Supporting Information

The Triple Role of Rongalite in Aminosulfonylation of Aryldiazonium Tetrafluoroborates: Synthesis of N-aminosulfonamides via a Radical Coupling Reaction

Miao Wang,[a] Bo-Cheng Tang,[a] Jun-Gang Wang,[b] Jia-Chen Xiang,[a] Ao-Yu Guan,[a] Ping-Ping Huang,[a] Wu-Yinzheng Guo,[a] Yan-Dong Wu,[a] and An-Xin Wu*[,a]

[a] Key Laboratory of Pesticide & Chemical Biology, Ministry of Education, College of Chemistry, Central China Normal University, Hubei, Wuhan 430079, P. R. China

[b] School of Chemical Engineering, Guizhou Minzu University, Guiyang, Guizhou, 550025, P. R. China

E-mail: chwuax@mail.ccnu.edu.cn

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1. General.

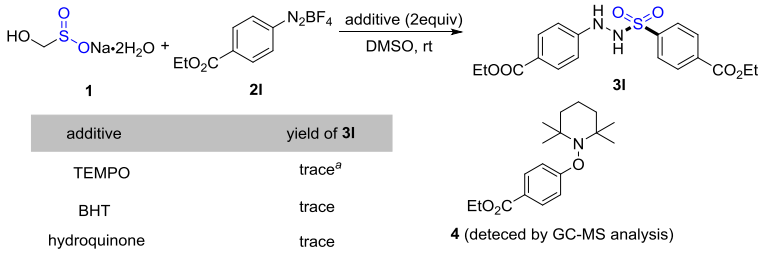
All kinds of aryldiazonium tetrafluoroborates **2a-2v** were prepared according to literature procedures[1] and other substrates and reagents were commercially available and used without further purification. TLC analysis was performed using pre-coated glass plates. Column chromatography was performed using silica gel (200–300 mesh). IR spectra were recorded on a Perkin-Elmer PE-983 infrared spectrometer as KBr pellets with absorption in cm^{-1}. ^1H spectra were recorded in DMSO-d_6 on 600 MHz NMR spectrometers and resonances (δ) are given in parts per million relative to tetramethylsilane. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quadruple), coupling constants (Hz) and integration. ^{13}C spectra were recorded in DMSO-*d6* on 150 MHz NMR spectrometers and resonances (δ) are given in ppm. HRMS were obtained on a Bruker 7-tesla FT-ICR MS equipped with an electrospray source. The X-ray crystal structure determinations of **3b** were obtained on a Bruker SMART APEX CCD system. Melting points were determined using XT-4 apparatus and not corrected.

2. General procedure for the synthesis of 3 (3a as an example).

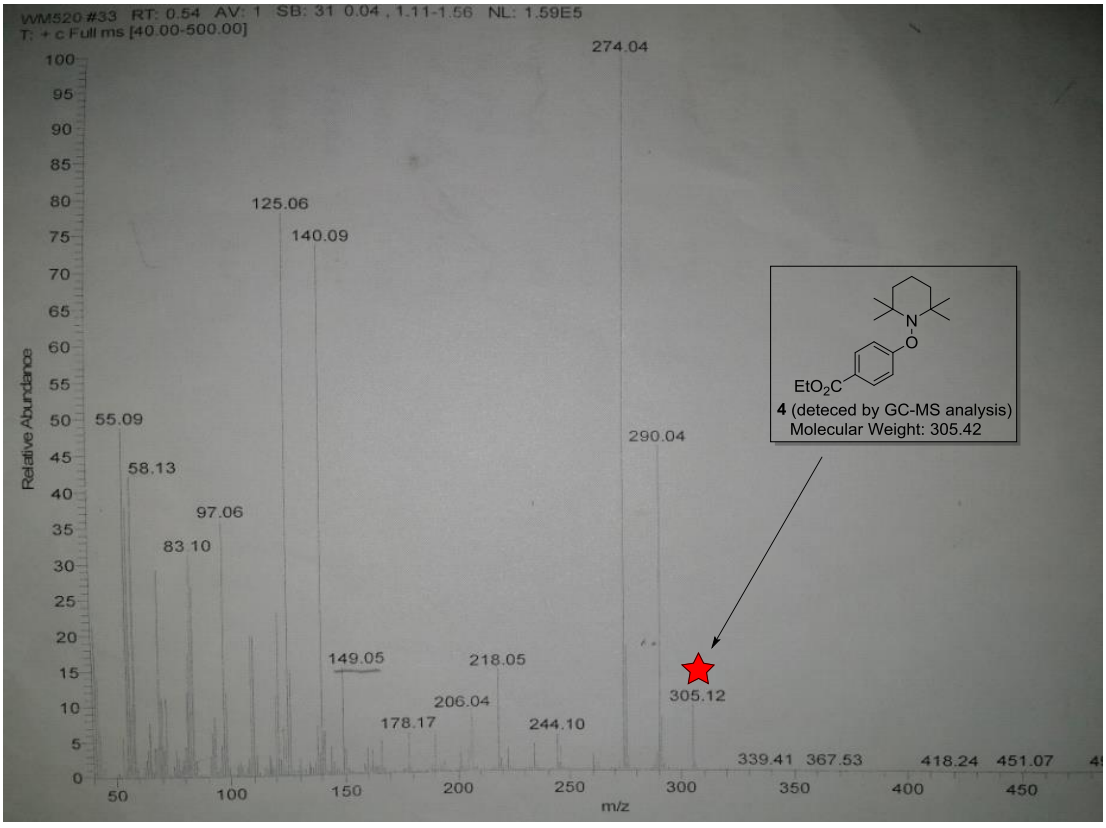
A mixture of **1** rongalite (1.2 mmol) and **2a** (0.6 mmol) in DMSO (3.0 mL) was stirred at rt for 10min in a pressure vessel. The resulting mixture was dropped into 50 mL H_2O and extracted with EtOAc 3 times (3 ×50 mL). The organic extract was dried with anhydrous Na_2SO_4, filtered and concentrated. The crude product was purified by column chromatography on silica gel (alkalization with 1% Et_3N) (eluent: petroleum ether/EtOAc = 5/1) to afford the product **3a** as light yellow oil (40.9 mg, 55%).

3. Evidence in support of the mechanism.

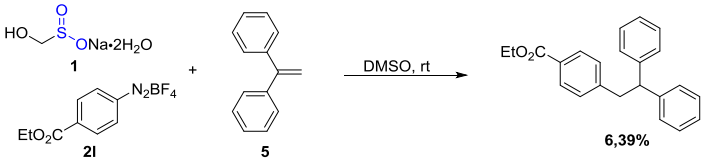
(1) We performed a series of radical trapping experiments, and such as TEMPO, BHT, and hydroquinone, no desired product (**3l**) was observed. And in the reaction containing TEMPO, a TEMPO-PhCO$_2$Et product **4** was detected by GC−MS.



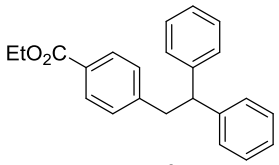
additive	yield of **3l**
TEMPO	trace[a]
BHT	trace
hydroquinone	trace



(2) The further radical trapping experiment using 1,1-diphenylethylene **(5)** as a trapping reagent and ethane-1,1,2-triyltribenzene **(6)** was obtained.



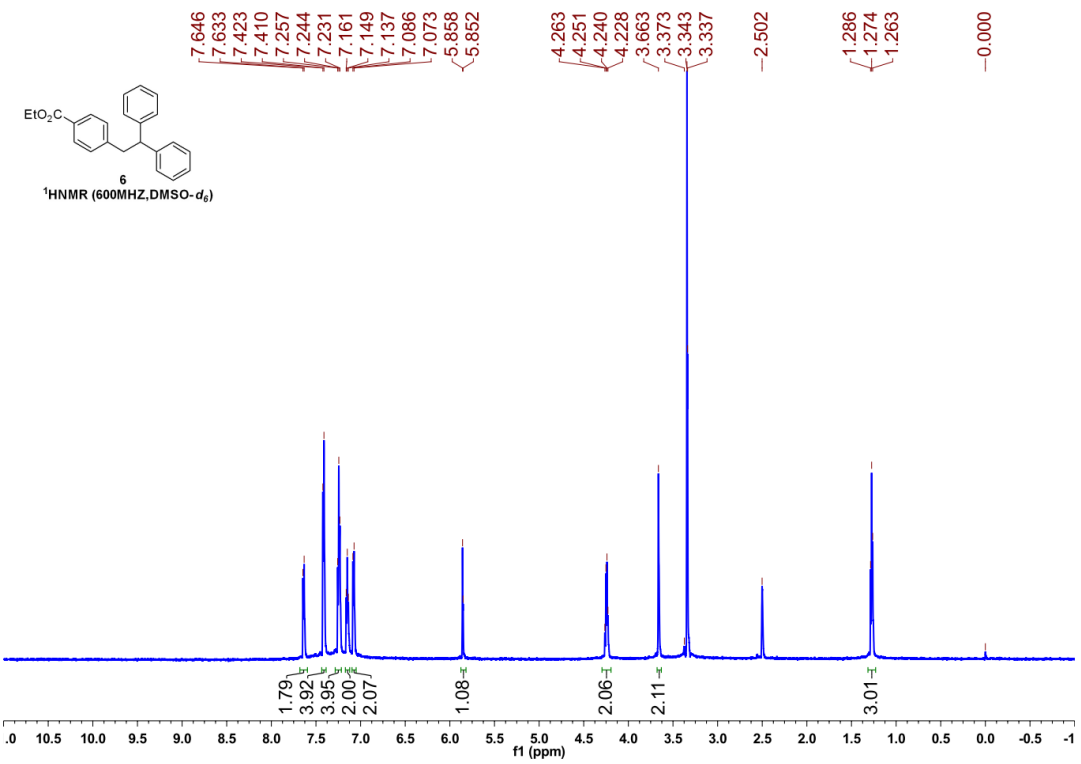
And the characterization data of **6** are listed below:

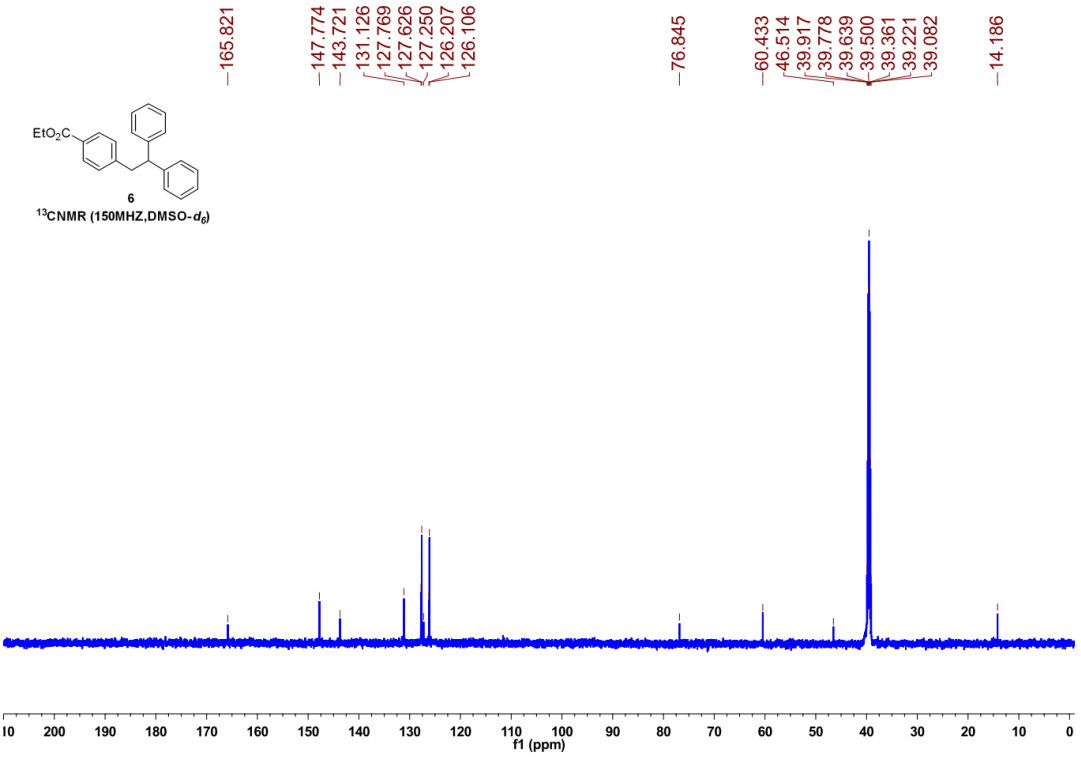


6

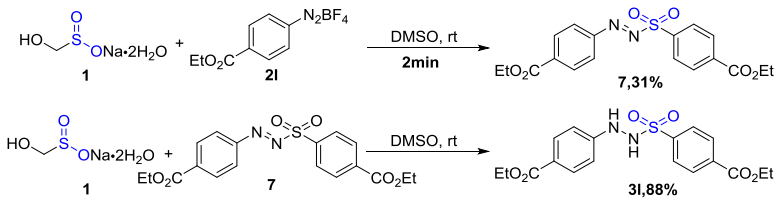
Ethyl 4-(2,2-diphenylethyl)benzoate:

IR (KBr): 2925, 2361, 1687, 1490, 1416, 1281, 1108, 1026, 1003, 759, 697 cm^{-1}**;** ^{1}H NMR (600 MHz, DMSO-*d6*) δ = 7.64 (d, *J* = 7.8 Hz, 2H), 7.42 (d, *J* = 7.8 Hz, 4H), 7.24 (t, *J* = 7.8 Hz, 4H), 7.15 (t, *J* = 7.2 Hz, 2H), 7.08 (d, *J* = 7.8 Hz, 2H), 5.85 (d, *J* = 3.6 Hz, 1H), 4.26-4.23(m, 2H), 3.66 (s, 2H), 1.27 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-*d6*) δ= 165.8, 147.8, 143.7, 131.1, 127.8, 127.6, 127.3, 126.2, 126.1, 76.8, 60.4, 16.5, 14.2; HRMS (ESI) m/z calcd for $C_{23}H_{21}O_2^+$ $(M+H)^+$ 329.1536, found 329.1536.

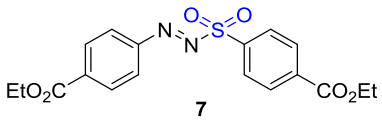




(3) The reaction of rongalite **(1)** and **2l** under the standard conditions was stopped at 2 min, the corresponding sulfonyl diazo **(7)** was obtained in 31% yield, which was then reduced to corresponding sulfonyl hydrazide **(3l)** in 88% yield by rongalite.

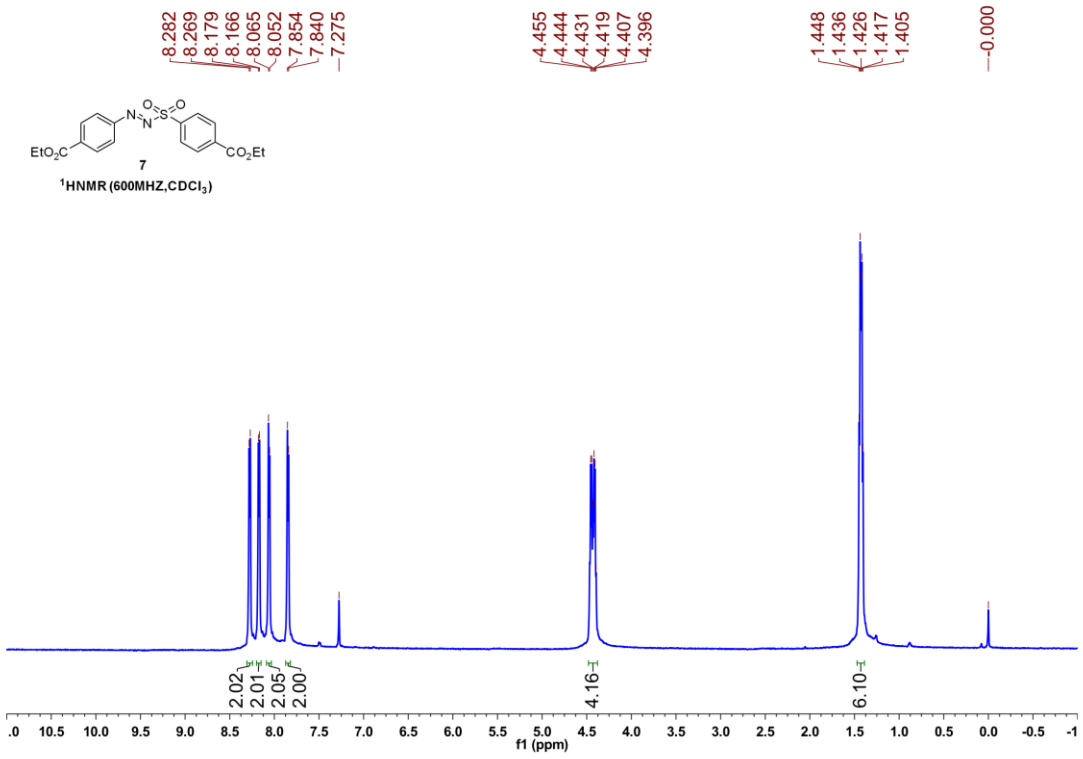


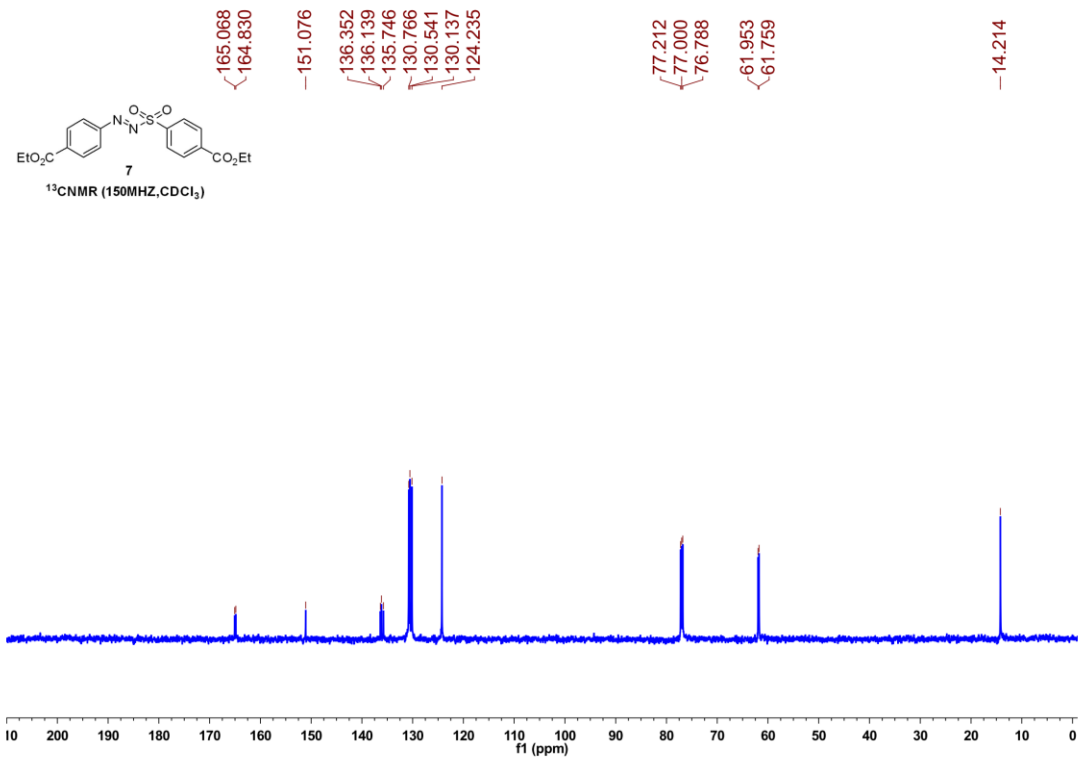
And the characterization data of **7** are listed below:



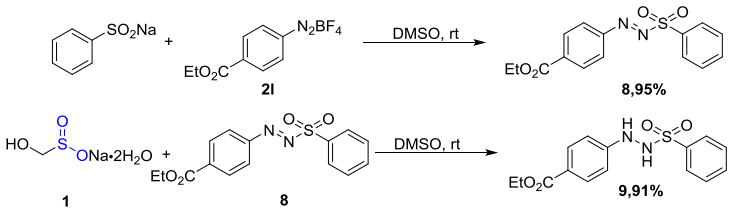
ethyl (E)-4-(((4-(ethoxycarbonyl)phenyl)diazenyl)sulfonyl)benzoate:

IR (KBr): 1721, 1626, 1360, 1271, 1166, 1101, 1012, 772, 687, 645, 597 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ = 8.28 (d, J = 7.8 Hz, 2H), 8.17 (d, J = 7.8 Hz, 2H), 8.06 (d, J = 7.8 Hz, 2H), 7.85 (d, J = 8.4 Hz, 2H), 4.47 – 4.40 (m, 4H), 1.45 – 1.41 (m, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ= 165.1, 164.8, 151.1, 136.4, 136.1, 135.8, 130.8, 130.5, 130.1, 124.2, 62.0, 61.8, 14.2; HRMS (ESI) m/z calcd for $C_{18}H_{19}N_2O_6S^+$ $(M+H)^+$ 391.0958, found 391.0953.

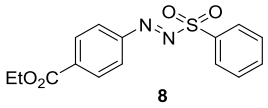

8.282
8.269
8.179
8.166
8.065
8.052
7.854
7.840
7.275
4.455
4.444
4.431
4.419
4.407
4.396
1.448
1.436
1.426
1.417
1.405
0.000
EtO2C
CO2Et
7
1HNMR (600MHZ,CDCl3)
2.02
2.01
2.05
2.00
4.16
6.10
f1 (ppm)


165.068
164.830
151.076
136.352
136.139
135.746
130.766
130.541
130.137
124.235
77.212
77.000
76.788
61.953
61.759
14.214
EtO2C
CO2Et
7
13CNMR (150MHZ,CDCl3)
f1 (ppm)

(4) The reaction of sodium benzenesulfinate and **2l** gave corresponding sulfonyl diazo **8** in 95% yield, which was then reduced to corresponding sulfonyl hydrazide **9** in 91% yield by rongalite.



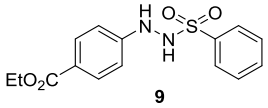
And the characterization data of **8** and **9** are listed below:



8

ethyl (E)-4-((phenylsulfonyl)diazenyl)benzoate:

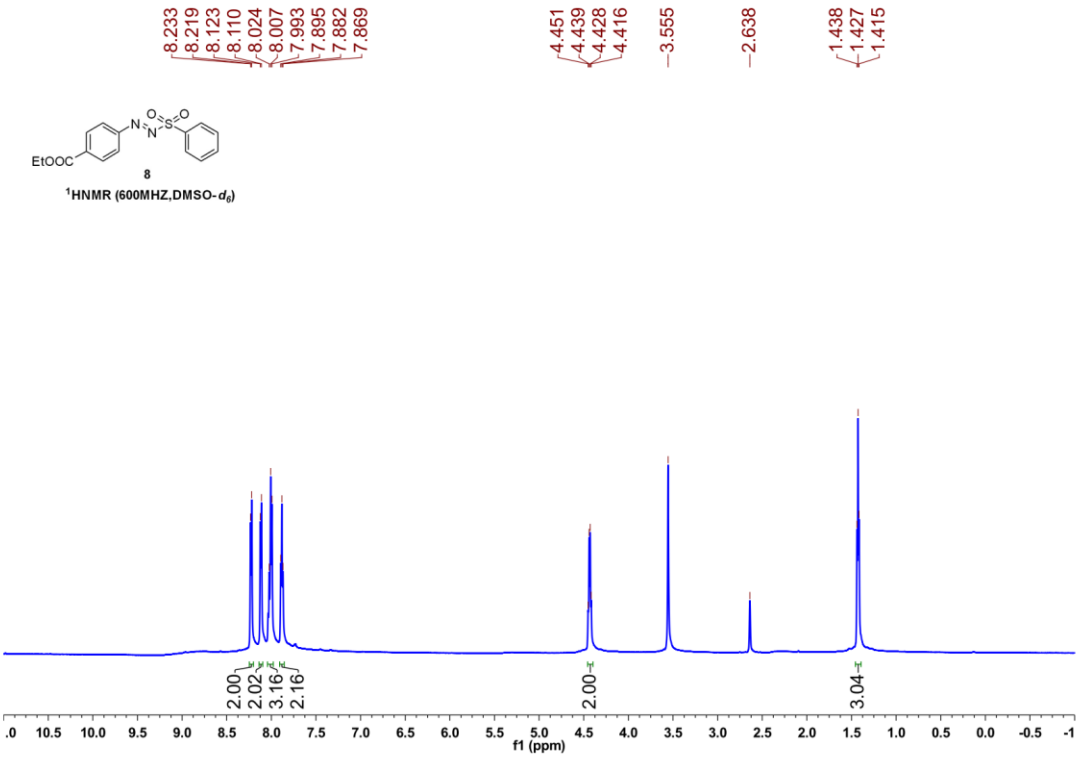
IR (KBr): 2978, 2361, 1721, 1604, 1446, 1355, 1272, 1165, 1012, 768, 683, 548 cm^{-1}**;** ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 8.23 (d, *J* = 8.4 Hz, 2H), 8.12 (d, *J* = 7.8 Hz, 2H), 8.04 – 7.99 (m, 3H), 7.88 (t, *J* = 7.8 Hz, 2H), 4.45 – 4.42 (m, 2H), 1.43 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 164.5, 150.7, 135.7, 135.1, 131.6, 130.8, 130.2, 129.9, 124.2, 61.5, 14.0; HRMS (ESI) m/z calcd for $C_{15}H_{15}N_2O_4S^+$ $(M+H)^+$ 319.0747, found 319.0745.



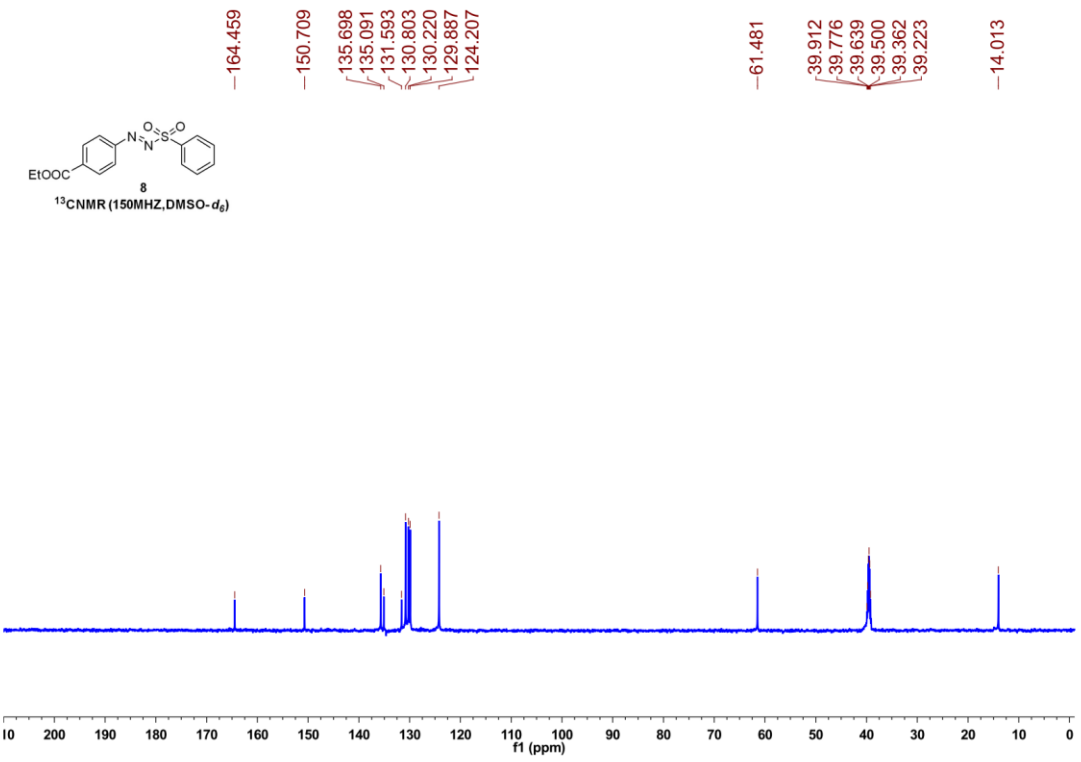
9

ethyl 4-(2-(phenylsulfonyl)hydrazinyl)benzoate:

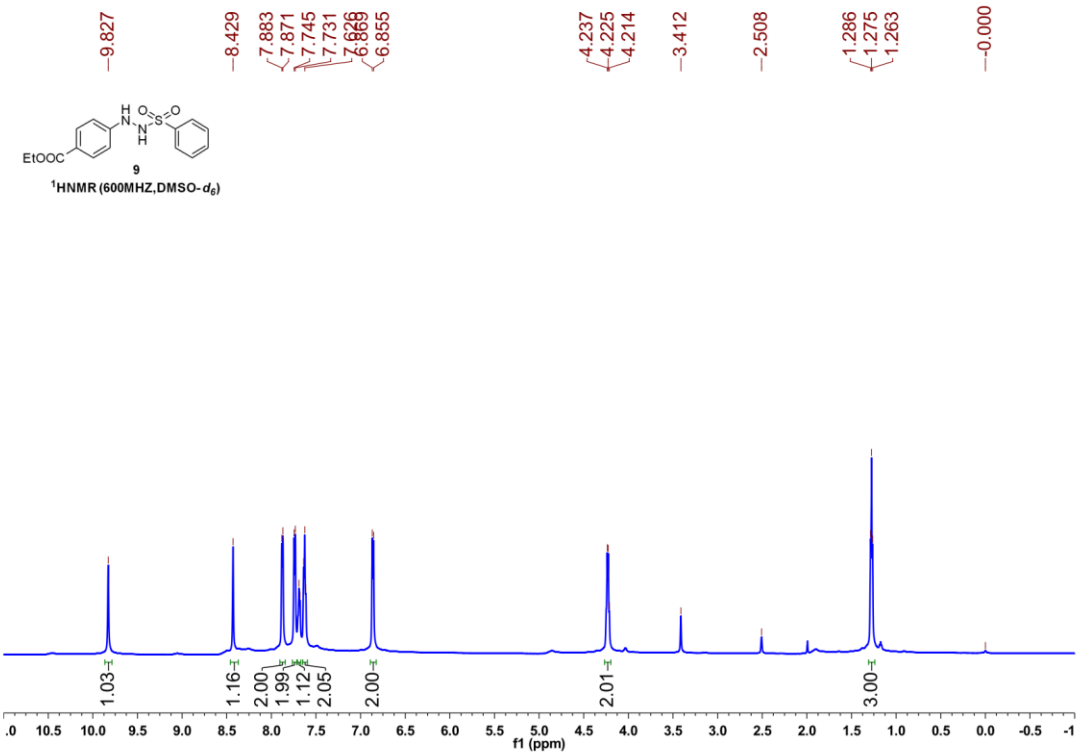
IR (KBr): 2361, 1683, 1609, 1340, 1270, 1162, 1021, 874, 724, 614, 553 cm^{-1}**;** ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.83 (s, 1H), 8.43 (s, 1H), 7.88 (d, *J* = 7.2 Hz, 2H), 7.74 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 7.2 Hz, 1H), 7.63 (t, *J* = 7.2 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 4.24 – 4.21 (m, 2H), 1.27 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 165.7, 152.9, 138.8, 133.1, 130.5, 129.3, 127.6, 119.7, 111.6, 59.9, 14.3; HRMS (ESI) m/z calcd for $C_{15}H_{17}N_2O_4S^+$ $(M+H)^+$ 321.0904, found 321.0901.

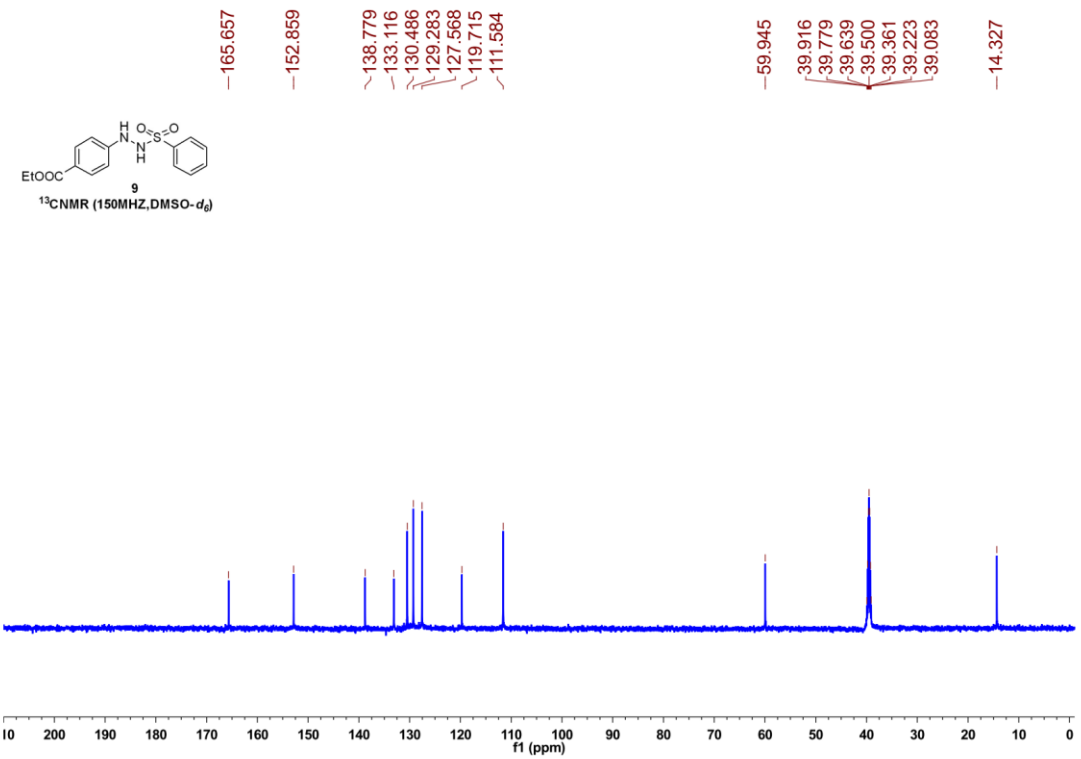


^{1}HNMR (600MHZ,DMSO-d_6)

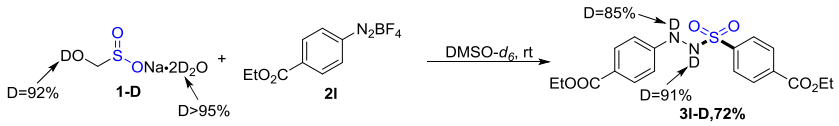


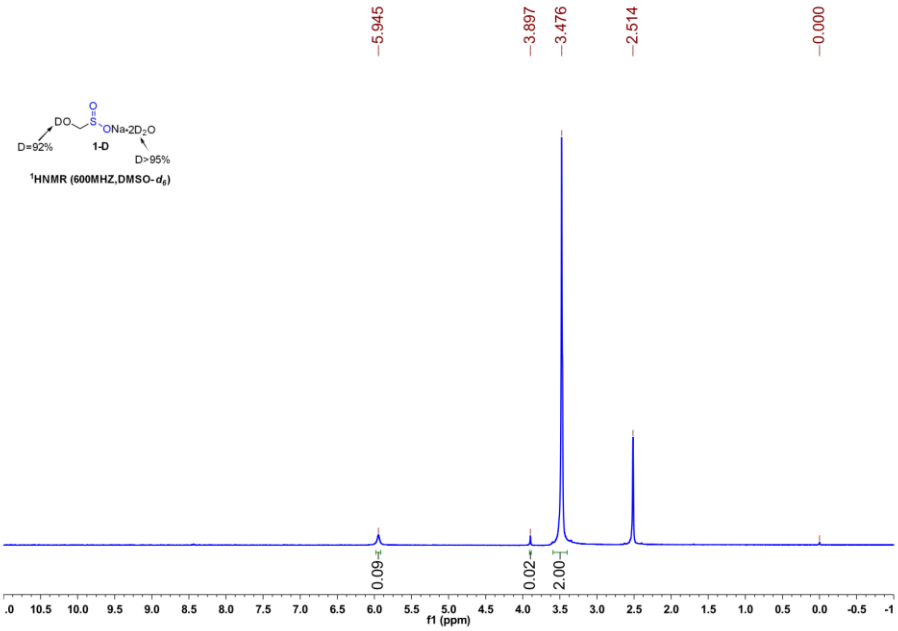
^{13}CNMR (150MHZ,DMSO-d_6)

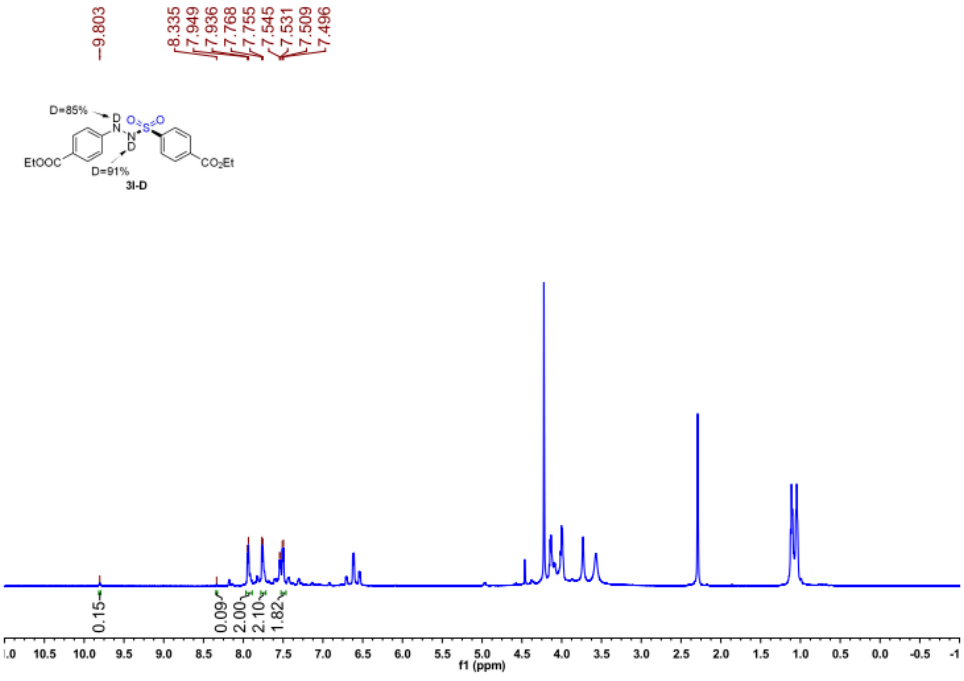

9.827
8.429
7.883
7.871
7.745
7.731
6.869
6.855
4.237
4.225
4.214
3.412
2.508
1.286
1.275
1.263
0.000
EtOOC
9
1HNMR (600MHZ,DMSO-d6)
1.03
1.16
2.00
1.99
1.12
2.05
2.00
2.01
3.00
f1 (ppm)


165.657
152.859
138.779
133.116
130.486
129.283
127.568
119.715
111.584
59.945
39.916
39.779
39.639
39.500
39.361
39.223
39.083
14.327
EtOOC
9
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

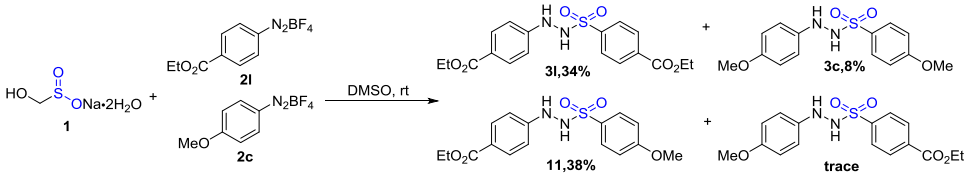
(5) To confirm the source of NH in the product of **3**, we performed the reaction of deuterated rongalite (**1-D**) and **2l** in the in DMSO-d_6 and the deuterated product **3l-D** generated in 72% yield with 85% and 91% deuteration of the two N–H groups, respectively. (Since the NH in **3** was too active to exchange with water, we performed the ^{1}H NMR within reaction solution).



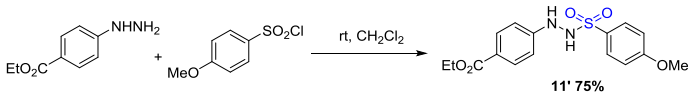




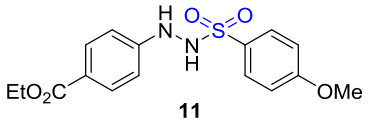
(6) We performed cross experiment of 4-(ethoxycarbonyl)benzenediazonium tetrafluoroborate **(2l)** and 4-methoxy-benzenediazonium tetrafluoroborate **(2c)**, and only three products was obtained.



It is not easy to determine the structure of the cross product **11**, so we try to synthesis the same compound in other way which was list below[2]. The characterization data of **11** are the same with **11'**.

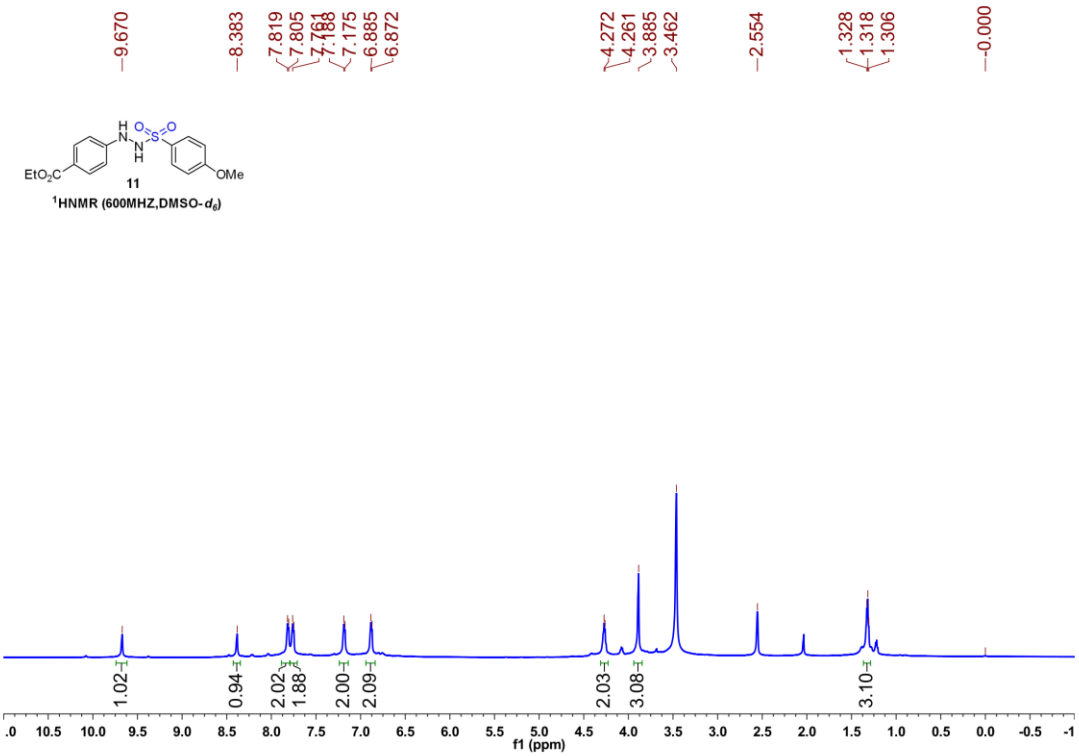


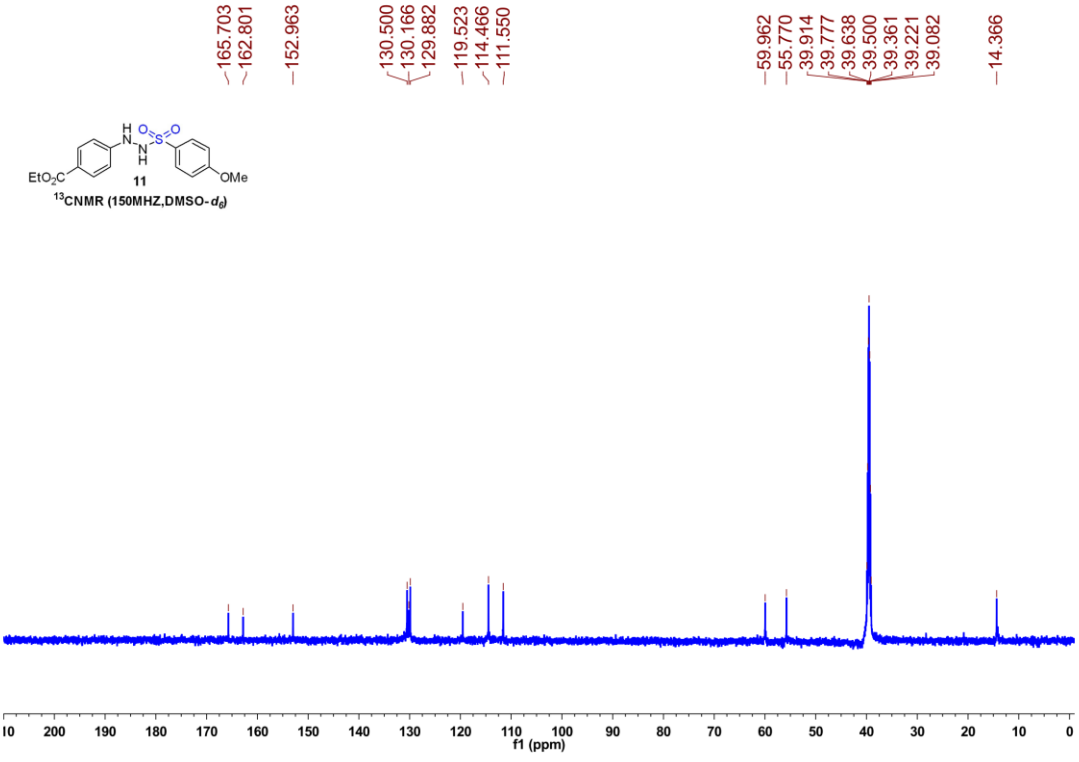
And the characterization data of cross product **11** are listed below:

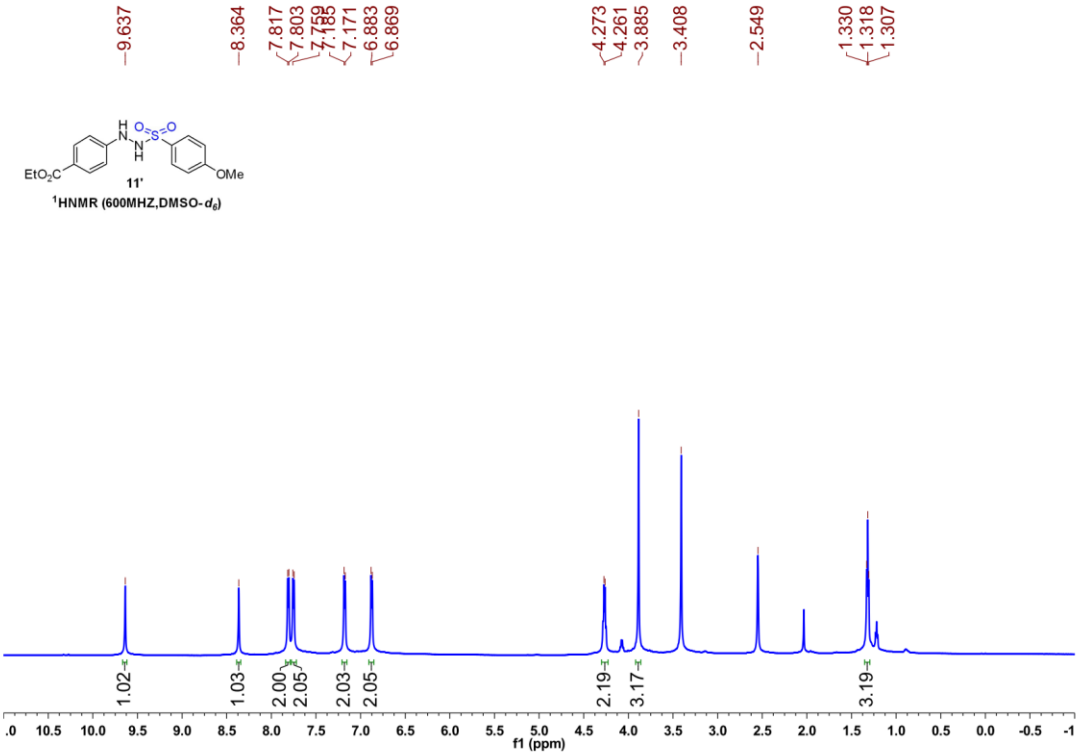


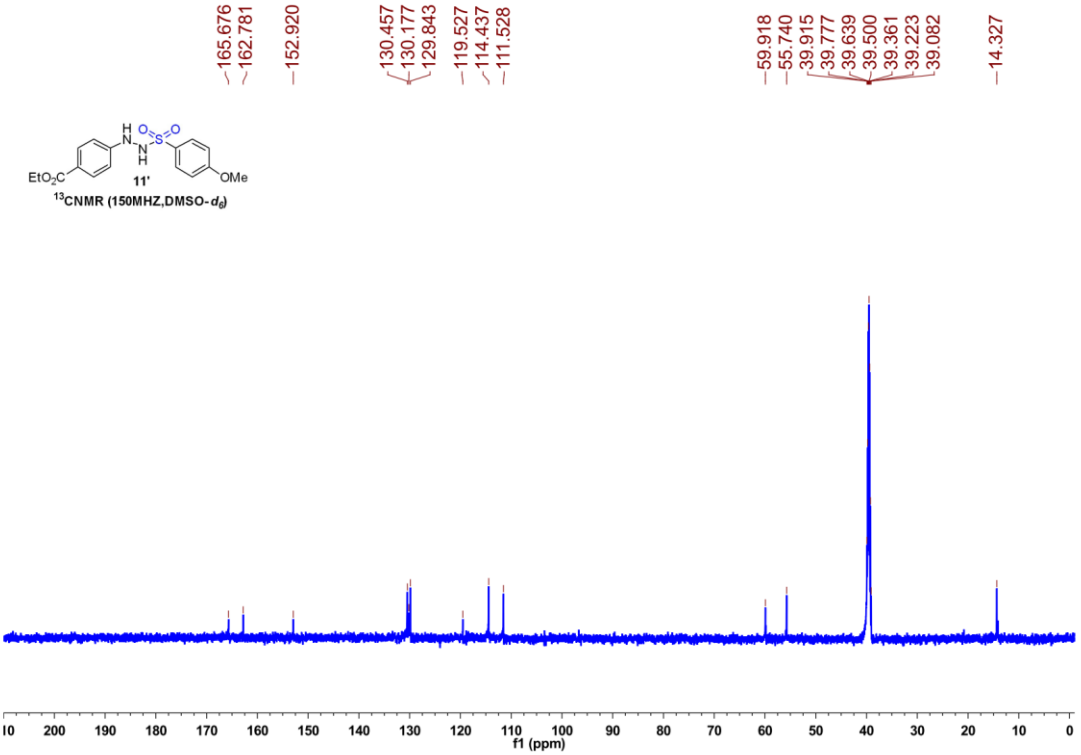
ethyl 4-(2-((4-methoxyphenyl)sulfonyl)hydrazinyl)benzoate:

IR (KBr): 2255, 1649, 1319, 1264, 1049, 1026, 1002, 826, 765, 629, 565 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d}$6) δ = 9.67 (s, 1H), 8.38 (s, 1H), 7.81 (d, J = 8.4 Hz, 2H), 7.75 (d, J = 7.8 Hz, 2H), 7.18 (d, J = 7.8 Hz, 2H), 6.88 (d, J = 7.8 Hz, 2H), 4.28 – 4.26 (m, 2H), 3.89 (s, 3H), 1.33 – 1.31 (m, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 165.7, 162.8, 153.0, 130.5, 130.2, 129.9, 119.5, 114.5, 111.6, 60.0, 55.8, 14.4; HRMS (ESI) m/z calcd for $C_{16}H_{19}N_2O_5S^+$ $(M+H)^+$ 351.1009, found 351.1006.


9.670
8.383
7.819
7.805
7.761
7.175
6.885
6.872
4.272
4.261
3.885
3.462
2.554
1.328
1.318
1.306
0.000
EtO2C
OMe
11
1HNMR (600MHZ,DMSO-d6)
1.02
0.94
2.02
1.88
2.00
2.09
2.03
3.08
3.10
f1 (ppm)


165.703
162.801
152.963
130.500
130.166
129.882
119.523
114.466
111.550
59.962
55.770
39.914
39.777
39.638
39.500
39.361
39.221
39.082
14.366
EtO2C
OMe
11
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)



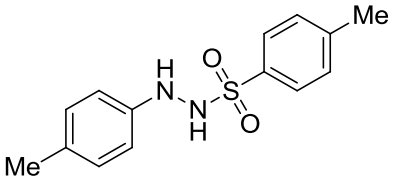


References:

(1) Wu, J.; Gu,Y.; Leng, X.; Shen, Q. *Angew. Chem., Int. Ed.*, 2015, **54**, 7648.

(2) M. Cheng, M. Hung, J. Song, S. Lin, F. Liao, M. Wu, W. Hsiao, C. Hsieh, J. Wu,Y. Chao, C. Shih, S. Wu, S. Ueng, *Bioorg. Med. Chem. Lett.*, 2014, **24**, 3403.

4. Crystallographic data of 3b.



3b

4-methyl-N'-(p-tolyl)benzenesulfonohydrazide: (3b)

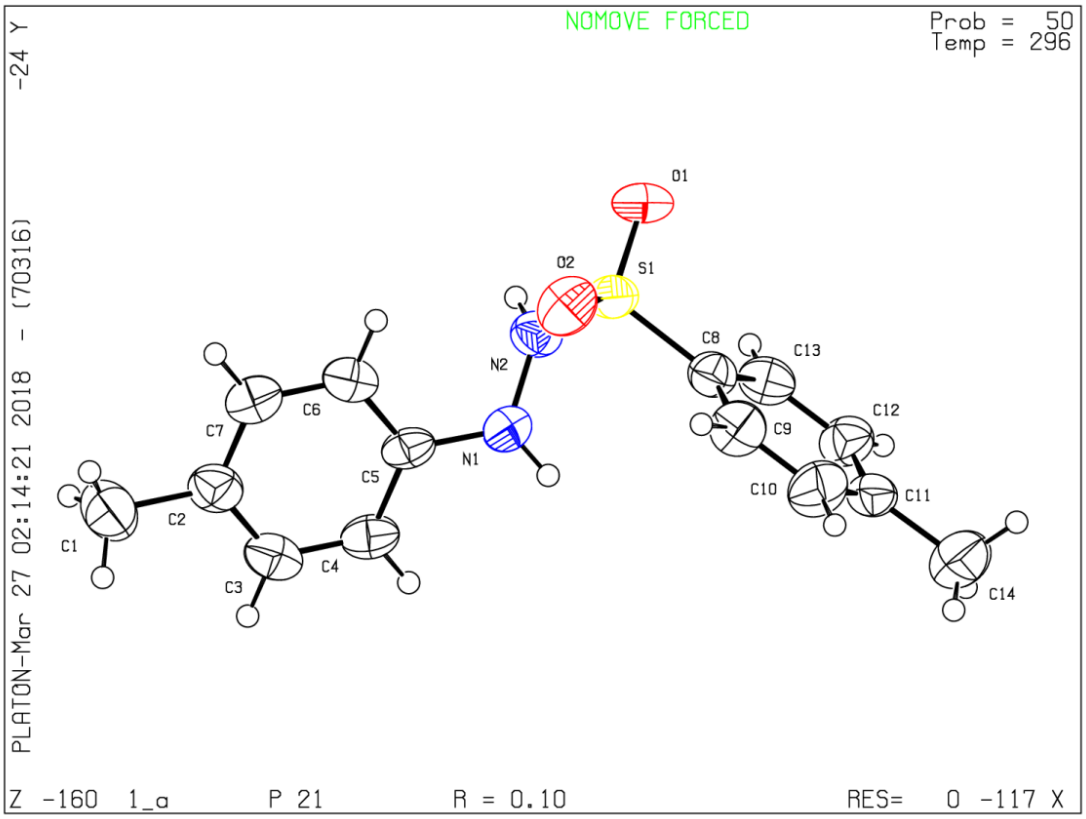


Figure S1. X-ray crystal structure of 3b.

Table S1. Crystal data and structure refinement for compound 3b (CCDC: 1839767)

Bond precision: C-C = 0.0088 A Wavelength=0.71073

Cell: a=9.955(3) b=5.5629(19) c=12.732(4)
alpha=90 beta=92.902(9) gamma=90
Temperature: 296 K

	Calculated	Reported
Volume	704.2(4)	704.2(4)
Space group	P 21	P 21
Hall group	P 2yb	P 2yb
Moiety formula	C14 H16 N2 O2 S	?
Sum formula	C14 H16 N2 O2 S	C14 H16 N2 O2 S
Mr	276.35	276.35
Dx,g cm-3	1.303	1.303
Z	2	2
Mu (mm-1)	0.229	0.229
F000	292.0	292.0
F000’	292.36	
h,k,lmax	11,6,15	11,6,15
Nref	2477[1378]	1842
Tmin,Tmax	0.951,0.960	0.864,0.864
Tmin’	0.951	

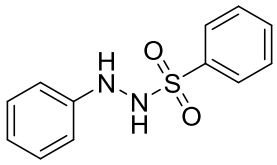
Correction method= # Reported T Limits: Tmin=0.864 Tmax=0.864
AbsCorr = MULTI-SCAN

Data completeness= 1.34/0.74 Theta(max)= 24.994

R(reflections)= 0.0975(1580) wR2(reflections)= 0.2910(1842)

S = 1.243 Npar= 151

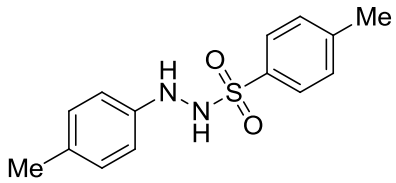
5. Characterization data for target compound.



3a

N'-phenylbenzenesulfonohydrazide:

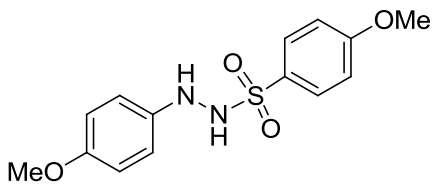
Yield: 55% (40.9 mg); light yellow oil; IR (KBr): 3266, 1640, 1604, 1500, 1329, 1158, 732, 687, 517 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.59 (s, 1H), 7.86 (d, *J* = 7.2Hz, 2H), 7.67 (t, *J* = 7.2 Hz, 2H), 7.61 (t, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 7.8 Hz, 2H), 6.81 (d, *J* = 8.4 Hz, 2H), 6.70 (t, *J* = 7.2 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ=148.7, 139.2, 132.9,129.2, 128.5, 127.5, 119.1, 112.8; HRMS (ESI) m/z calcd for $C_{12}H_{13}N_2O_2S^+$ $(M+H)^+$ 249.0692, found 249.0689.



3b

4-methyl-N'-(p-tolyl)benzenesulfonohydrazide:

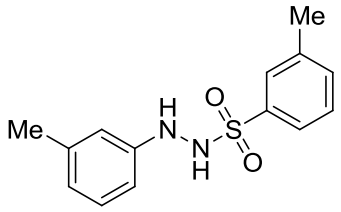
Yield: 52% (42.9 mg); gray solid; mp: 128.6-131.0 ℃; IR (KBr): 1642, 1324, 1159, 1091, 1025, 998, 816, 659, 556 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.40 (s, 1H), 7.71 (d, *J* = 7.8 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 3H), 6.89 (d, *J* = 7.8 Hz, 2H), 6.69 (d, *J* = 8.4 Hz, 2H), 2.39 (s, 3H), 2.15 (s, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 146.4, 143.1, 136.3, 129.5, 128.9, 127.5, 113.0, 21.0, 20.2; HRMS (ESI) m/z calcd for $C_{14}H_{17}N_2O_2S^+$ $(M+H)^+$ 277.1005, found 277.1006.



3c

4-methoxy-N'-(4-methoxyphenyl)benzenesulfonohydrazide:

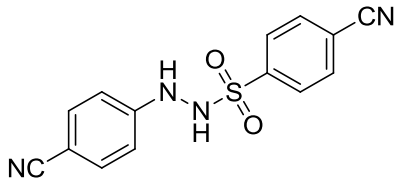
Yield: 47% (43.4 mg); gray solid; mp: 110.1-113.2°C; IR (KBr): 3399, 2921, 1644, 1503, 1403, 1260, 1156, 1087, 831, 803, 559 cm^{-1}; ^1H NMR (600 MHz, DMSO-$_{d6}$) δ =9.29 (s, 1H), 7.76 – 7.74(m, 2H), 7.23 (s, 1H), 7.12 – 7.11 (m, 2H), 6.75 – 6.74 (m, 2H), 6.71 – 6.69 (m, 2H), 3.84 (s, 3H), 3.64 (s, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 152.8, 142.4, 130.8, 129.7, 114.2, 113.9, 109.6, 55.7, 55.2; HRMS (ESI) m/z calcd for $C_{14}H_{17}N_2O_4S^+$ $(M+H)^+$ 309.0904, found 309.0905.



3d

3-methyl-N'-(m-tolyl)benzenesulfonohydrazide:

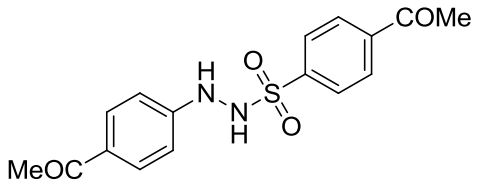
Yield: 51% (42.2 mg); light yellow solid; mp: 96.8-99.3 °C; IR (KBr): 2253, 1648, 1610, 1333, 1158, 1050, 1028, 1004, 824, 764, 620, 589 cm^{-1}; ^1H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.50 (s, 1H), 7.64 (d, *J* = 12.6 Hz, 2H), 7.55 (s, 1H), 7.49 – 7.47 (m, 2H), 6.96 (t, *J* = 7.8 Hz, 1H), 6.60 (s, 2H), 6.51 (d, *J* = 7.2 Hz, 1H), 2.38 (s, 3H), 2.15 (s, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 148.6, 139.2, 138.8, 137.6, 133.4, 129.0, 128.4, 127.7, 124.7, 119.8, 113.3, 110.2, 21.3, 20.9; HRMS (ESI) m/z calcd for $C_{14}H_{17}N_2O_2S^+$ $(M+H)^+$ 277.1005, found 277.1003.



3e

4-cyano-N'-(4-cyanophenyl)benzenesulfonohydrazide:

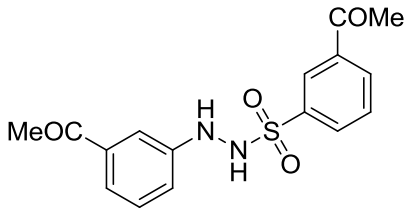
Yield: 81% (72.4 mg); gray solid; mp:187.1-190.5 °C; IR (KBr): 2219, 1640, 1609, 1164, 1025, 1001, 828, 667, 568 cm^{-1}; ^1H NMR (600 MHz, DMSO-$_{d6}$) δ = 10.17 (s, 1H), 8.60 (s, 1H), 8.14 (d, *J* = 7.2 Hz, 2H), 8.00 (d, *J* = 7.8 Hz, 2H), 7.55 (d, *J* = 7.8 Hz, 2H), 6.88 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 152.2, 142.6, 133.5, 133.4, 128.4, 119.9, 117.7, 115.6, 112.4, 100.0; HRMS (ESI) m/z calcd for $C_{14}H_{11}N_4O_2S^+$ $(M+H)^+$ 299.0597, found 299.0598.



3f

4-acetyl-N'-(4-acetylphenyl)benzenesulfonohydrazide:

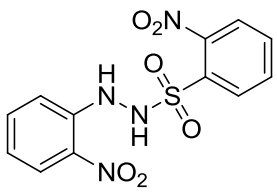
Yield: 80% (79.6 mg); gray solid; mp: 180.4-183.0 °C; IR (KBr): 3334, 1685, 1643, 1605, 1163, 831, 636, 594 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 10.02 (s, 1H), 8.46 (s, 1H), 8.17 (d, *J* = 7.8 Hz, 2H), 7.98 (d, *J* = 8.4 Hz, 2H), 7.76 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 2H), 2.66 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 197.8, 196.1, 153.1, 142.7, 140.3, 130.3, 129.4, 128.4, 128.3, 111.8, 27.5, 26.5; HRMS (ESI) m/z calcd for $C_{16}H_{17}N_2O_4S^+$ $(M+H)^+$ 333.0904, found 333.0901.



3g

3-acetyl-N'-(3-acetylphenyl)benzenesulfonohydrazide:

Yield: 67% (66.7 mg); yellow oil; IR (KBr): 3336, 1683, 1593, 1358, 1288, 1260, 1162, 1024, 797, 632, 582 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.88 (s, 1H), 8.35 (s, 1H), 8.24 (d, *J* = 7.2 Hz, 1H), 8.08 (d, *J* = 7.8 Hz, 1H), 8.01 (s, 1H), 7.77 (t, *J* = 7.8 Hz, 1H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.26 (t, *J* = 7.8 Hz, 1H), 7.06 (d, *J* = 6.6 Hz, 1H), 2.64 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 197.8, 196.9, 148.7, 139.7, 137.3, 132.6, 131.8 130.0, 129.0, 126.8, 119.5, 117.5, 111.5, 26.9, 26.7; HRMS (ESI) m/z calcd for $C_{16}H_{17}N_2O_4S^+$ $(M+H)^+$ 333.0904, found 333.0904.

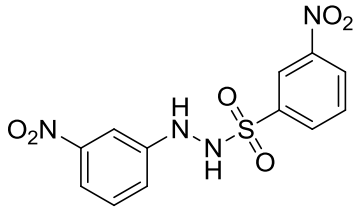


3h

2-nitro-N'-(2-nitrophenyl)benzenesulfonohydrazide:

Yield: 45% (45.6 mg); gray solid; mp: 147.6-151.2 °C; IR (KBr): 3257, 1613, 1577, 1524, 1498, 1433, 1334, 1271, 1165, 856, 738, 588 cm^{-1}; ^{1}H NMR (600 MHz,

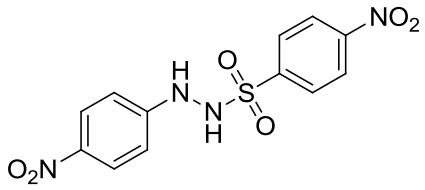
DMSO-$_{d6}$) δ = 10.45 (s, 1H), 9.20 (s, 1H), 8.06 – 8.04 (m, 3H), 7.93 (t, *J* = 7.8 Hz, 1H), 7.87 (t, *J* = 7.8 Hz, 1H), 7.56 (t, *J* = 7.8 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 1H), 6.91 (t, *J* = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 147.9, 144.3, 136.1, 135.2, 132.8, 132.5, 131.1, 130.3, 125.7, 124.6, 118.9, 115.6; HRMS (ESI) m/z calcd for $C_{12}H_{11}N_4O_6S^+$ $(M+H)^+$ 339.0394, found 339.0399.



3i

3-nitro-N'-(3-nitrophenyl)benzenesulfonohydrazide:

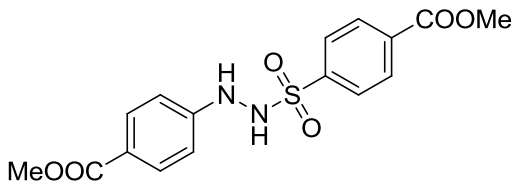
Yield: 62% (62.8 mg); yellow oil; IR (KBr): 3090, 1624, 1530, 1352, 1126, 1025, 1001, 876, 780, 732, 672, 586 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 10.23 (s, 1H), 8.58 – 8.55 (m, 2H), 8.46 (s, 1H), 8.26 (d, *J* = 8.4 Hz, 1H), 7.94 (t, *J* = 7.8 Hz, 1H), 7.57 – 7.56 (m, 2H), 7.41 (t, *J* = 8.4 Hz, 1H), 7.19 (d, *J* = 7.4 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 149.5, 148.4, 147.9, 140.1, 133.8, 131.5, 130.2, 127.8, 122.4, 118.9, 113.7, 106.2; HRMS (ESI) m/z calcd for $C_{12}H_{11}N_4O_6S^+$ $(M+H)^+$ 339.0394, found 339.0397.



3j

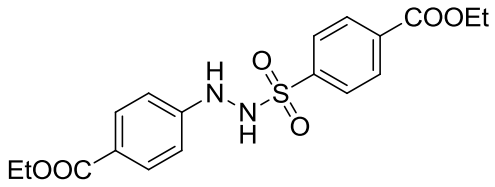
4-nitro-N'-(4-nitrophenyl)benzenesulfonohydrazide:

Yield: 78% (78.9 mg); yellow solid; mp: 106.8-109.4 °C; IR (KBr): 1632, 1526, 1350, 1325, 1166, 1110, 1022, 987, 780, 686, 615 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 10.39 (s, 1H), 9.02 (s, 1H), 8.49 (d, *J* = 6.6 Hz, 2H), 8.13 (d, *J* = 7.2 Hz, 2H), 8.08 (d, *J* = 7.2 Hz, 2H), 6.92 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 154.3, 150.2, 143.8, 138.9, 129.4, 125.7, 124.7, 111.5; HRMS (ESI) m/z calcd for $C_{12}H_{10}N_4O_6SNa^+$ $(M+Na)^+$ 361.0213, found 361.0207.



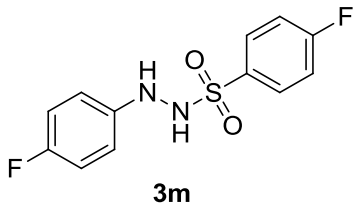
3k

methyl 4-((2-(4-(methoxycarbonyl)phenyl)hydrazinyl)sulfonyl)benzoate:
Yield: 75% (81.9 mg); yellow solid; mp: 87.7-91.3 °C; IR (KBr): 2252, 1644, 1610, 1245, 1111, 1048, 1026, 1003, 826, 774, 692, 604 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 10.00 (s, 1H), 8.43 (s, 1H), 8.17 (d, *J* = 7.8 Hz, 2H), 7.98 (d, *J* = 7.8 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 9.0 Hz, 2H), 3.91 (s, 3H), 3.76 (s, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 166.1, 165.3, 152.7, 142.7, 133.4, 130.6, 130.1, 128.1, 119.6, 111.6, 52.7, 51.5; HRMS (ESI) m/z calcd for $C_{16}H_{17}N_2O_6S^+$ $(M+H)^+$ 365.0802, found 365.0803.



3l

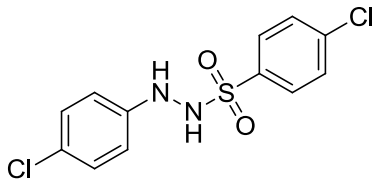
ethyl 4-((2-(4-(ethoxycarbonyl)phenyl)hydrazinyl)sulfonyl)benzoate:
Yield: 74% (87.1 mg); yellow solid; mp: 135.8-137.5 °C; IR (KBr): 3327, 3154, 1717, 1682, 1607, 1309, 1175, 1161, 1023, 844, 691, 607, 548 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 10.00 (s, 1H), 8.42 (s, 1H), 8.18 (d, *J* = 8.4 Hz, 2H), 8.00 (d, *J* = 7.8 Hz, 2H), 7.73 (d, *J* = 9.0 Hz, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 4.39 – 4.36 (m, 2H), 4.25 – 4.21 (m, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 1.28 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 165.6, 164.8, 152.6, 142.7, 133.7, 130.5, 130.0, 128.1, 119.9, 111.6, 61.5, 60.0, 14.3, 14.1; HRMS (ESI) m/z calcd for $C_{18}H_{21}N_2O_6S^+$ $(M+H)^+$ 393.1115, found 393.1113.



3m

4-fluoro-N'-(4-fluorophenyl)benzenesulfonohydrazide:

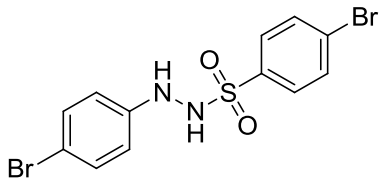
Yield: 69% (58.7 mg); yellow solid; mp: 130.4-133.7 °C; IR (KBr): 3333, 3246, 1639, 1591, 1506, 1346, 1170, 1091, 1021, 830, 718, 551 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.64 (d, *J* = 1.2 Hz, 1H), 7.91 – 7.89 (m, 2H), 7.66 (s, 1H), 7.46 (t, *J* = 9.0 Hz, 2H), 6.95 (t, *J* = 9.0 Hz, 2H), 6.82 – 6.79 (m, 2H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 165.4, 163.7, 156.9, 155.3, 145.0, 135.3, 130.7, 130.6, 116.4, 116.3, 115.1, 115.0, 114.0; HRMS (ESI) m/z calcd for $C_{12}H_{11}F_2N_2O_2S^+$ $(M+H)^+$ 285.0504, found 285.0505.



3n

4-chloro-N'-(4-chlorophenyl)benzenesulfonohydrazide:

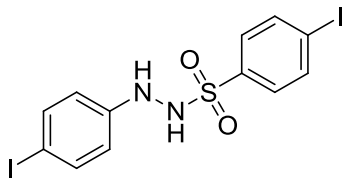
Yield: 72% (68.4 mg); yellow solid; mp: 214.3-216.7 °C; IR (KBr): 3253, 1594, 1510, 1485, 1327, 1163, 1088, 823, 753, 724, 645, 549 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.74 (s, 1H), 7.86 (s, 1H), 7.83 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.15 (d, *J* = 9.0 Hz, 2H), 6.80 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 147.5, 137.9, 137.6 129.5, 129.3, 128.4, 122.4, 114.1; HRMS (ESI) m/z calcd for $C_{12}H_{11}Cl_2N_2O_2S^+$ $(M+H)^+$ 316.9913, found 316.9908.



3o

4-bromo-N'-(4-bromophenyl)benzenesulfonohydrazide:

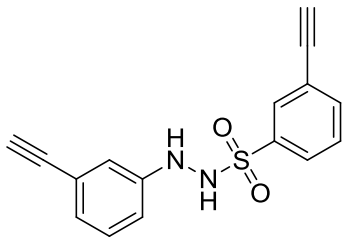
Yield: 78% (94.9 mg); gray solid; mp:125.7-128.1 °C; IR (KBr): 3242, 1637, 1574, 1486, 1389, 1325, 1157, 1089, 1005, 819, 737, 601 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.74 (s, 1H), 7.87 (s, 1H), 7.84 (d, *J* = 8.4 Hz, 2H), 7.75 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 6.76 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 147.9, 138.0, 132.3, 131.3, 129.6, 127.0, 114.7, 110.0; (EI-MS) m/z: 403.90.



3p

4-iodo-N'-(4-iodophenyl)benzenesulfonohydrazide:

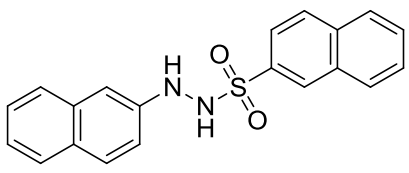
Yield: 68% (101.8 mg); gray solid; mp: 142.1-144.6 °C; IR (KBr): 3324, 3225, 1637, 1483, 1328, 1252, 1159, 1087, 1003, 815, 732, 496 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.70 (s, 1H), 8.01 (d, *J* = 7.2 Hz, 2H), 7.85 (s, 1H), 7.58 (d, *J* = 7.8 Hz, 2H), 7.41 (d, *J* = 7.2 Hz, 2H), 6.65 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 148.4, 138.4, 138.1, 137.0, 129.2, 115.2, 101.4, 80.6; (EI-MS) m/z: 499.90.



3q

3-ethynyl-N'-(3-ethynylphenyl)benzenesulfonohydrazide:

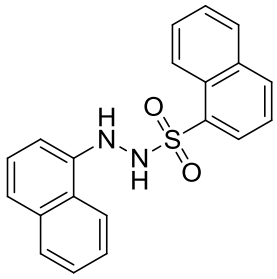
Yield: 43% (38.1 mg); gray solid; mp: 136.9-140.3 °C; IR (KBr): 1639, 1330, 1208, 1157, 1024, 994, 782, 688 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.77 (s, 1H), 7.90 (s, 1H), 7.85 (d, *J* = 6.6 Hz, 2H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.65 (t, *J* = 8.4 Hz, 1H), 7.12 (t, *J* = 7.8 Hz, 1H), 6.88 (s, 1H), 6.82 (d, *J* = 7.8 Hz, 2H), 4.43 (s, 1H), 4.07 (s, 1H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 149.0, 139.8, 136.3, 130.6, 130.2, 129.4, 128.3, 123.1, 122.9, 122.2, 115.8, 113.9, 84.2, 83.1, 82.3, 80.3; HRMS (ESI) m/z calcd for $C_{16}H_{13}N_2O_2S^+$ $(M+H)^+$ 297.0692, found 297.0692.



3r

N'-(naphthalen-2-yl)naphthalene-2-sulfonohydrazide:

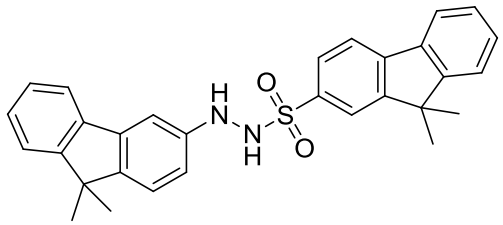
Yield: 61% (63.6 mg) ; gray solid; mp: 159.3-162.5 °C; IR (KBr): 3173, 1631, 1605, 1524, 1313, 1158, 1046, 1022, 775, 708, 523 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-$_{d6}$) δ = 9.79 (s, 1H), 8.51 (s, 1H), 8.17 – 8.13 (m, 2H), 8.03 (d, *J* = 8.0 Hz, 1H), 7.94 (s, 1H), 7.90 (d, *J* = 8.8 Hz, 1H), 7.70 – 7.60 (m, 4H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.30 (t, *J* = 8.0 Hz, 1H), 7.18 – 7.13 (m, 2H), 7.03 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-$_{d6}$) δ=146.1, 135.8, 134.2, 133.8, 131.5, 129.1, 129.0, 128.6, 128.0, 127.7, 127.5, 127.3, 127.2, 125.9, 125.8, 122.8, 122.2, 115.8, 105.8; HRMS (ESI) m/z calcd for $C_{20}H_{17}N_2O_2S^+$ $(M+H)^+$ 349.1005, found 349.1002.



3s

N'-(naphthalen-1-yl)naphthalene-1-sulfonohydrazide:

Yield: 58% (60.5 mg); yellow oil; IR (KBr): 1637, 1383, 1292, 1161, 1024, 995, 781, 713 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 10.01 (s, 1H), 8.85 (d, *J* = 8.4 Hz, 1H), 8.30 (s, 1H), 8.23 (d, *J* = 8.4 Hz, 1H), 8.19 (d, *J* = 6.6 Hz, 1H), 8.10 (d, *J* = 7.8 Hz, 1H), 7.92 (d, *J* = 7.8 Hz, 1H), 7.75 – 7.68 (m, 3H), 7.61 – 7.59 (m, 1H), 7.40 (d, *J* = 6.6 Hz, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 7.21 (d, *J* = 7.8 Hz, 1H), 7.11 – 7.09 (m, 1H), 6.92 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 142.9, 134.2, 133.6, 133.3, 133.0, 129.3, 128.3, 127.6, 127.1, 126.2, 125.3, 125.1, 124.7, 123.9, 123.8, 121.5, 121.1, 118.3, 106.0; HRMS (ESI) m/z calcd for $C_{20}H_{17}N_2O_2S^+$ $(M+H)^+$ 349.1005, found 349.1004.

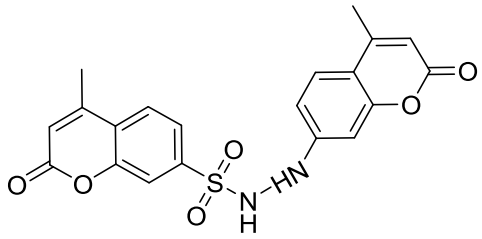


3t

N'-(9,9-dimethyl-9H-fluoren-3-yl)-9,9-dimethyl-9H-fluorene-2-sulfonohydrazide:

Yield: 63% (90.7 mg); light yellow oil; IR (KBr): 2960, 1613, 1446, 1300, 1212, 1157, 1026, 1001, 761, 735, 670, 567 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 9.62 (s, 1H), 8.06 (d, *J* = 7.8 Hz, 1H), 7.96 – 7.92 (m, 3H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.56 (d, *J* = 7.8 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 1H), 7.39 – 7.36 (m, 3H), 7.21 (t, *J* = 7.2 Hz, 1H), 7.13 (t, *J* = 7.2 Hz, 1H), 6.75 – 6.63 (m, 2H), 1.39 (s, 6H), 1.19 (s, 6H); ^{13}C

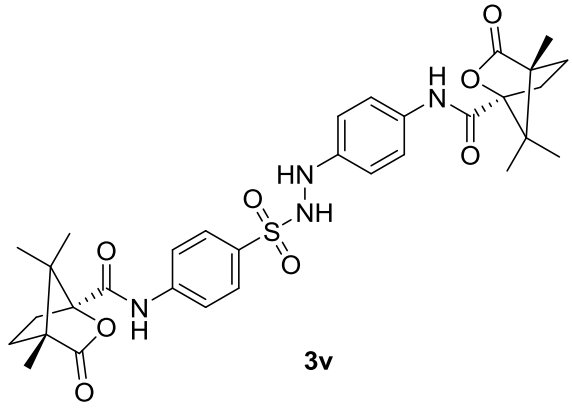
NMR (150 MHz, DMSO-$_{d6}$) δ= 154.2, 153.7, 152.3, 148.3, 143.1, 139.0, 137.8, 136.9, 129.9, 128.8, 127.4, 126.8, 126.7, 125.5, 123.0, 122.4, 122.0, 121.2, 120.7, 120.3, 118.6, 111.8, 106.6, 46.8, 46.0, 27.0, 26.3; HRMS (ESI) m/z calcd for $C_{30}H_{29}N_2O_2S^+$ $(M+H)^+$ 481.1944, found 481.1950.



3u

4-methyl-N'-(4-methyl-2-oxo-2H-chromen-7-yl)-2-oxo-2H-chromene-7-sulfonohydrazide:

Yield: 53% (65.5 mg); yellow solid; mp: 257.1- 260.3 °C; IR (KBr): 3251, 2382, 1726, 1618, 1393, 1304, 1162, 864, 583 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 10.16 (s, 1H), 8.54 (s, 1H), 8.02 (d, *J* = 7.8 Hz, 1H), 7.77 (d, *J* = 7.8 Hz, 1H), 7.71 (s, 1H), 7.50 (d, *J* = 9.0 Hz, 1H), 6.78 (d, *J* = 9.0 Hz, 1H), 6.59 (d, *J* = 8.4 Hz, 2H), 6.04 (s, 1H), 2.47 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 160.4, 159.1, 154.6, 153.6, 152.6, 152.3, 152.0, 141.2, 126.9, 126.2, 123.3, 123.0, 116.8, 115.6, 111.6, 109.7, 109.5, 98.3, 18.2, 18.1; HRMS (ESI) m/z calcd for $C_{20}H_{17}N_2O_6S^+$ $(M+H)^+$ 413.0802, found 413.0803.

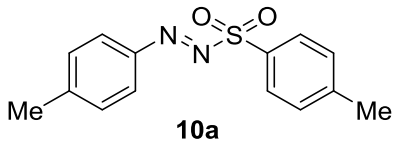


3v

(1S,4R)-4,7,7-trimethyl-3-oxo-N-(4-((2-(4-((1S,4R)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxamido)phenyl)hydrazinyl)sulfonyl)phenyl)-2-oxabicyclo[2.2.1]heptane-1-carboxamide:

Yield: 58% (110.8 mg); light yellow oil; IR (KBr): 1788, 1642, 1529, 1440, 1322, 1267, 1162, 1099, 991, 923, 782, 687 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-$_{d6}$) δ = 10.24 (s, 1H), 9.55 (s, 1H), 9.47 (s, 1H), 7.97 (d, *J* = 8.4 Hz, 2H), 7.78 (d, *J* = 9.0 Hz, 2H), 7.56 (s, 1H), 7.38 (d, *J* = 9.0 Hz, 2H), 6.74 (d, *J* = 9.0 Hz, 2H), 1.05 (s, 9H), 1.03 (d, *J* = 2.4 Hz, 8H), 0.91 (s, 5H), 0.88 (s, 4H); ^{13}C NMR (150 MHz, DMSO-$_{d6}$) δ= 178.0,

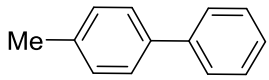
177.8, 165.9, 164.5, 145.3, 141.6, 133.8, 129.5, 128.3, 122.1, 120.6, 112.4, 91.8, 91.7, 54.5, 54.4, 53.7, 53.4, 29.9, 29.8, 28.3, 16.4, 16.3, 9.5; HRMS (ESI) m/z calcd for $C_{32}H_{39}N_4O_8S^+$ $(M+H)^+$ 639.2483, found 639.2483.



10a

(E)-1-(p-tolyl)-2-tosyldiazene:

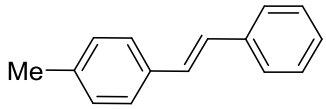
IR (KBr): 2361, 1923, 1596, 1459, 1338, 1164, 1039, 885, 808, 656, 579 cm^{-1}**;** ^1H NMR (600 MHz, $CDCl_3$) δ= 7.86 (d, *J* = 7.2 Hz, 2H), 7.72 (d, *J* = 7.2 Hz, 2H), 7.39 (d, *J* = 7.8 Hz, 2H), 7.28 (d, *J* = 7.2 Hz, 2H), 2.47 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 147.1, 146.4, 145.8, 130.2, 130.1, 129.8, 124.6, 21.8, 21.7 (5); HRMS (ESI) m/z calcd for $C_{14}H_{15}N_2O_2S^+$ $(M+H)^+$ 275.0849, found 275.0857.



10b

4-methyl-1,1'-biphenyl:

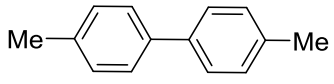
IR (KBr): 2292, 2361, 1654, 1486, 1262, 1108, 1036, 820, 757, 697 cm-1; ^1H NMR (600 MHz, DMSO-*d6*) δ= 7.50 (d, *J* = 7.2 Hz, 4H), 7.22 (d, *J* = 7.2 Hz, 4H), 2.30 (s, 6H); ^{13}C NMR (150 MHz, DMSO-*d6*) δ= 137.2, 136.4, 129.6, 126.3, 20.7; HRMS (ESI) m/z calcd for $C_{13}H_{13}^+$ $(M+H)^+$ 169.1012, found 169.1007.



10c

(E)-1-methyl-4-styrylbenzene:

IR (KBr): 2362, 2336, 1652, 1446, 1264, 1025, 966, 807, 747, 688, 529 cm^{-1}**;** ^1H NMR (600 MHz, $CDCl_3$) δ= 7.50 (d, *J* = 7.2 Hz, 2H), 7.41 (d, *J* = 7.8 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 2H), 7.25 – 7.23 (m, 1H), 7.16 (d, *J* = 7.2 Hz, 2H), 7.10 – 7.04 (m, 2H), 2.35 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 137.5, 137.4, 134.5, 129.4, 128.6, 128.5, 127.6, 127.4, 126.4, 126.3, 21.3; HRMS (ESI) m/z calcd for $C_{15}H_{15}^+$ $(M+H)^+$ 195.1168, found 195.1166.

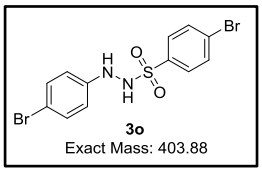


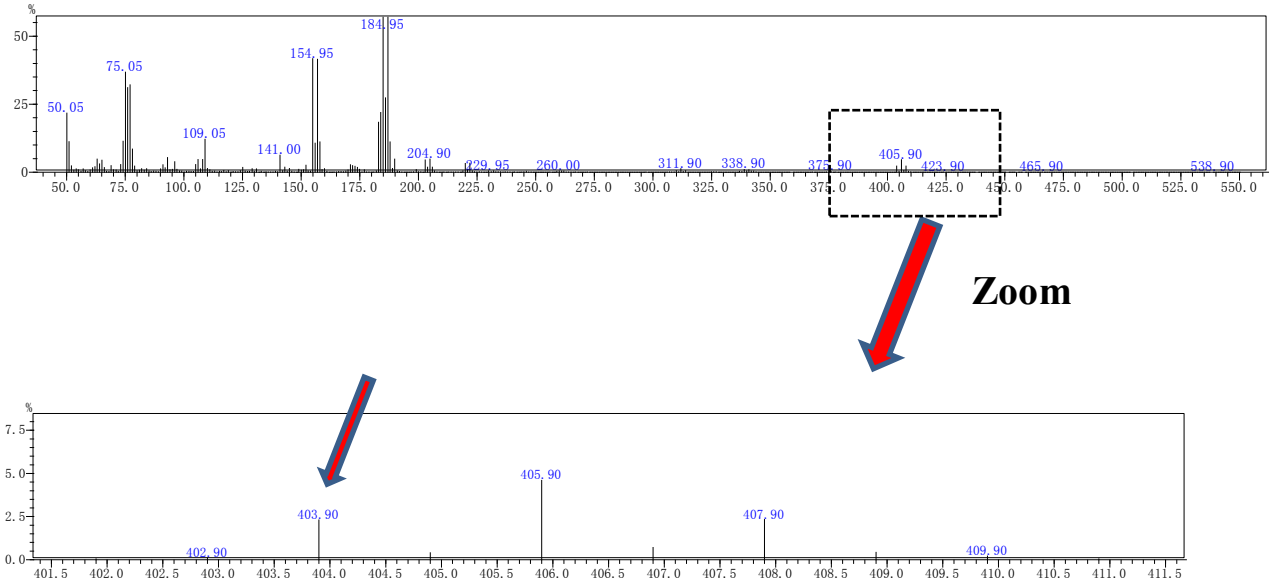
10d

4,4'-dimethyl-1,1'-biphenyl:

IR (KBr): 2922, 2854, 2361, 2336, 1648, 1452, 1397, 1270, 1030, 829, 670, 547 cm^{-1}**;** ^1H NMR (600 MHz, $CDCl_3$) δ= 7.60-7.57 (m, 2H), 7.50 (d, *J* = 7.8 Hz, 2H), 7.46 – 7.41 (m, 2H), 7.36 – 7.31 (m, 1H), 7.25 (t, *J* = 3.6 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ = 129.5, 128.7, 128.6(8), 127.2, 127.1, 127.0, 126.9, 126.8, 21.1; HRMS (ESI) m/z calcd for $C_{14}H_{15}^+$ $(M+H)^+$ 183.1168, found 183.1161.

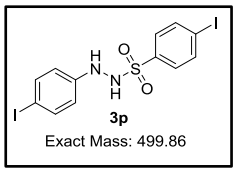
The EI-MS of **3n**

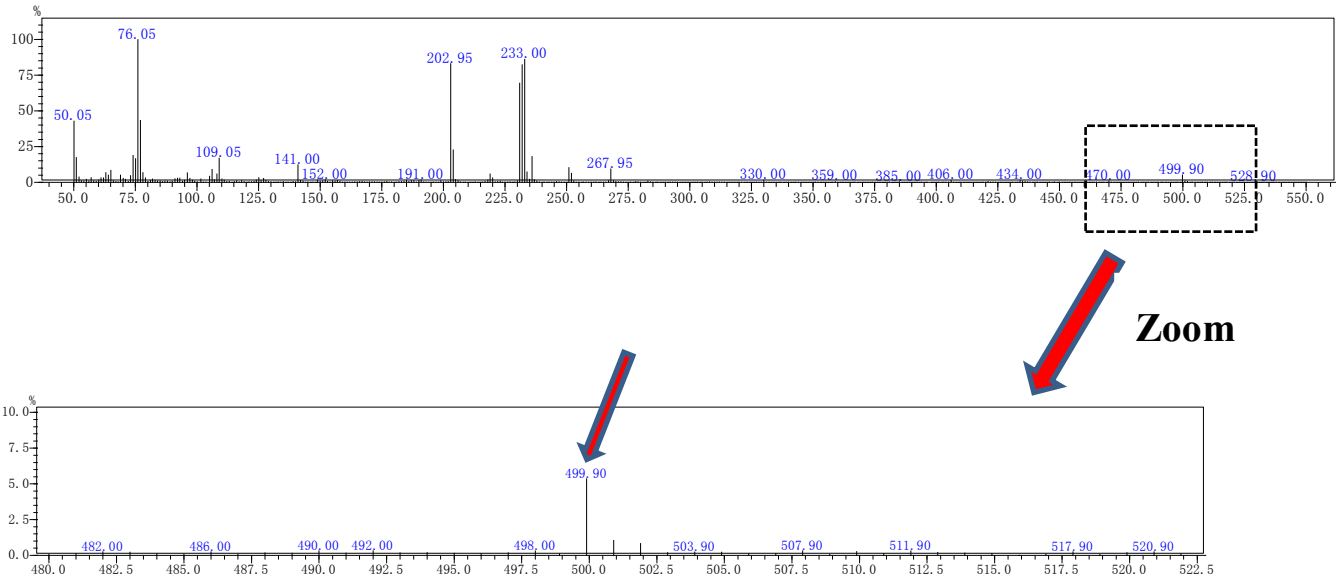




Zoom

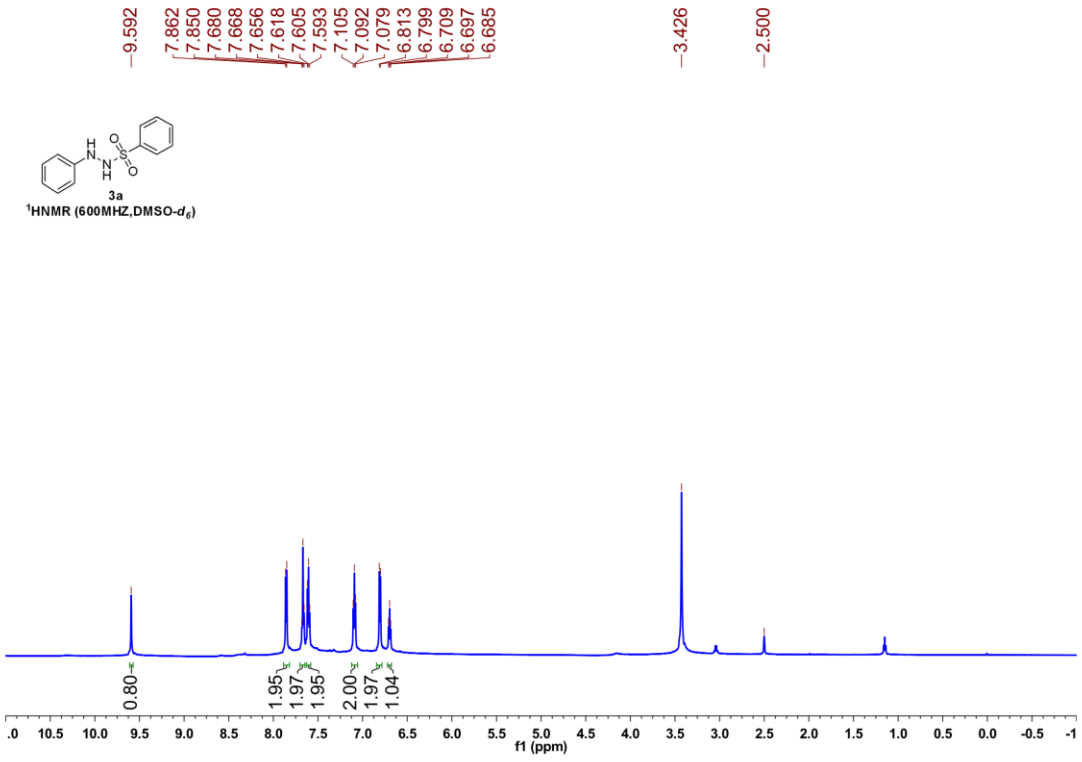
The EI-MS of **3p**

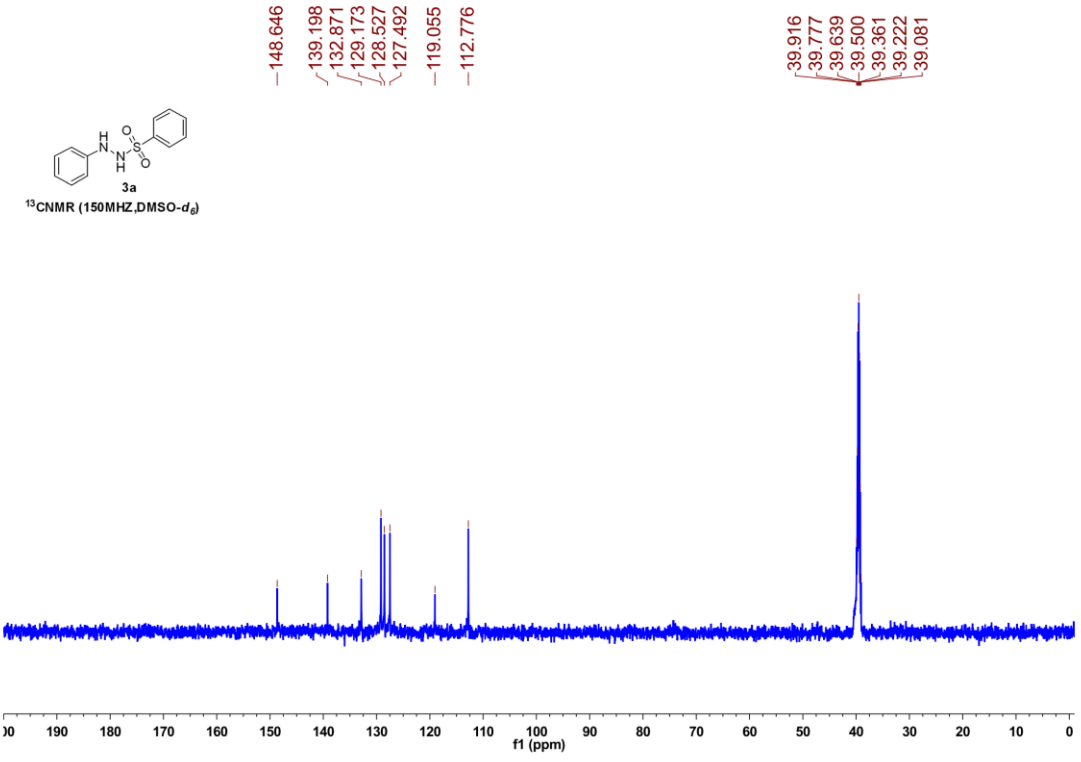


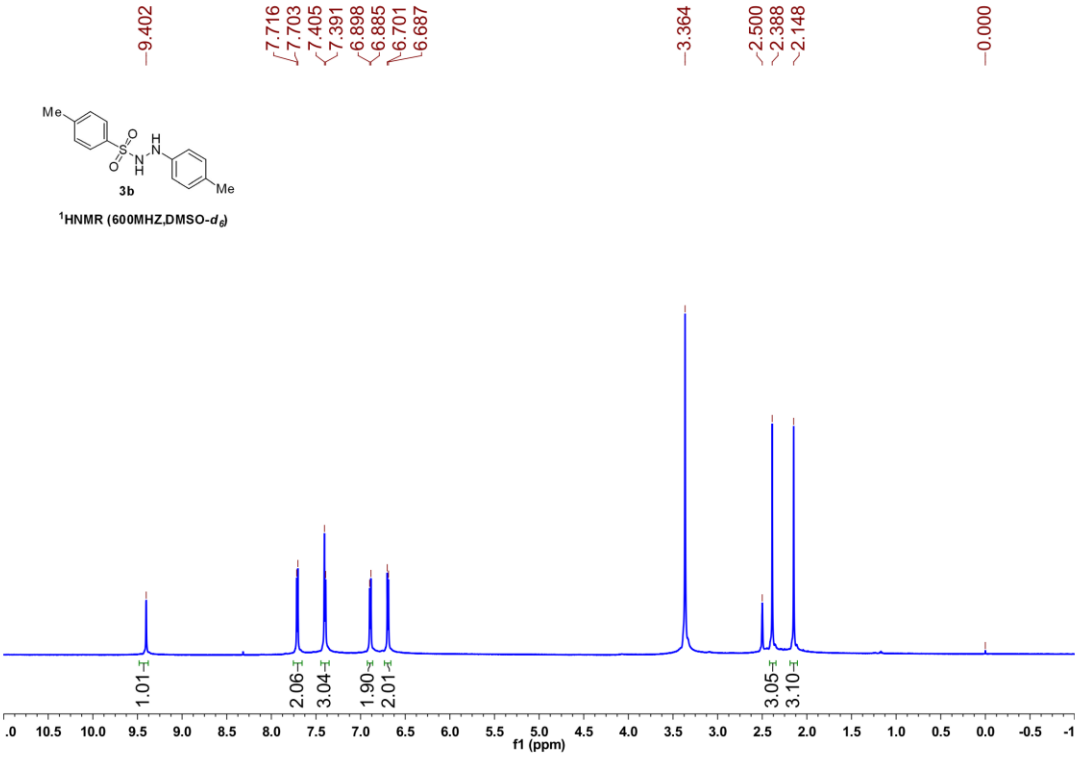


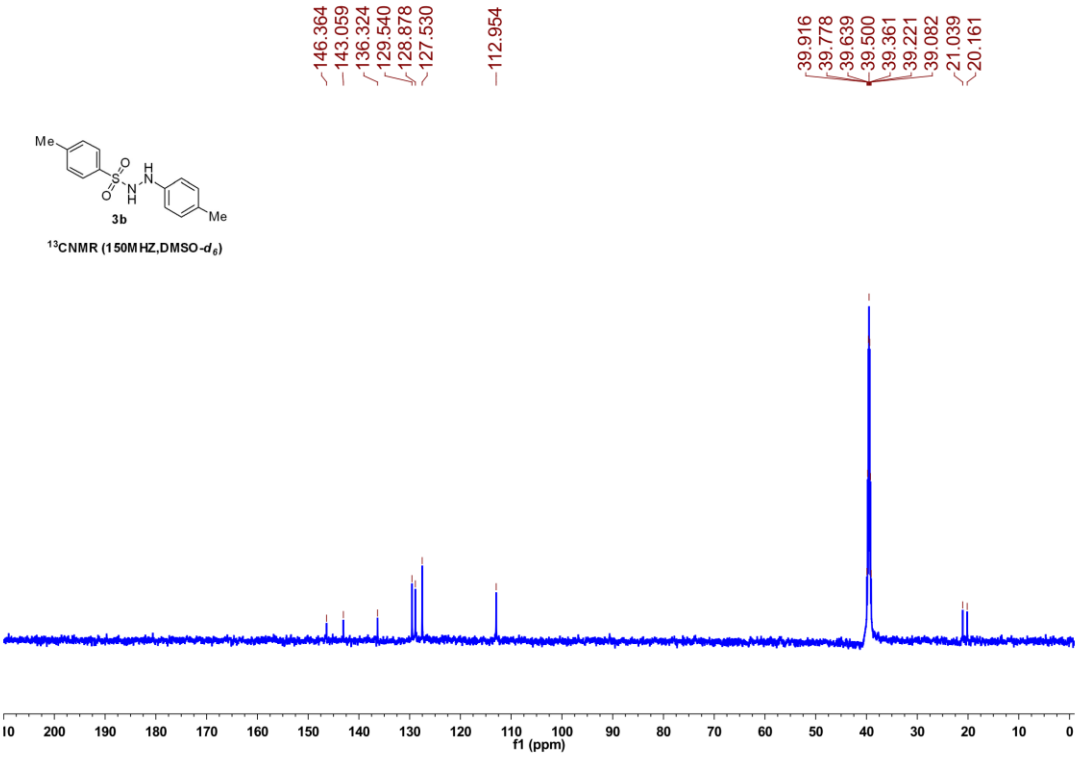
Zoom

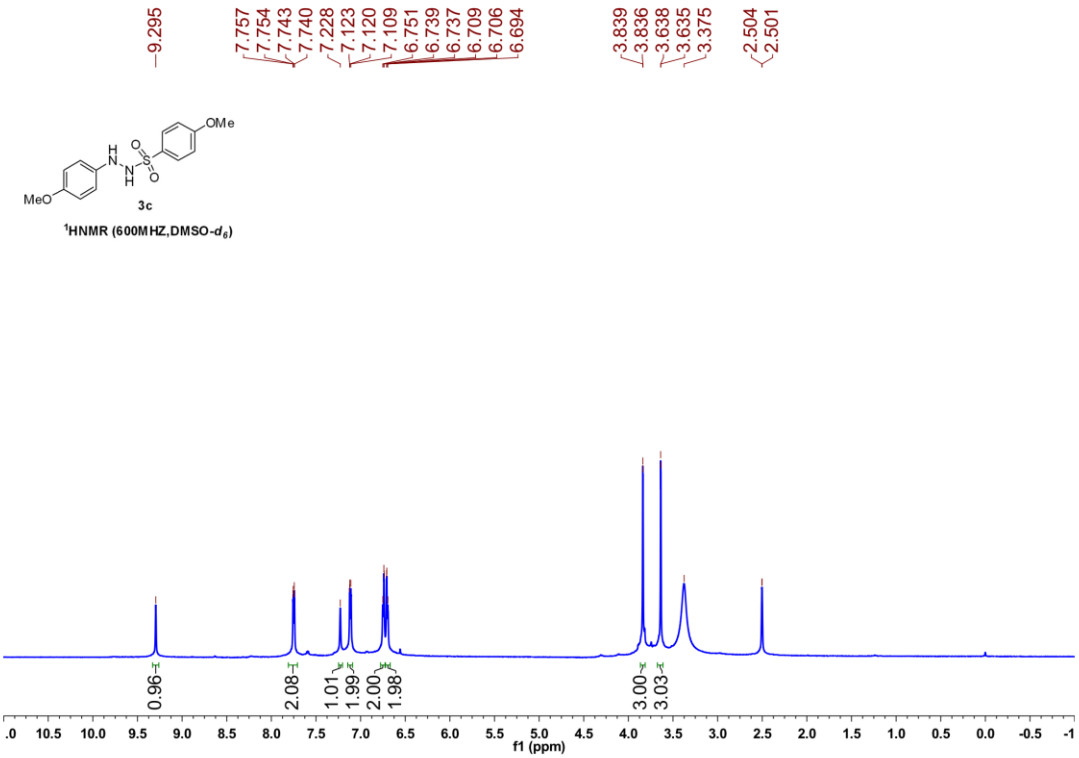
6. Copies of ^{1}H NMR, ^{13}C NMR

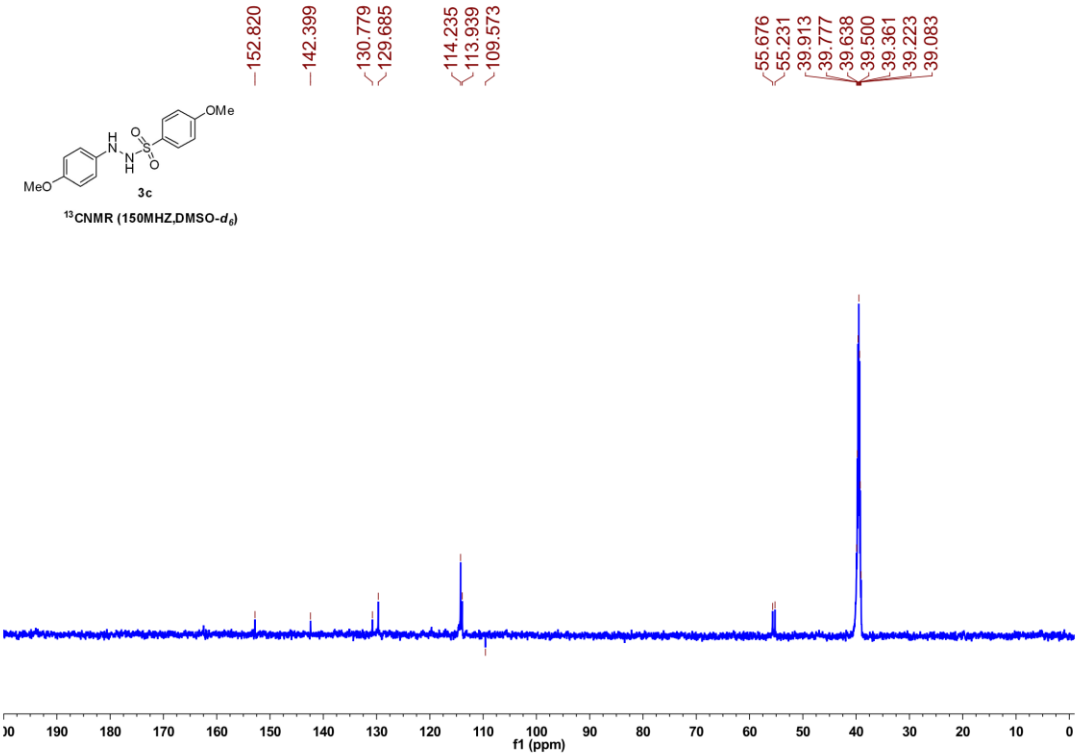


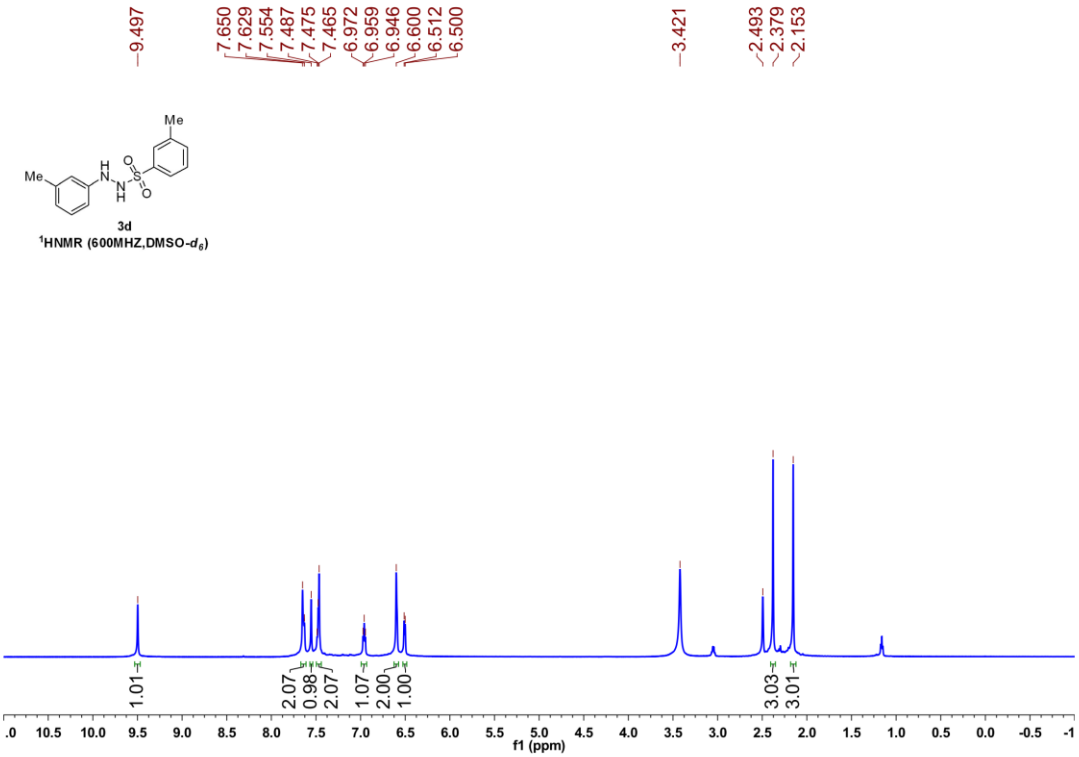


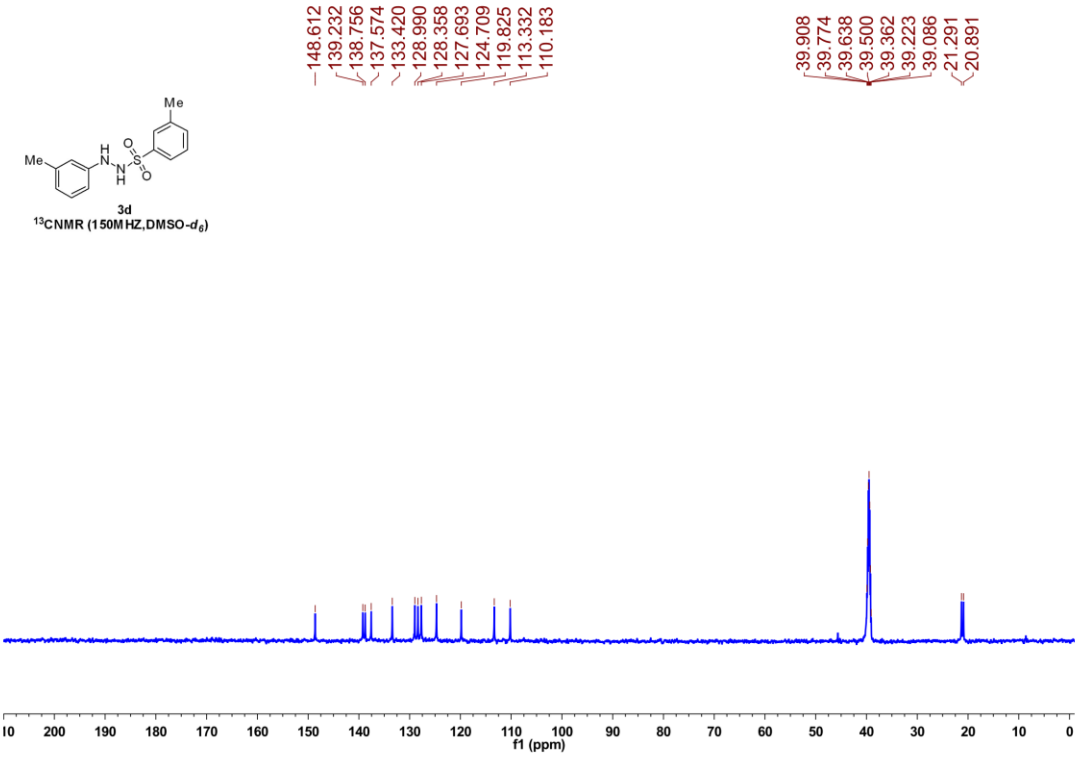

9.402
7.716
7.703
7.405
7.391
6.898
6.885
6.701
6.687
3.364
2.500
2.388
2.148
0.000
Me
Me
3b
1HNMR (600MHZ,DMSO-d6)
1.01
2.06
3.04
1.90
2.01
3.05
3.10
f1 (ppm)

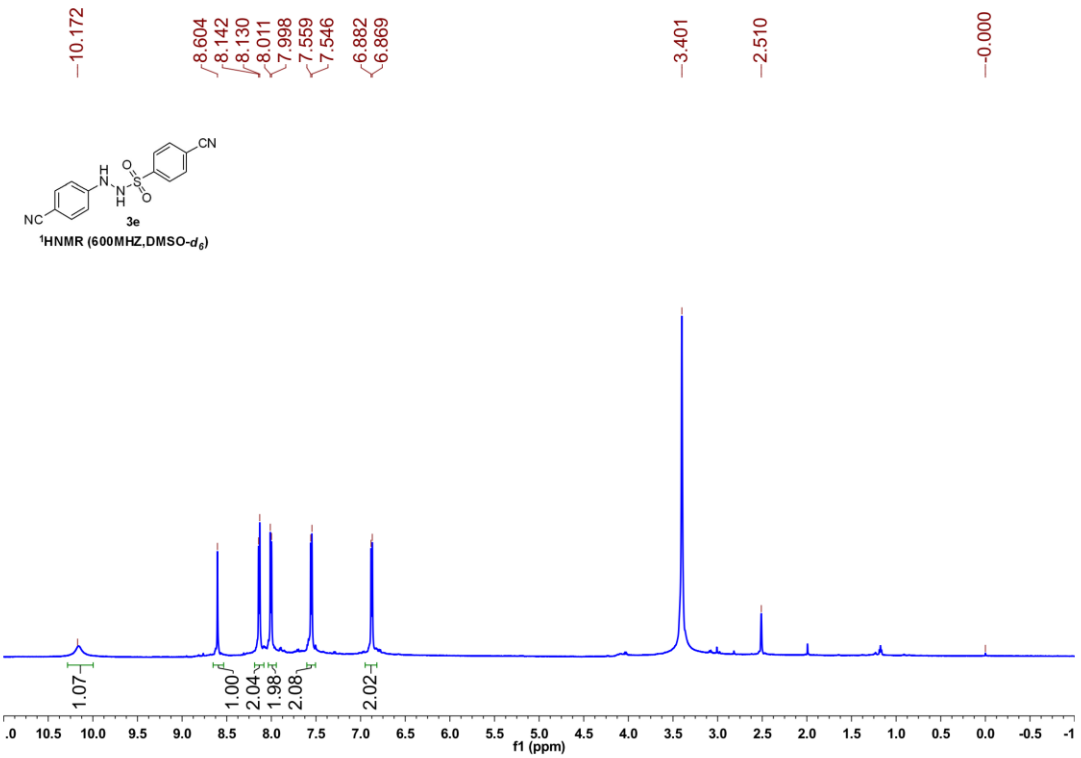

146.364
143.059
136.324
129.540
128.878
127.530
112.954
39.916
39.778
39.639
39.500
39.361
39.221
39.082
21.039
20.161
Me
Me
3b
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

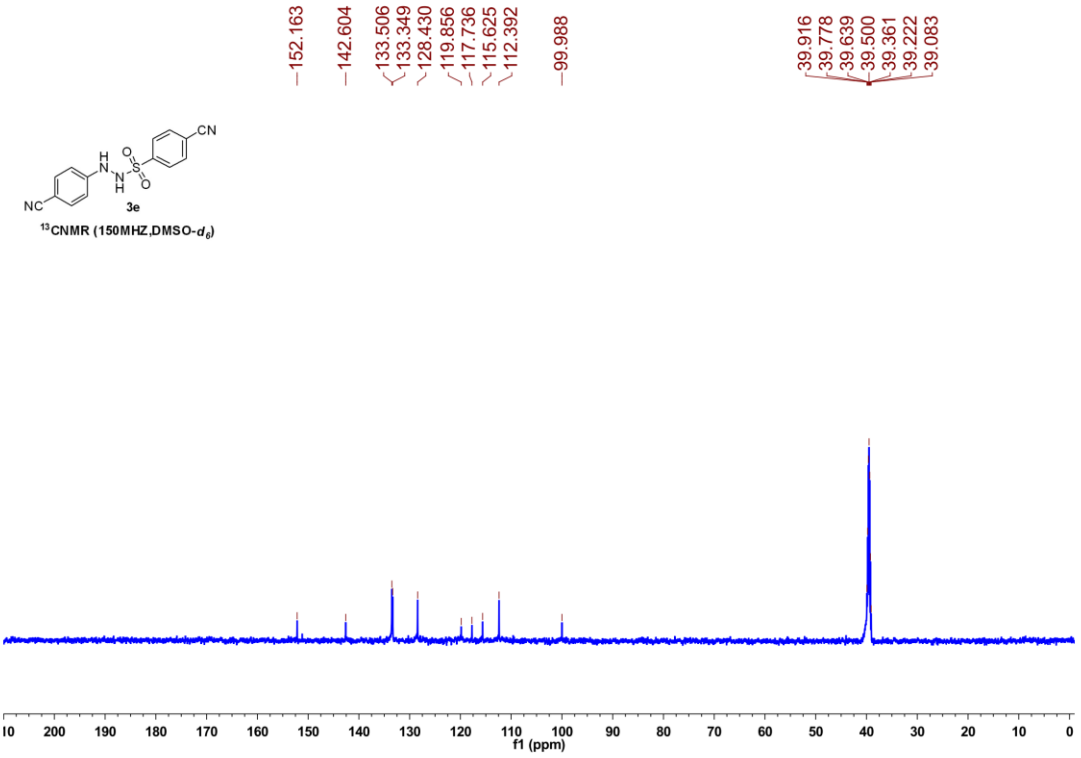

9.295
7.757
7.754
7.743
7.740
7.228
7.123
7.120
7.109
6.751
6.739
6.737
6.709
6.706
6.694
3.839
3.836
3.638
3.635
3.375
2.504
2.501
OMe
MeO
3c
1HNMR (600MHZ,DMSO-d6)
0.96
2.08
1.01
1.99
2.00
1.98
3.00
3.03
f1 (ppm)

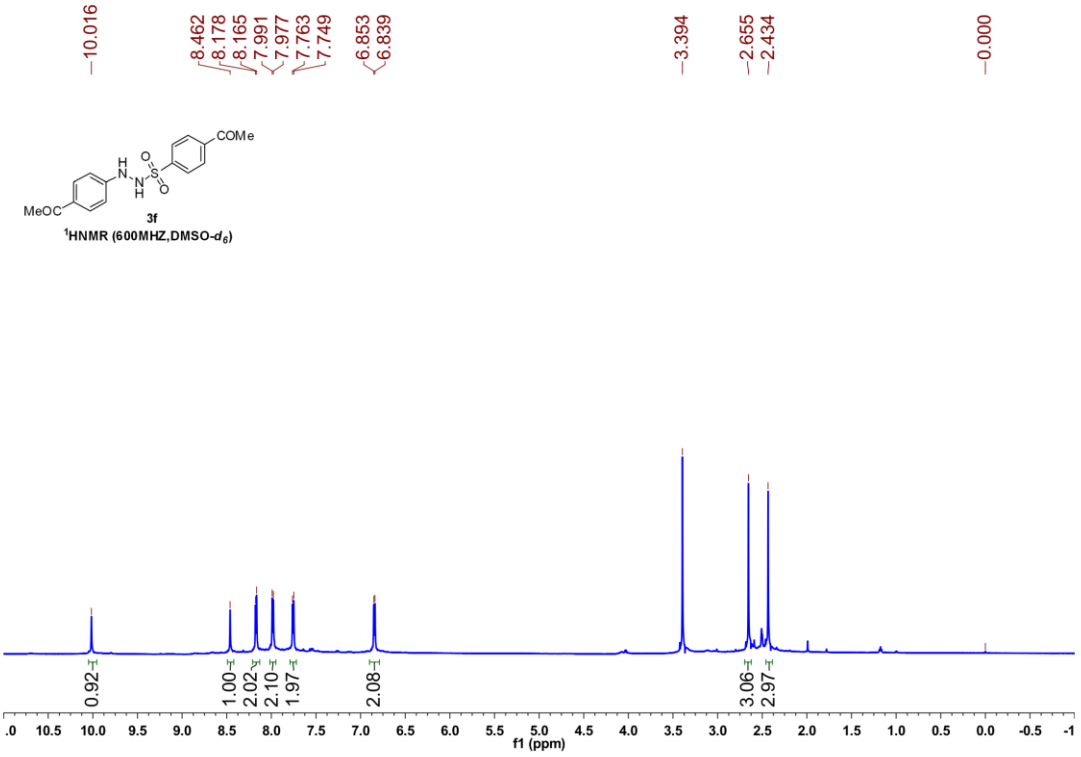

152.820
142.399
130.779
129.685
114.235
113.939
109.573
55.676
55.231
39.913
39.777
39.638
39.500
39.361
39.223
39.083
OMe
MeO
3c
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

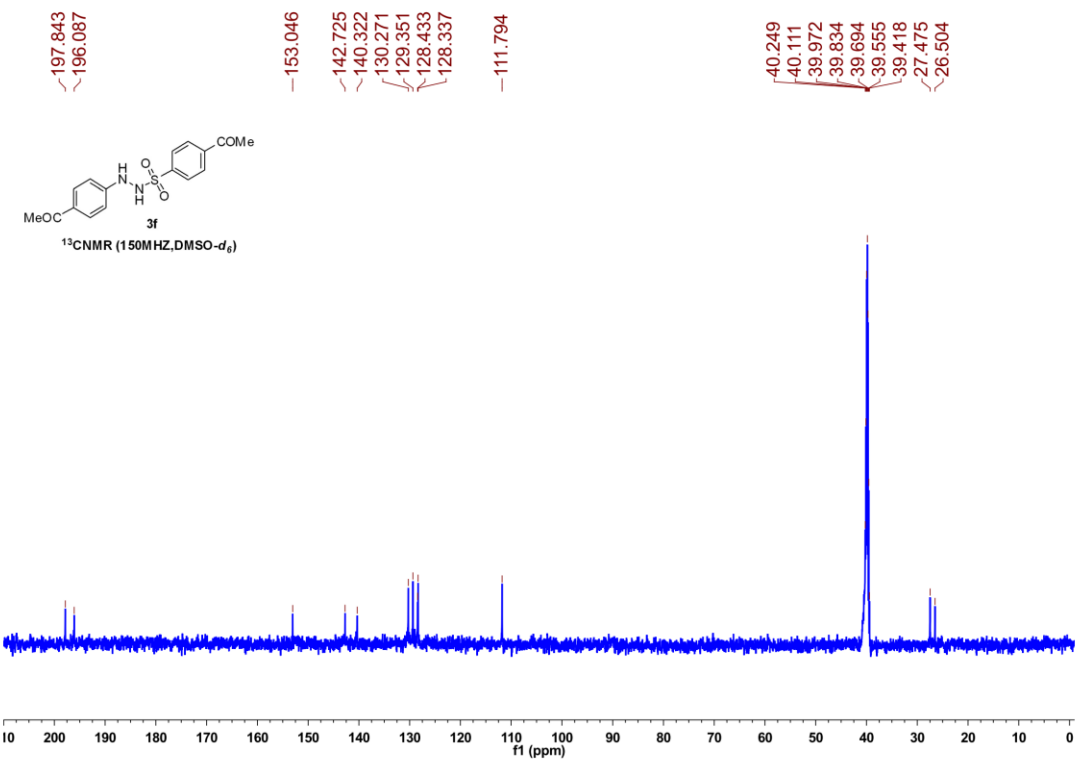

9.497
7.650
7.629
7.554
7.487
7.475
7.465
6.972
6.959
6.946
6.600
6.512
6.500
3.421
2.493
2.379
2.153
Me
Me
3d
1HNMR (600MHZ,DMSO-d6)
1.01
2.07
0.98
2.07
1.07
2.00
1.00
3.03
3.01
f1 (ppm)

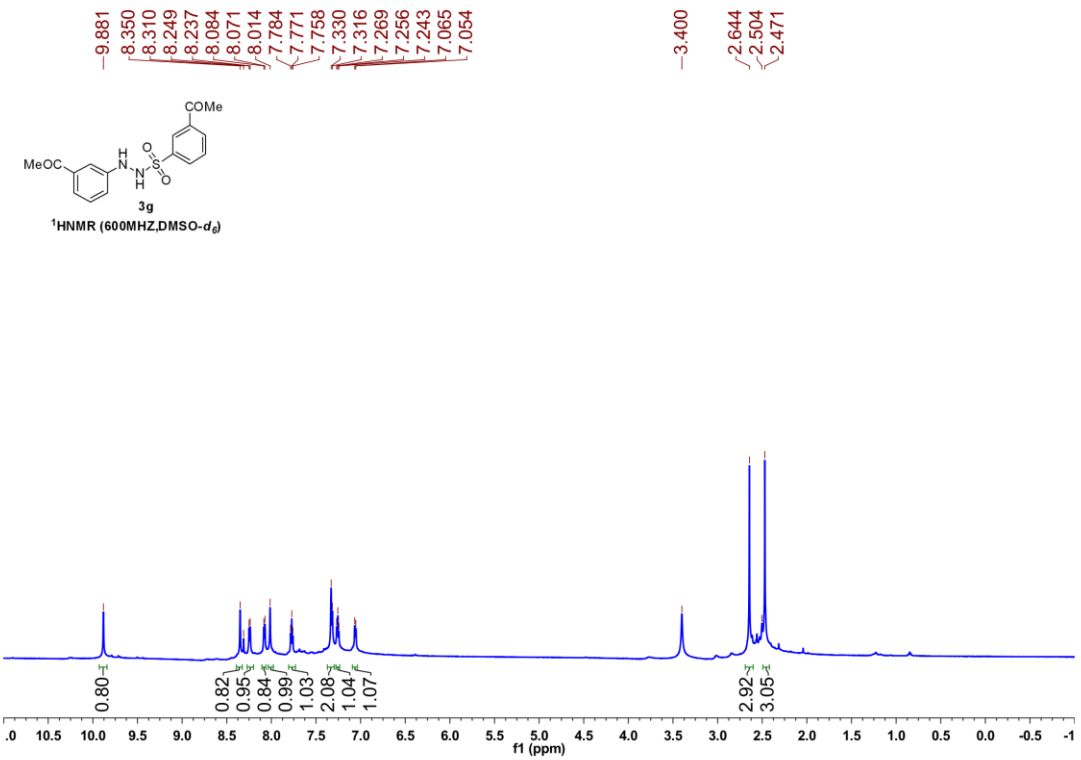

148.612
139.232
138.756
137.574
133.420
128.990
128.358
127.693
124.709
119.825
113.332
110.183
39.908
39.774
39.638
39.500
39.362
39.223
39.086
21.291
20.891
Me
Me
3d
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)


10.172
8.604
8.142
8.130
8.011
7.998
7.559
7.546
6.882
6.869
3.401
2.510
0.000
CN
NC
3e
1HNMR (600MHZ,DMSO-d6)
1.07
1.00
2.04
1.98
2.08
2.02
f1 (ppm)

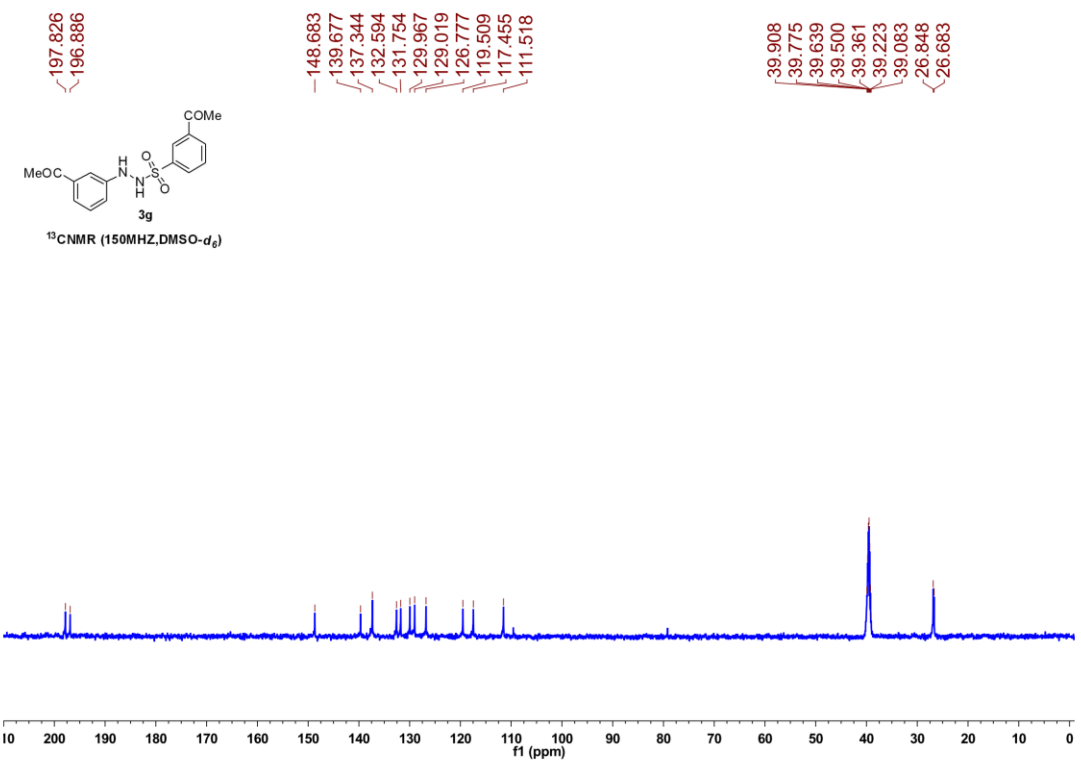

152.163
142.604
133.506
133.349
128.430
119.856
117.736
115.625
112.392
99.988
39.916
39.778
39.639
39.500
39.361
39.222
39.083
CN
NC
3e
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)


10.016
8.462
8.178
8.165
7.991
7.977
7.763
7.749
6.853
6.839
3.394
2.655
2.434
0.000
COMe
MeOC
3f
1HNMR (600MHZ,DMSO-d6)
0.92
1.00
2.02
2.10
1.97
2.08
3.06
2.97
f1 (ppm)

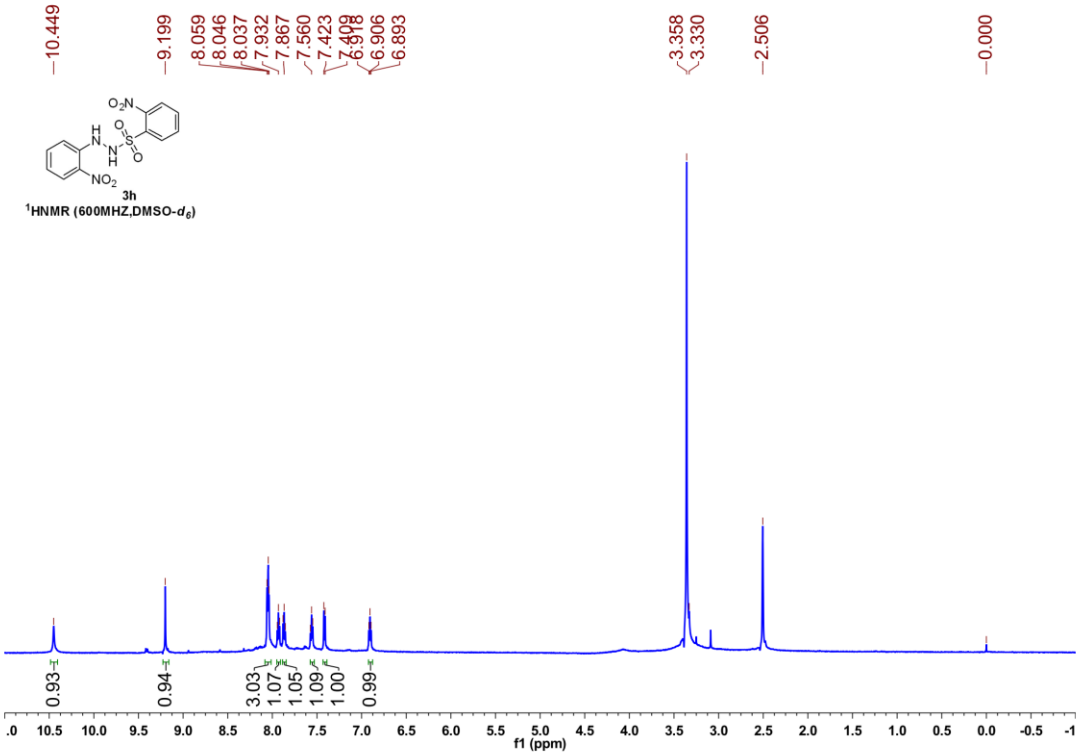

197.843
196.087
153.046
142.725
140.322
130.271
129.351
128.433
128.337
111.794
40.249
40.111
39.972
39.834
39.694
39.555
39.418
27.475
26.504
COMe
MeOC
3f
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

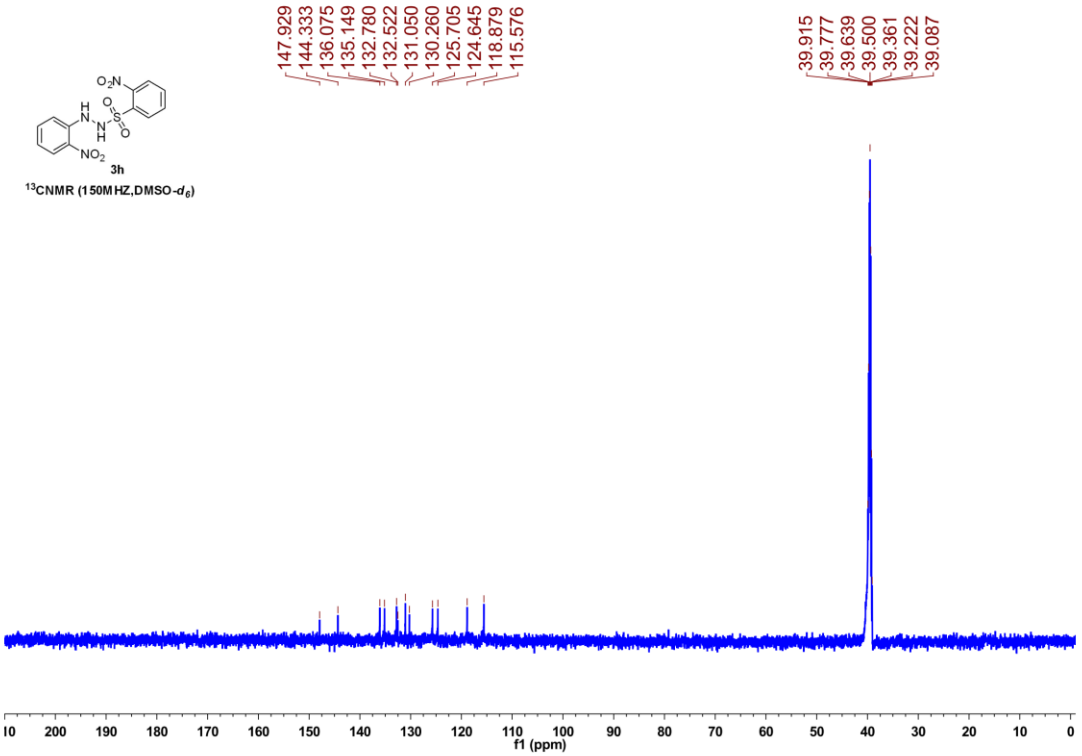


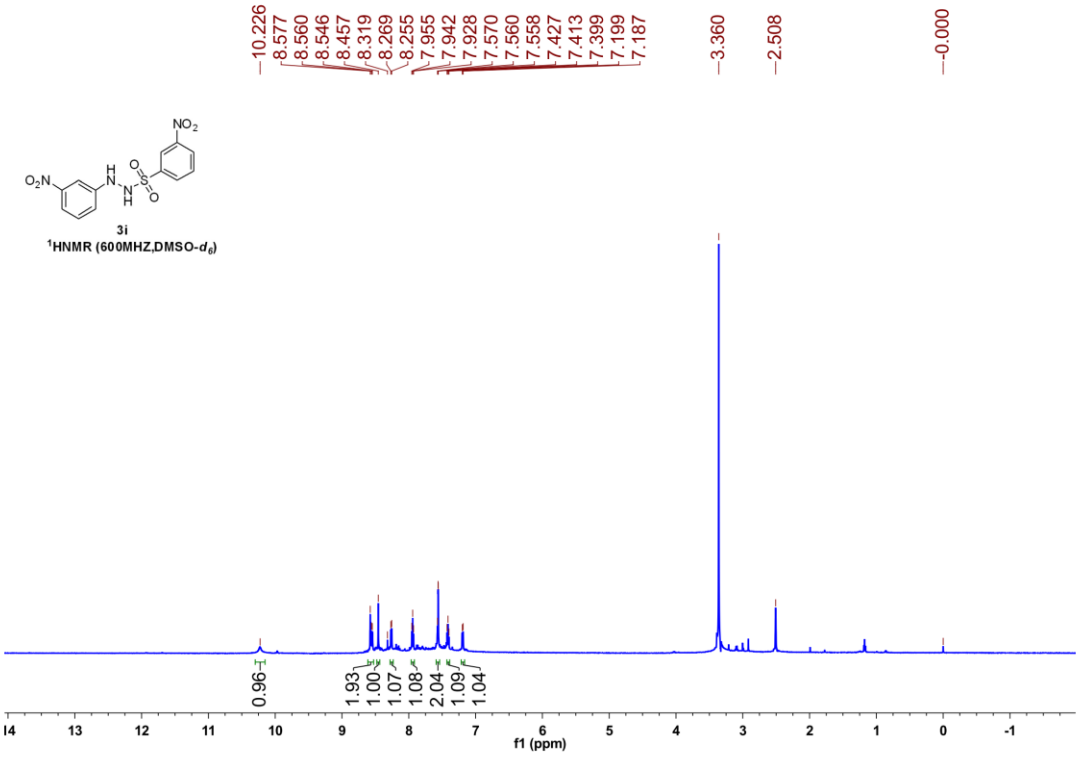
^{1}HNMR (600MHZ,DMSO-d_6)

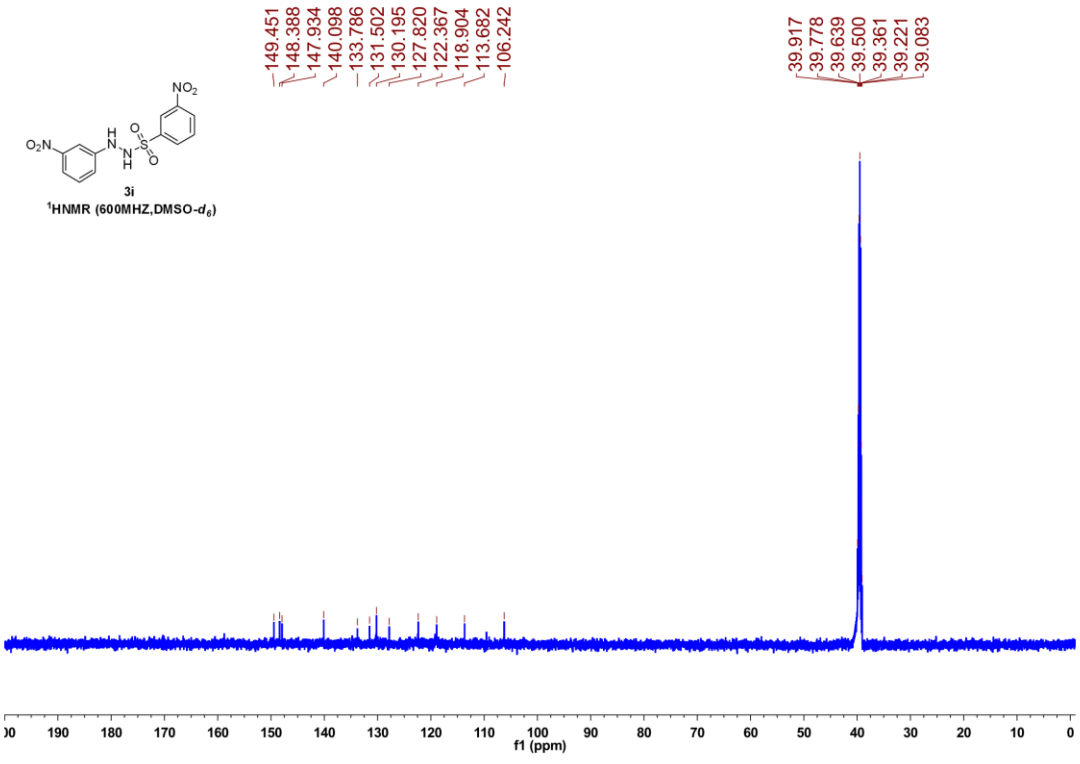


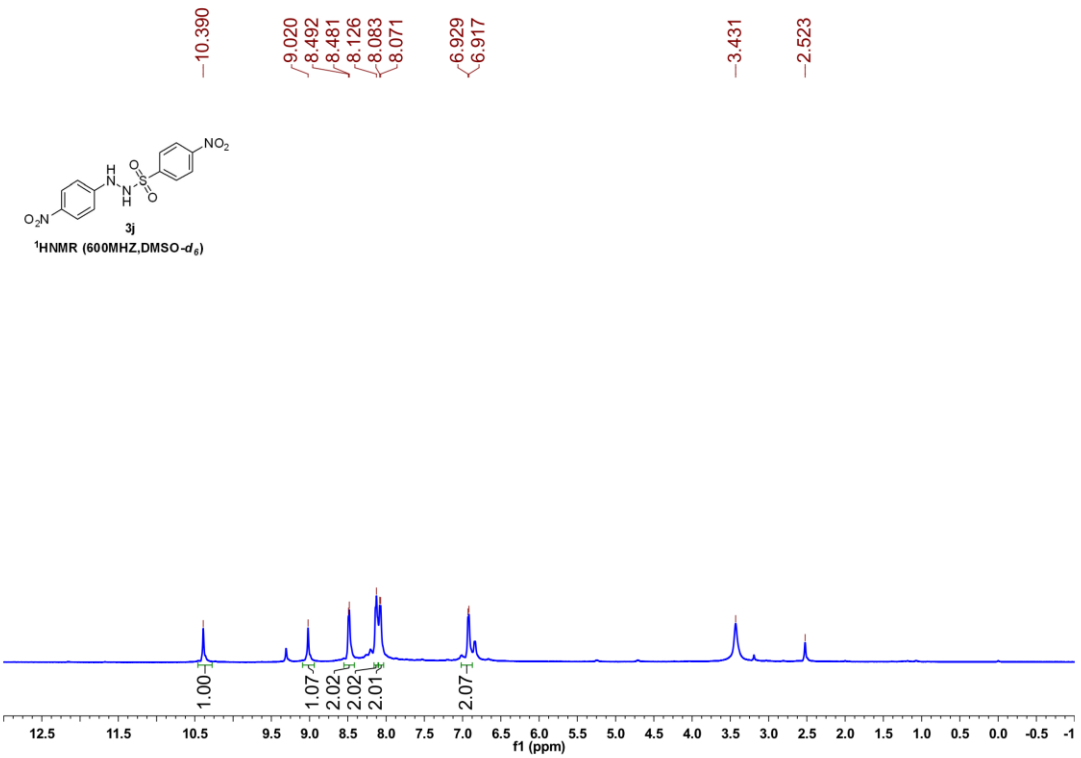
^{13}CNMR (150MHZ,DMSO-d_6)

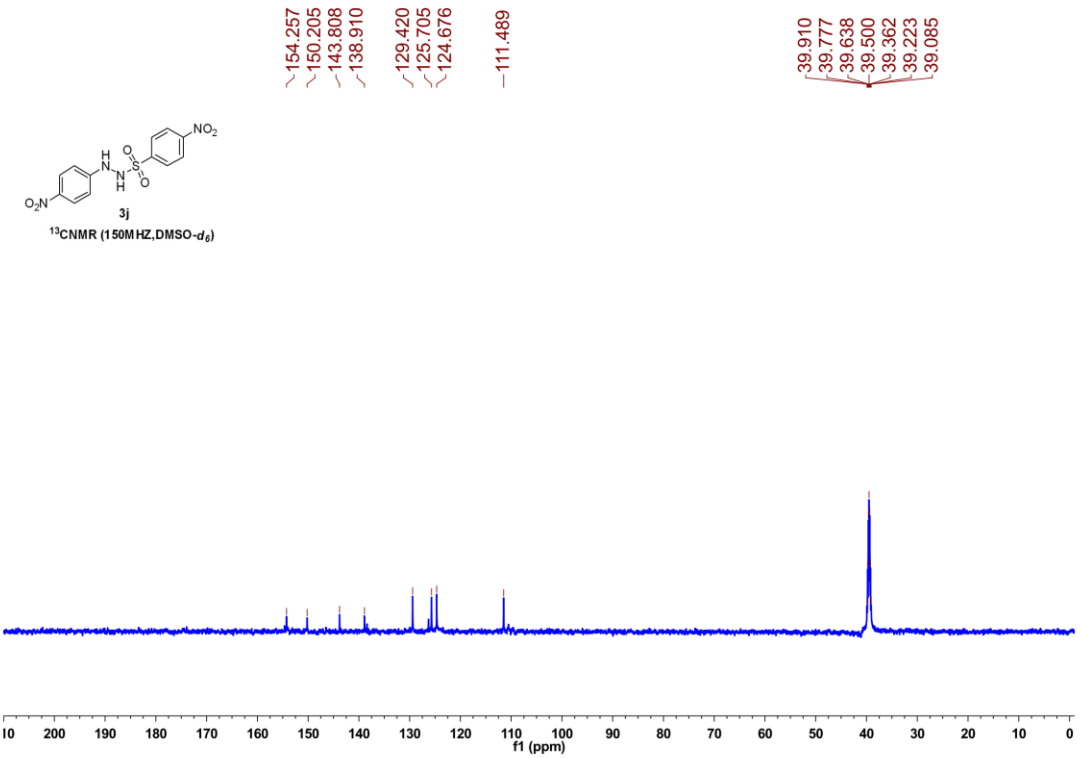

10.449
9.199
8.059
8.046
8.037
7.932
7.867
7.560
7.423
7.409
6.918
6.906
6.893
3.358
3.330
2.506
0.000
3h
1HNMR (600MHZ,DMSO-d6)
0.93
0.94
3.03
1.07
1.05
1.09
1.00
0.99
f1 (ppm)

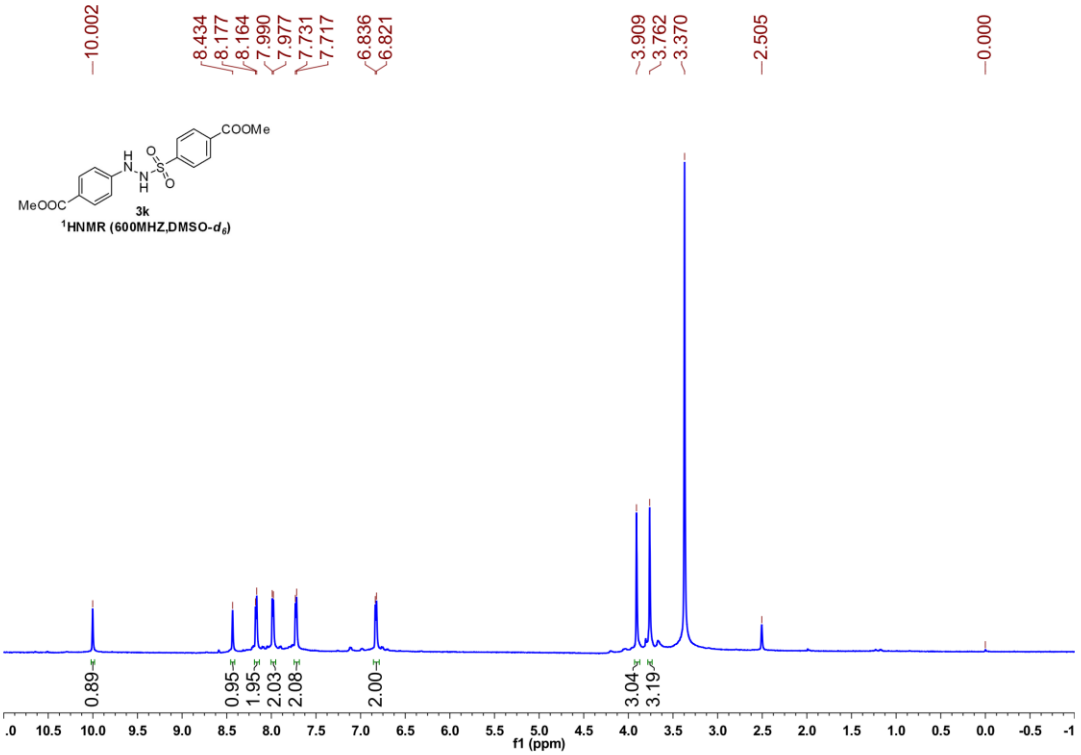

147.929
144.333
136.075
135.149
132.780
132.522
131.050
130.260
125.705
124.645
118.879
115.576
39.915
39.777
39.639
39.500
39.361
39.222
39.087
3h
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

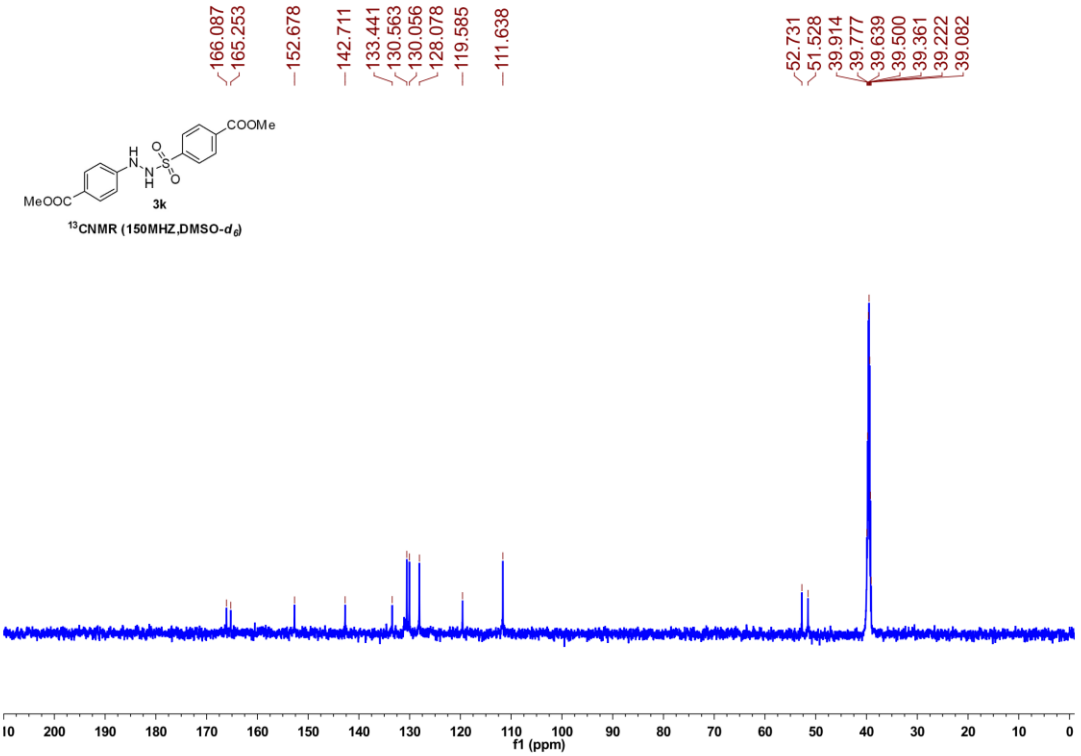

10.226
8.577
8.560
8.546
8.457
8.319
8.269
8.255
7.955
7.942
7.928
7.570
7.560
7.558
7.427
7.413
7.399
7.199
7.187
3.360
2.508
0.000
3i
1HNMR (600MHZ,DMSO-d6)
0.96
1.93
1.00
1.07
1.08
2.04
1.09
1.04
f1 (ppm)

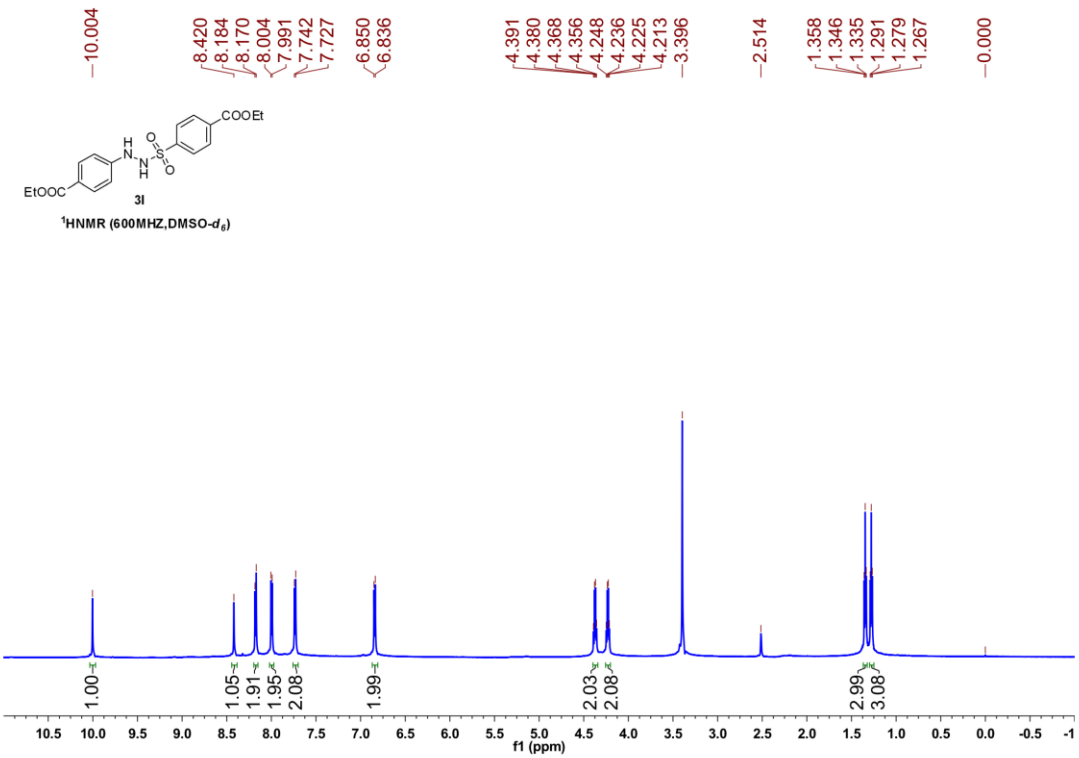

149.451
148.388
147.934
140.098
133.786
131.502
130.195
127.820
122.367
118.904
113.682
106.242
39.917
39.778
39.639
39.500
39.361
39.221
39.083
3i
1HNMR (600MHZ,DMSO-d6)
f1 (ppm)

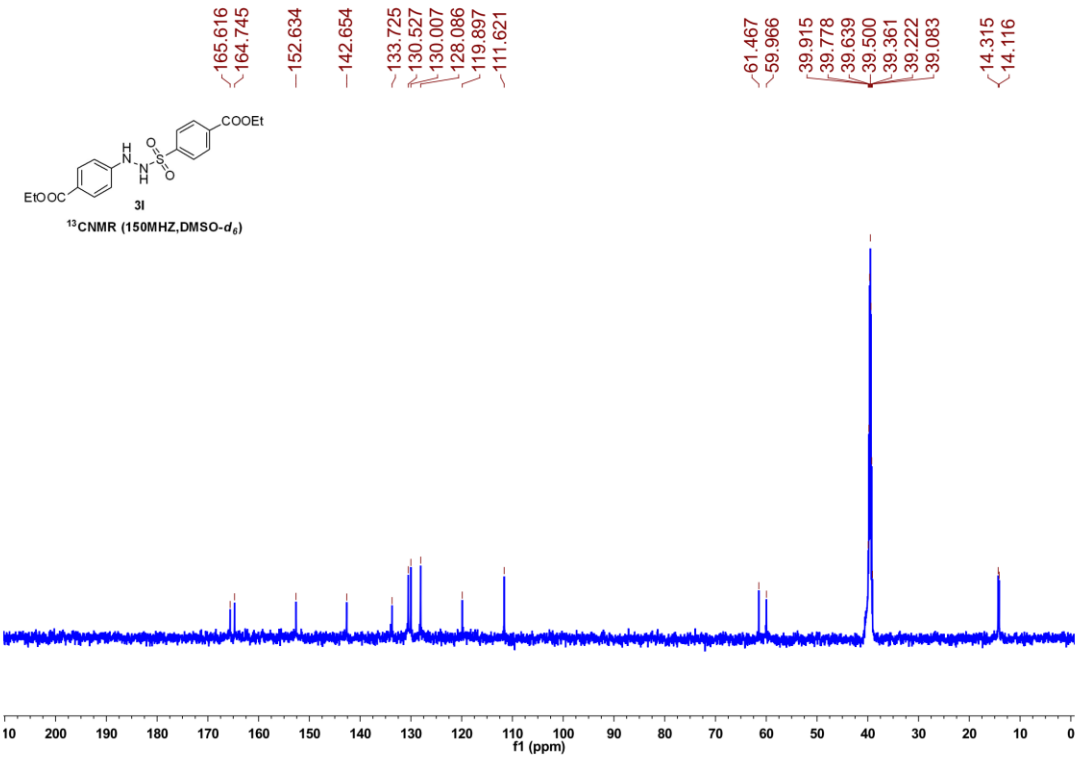

10.390
9.020
8.492
8.481
8.126
8.083
8.071
6.929
6.917
3.431
2.523
3j
1HNMR (600MHZ,DMSO-d6)
1.00
1.07
2.02
2.02
2.01
2.07
f1 (ppm)

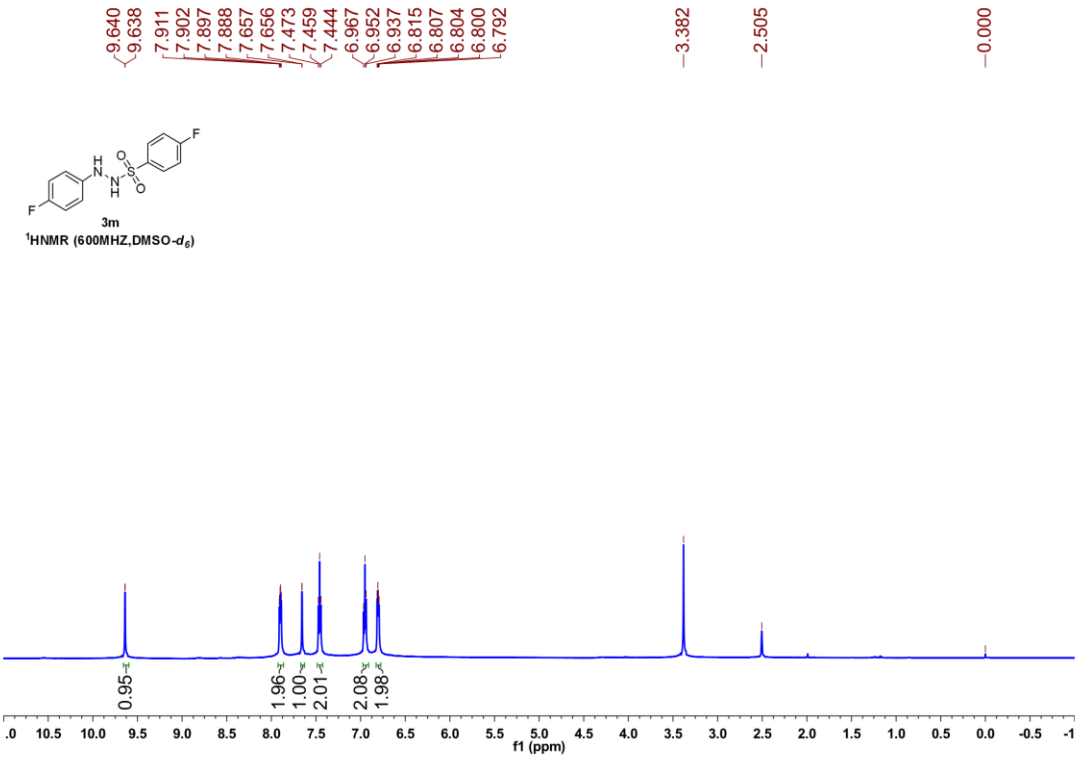

154.257
150.205
143.808
138.910
129.420
125.705
124.676
111.489
39.910
39.777
39.638
39.500
39.362
39.223
39.085
3j
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

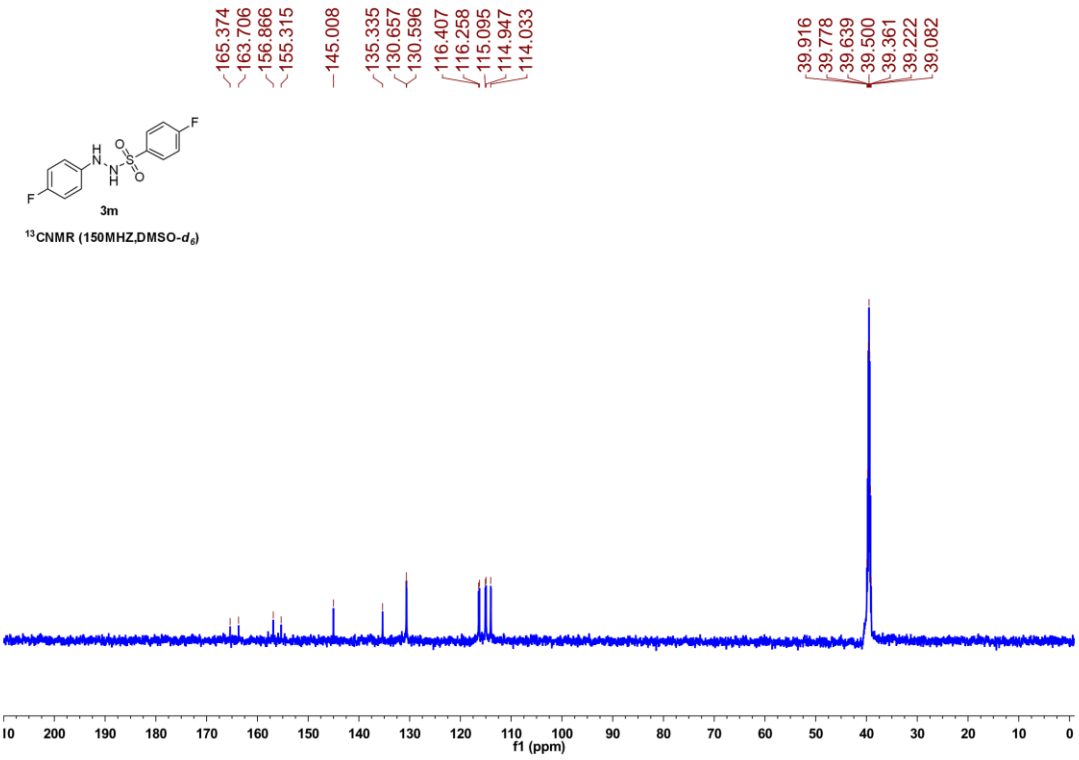

10.002
8.434
8.177
8.164
7.990
7.977
7.731
7.717
6.836
6.821
3.909
3.762
3.370
2.505
0.000
COOMe
MeOOC
3k
1HNMR (600MHZ,DMSO-d6)
0.89
0.95
1.95
2.03
2.08
2.00
3.04
3.19
f1 (ppm)

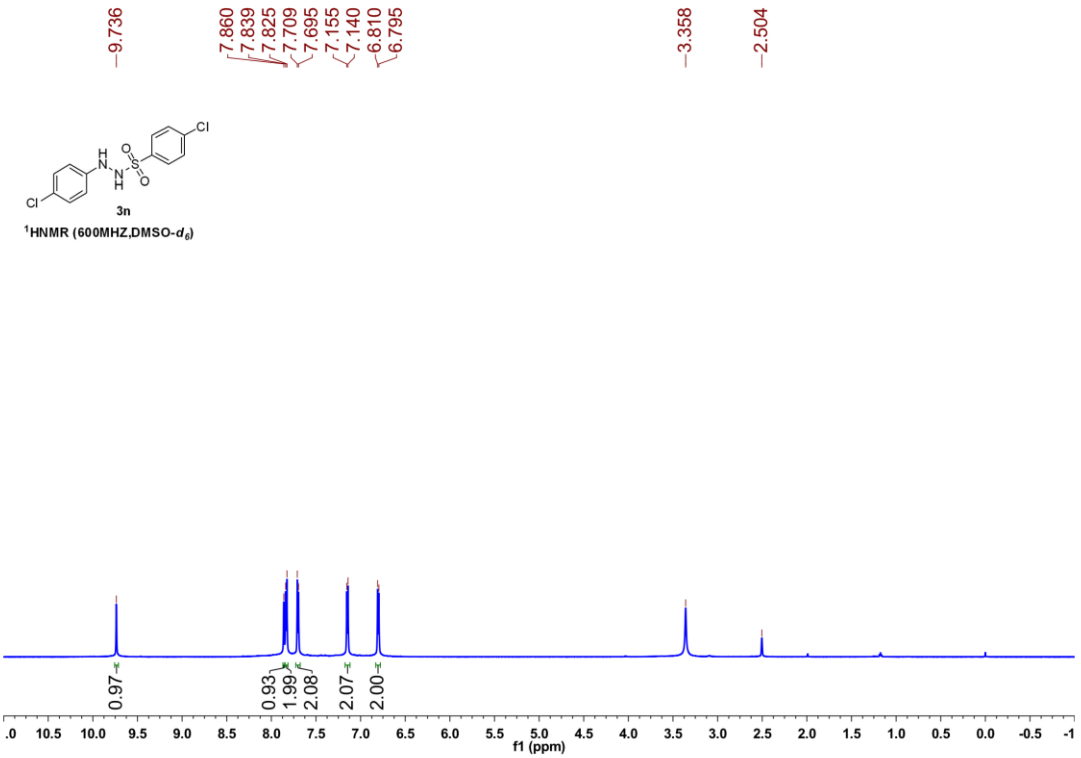

166.087
165.253
152.678
142.711
133.441
130.563
130.056
128.078
119.585
111.638
52.731
51.528
39.914
39.777
39.639
39.500
39.361
39.222
39.082
COOMe
MeOOC
3k
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

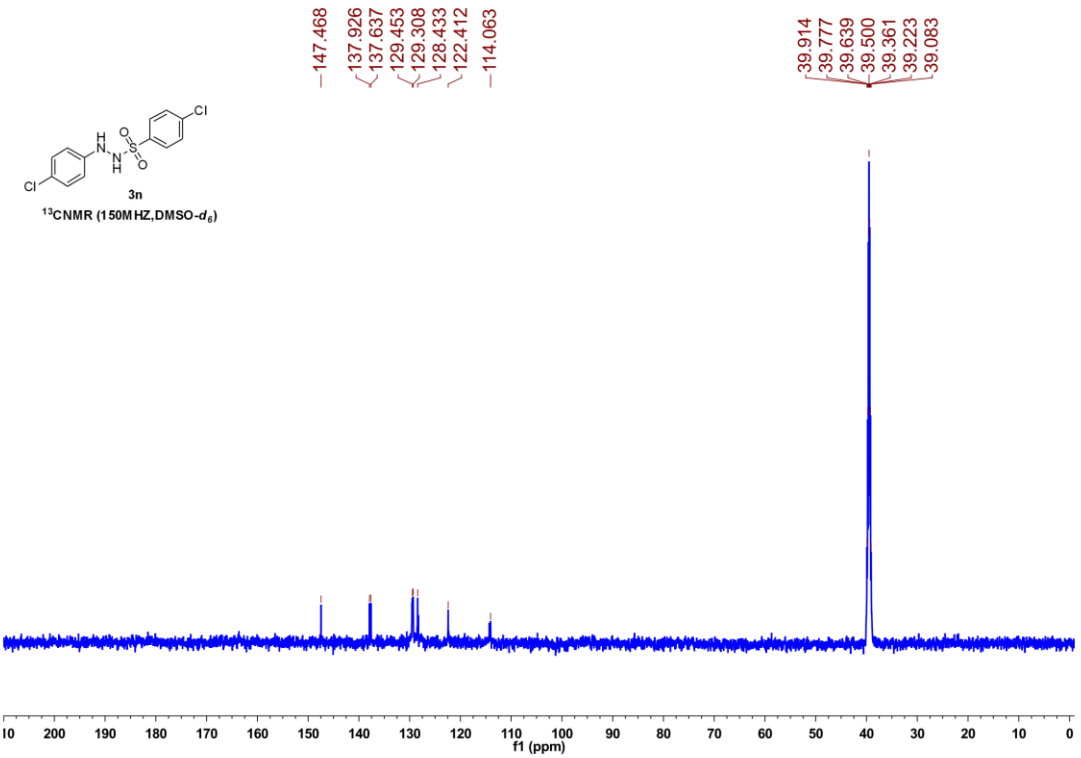

10.004
8.420
8.184
8.170
8.004
7.991
7.742
7.727
6.850
6.836
4.391
4.380
4.368
4.356
4.248
4.236
4.225
4.213
3.396
2.514
1.358
1.346
1.335
1.291
1.279
1.267
0.000
COOEt
EtOOC
3l
1HNMR (600MHZ,DMSO-d6)
1.00
1.05
1.91
1.95
2.08
1.99
2.03
2.08
2.99
3.08
f1 (ppm)

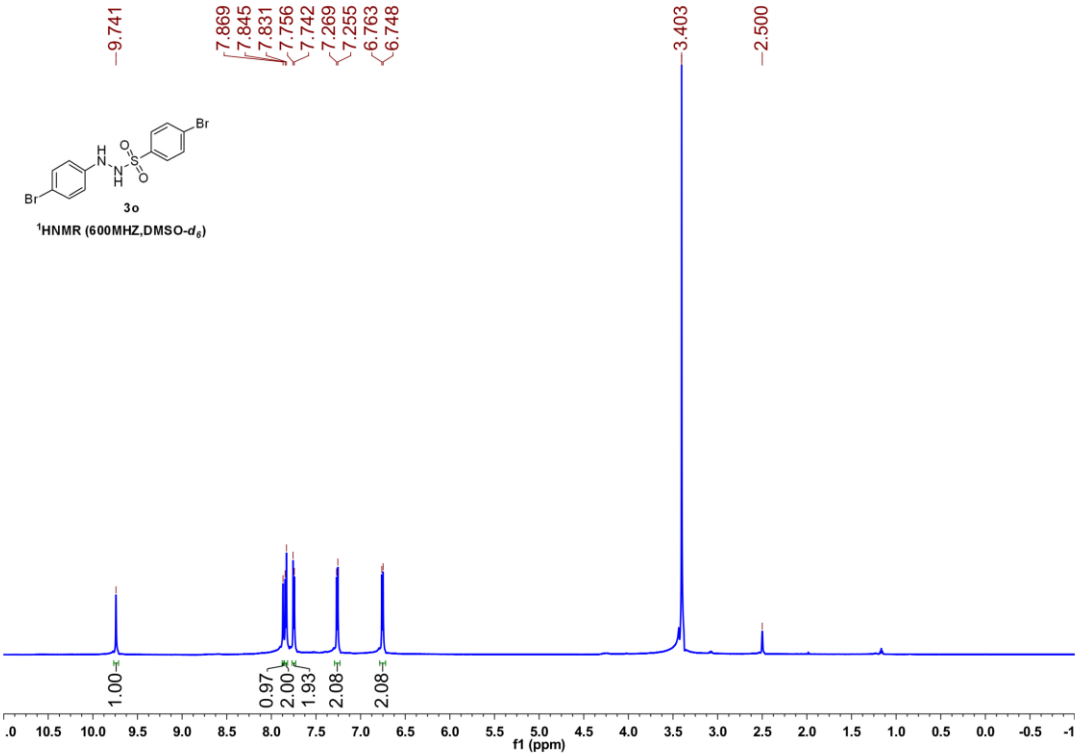

165.616
164.745
152.634
142.654
133.725
130.527
130.007
128.086
119.897
111.621
61.467
59.966
39.915
39.778
39.639
39.500
39.361
39.222
39.083
14.315
14.116
COOEt
EtOOC
3l
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

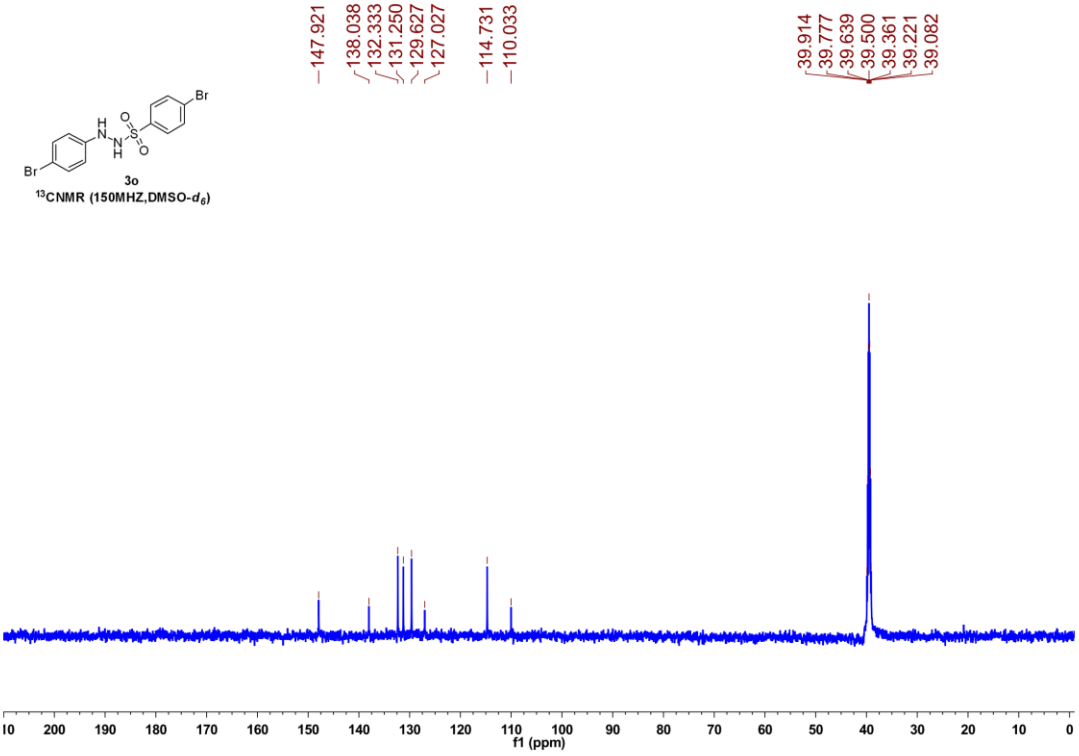

9.640
9.638
7.911
7.902
7.897
7.888
7.657
7.656
7.473
7.459
7.444
6.967
6.952
6.937
6.815
6.807
6.804
6.800
6.792
3.382
2.505
0.000
3m
1HNMR (600MHZ,DMSO-d6)
0.95
1.96
1.00
2.01
2.08
1.98
f1 (ppm)

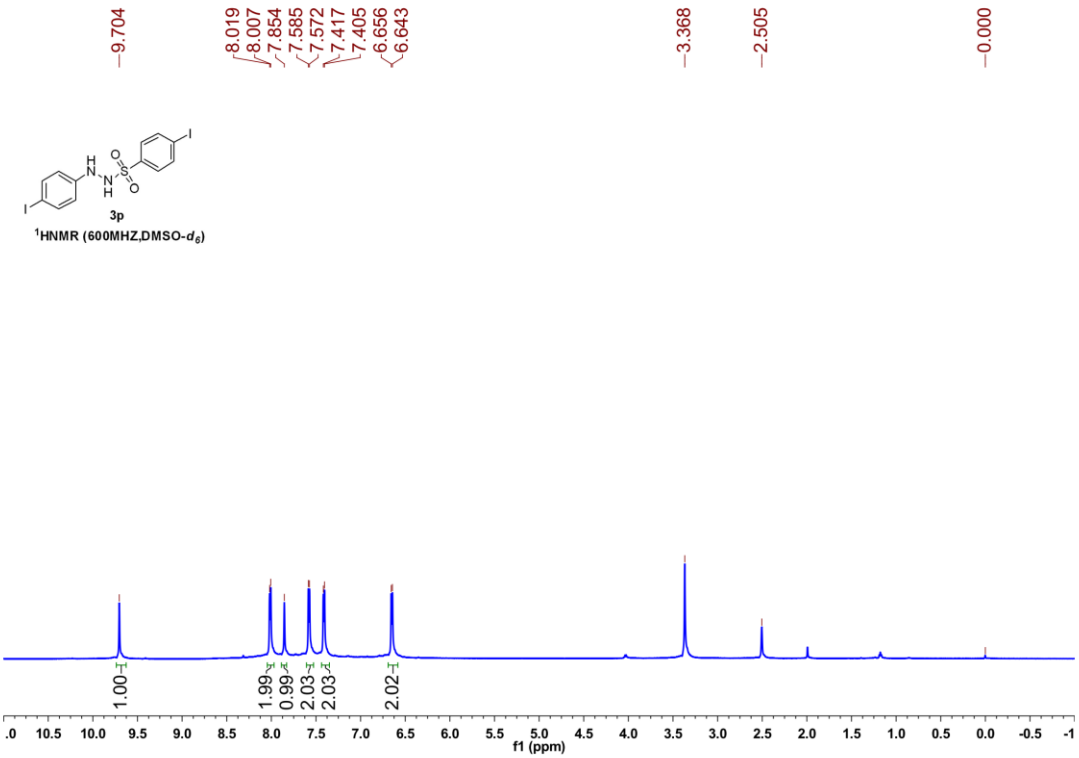

165.374
163.706
156.866
155.315
145.008
135.335
130.657
130.596
116.407
116.258
115.095
114.947
114.033
39.916
39.778
39.639
39.500
39.361
39.222
39.082
3m
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

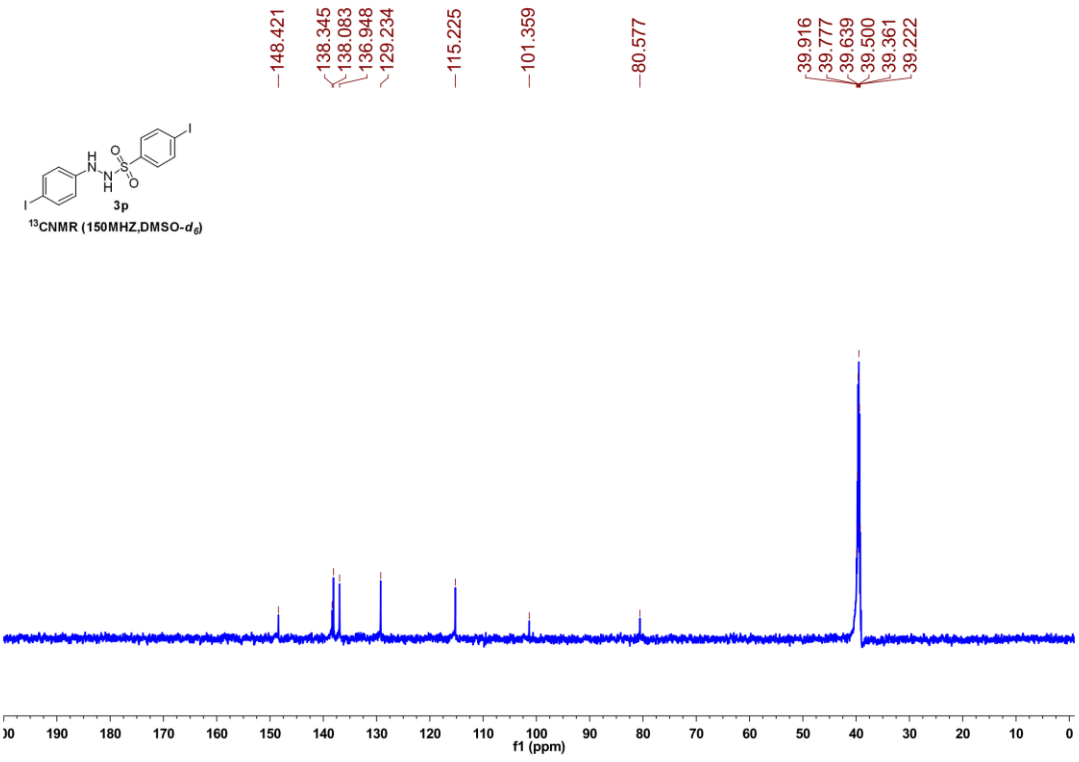

9.736
7.860
7.839
7.825
7.709
7.695
7.155
7.140
6.810
6.795
3.358
2.504
Cl
Cl
3n
1HNMR (600MHZ,DMSO-d6)
0.97
0.93
1.99
2.08
2.07
2.00
f1 (ppm)

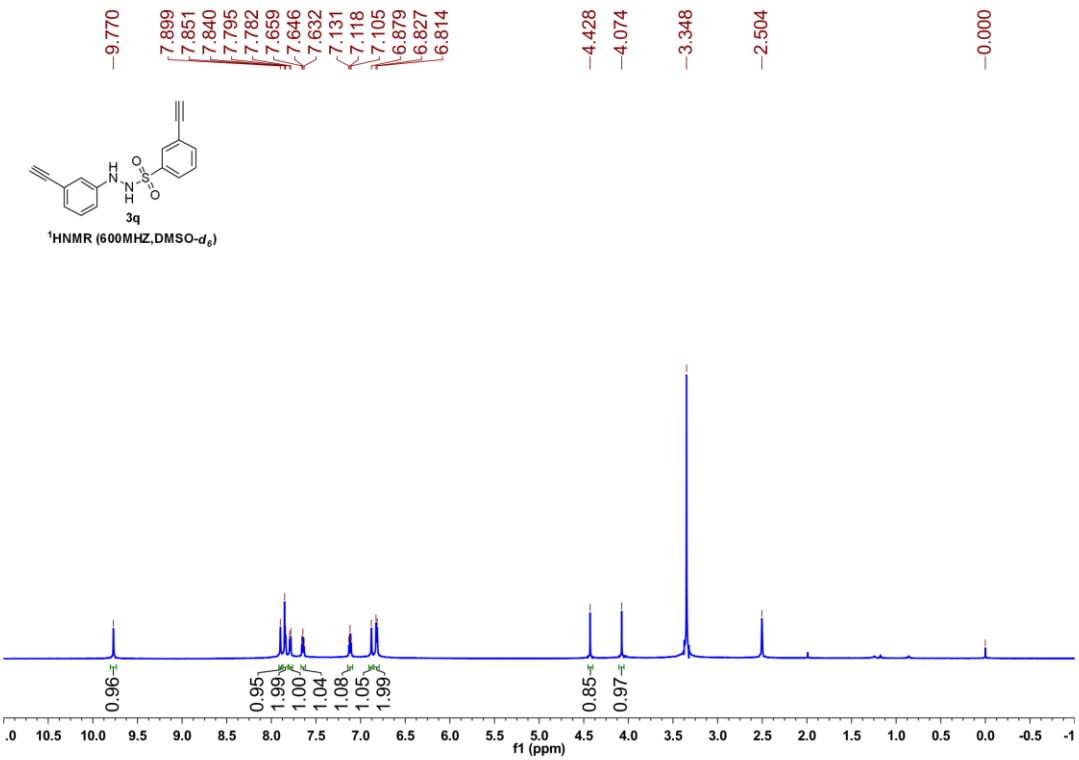

147.468
137.926
137.637
129.453
129.308
128.433
122.412
114.063
39.914
39.777
39.639
39.500
39.361
39.223
39.083
Cl
Cl
3n
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

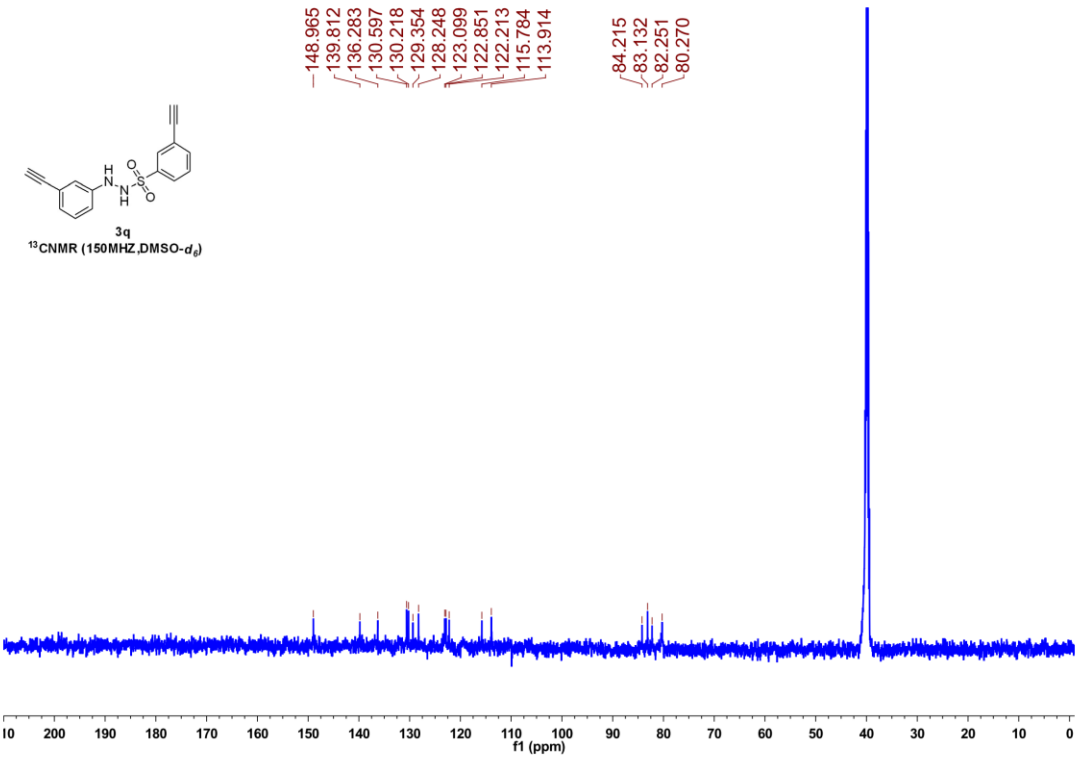

9.741
7.869
7.845
7.831
7.756
7.742
7.269
7.255
6.763
6.748
3.403
2.500
Br
Br
3o
1HNMR (600MHZ,DMSO-d6)
1.00
0.97
2.00
1.93
2.08
2.08
f1 (ppm)

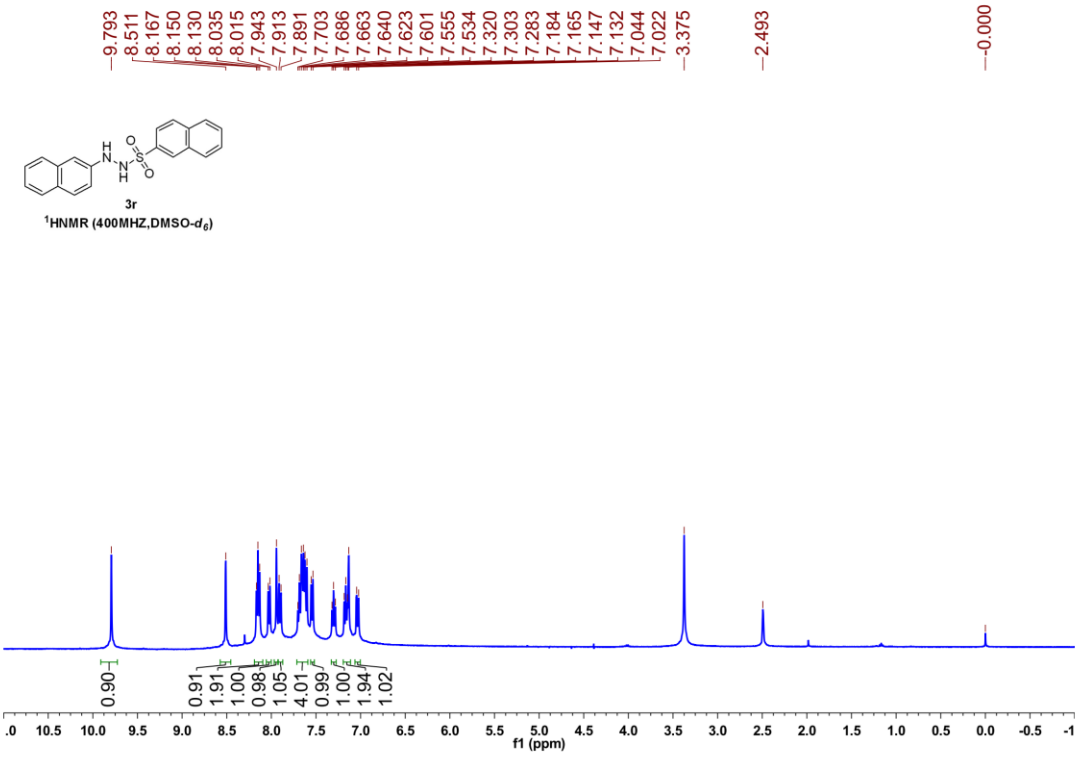

147.921
138.038
132.333
131.250
129.627
127.027
114.731
110.033
39.914
39.777
39.639
39.500
39.361
39.221
39.082
Br
Br
3o
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

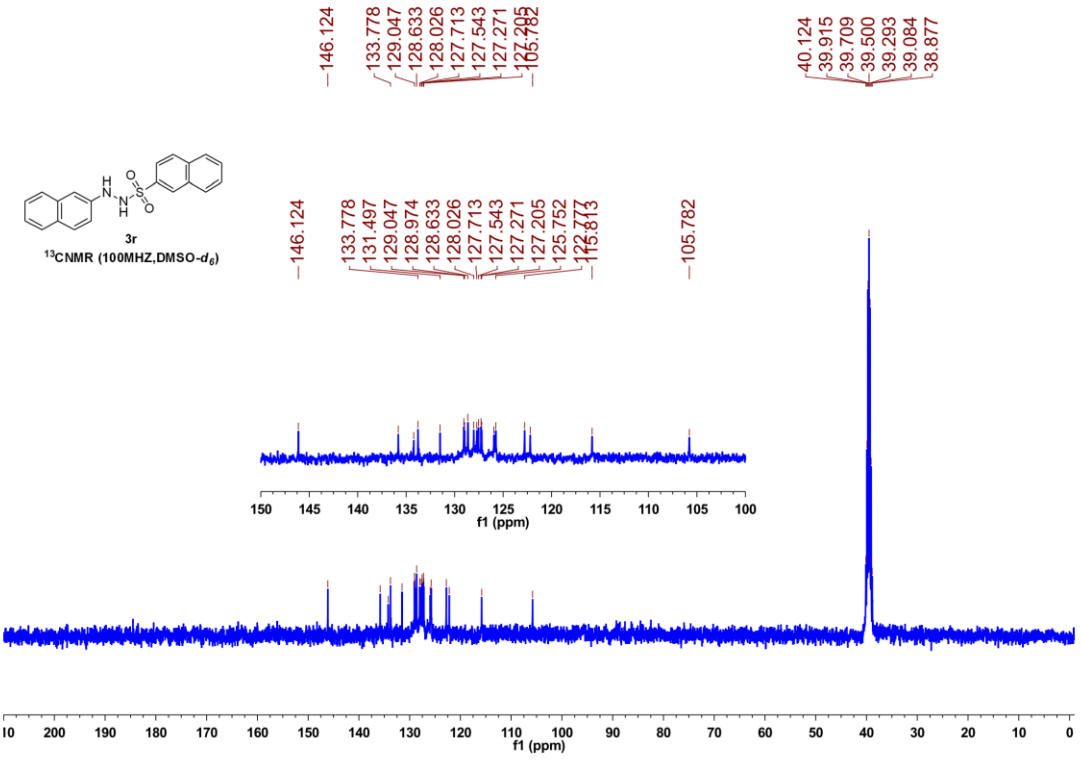

9.704
8.019
8.007
7.854
7.585
7.572
7.417
7.405
6.656
6.643
3.368
2.505
0.000
3p
1HNMR (600MHZ,DMSO-d6)
1.00
1.99
0.99
2.03
2.03
2.02
f1 (ppm)

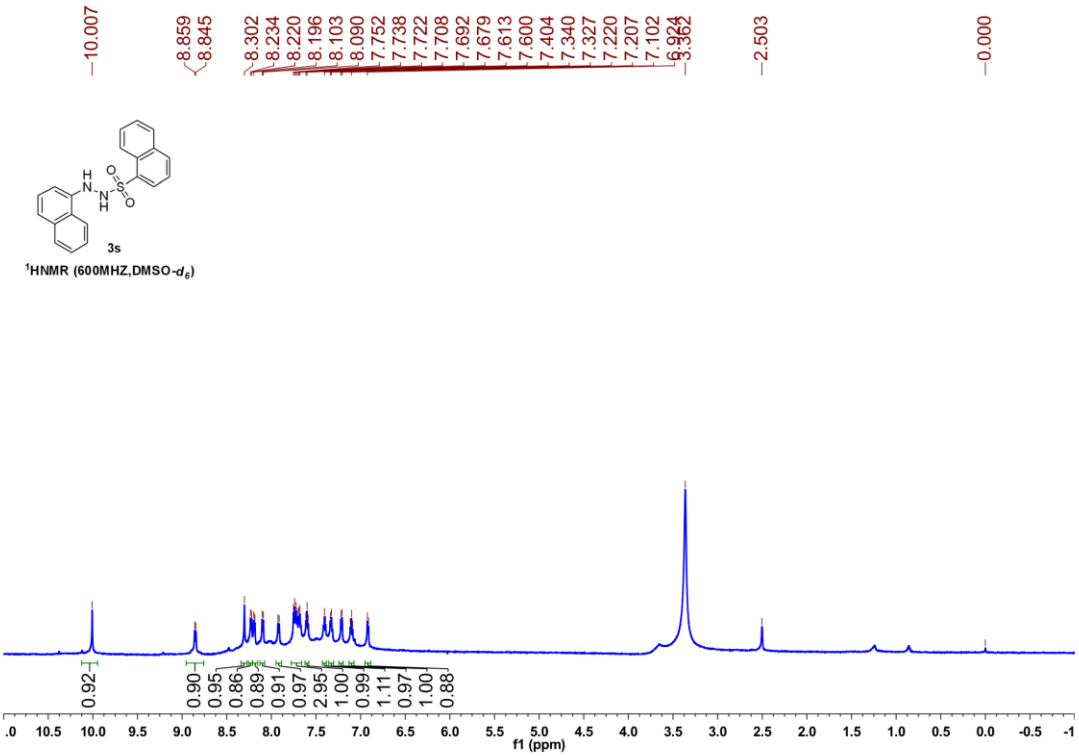

148.421
138.345
138.083
136.948
129.234
115.225
101.359
80.577
39.916
39.777
39.639
39.500
39.361
39.222
3p
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

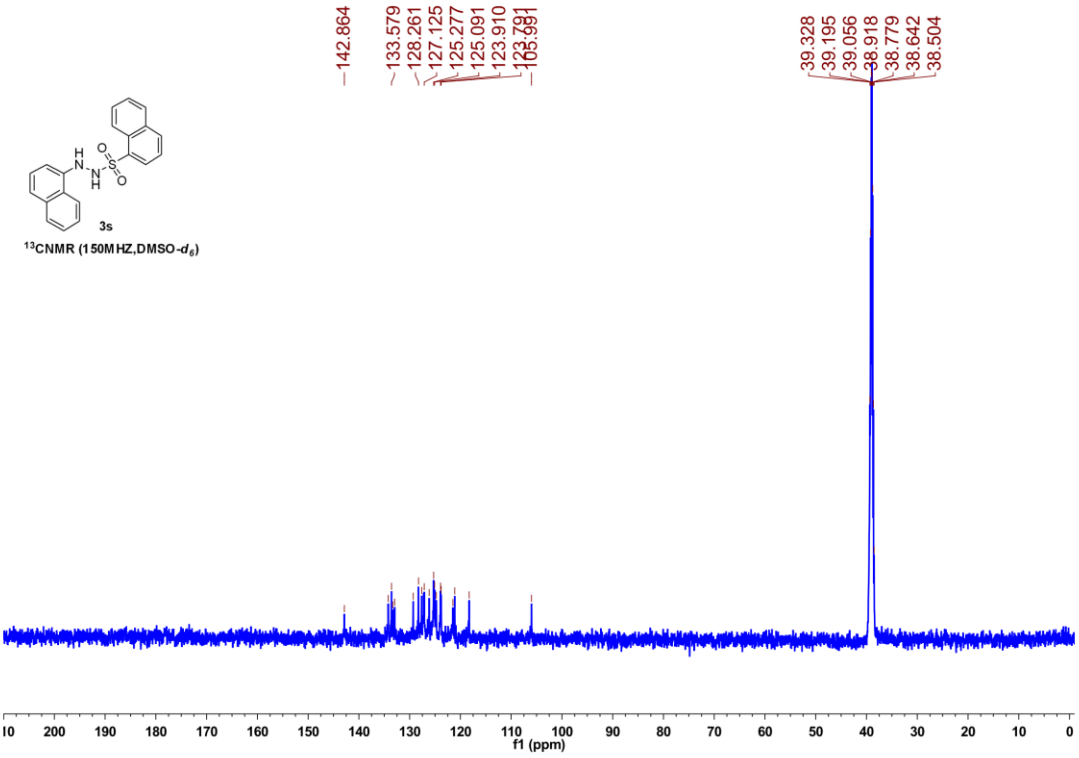

9.770
7.899
7.851
7.840
7.795
7.782
7.659
7.646
7.632
7.131
7.118
7.105
6.879
6.827
6.814
4.428
4.074
3.348
2.504
0.000
3q
1HNMR (600MHZ,DMSO-d6)
0.96
0.95
1.99
1.00
1.04
1.08
1.05
1.99
0.85
0.97
f1 (ppm)


148.965
139.812
136.283
130.597
130.218
129.354
128.248
123.099
122.851
122.213
115.784
113.914
84.215
83.132
82.251
80.270
3q
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)


9.793
8.511
8.167
8.150
8.130
8.035
8.015
7.943
7.913
7.891
7.703
7.686
7.663
7.640
7.623
7.601
7.555
7.534
7.320
7.303
7.283
7.184
7.165
7.147
7.132
7.044
7.022
3.375
2.493
0.000
3r
1HNMR (400MHZ,DMSO-d6)
0.90
0.91
1.91
1.00
0.98
1.05
4.01
0.99
1.00
1.94
1.02
f1 (ppm)

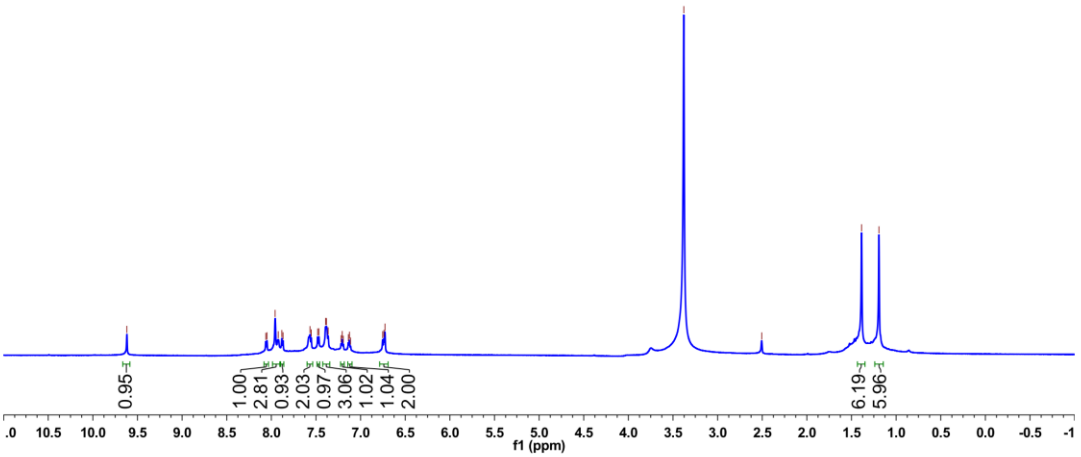

146.124
133.778
129.047
128.633
128.026
127.713
127.543
127.271
105.782
40.124
39.915
39.709
39.500
39.293
39.084
38.877
3r
13CNMR (100MHZ,DMSO-d6)
146.124
133.778
131.497
129.047
128.974
128.633
128.026
127.713
127.543
127.271
127.205
125.752
105.782
f1 (ppm)

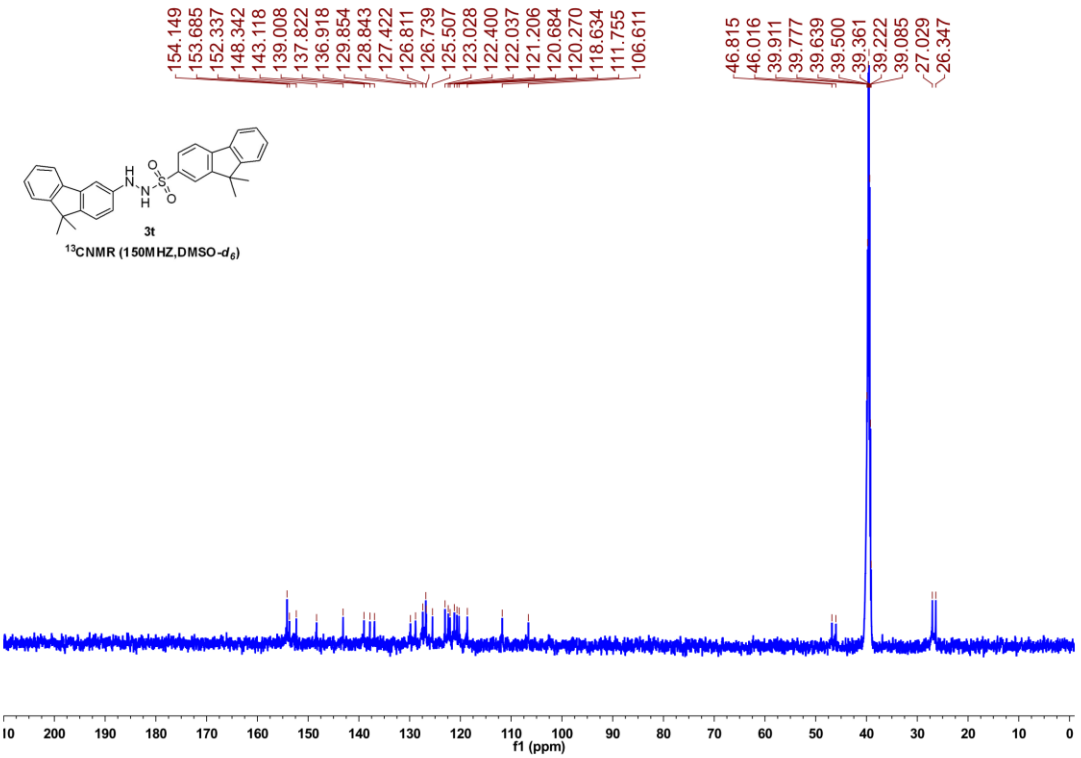

10.007
8.859
8.845
8.302
8.234
8.220
8.196
8.103
8.090
7.752
7.738
7.722
7.708
7.692
7.679
7.613
7.600
7.404
7.340
7.327
7.220
7.207
7.102
6.924
3.362
2.503
0.000
3s
1HNMR (600MHZ,DMSO-d6)
0.92
0.90
0.95
0.86
0.89
0.91
0.97
2.95
1.00
0.99
1.11
0.97
1.00
0.88
f1 (ppm)


142.864
133.579
128.261
127.125
125.277
125.091
123.910
39.328
39.195
39.056
38.918
38.779
38.642
38.504
3s
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

3t

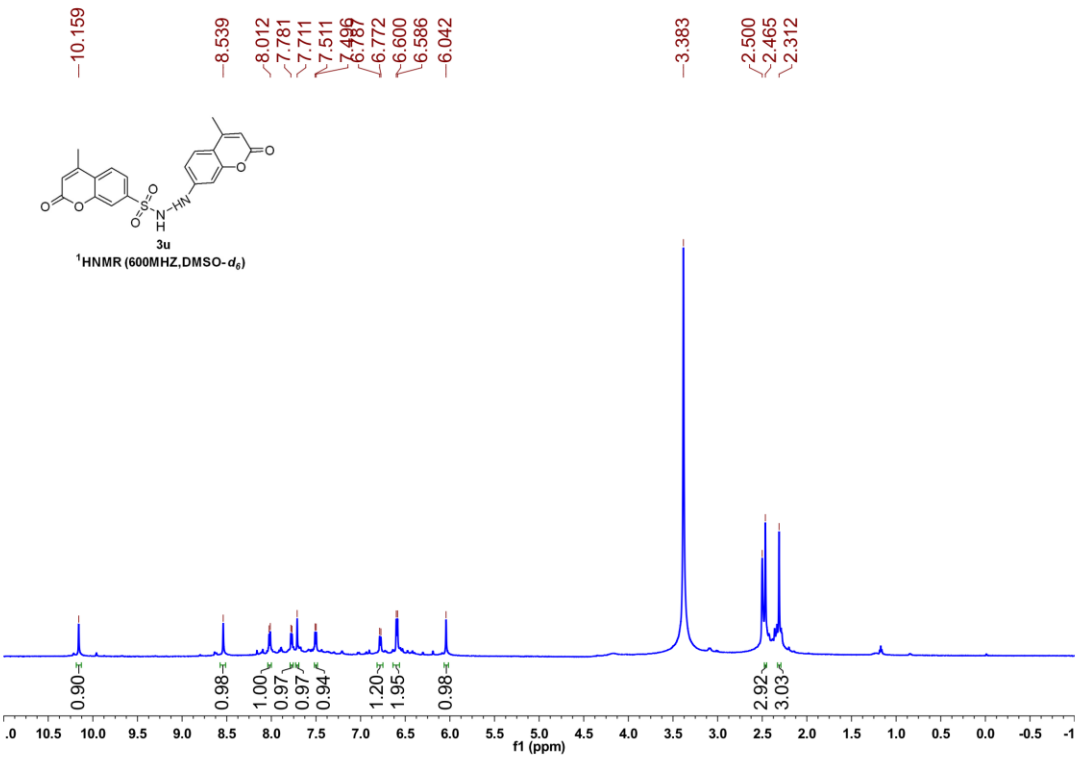
^{1}HNMR (600MHZ,DMSO-d_6)

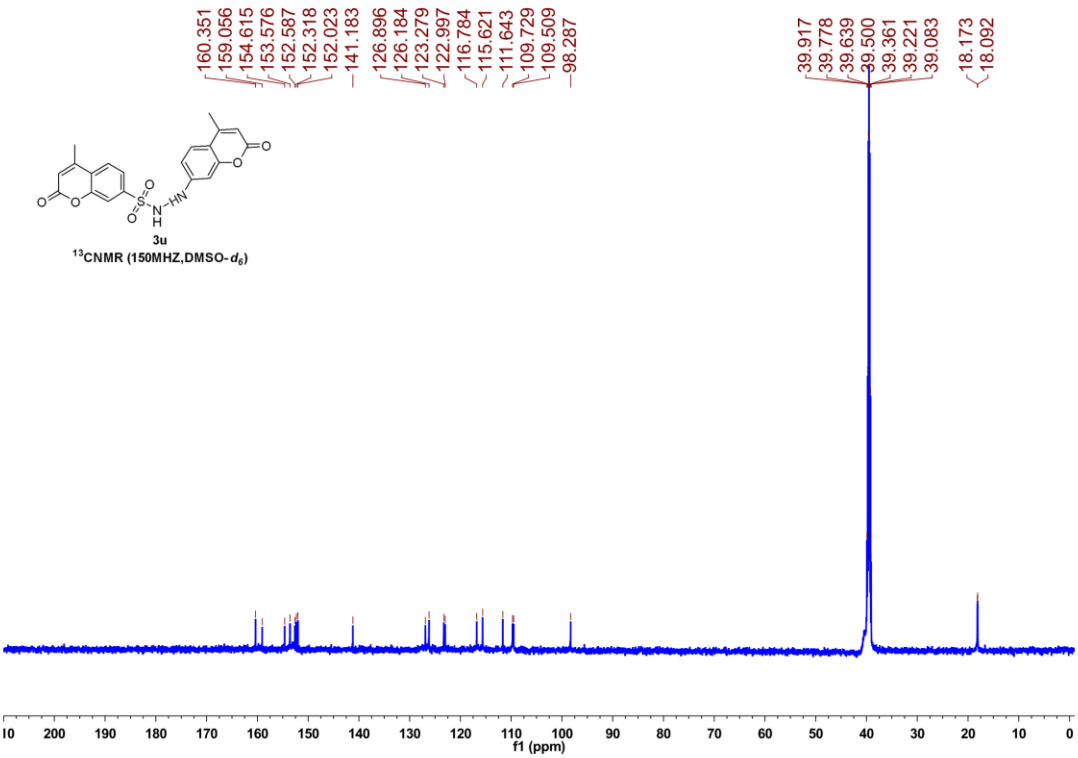


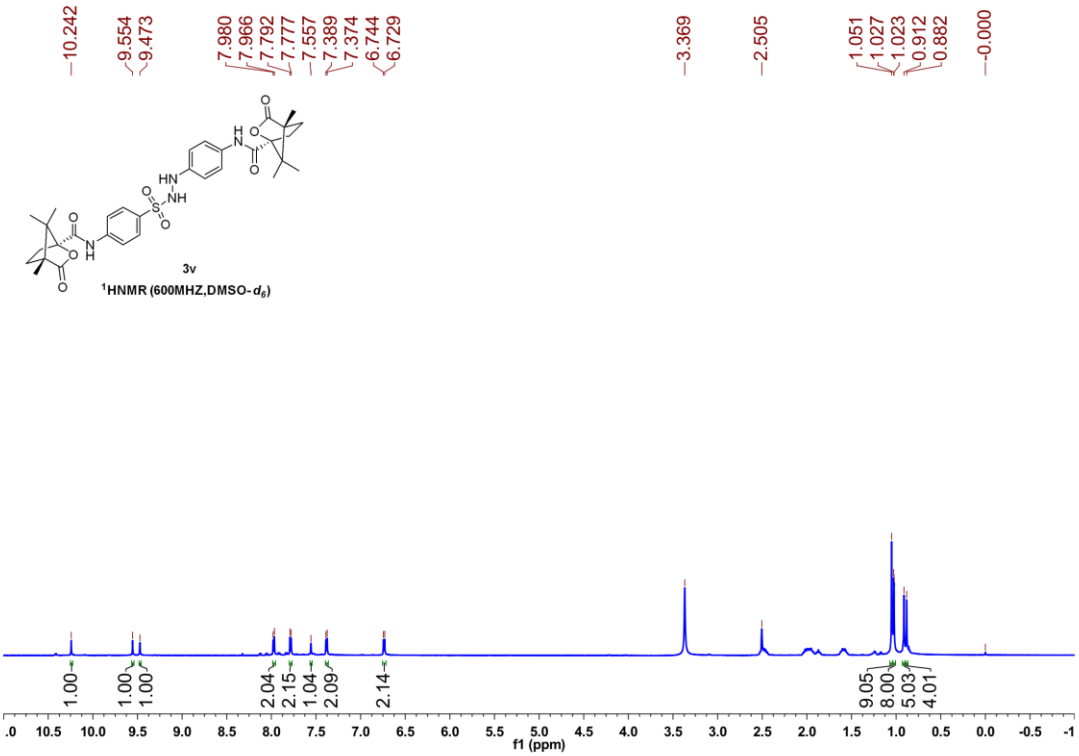


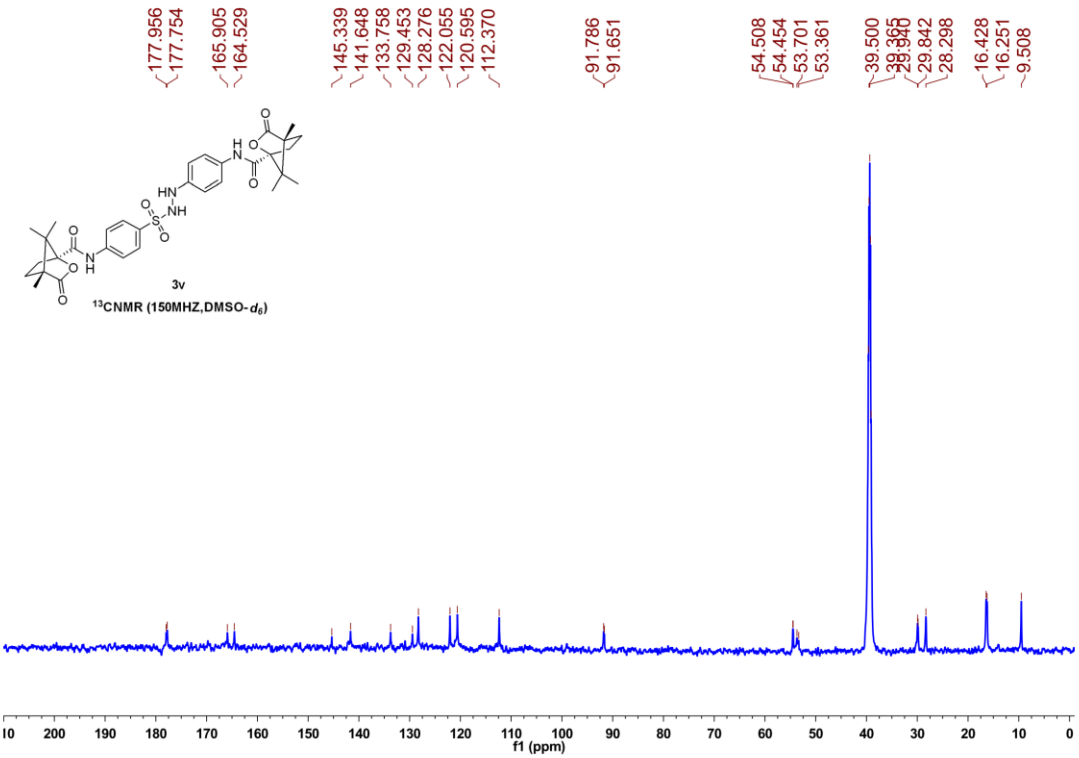
3t

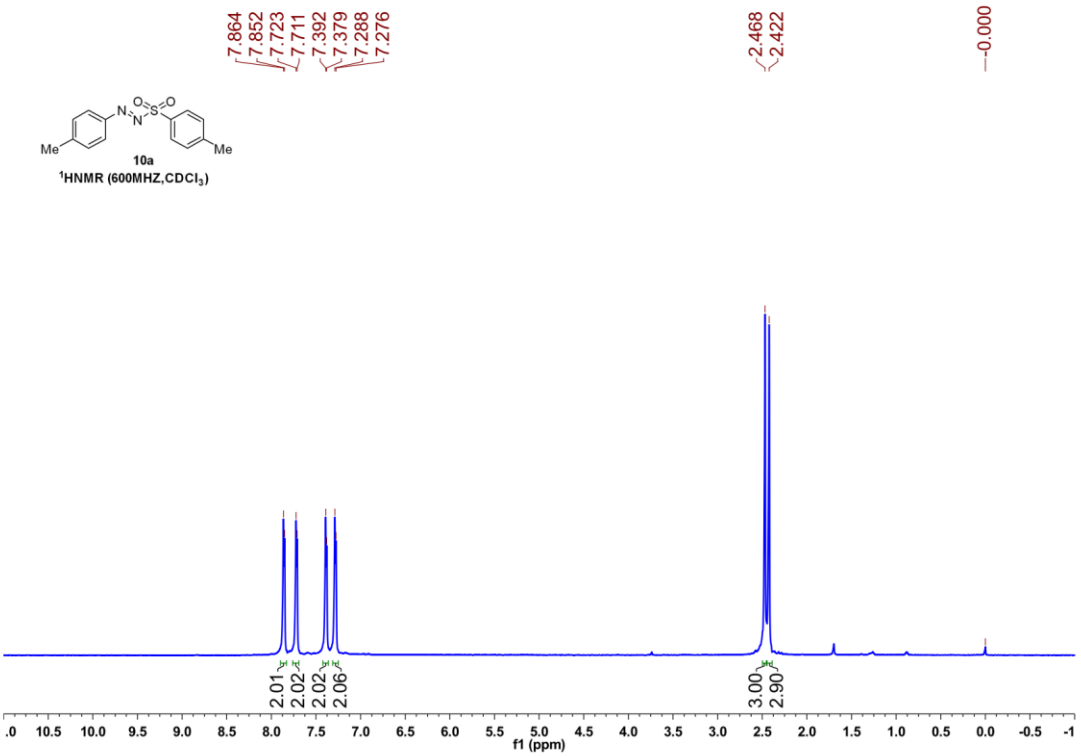
^{13}CNMR (150MHZ,DMSO-d_6)

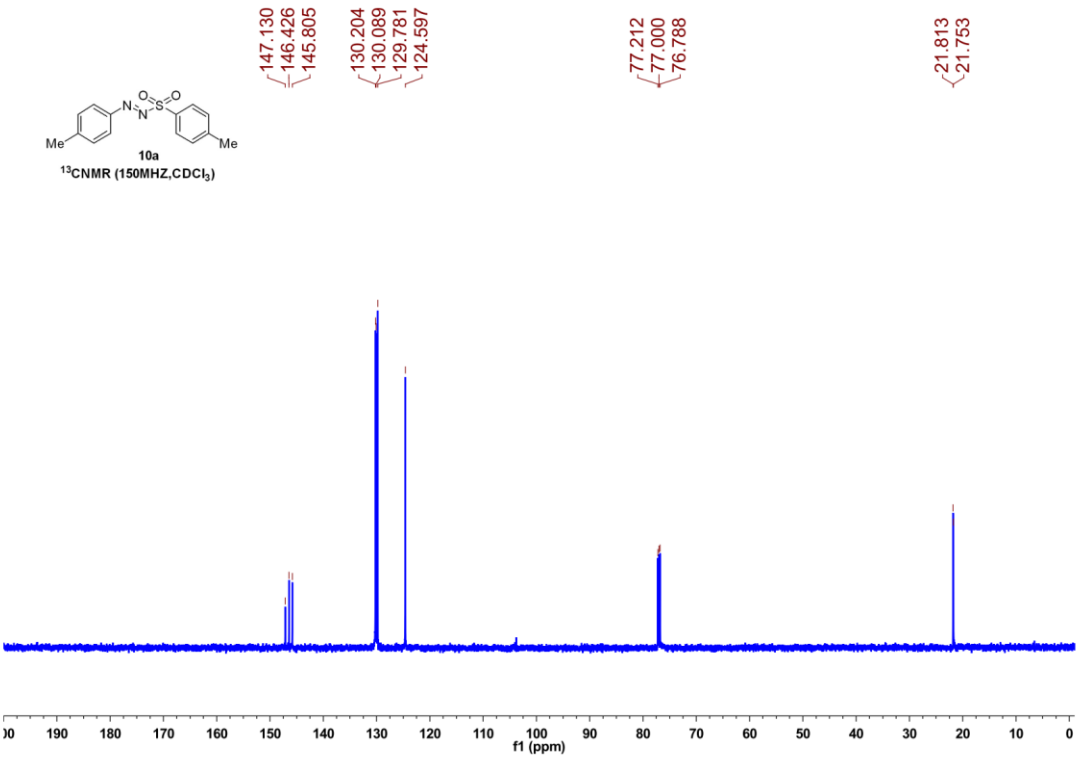

10.159
8.539
8.012
7.781
7.711
7.511
7.496
7.487
6.772
6.600
6.586
6.042
3.383
2.500
2.465
2.312
3u
1HNMR (600MHZ,DMSO-d6)
0.90
0.98
1.00
0.97
0.97
0.94
1.20
1.95
0.98
2.92
3.03
f1 (ppm)

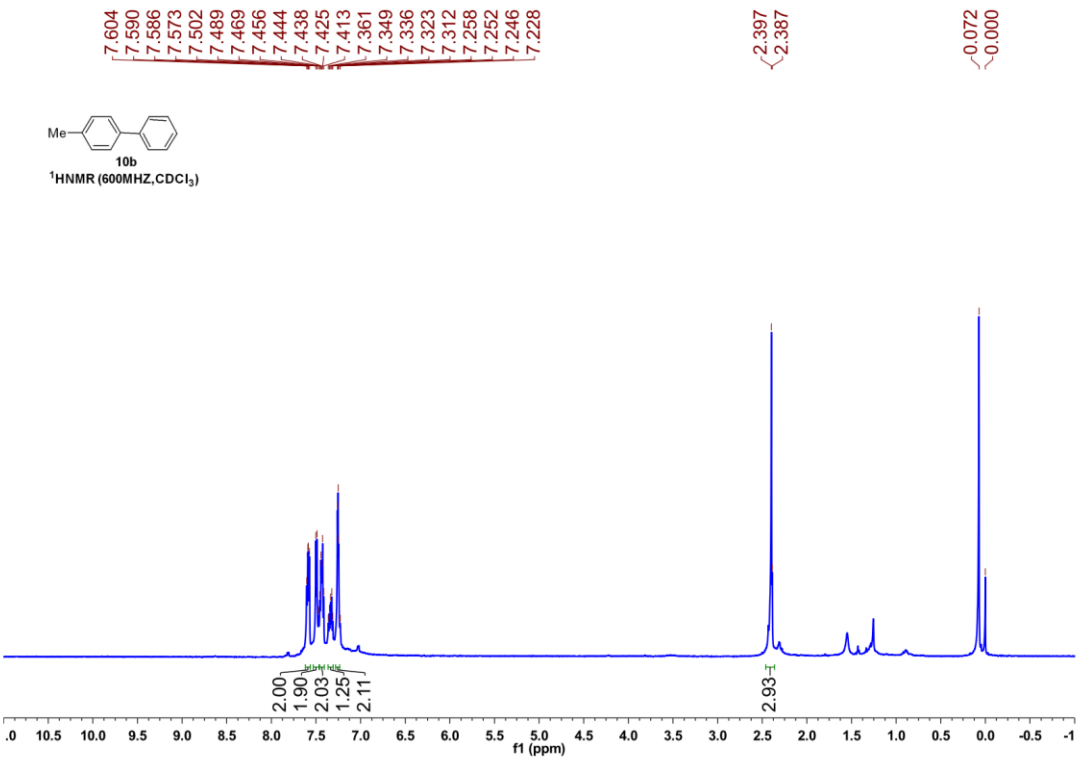

160.351
159.056
154.615
153.576
152.587
152.318
152.023
141.183
126.896
126.184
123.279
122.997
116.784
115.621
111.643
109.729
109.509
98.287
39.917
39.778
39.639
39.500
39.361
39.221
39.083
18.173
18.092
3u
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

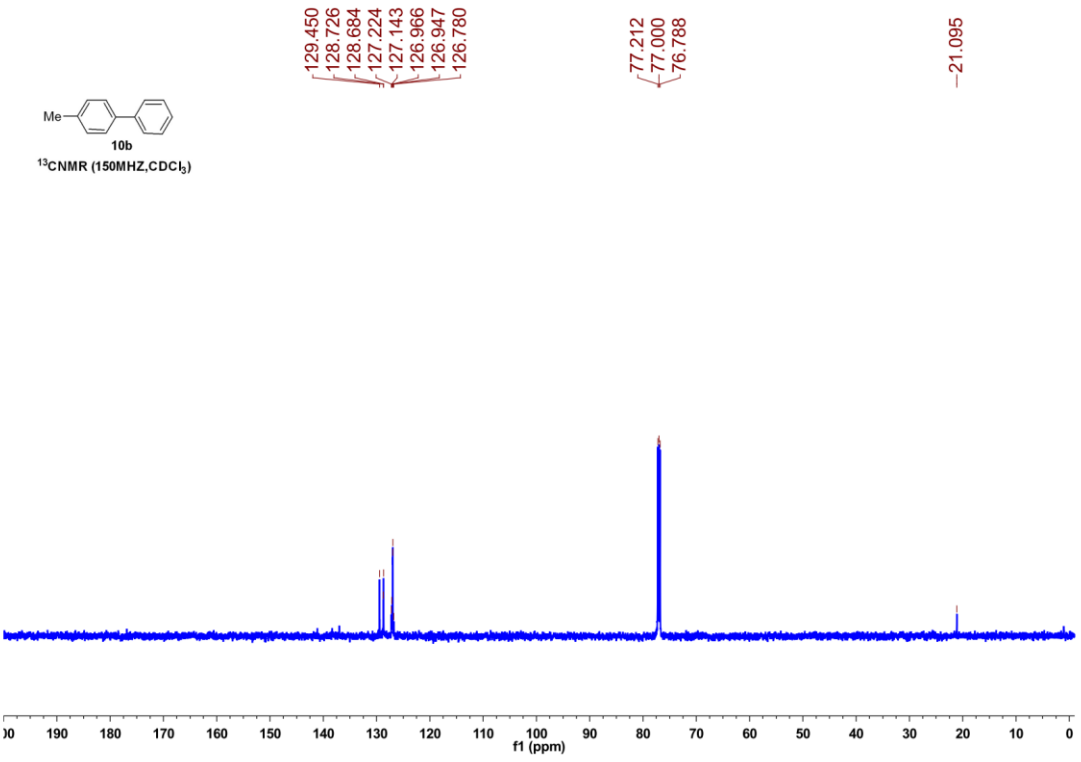

10.242
9.554
9.473
7.980
7.966
7.792
7.777
7.557
7.389
7.374
6.744
6.729
3.369
2.505
1.051
1.027
1.023
0.912
0.882
0.000
3v
1HNMR (600MHZ,DMSO-d6)
1.00
1.00
1.00
2.04
2.15
1.04
2.09
2.14
9.05
8.00
5.03
4.01
f1 (ppm)

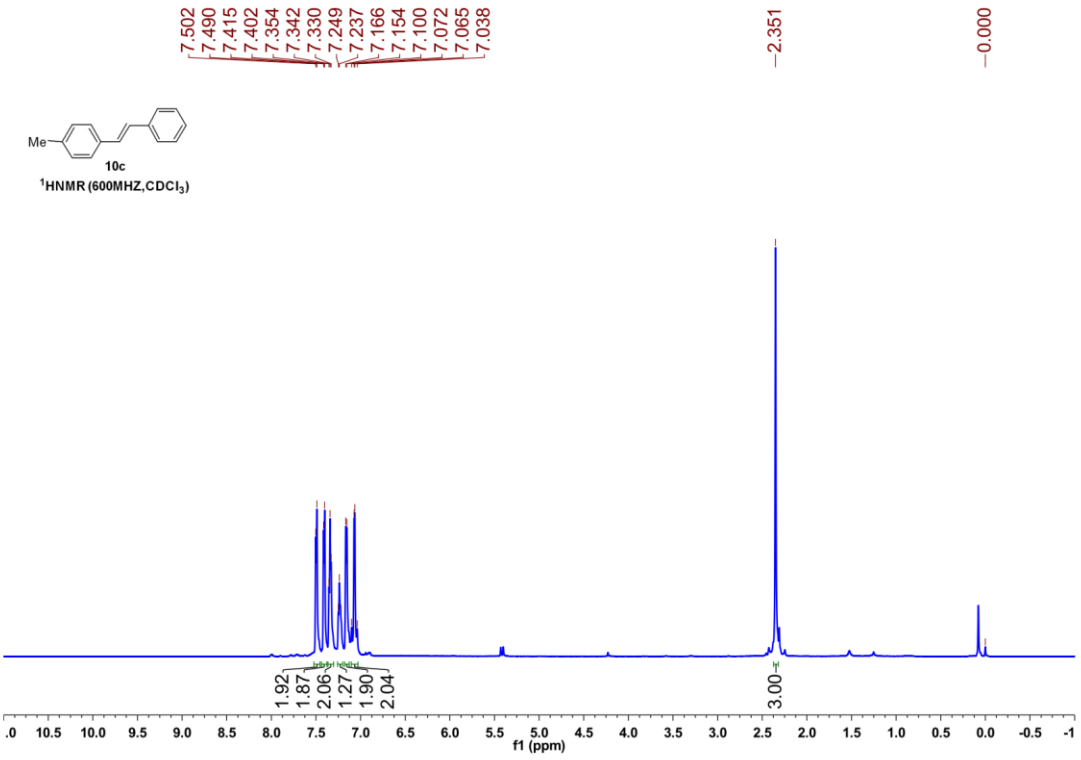

177.956
177.754
165.905
164.529
145.339
141.648
133.758
129.453
128.276
122.055
120.595
112.370
91.786
91.651
54.508
54.454
53.701
53.361
39.500
39.365
29.842
28.298
16.428
16.251
9.508
3v
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)

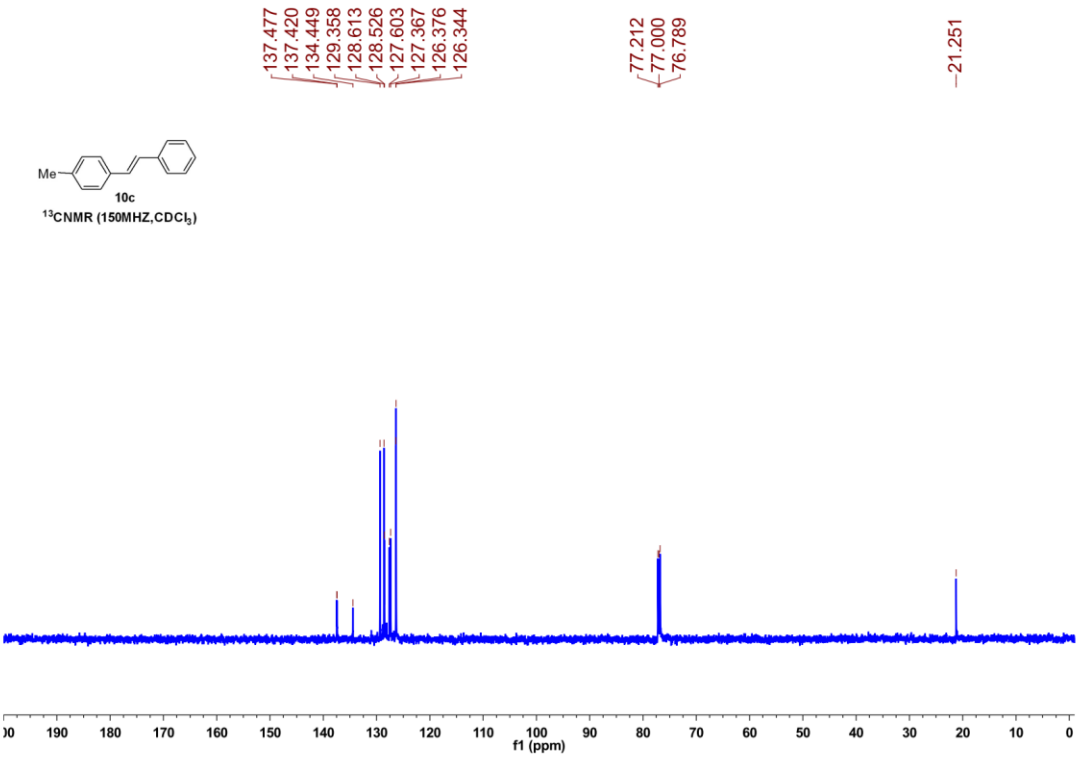

7.864
7.852
7.723
7.711
7.392
7.379
7.288
7.276
2.468
2.422
0.000
Me
Me
10a
1HNMR (600MHZ,CDCl3)
2.01
2.02
2.02
2.06
3.00
2.90
f1 (ppm)

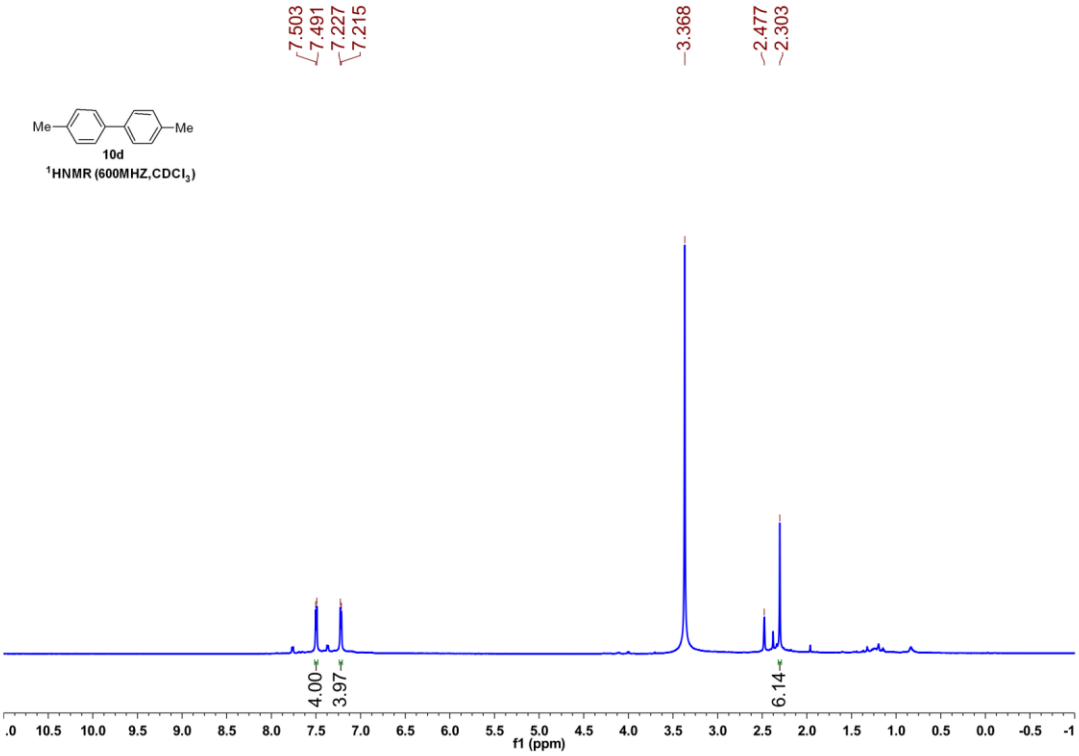

147.130
146.426
145.805
130.204
130.089
129.781
124.597
77.212
77.000
76.788
21.813
21.753
Me
Me
10a
13CNMR (150MHZ,CDCl3)
f1 (ppm)

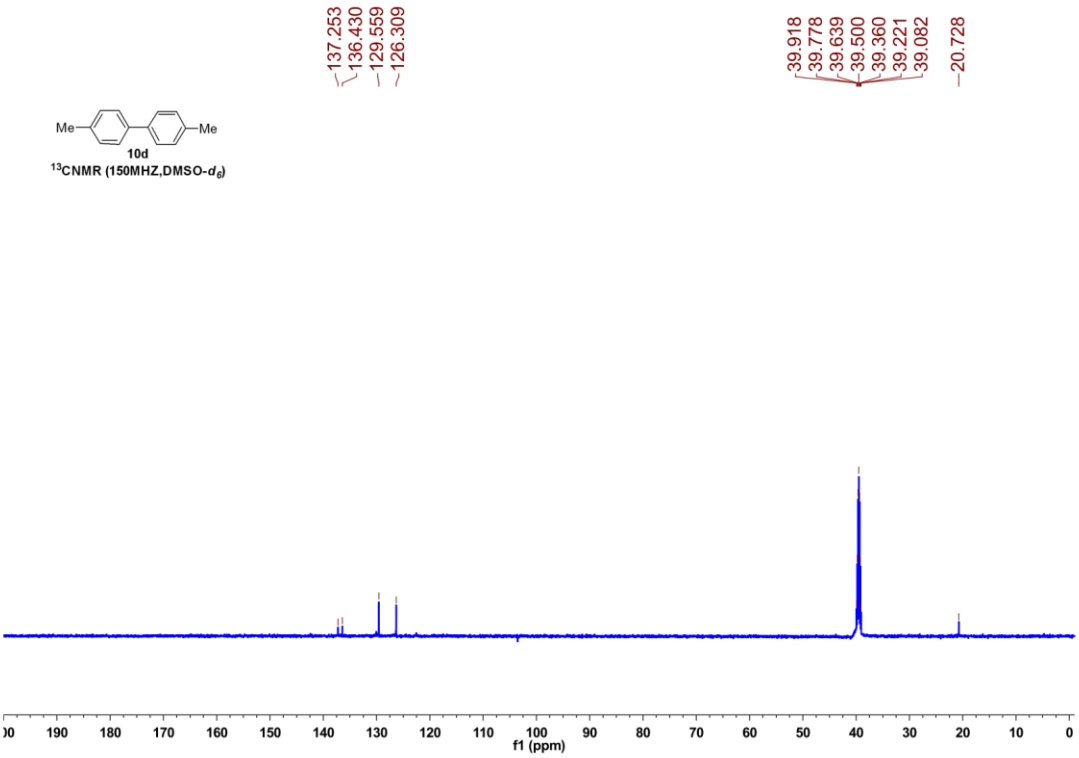

7.604
7.590
7.586
7.573
7.502
7.489
7.469
7.456
7.444
7.438
7.425
7.413
7.361
7.349
7.336
7.323
7.312
7.258
7.252
7.246
7.228
2.397
2.387
0.072
0.000
Me
10b
1HNMR (600MHZ,CDCl3)
2.00
1.90
2.03
1.25
2.11
2.93
f1 (ppm)


129.450
128.726
128.684
127.224
127.143
126.966
126.947
126.780
77.212
77.000
76.788
21.095
Me
10b
13CNMR (150MHZ,CDCl3)
f1 (ppm)


7.502
7.490
7.415
7.402
7.354
7.342
7.330
7.249
7.237
7.166
7.154
7.100
7.072
7.065
7.038
2.351
0.000
Me
10c
1HNMR (600MHZ,CDCl3)
1.92
1.87
2.06
1.27
1.90
2.04
3.00
f1 (ppm)


137.477
137.420
134.449
129.358
128.613
128.526
127.603
127.367
126.376
126.344
77.212
77.000
76.789
21.251
Me
10c
13CNMR (150MHZ,CDCl3)
f1 (ppm)


7.503
7.491
7.227
7.215
3.368
2.477
2.303
Me
Me
10d
1HNMR (600MHZ,CDCl3)
4.00
3.97
6.14
f1 (ppm)


137.253
136.430
129.559
126.309
39.918
39.778
39.639
39.500
39.360
39.221
39.082
20.728
Me
Me
10d
13CNMR (150MHZ,DMSO-d6)
f1 (ppm)