

Supporting Information for:

## **Zinc/PyBisulidine Catalyzed Asymmetric Mannich Reaction of *N*-Tosyl Imines with 3-Acyloxy-2-Oxindoles**

Zinan Yang,<sup>a</sup> Huakang He,<sup>a</sup> Rui Tian,<sup>a,b</sup> Ruoran Wu,<sup>a</sup> Sheng Hu,<sup>a,c</sup> Yue Wu<sup>a,\*</sup> and Hui Zhou<sup>a,\*</sup>

*hzhou@cqmu.edu.cn (H. Zhou); wuyue511@cqmu.edu.cn (Y. Wu)*

<sup>a</sup> *Chongqing Research Center for Pharmaceutical Engineering, College of Pharmacy, Chongqing Medical University, Chongqing 400016, China*

<sup>b</sup> *Pharmaceutical Teaching Laboratory Center, Chongqing Medical University, Chongqing, China*

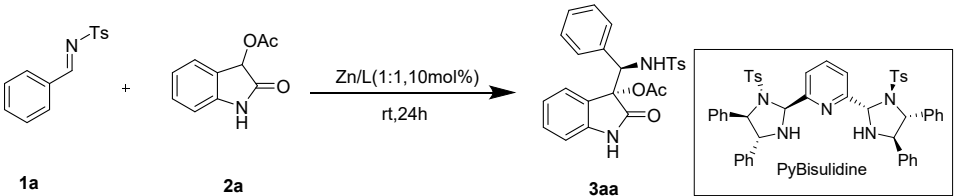
<sup>c</sup> *Department of hepatobiliary surgery, Chongqing 4th hospital, Chongqing, China*

### **CONTENTS**

1. Other optimizations of the Mannich reaction .....	S2
2. Crystal data and structure refinement .....	S4
3. Copies of HPLC.....	S6
4. Copies of NMR Spectra.....	S36

## 1. Other optimizations of the Mannich reaction

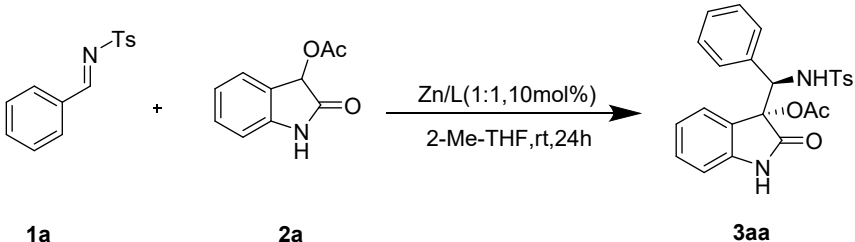
**Table S1:** The effects of different solvents.<sup>a</sup>



Entry	Solvent	Yield (%) <sup>b</sup>	<i>dr</i> <sup>c</sup>	<i>ee</i> (%) <sup>d</sup>
1	2-Me-THF	50	15:1	97
2	THF	34	15:1	97
3	AcOEt	38	24:1	98
4	Toluene	37	25:1	96
5	CH <sub>2</sub> Cl <sub>2</sub>	29	4:1	71
6	1,4-dioxane	25	22:1	95

<sup>a</sup>All reactions were performed with Zn(acac)<sub>2</sub>/L (1:1, 10 mol%), **1a** (0.1 mmol), **2a** (0.1 mmol), solvent (1 mL), at rt for 24 h. <sup>b</sup>Isolated yield. <sup>c</sup>Determined by HPLC. <sup>d</sup>Enantiomeric excess values of major diastereomers; Determined by chiral HPLC.

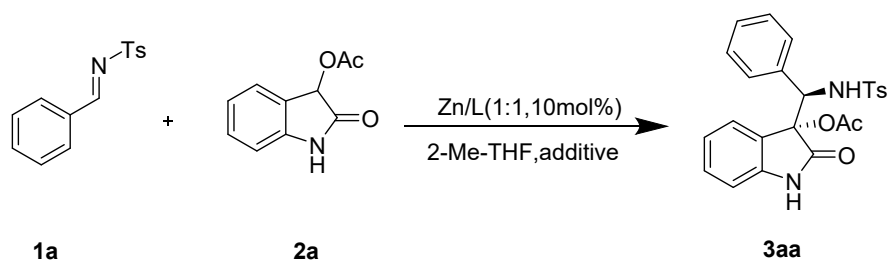
**Table S2:** Effects of the temperature and the ratio of **1a/2a**.<sup>a</sup>



Entry	The amount of <b>1a</b> (mmol)	The amount of <b>2a</b> (mmol)	Yield (%) <sup>b</sup>	<i>dr</i> <sup>c</sup>	<i>ee</i> (%) <sup>d</sup>
1	0.1	0.12	55	15:1	98
2	0.1	0.14	44	13:1	99
3	0.1	0.2	66	10:1	97
4	0.12	0.1	40	8:1	98
5	0.14	0.1	43	9:1	98
6	0.2	0.1	30	13:1	97
7 <sup>e</sup>	0.1	0.12	40	12:1	99
8 <sup>f</sup>	0.1	0.12	58	10:1	94

<sup>a</sup> Unless otherwise noted, the reactions were performed with Zn(acac)<sub>2</sub>/L (1:1, 10 mol%), 2-Me-THF (1 mL), at rt for 48 h. <sup>b</sup>Isolated yield. <sup>c</sup>Determined by HPLC. <sup>d</sup>Enantiomeric excess values of major diastereomers; Determined by chiral HPLC. <sup>e</sup>At 0 °C. <sup>f</sup>At 40 °C.

**Table S3:** The effect of additive.<sup>a</sup>

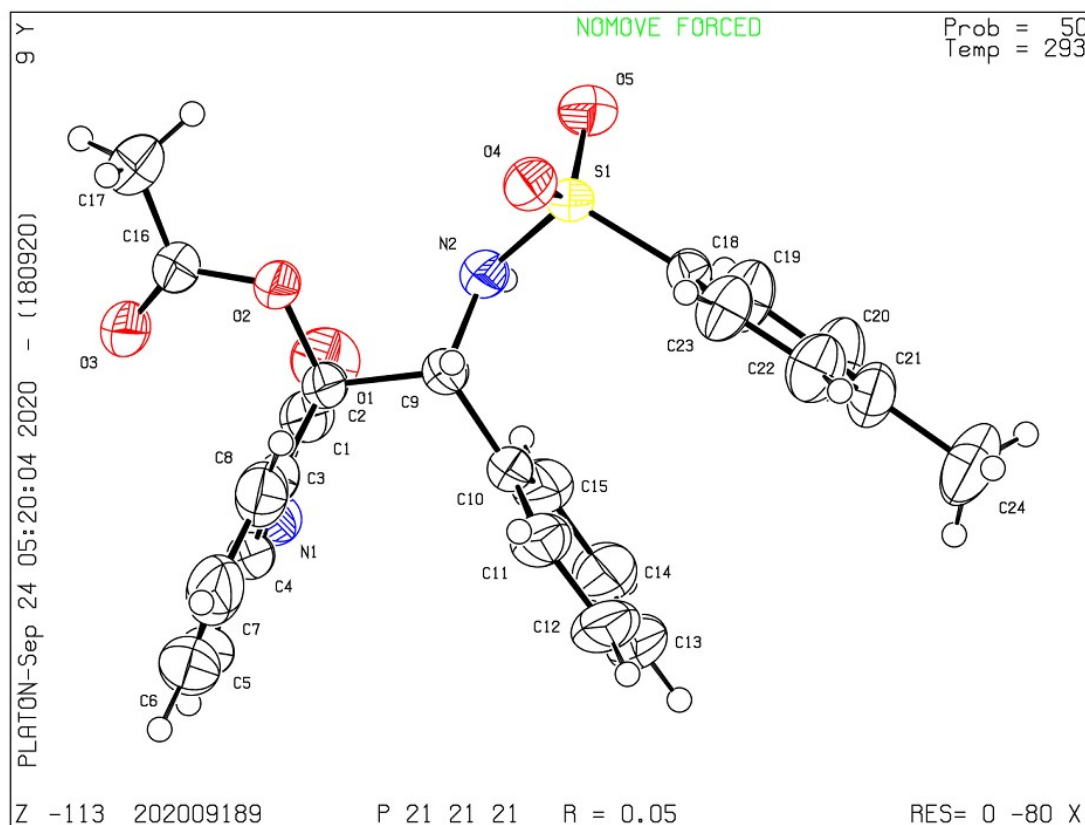


Entry	Additive	The amount of additive	Yield (%) <sup>b</sup>	<i>dr</i> <sup>c</sup>	<i>ee</i> (%) <sup>d</sup>
1	none	-	55	15:1	98
2	<i>N</i> -methyl morpholine	20 mmol%	54	15:1	98
3	DIPEA	20 mmol%	33	15:1	87
4	Et <sub>3</sub> N	20 mmol%	46	7:1	98
5	4-Methylphenol	20 mmol%	39	15:1	98
6	3Å MS	3 mg	54	11:1	98
7	4Å MS	3 mg	40	15:1	97
8	5Å MS	3 mg	72	15:1	99
9	5Å MS	30 mg	73	15:1	99
10 <sup>e</sup>	5Å MS	30 mg	94	15:1	97
11 <sup>e</sup>	5Å MS	45 mg	76	6:1	96

<sup>a</sup>Unless otherwise noted, the reactions were performed with Zn(acac)<sub>2</sub>/L (1:1, 10 mol%), **1a** (0.1 mmol), **2a** (0.12 mmol), 2-Me-THF (1 mL), at rt for 24 h. <sup>b</sup>Isolated yield. <sup>c</sup>Determined by HPLC. <sup>d</sup>Enantiomeric excess values of major diastereomers; Determined by chiral HPLC. <sup>e</sup>At rt for 48 h.

## 2. Crystal data and structure refinement

**Preparation of the single crystals of enantiopure 3aa:** Compound **3aa** (20.0 mg, 20:1 dr, 98% ee) was dissolved in ethyl acetate (0.5 mL), then followed by the slowly addition of petroleum ether until a solid just appeared. Shaked the tube to make the solution clear. The tube was sealed, thus allowing slow evaporation of the solvents at room temperature. After a week, several small particles could be observed at the bottom of the tube. The crystals were chosen and subjected to the single crystal X-ray diffraction analysis for the determination of the absolute configuration of **3aa**. The data were collected by a Rigaku Gemini equipped with a Cu radiation source ( $K\alpha = 1.54184 \text{ \AA}$ ) at 293(2) K. CCDC 2085480 (**3aa**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).



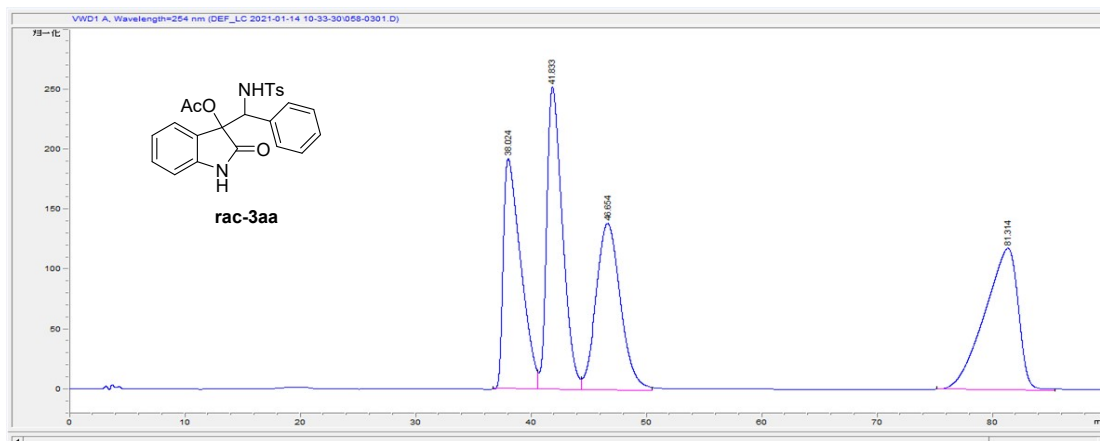
(ellipsoid contour probability 30%)

Table S4. Crystal data and structure refinement for 202009189 (**3aa**).

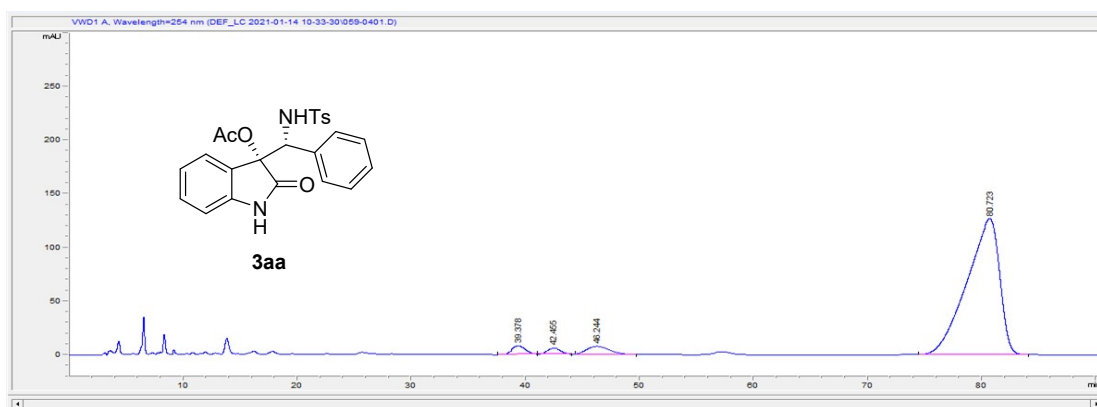
Identification code	202009189
Empirical formula	$C_{24}H_{22}N_2O_5S$
Formula weight	450.49
Temperature/K	293(2)
Crystal system	orthorhombic

Space group	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
a/Å	9.42743(15)
b/Å	9.8509(2)
c/Å	24.0927(4)
$\alpha$ /°	90
$\beta$ /°	90
$\gamma$ /°	90
Volume/Å <sup>3</sup>	2237.46(7)
Z	4
$\rho_{\text{calc}}$ /cm <sup>3</sup>	1.337
$\mu$ /mm <sup>-1</sup>	1.610
F(000)	944.0
Crystal size/mm <sup>3</sup>	0.14 × 0.1 × 0.09
Radiation	CuK $\alpha$ ( $\lambda$ = 1.54184)
2 $\Theta$ range for data collection/°	7.338 to 134.116
Index ranges	-11 ≤ h ≤ 7, -11 ≤ k ≤ 10, -28 ≤ l ≤ 28
Reflections collected	8071
Independent reflections	3985 [ $R_{\text{int}}$ = 0.0367, $R_{\text{sigma}}$ = 0.0559]
Data/restraints/parameters	3985/0/295
Goodness-of-fit on F <sup>2</sup>	1.036
Final R indexes [ $I \geq 2\sigma(I)$ ]	$R_1$ = 0.0499, $wR_2$ = 0.1299
Final R indexes [all data]	$R_1$ = 0.0561, $wR_2$ = 0.1372
Largest diff. peak/hole / e Å <sup>-3</sup>	0.28/-0.25
Flack parameter	0.07(3)

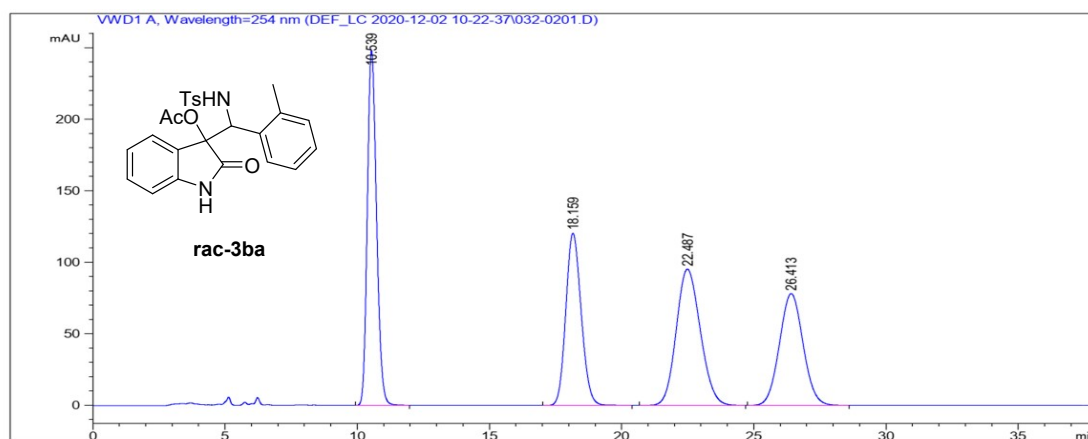
### 3. Copies of HPLC



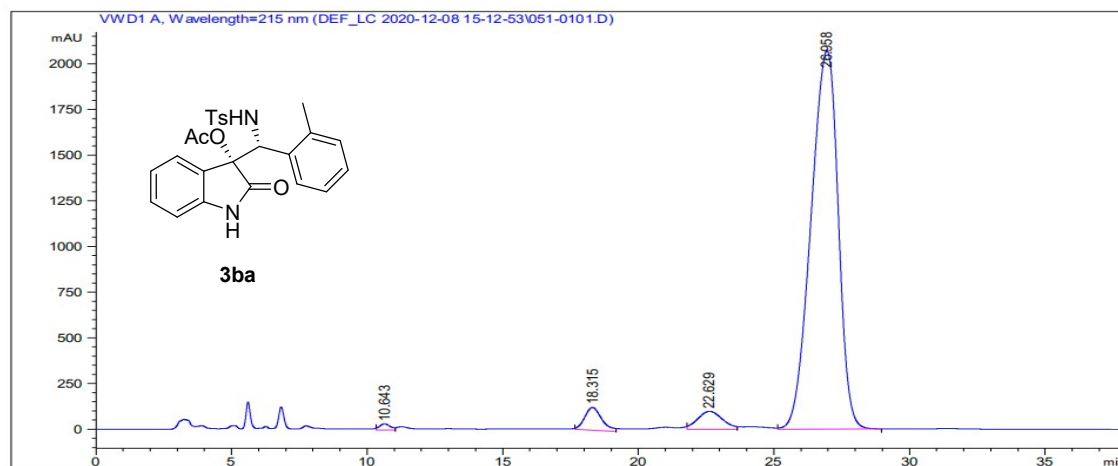
Peak#	Retention Time	Area	Area%
1	38.04	2.00101e4	22.6617
2	41.833	2.37290e4	26.8734
3	46.654	2.05981e4	23.3276
4	81.313	2.39621e4	27.1373



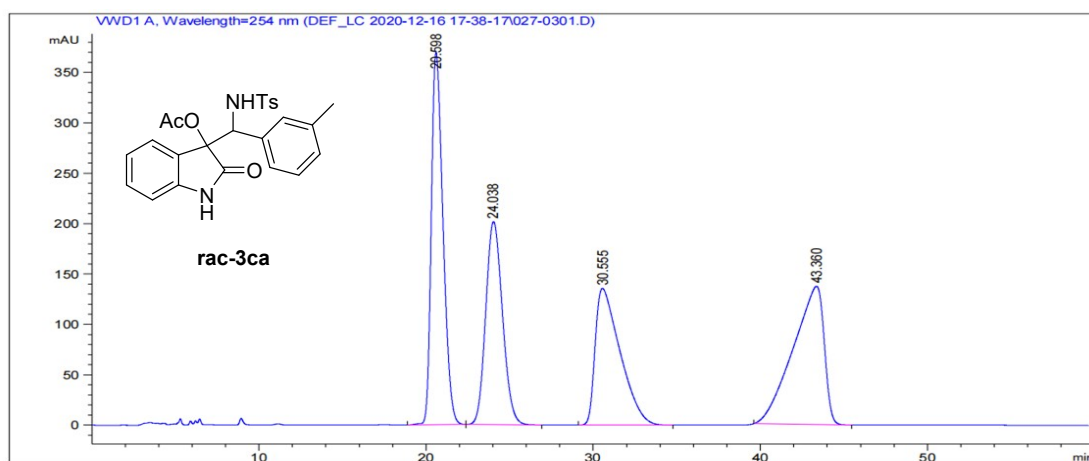
Peak#	Retention Time	Area	Area%
1	39.378	755.83234	2.6972
2	42.455	438.55524	1.5650
3	46.244	1027.25305	3.6658
4	80.723	2.58013e4	92.0721



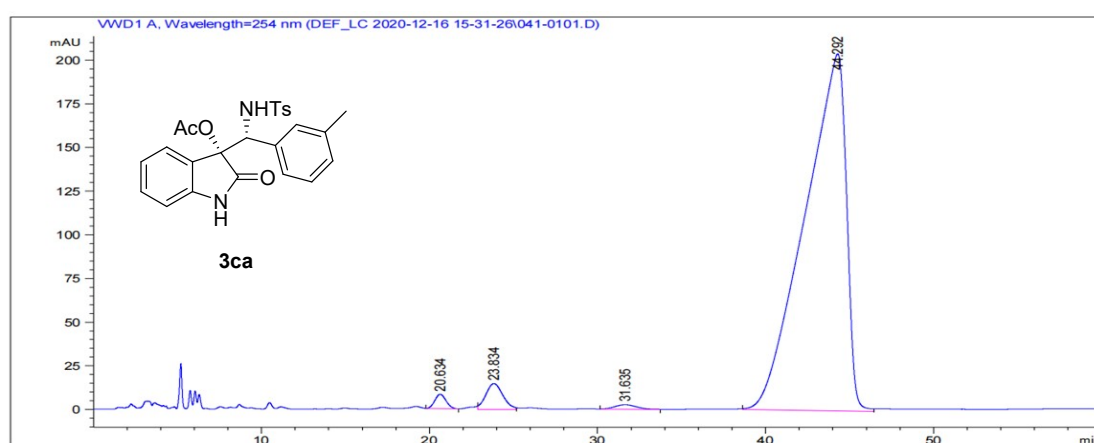
Peak#	Retention Time	Area	Area%
1	10.539	6008.21338	27.6389
2	18.159	4858.50684	22.3500
3	22.487	6024.83691	27.7154
4	26.413	4846.69434	22.2957



Peak#	Retention Time	Area	Area%
1	10.643	933.87533	0.5950
2	18.315	5368.28467	3.4223
3	22.629	6194.30225	3.9489
4	26.958	144364	92.0337

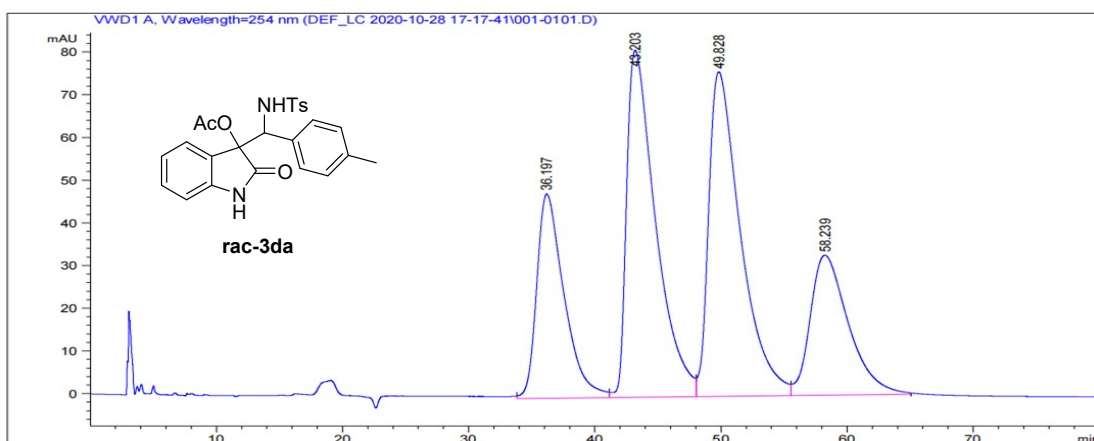


Peak#	Retention Time	Area	Area%
1	11.455	4.90752e4	37.2361
2	14.934	1.62817e4	12.3538
3	22.995	1.68415e4	12.7786
4	40.074	4.95964e4	37.6315

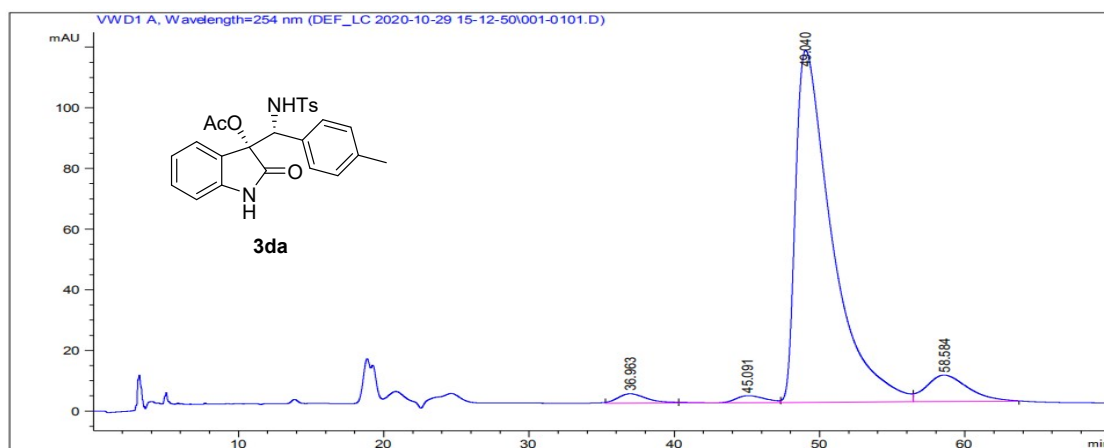


Peak#	Retention Time	Area	Area%
1	11.497	376.01935	0.8786
2	14.705	1038.41614	3.4886
3	22.351	214.11430	9.4882
4	40.733	3.49961e4	86.1445

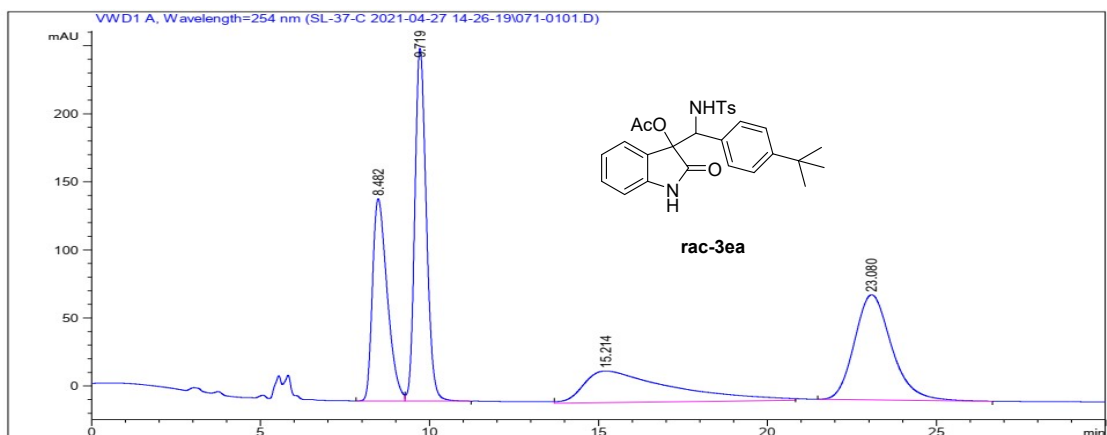




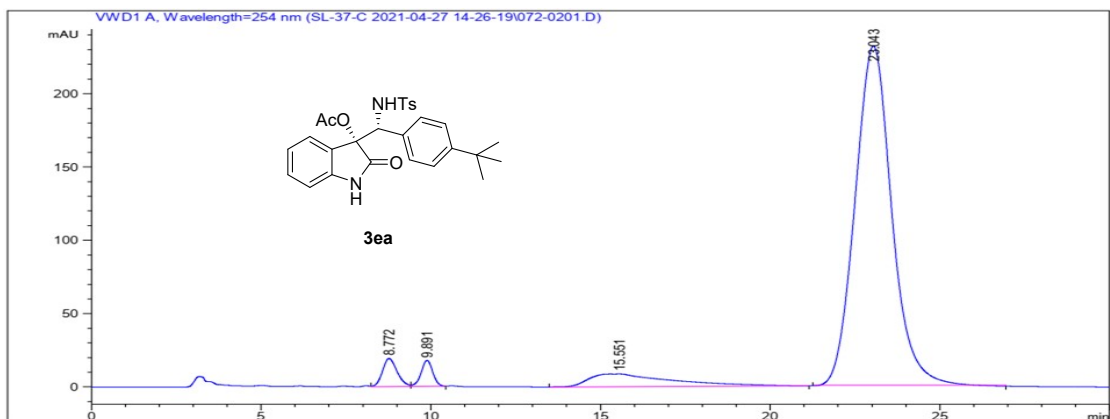
Peak#	Retention Time	Area	Area%
1	36.197	7208.13867	17.8165
2	43.203	1.29935e4	32.1163
3	49.828	1.33438e4	32.9821
4	58.239	6912.24170	17.0851



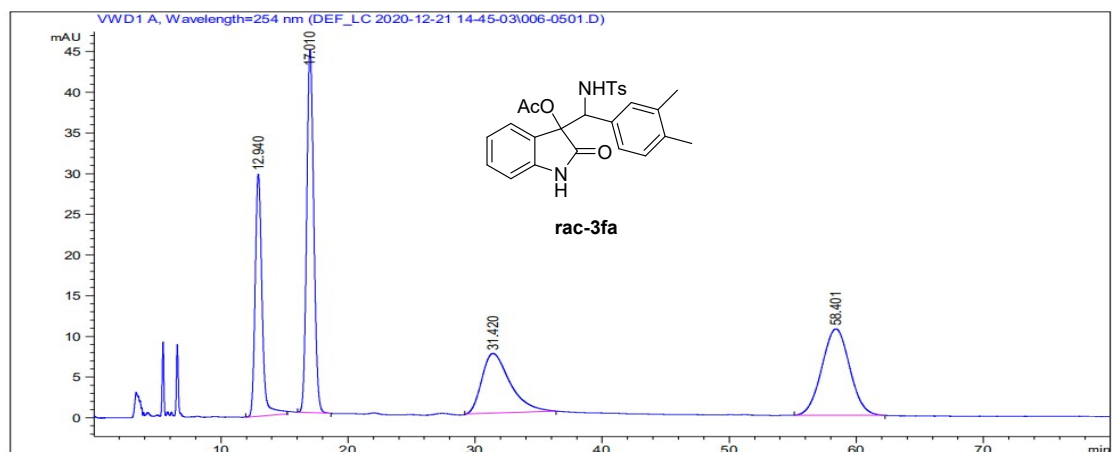
Peak#	Retention Time	Area	Area%
1	36.963	442.07983	1.9516
2	45.091	329.50961	1.4547
3	49.040	2.01339e4	88.8831
4	58.584	1746.61963	7.7106



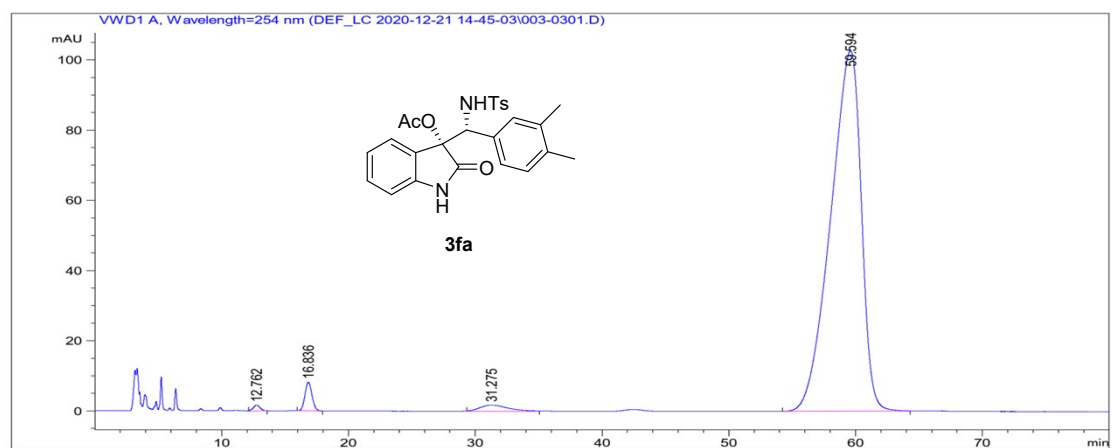
Peak#	Retention Time	Area	Area%
1	8.482	4517.29297	21.8871
2	9.719	6113.34619	29.6203
3	15.214	4123.76074	19.9804
4	23.080	5884.63721	28.5122



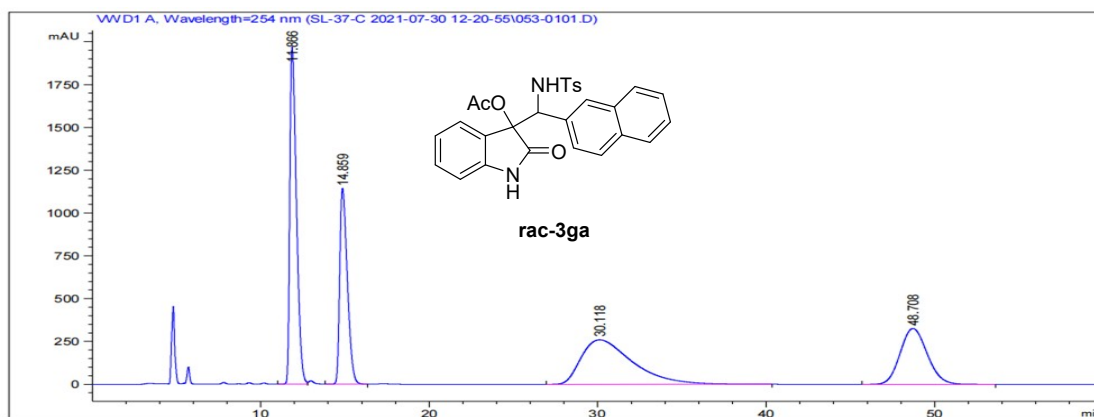
Peak#	Retention Time	Area	Area%
1	8.772	568.63776	2.8722
2	9.891	418.27148	2.1127
3	15.551	1475.18640	7.4511
4	23.043	1.73360e4	87.5640



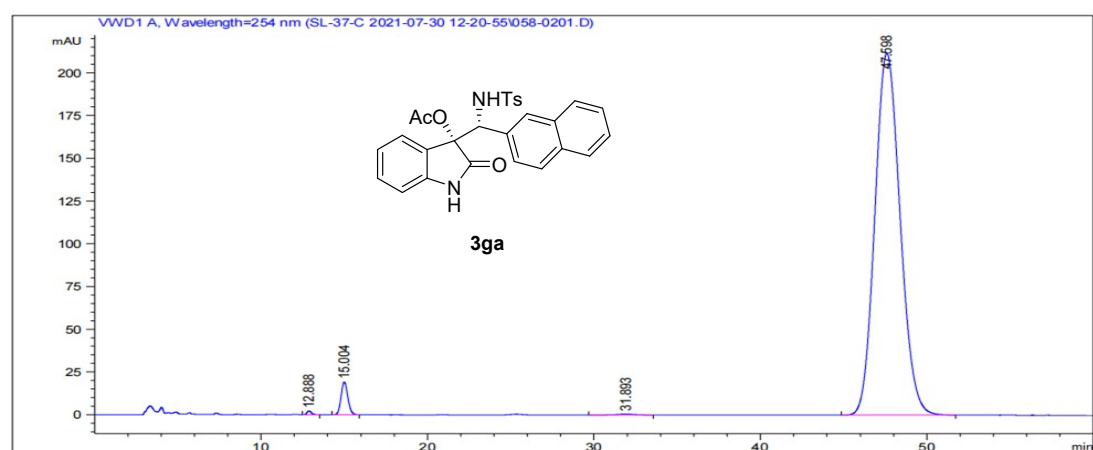
Peak#	Retention Time	Area	Area%
1	12.940	1122.10889	20.0834
2	17.010	1719.66980	30.7785
3	31.420	1126.57922	20.1634
4	58.401	1618.87695	28.9746



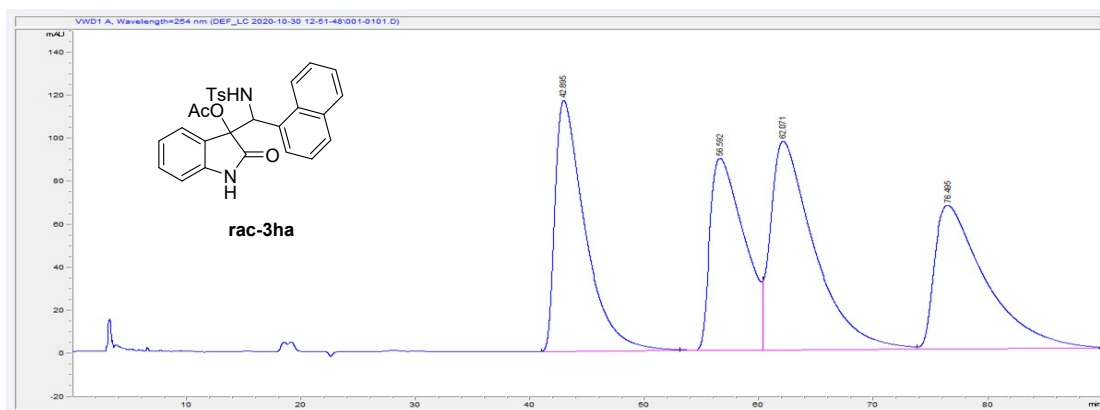
Peak#	Retention Time	Area	Area%
1	12.762	84.59418	0.4686
2	16.836	314.98401	1.7448
3	31.275	443.69452	2.4577
4	59.594	17210.0	95.3290



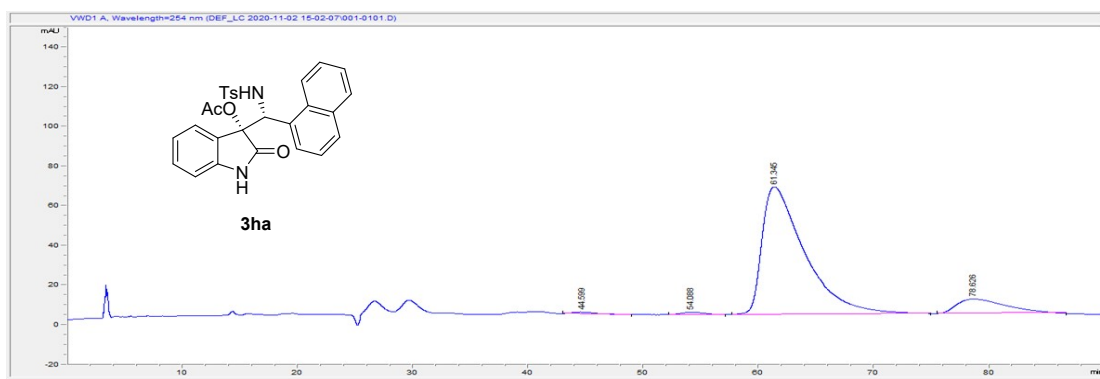
Peak#	Retention Time	Area	Area%
1	11.866	5.37464e4	29.7757
2	14.859	3.58072e4	19.8373
3	30.118	5.47544e4	30.3342
4	48.708	3.61959e4	20.0527



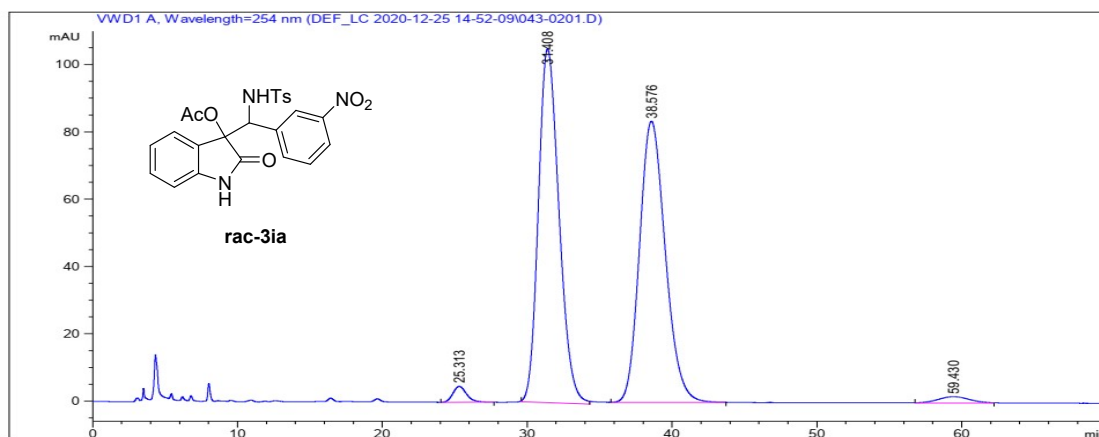
Peak#	Retention Time	Area	Area%
1	12.888	41.12314	0.1793
2	15.004	542.54950	2.3653
3	31.893	69.35703	0.302
4	47.598	2.22848e4	97.1530



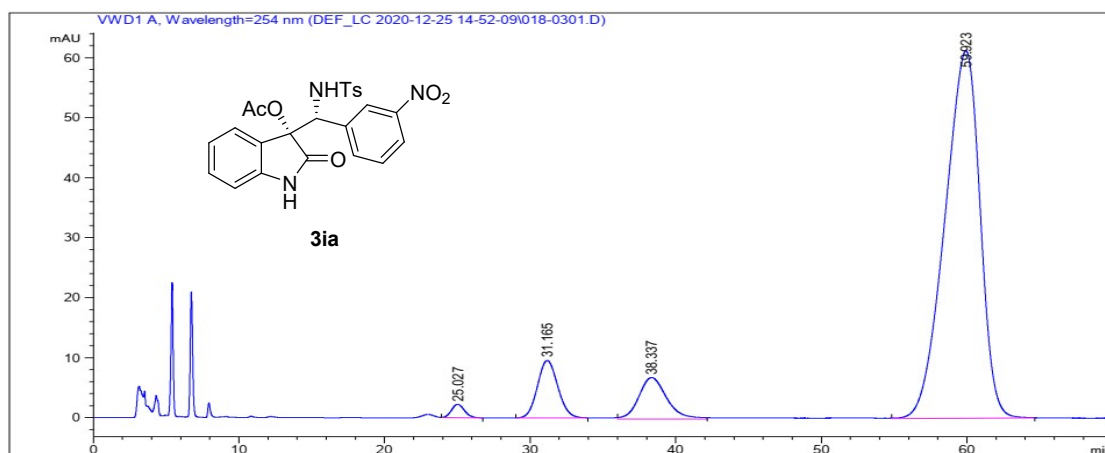
Peak#	Retention Time	Area	Area%
1	42.895	2.13946e4	24.5576
2	56.592	1.88732e4	21.6633
3	62.071	2.57895e4	29.6022
4	76.495	2.10630e4	24.1769



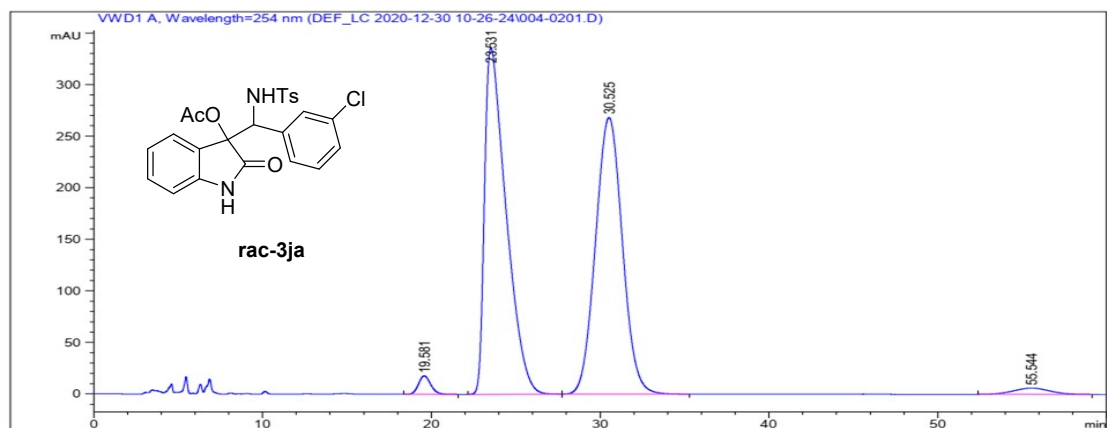
Peak#	Retention Time	Area	Area%
1	44.599	151.77515	0.8013
2	54.088	200.00208	1.0559
3	61.345	1.64311e4	86.7509
4	78.626	2157.67505	11.3918



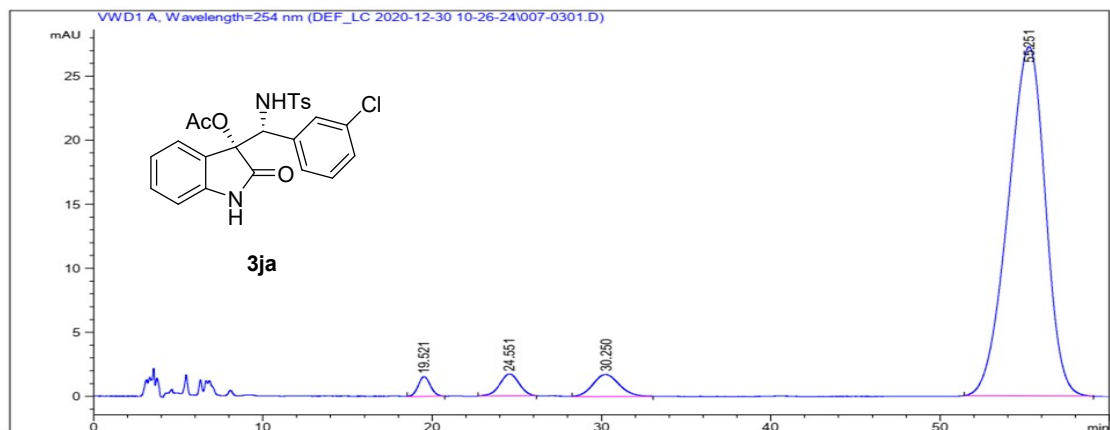
Peak#	Retention Time	Area	Area%
1	25.313	312.20825	1.4632
2	31.408	10399.11	48.7378
3	38.576	10323.40	48.3829
4	59.429	302.14886	1.4161



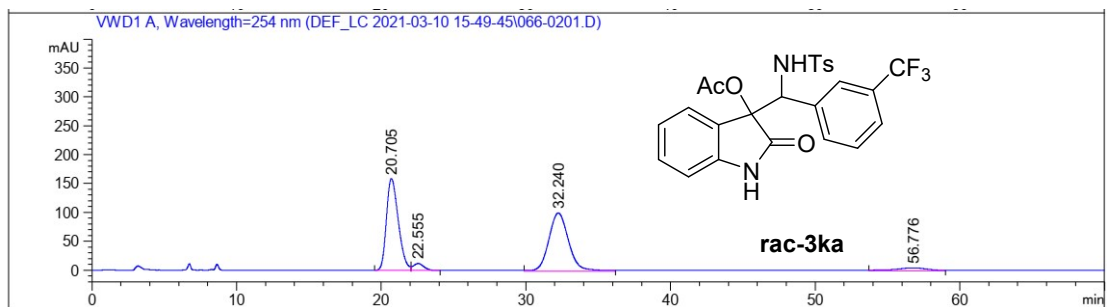
Peak#	Retention Time	Area	Area%
1	25.027	135.86183	1.0830
2	31.165	923.98108	7.3652
3	38.337	918.82520	7.3241
4	59.922	10436.1	84.2278



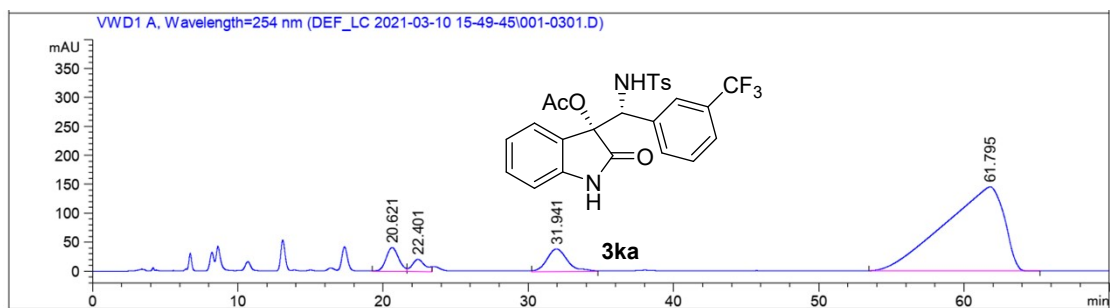
Peak#	Retention Time	Area	Area%
1	19.581	902.55109	1.4748
2	23.531	2.97761e4	48.6540
3	30.525	2.96240e4	48.4055
4	55.544	897.03772	1.4658



Peak#	Retention Time	Area	Area%
1	19.521	75.68641	1.6617
2	24.551	135.68826	2.9791
3	30.251	186.78535	4.1009
4	55.251	4156.52881	91.2582

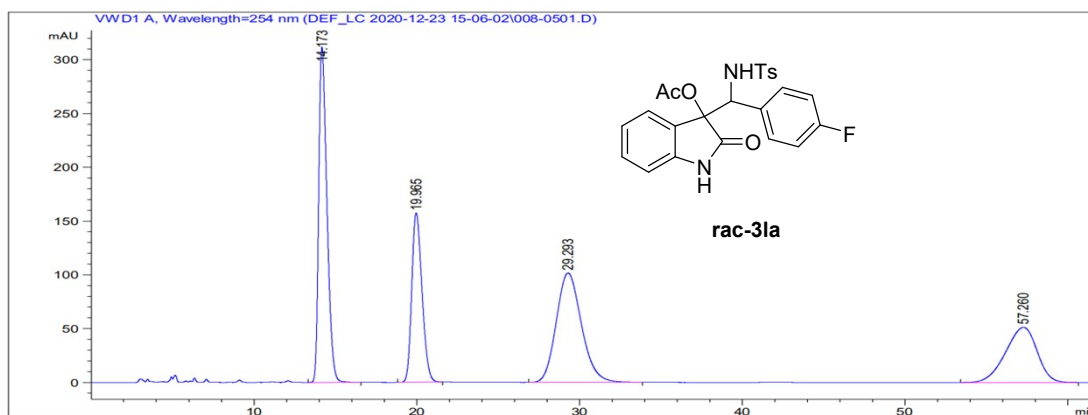


Peak#	Retention Time	Area	Area%
1	20.705	9042.71191	45.3333
2	22.555	595.91644	2.9875
3	32.240	9607.39746	48.1642
4	56.776	701.13434	3.5150

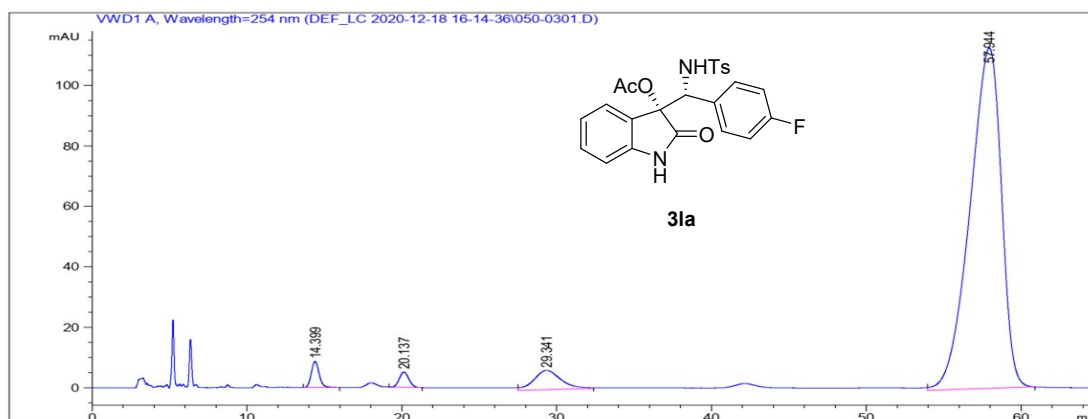


Peak#	Retention Time	Area	Area%
1	20.621	2450.21802	5.0064
2	22.401	1296.14758	2.6484
3	31.941	3771.41333	7.7060
4	61.795	4.14237e4	84.6393

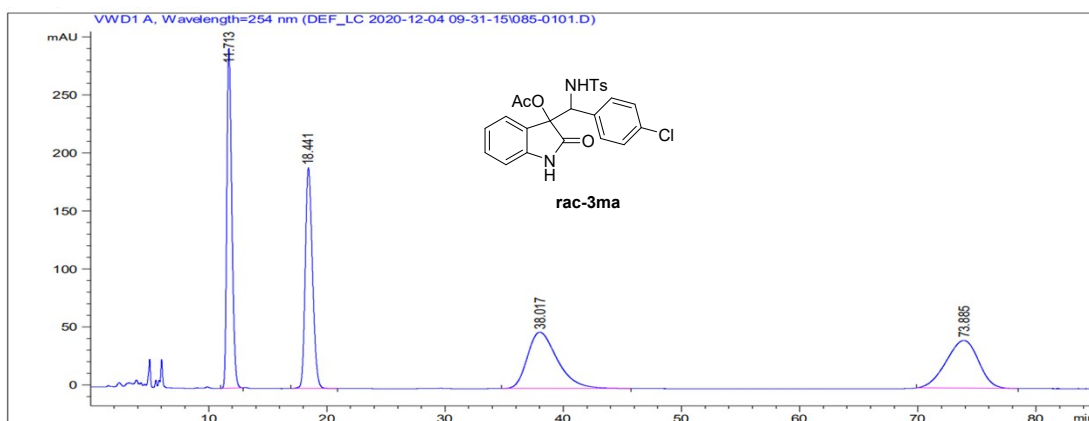




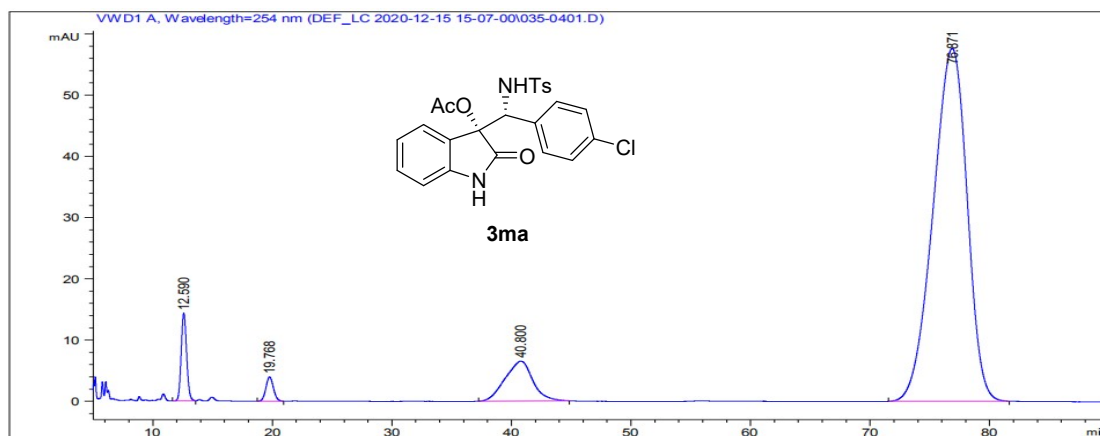
Peak#	Retention Time	Area	Area%
1	14.173	1.09381e4	30.7177
2	19.965	6865.47168	19.2804
3	29.293	1.09157e4	30.6547
4	57.260	6889.31152	19.3473



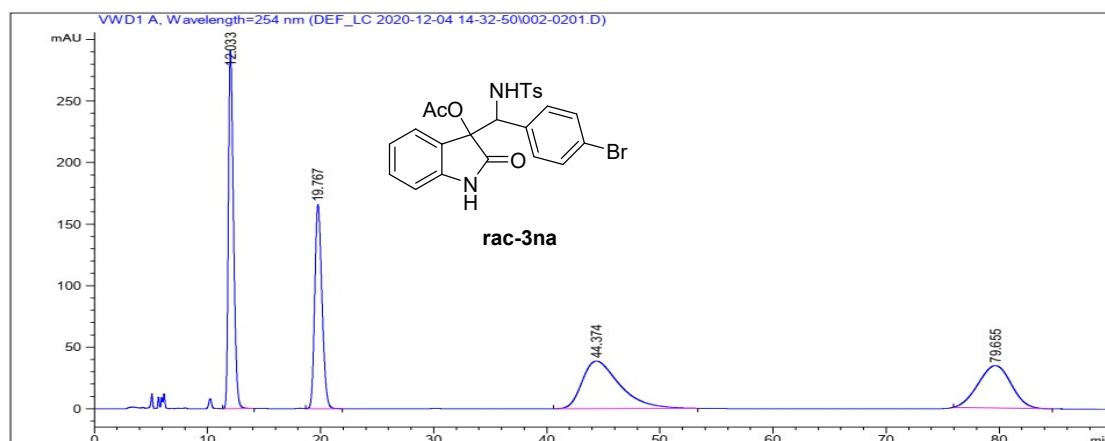
Peak#	Retention Time	Area	Area%
1	14.399	303.42355	1.7706
2	20.137	214.91441	1.2541
3	29.347	772.27985	4.5065
4	57.944	1.58463e4	92.4688



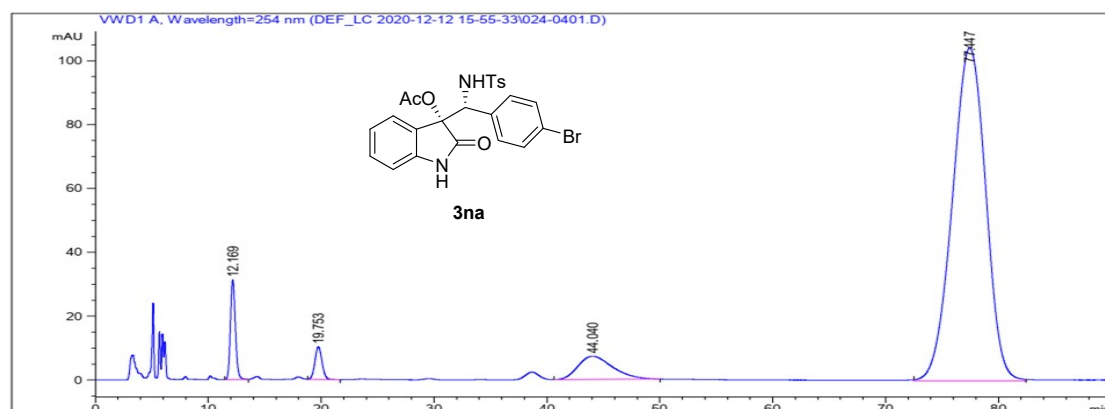
Peak#	Retention Time	Area	Area%
1	11.713	8580.37598	25.9683
2	18.441	8071.57471	24.4284
3	38.017	8530.20801	25.8164
4	73.885	7859.62939	23.7869



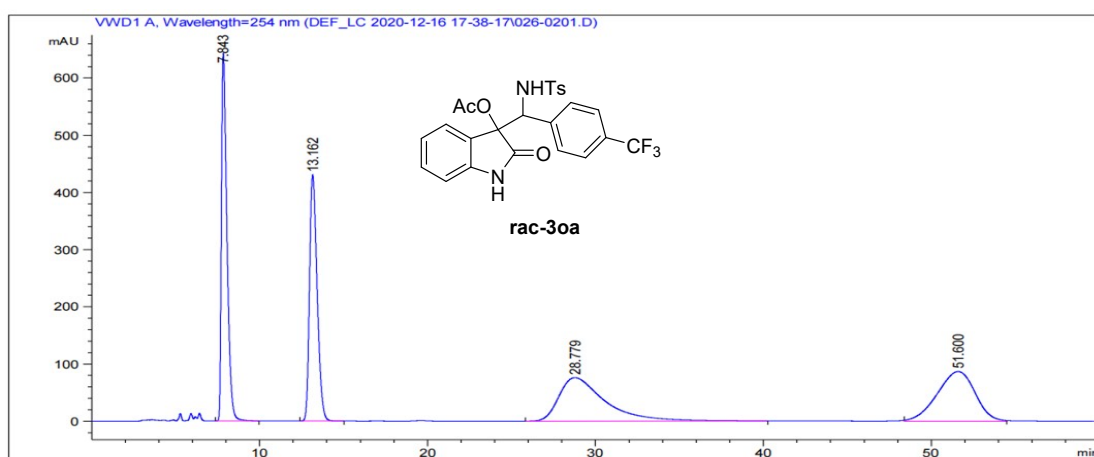
Peak#	Retention Time	Area	Area%
1	12.590	452.06543	3.4616
2	19.768	172.88231	1.3238
3	40.800	1045.86230	8.0085
4	76.871	1.13886e4	87.2061



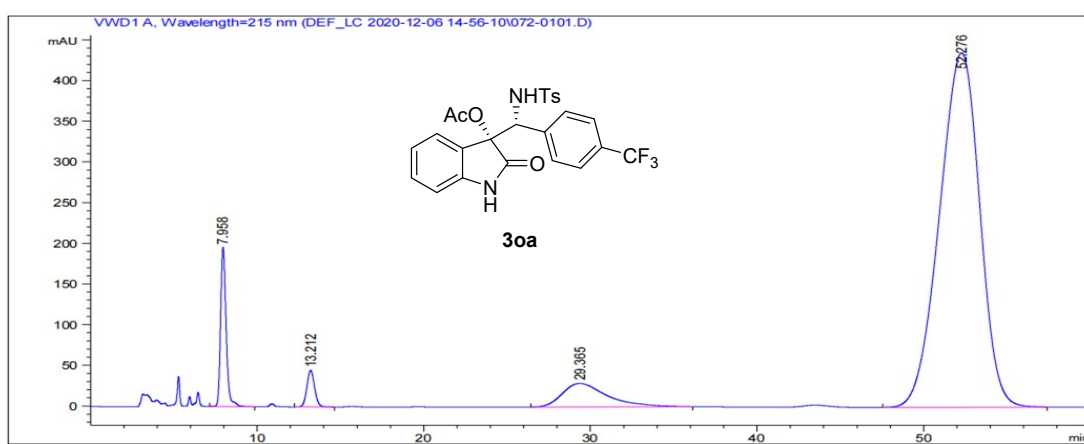
Peak#	Retention Time	Area	Area%
1	12.033	9060.74219	28.0799
2	19.767	7355.35400	22.7948
3	44.374	8885.72559	27.5375
4	79.655	6965.84863	21.5877



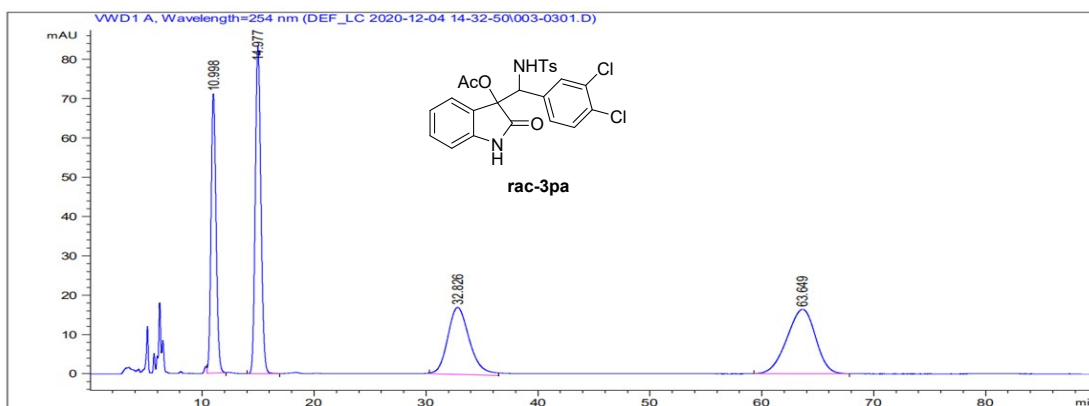
Peak#	Retention Time	Area	Area%
1	12.169	950.85400	3.9106
2	19.753	446.82974	1.8377
3	44.040	1560.74072	6.4189
4	77.447	2.13565e4	87.8329



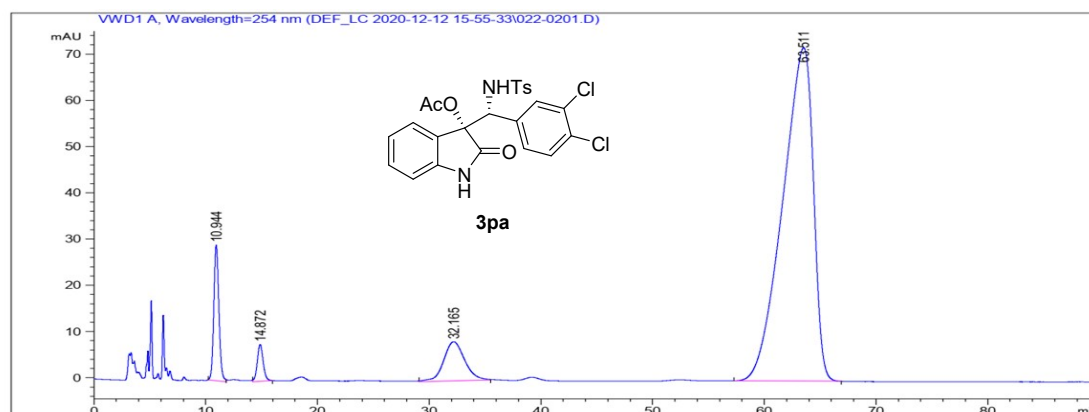
Peak#	Retention Time	Area	Area%
1	7.843	1.46729e4	26.0612
2	13.162	4.35984e4	24.1528
3	28.779	1.44482e4	25.6622
4	51.600	1.35820e4	24.1237



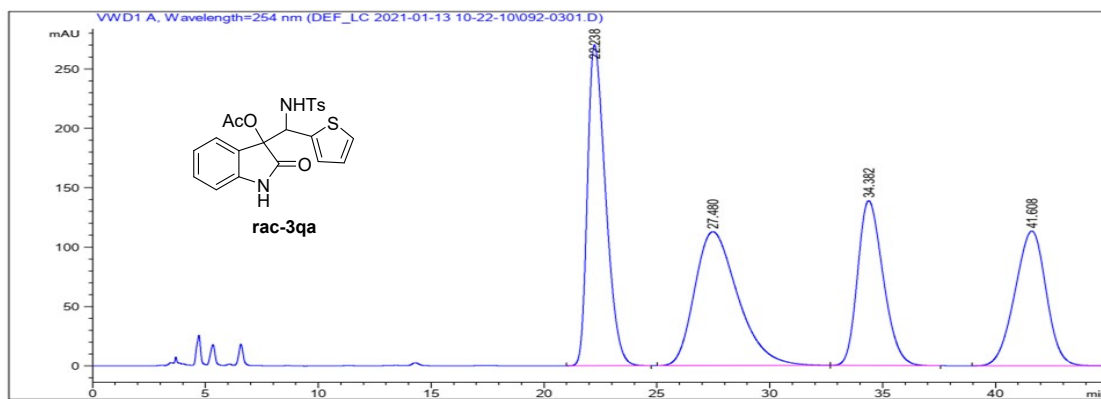
Peak#	Retention Time	Area	Area%
1	7.958	4485.00195	5.3290
2	13.212	1446.17529	1.7183
3	29.365	5391.12109	6.4057
4	52.276	7.28395e4	86.5470



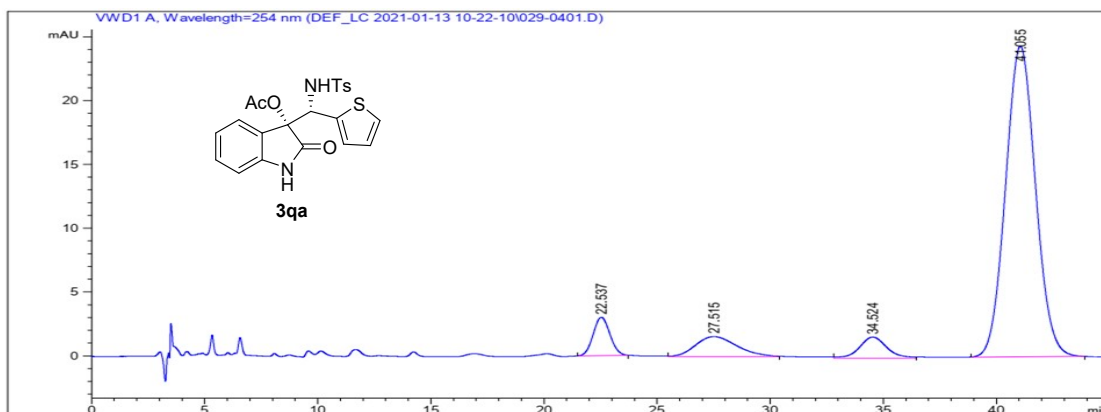
Peak#	Retention Time	Area	Area%
1	10.998	2214.74780	21.3503
2	14.977	2930.17188	28.2470
3	32.826	2318.61963	22.3516
4	63.649	2909.83960	28.0510



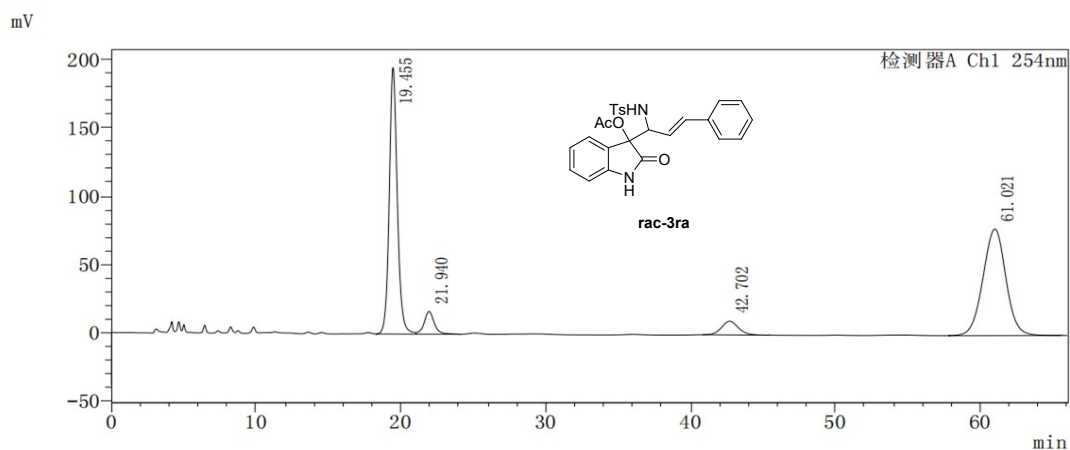
Peak#	Retention Time	Area	Area%
1	10.943	893.11395	5.6386
2	14.873	278.11292	1.7559
3	32.169	936.92523	5.9152
4	63.511	1.37310e4	86.6902



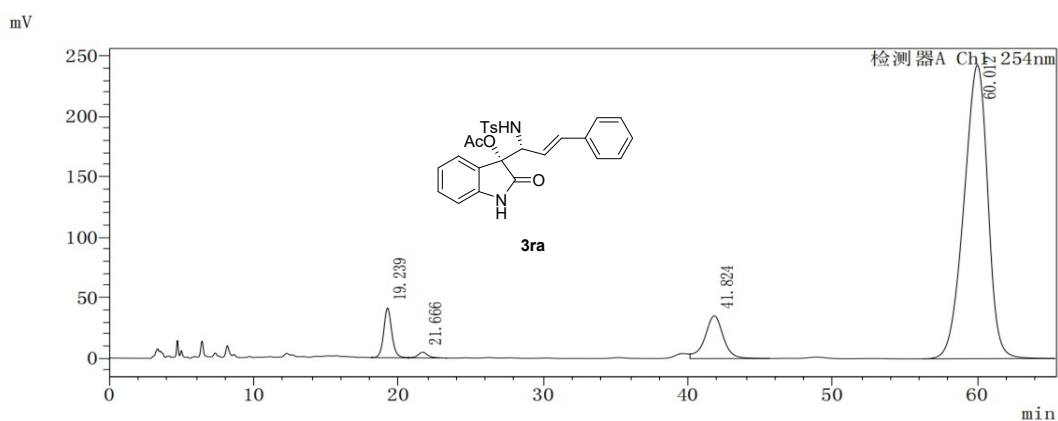
Peak#	Retention Time	Area	Area%
1	22.238	1.50651e4	29.0820
2	27.480	1.49438e4	28.8478
3	34.382 B	1.08961e4	21.0340
4	41.608	1.08972e4	21.0362



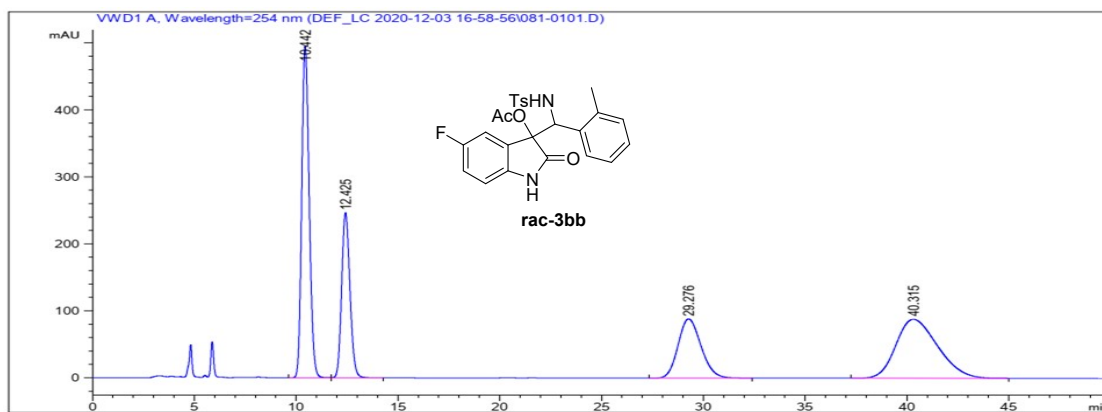
Peak#	Retention Time	Area	Area%
1	22.537	154.03752	5.5409
2	27.515	242.97783	8.7403
3	34.524	144.14236	5.1850
4	41.055	2238.82739	80.5338



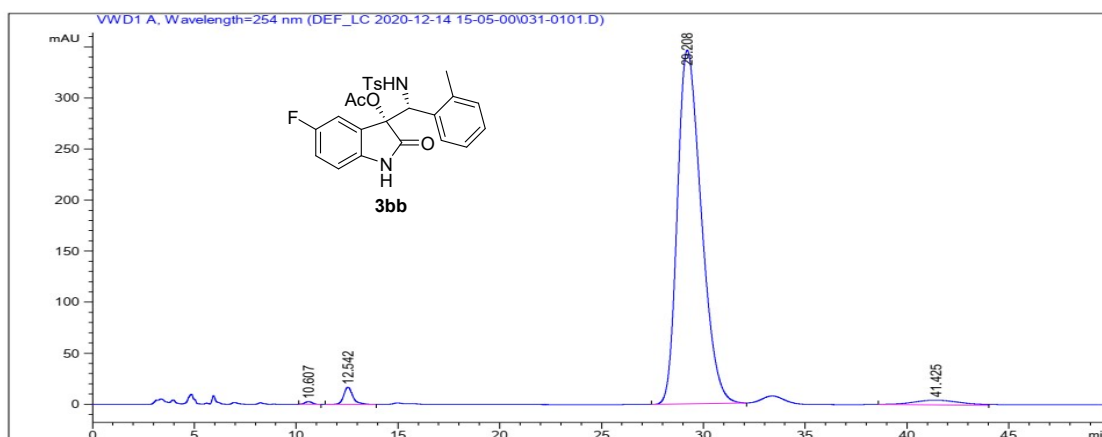
Peak#	Retention Time	Area	Area%
1	19.455	6013544	44.879
2	21.940	621333	4.637
3	42.702	698246	5.211
4	61.012	6066338	45.273



Peak#	Retention Time	Area	Area%
1	19.239	1609895	4.900
2	21.666	223093	0.679
3	41.824	3142175	9.563
4	60.012	27881707	84.858

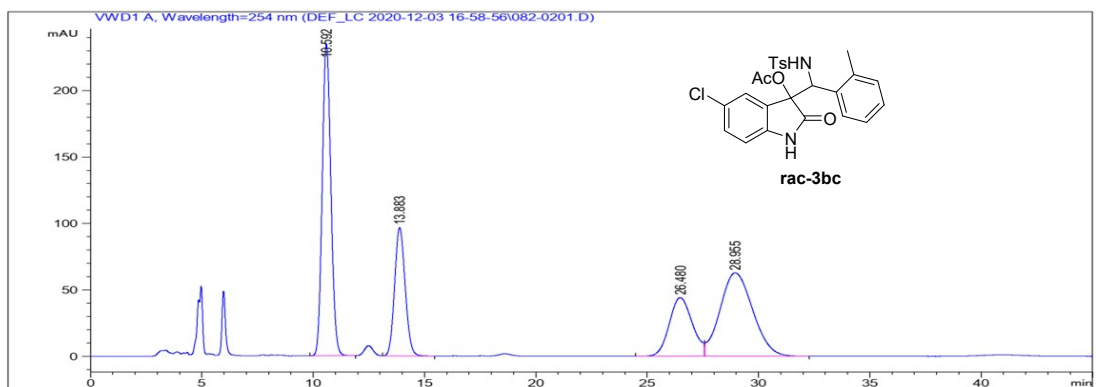


Peak#	Retention Time	Area	Area%
1	10.442	1.25945e4	31.3936
2	12.425	6942.06201	17.3041
3	29.276	6926.31201	17.2649
4	40.315	1.24909e4	31.1355

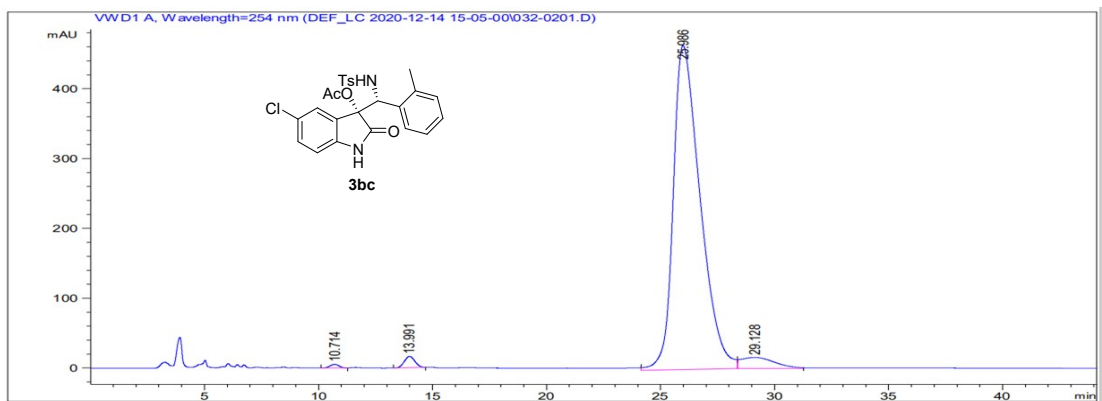


Peak#	Retention Time	Area	Area%
1	10.607	62.81887	0.2108
2	12.542	524.17572	1.7585
3	29.208	2.82197e4	94.6740
4	41.375	1000.53473	3.3567

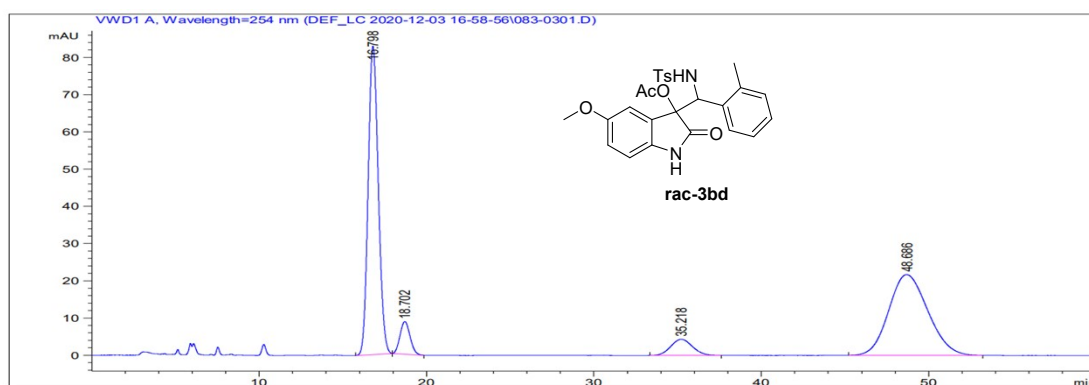




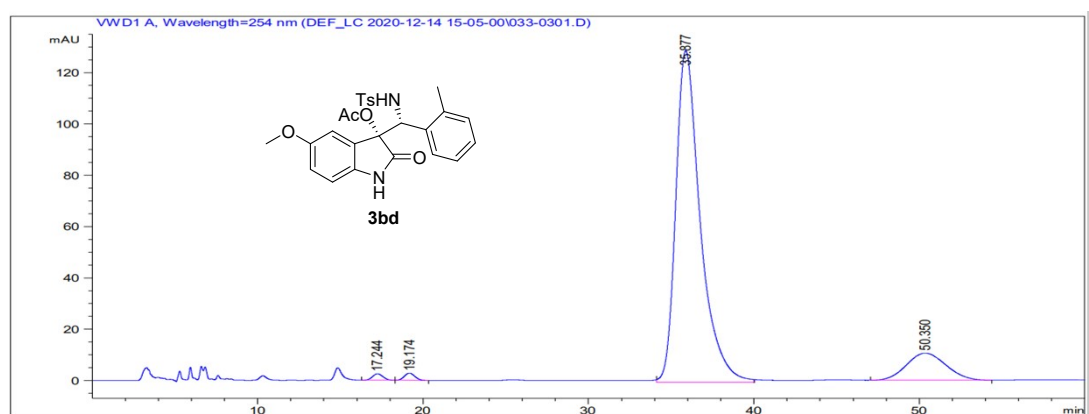
Peak#	Retention Time	Area	Area%
1	10.592	6357.72949	33.2376
2	13.883	3189.30786	16.6734
3	26.480	3170.62280	16.5757
4	28.955	6410.48975	33.5134



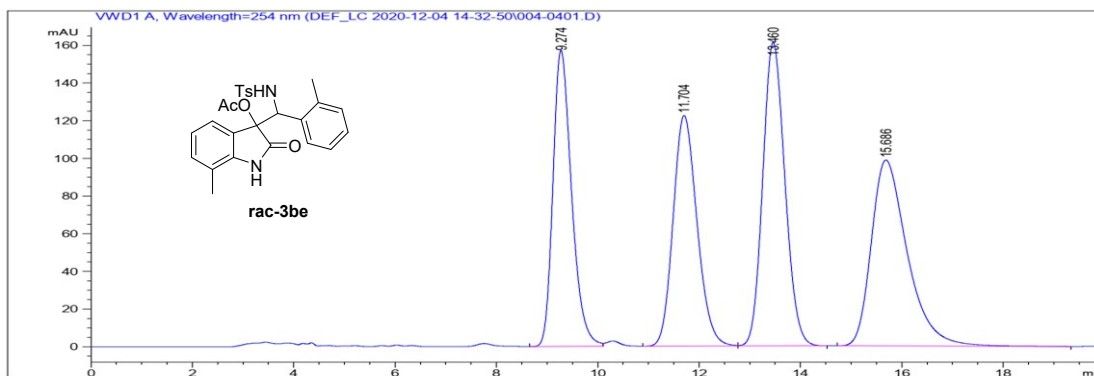
Peak#	Retention Time	Area	Area%
1	10.710	139.24260	0.3441
2	13.991	517.06921	1.2780
3	25.986	3.81326e4	94.2476
4	29.128	1671.08899	4.1302



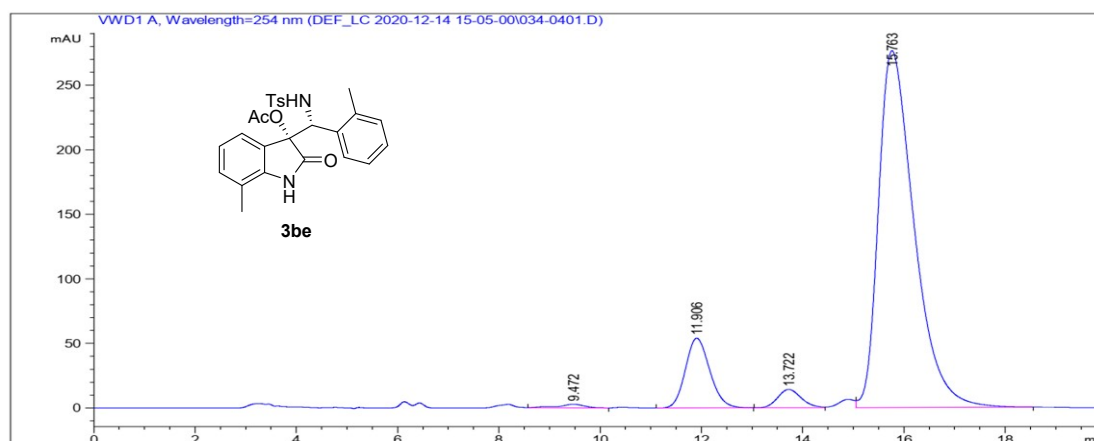
Peak#	Retention Time	Area	Area%
1	16.798	82.83369	44.9349
2	18.702	8.76689	4.6893
3	35.218	4.33215	5.0198
4	48.686	21.67719	45.3560



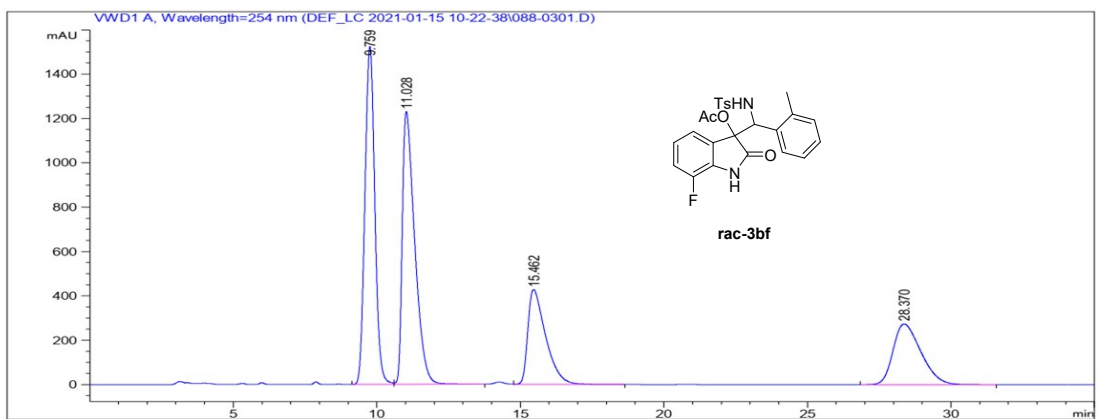
Peak#	Retention Time	Area	Area%
1	17.244	109.15645	0.7131
2	19.174	122.37705	0.7995
3	35.877	1.33228e4	87.0398
4	50.350	1752.22473	11.4476



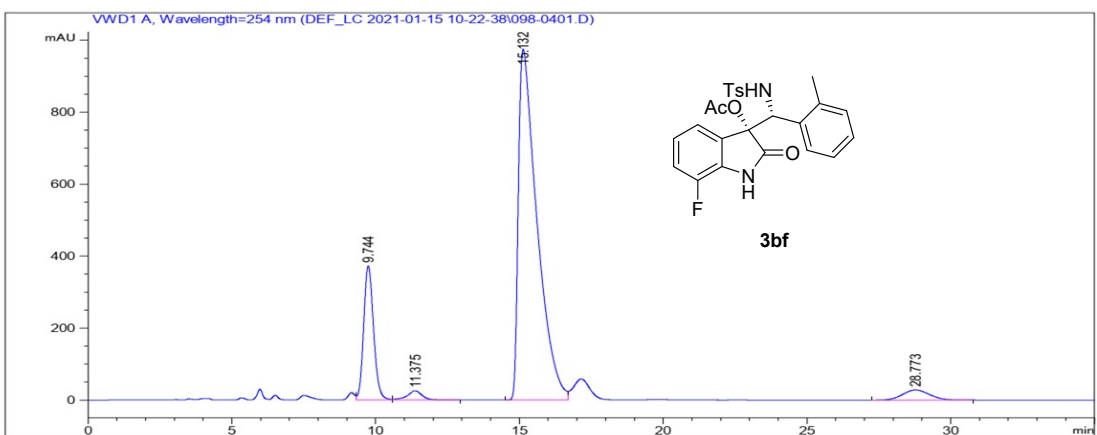
Peak#	Retention Time	Area	Area%
1	9.590	4492.37256	27.8723
2	12.126	4462.75000	27.6885
3	14.052	3572.83643	22.1671
4	16.370	3589.76025	22.2721



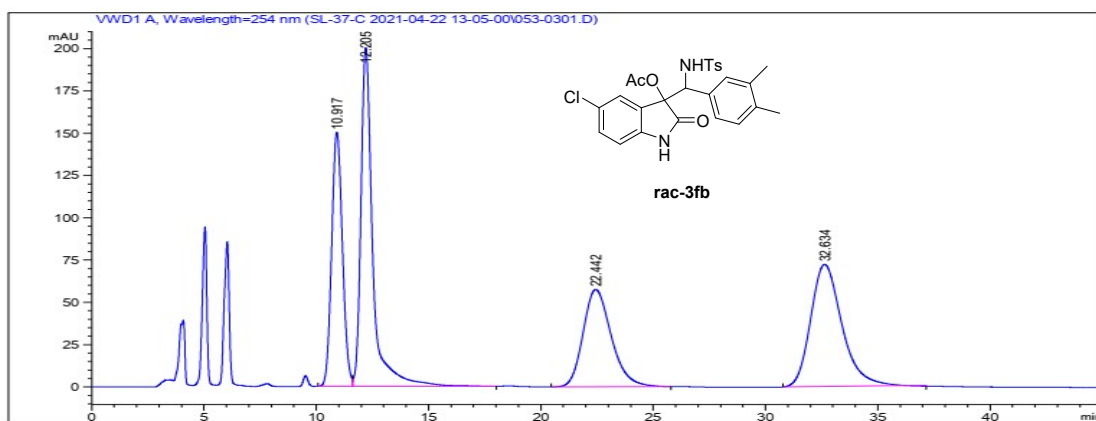
Peak#	Retention Time	Area	Area%
1	9.666	177.62604	0.4929
2	12.131	4105.70996	11.3941
3	14.078	1050.18042	2.9144
4	15.962	3.07001e4	85.1985



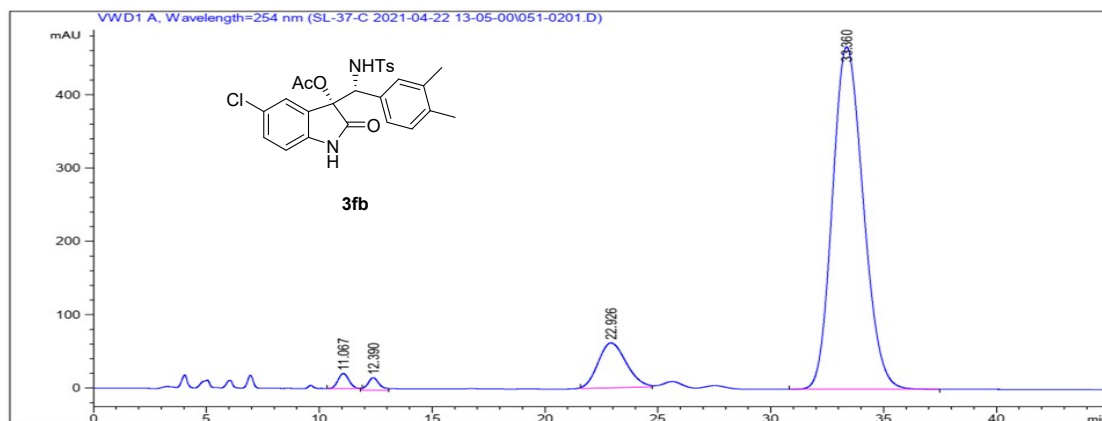
Peak#	Retention Time	Area	Area%
1	9.759	3.64769e4	33.0902
2	11.028	3.70050e4	33.5692
3	15.462	1.83792e4	16.6728
4	28.370	1.83737e4	16.6678



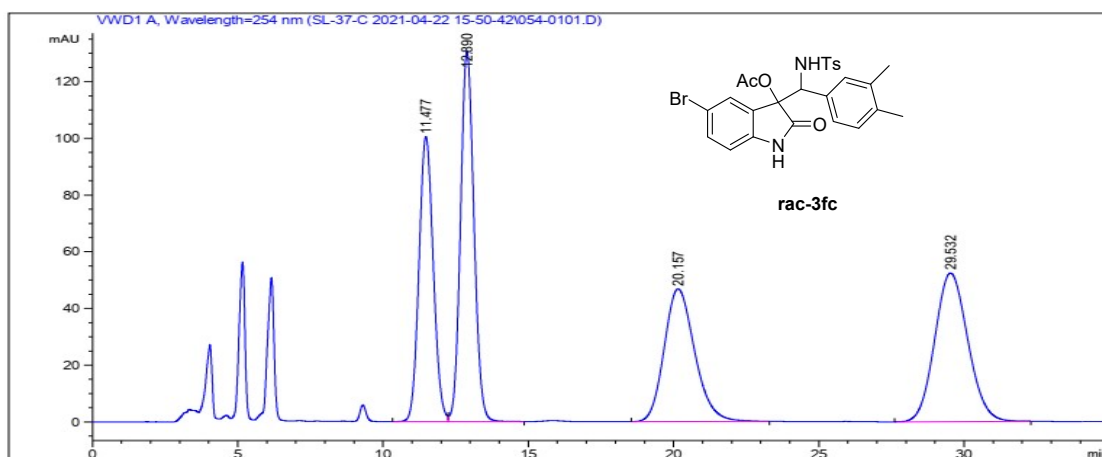
Peak#	Retention Time	Area	Area%
1	9.744	8897.38672	16.0299
2	11.375	911.02985	1.6413
3	15.132	4.38803e4	79.0565
4	28.773	1816.30945	3.2723



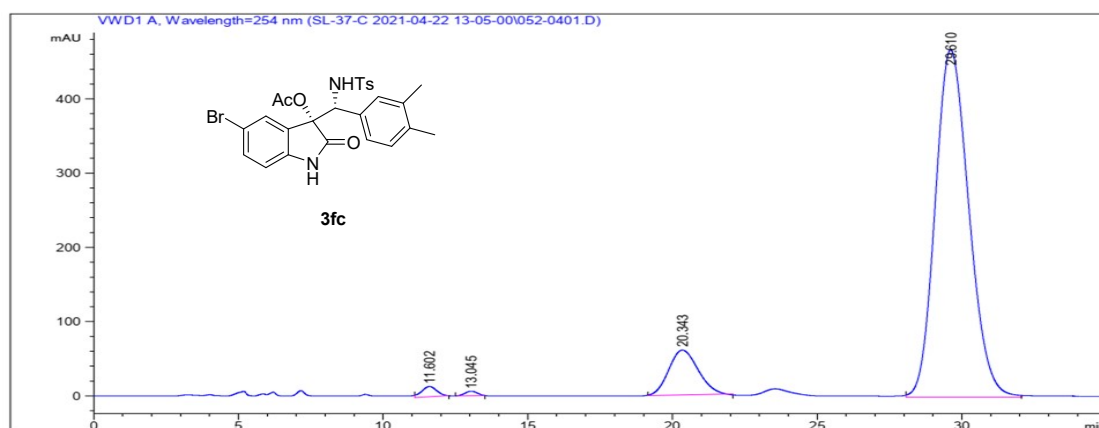
Peak#	Retention Time	Area	Area%
1	10.917	5008.21680	20.6105
2	12.205	7331.53809	30.1718
3	22.442	4998.07568	20.5688
4	32.634	6961.47070	28.6489



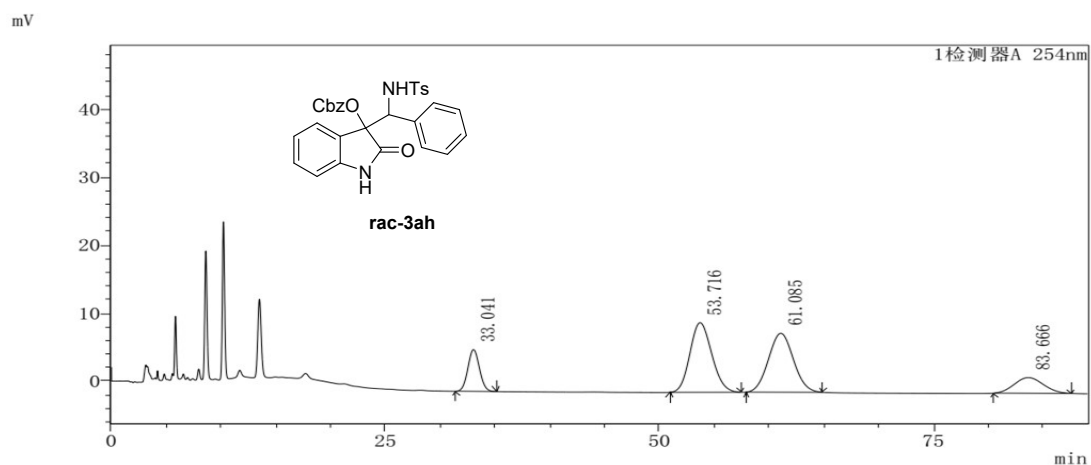
Peak#	Retention Time	Area	Area%
1	11.067	698.77124	1.3748
2	12.390	595.81995	1.1722
3	22.926	5309.32568	10.4455
4	33.360	4.42247e4	87.0075



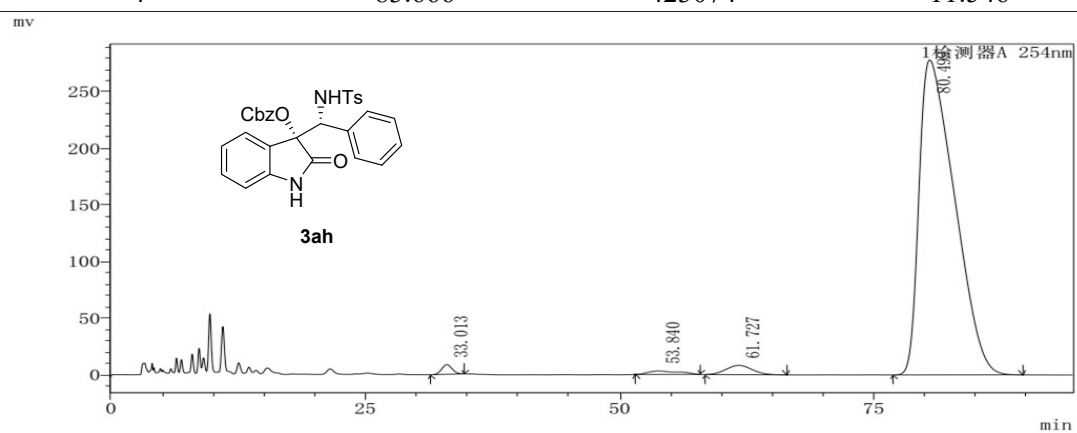
Peak#	Retention Time	Area	Area%
1	11.477	3434.51831	22.6216
2	12.890	4189.20654	27.5924
3	20.157	3412.87207	22.4790
4	29.532	4145.86572	27.3069



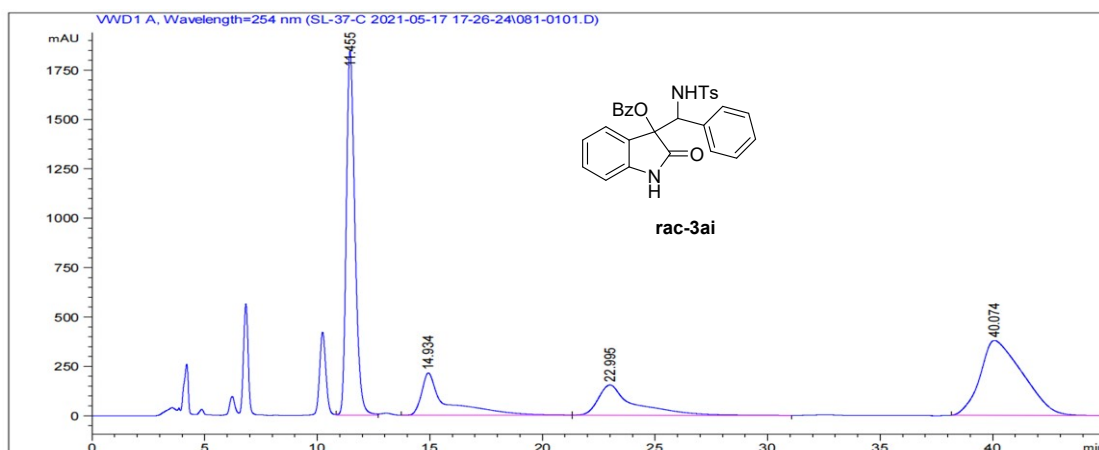
Peak#	Retention Time	Area	Area%
1	11.602	496.39383	1.1611
2	13.045	184.28030	0.4310
3	20.343	4267.55420	9.9822
4	29.610	3.78034e4	88.4256



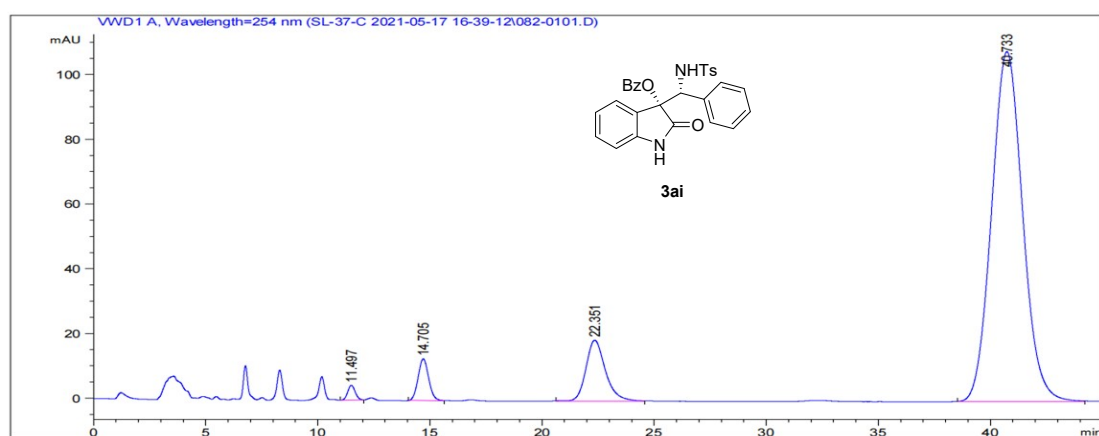
Peak#	Retention Time	Area	Area%
1	33.041	450915	12.305
2	53.716	1417615	38.686
3	61.085	1372766	37.463
4	83.666	423074	11.546



Peak#	Retention Time	Area	Area%
1	33.013	8525	0.869
2	53.840	2947	0.839
3	61.727	8175	2.077
4	80.499	278417	96.215

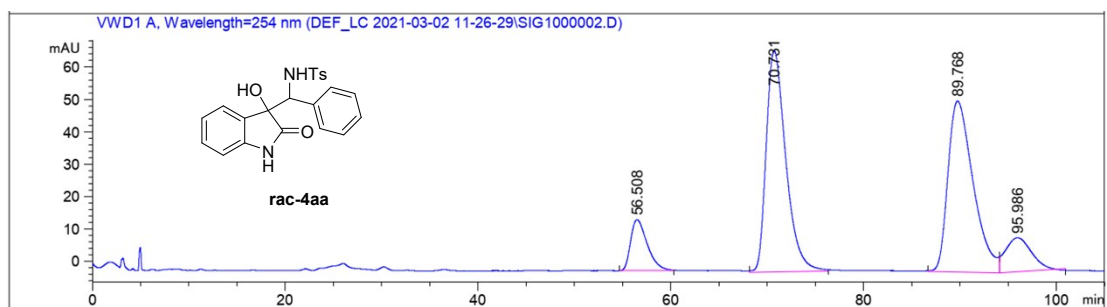


Peak#	Retention Time	Area	Area%
1	33.013	8525	0.869
2	53.840	2947	0.839
3	61.727	8175	2.077
4	80.499	278417	96.215

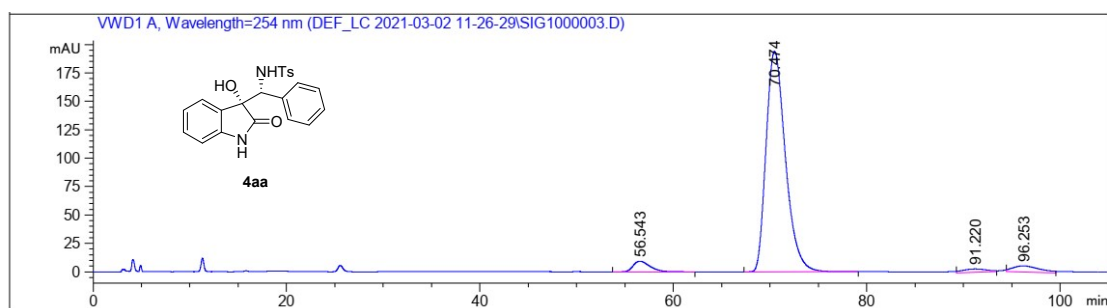


Peak#	Retention Time	Area	Area%
1	33.013	8525	0.869
2	53.840	2947	0.839
3	61.727	8175	2.077
4	80.499	278417	96.215

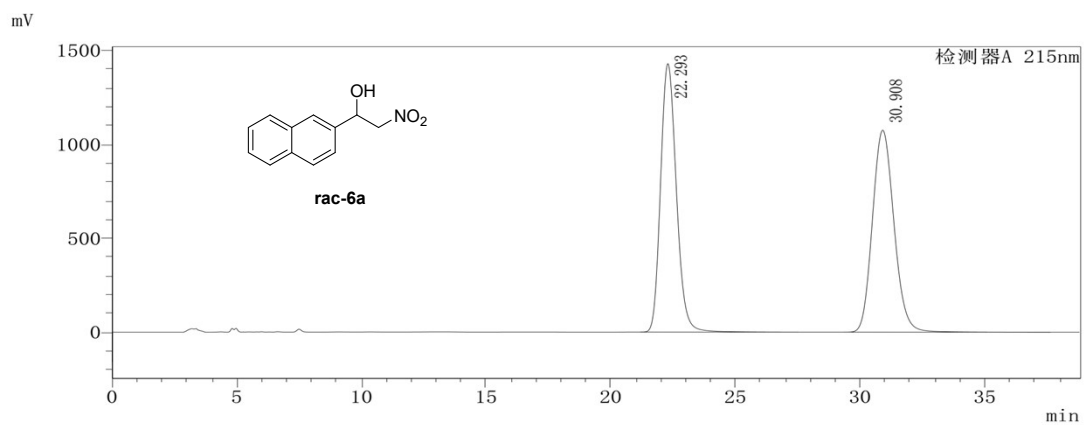




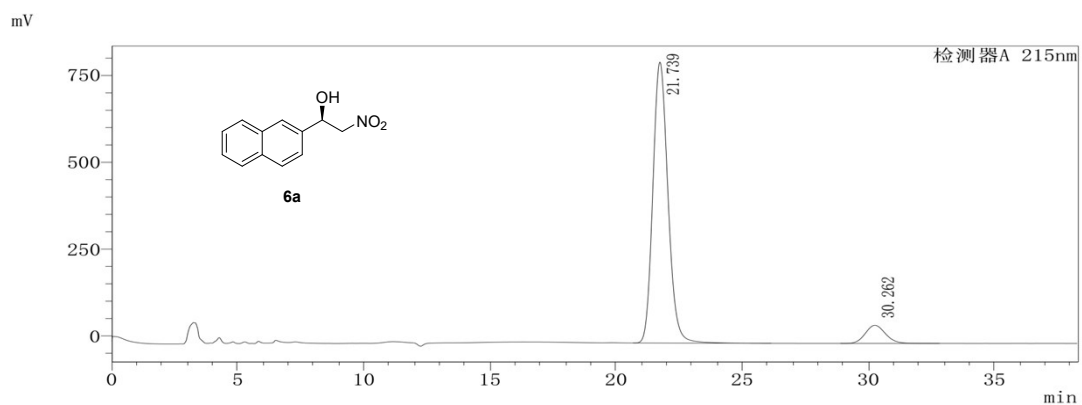
Peak#	Retention Time	Area	Area%
1	56.508	1893.79529	8.1879
2	70.731	9629.56152	41.6297
3	89.768	9645.88867	41.7003
4	95.986	1962.22485	8.4829



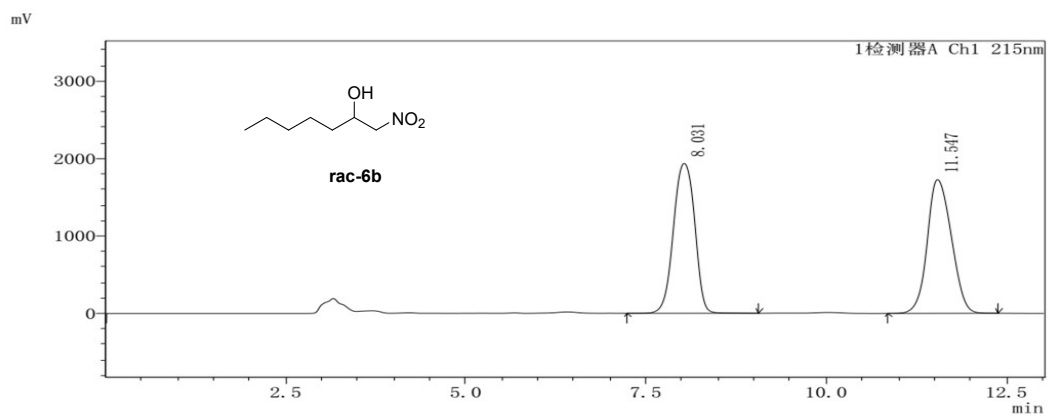
Peak#	Retention Time	Area	Area%
1	56.543	1132.86096	3.8009
2	70.474	2.70235e4	90.6677
3	91.220	548.59631	1.8406
4	96.253	1100.04626	3.6908



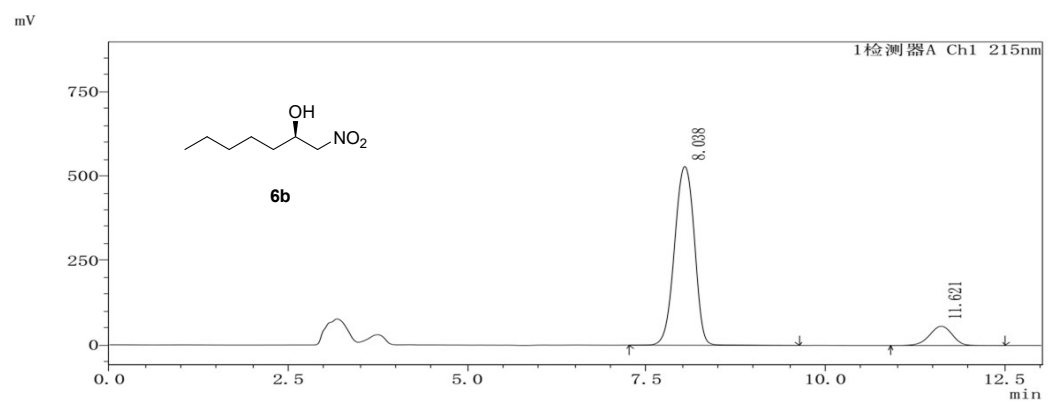
Peak#	Retention Time	Area	Area%
1	22.293	62945527	49.332
2	30.908	64649218	50.668



Peak#	Retention Time	Area	Area%
1	21.739	33546586	92.064
2	30.262	2891620	7.936

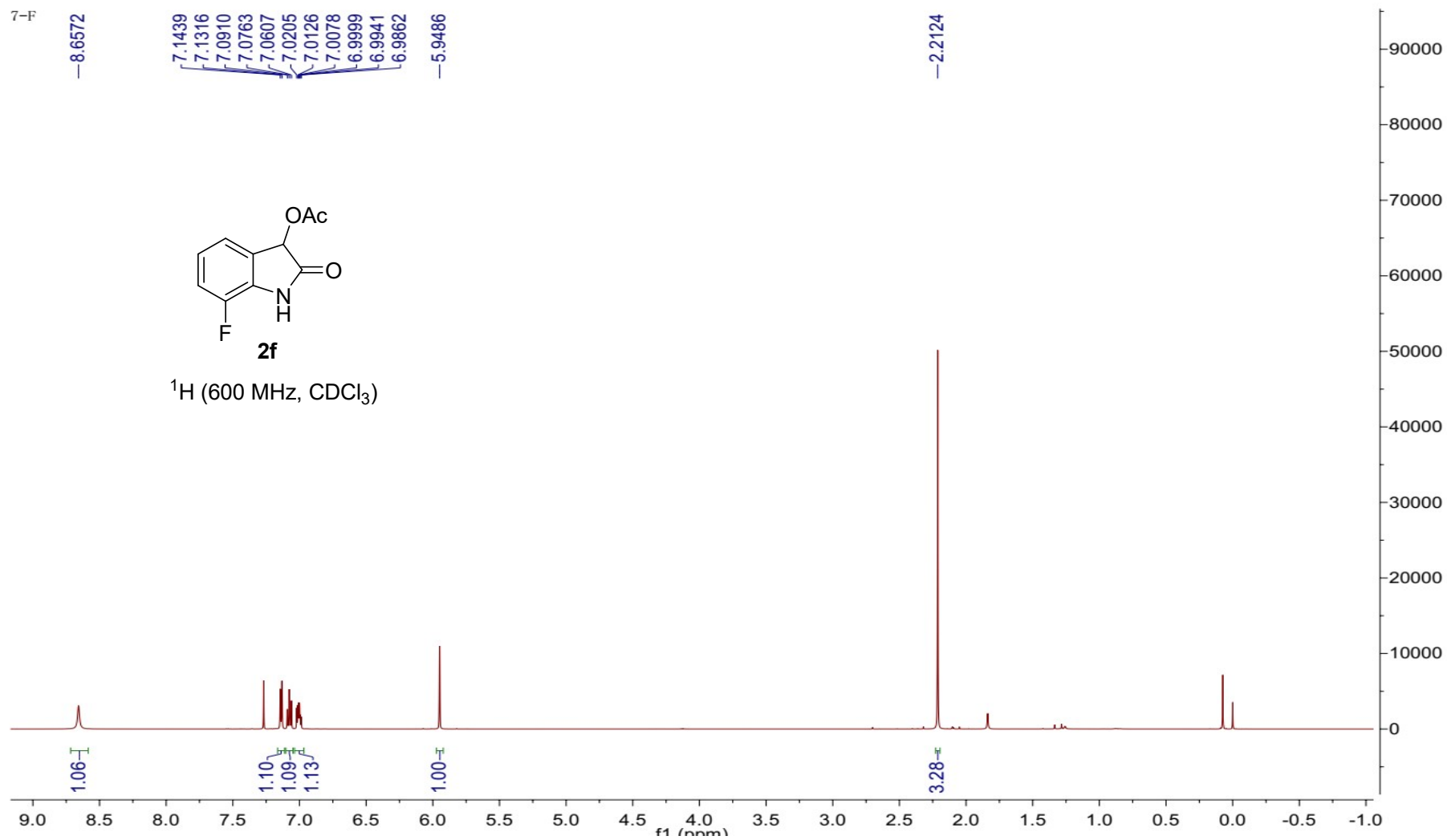


Peak#	Retention Time	Area	Area%
1	8.031	39761355	49.610
2	11.547	40386669	50.390

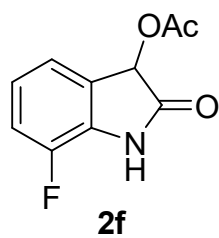


Peak#	Retention Time	Area	Area%
1	8.038	10198318	89.037
2	11.621	1255764	10.963

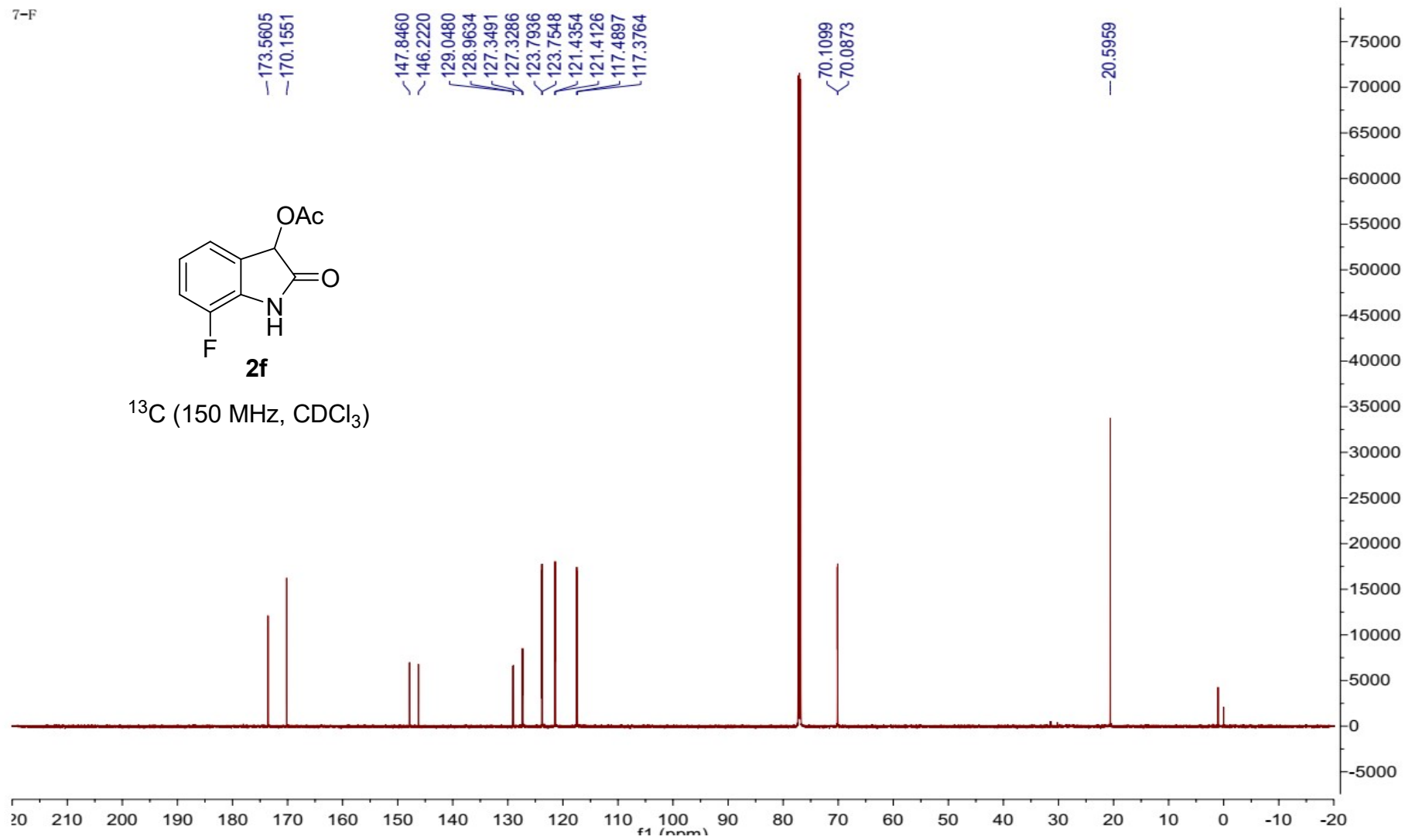
## 4. Copies of NMR Spectra

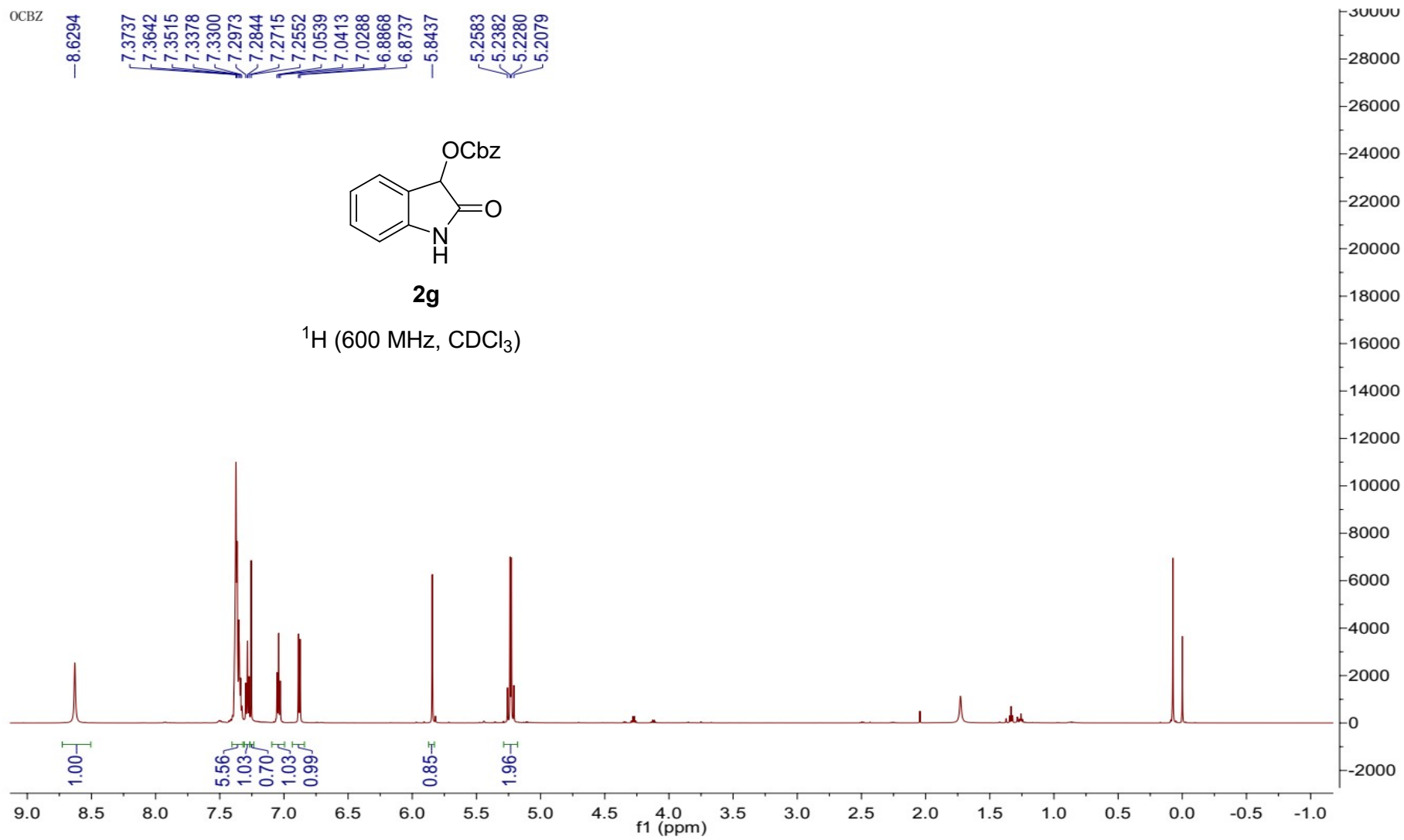


7-F

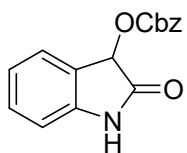


$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )



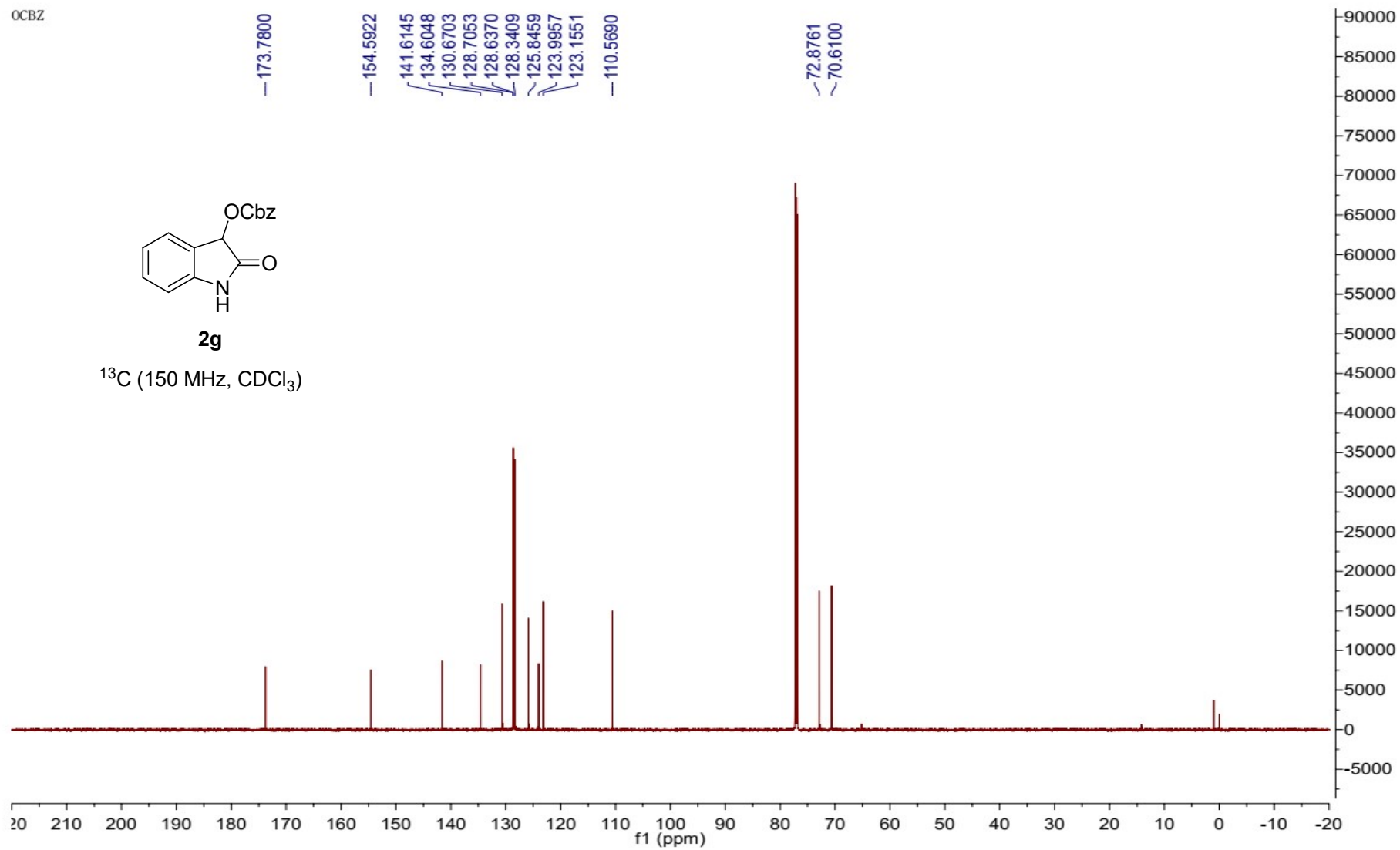


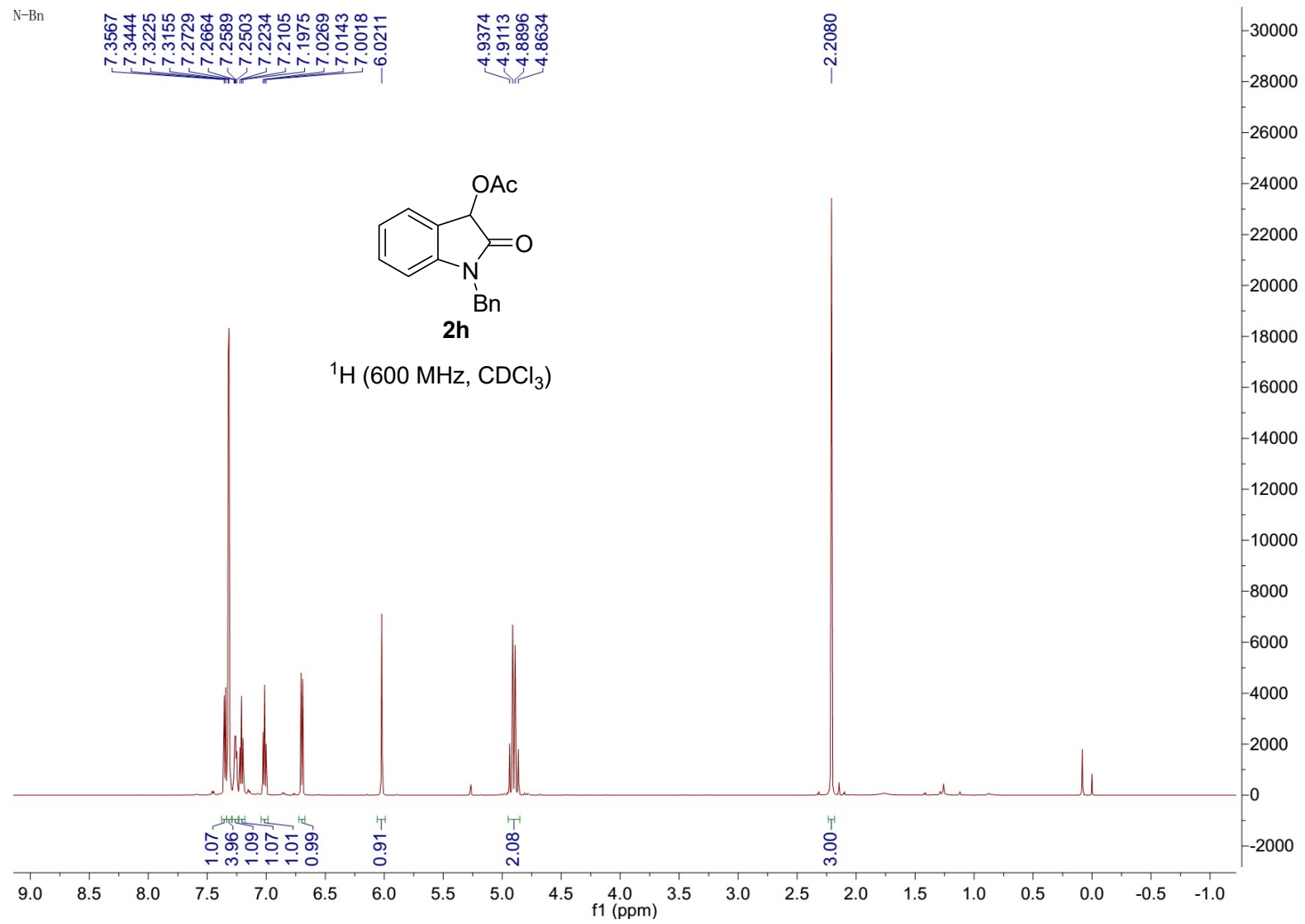
OCBZ



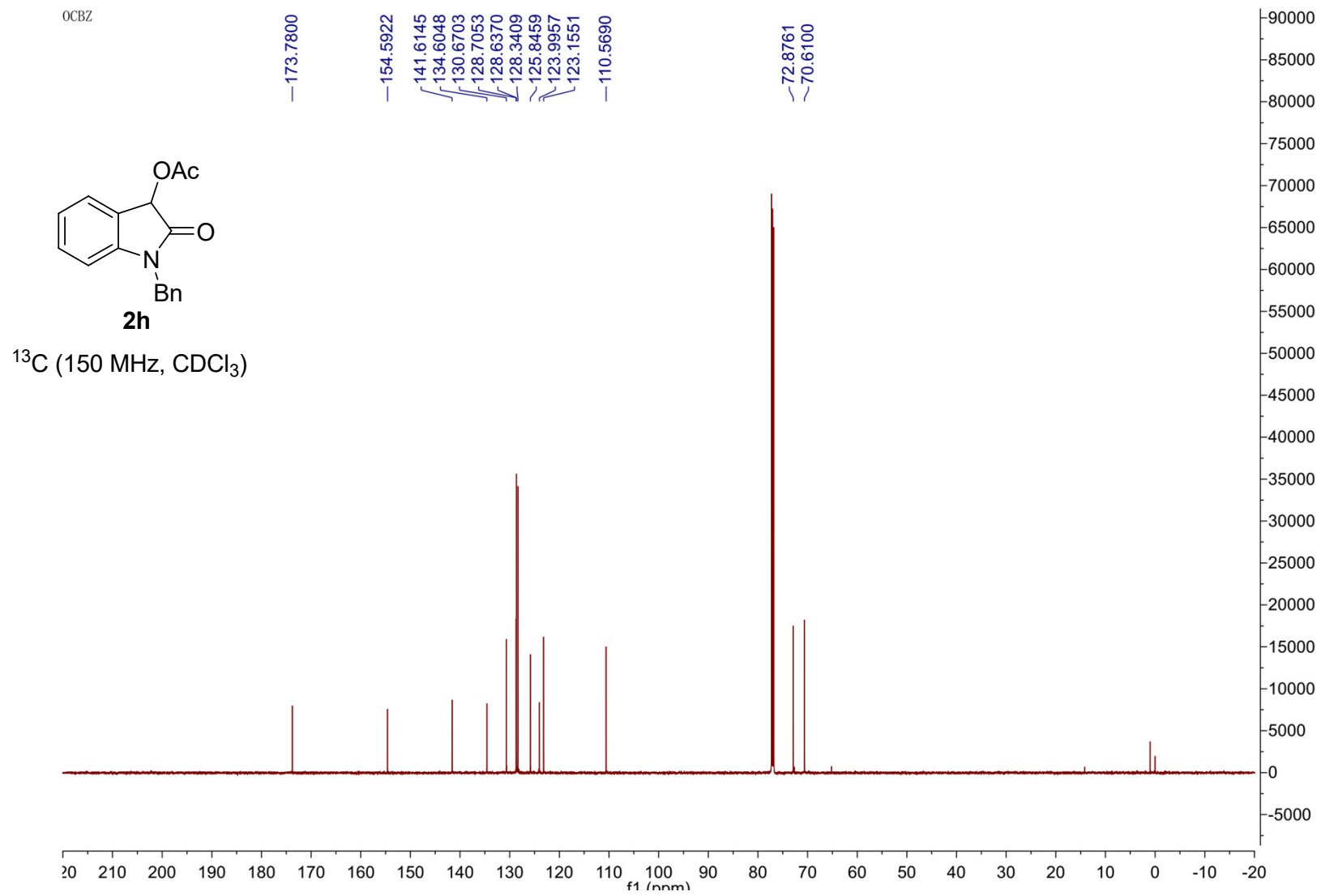
**2g**

$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )





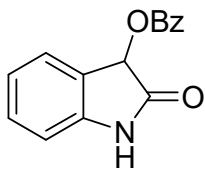




BTSZYL

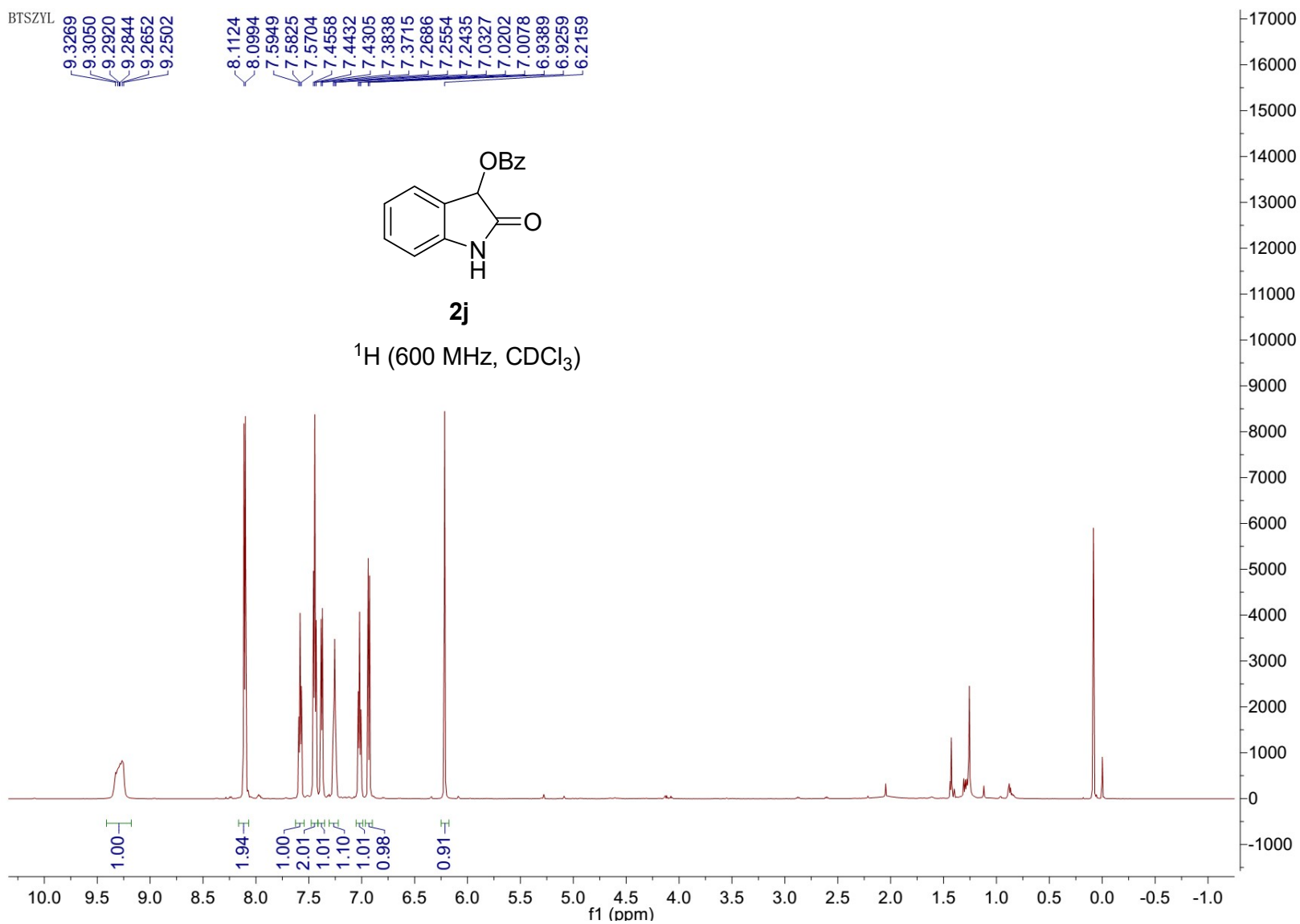
9.3269  
9.3050  
9.2920  
9.2844  
9.2652  
9.2502

8.1124  
8.0994  
7.5949  
7.5825  
7.5704  
7.4558  
7.4432  
7.4305  
7.3838  
7.3715  
7.2686  
7.2554  
7.2435  
7.0327  
7.0202  
7.0078  
6.9389  
6.9259  
6.2159

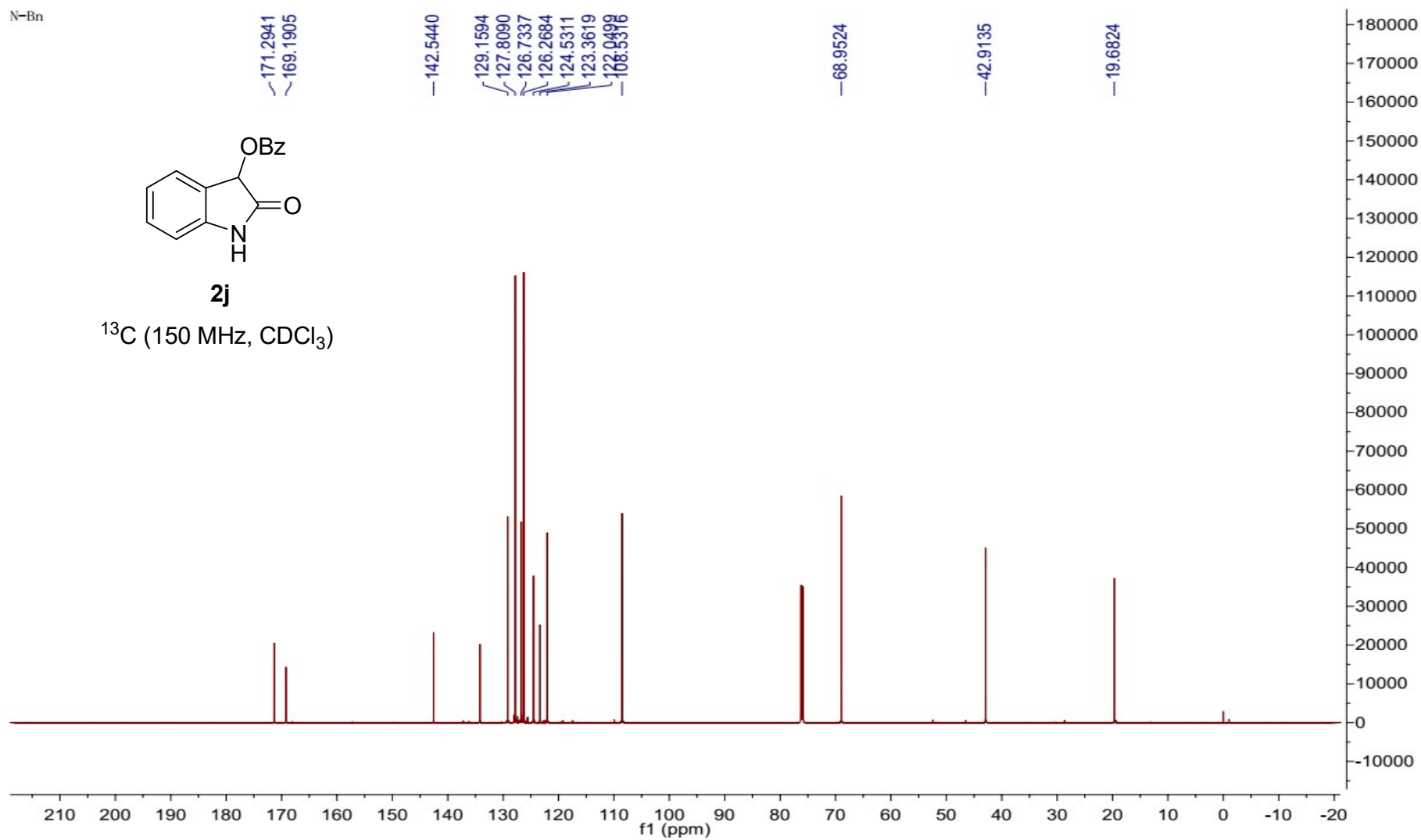
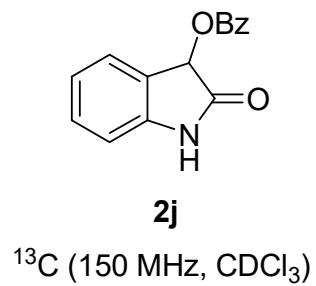


**2j**

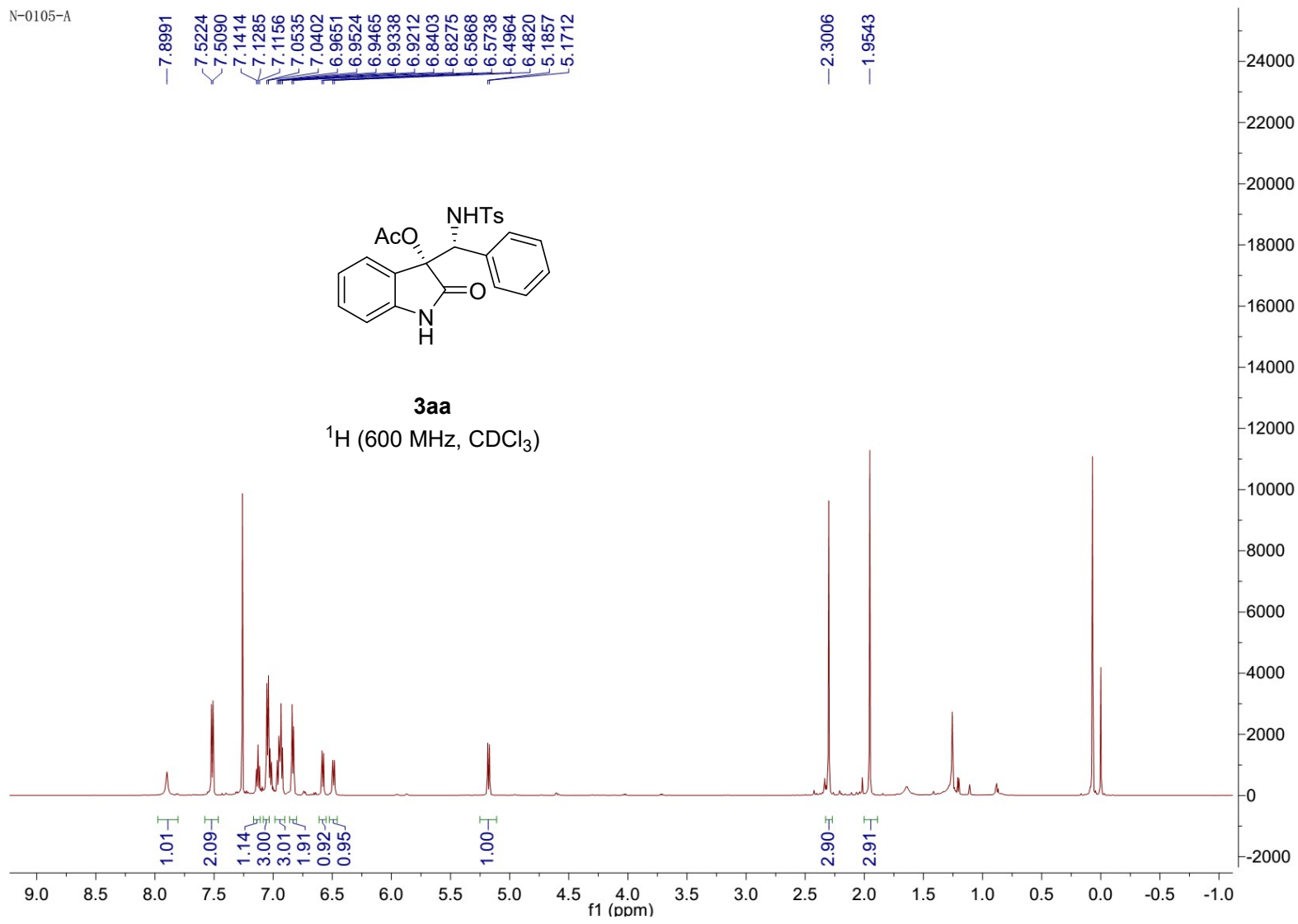
<sup>1</sup>H (600 MHz, CDCl<sub>3</sub>)



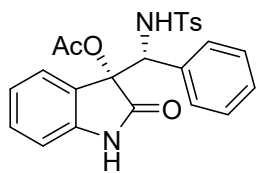
N-Bn



N-0105-A

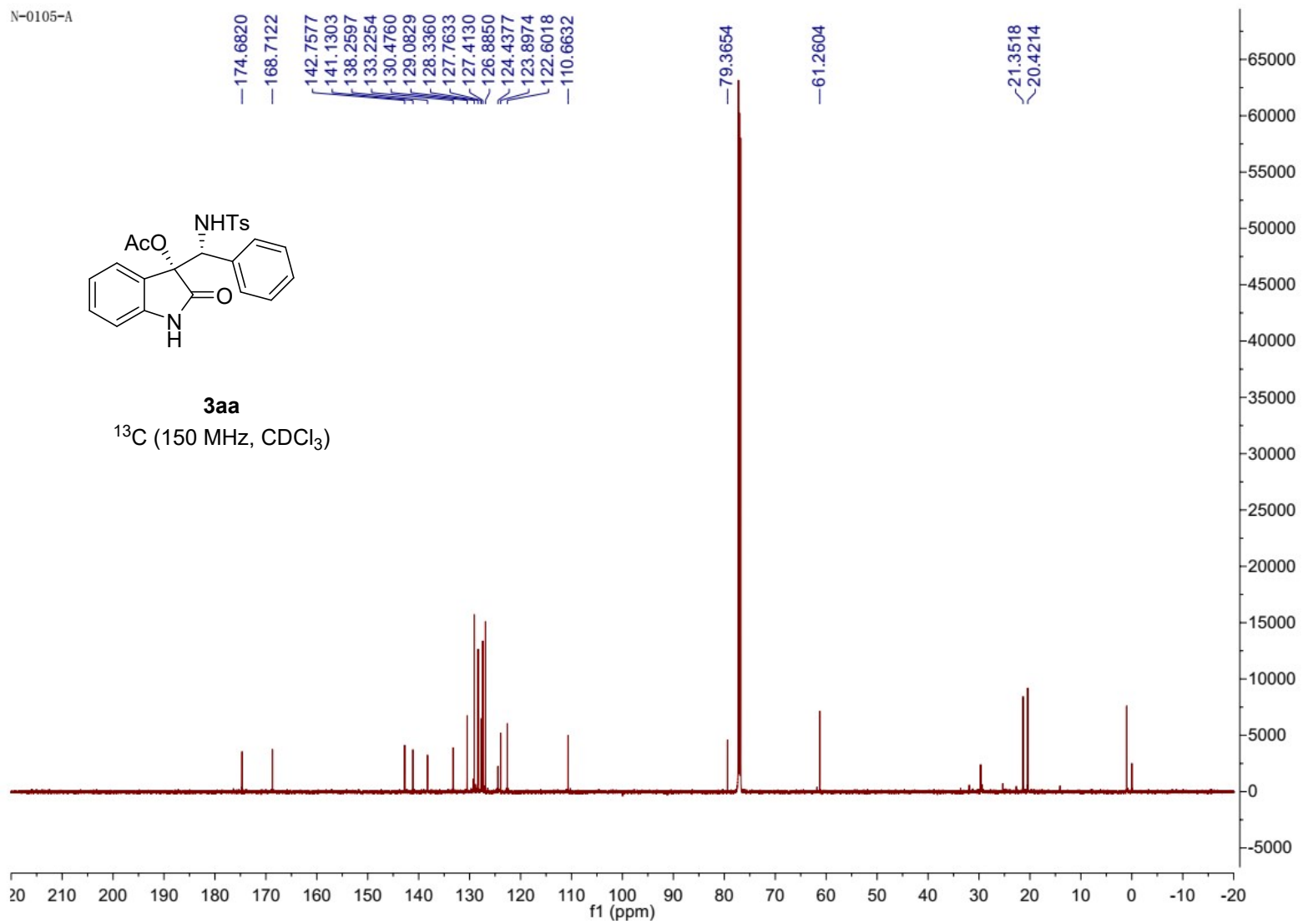


N-0105-A

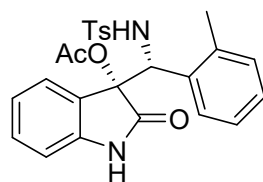


**3aa**

$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )

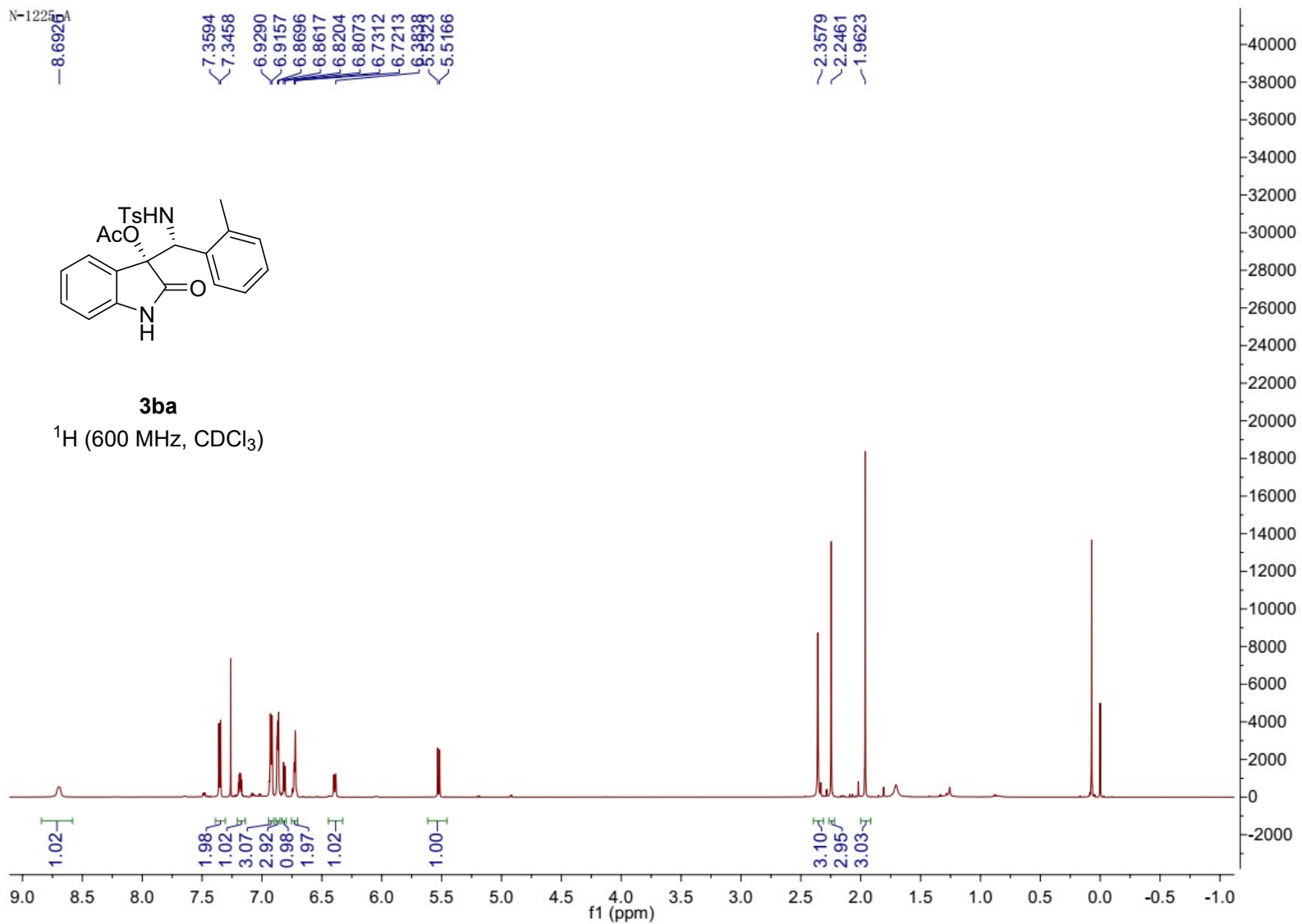


N-1225-A

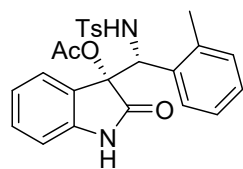


**3ba**

$^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ )

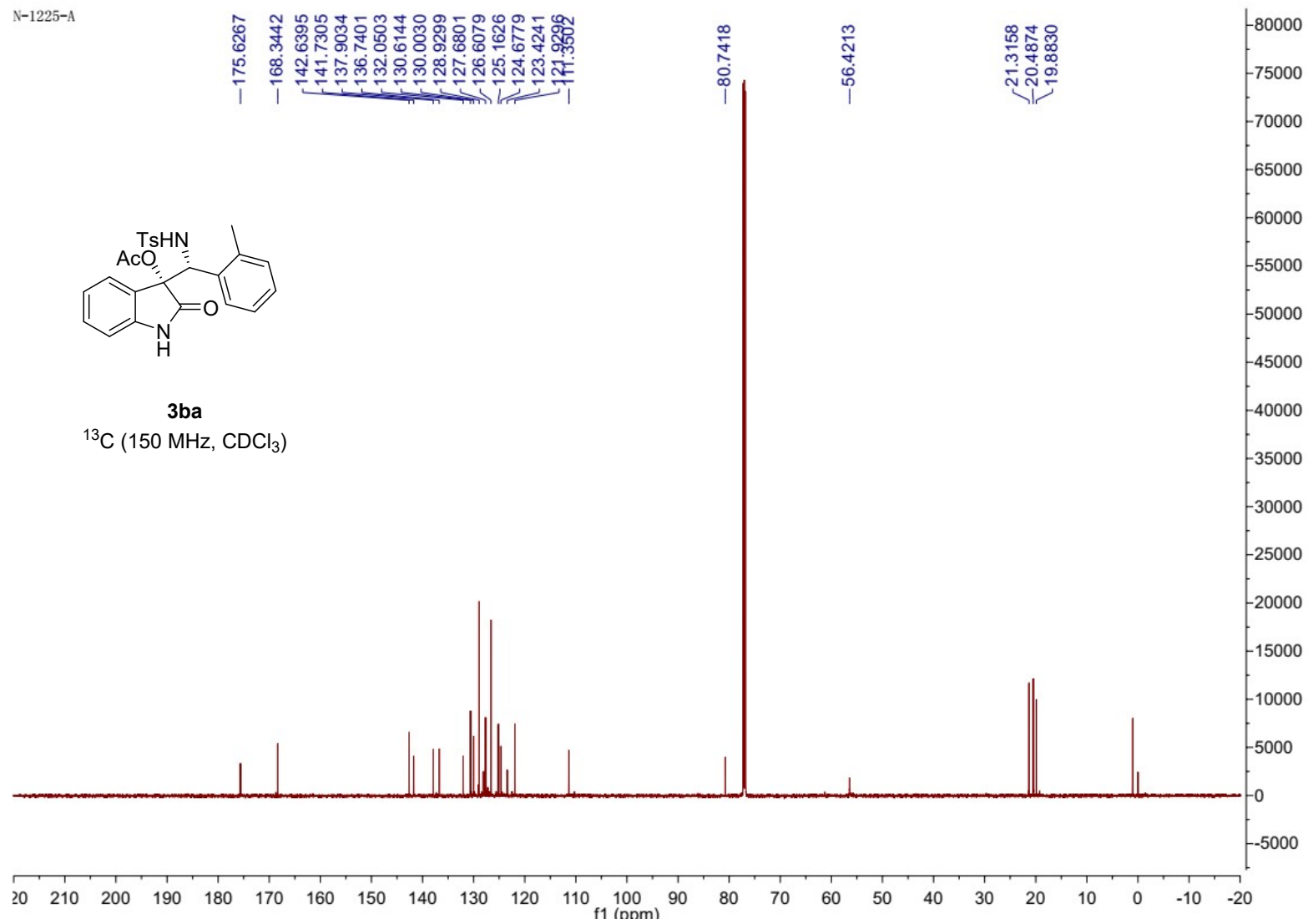


N-1225-A

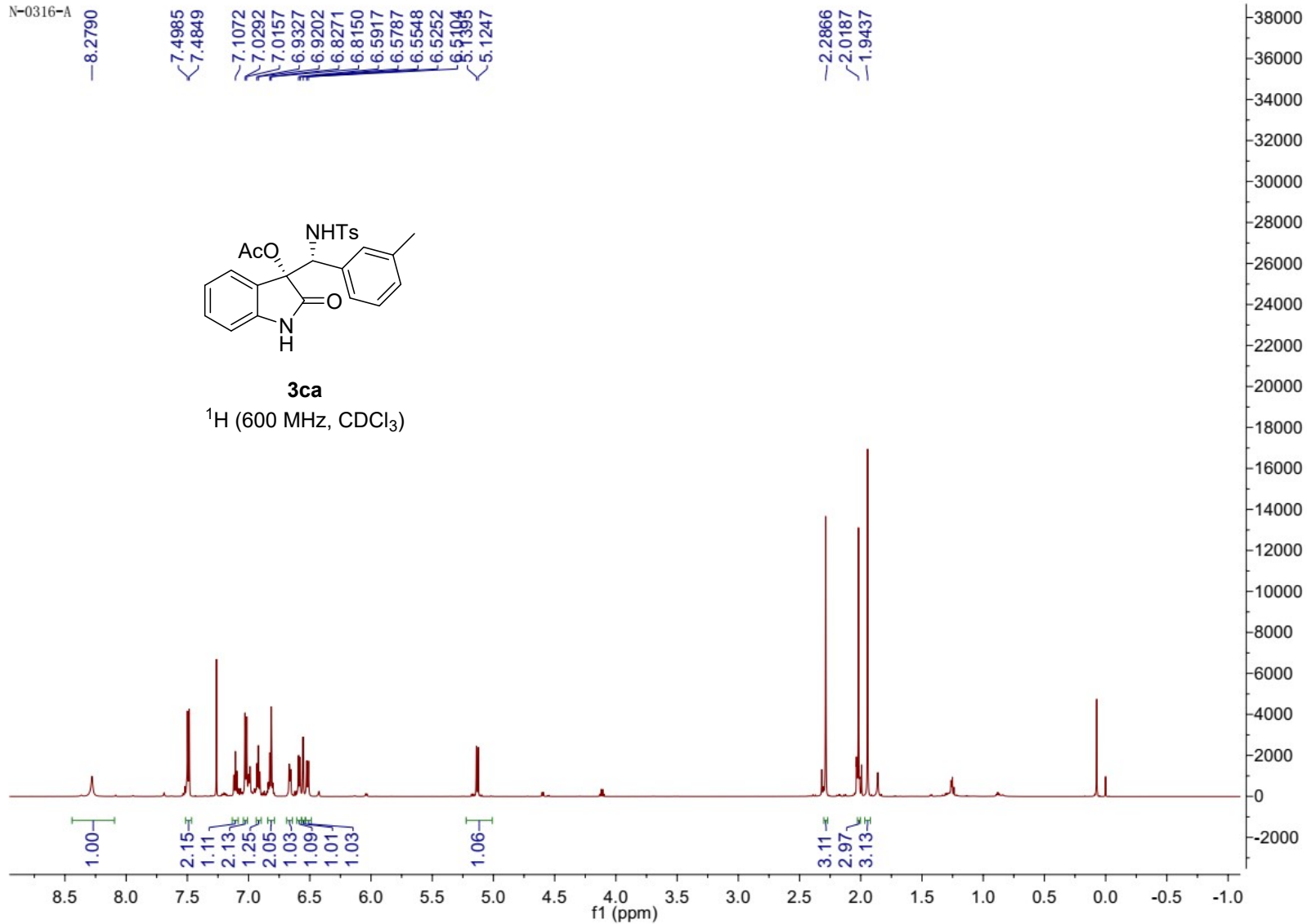


**3ba**

$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )

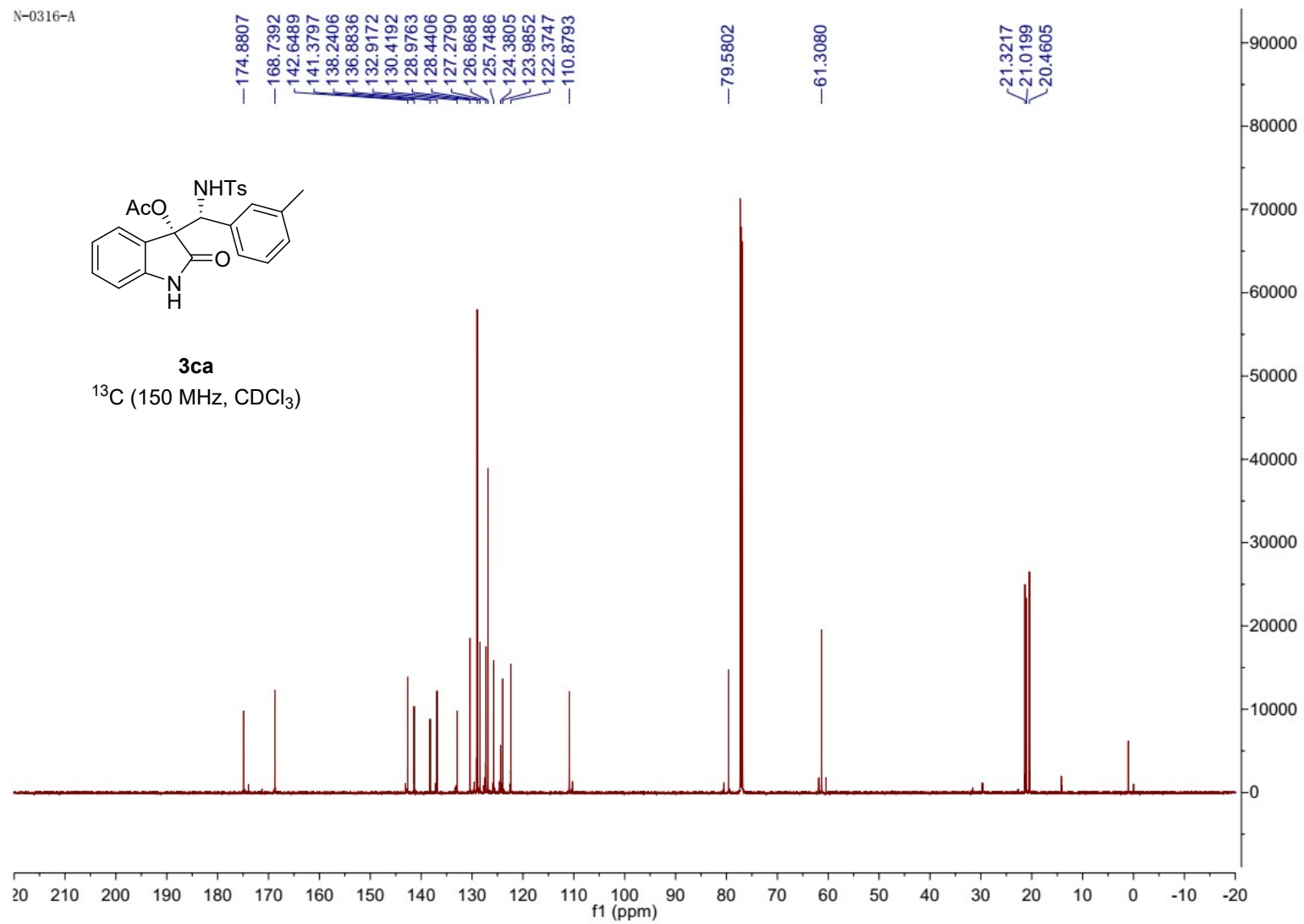


N-0316-A





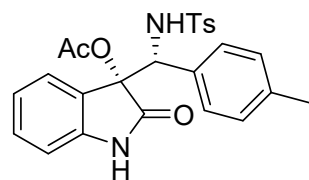
N-0316-A



1225-B

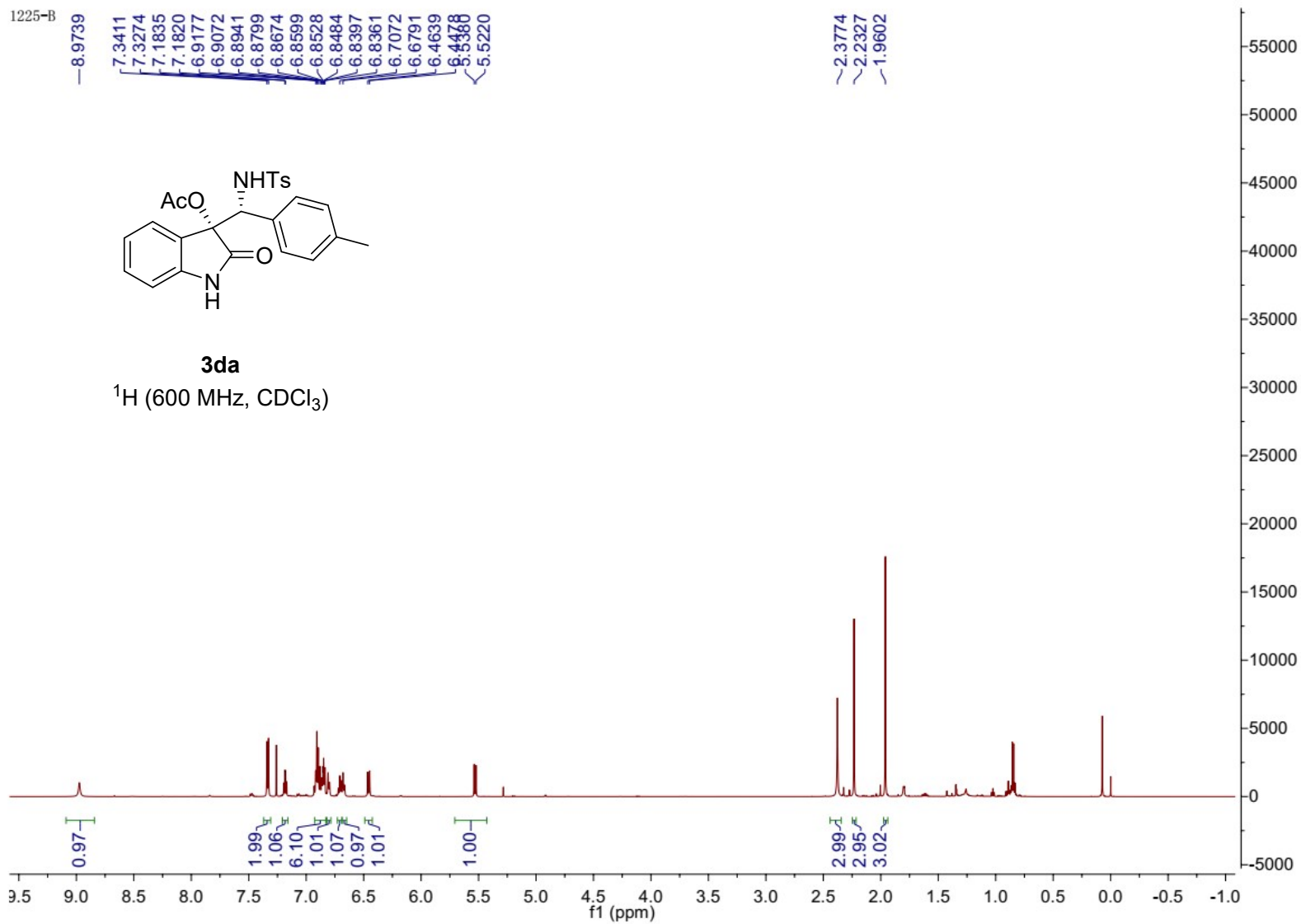
8.9739  
7.3411  
7.3274  
7.1835  
7.1820  
6.9177  
6.9072  
6.8941  
6.8799  
6.8674  
6.8599  
6.8528  
6.8484  
6.8397  
6.8361  
6.7072  
6.6791  
6.4639  
5.5220

2.3774  
2.2327  
1.9602

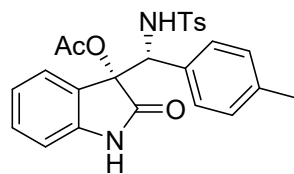


**3da**

<sup>1</sup>H (600 MHz, CDCl<sub>3</sub>)

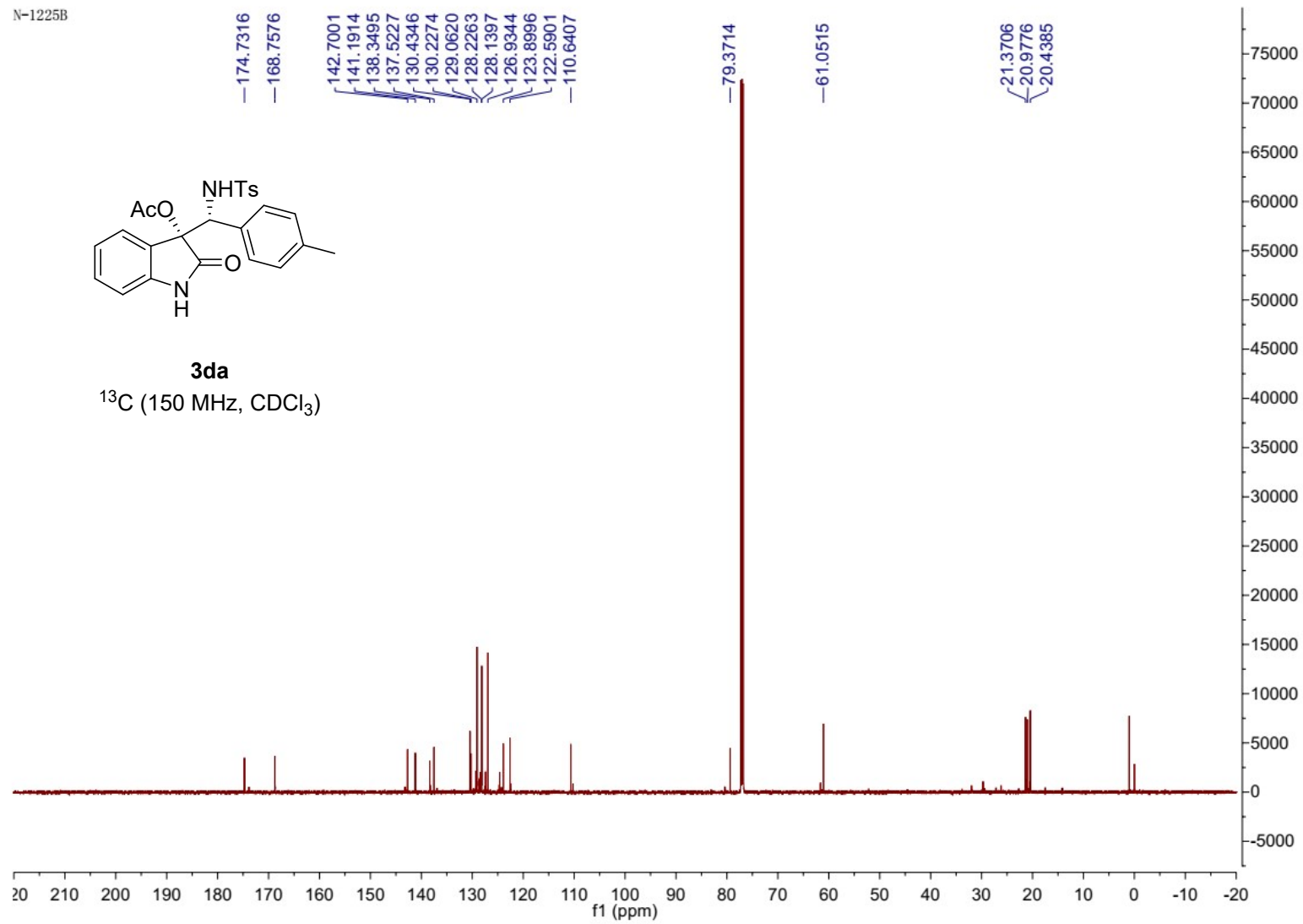


N-1225B

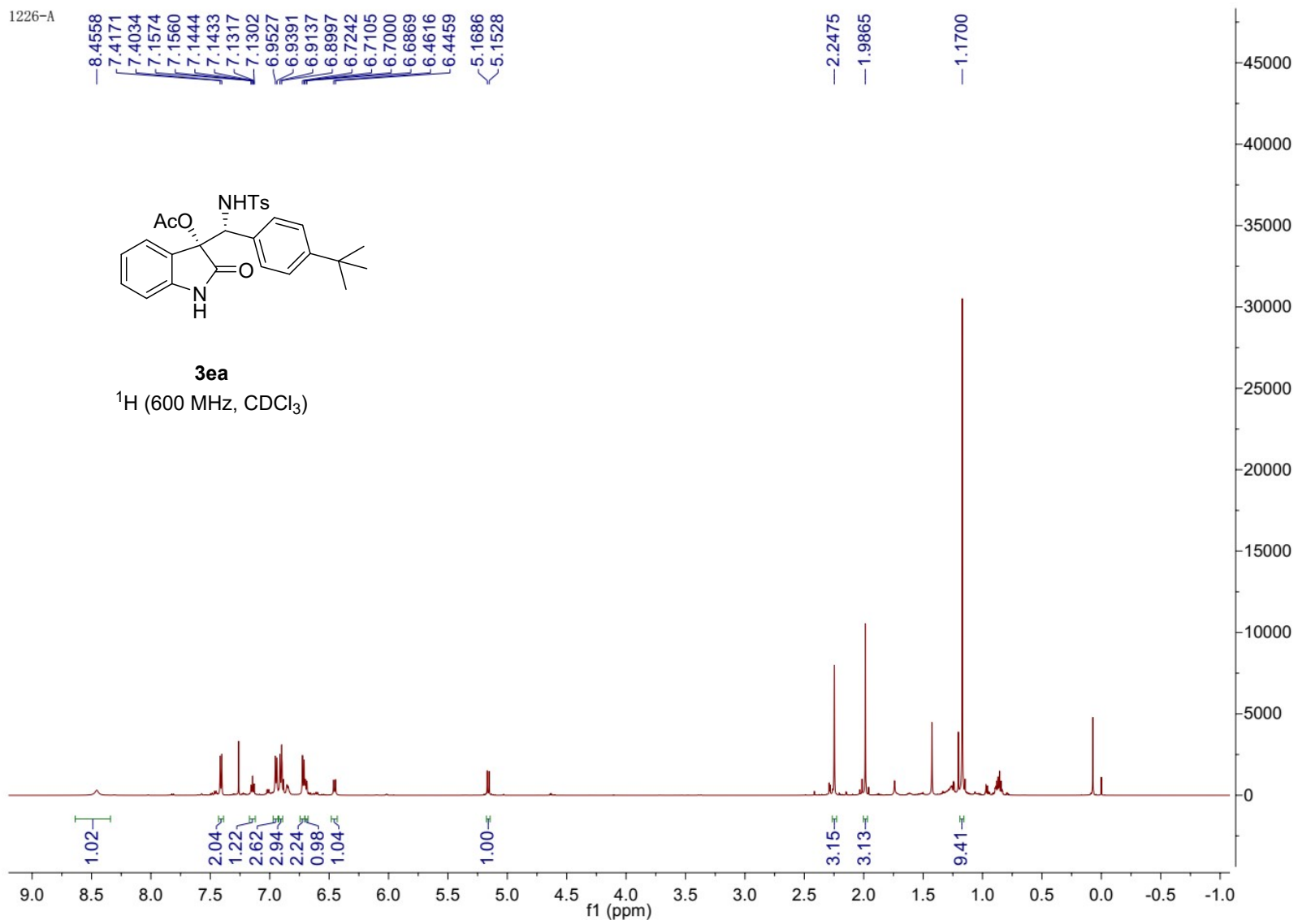


**3da**

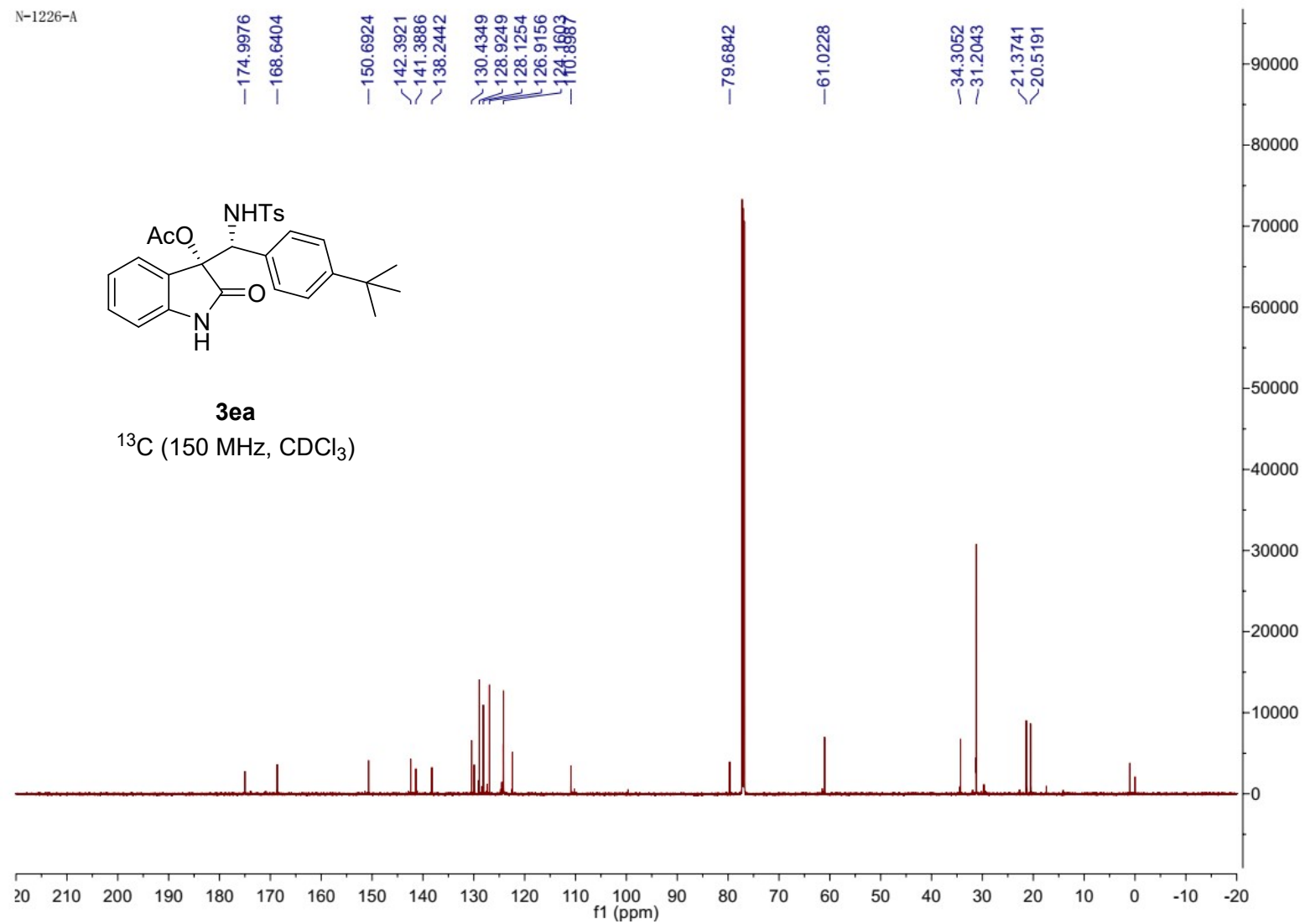
<sup>13</sup>C (150 MHz, CDCl<sub>3</sub>)



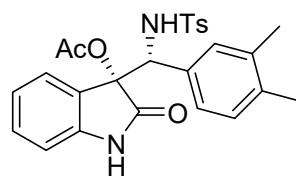
1226-A



N-1226-A

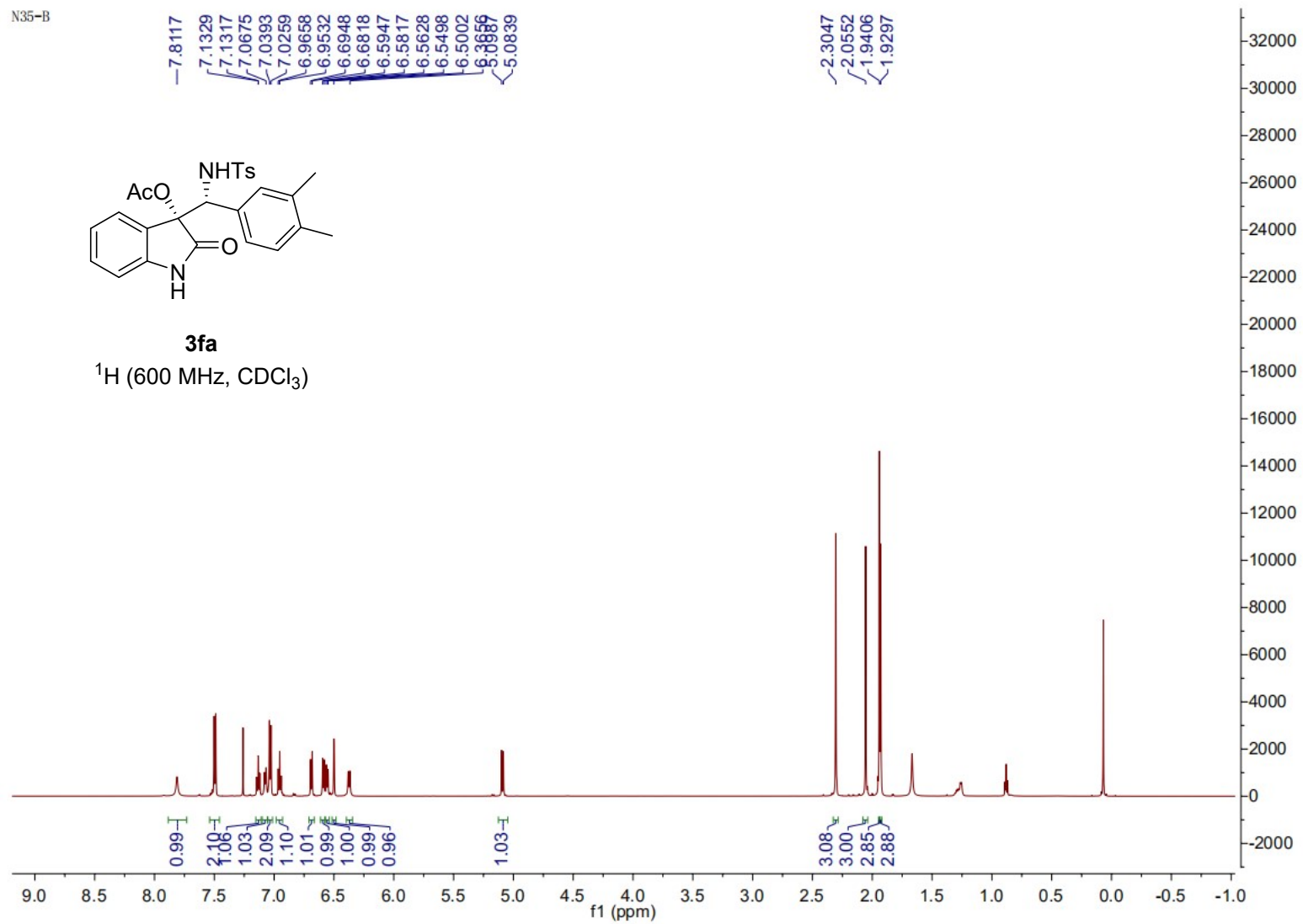


N35-B

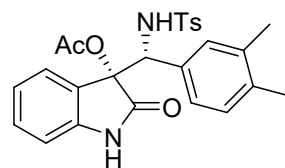


**3fa**

$^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ )

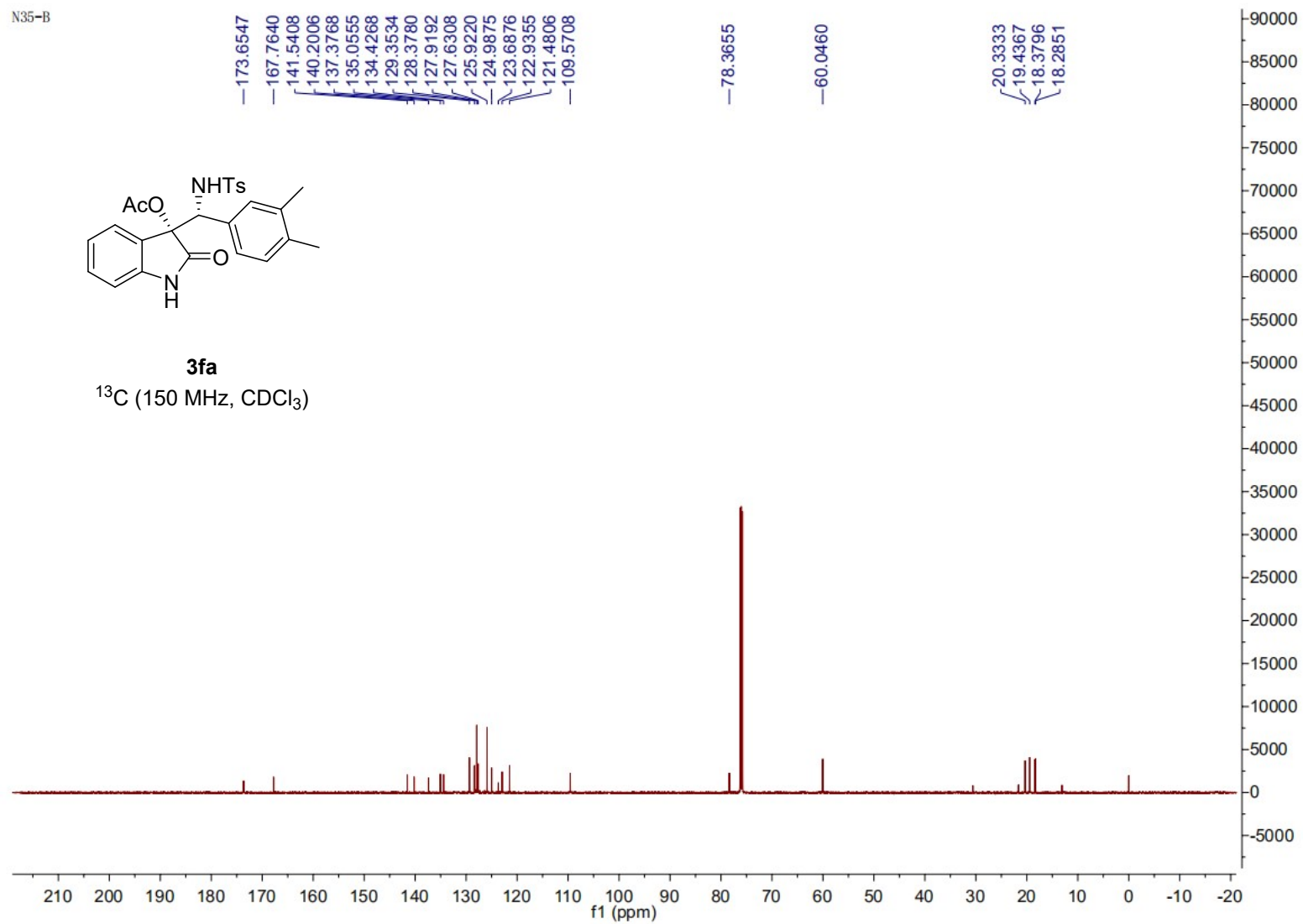


N35-B

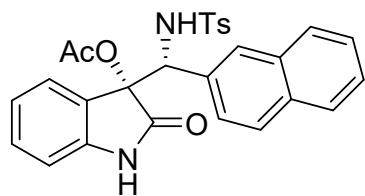


**3fa**

$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )

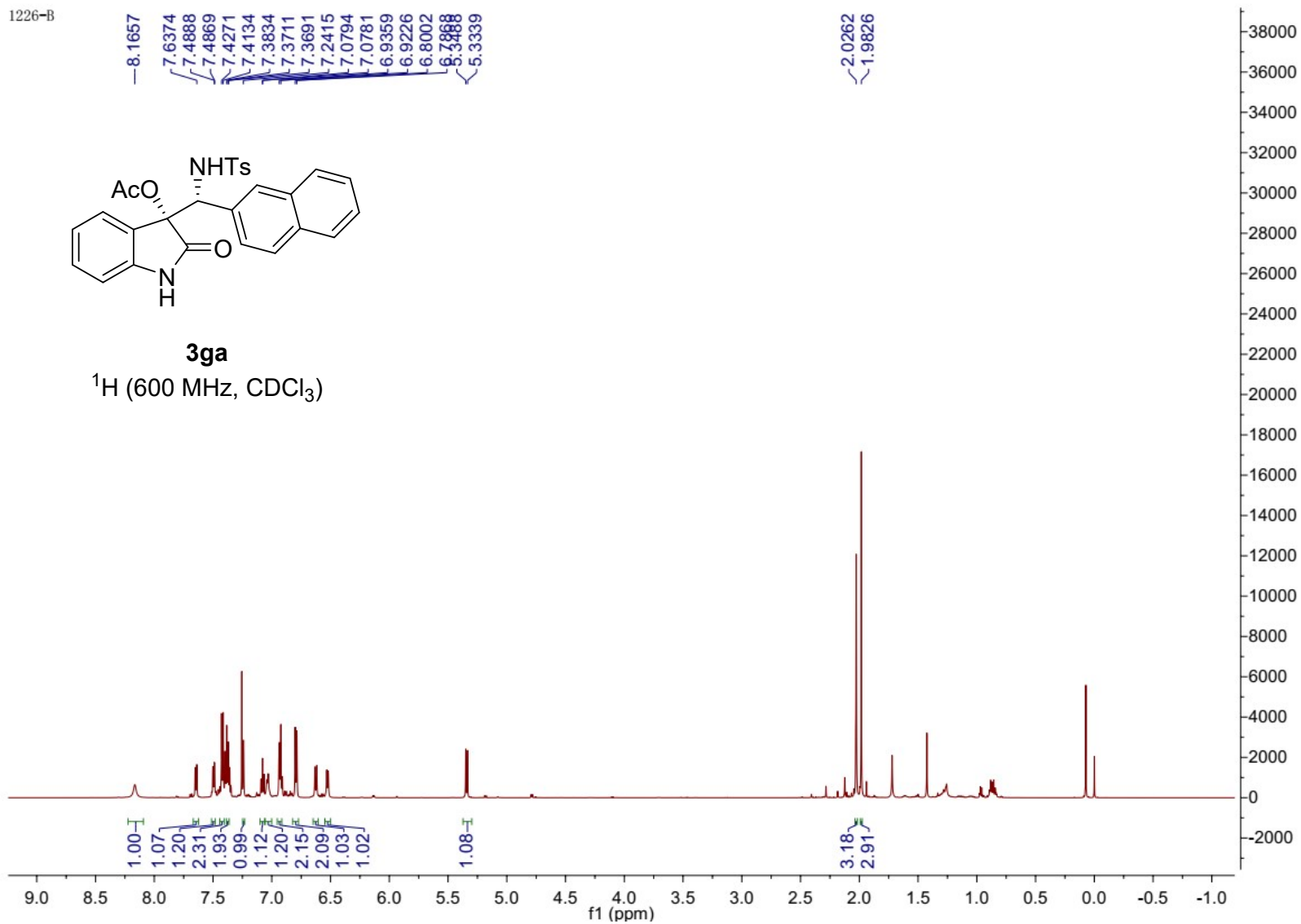


1226-B



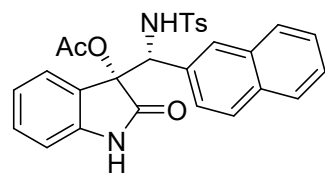
**3ga**

$^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ )

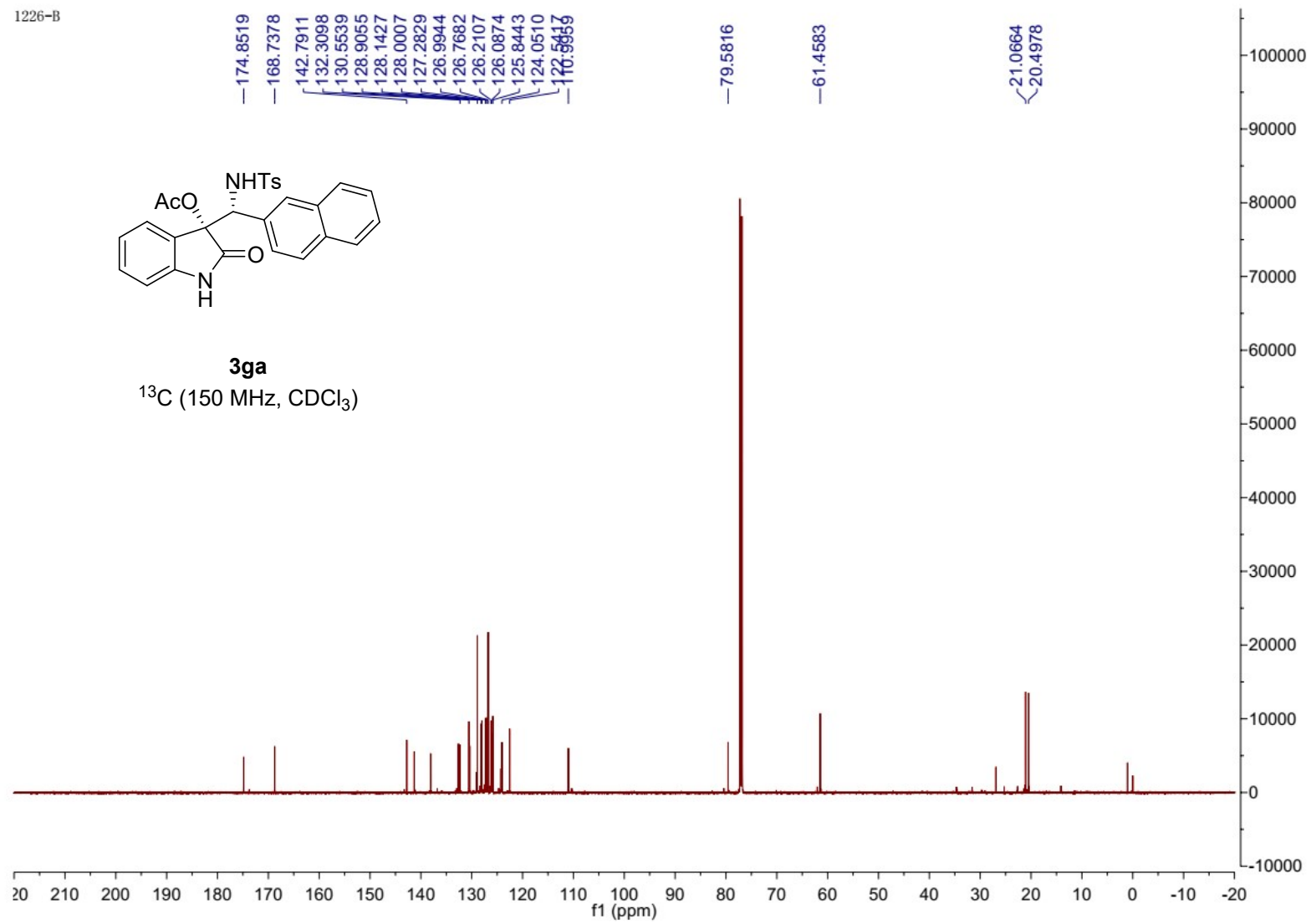


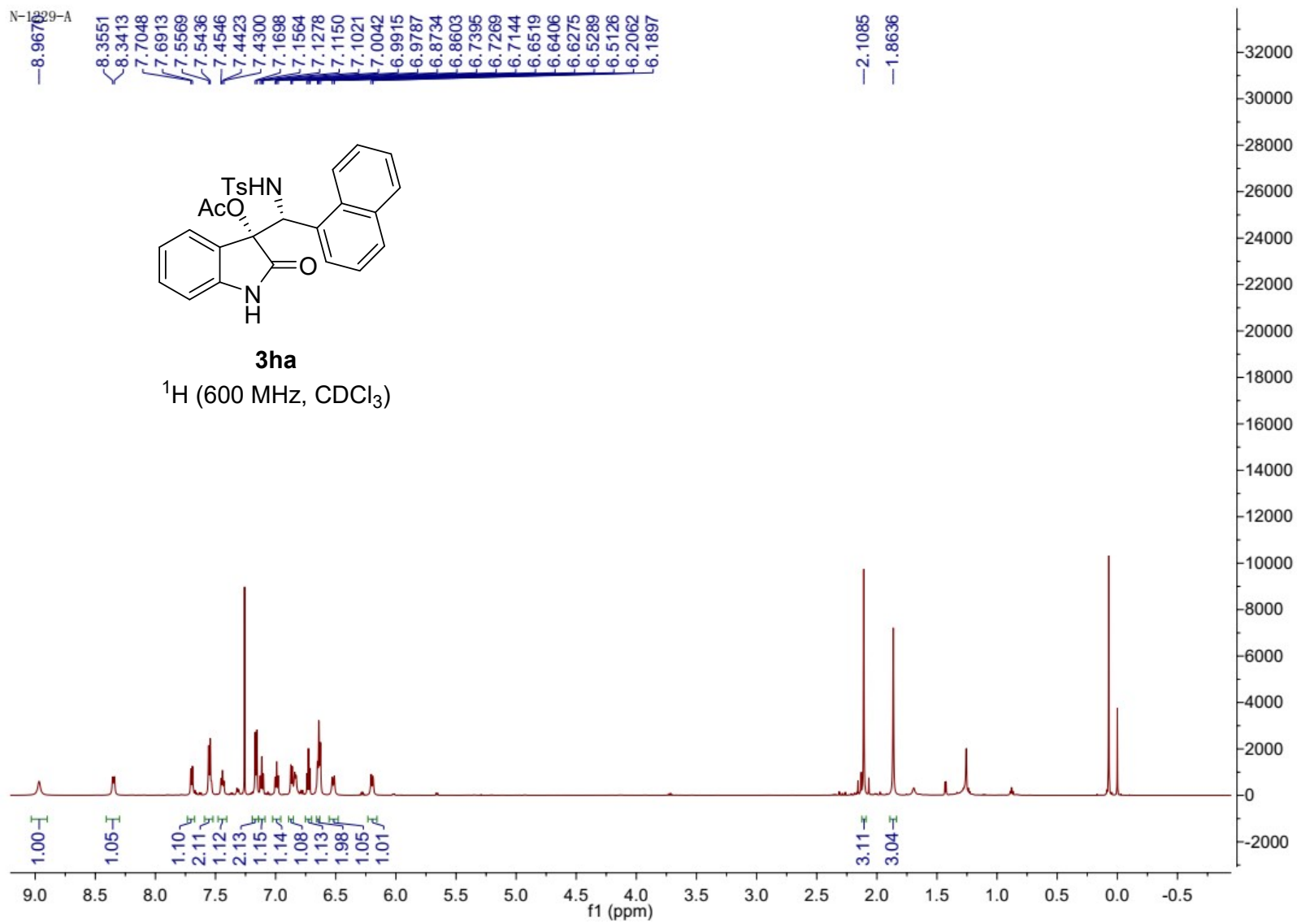


1226-B

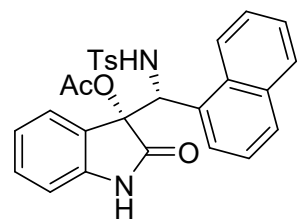


**3ga**  
 $^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )



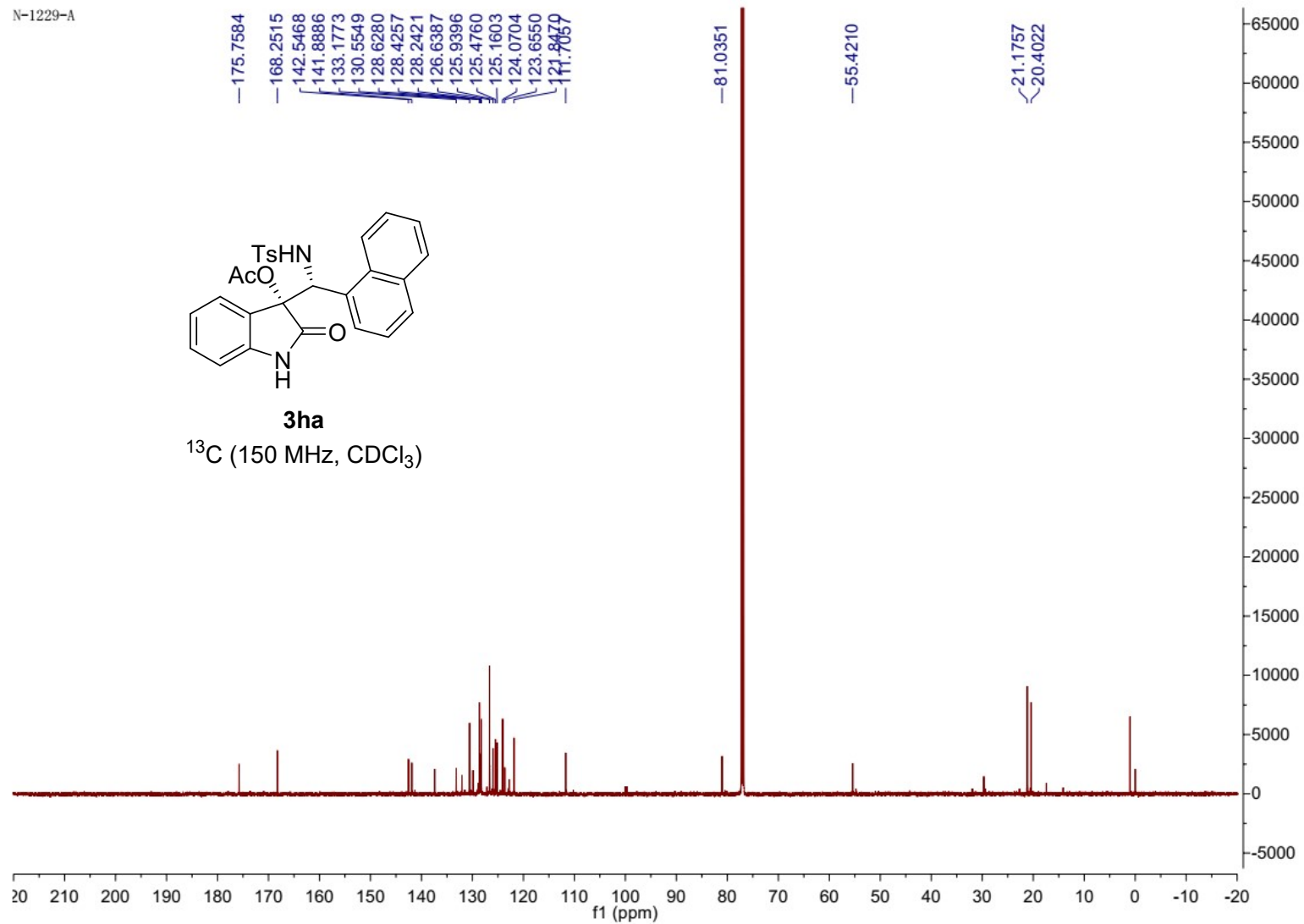


N-1229-A



**3ha**

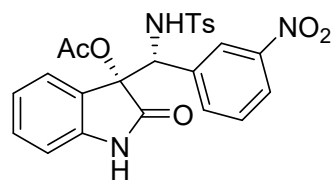
$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )



1226-D

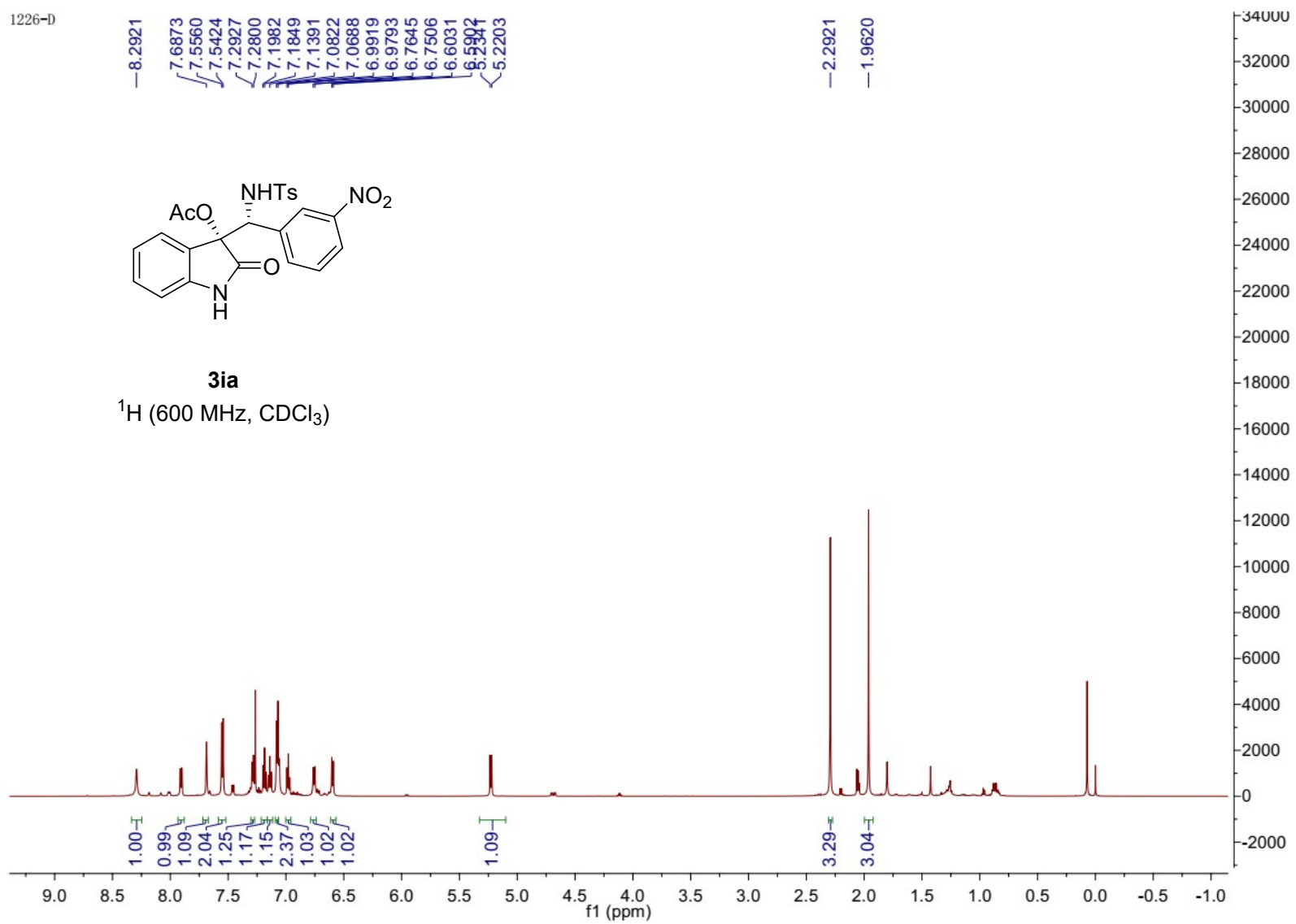
8.2921  
7.6873  
7.5560  
7.5424  
7.2927  
7.2800  
7.1982  
7.1849  
7.1391  
7.0822  
7.0688  
6.9919  
6.9793  
6.7645  
6.7506  
6.6031  
5.5907  
5.2203

2.2921  
1.9620

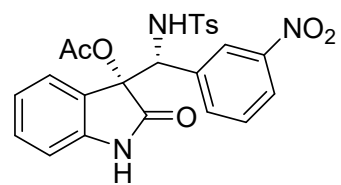


**3ia**

<sup>1</sup>H (600 MHz, CDCl<sub>3</sub>)

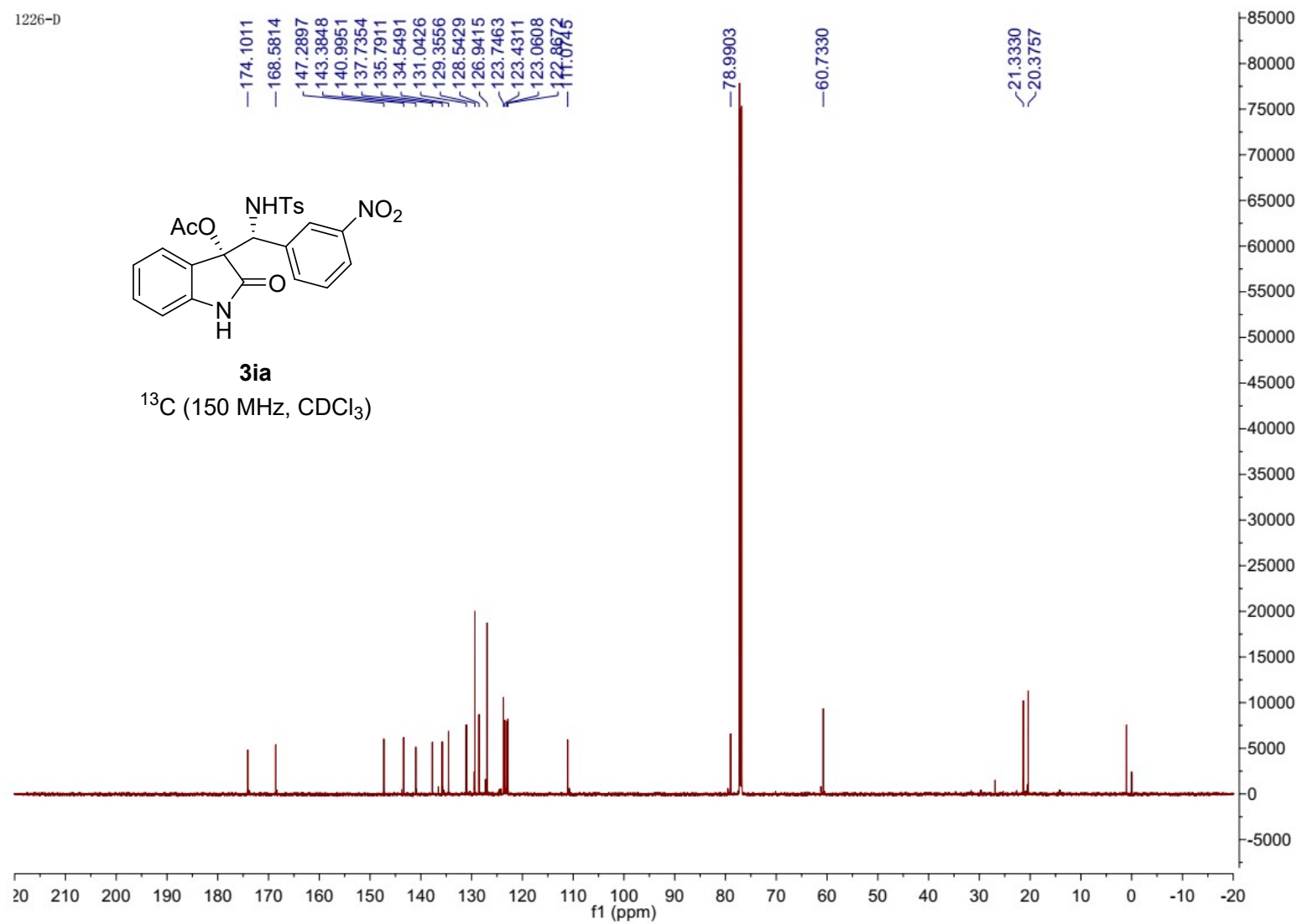


1226-D

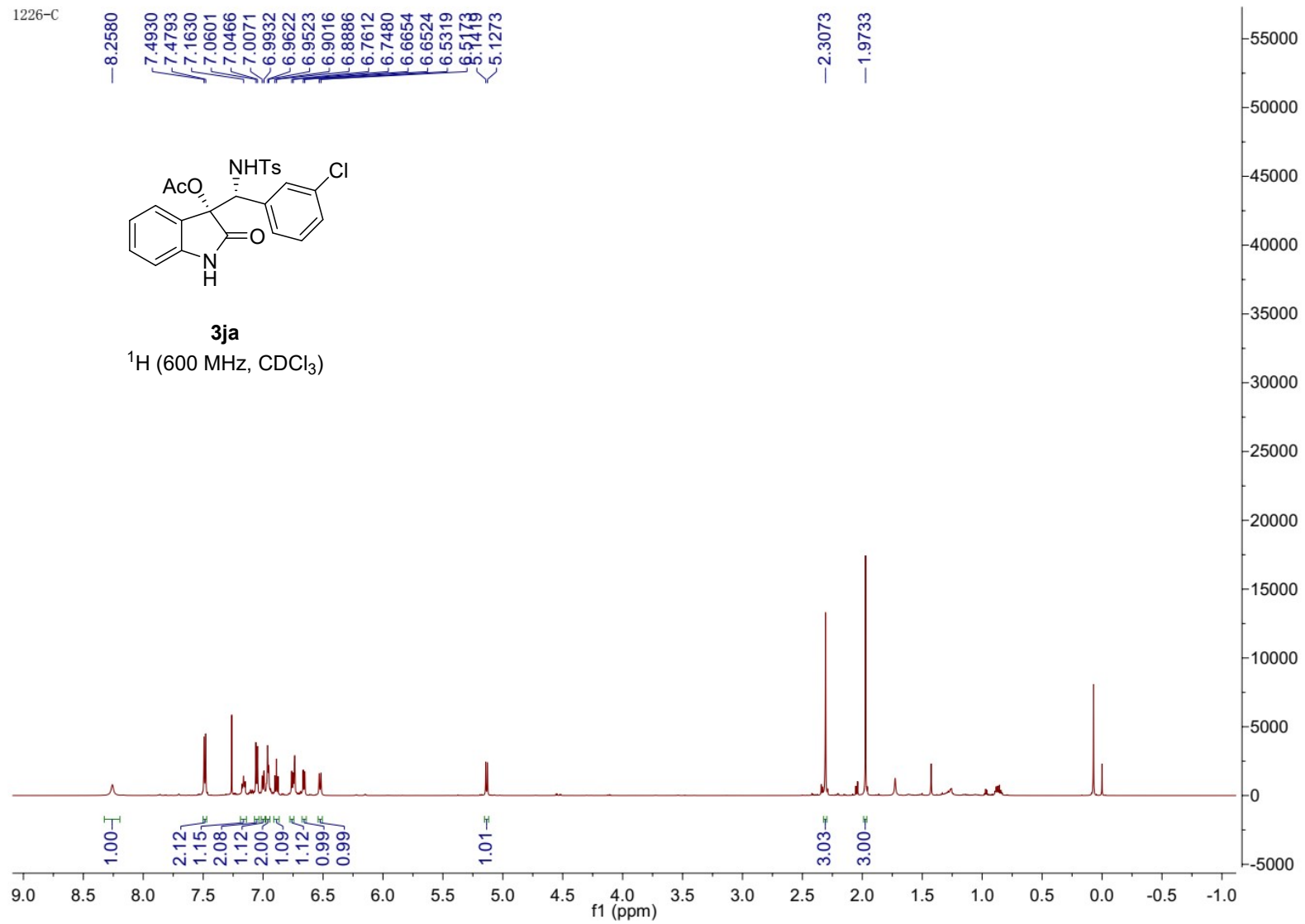


**3ia**

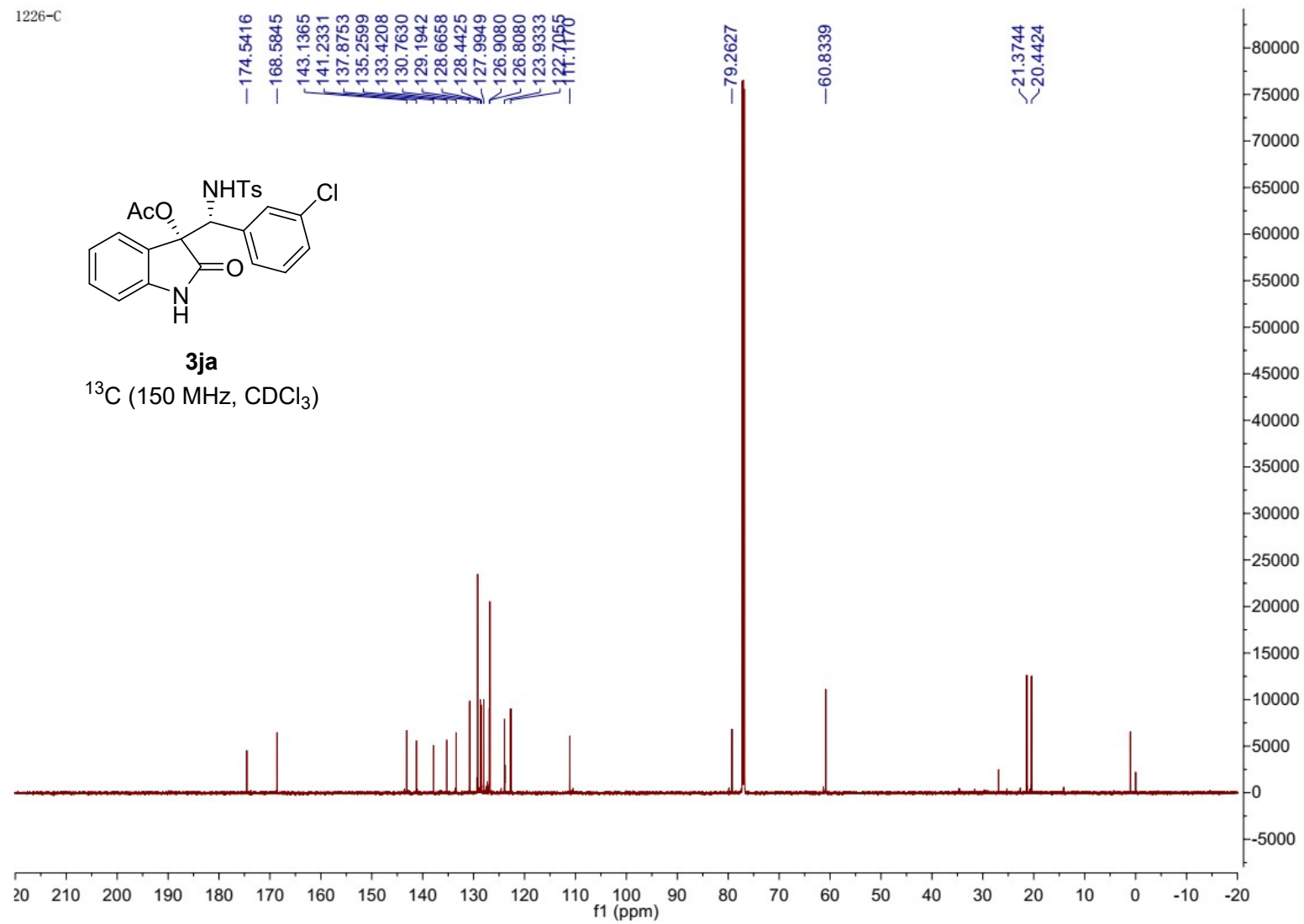
<sup>13</sup>C (150 MHz, CDCl<sub>3</sub>)



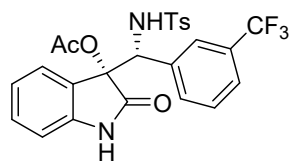
1226-C



1226-C

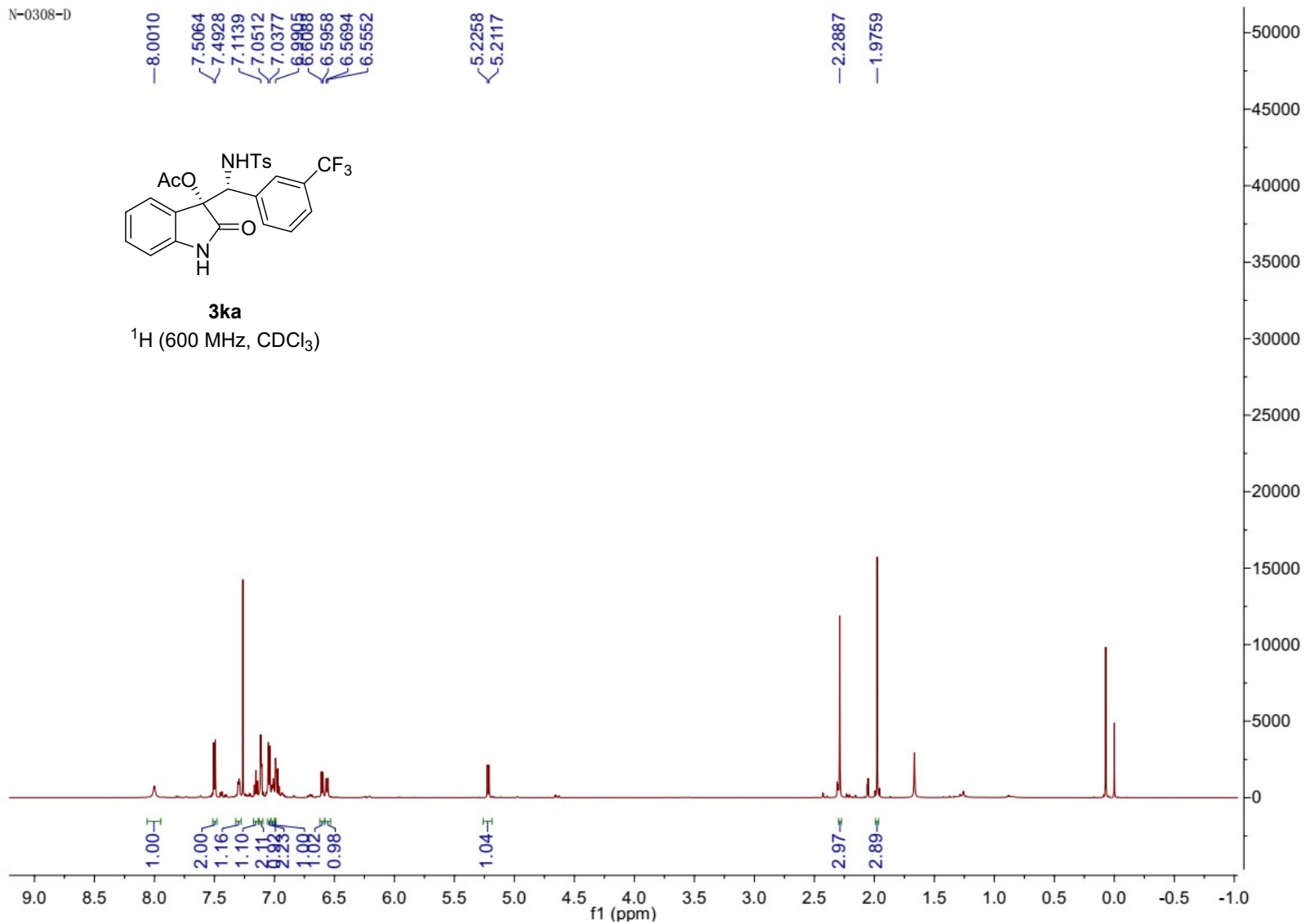


N-0308-D



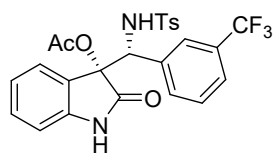
**3ka**

<sup>1</sup>H (600 MHz, CDCl<sub>3</sub>)



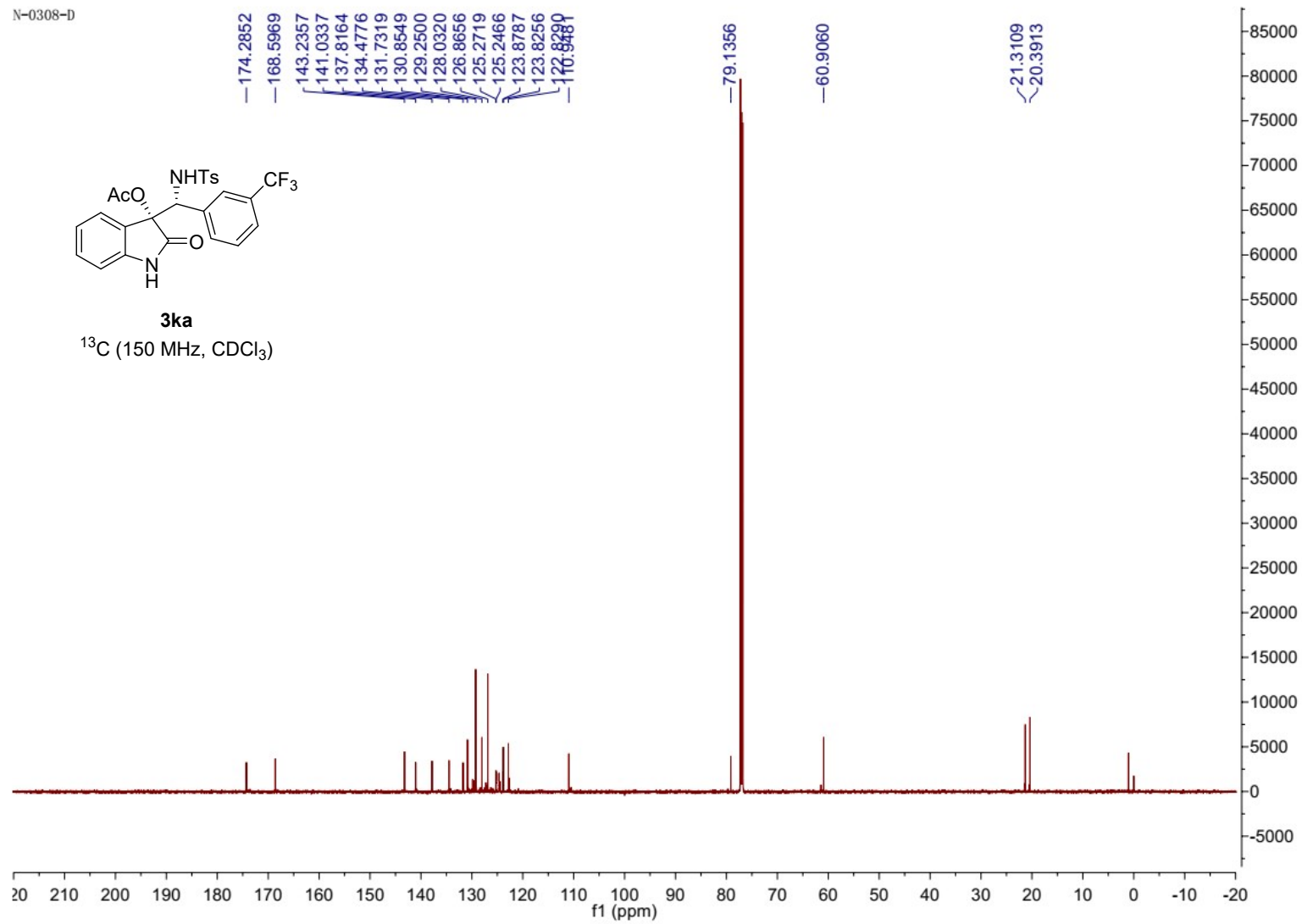


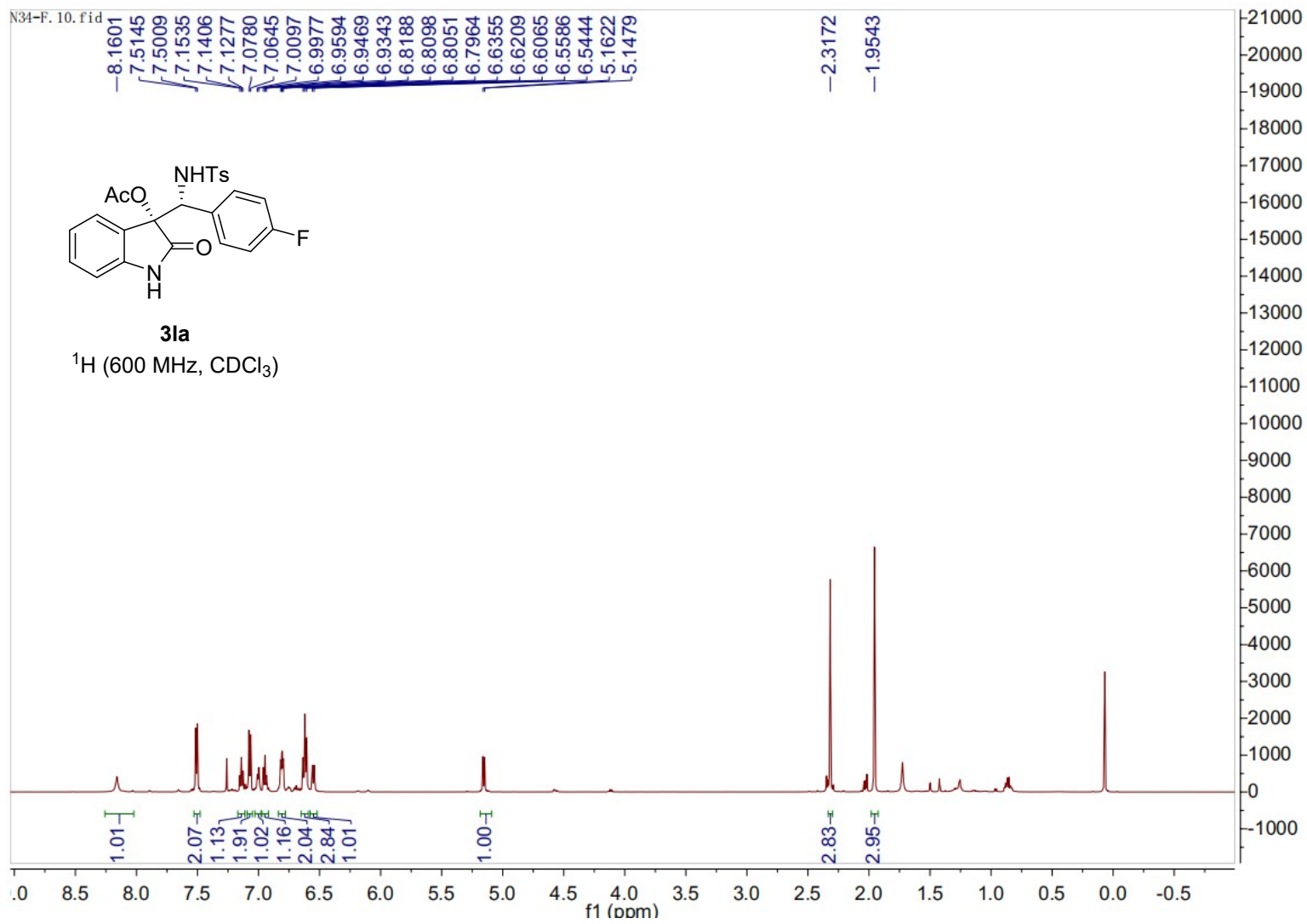
N-0308-D

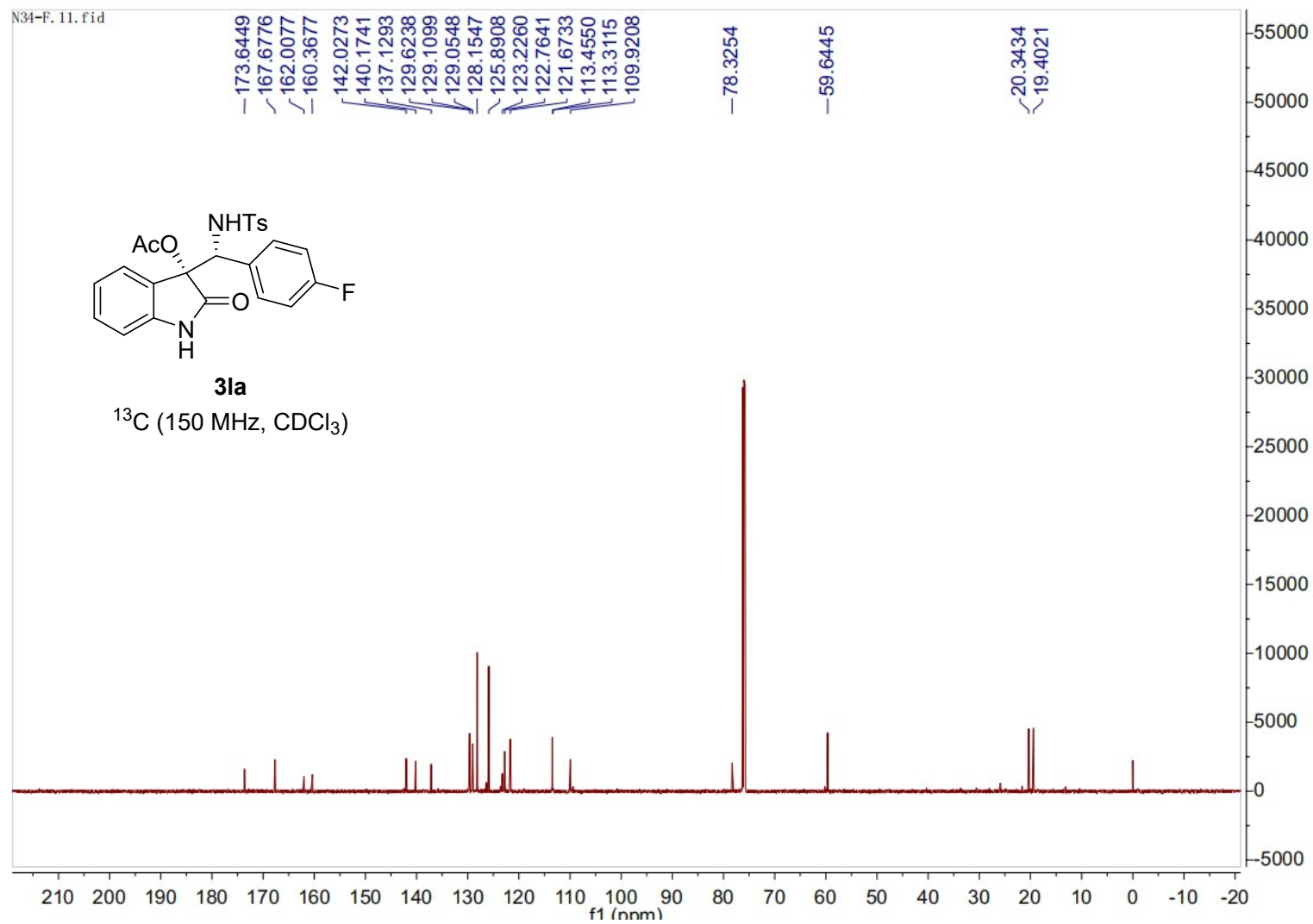


**3ka**

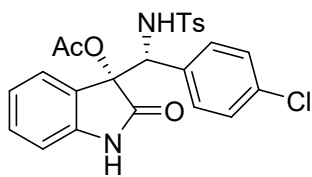
<sup>13</sup>C (150 MHz, CDCl<sub>3</sub>)





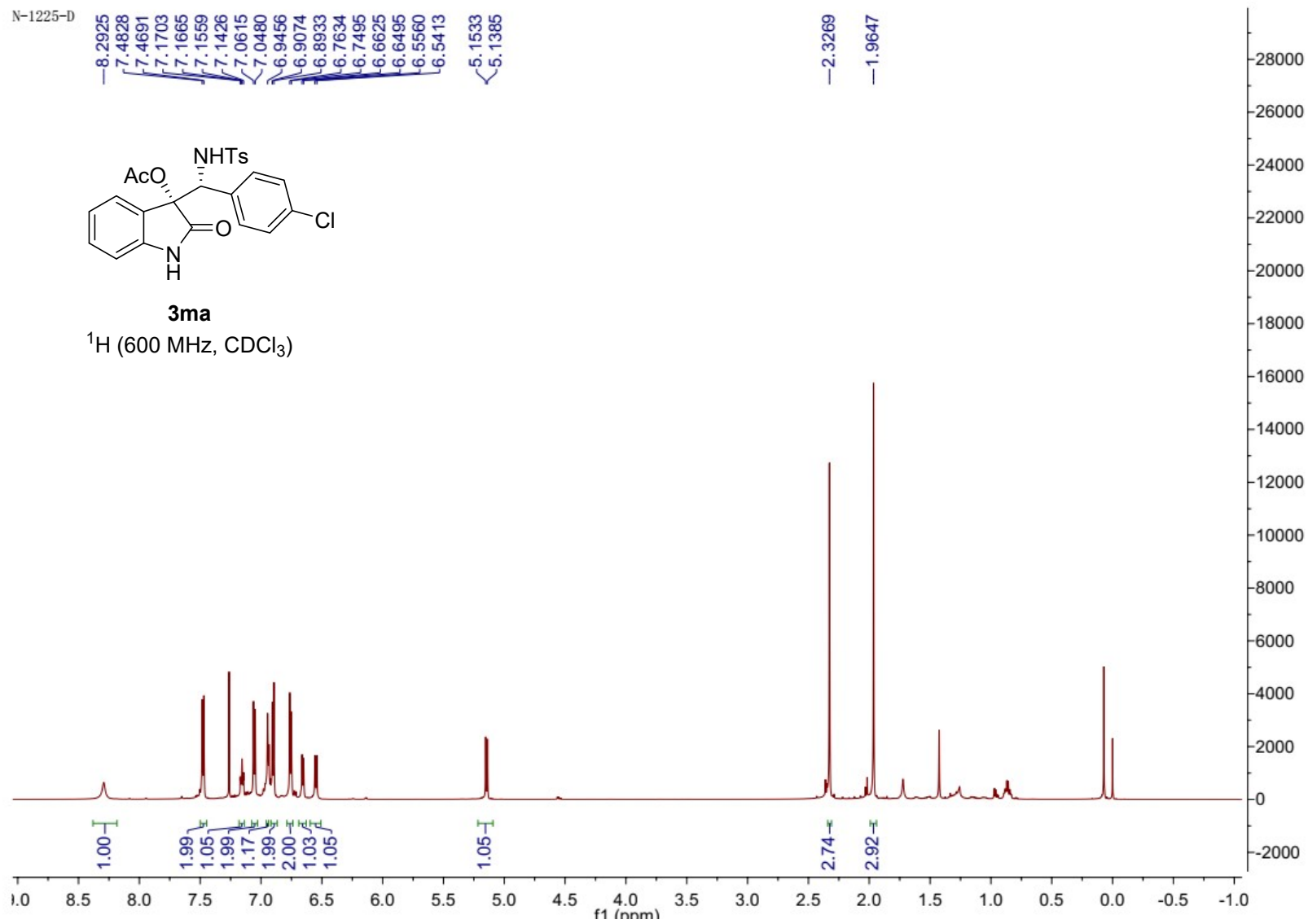


N-1225-D

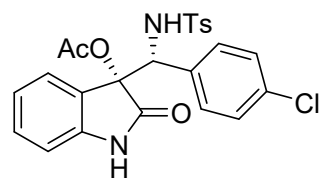


**3ma**

$^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ )

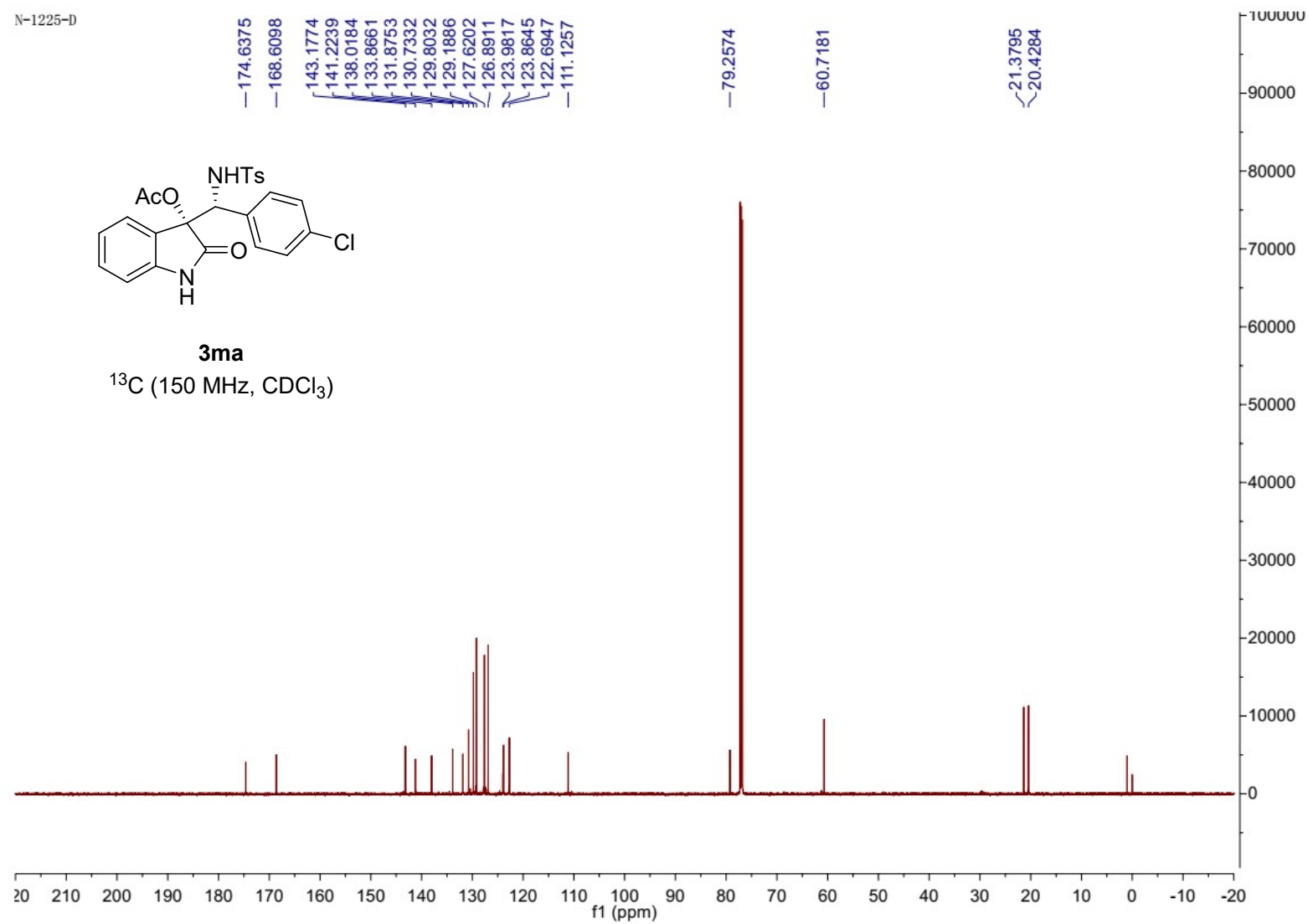


N-1225-D

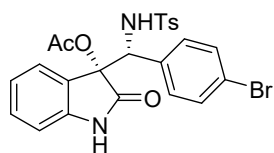


**3ma**

$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )

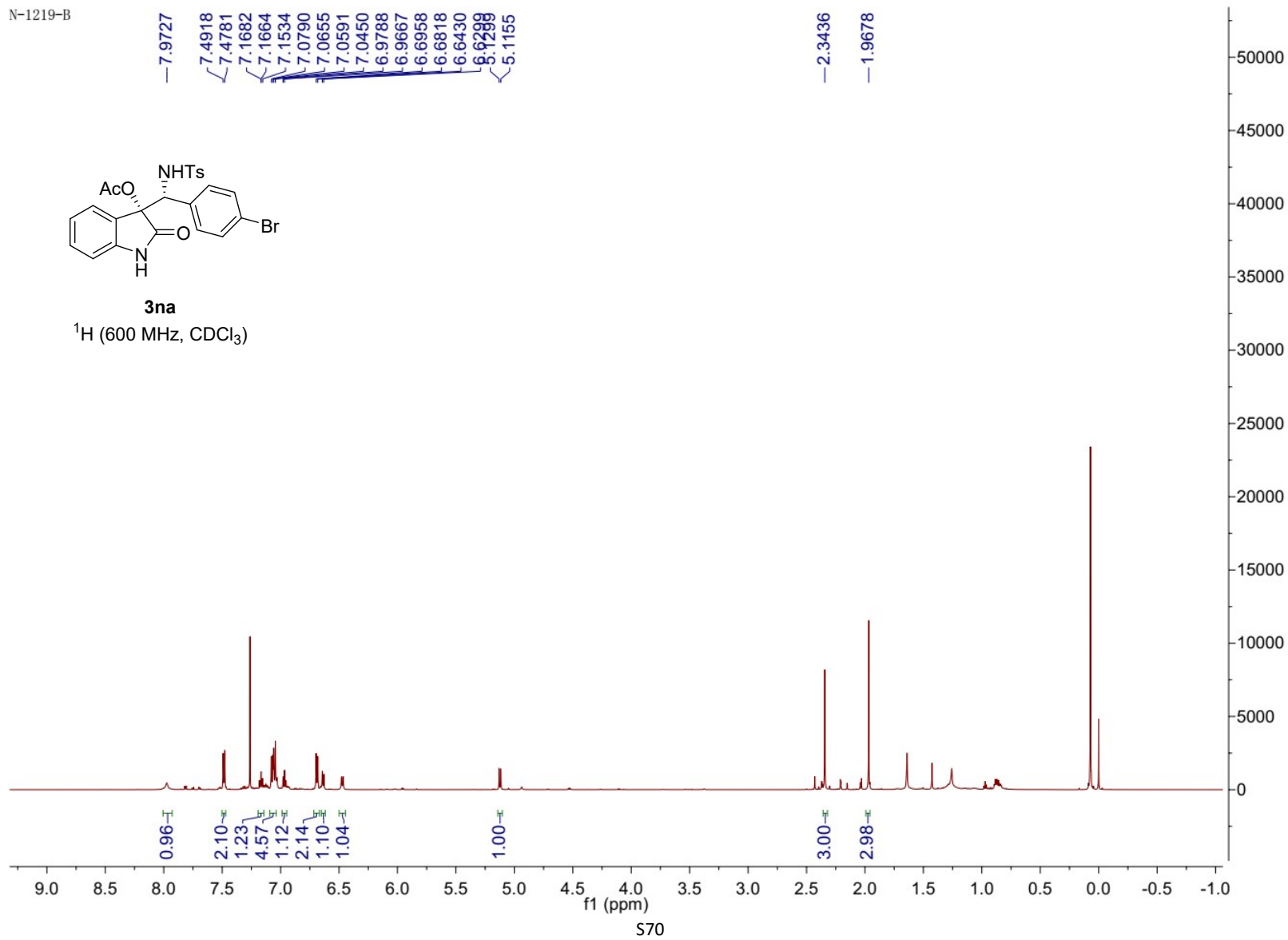


N-1219-B

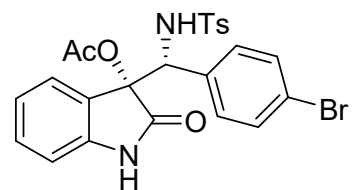


**3na**

$^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ )

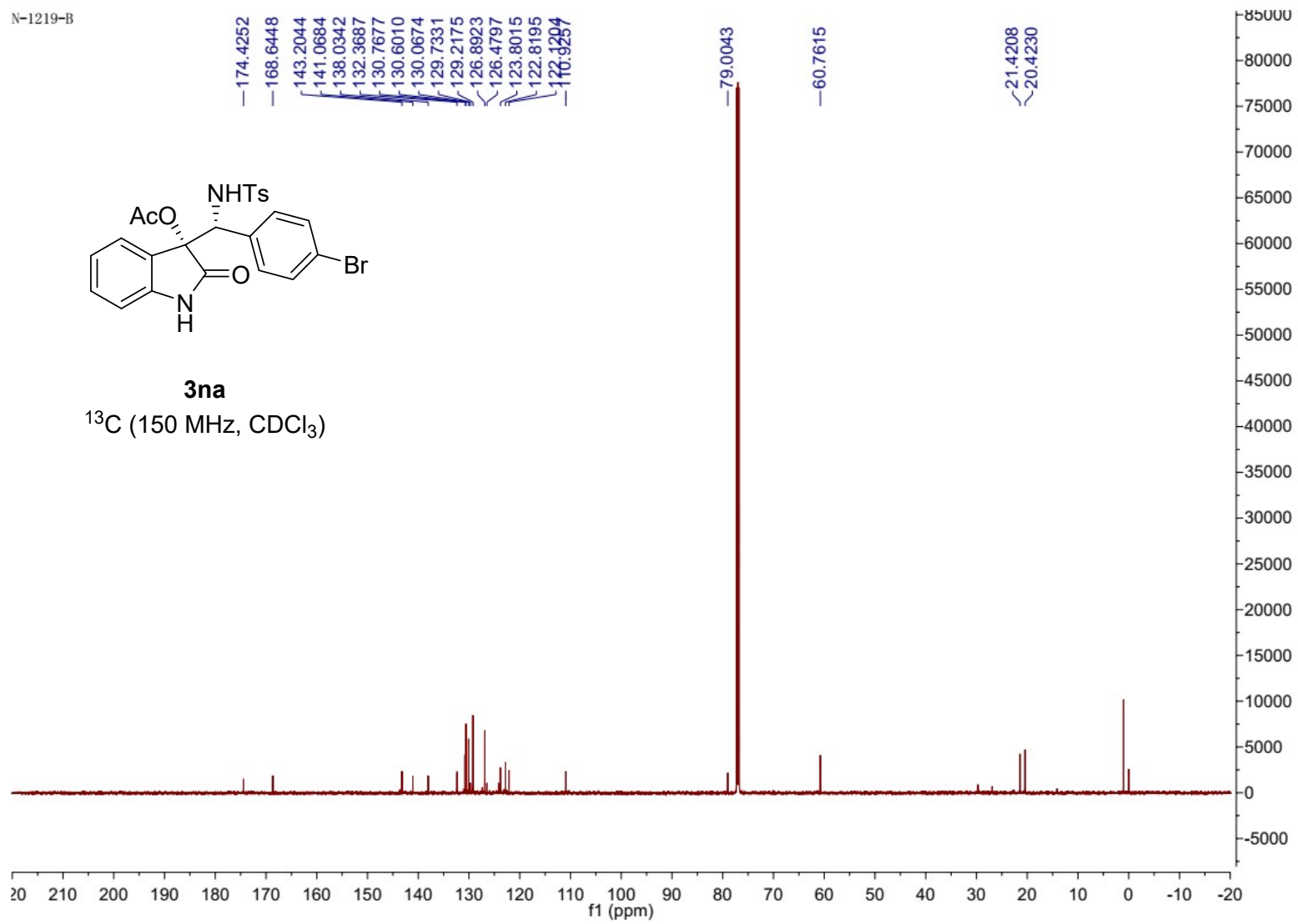


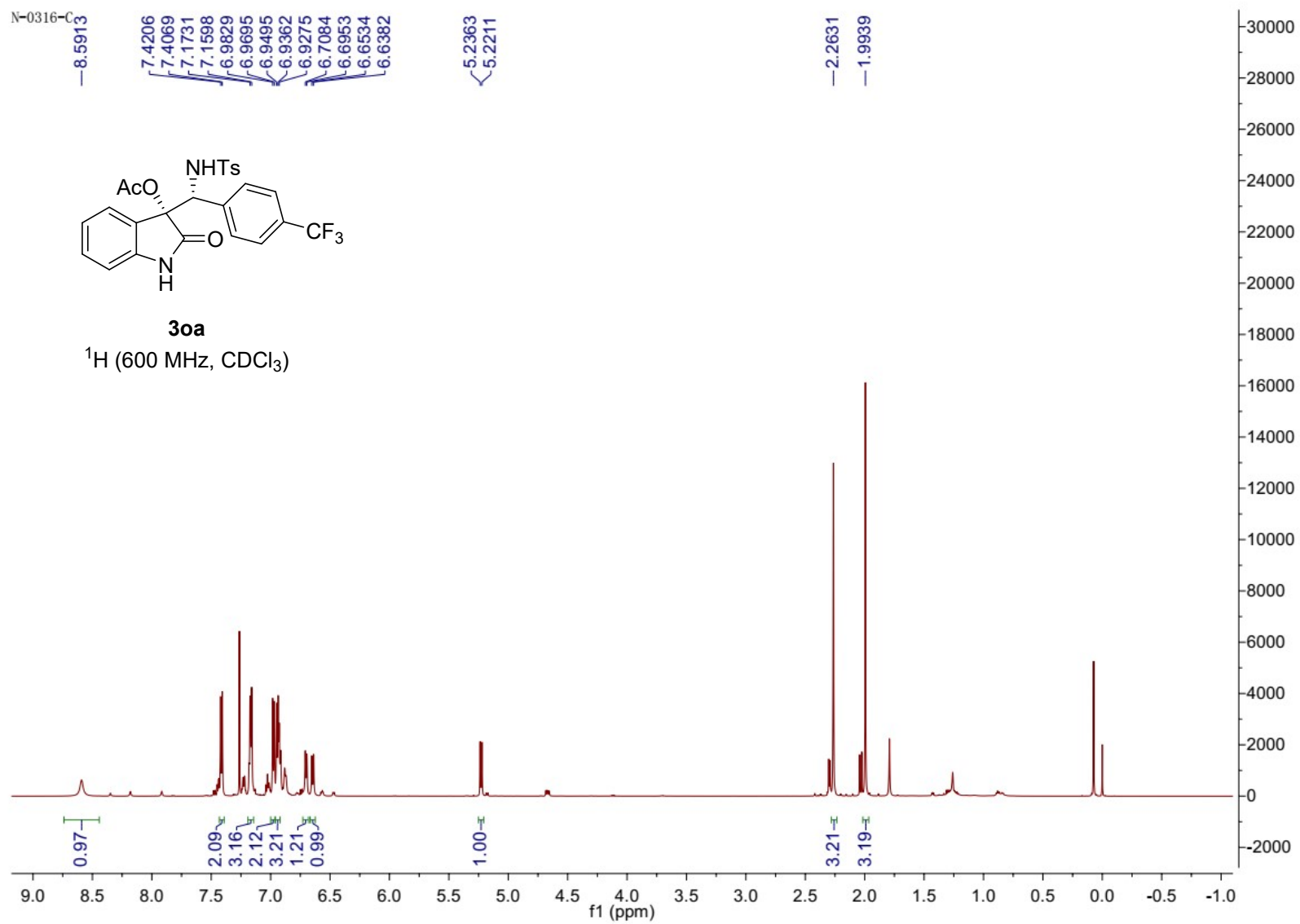
N-1219-B



**3na**

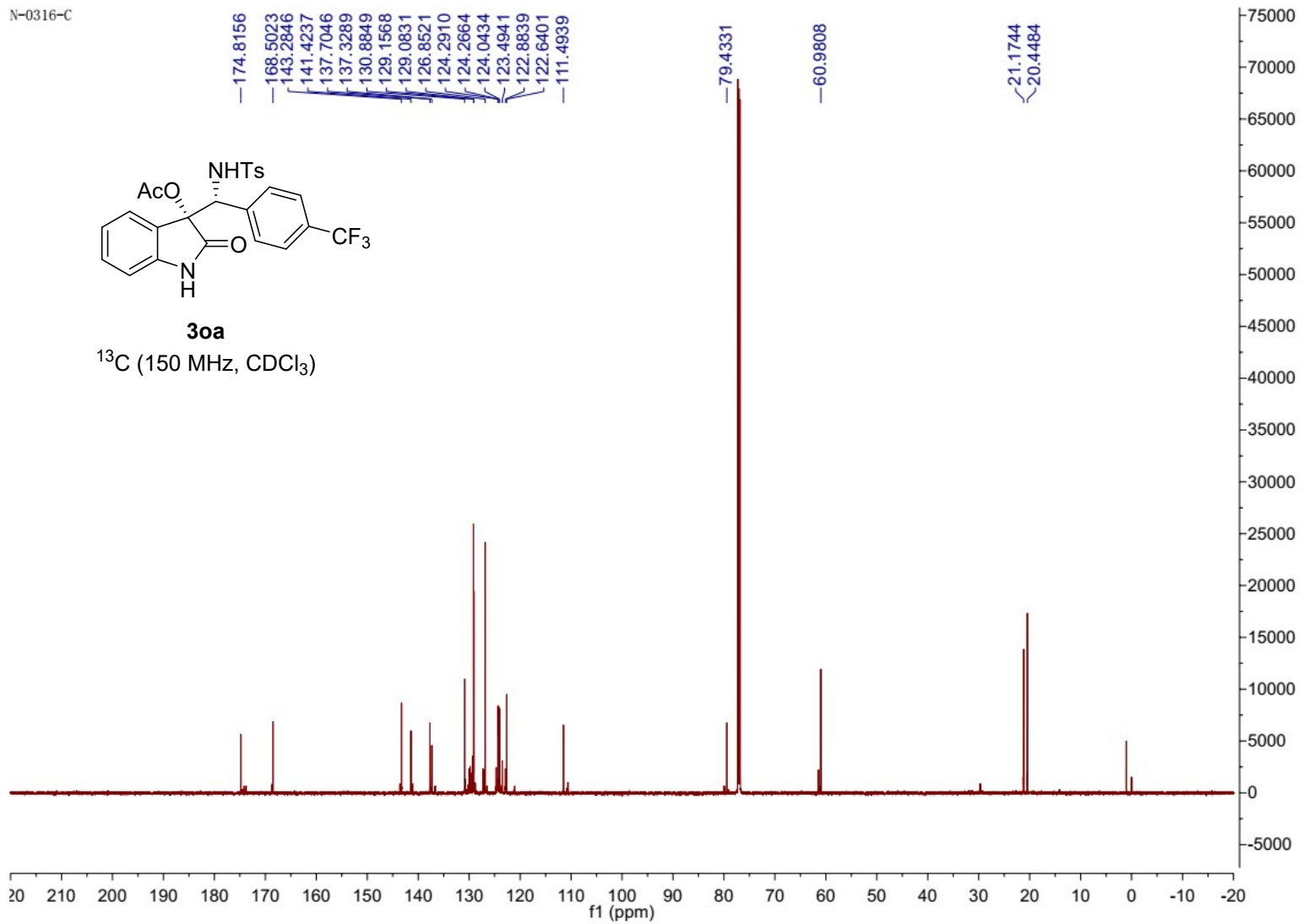
$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )



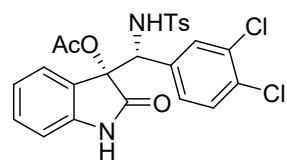




N-0316-C

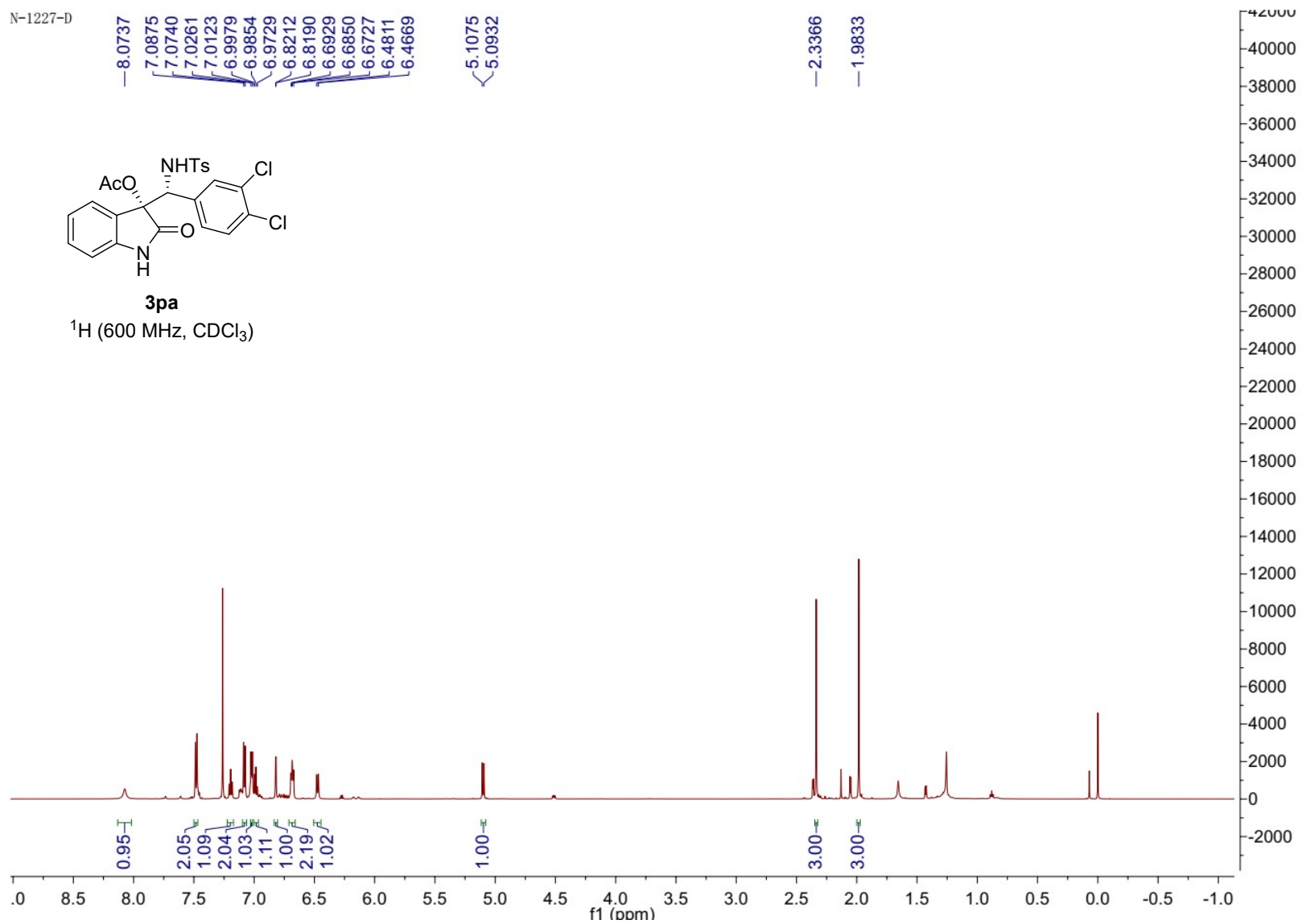


N-1227-D

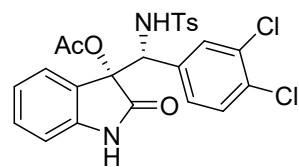


**3pa**

<sup>1</sup>H (600 MHz, CDCl<sub>3</sub>)

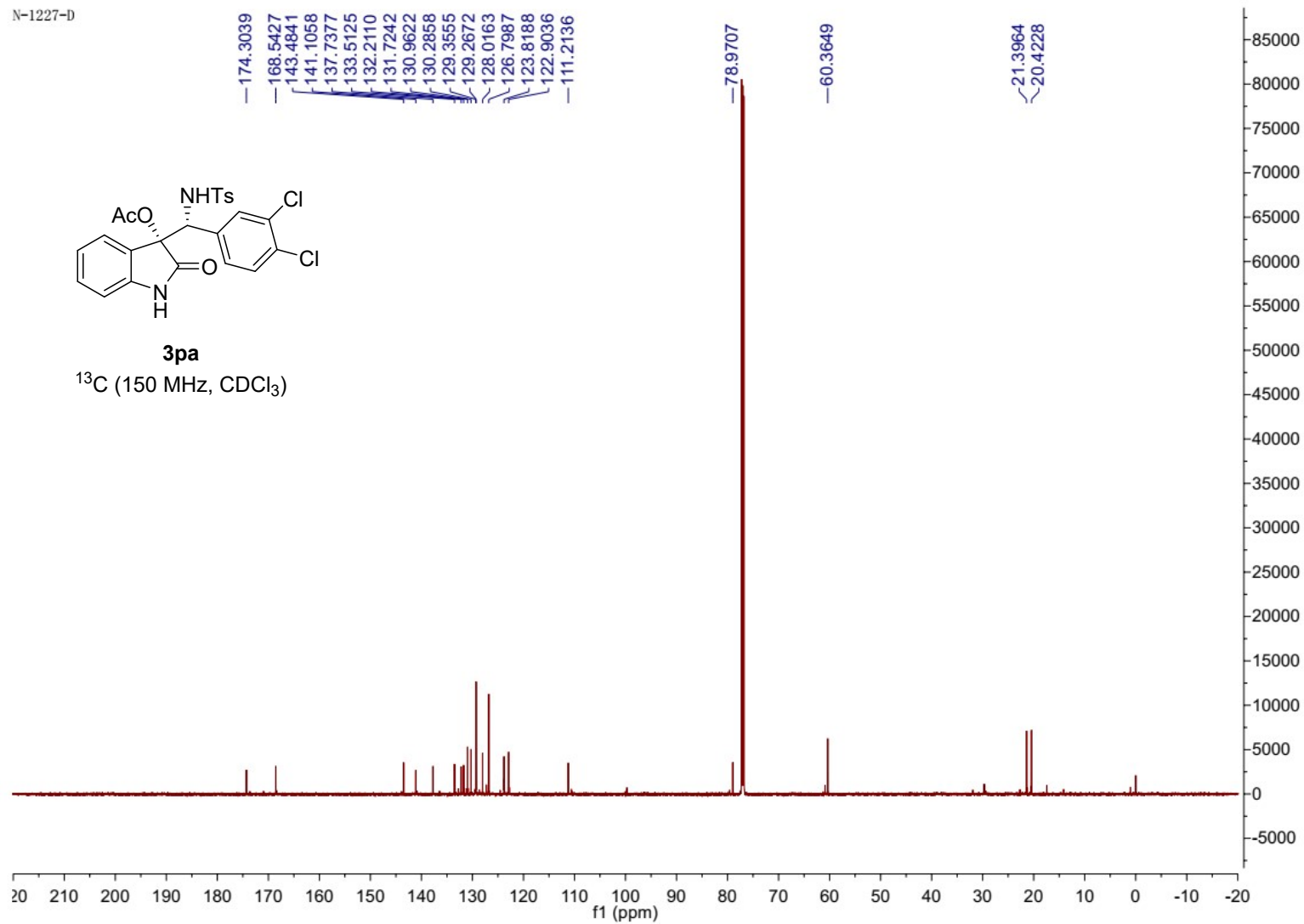


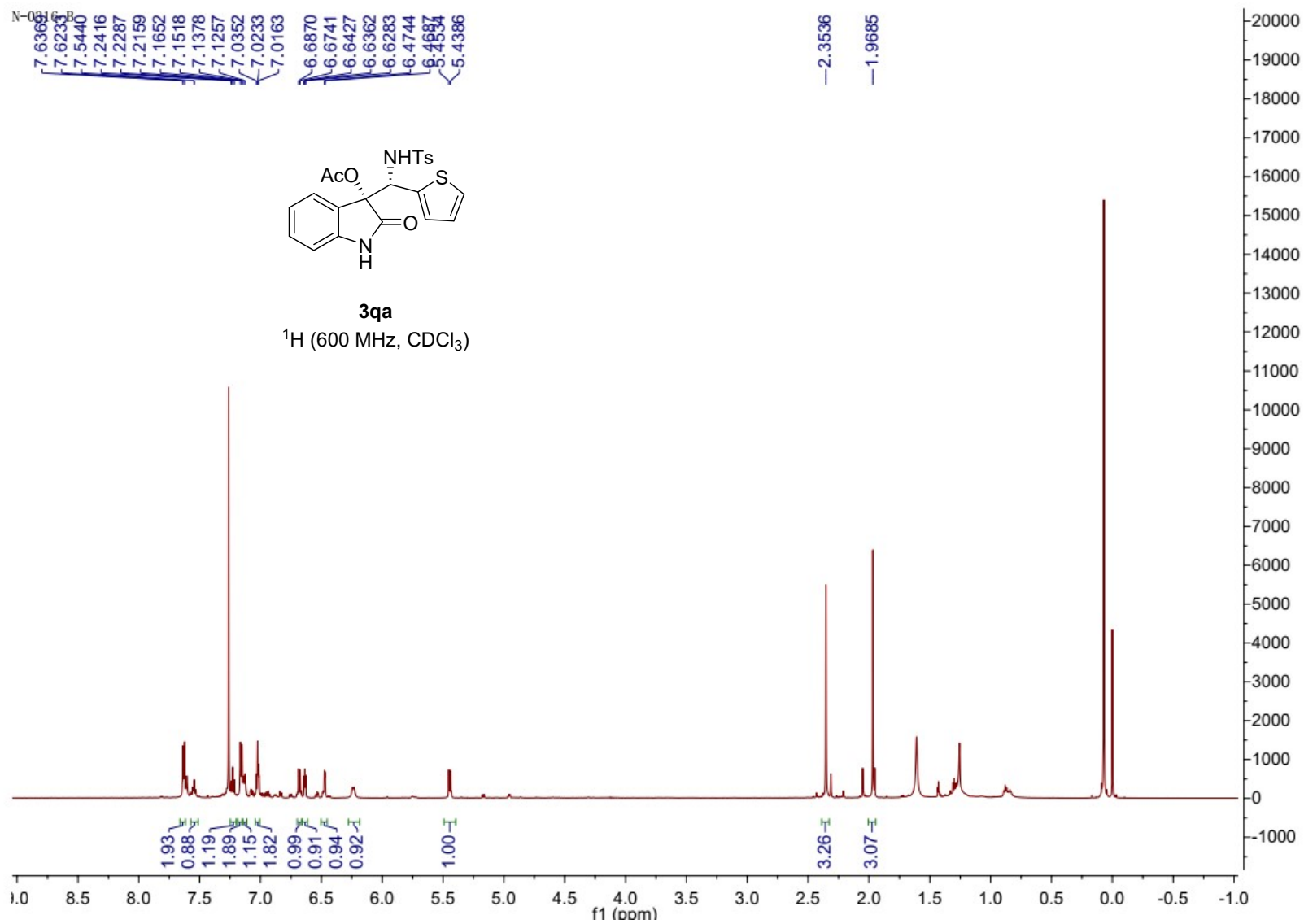
N-1227-D



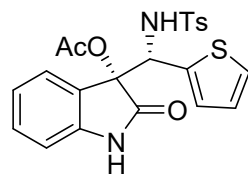
**3pa**

<sup>13</sup>C (150 MHz, CDCl<sub>3</sub>)



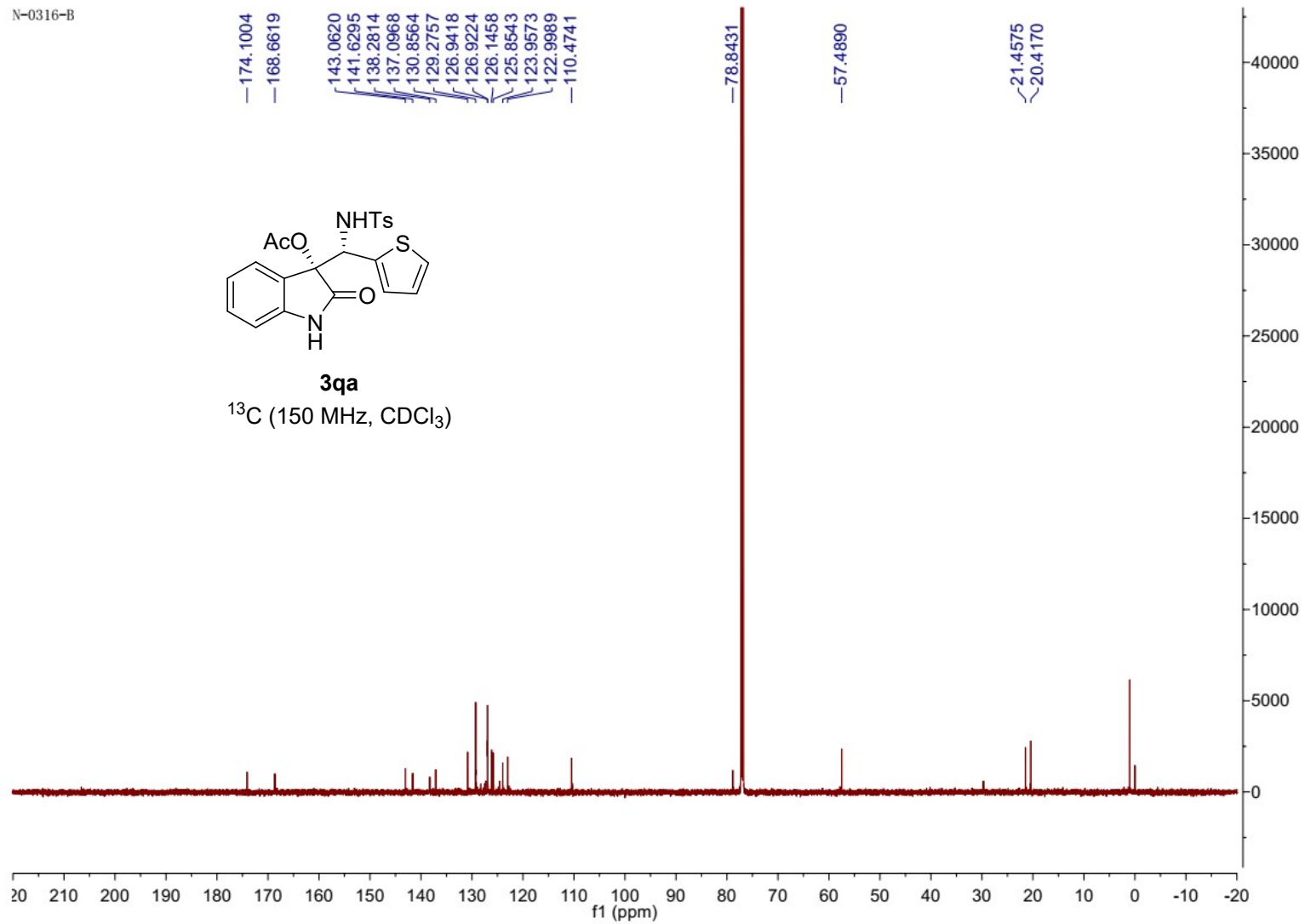


N-0316-B

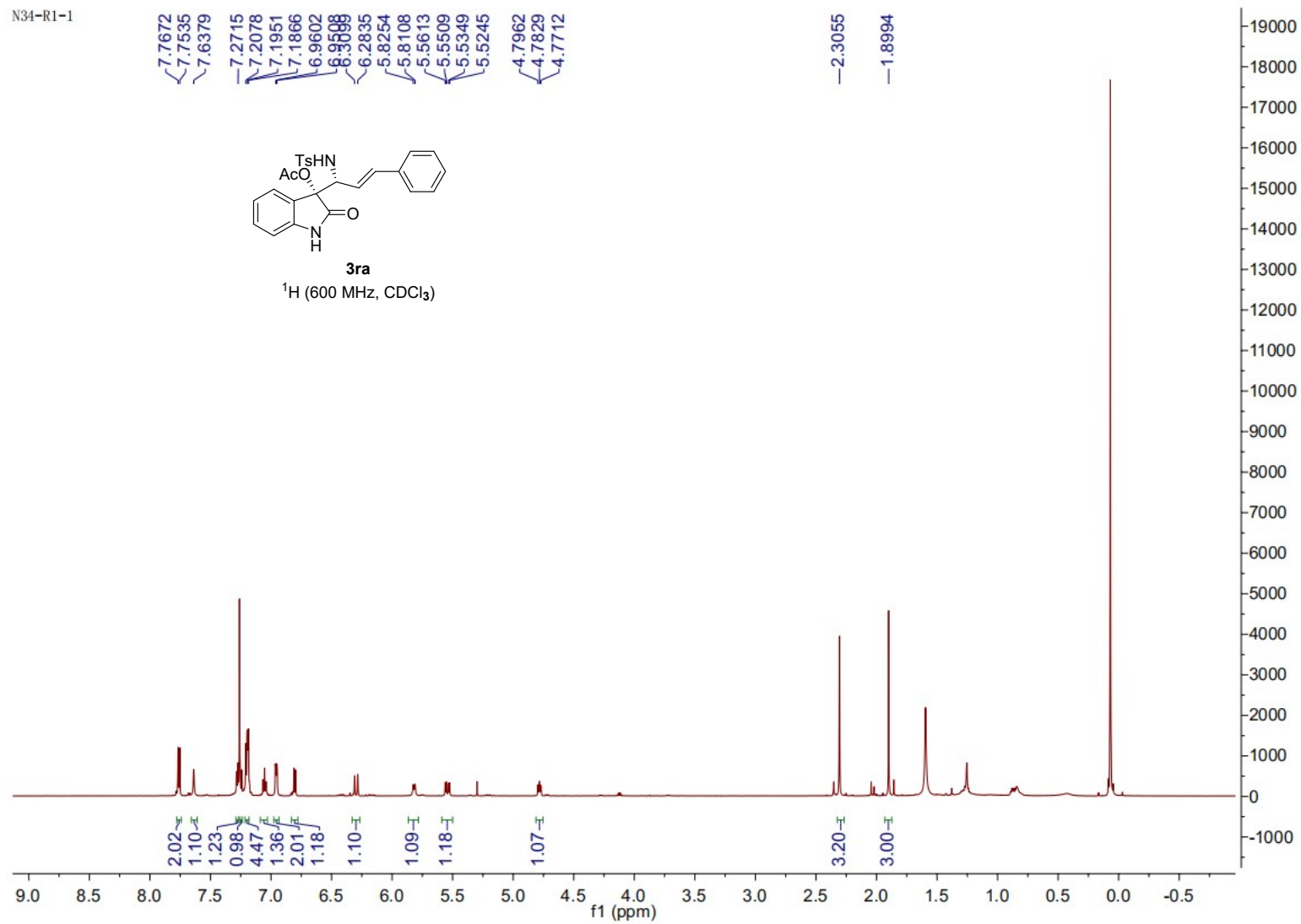


**3qa**

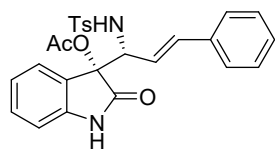
$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )



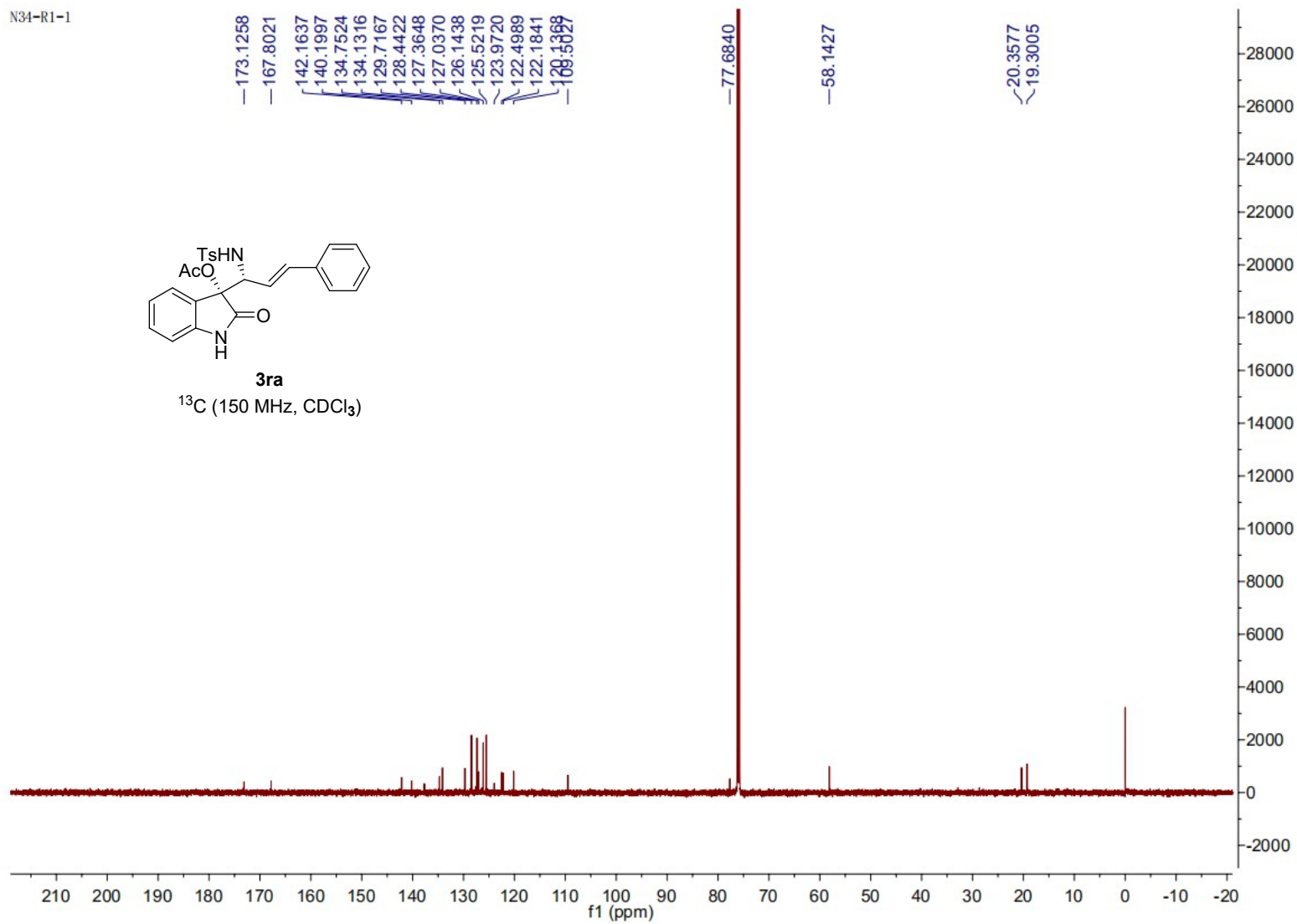
N34-R1-1



N34-R1-1



<sup>13</sup>C (150 MHz, CDCl<sub>3</sub>)



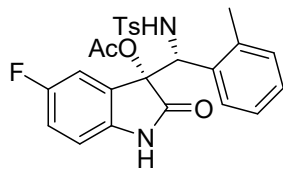
N-1223-A

8.9475

7.3224  
7.3087

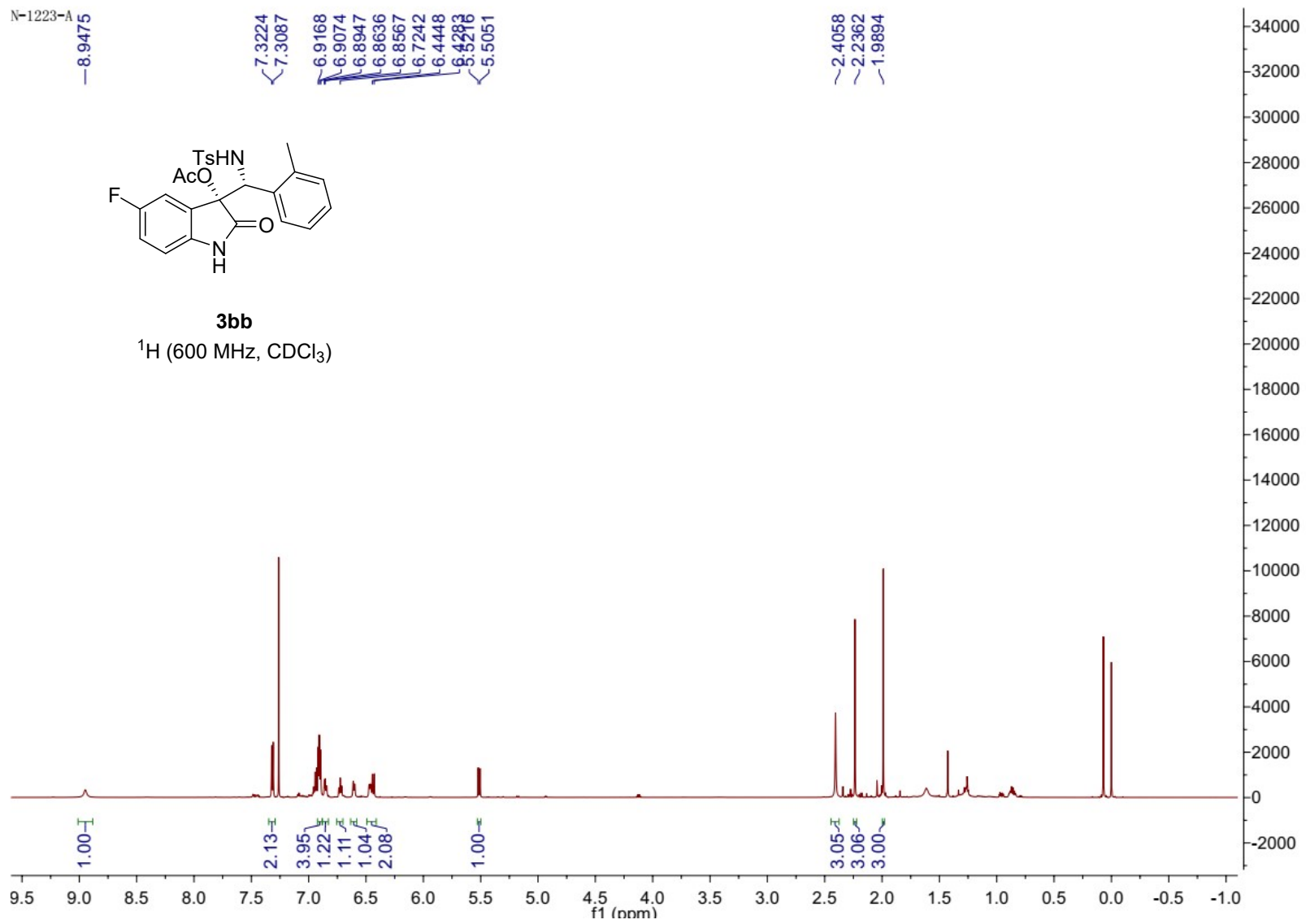
6.9168  
6.9074  
6.8947  
6.8636  
6.8567  
6.7242  
6.4448  
6.4283  
5.5051

2.4058  
2.2362  
1.9894



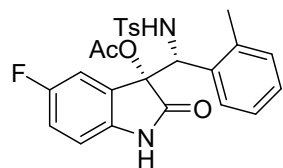
**3bb**

<sup>1</sup>H (600 MHz, CDCl<sub>3</sub>)



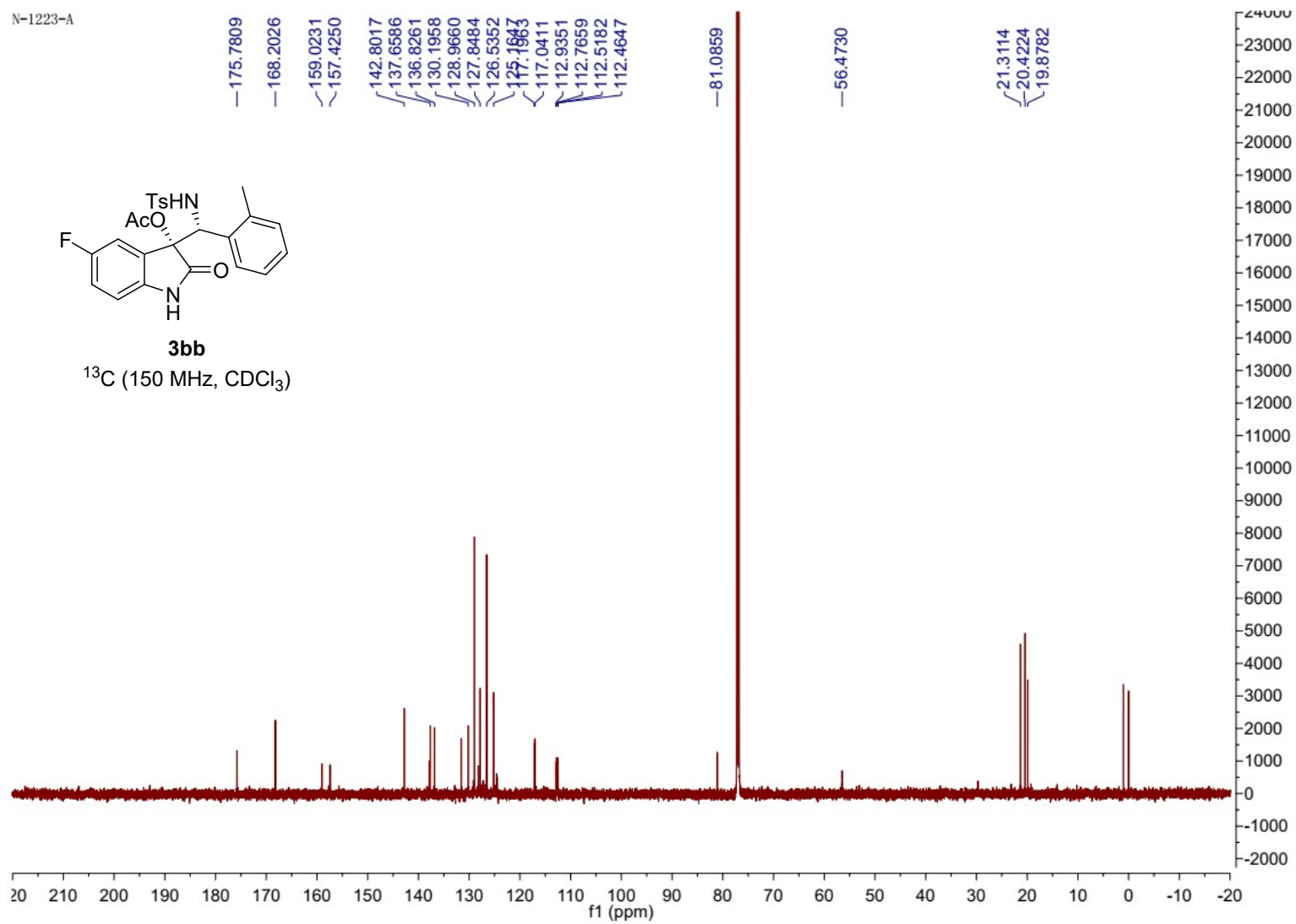


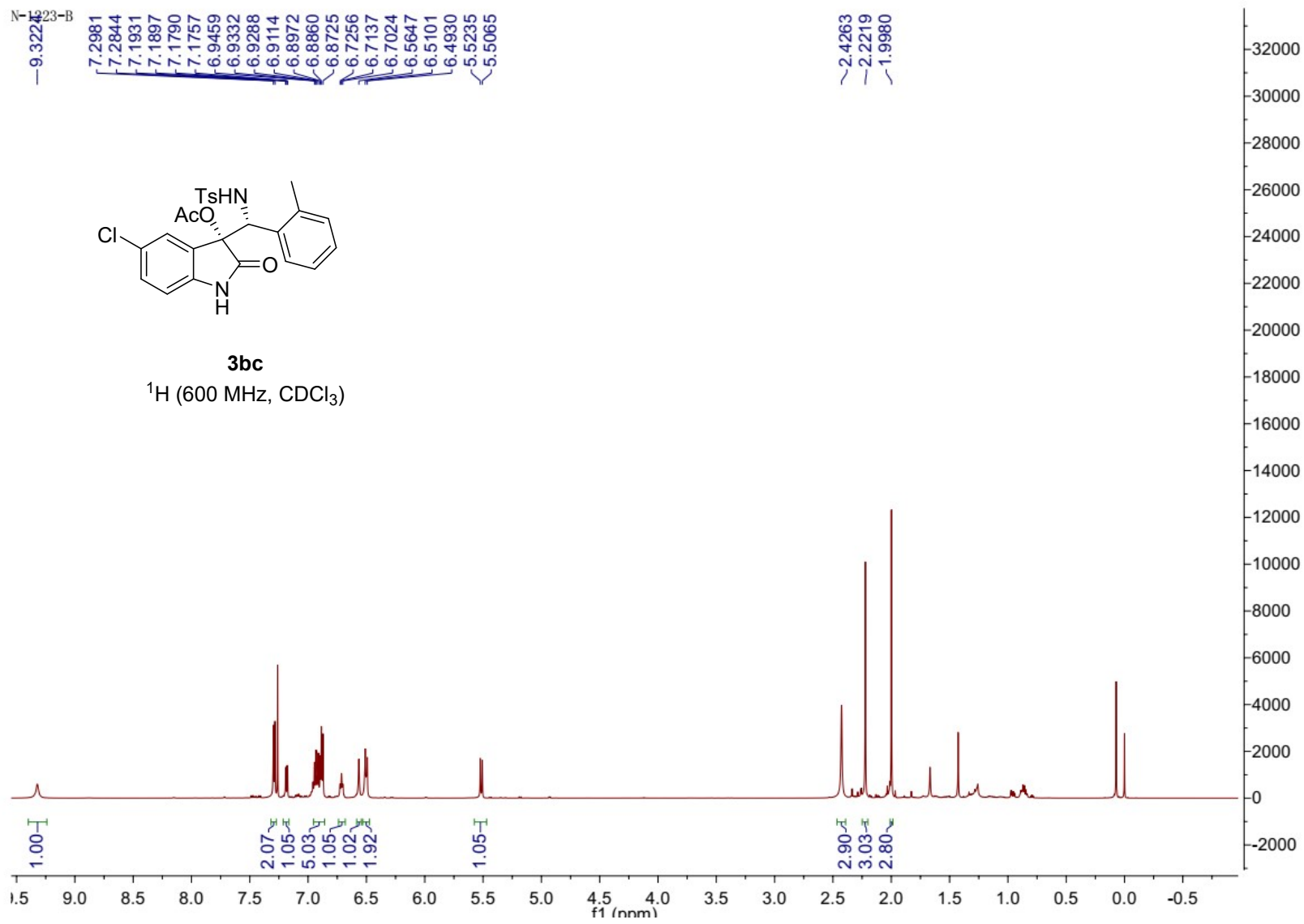
N-1223-A



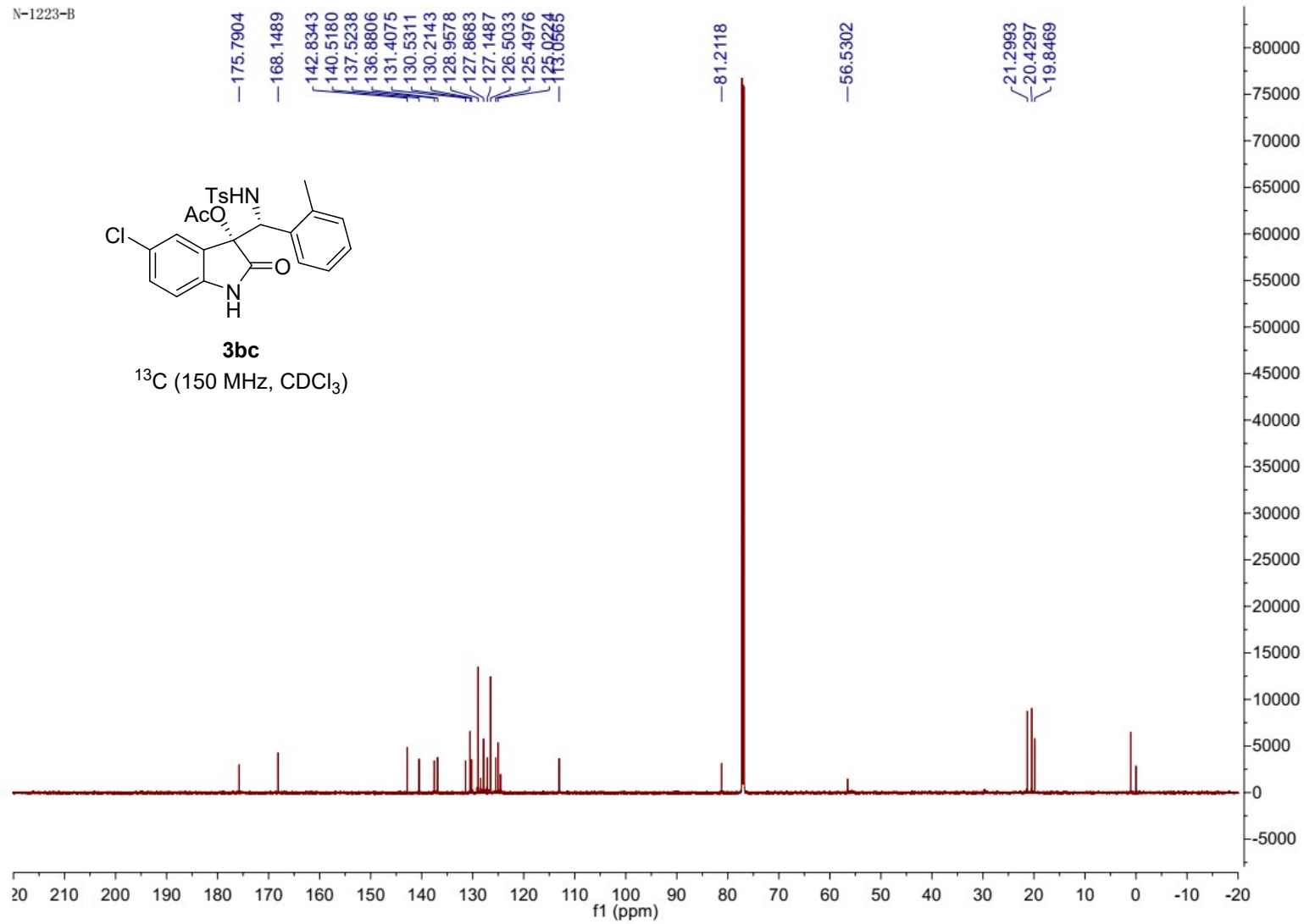
**3bb**

$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )





N-1223-B



N-1223-C

8.4601

7.3698

7.3562

6.9418

6.9285

6.8863

6.7556

6.7499

6.7175

6.4661

6.3811

6.3655

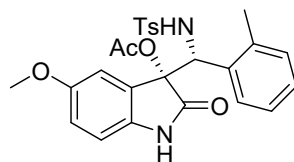
5.5014

3.6438

2.3681

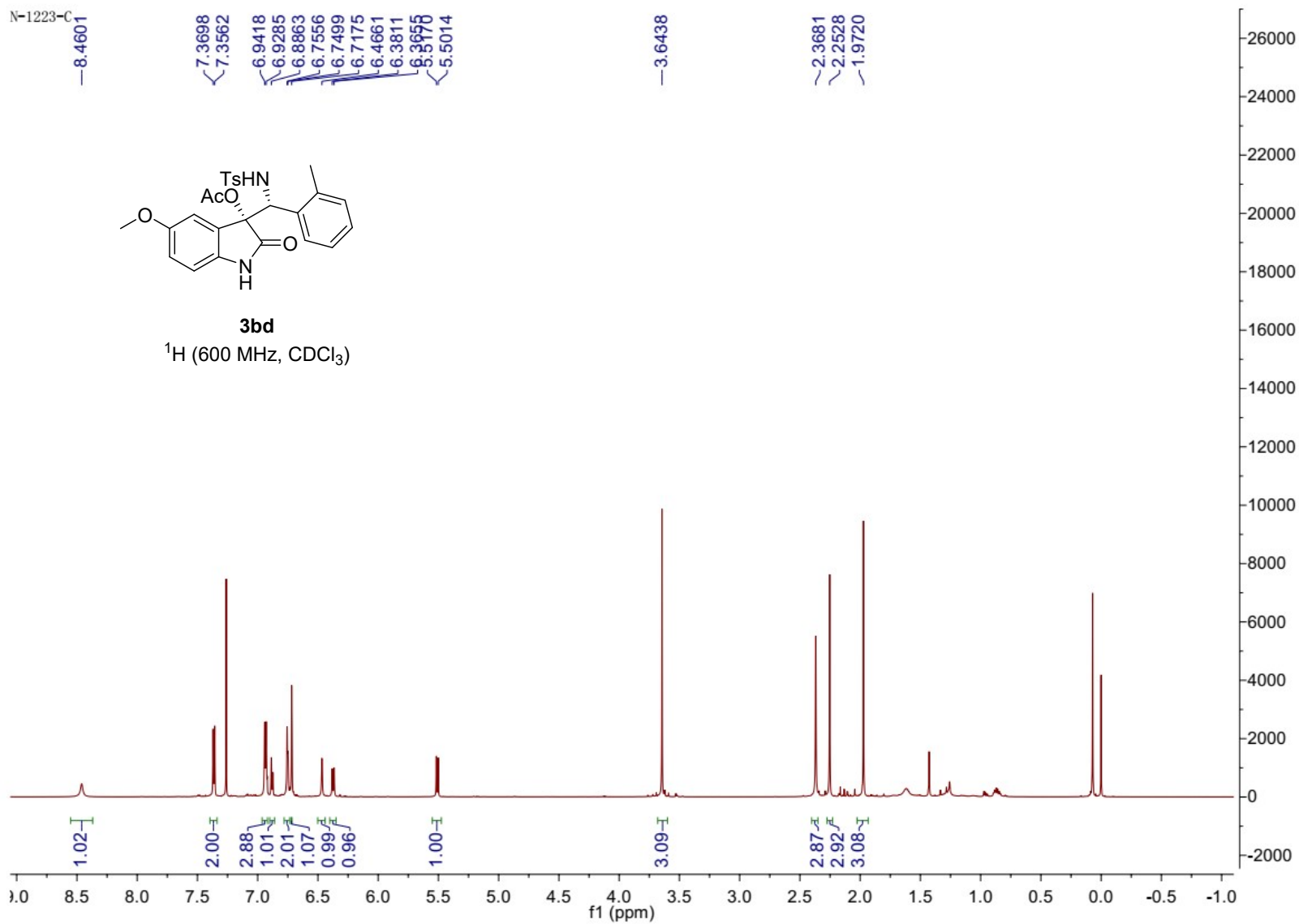
2.2528

1.9720

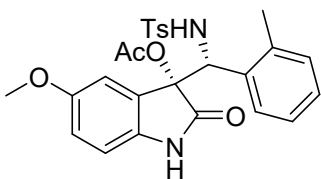


**3bd**

$^1\text{H}$  (600 MHz,  $\text{CDCl}_3$ )

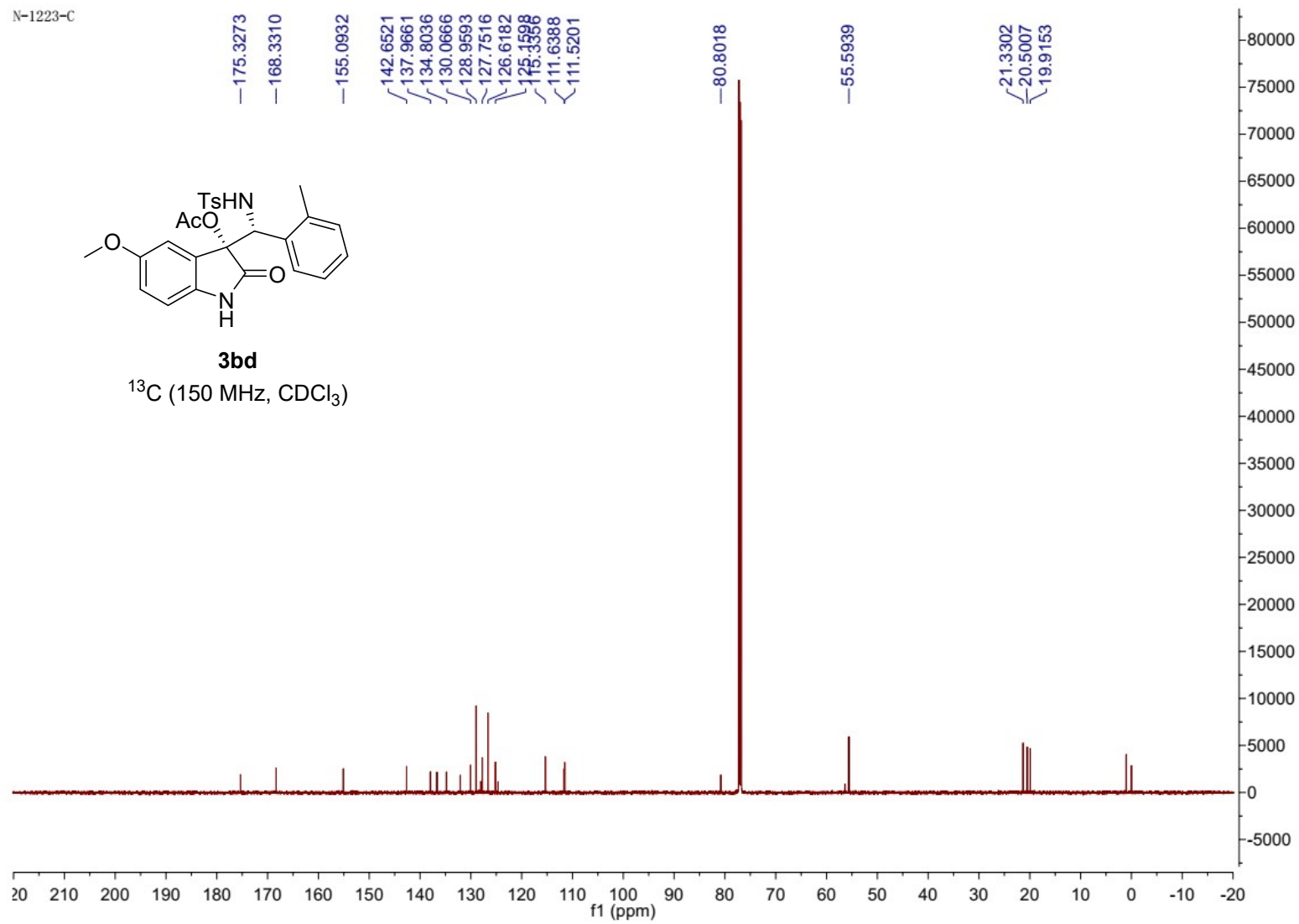


N-1223-C



**3bd**

<sup>13</sup>C (150 MHz, CDCl<sub>3</sub>)

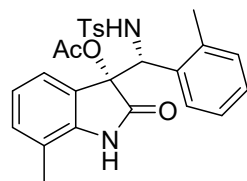


N-0322A

— 9.0224

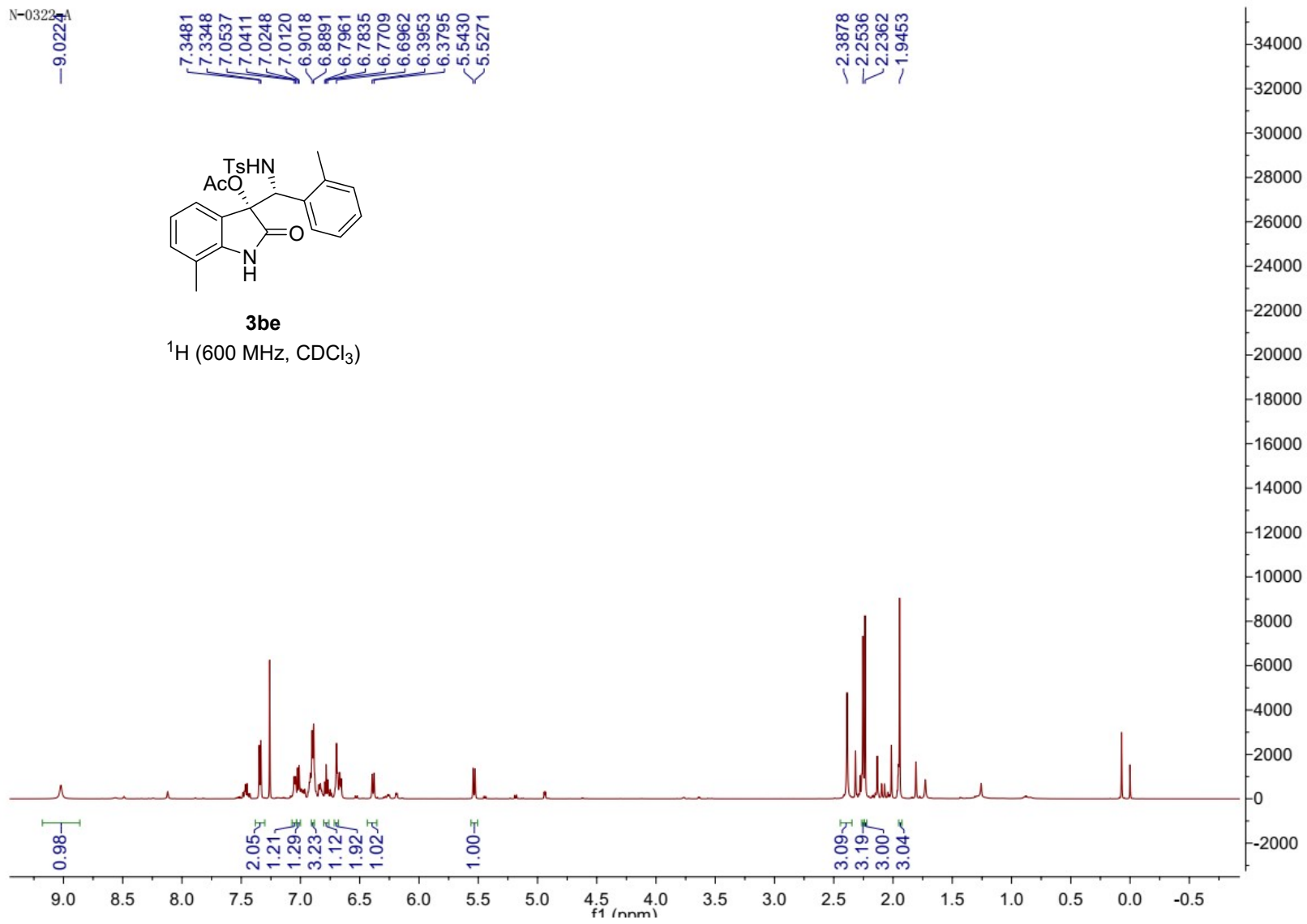
7.3481  
7.3348  
7.0537  
7.0411  
7.0248  
7.0120  
6.9018  
6.8891  
6.7961  
6.7835  
6.7709  
6.6962  
6.3953  
6.3795  
5.5430  
5.5271

2.3878  
2.2536  
2.2362  
1.9453

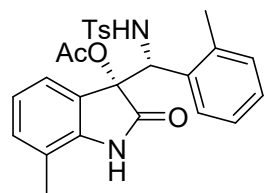


**3be**

<sup>1</sup>H (600 MHz, CDCl<sub>3</sub>)

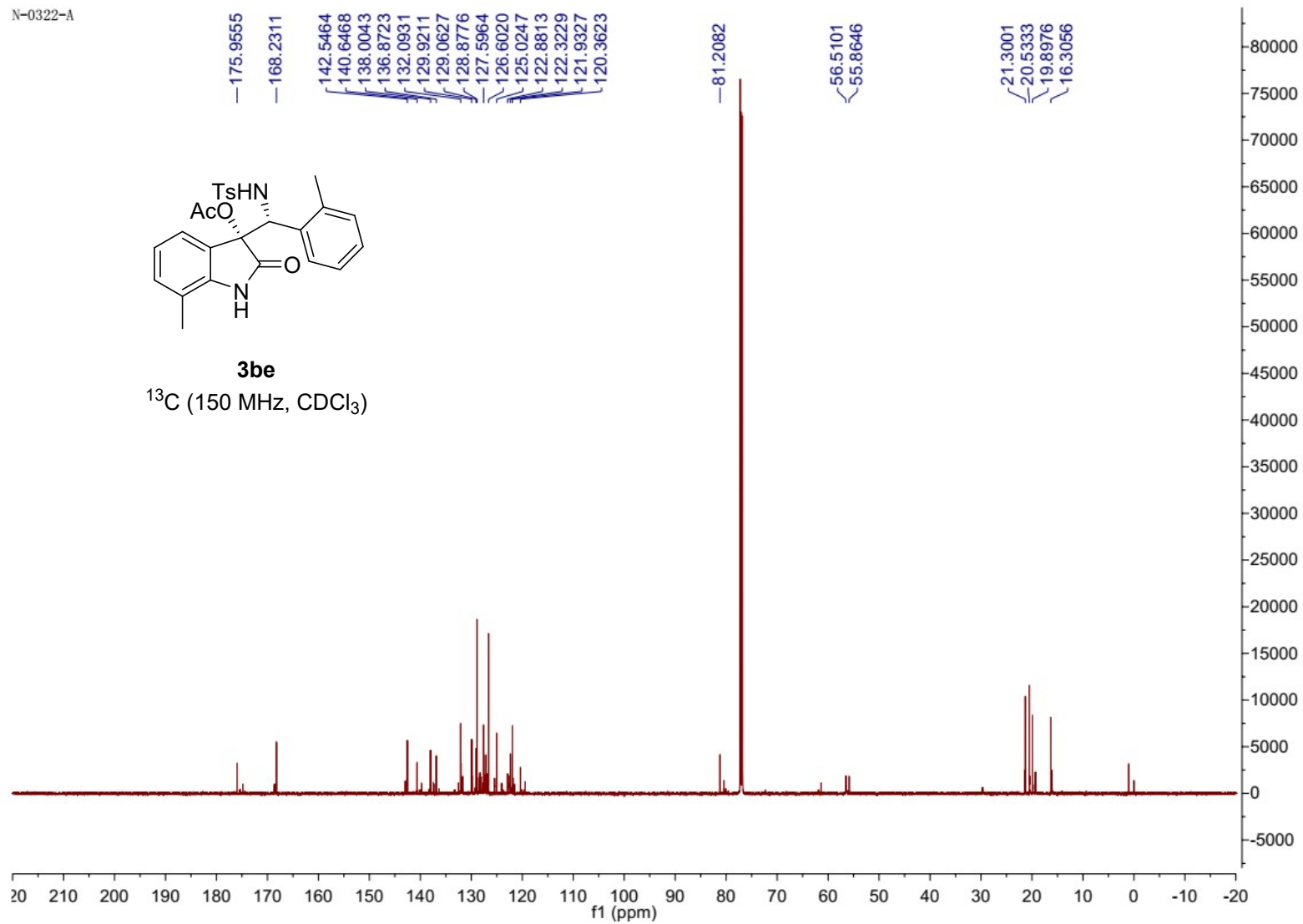


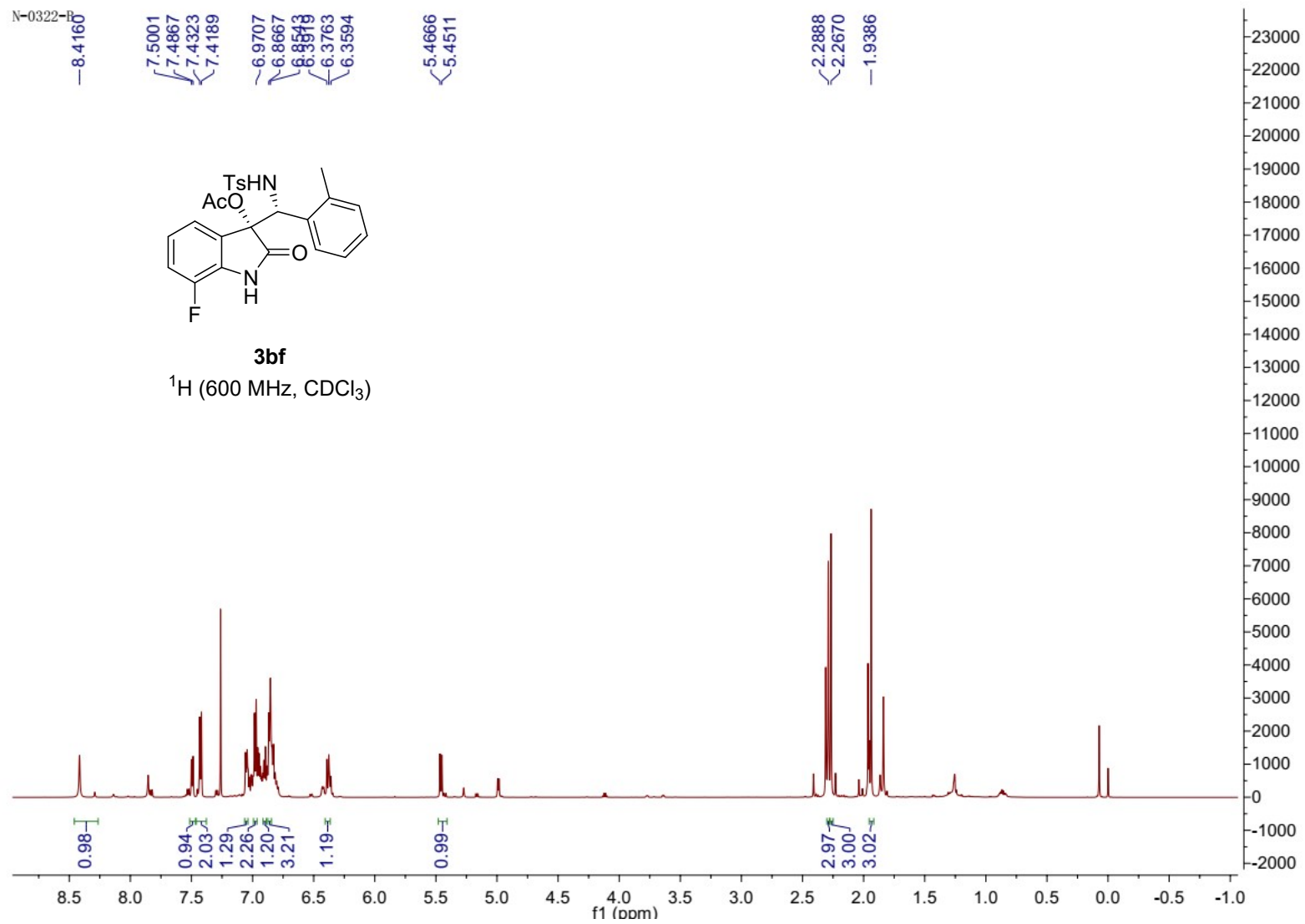
N-0322-A



**3be**

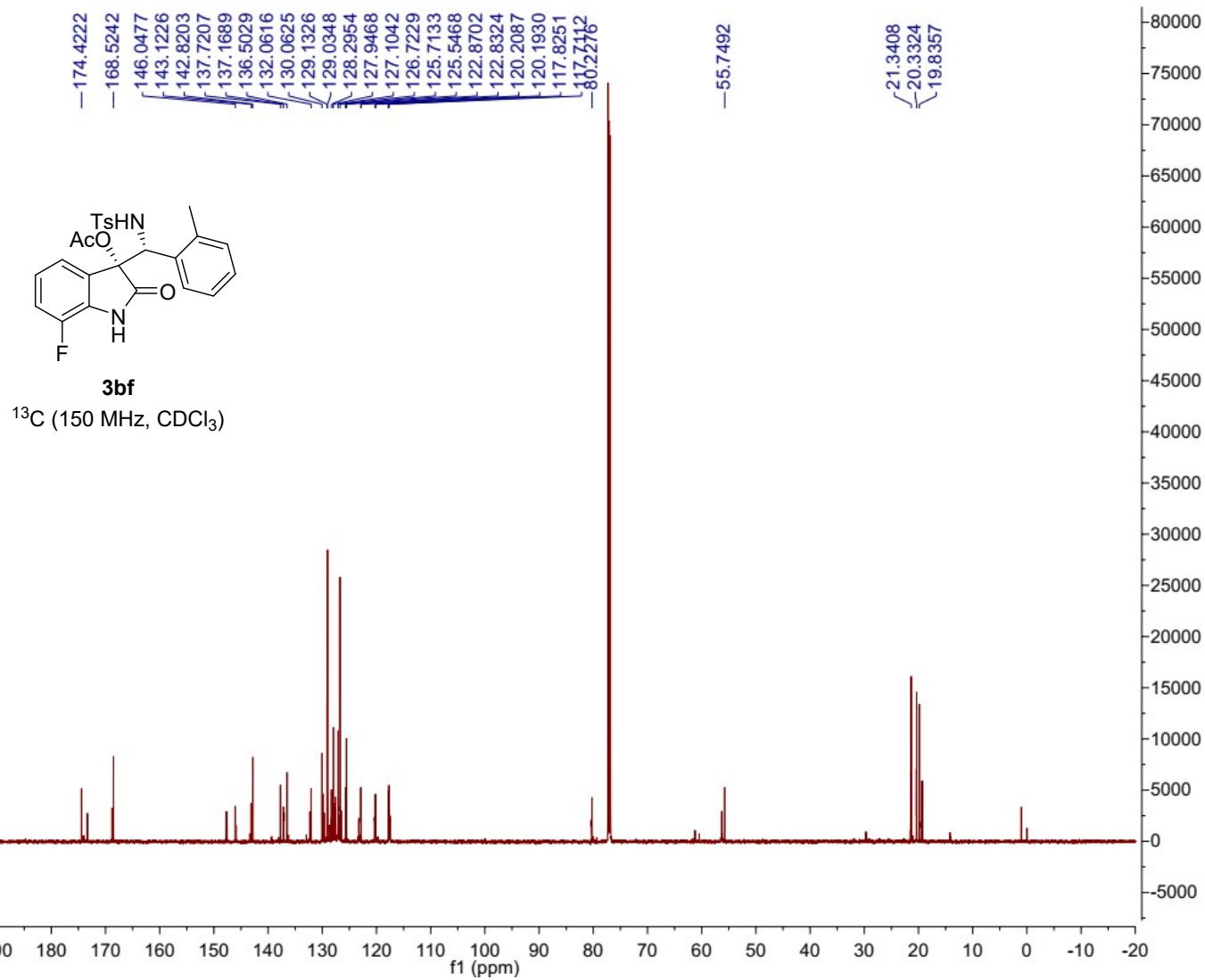
$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )



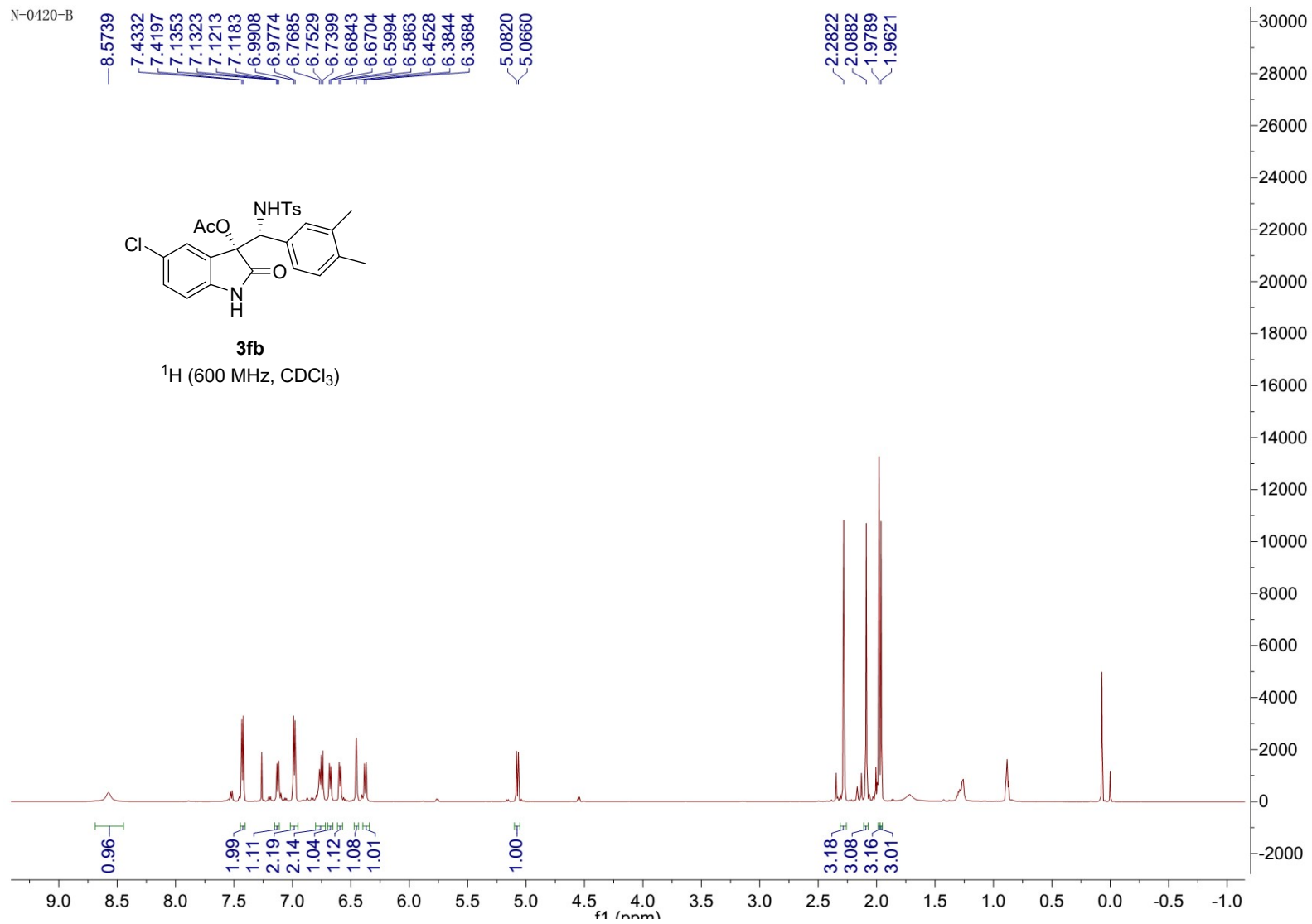




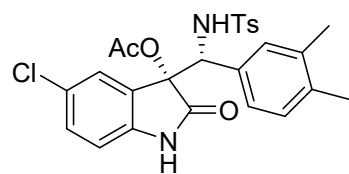
N-0322-B



N-0420-B

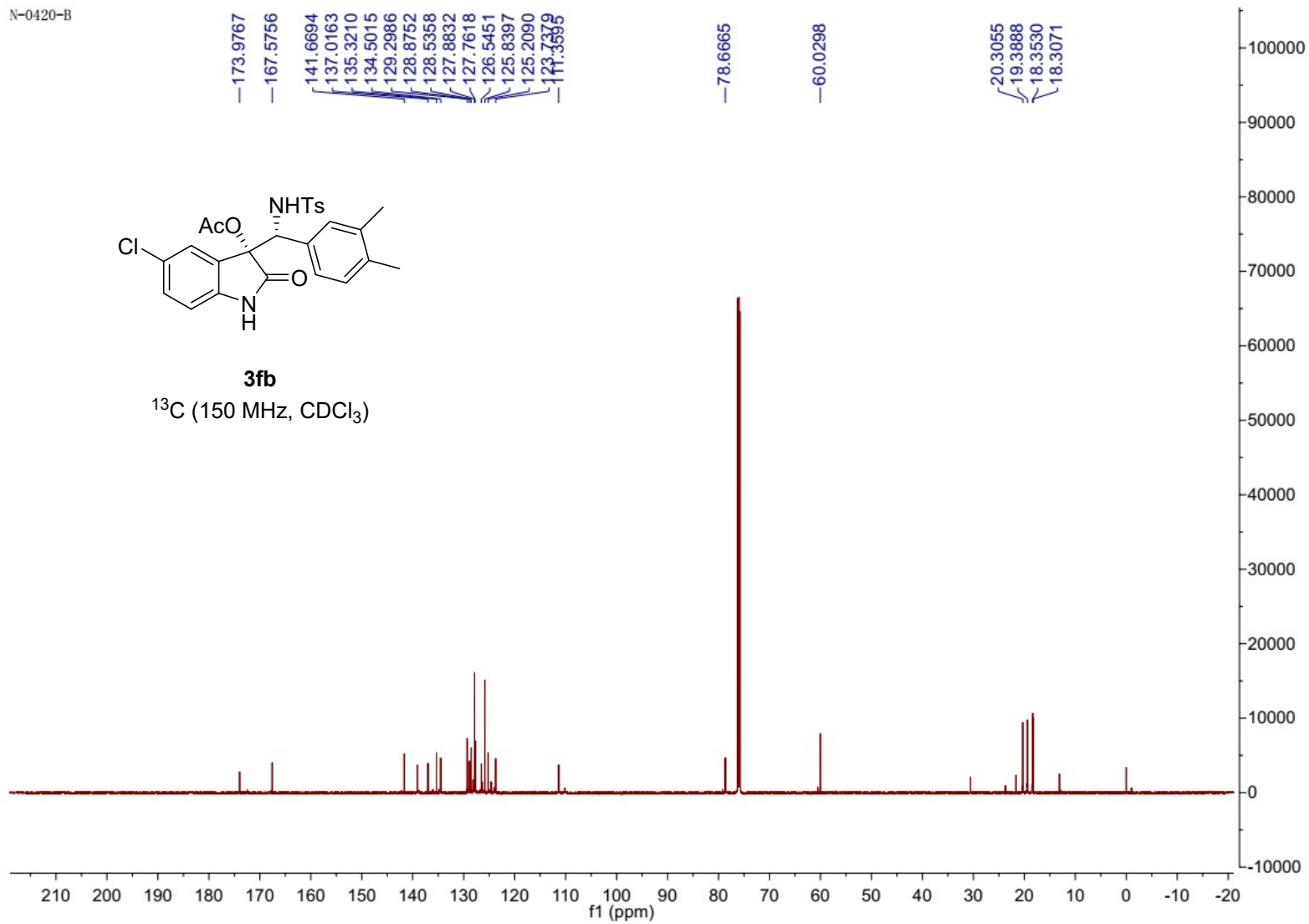


N-0420-B

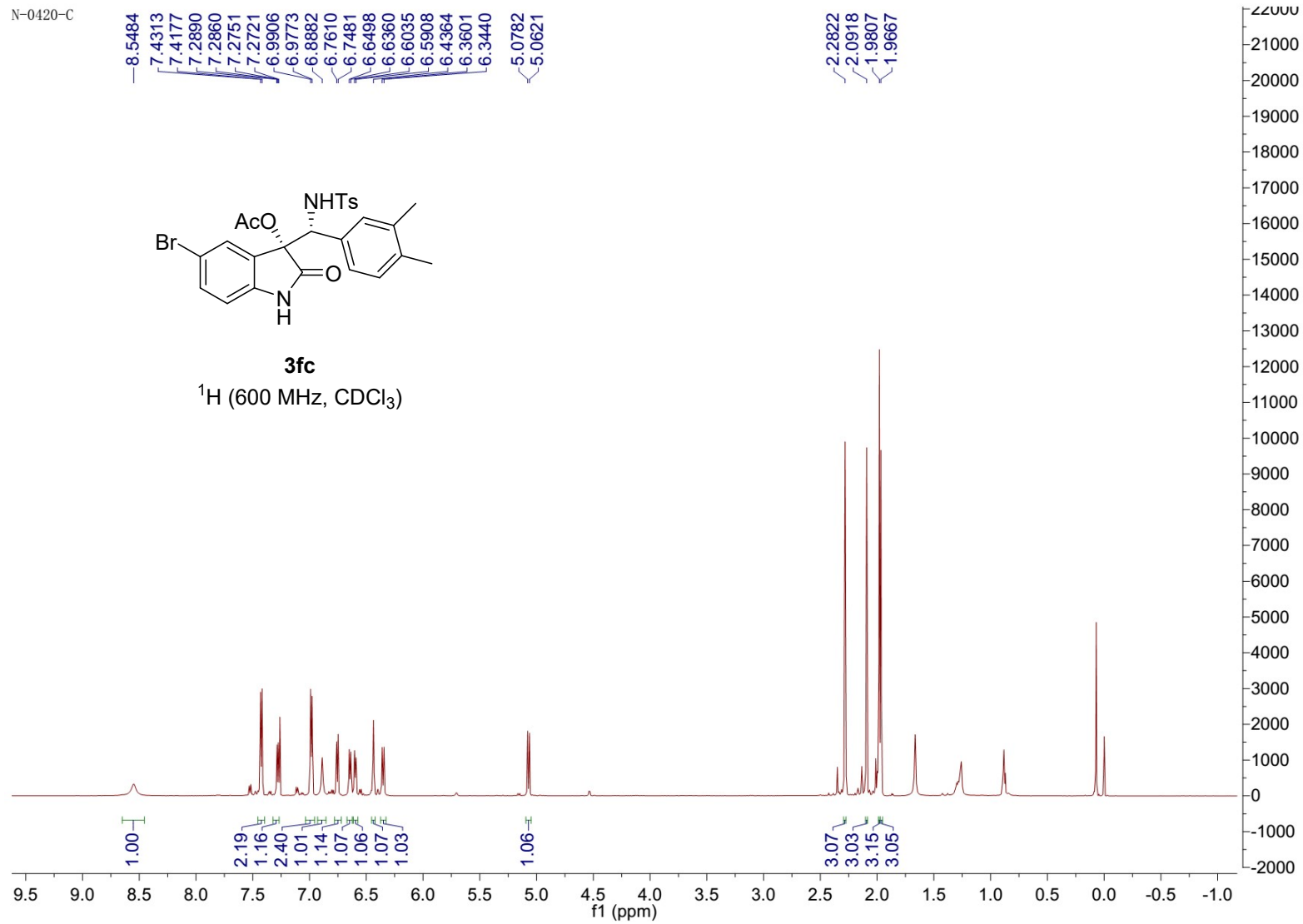


**3b**

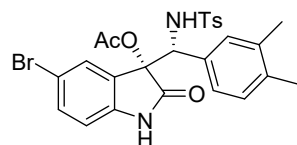
<sup>13</sup>C (150 MHz, CDCl<sub>3</sub>)



N-0420-C

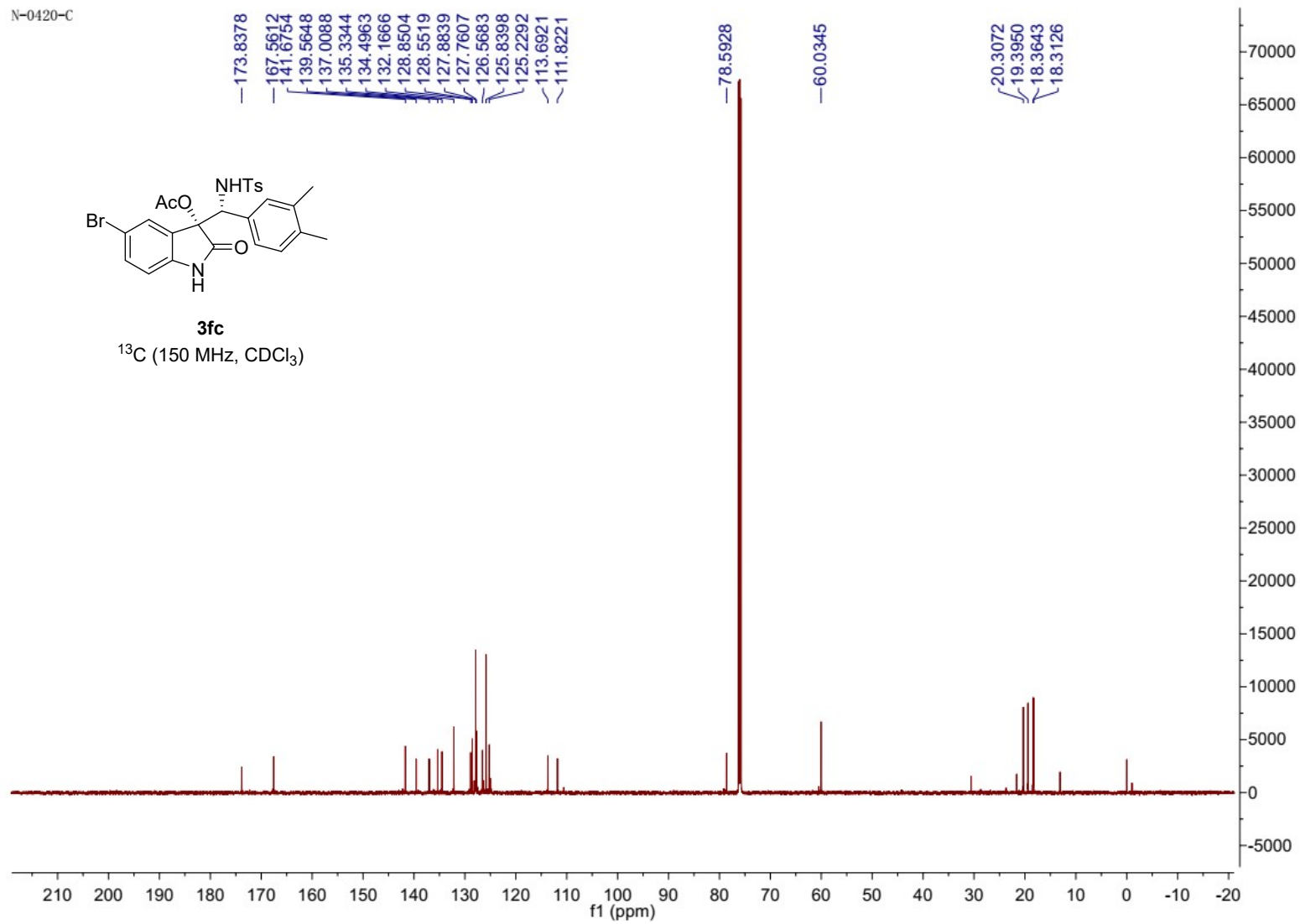


N-0420-C

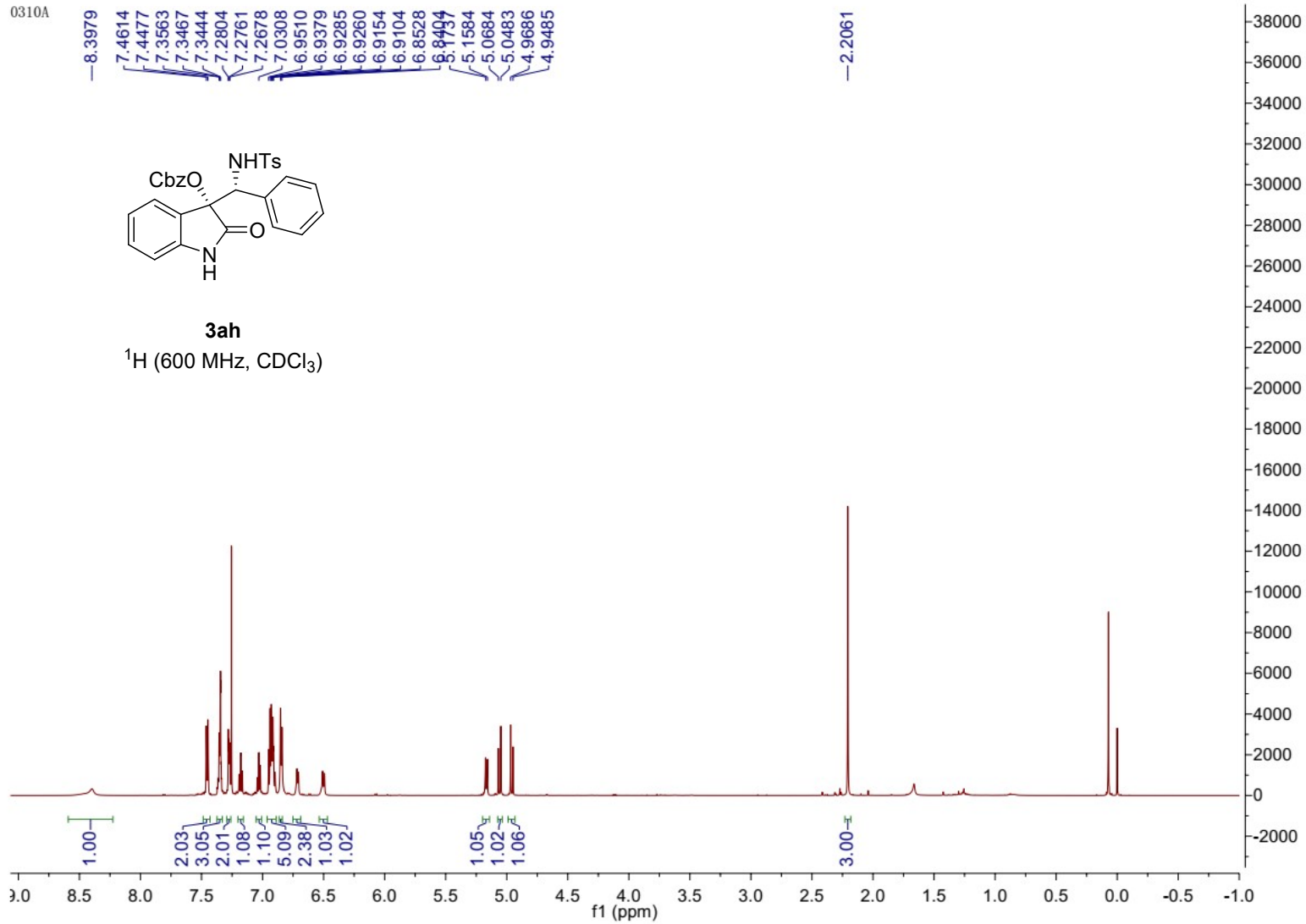


**3fc**

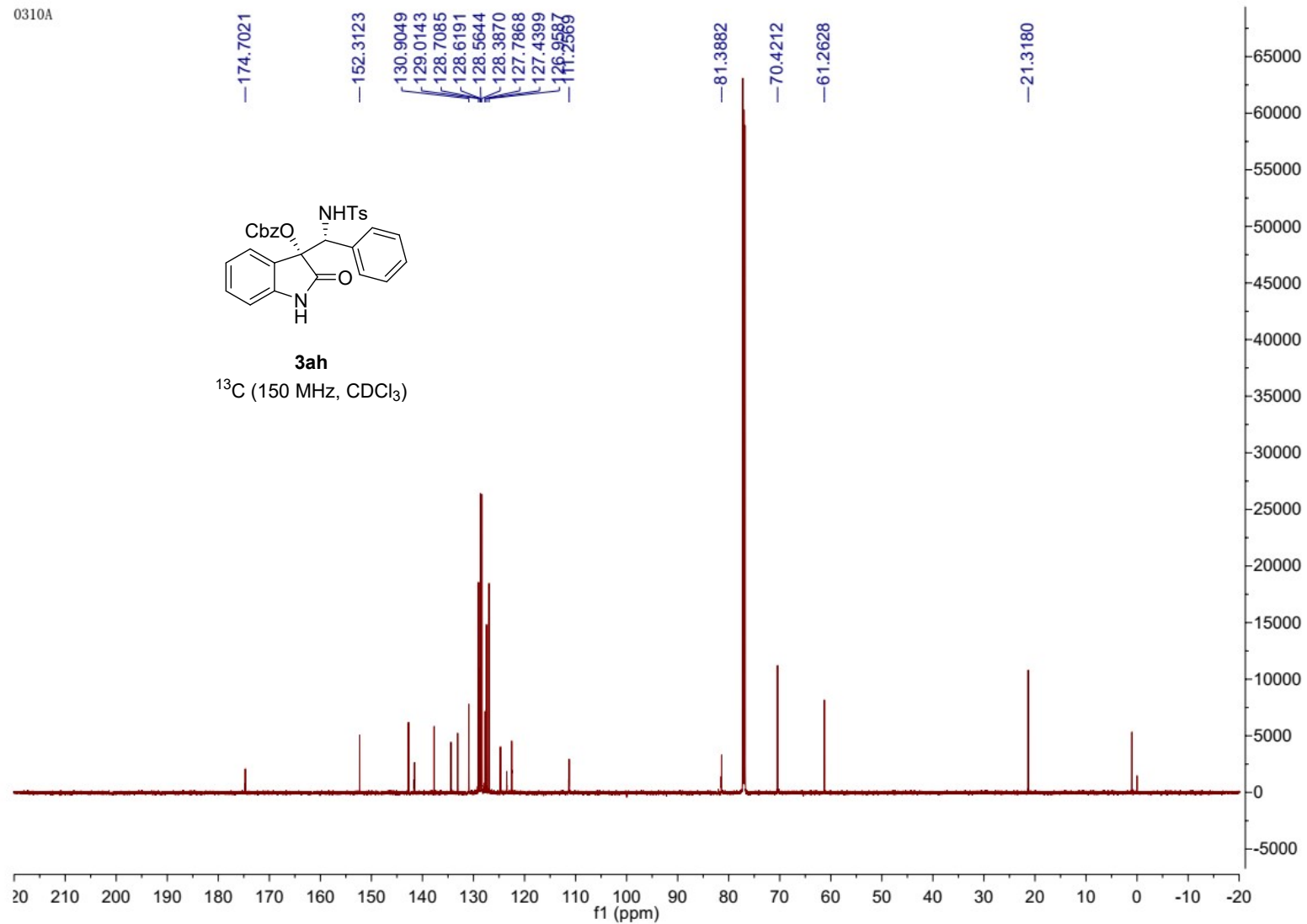
$^{13}\text{C}$  (150 MHz,  $\text{CDCl}_3$ )



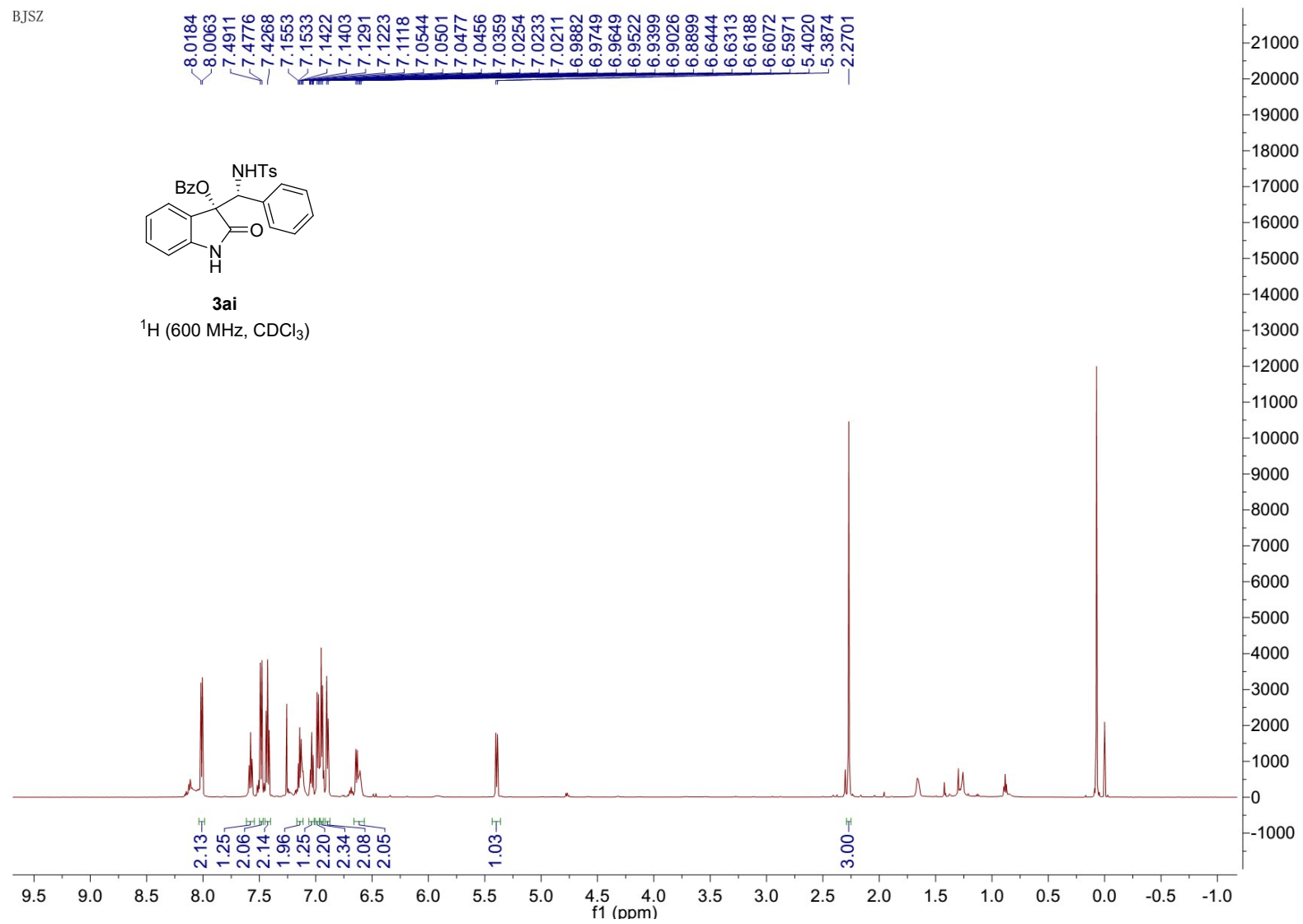
0310A



0310A

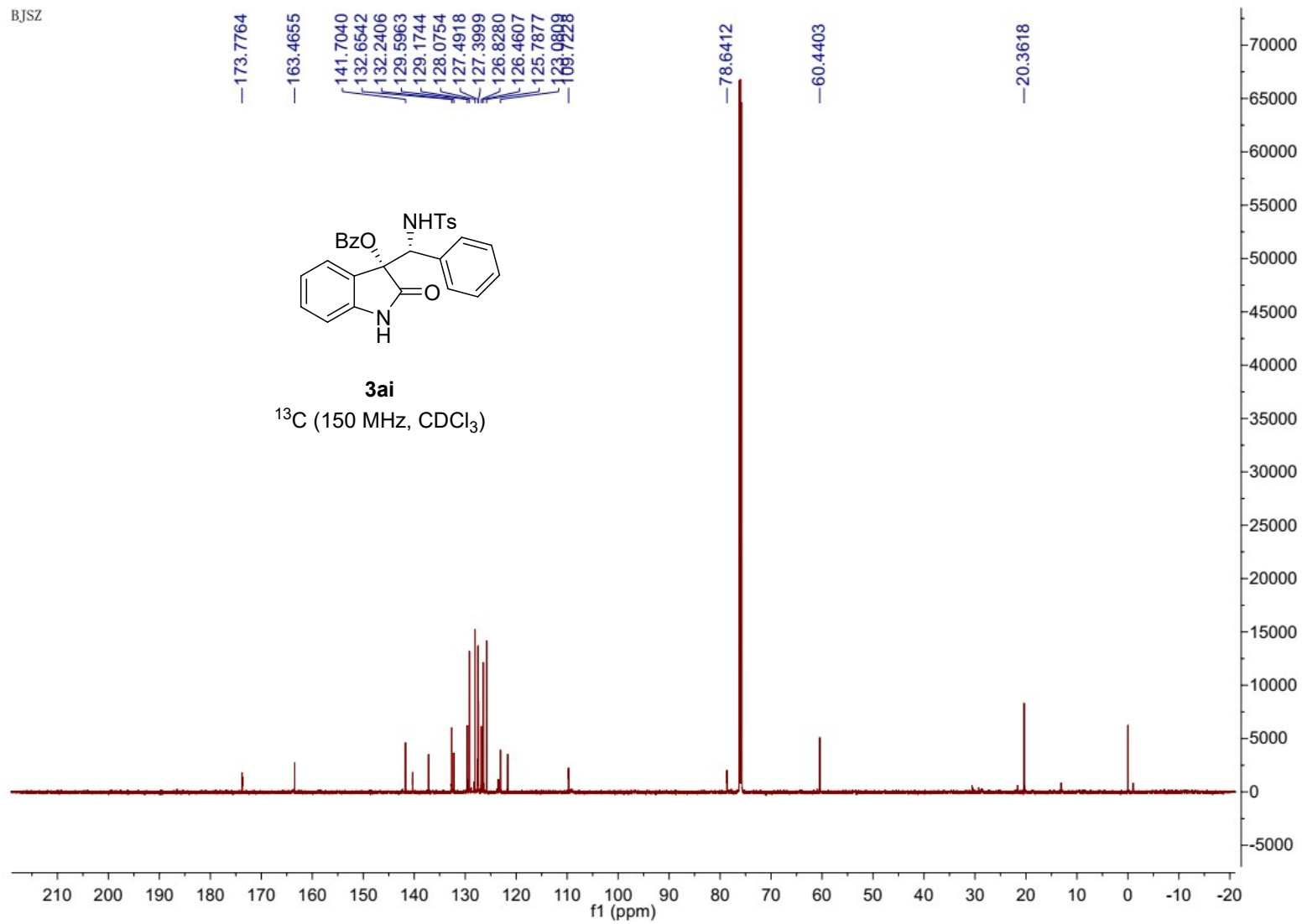


BJSZ





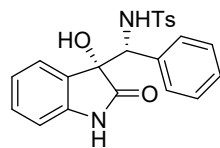
BJSZ



MOVEAC

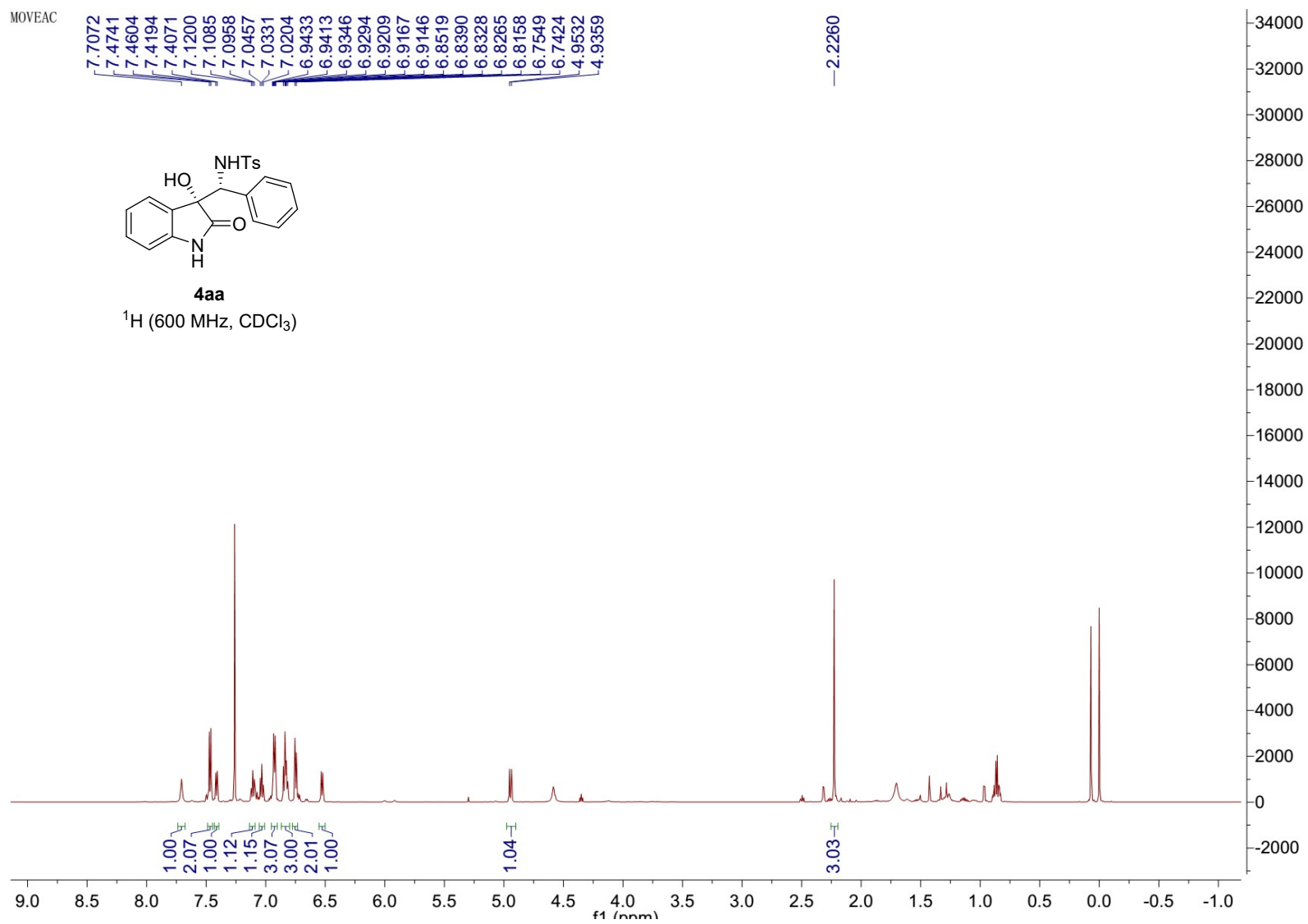
7.7072  
7.4741  
7.4604  
7.4194  
7.4071  
7.1200  
7.1085  
7.0958  
7.0457  
7.0331  
7.0204  
6.9433  
6.9413  
6.9346  
6.9294  
6.9209  
6.9167  
6.9146  
6.8519  
6.8390  
6.8328  
6.8265  
6.8158  
6.7549  
6.7424  
4.9532  
4.9359

—2.2260

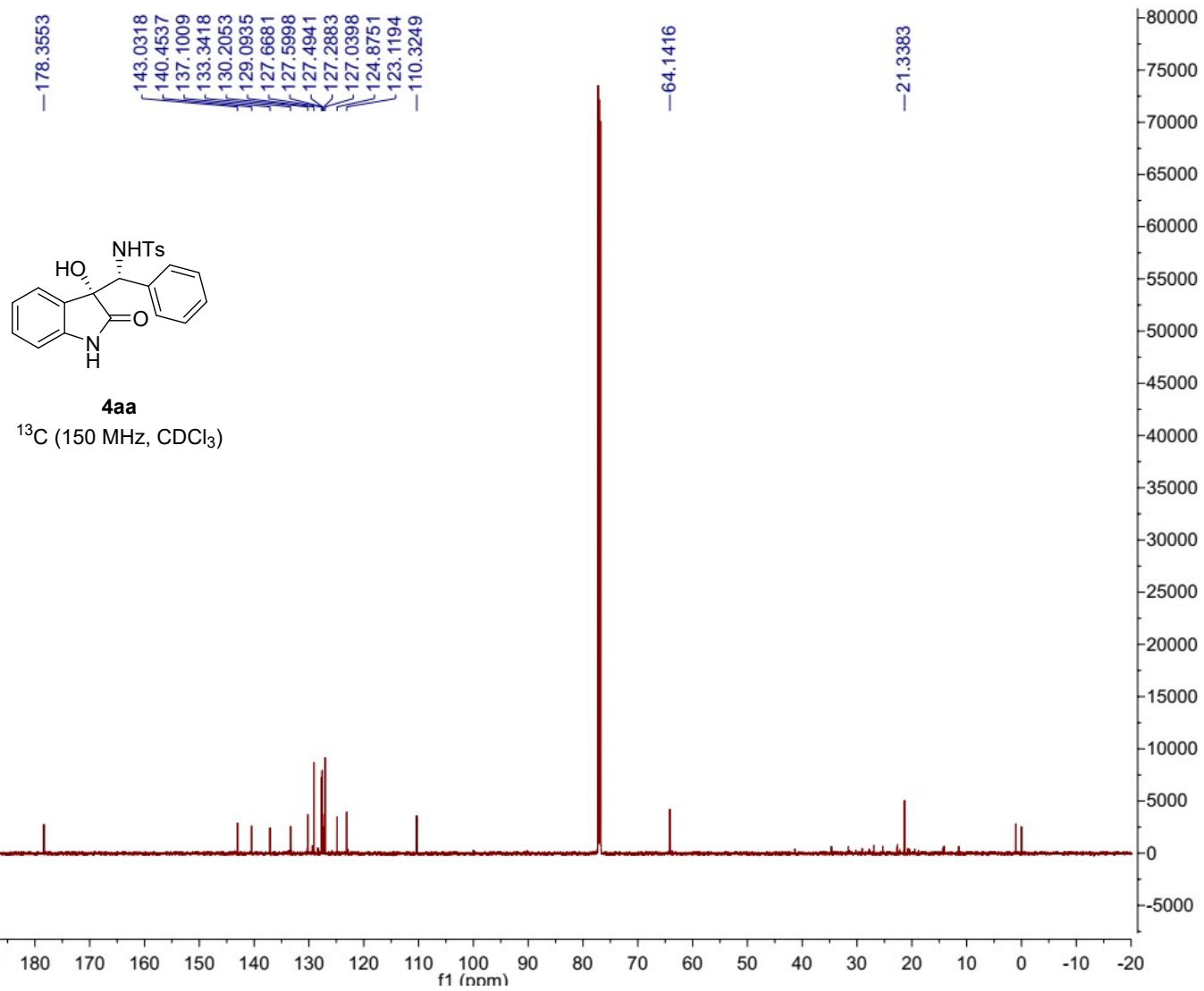


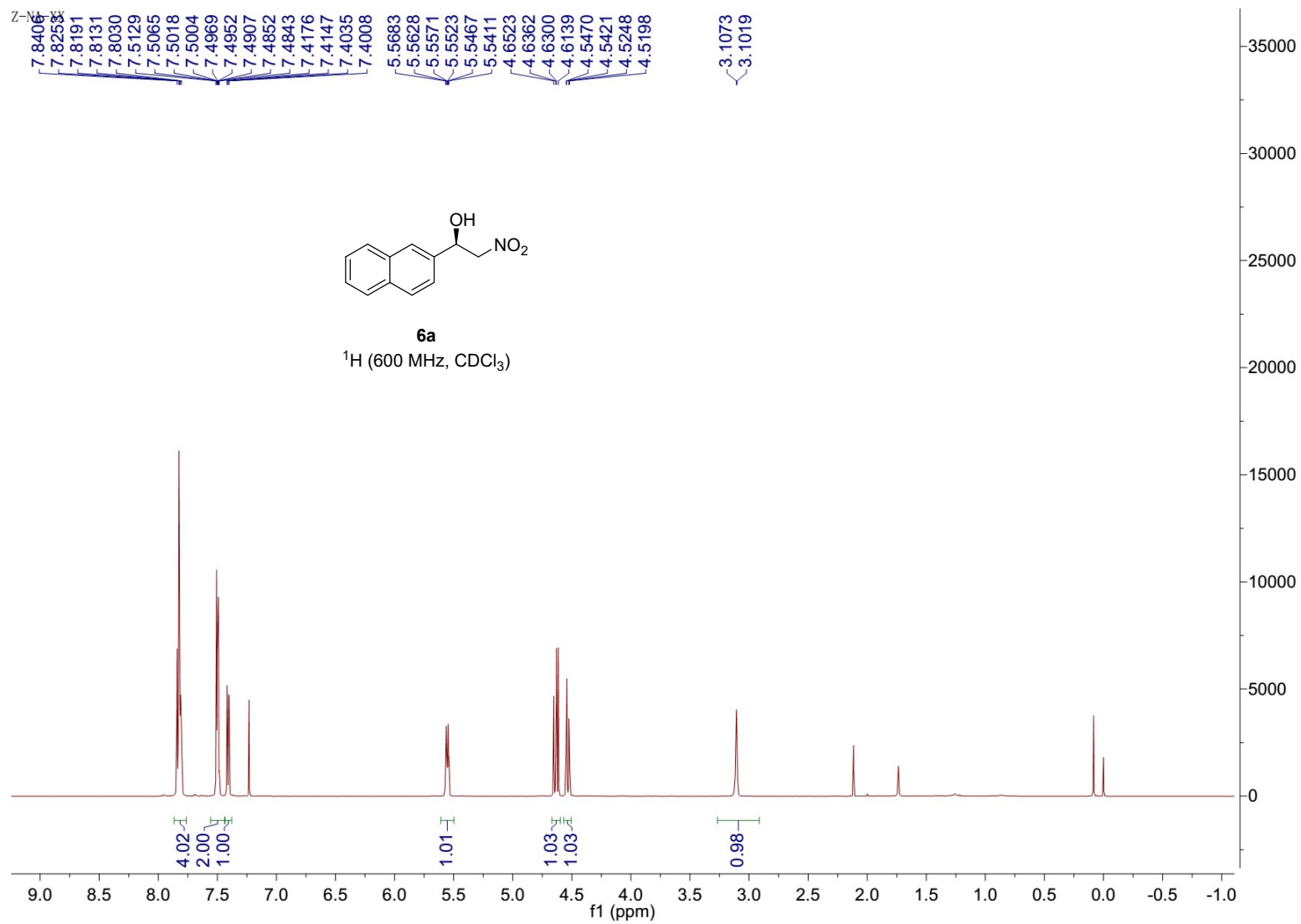
**4aa**

<sup>1</sup>H (600 MHz, CDCl<sub>3</sub>)



MOVEAC





N-4ab

