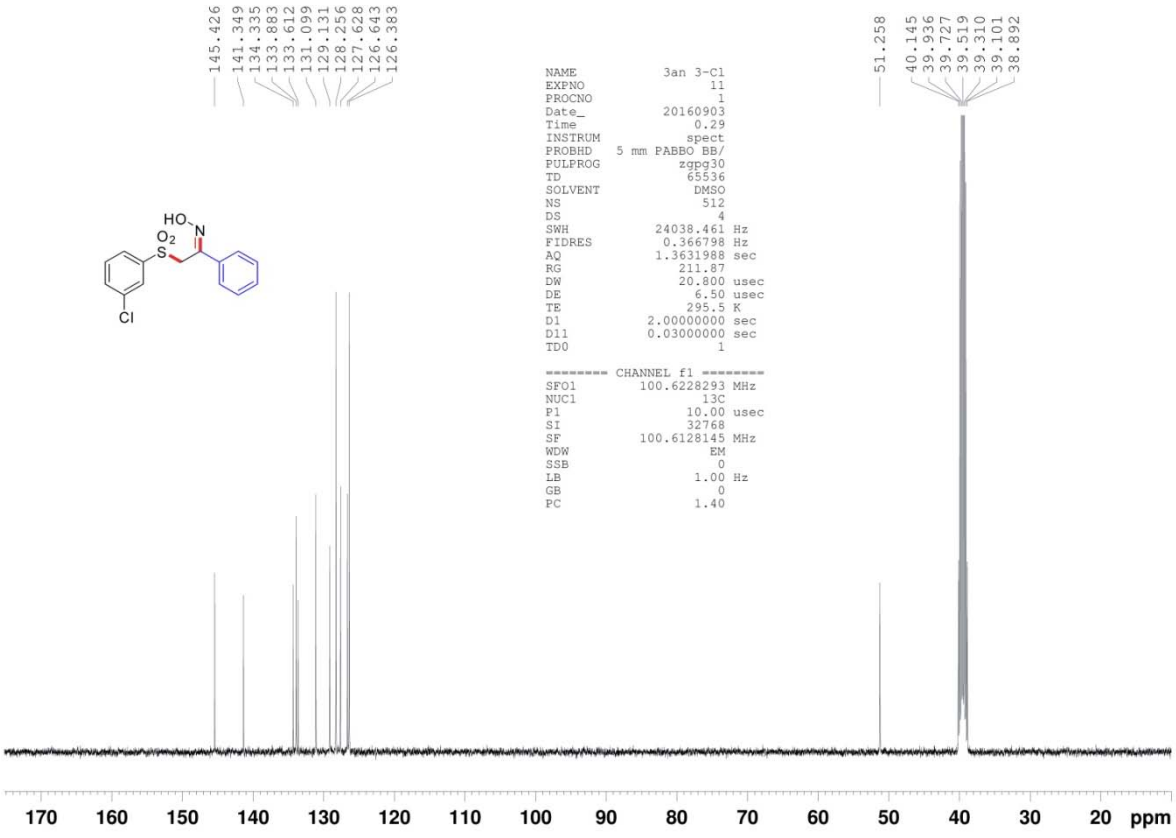
2-((3-nitrophenyl)sulfonyl)-1-phenylethan-1-one oxime(3al).



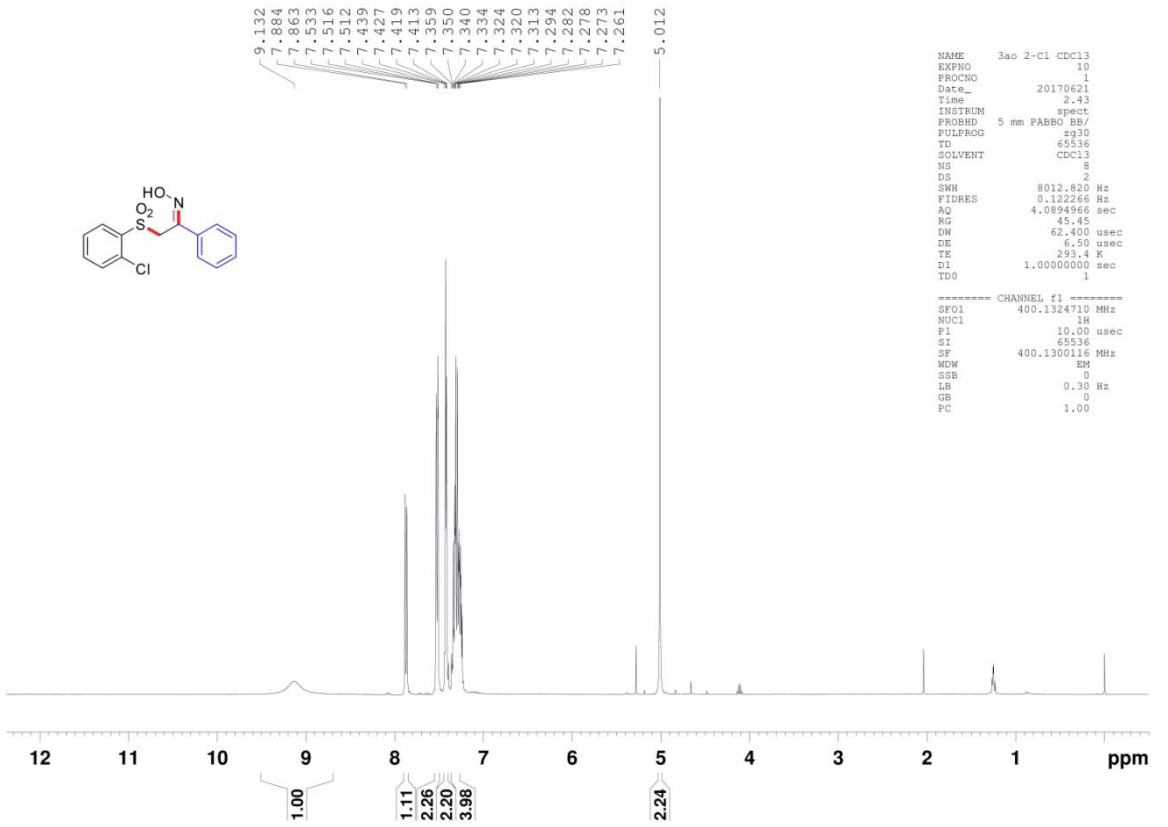


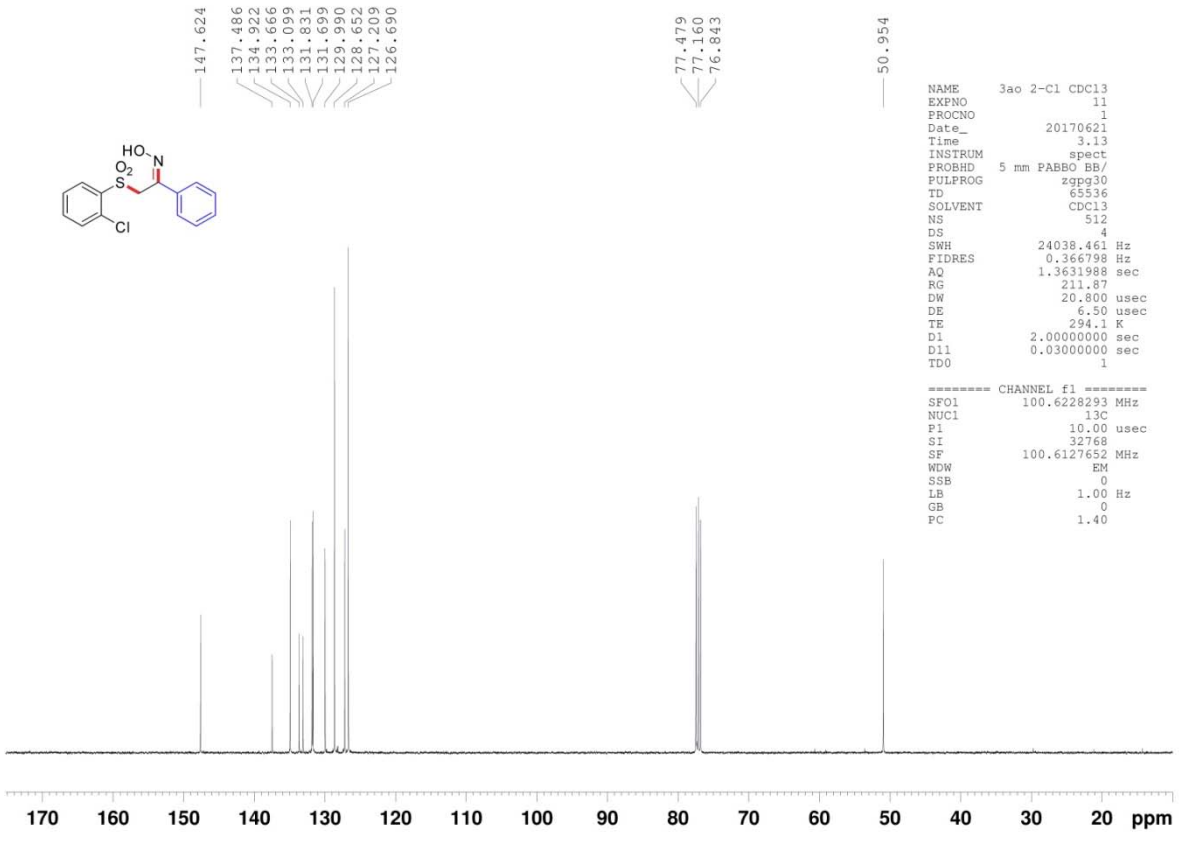
2-((3-chlorophenyl)sulfonyl)-1-phenylethan-1-one oxime(3am).



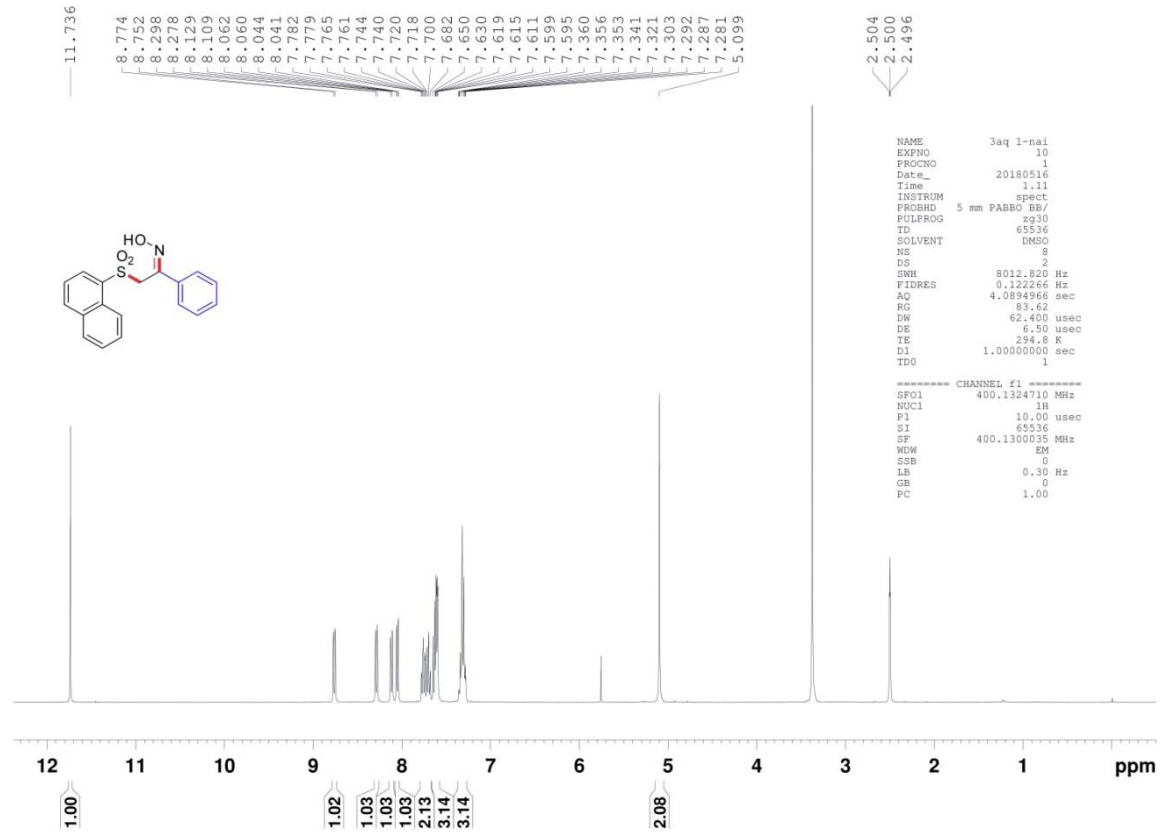


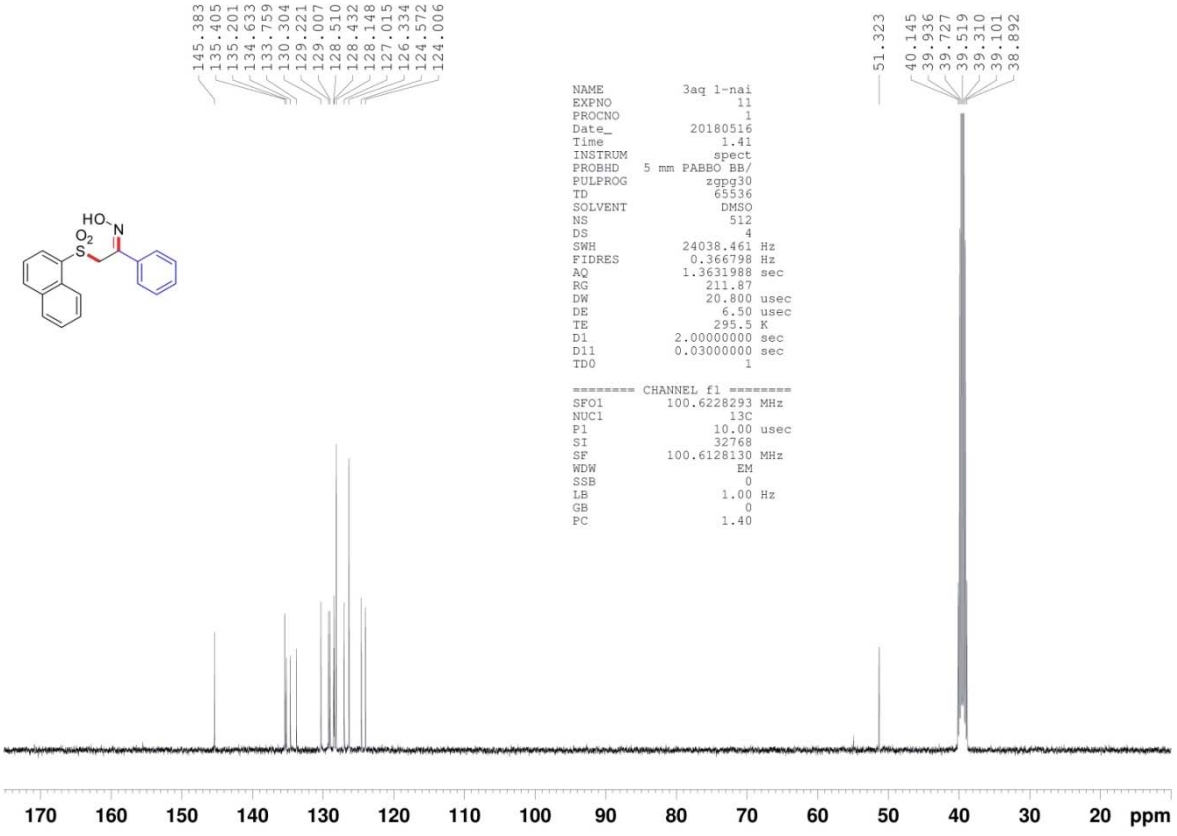
2-((2-chlorophenyl)sulfonyl)-1-phenylethan-1-one oxime(3an).



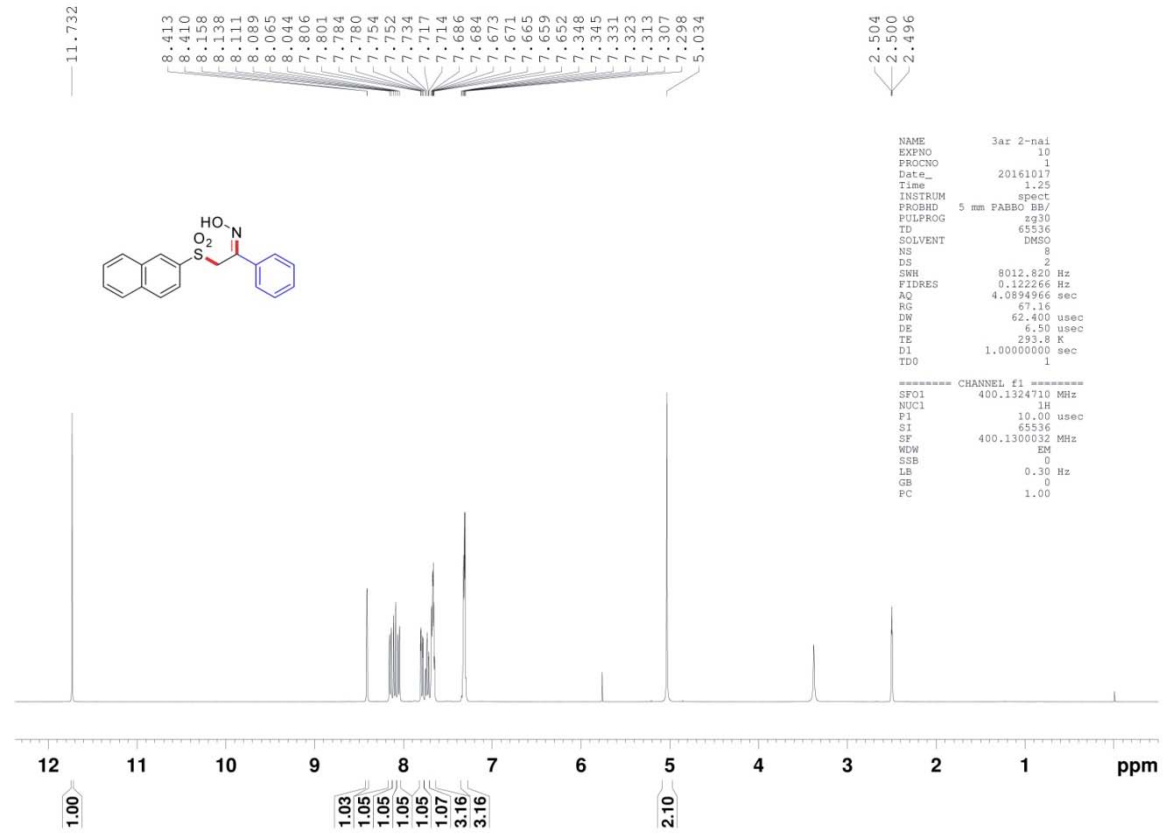


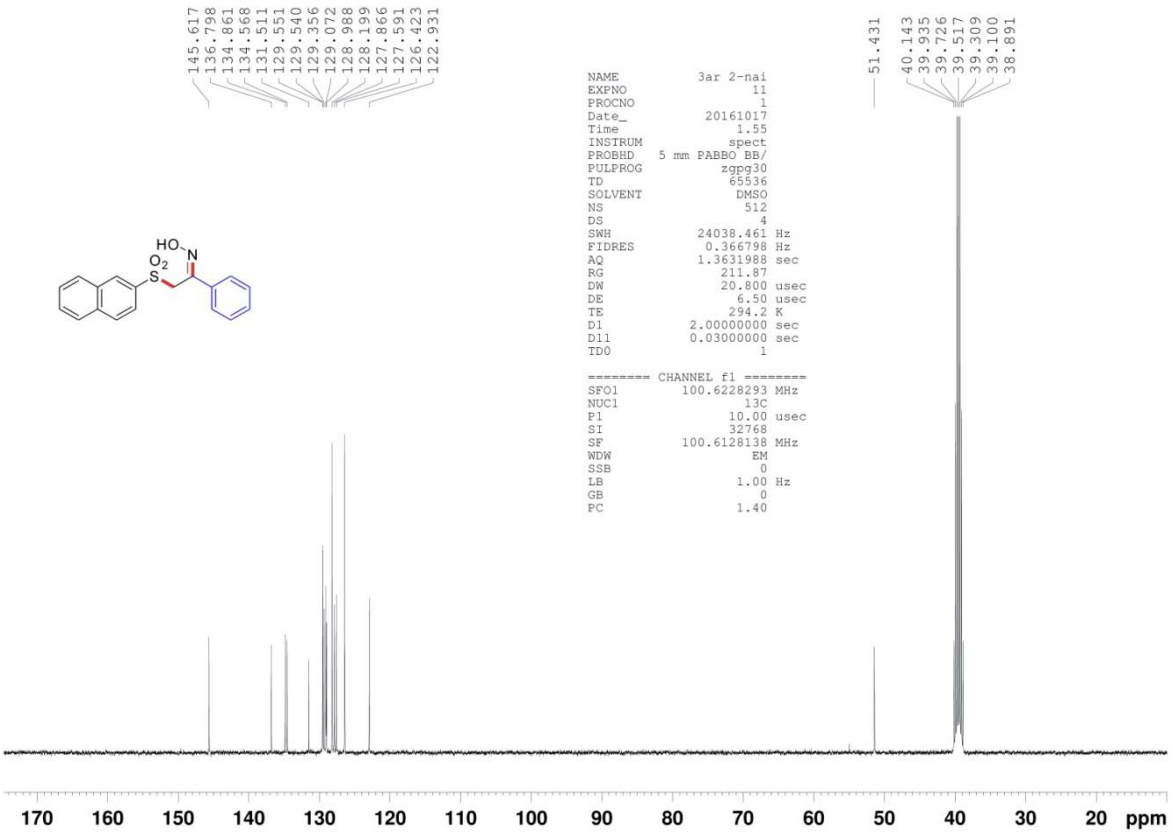
2-(naphthalen-1-ylsulfonyl)-1-phenylethan-1-one oxime(3aq)



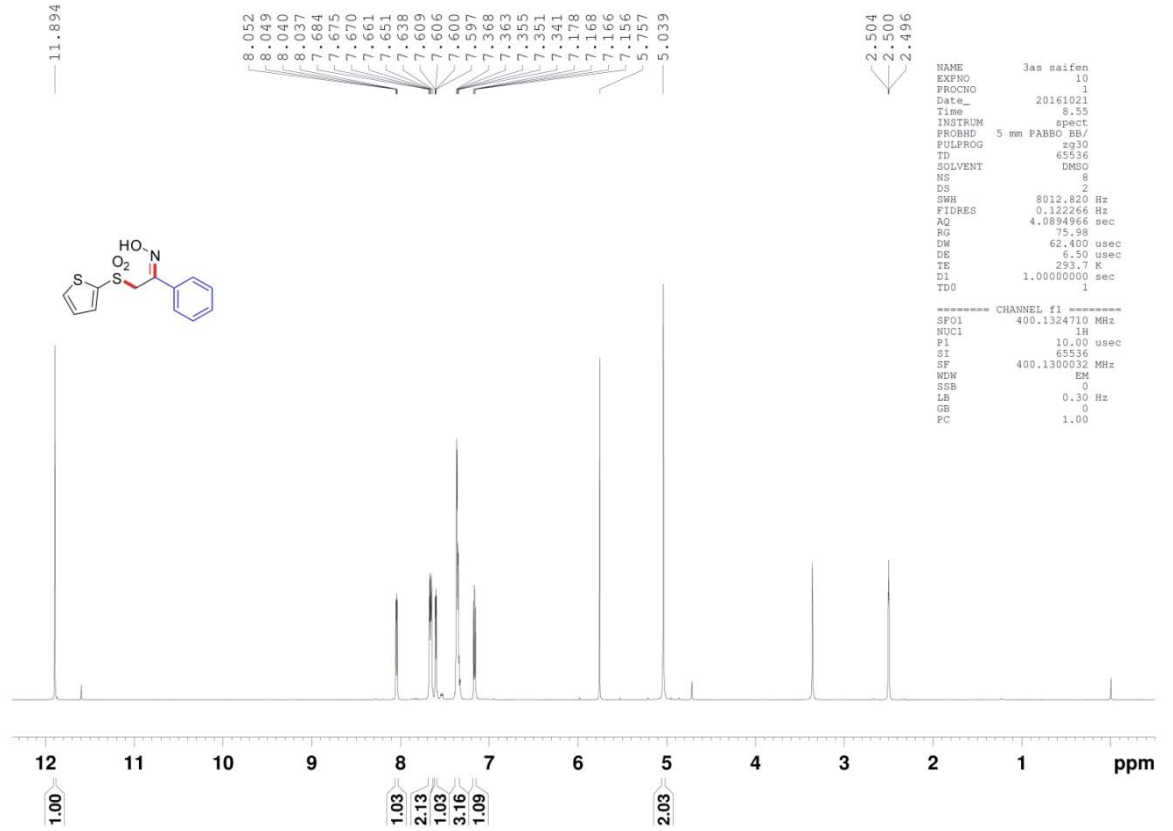


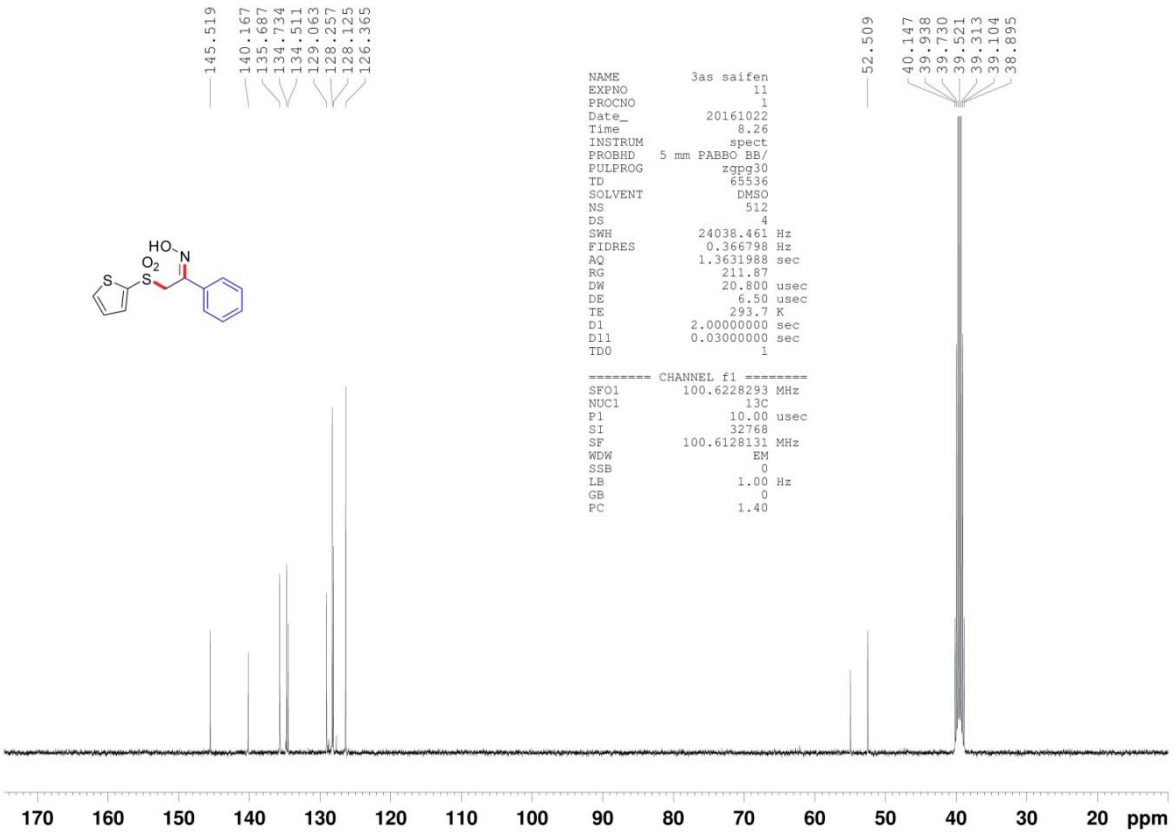
2-(naphthalen-2-ylsulfonyl)-1-phenylethan-1-one oxime(3ar).



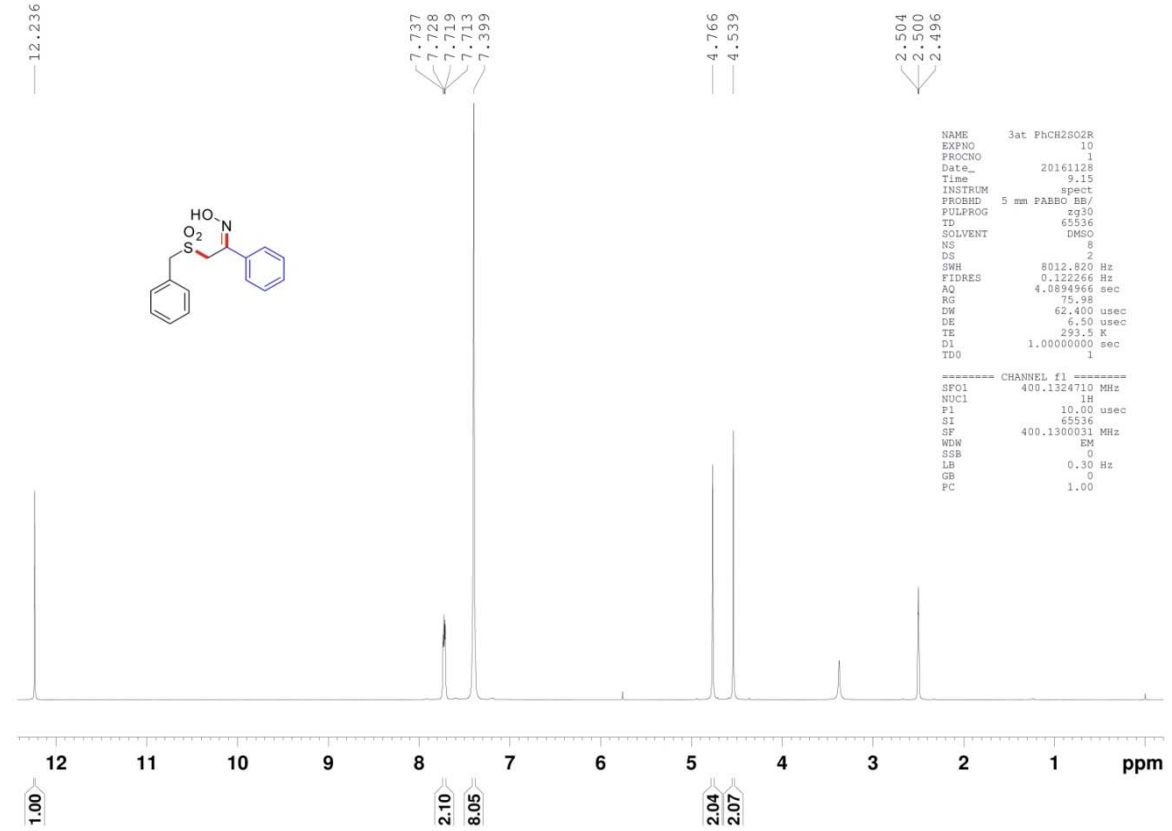


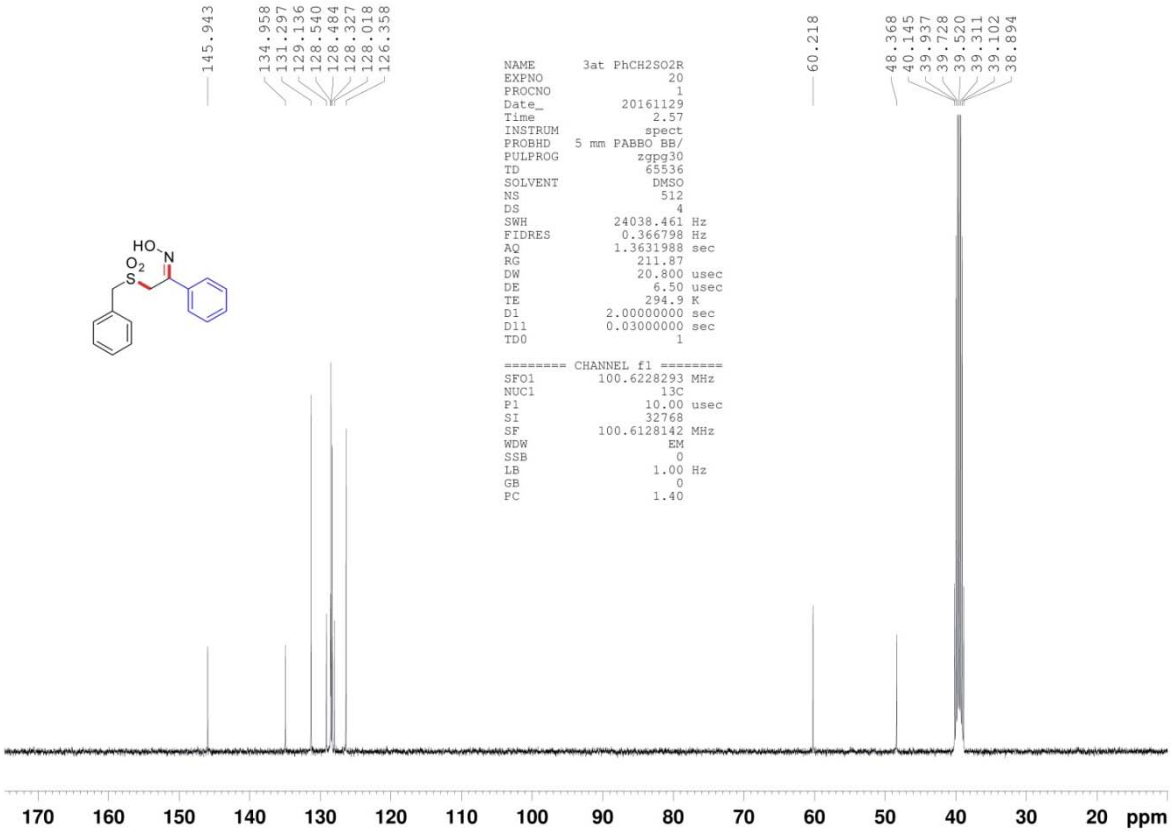
1-phenyl-2-(thiophen-2-ylsulfonyl)ethan-1-one oxime(3as).



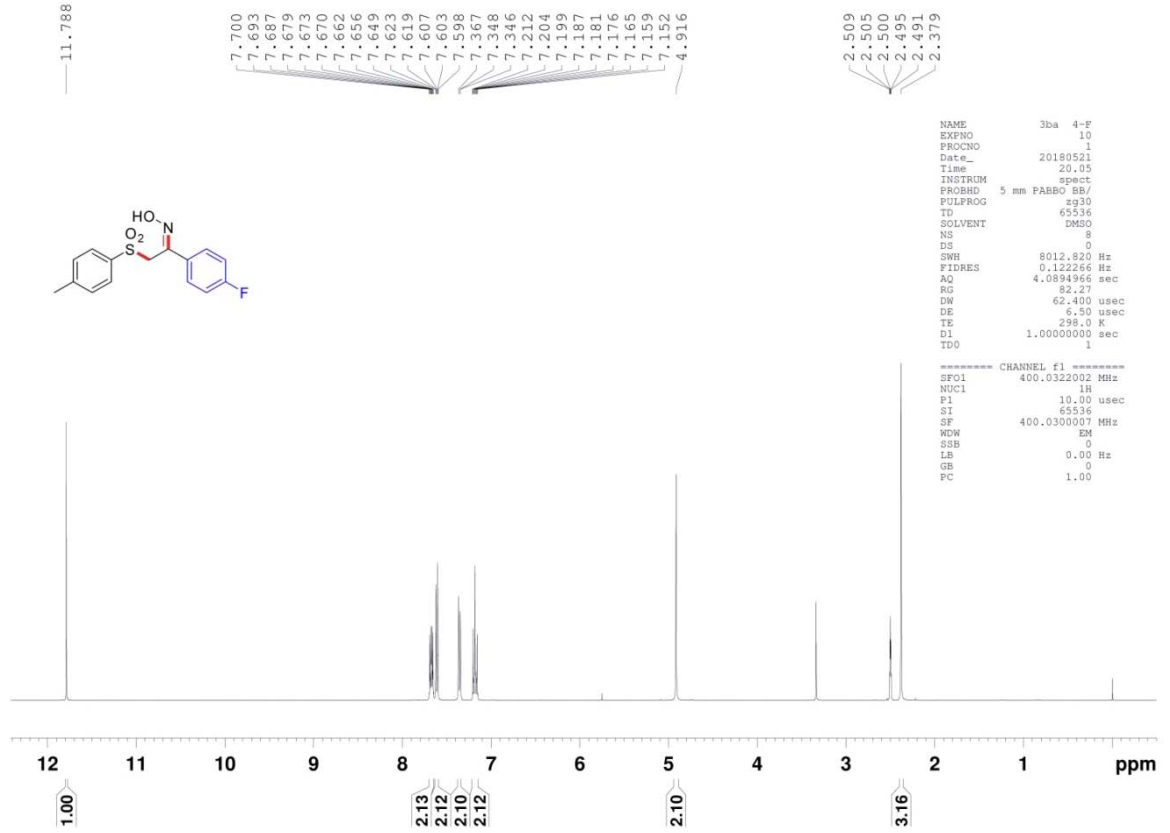


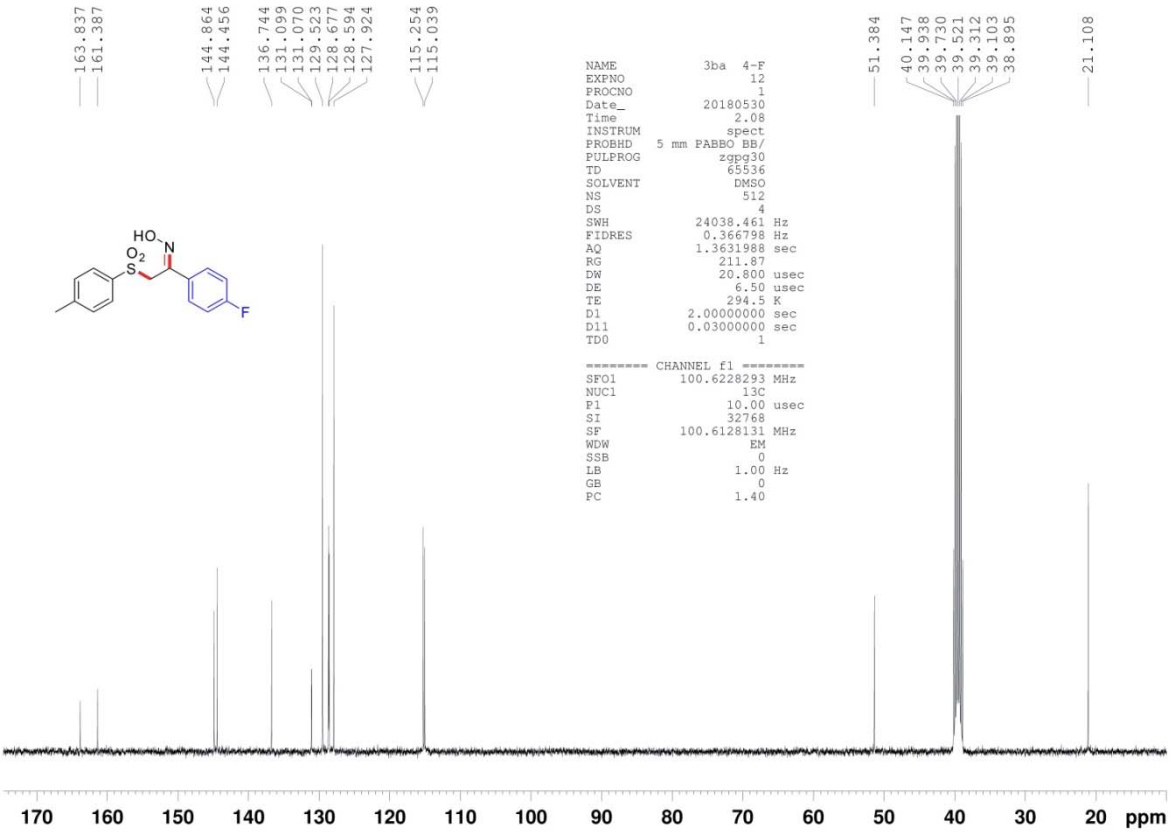
2-(benzylsulfonyl)-1-phenylethan-1-one oxime (3at).

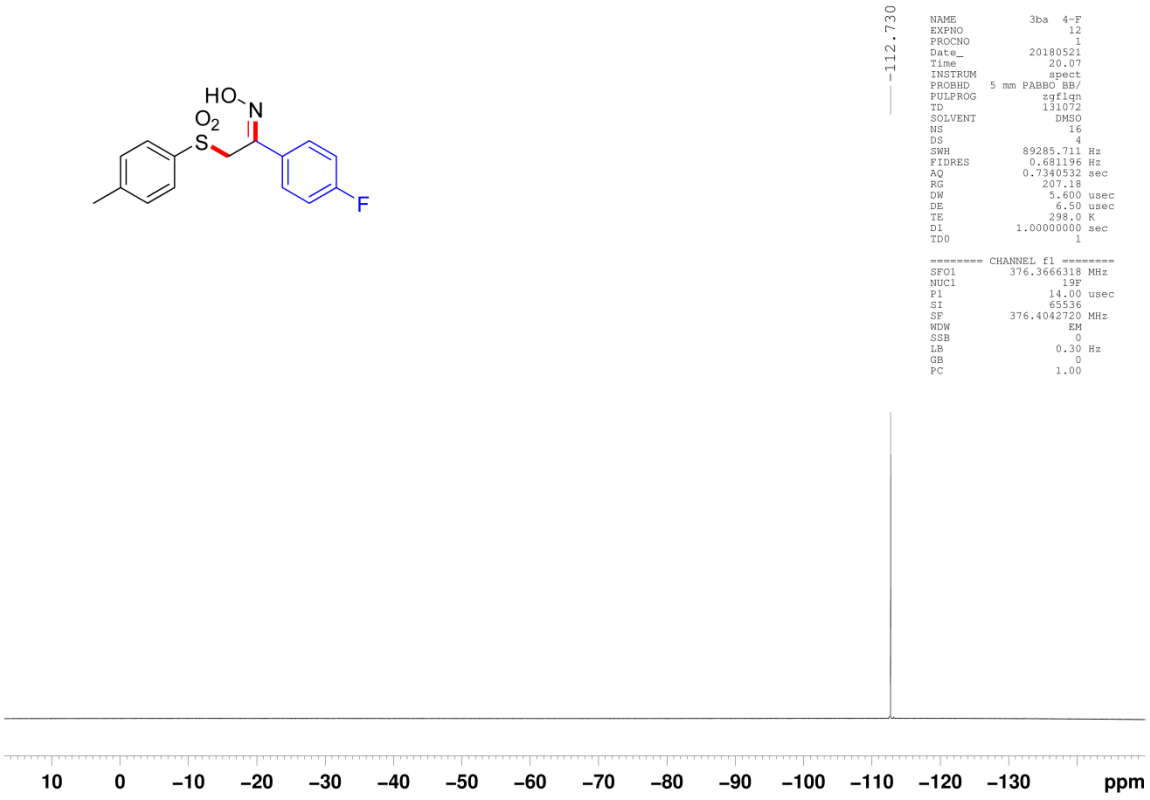




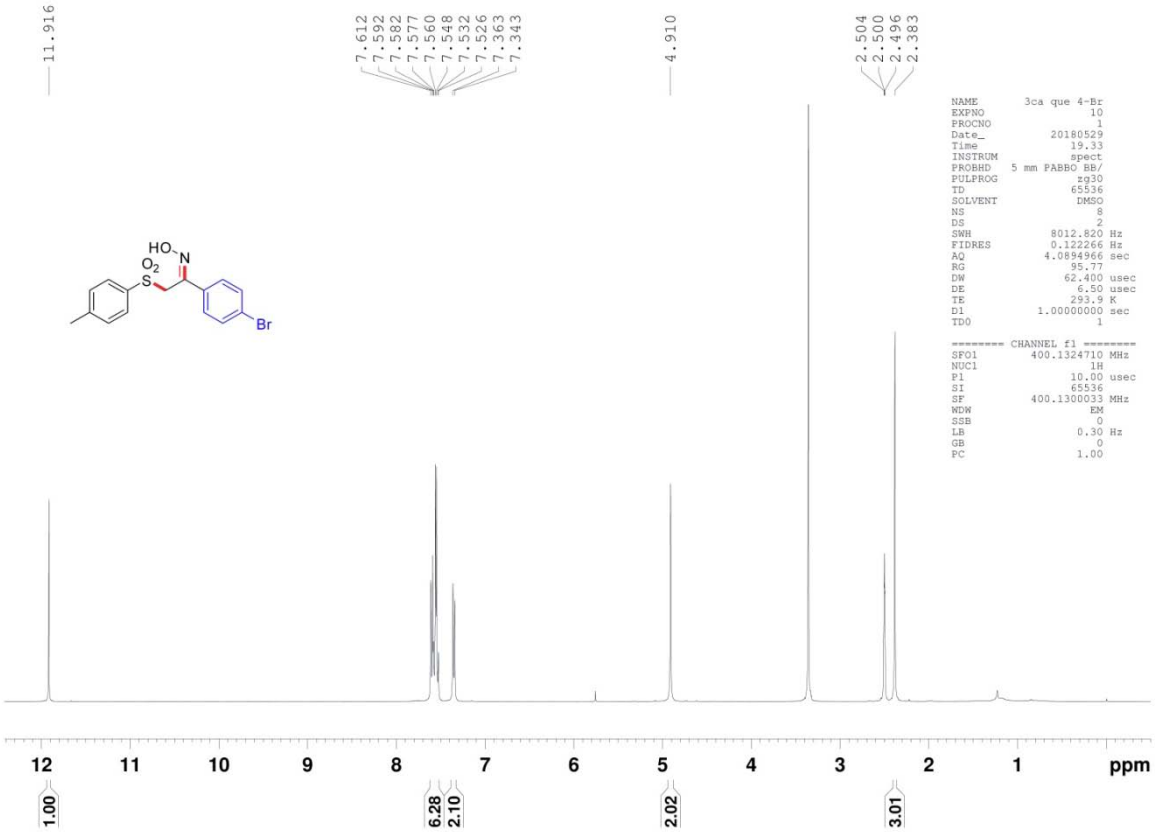
1-(4-fluorophenyl)-2-tosylethan-1-one oxime(3ba).

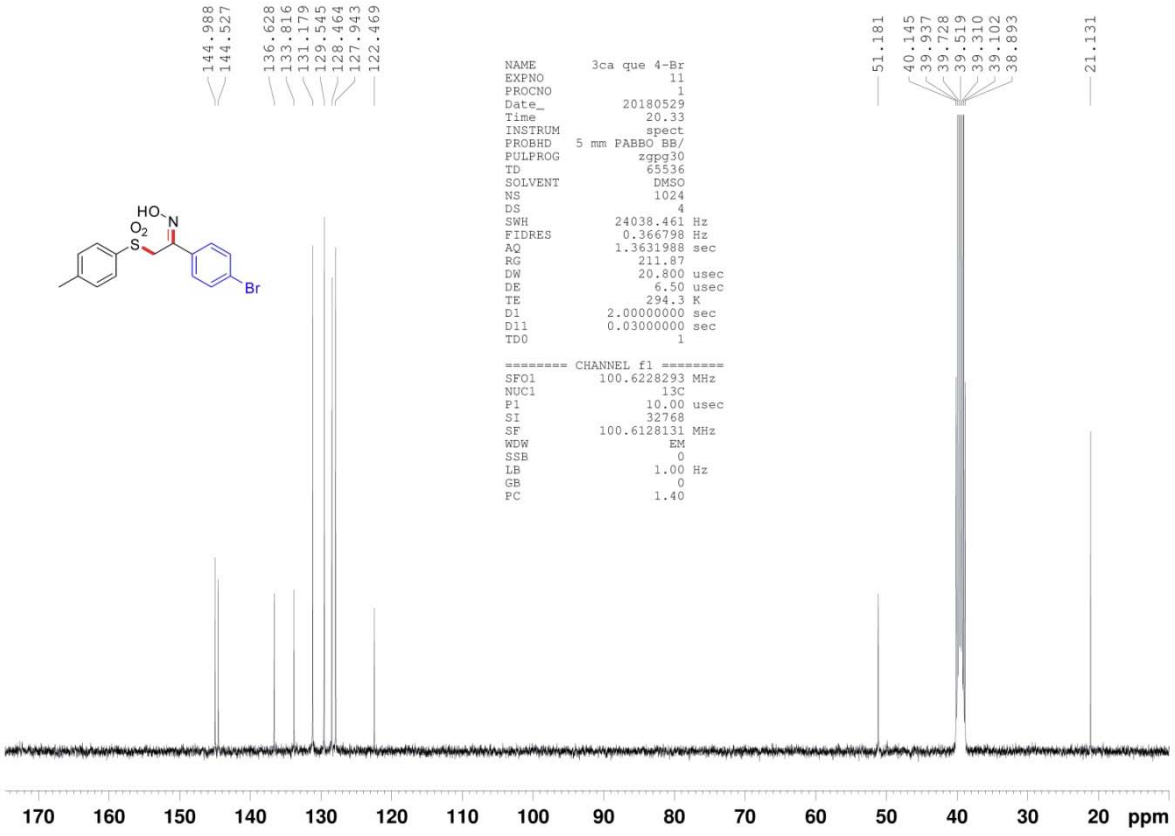




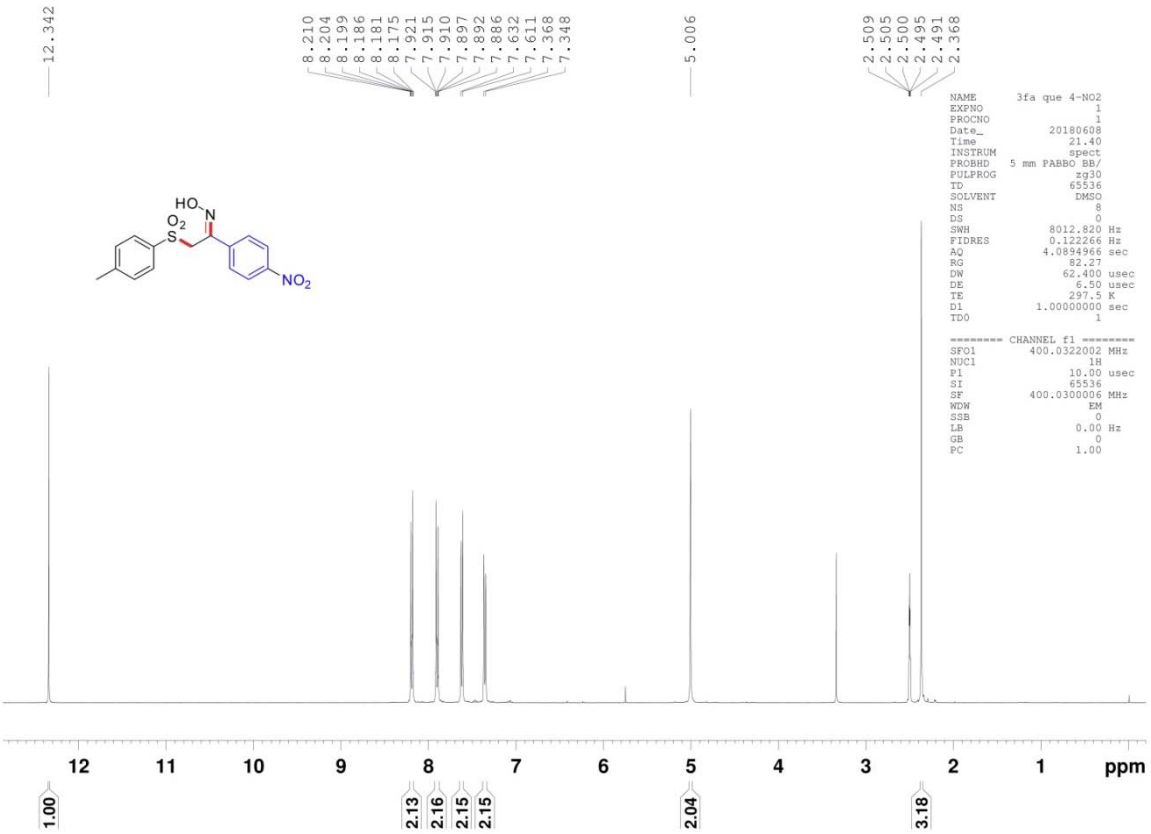


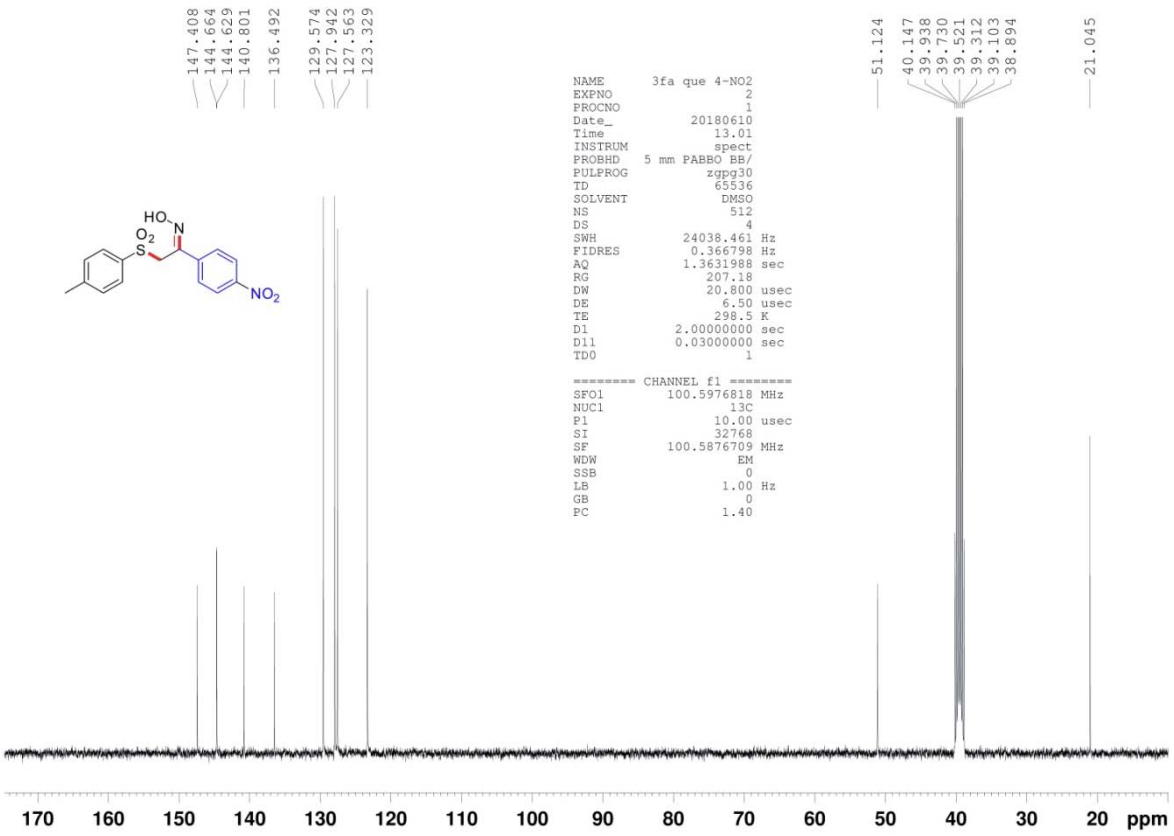
1-(4-bromophenyl)-2-tosylethan-1-one oxime(3ca).



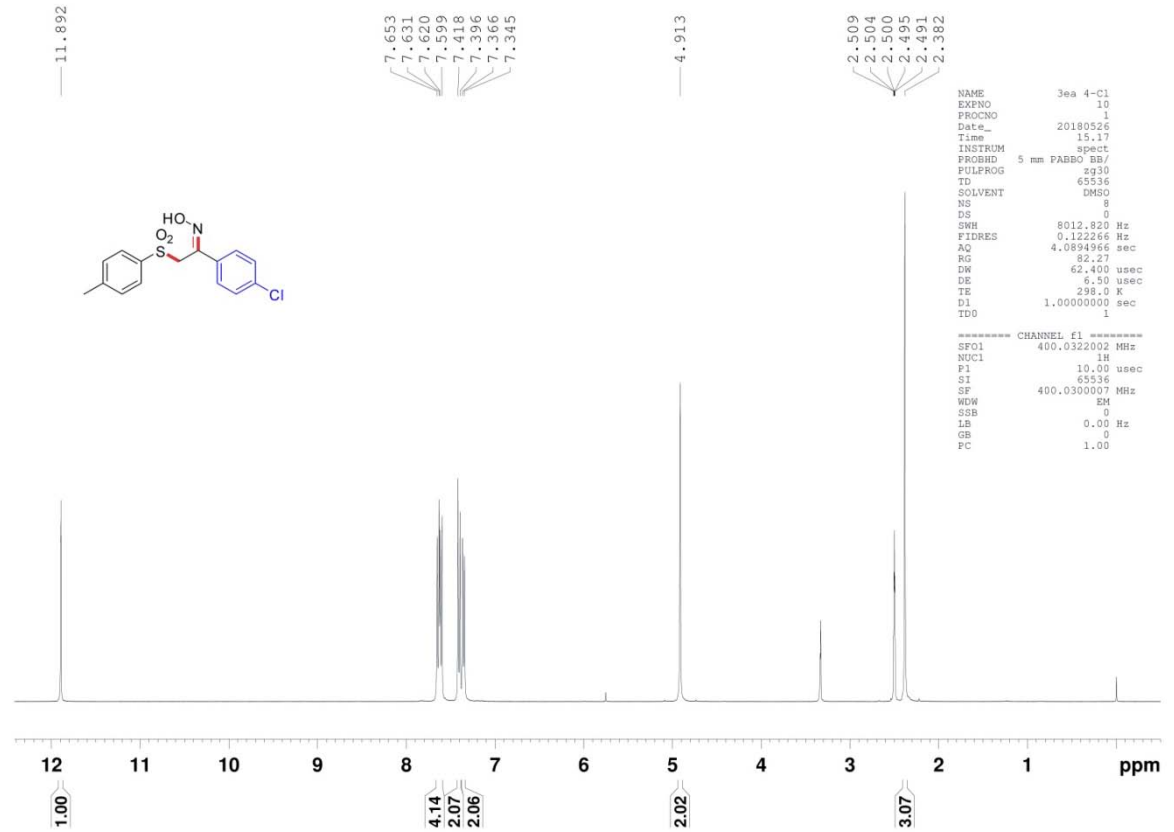


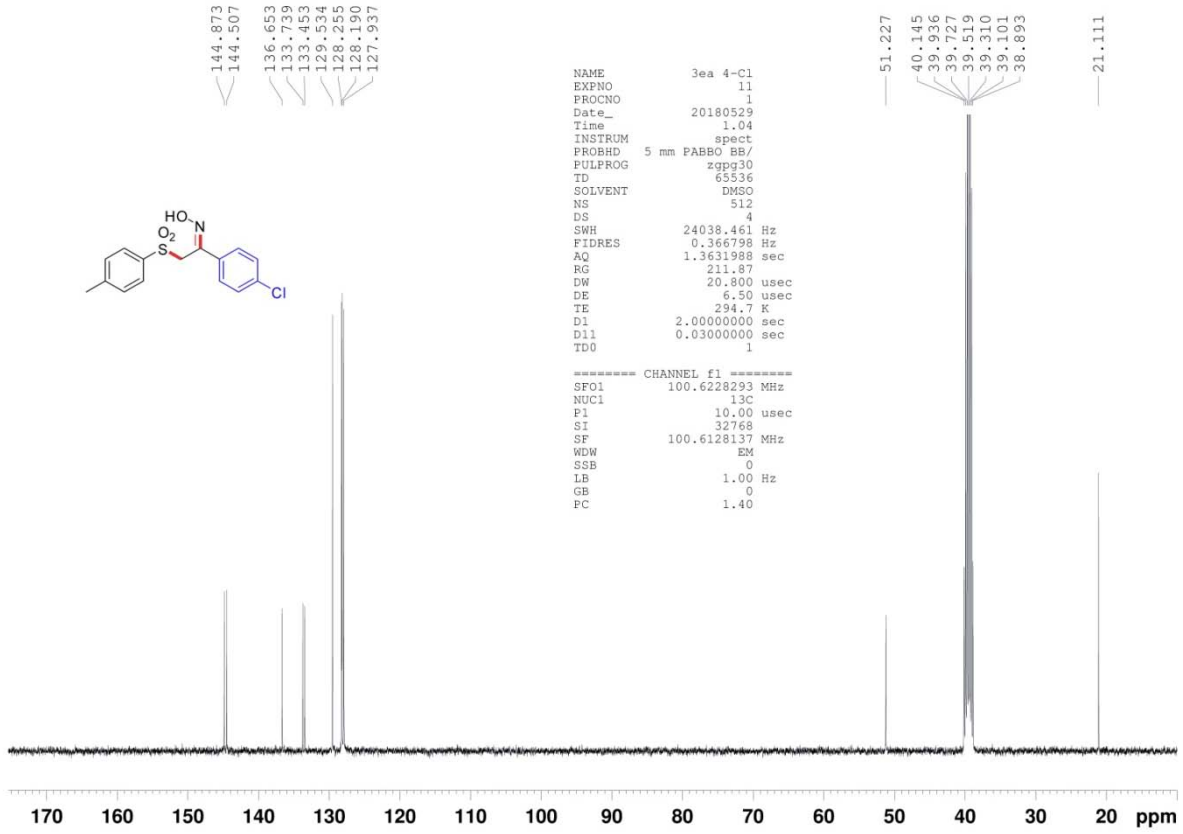
1-(4-nitrophenyl)-2-tosylethan-1-one oxime(3da).



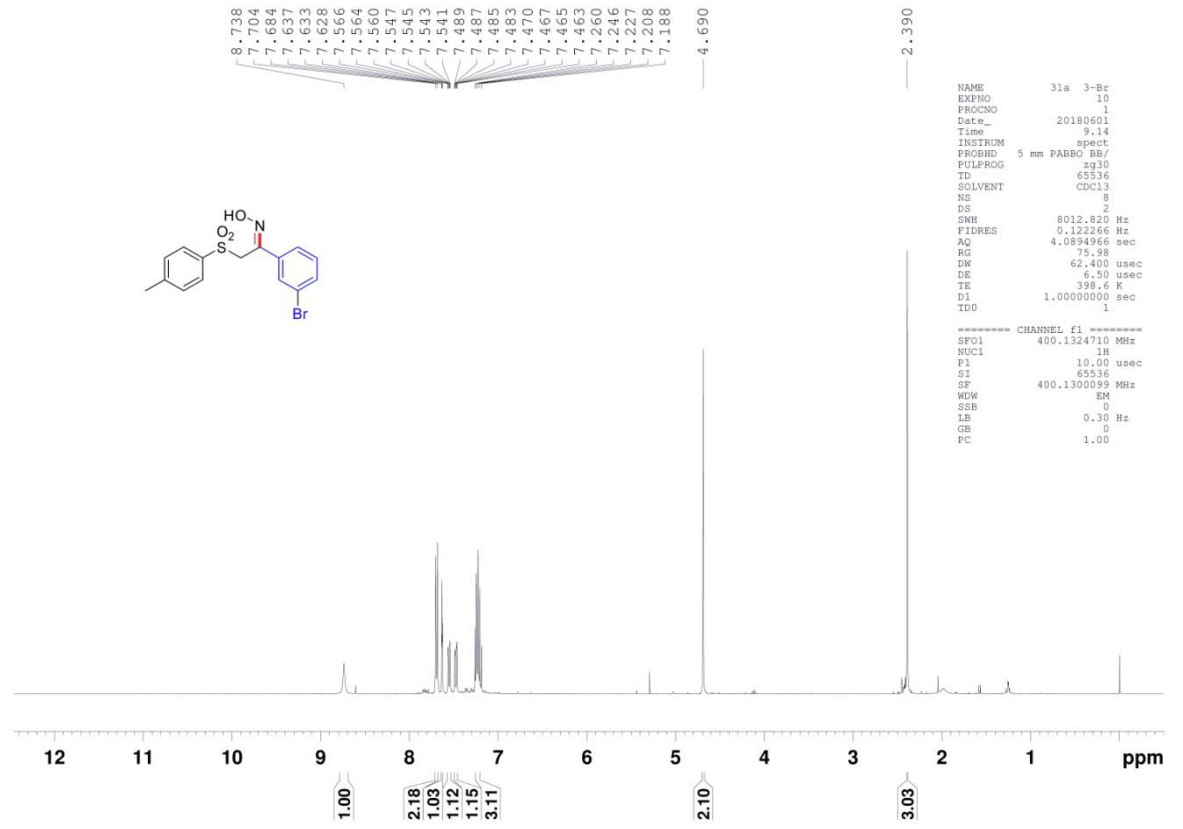


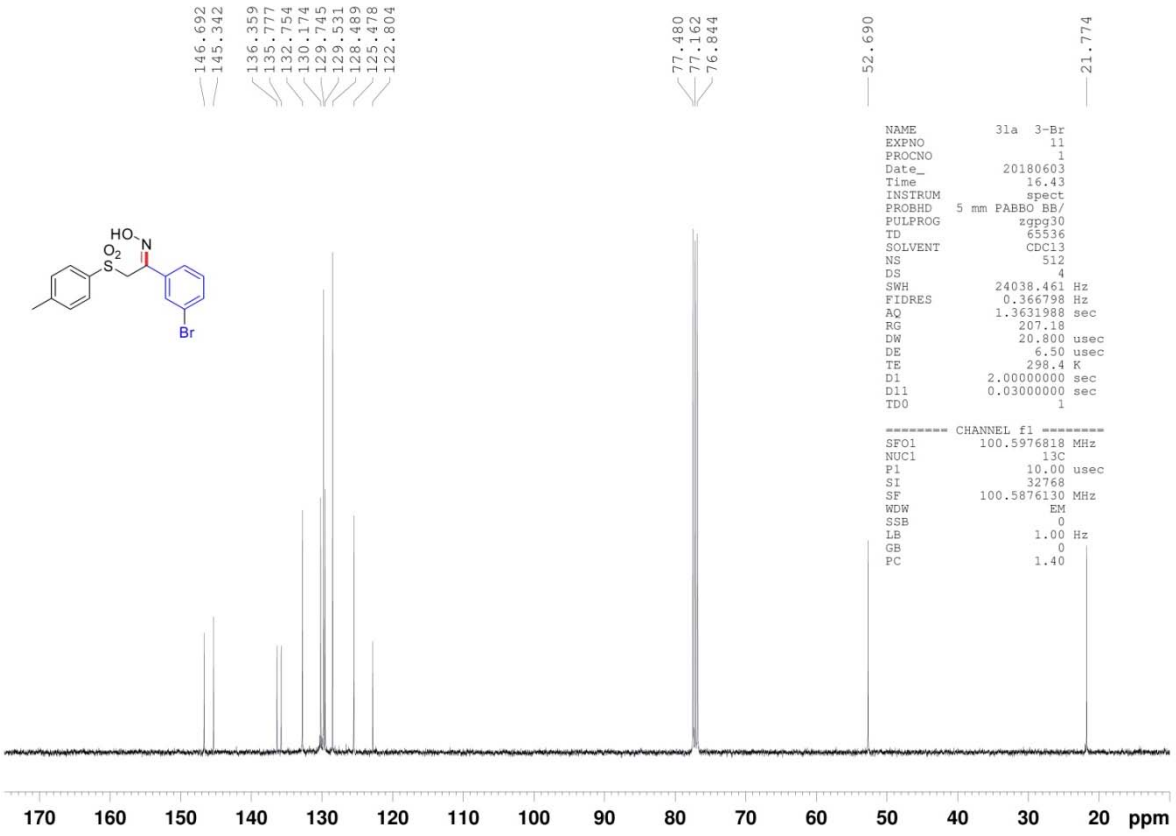
1-(4-chlorophenyl)-2-tosylethan-1-one oxime(3ea).



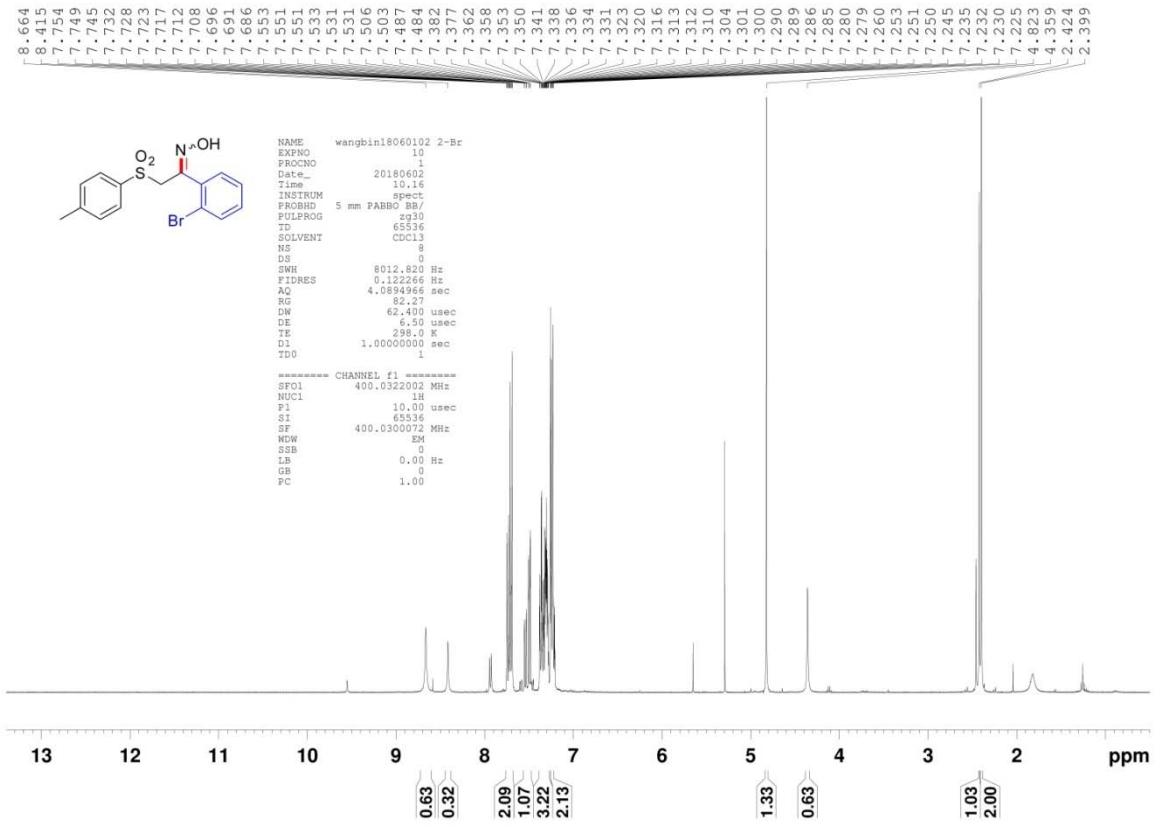


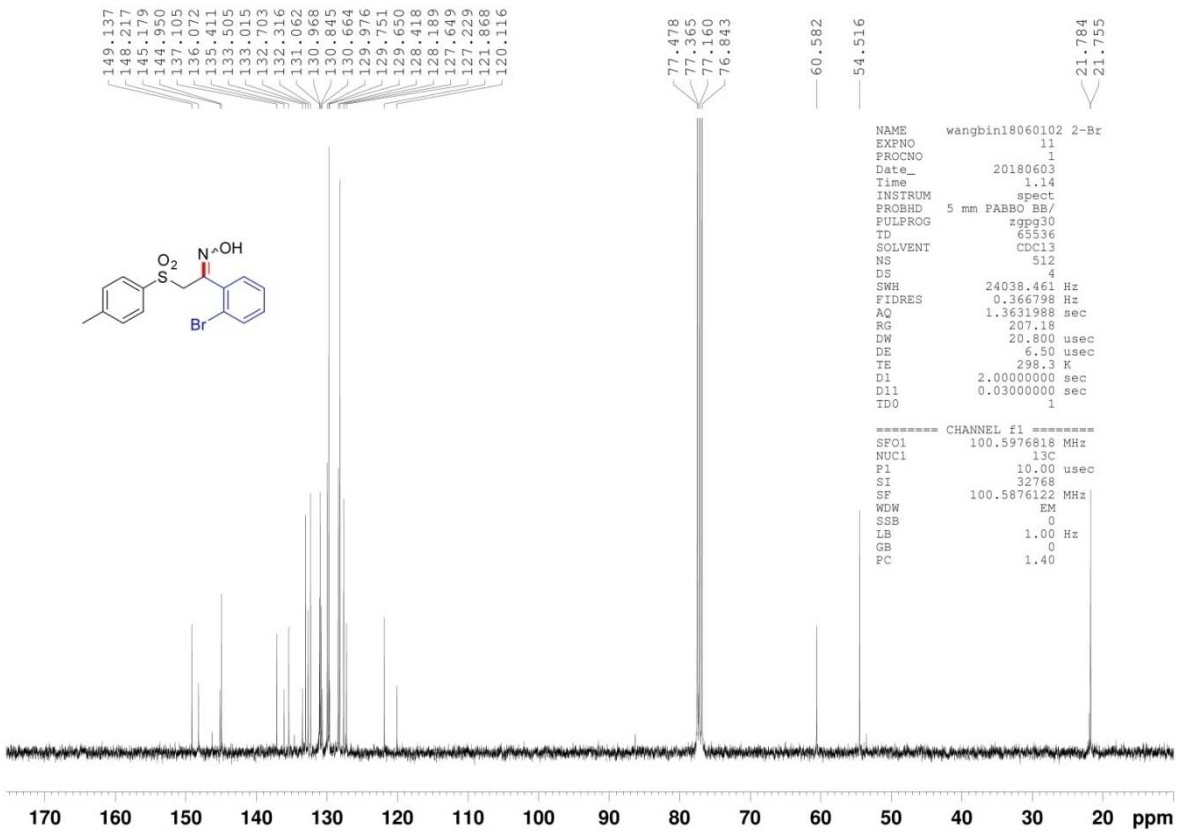
1-(3-bromophenyl)-2-tosylethan-1-one oxime(3fa).



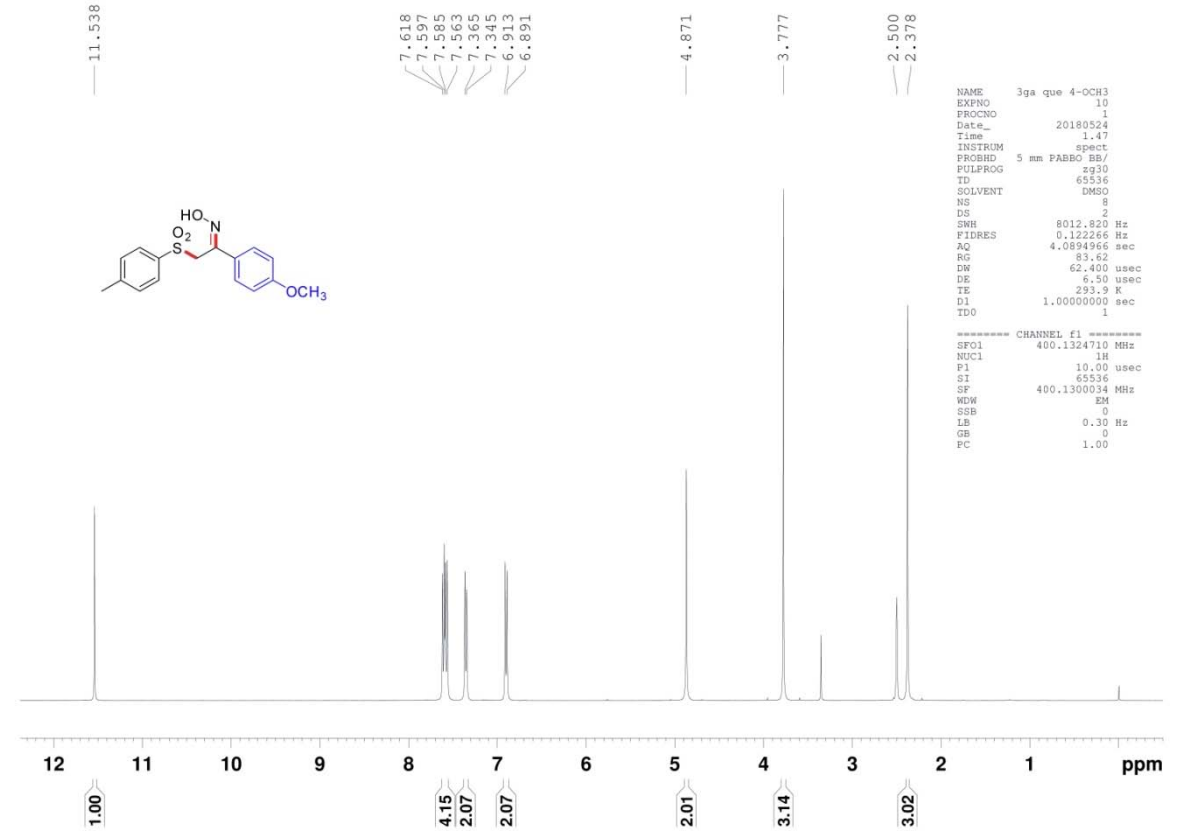


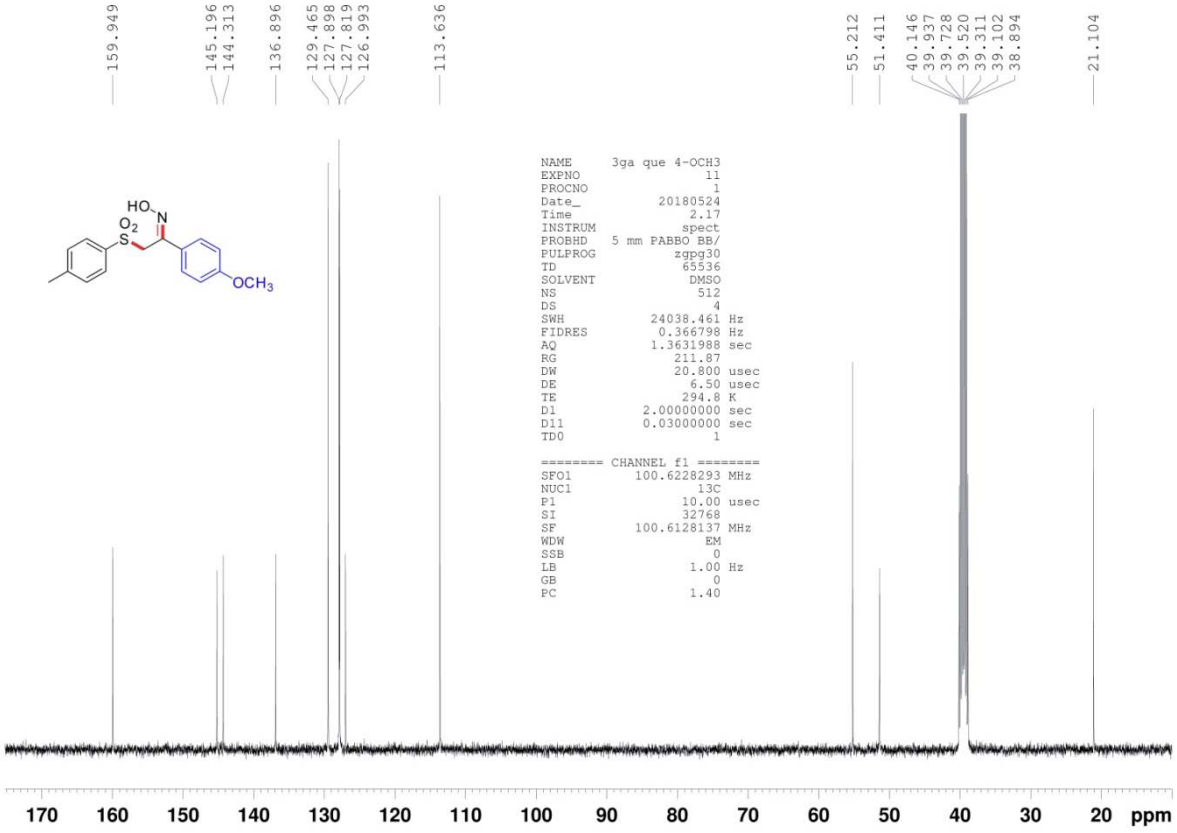
(Z/E)-1-(2-bromophenyl)-2-tosylethan-1-one oxime (3ha).



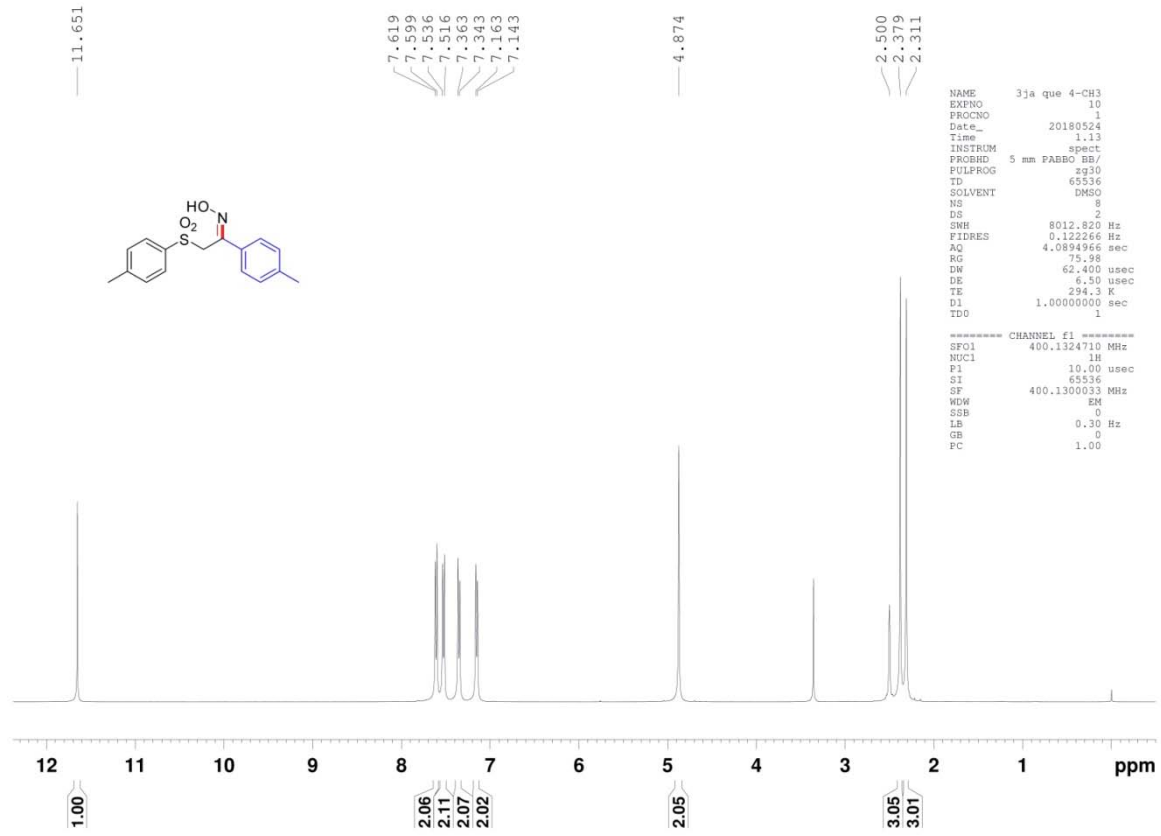


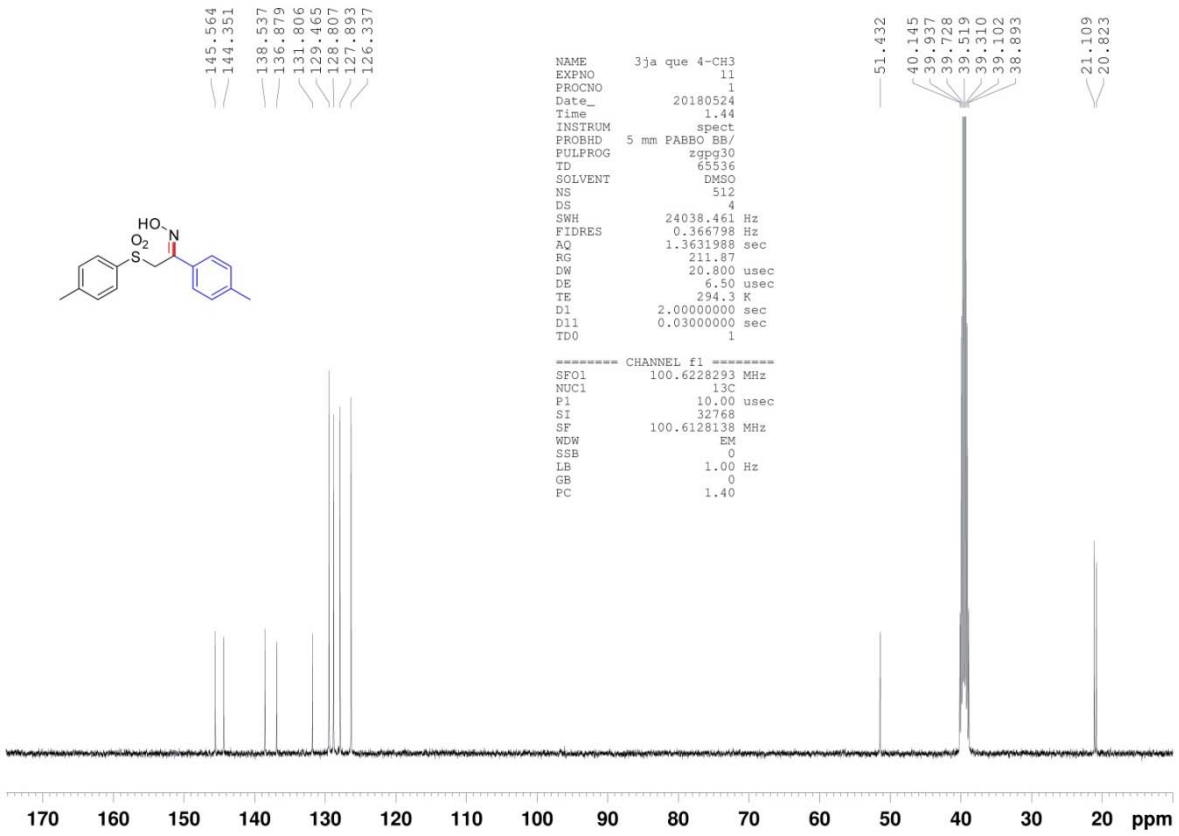
1-(4-methoxyphenyl)-2-tosylethan-1-one oxime(3ia).



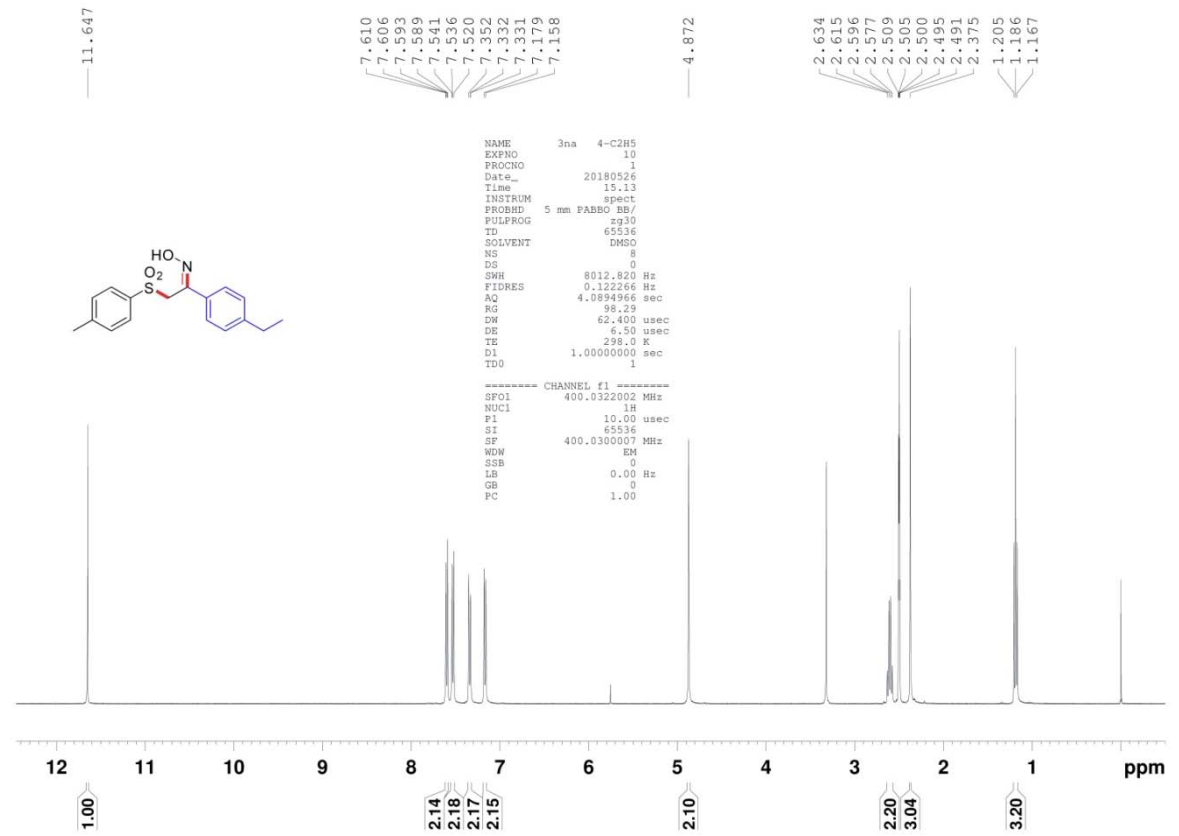


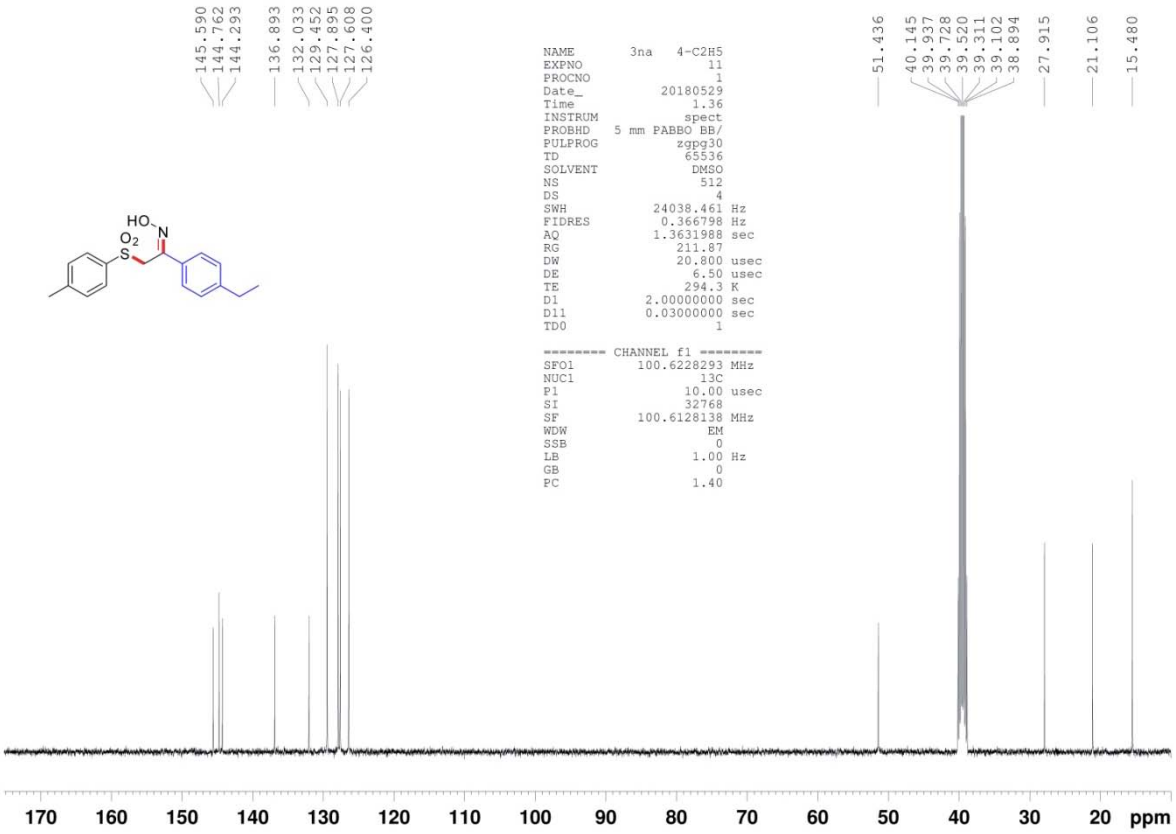
1-(p-tolyl)-2-tosylethan-1-one oxime(3ja).



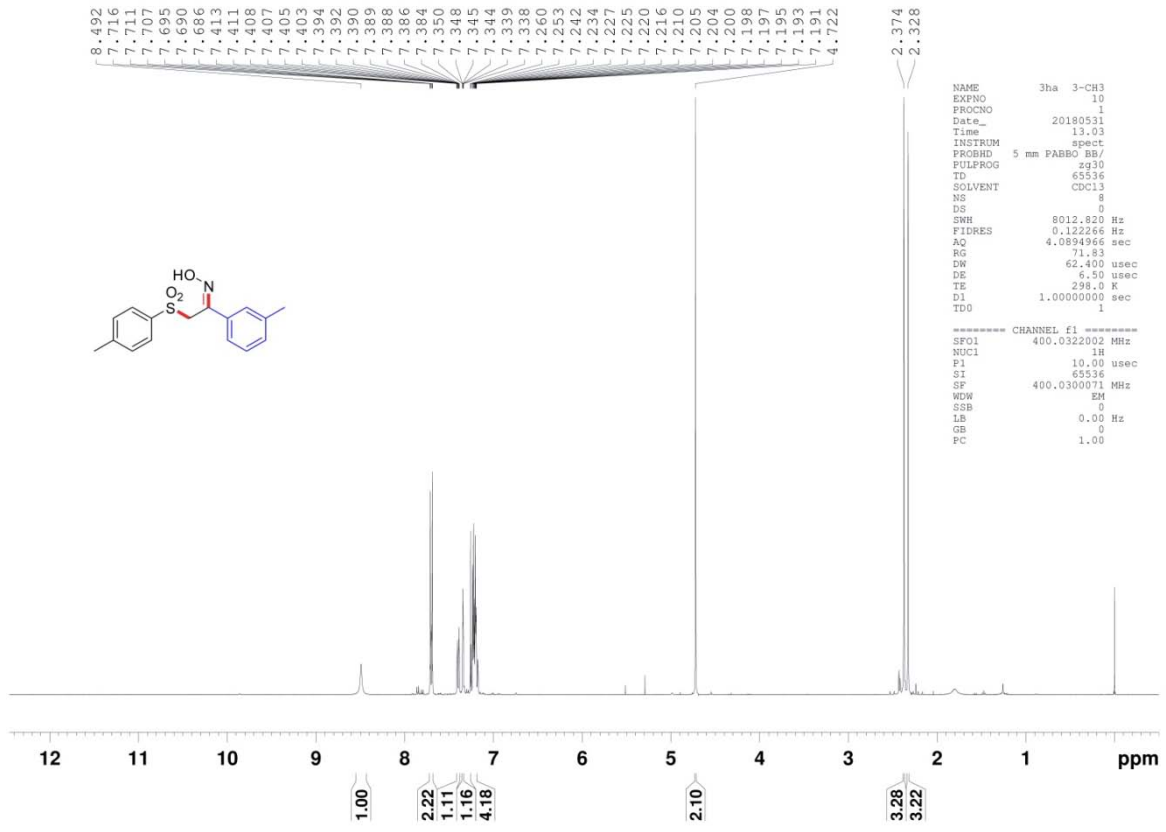


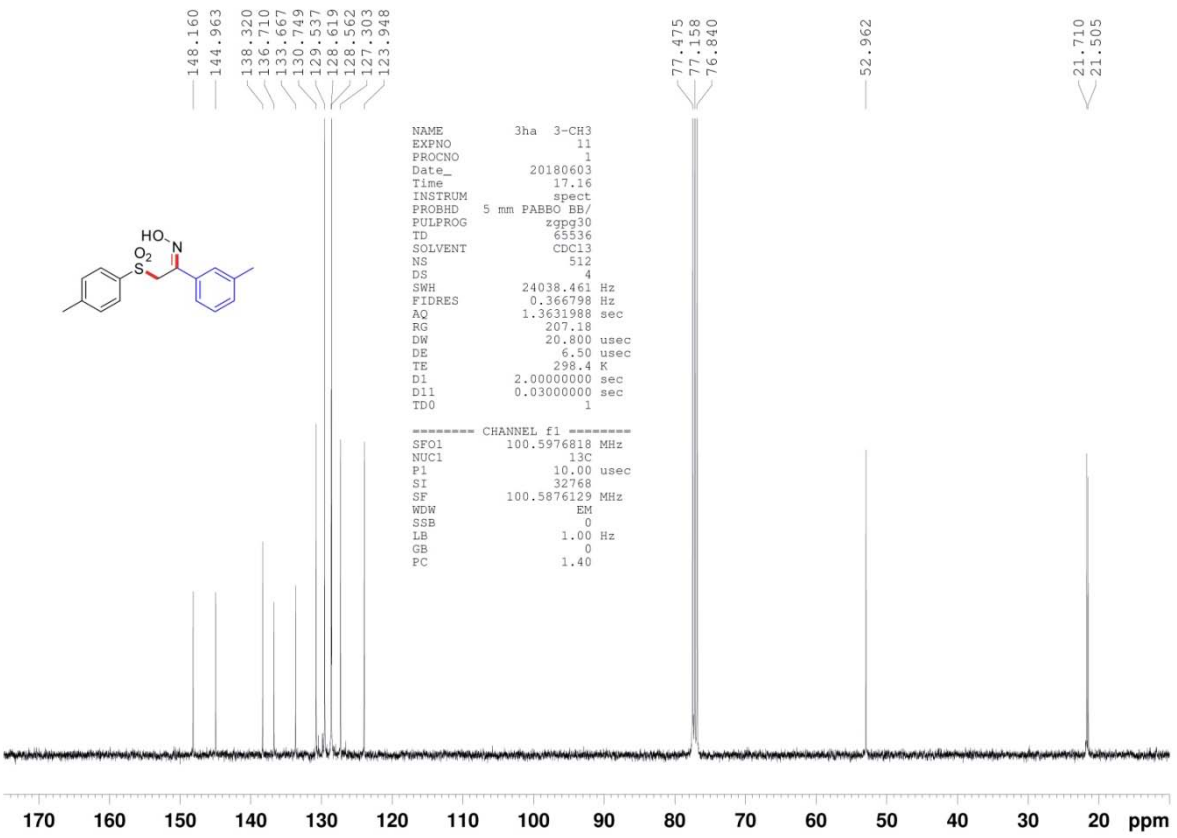
1-(4-ethylphenyl)-2-tosylethan-1-one oxime(3ka).



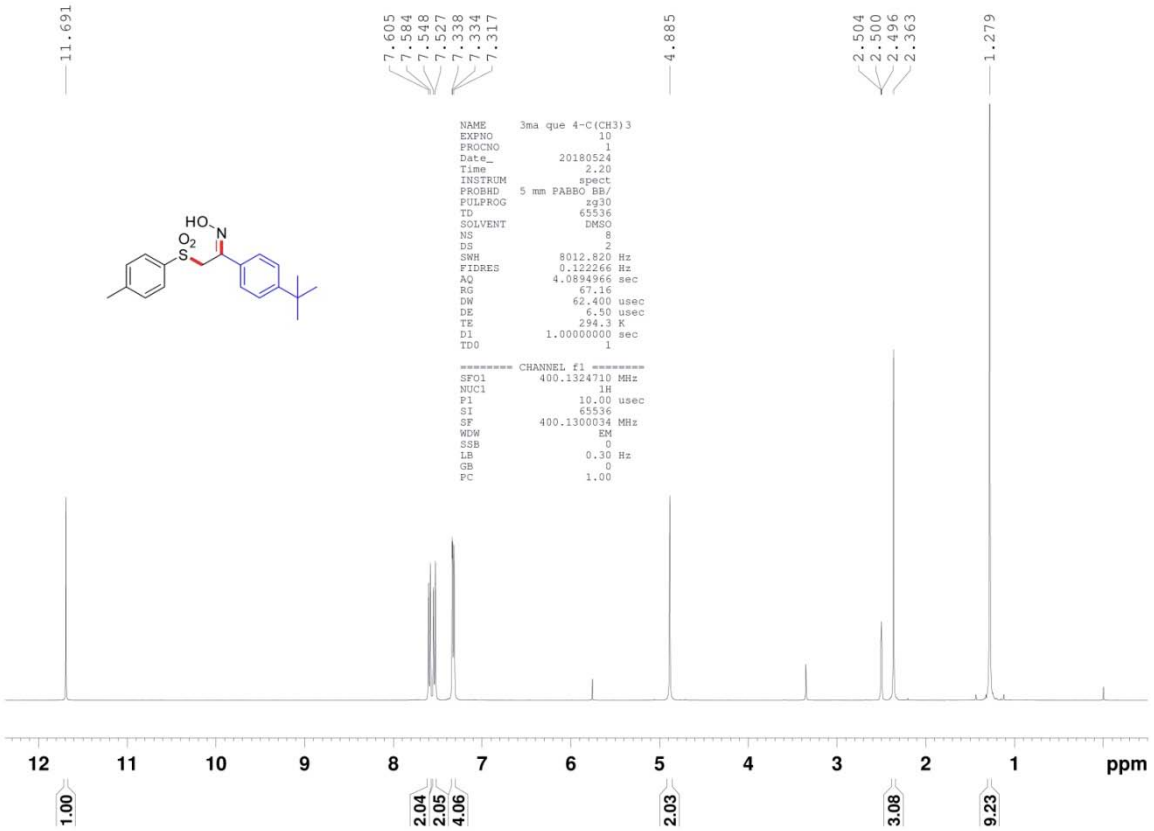


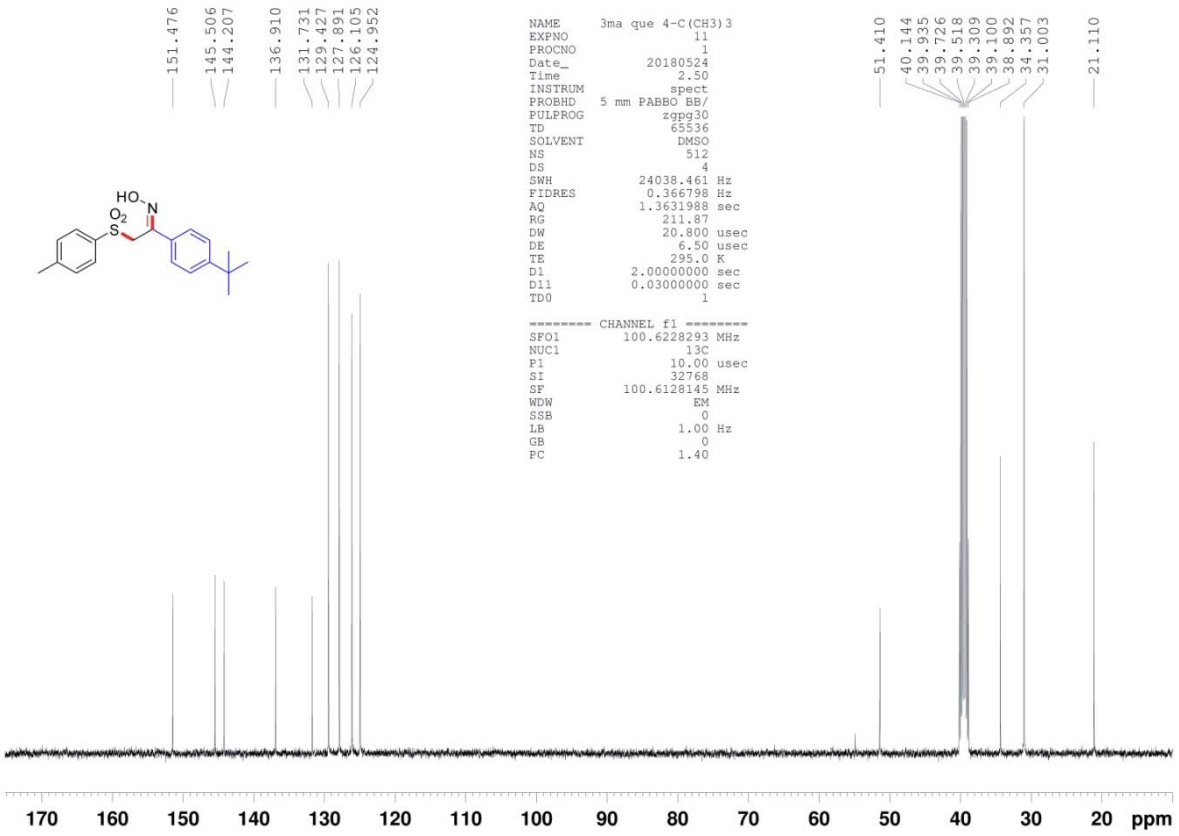
1-(m-tolyl)-2-tosylethan-1-one oxime(3la).



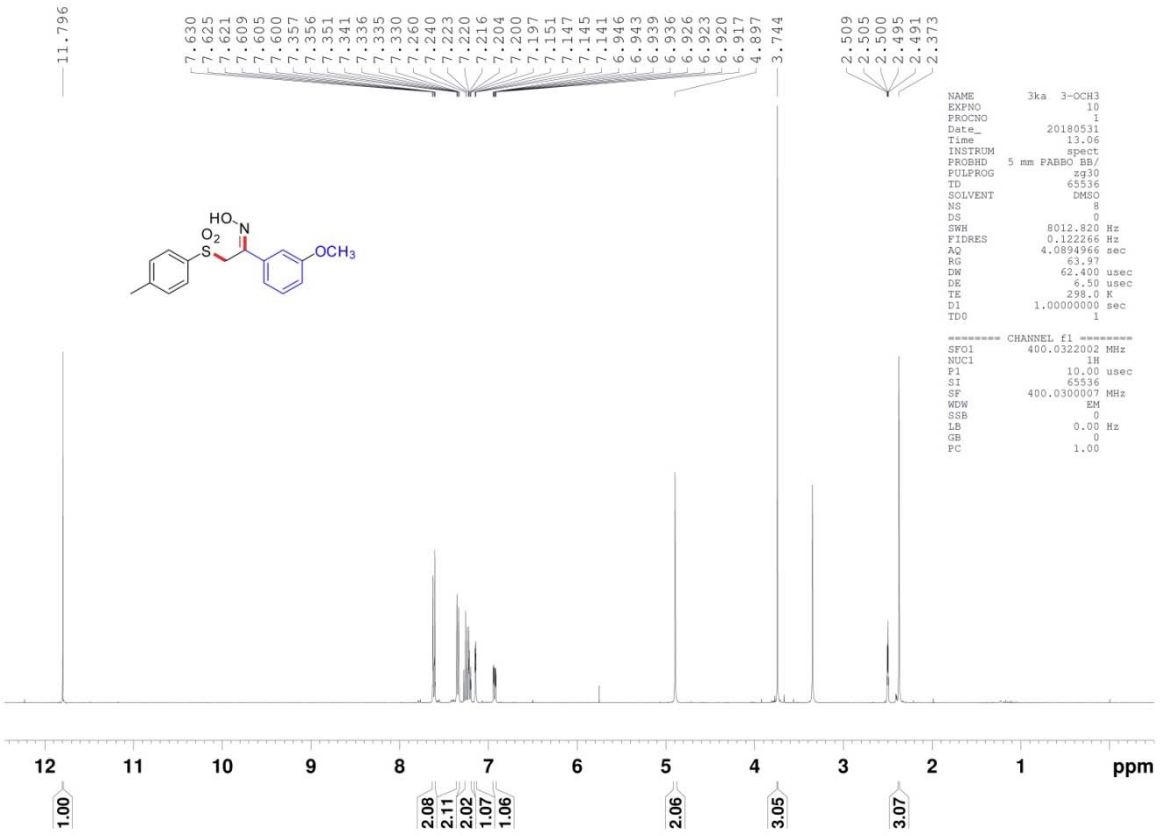


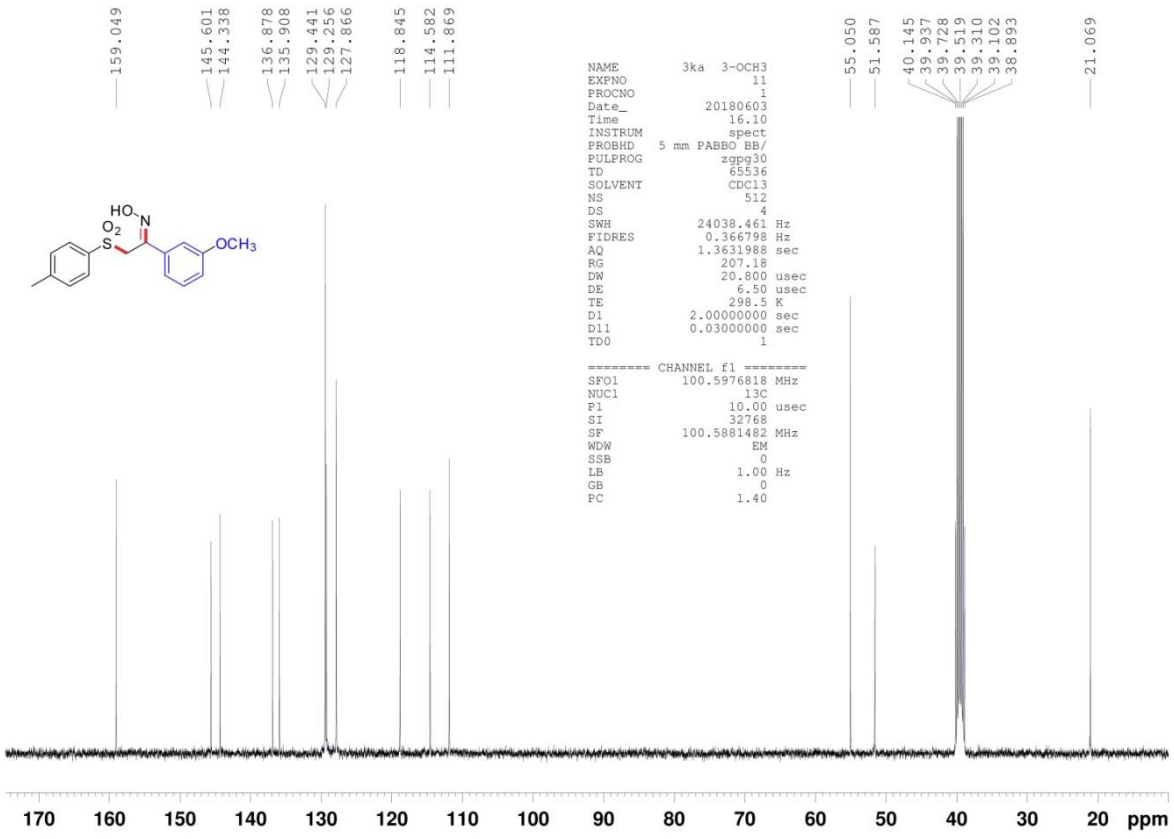
1-(4-(tert-butyl)phenyl)-2-tosylethan-1-one oxime(3ma).



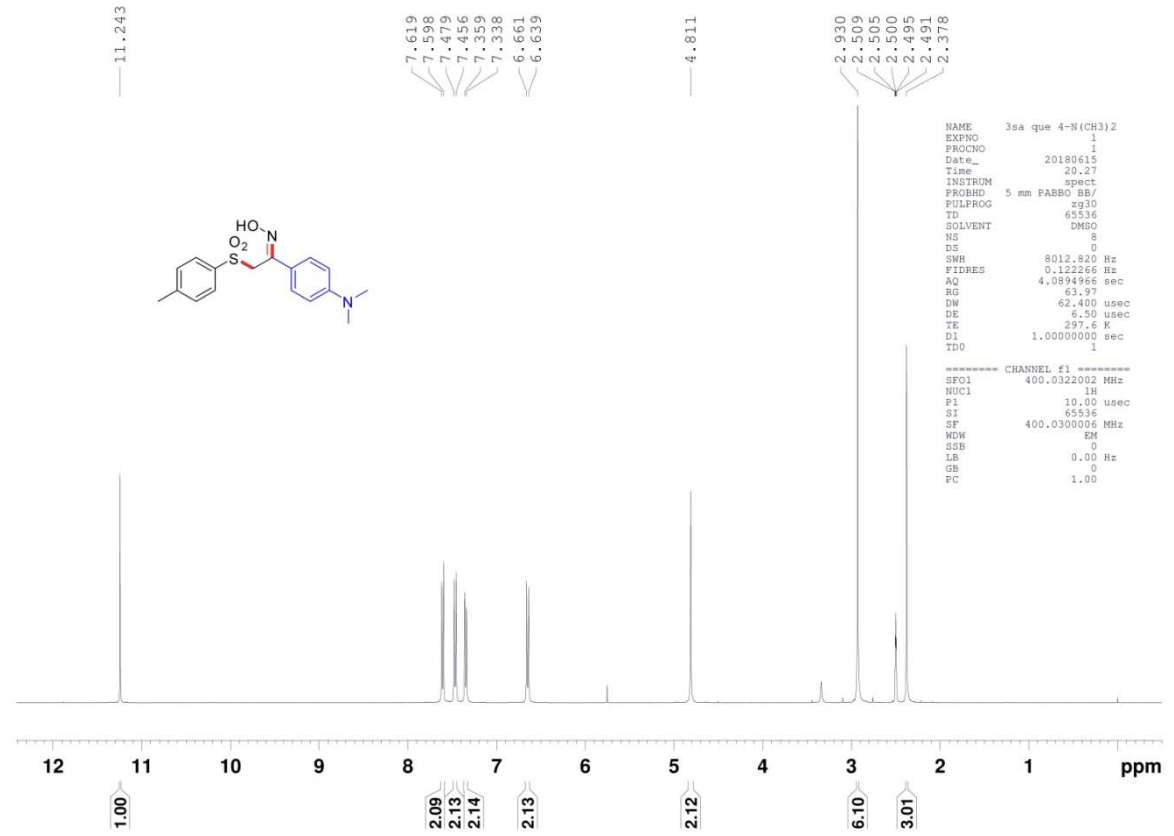


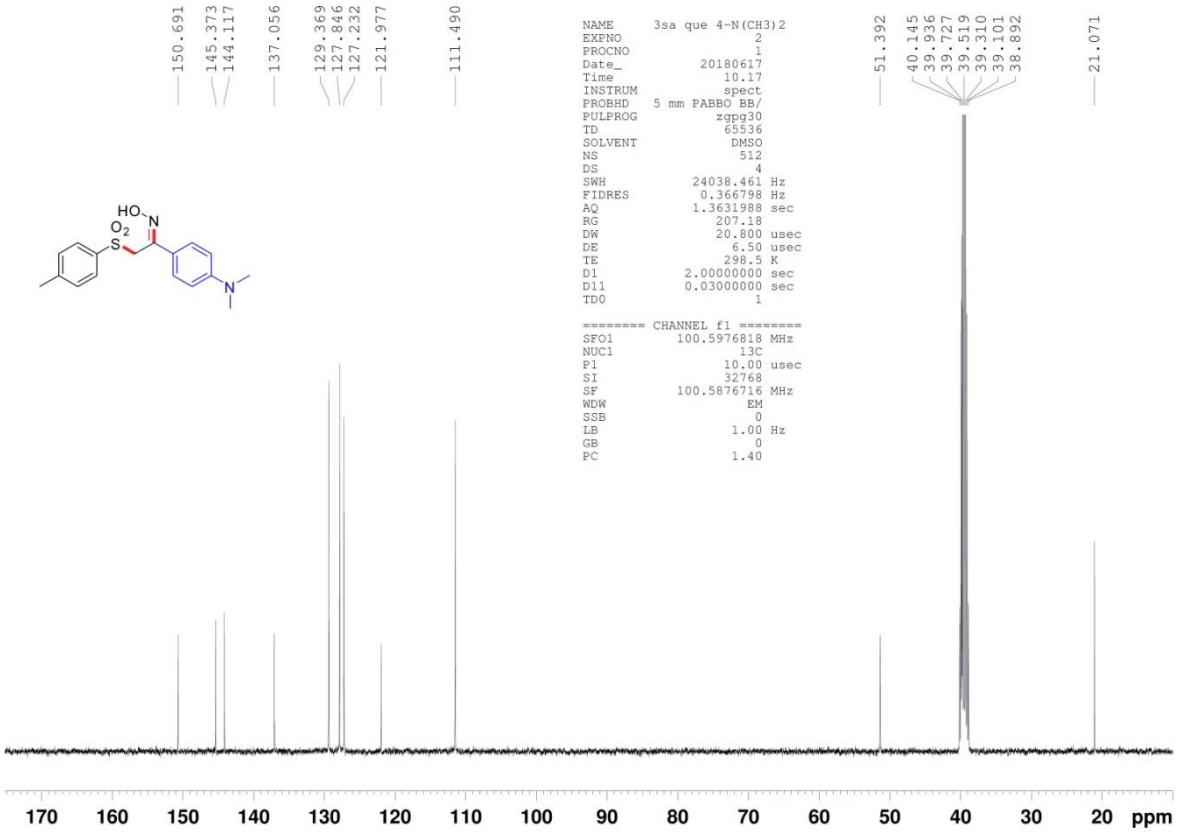
1-(3-methoxyphenyl)-2-tosylethan-1-one oxime(3na).



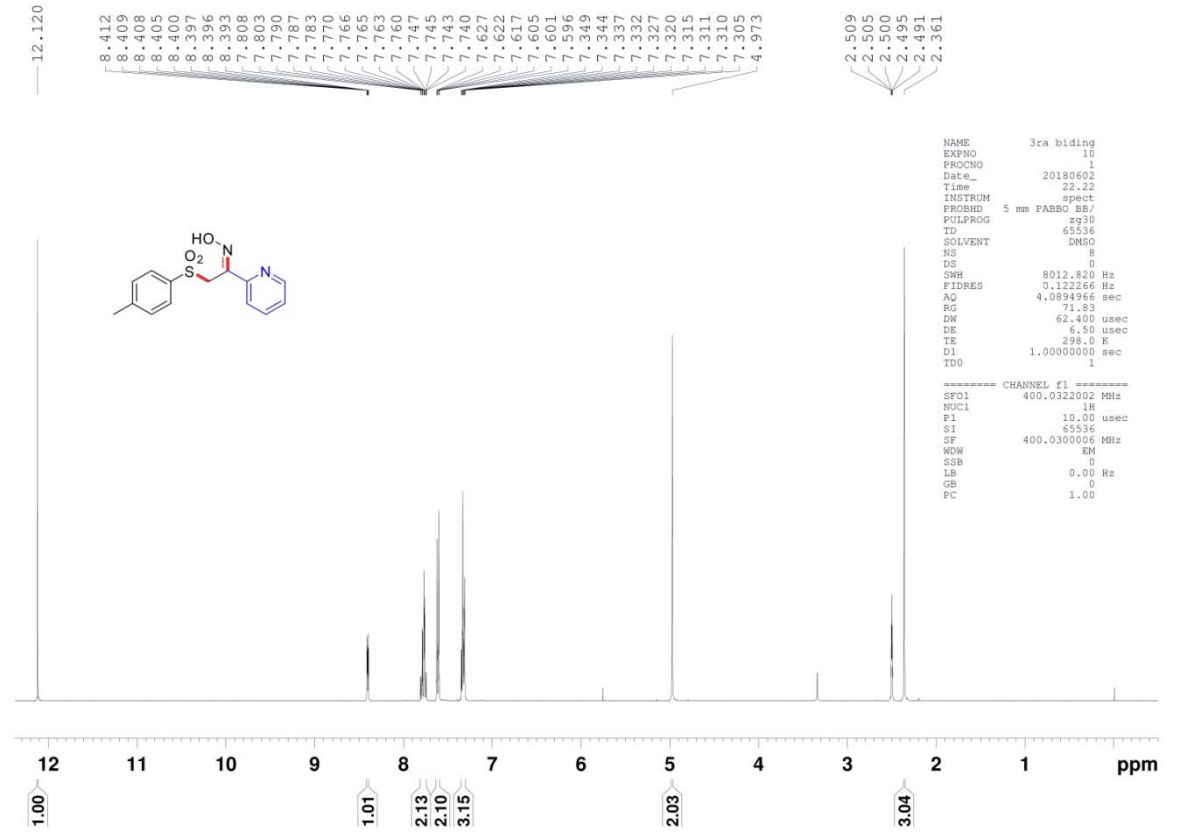


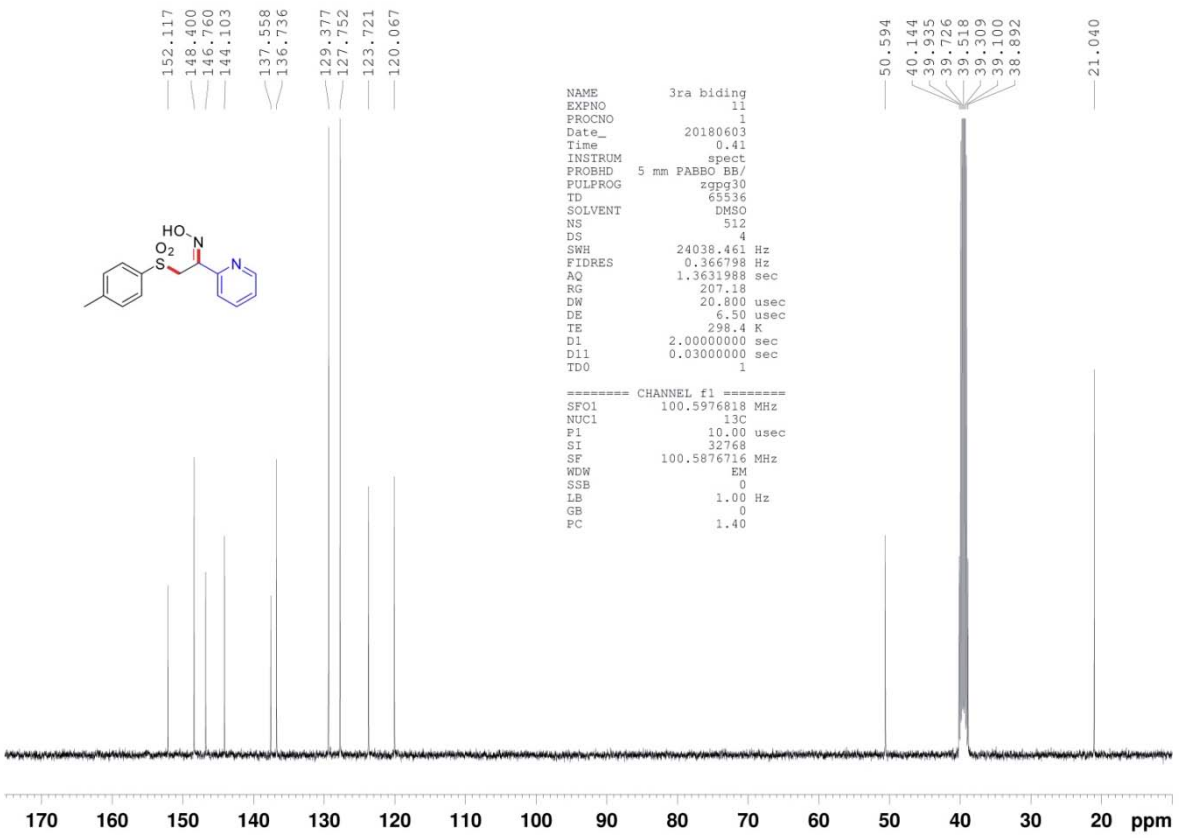
1-(4-(dimethylamino)phenyl)-2-tosylethan-1-one oxime(3oa).



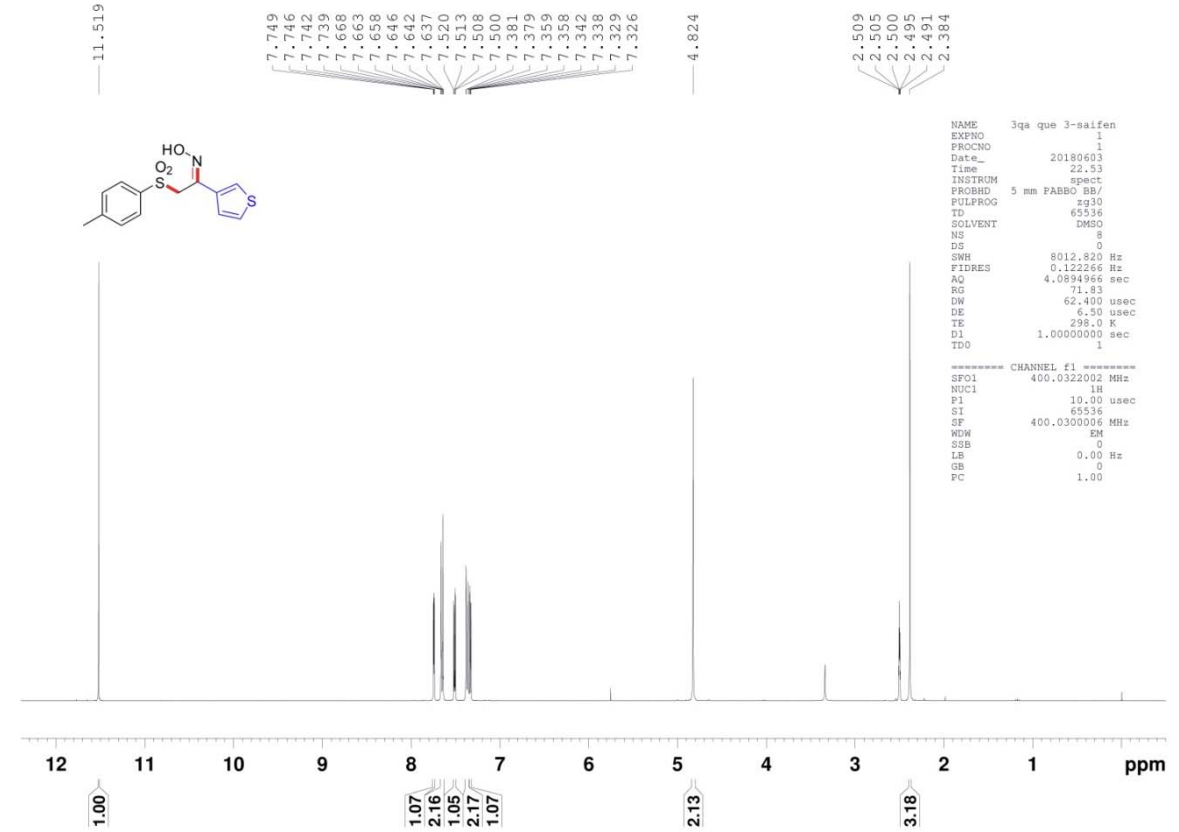


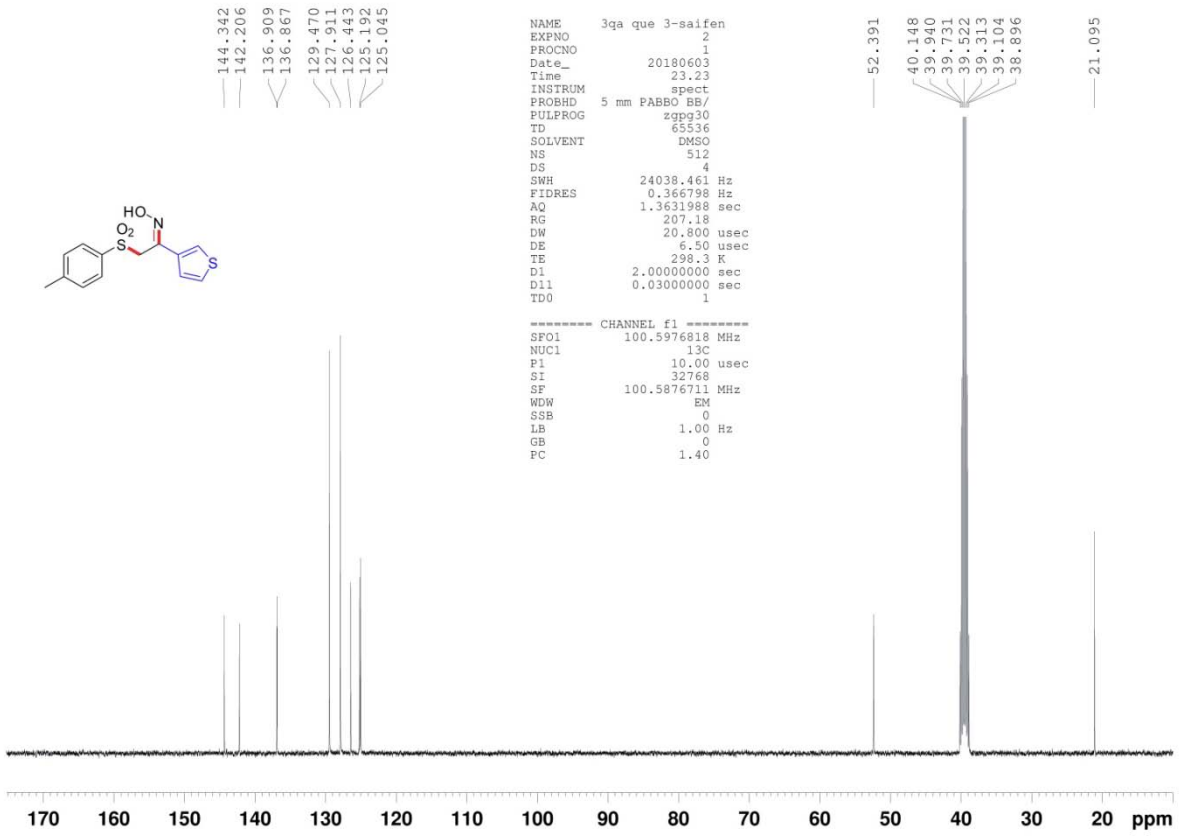
1-(pyridin-2-yl)-2-tosylethan-1-one oxime(3sa).



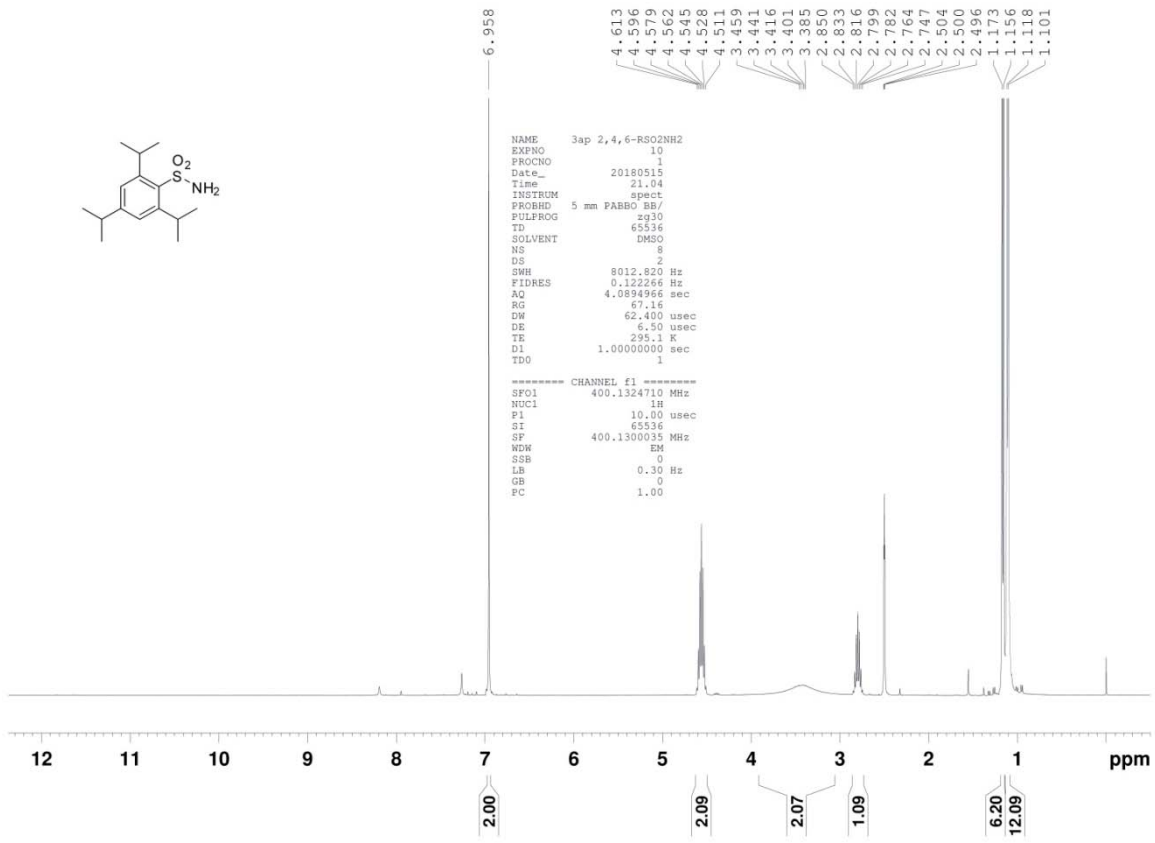


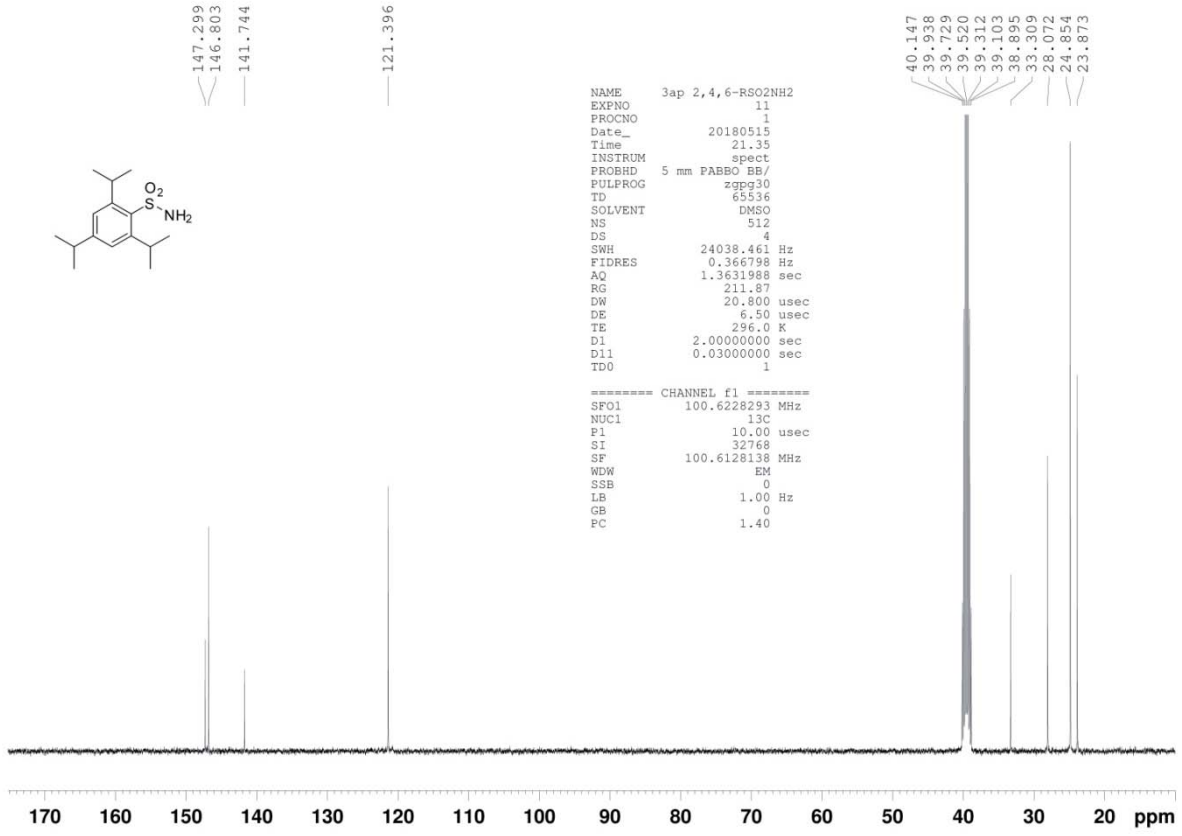
1-(thiophen-3-yl)-2-tosylethan-1-one oxime(3ta).



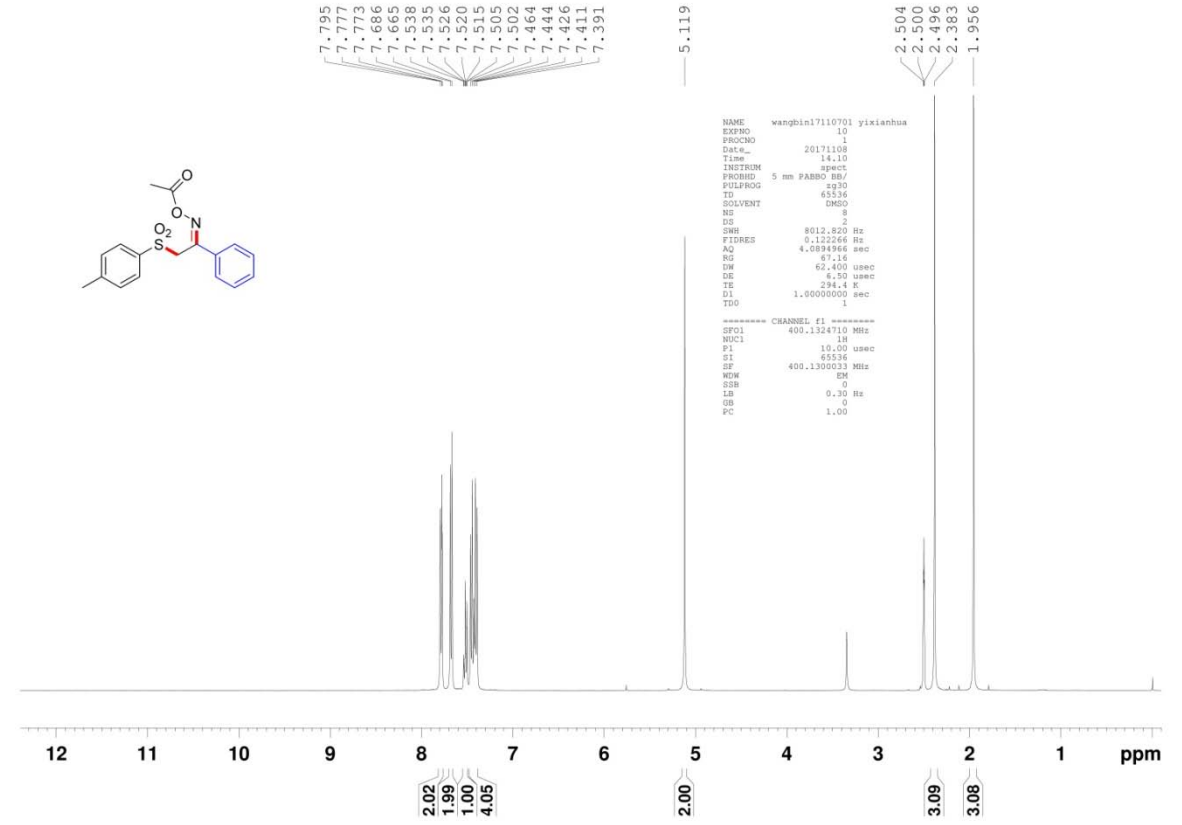


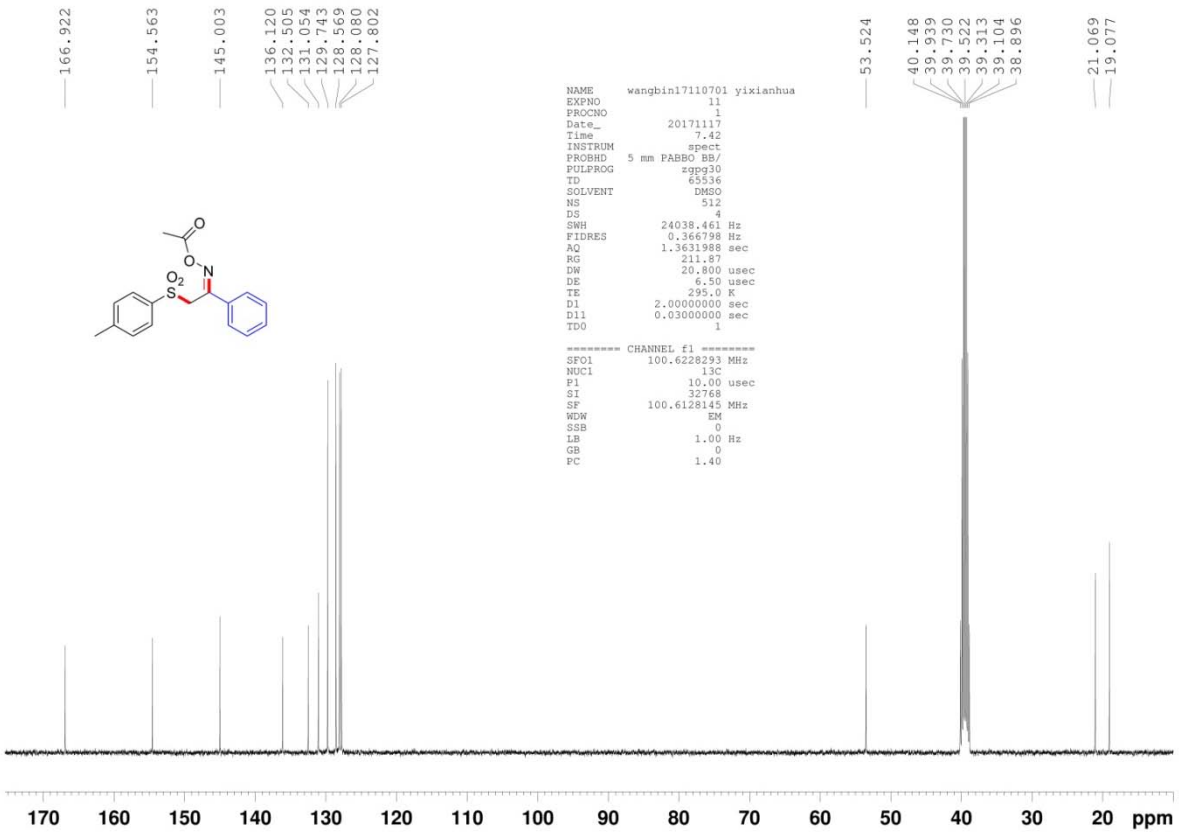
2,4,6-triisopropylbenzenesulfonamide (4d).





1-phenyl-2-tosylethan-1-one O-acetyl oxime (4i).

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FIDRES 0.366798 Hz
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DW 20.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
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NUC1 13C
P1 10.00 usec
SI 32768
SF 100.6128145 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
N
O2
S
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm