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Supporting Information

Diastereo- and Enantioselective Mannich/Cyclization Cascade Reaction of Isocyanoacetates with Cyclic sulfamide imines by Cinchona Alkaloid Squaramide/AgOAc Cooperative Catalysis

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1. X-Ray crystal data of compound 4d

Table 1. Crystal data and structure refinement for **4d** (CCDC 1587296).

Empirical formula	$C_{19}H_{16}BrN_3O_5S$	
Formula weight	478.32	
Temperature	296(2)	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.3579(3)Å	α= 85.4310(10)°.
	b = 8.8158(4)Å	β= 88.5430(10)°.
	c = 15.8511(7) Å	γ = 72.7390(10)°.
Volume	978.77(7) $Å^3$	
Z	2	
Density (calculated)	1.623 Mg/m^3	
Absorption coefficient	2.244 mm^{-1}	
F(000)	484	
Crystal size	0.46 x 0.35 x 0.28 mm^3	
Theta range for data collection	2..43 to 28.24°.	
Index ranges	-8<=h<=8, -10<=k<=10, -19<=l<=17	
Reflections collected	11881	
Independent reflections	3594 [R(int) = 0.0188]	
Data / restraints / parameters	3594 /0 / 262	
Goodness-of-fit on F^2	1.057	
Final R indices [I>2sigma(I)]	R1 = 0.0342, wR2 =0.0907	
R indices (all data)	R1 = 0.0397, wR2 = 0.0941	
Largest diff. peak and hole	0.764 and -0.768 $e.Å^{-3}$	

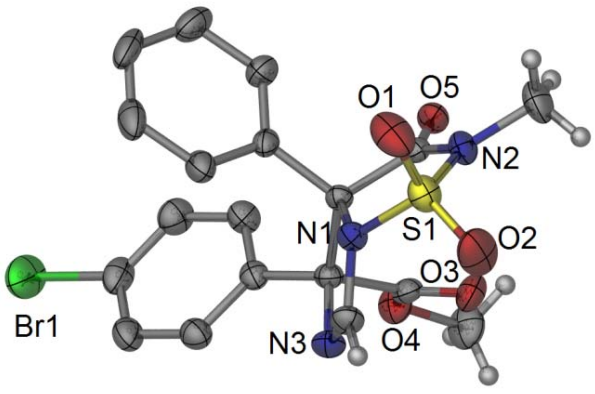
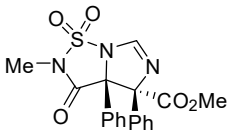


Figure S1. ORTEP plot of the X-ray crystal structure of **4d** (displacement ellipsoids are drawn at the 50% probability level)

2. Copies of HPLC analysis spectra of compounds 4

4a (Table 3, entry 1)



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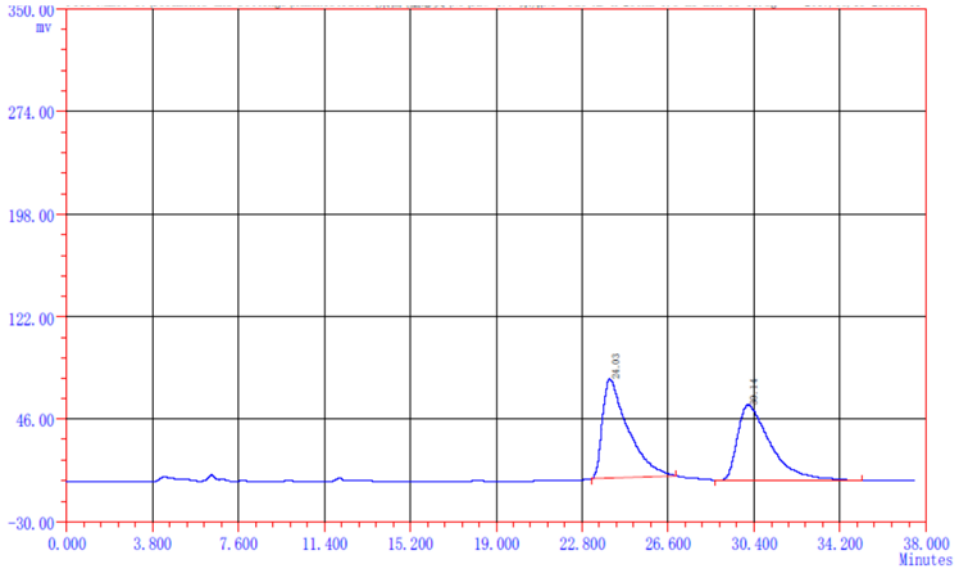
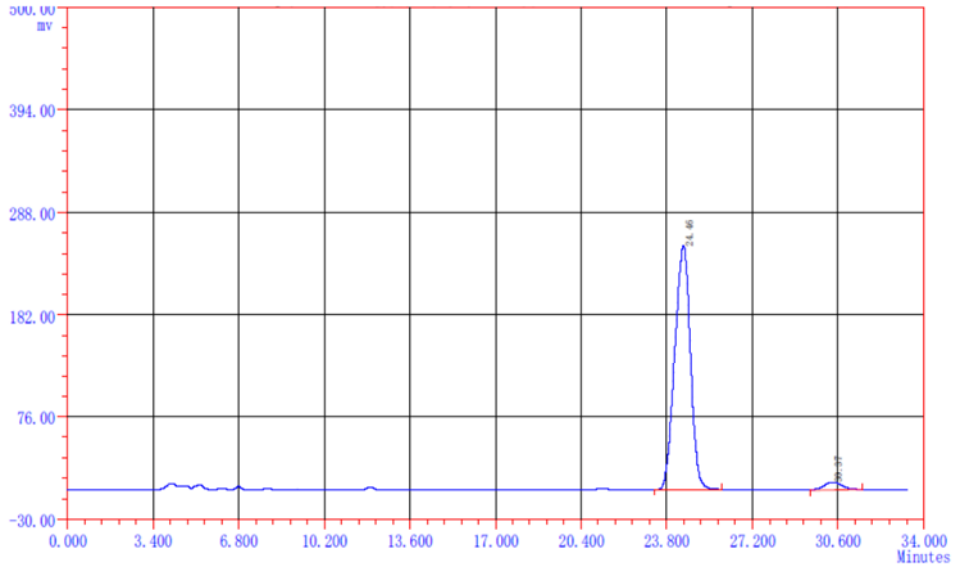


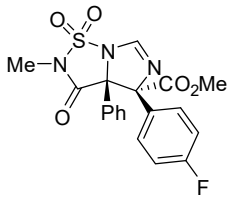
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		24.027	73355	5780649.3	50.0927	2.48	1853
2		30.140	55963	5759252.4	49.9073	2.08	1710
	Σ:		129318	11539901.7	100.0000		

Chiral



ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		24.462	252866	10989890.0	96.4733	0.95	6314
2		30.372	8067	401744.2	3.5267	1.18	7413
	Σ:		260933	11391634.2	100.0000		

4b (Table 3, entry 2)



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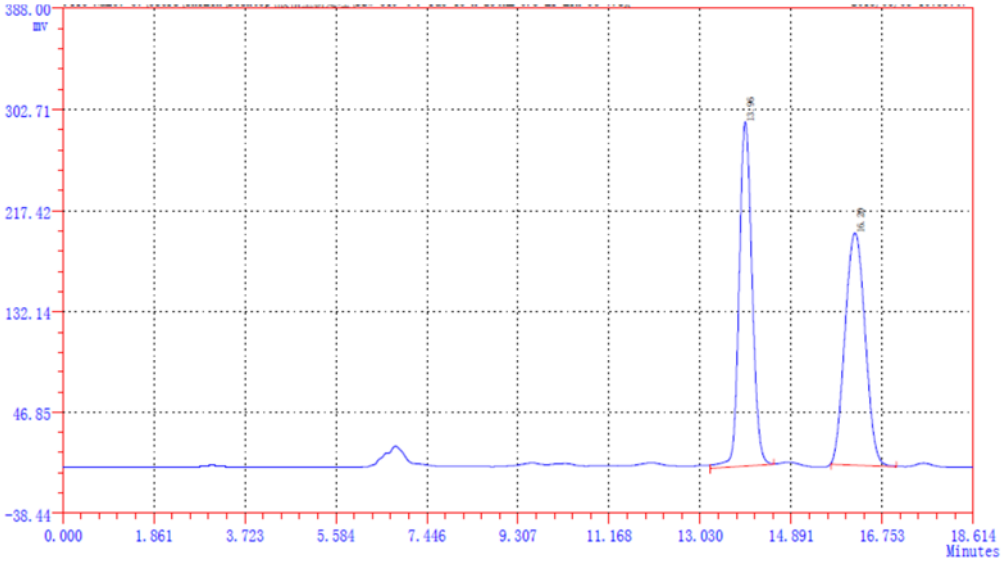

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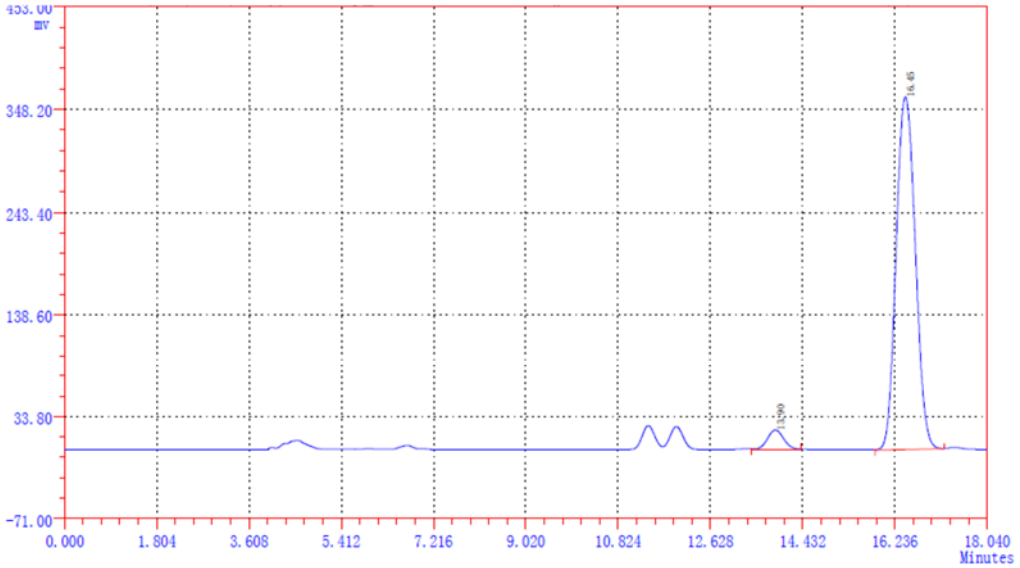
1		13.957	291030	5381597.4	48.8205	1.13	11354
2		16.203	196774	5641644.3	51.1795	1.04	6366
	Σ:		487804	11023241.7	100.0000		

Chiral



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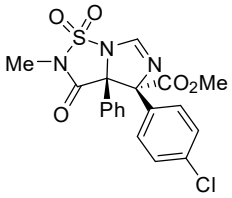
1		13.903	19607	425441.8	4.3180	1.06	8183
2		16.445	361115	9427378.0	95.6820	1.09	7909
	Σ:		380722	9852819.8	100.0000		

4c (Table 3, entry 3)



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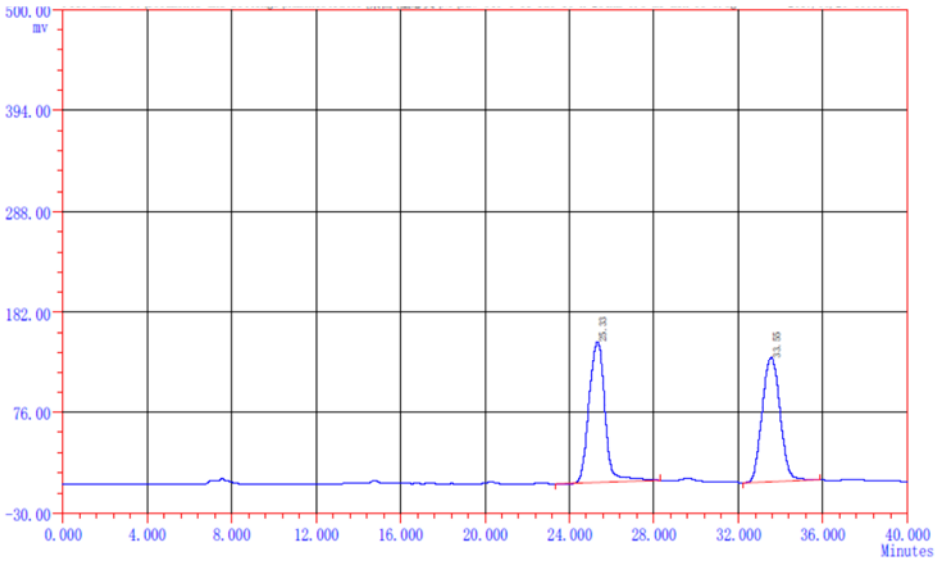

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1		25.325	149051	7835654.0	50.2227	1.07	4625
2		33.550	131931	7766173.4	49.7773	1.08	6474
	Σ:		280982	15601827.4	100.0000		

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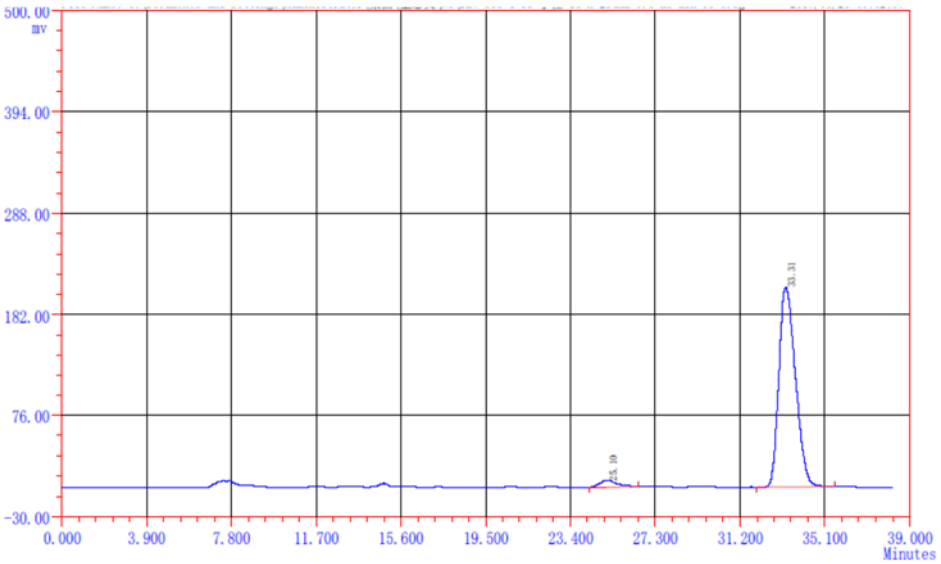

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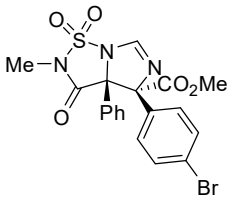
1		25.097	6861	382098.6	3.3152	1.51	4047
2		33.305	210343	11143601.6	96.6848	1.29	7877
	Σ:		217204	11525700.3	100.0000		

4d (Table 3, entry 4)



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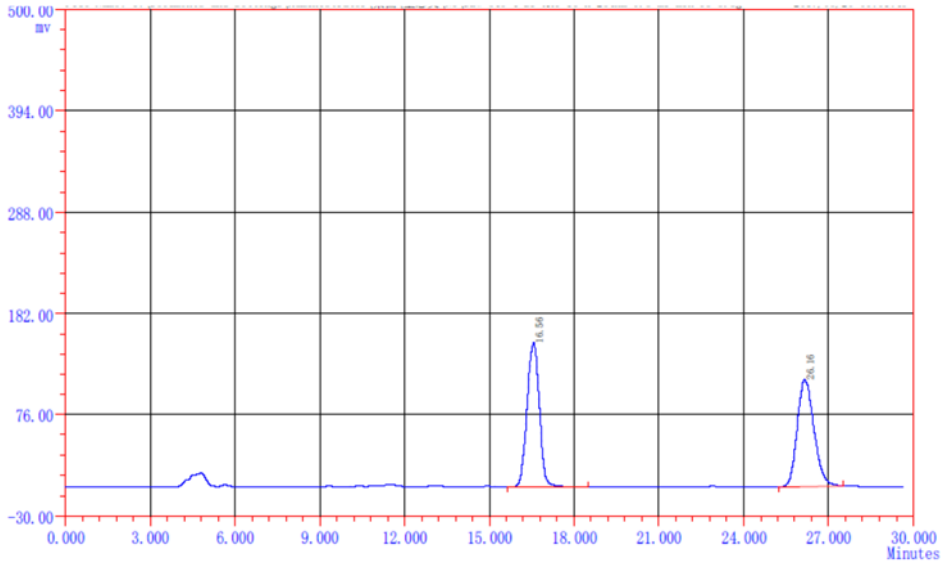

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1		16.562	151461	4610052.0	49.9817	0.98	5901
2		26.162	111821	4613432.9	50.0183	1.21	8014
	Σ:		263282	9223484.9	100.0000		

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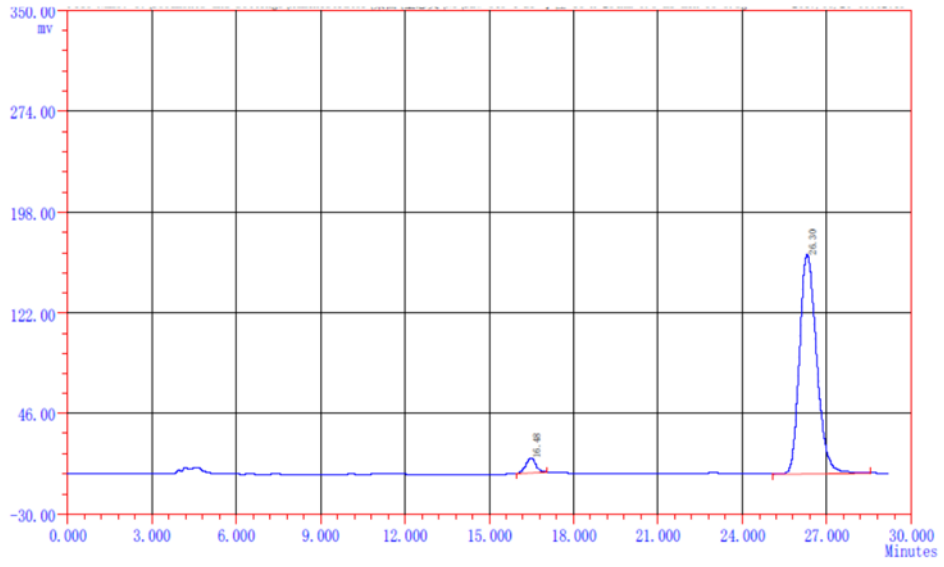

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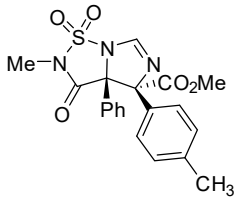
1		16.482	11178	284637.2	3.8878	1.21	8350
2		26.303	165792	7036697.2	96.1122	1.23	7655
	Σ:		176970	7321334.4	100.0000		

4e (Table 3, entry 5)



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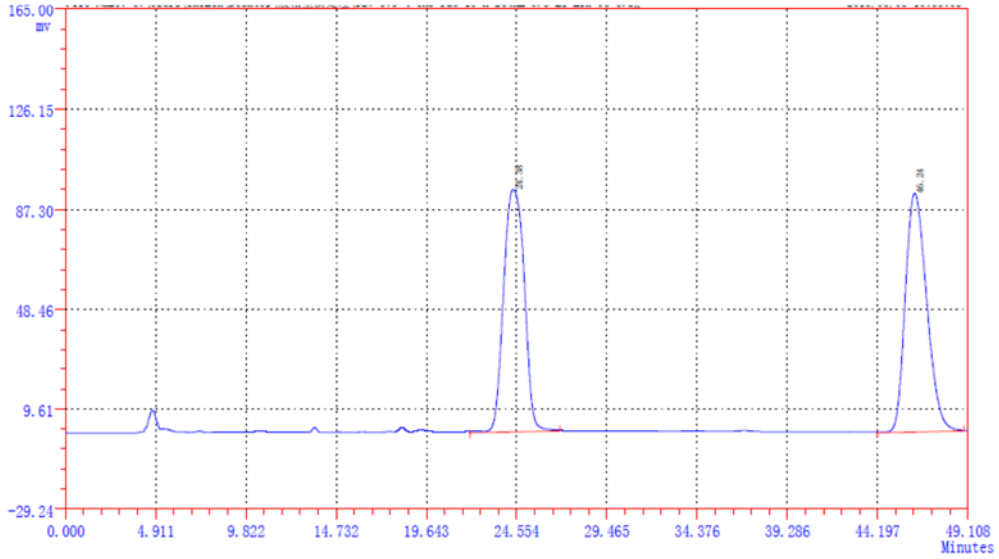

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1		24.378	94454	7400938.8	49.5102	1.06	1929
2		46.238	92987	7547380.4	50.4898	1.25	6468
	Σ:		187441	14948319.2	100.0000		

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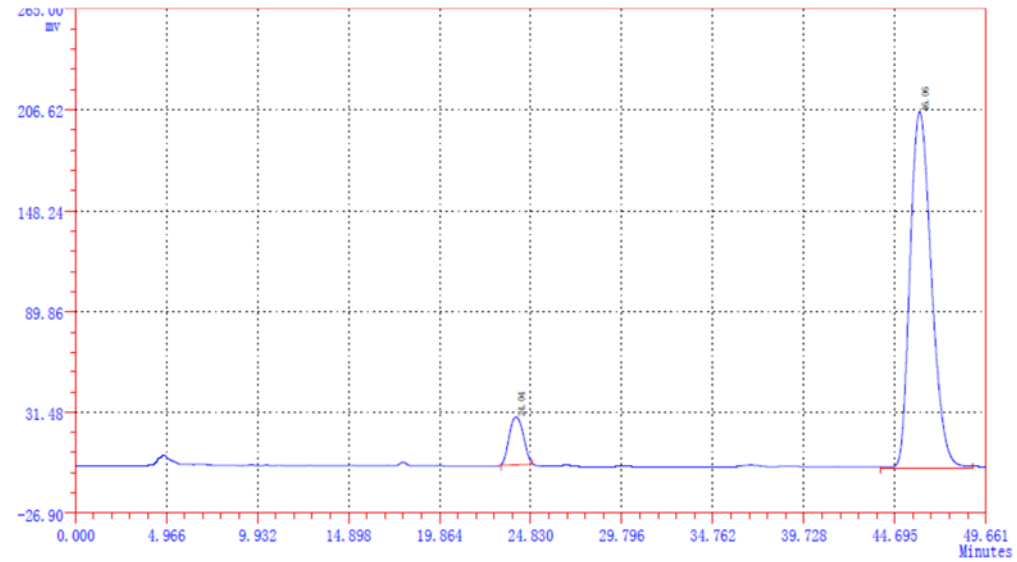

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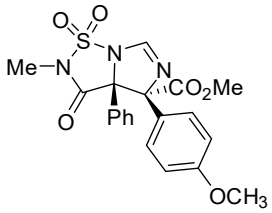
1		24.038	27577	1483080.1	8.1289	1.08	3982
2		46.062	206353	16761410.5	91.8711	1.24	6409
	Σ:		233930	18244490.6	100.0000		

4f (Table 3, entry 6)



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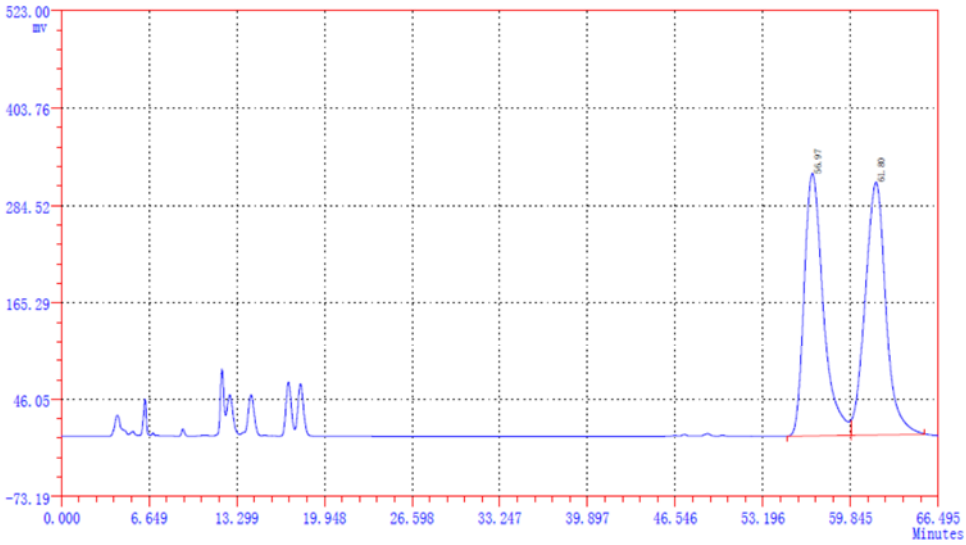

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1		56.972	322558	32669977.7	49.0573	1.64	6306
2		61.800	310545	33925558.6	50.9427	1.07	6378
	Σ:		633103	66595536.4	100.0000		

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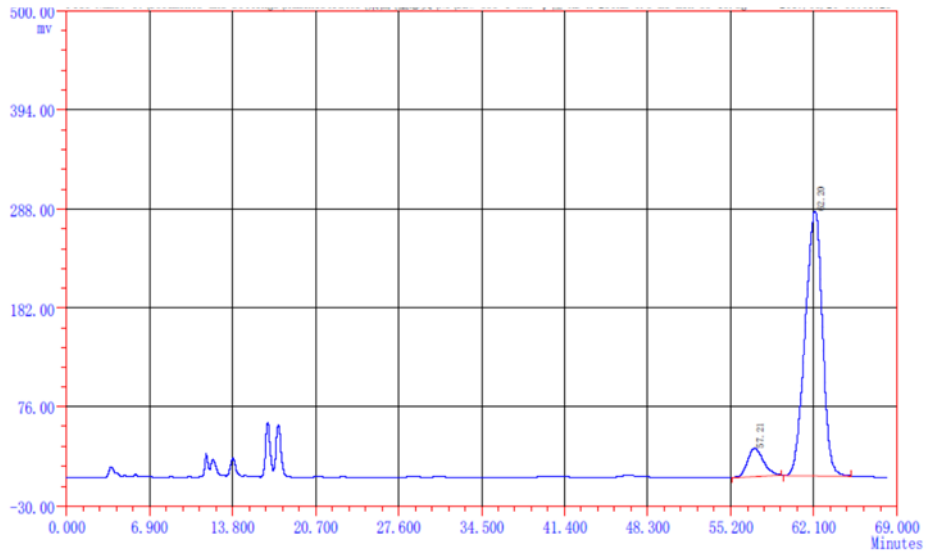

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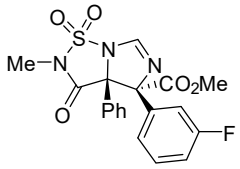
1		57.205	30296	2764355.2	8.8949	1.39	7834
2		62.202	284135	28313512.1	91.1051	0.91	7766
	Σ:		314431	31077867.2	100.0000		

4g (Table 3, entry 7)



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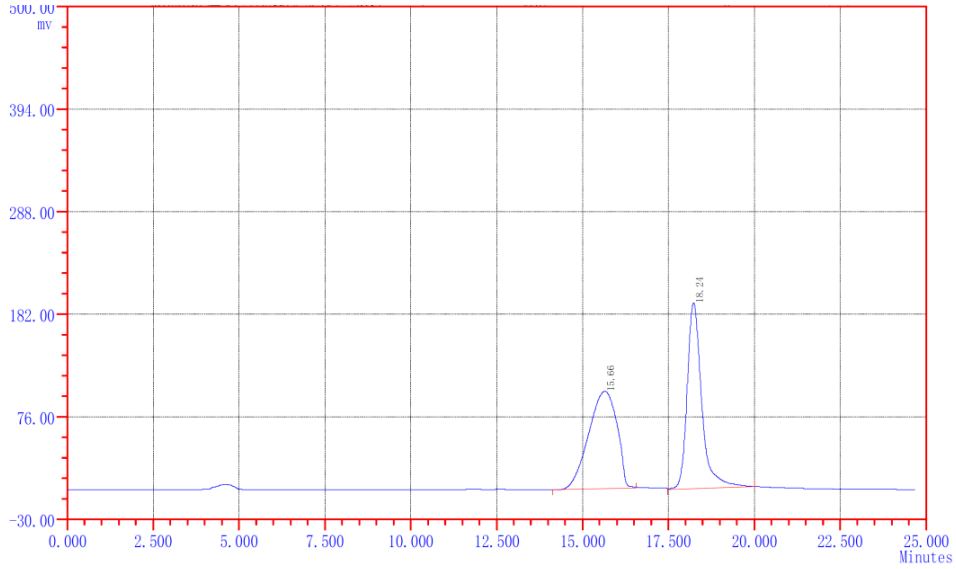

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		15.657	100764	5807639.8	50.5791	0.81	1471
2	组份 2	18.237	192365	5674644.5	49.4209	1.47	7617
	Σ:		293129	11482284.3	100.0000		

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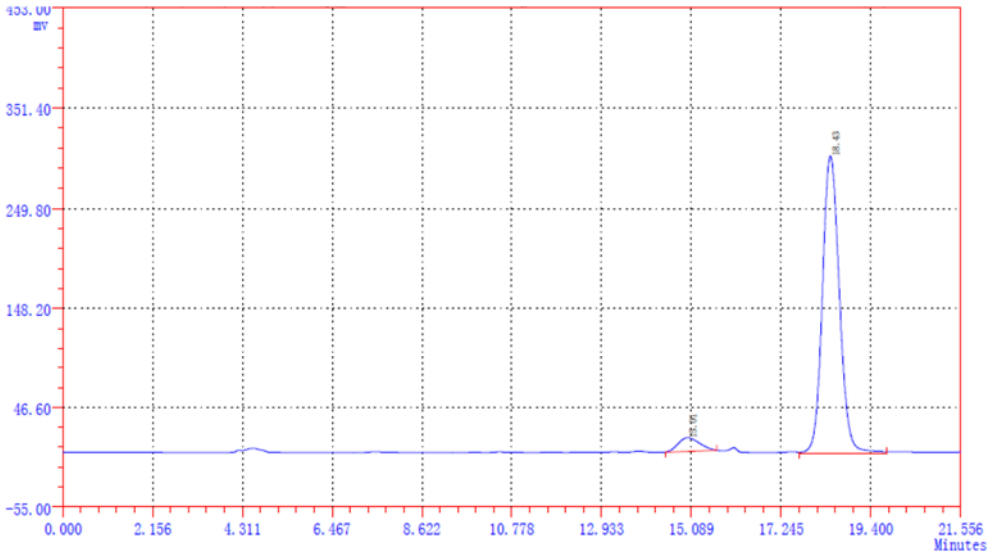

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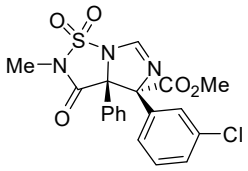
1		15.007	13742	493869.1	5.4123	1.31	3475
2		18.433	302882	8631141.0	94.5877	1.11	8340
	Σ:		316624	9125010.0	100.0000		

4h (Table 3, entry 8)



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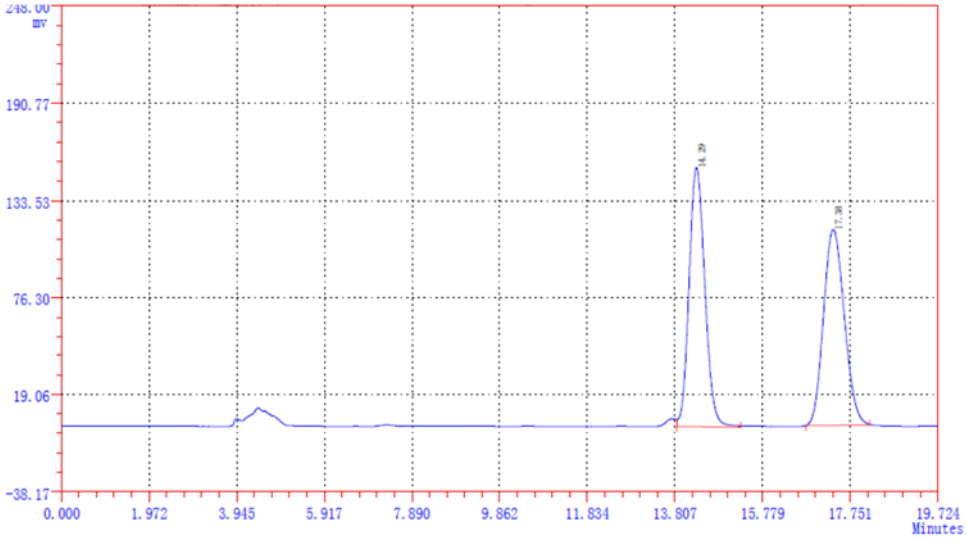

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1		14.293	152773	3781242.7	50.0459	1.13	6647
2		17.375	115874	3774311.5	49.9541	1.13	5671
	Σ:		268647	7555554.2	100.0000		

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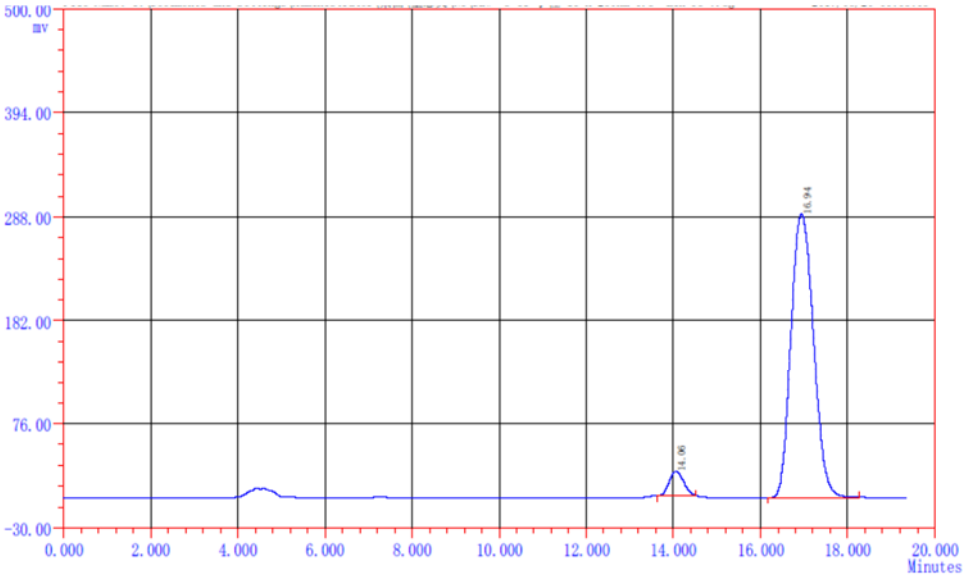

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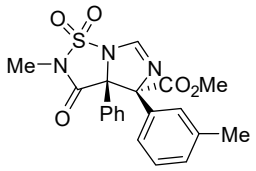
1		14.058	25315	588294.3	5.3331	1.11	7294
2		16.943	290592	10442657.3	94.6669	1.13	4431
	Σ:		315907	11030951.6	100.0000		

4i (Table 3, entry 9)



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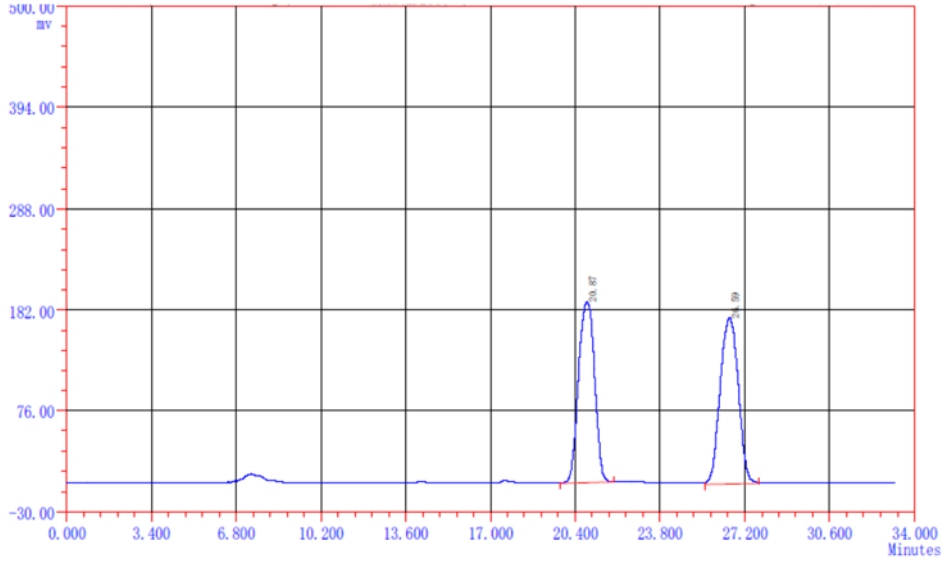

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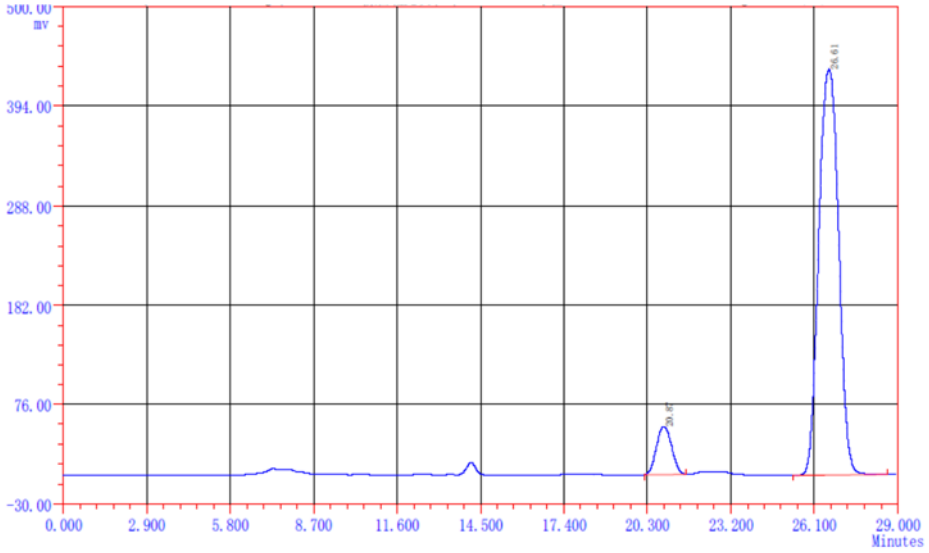
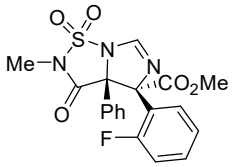
4	组份 4	20.870	190599	8512804.2	49.1174	0.98	4352
2		26.588	174462	8818750.6	50.8826	1.00	5514
	Σ:		365061	17331554.7	100.0000		

Chiral



ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
4	组份 4	20.872	51464	1903421.0	8.4022	1.07	6347
2		26.608	432744	20750452.8	91.5978	1.02	6137
	Σ:		484208	22653873.8	100.0000		

4j (Table 3, entry 10)



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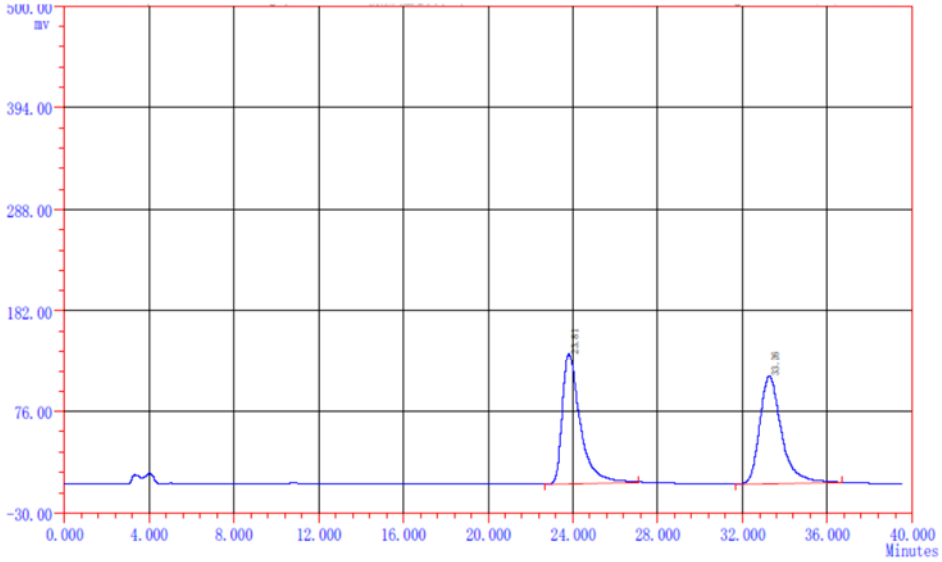

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1		23.807	136852	8070919.4	49.9834	1.88	3248
2		33.263	112845	8076268.4	50.0166	1.46	4305
	Σ:		249697	16147187.8	100.0000		

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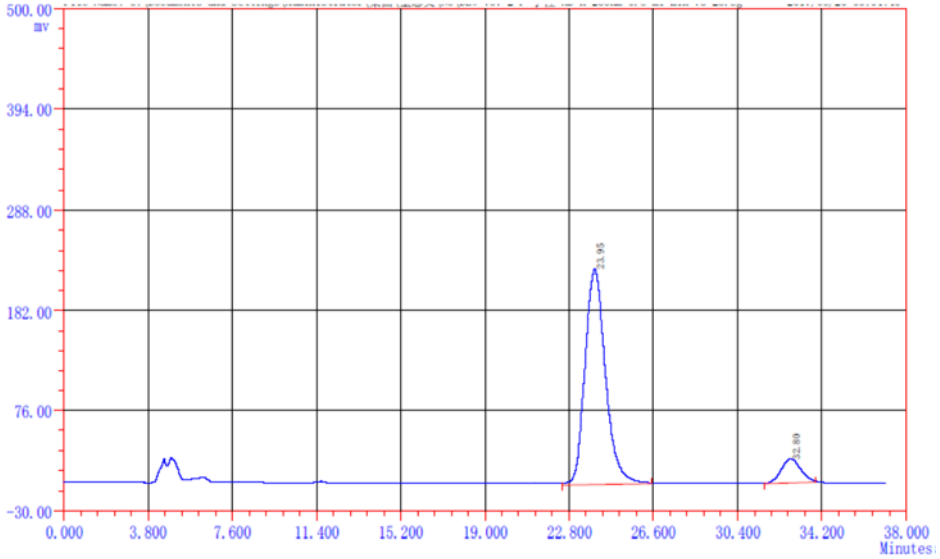

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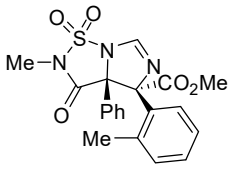
1		23.953	228616	14695524.6	90.4719	1.23	2768
2		32.795	25336	1547660.2	9.5281	1.14	5745
	Σ:		253952	16243184.9	100.0000		

4k (Table 3, entry 11)



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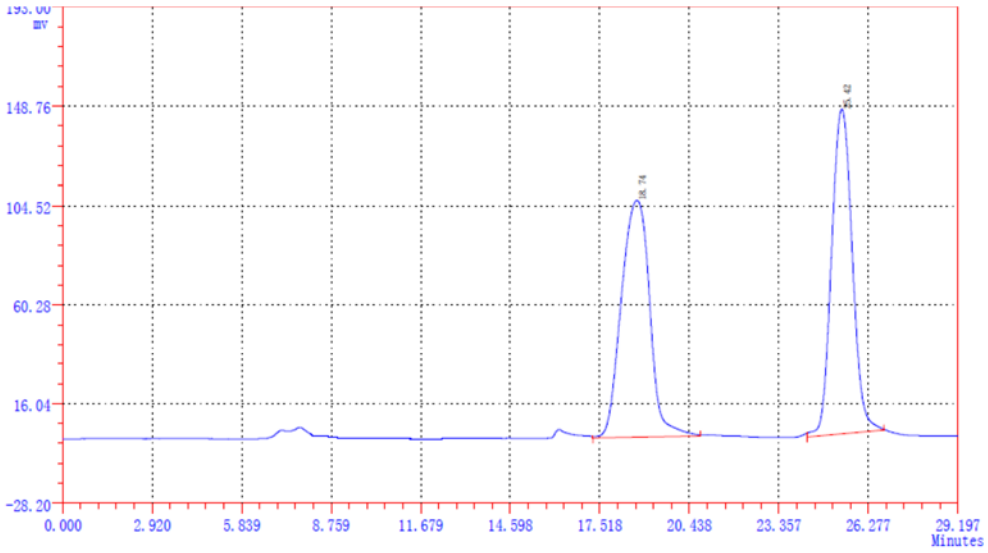
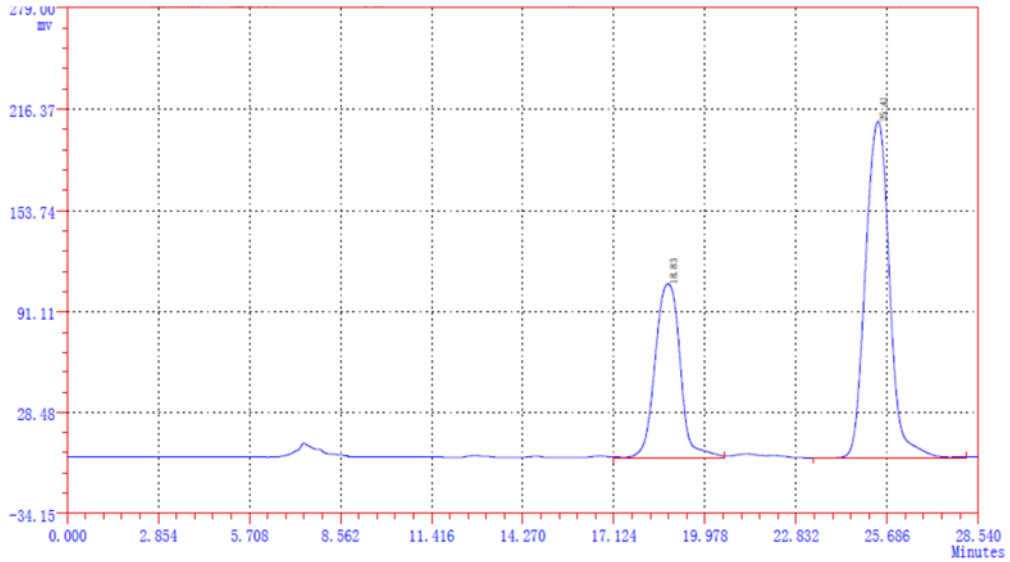


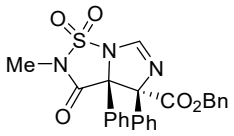
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
3		18.738	105843	6817643.7	50.0864	1.03	1687
2		25.418	144816	6794125.0	49.9136	1.12	5850
	Σ:		250659	13611768.7	100.0000		

Chiral

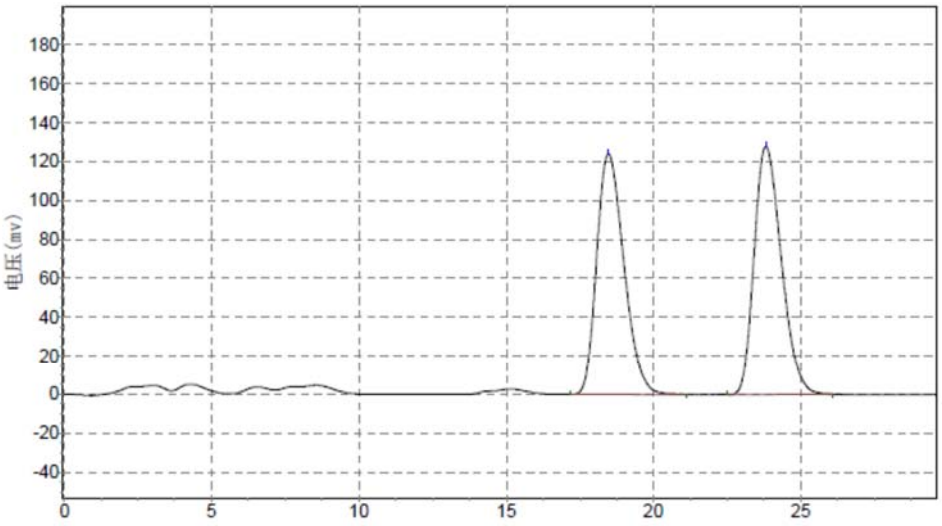


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
3		18.830	108289	5935613.5	35.1046	1.07	2352
2		25.410	208705	10972742.8	64.8954	1.07	4656
	Σ:		316994	16908356.3	100.0000		

4l (Table 3, entry 12)

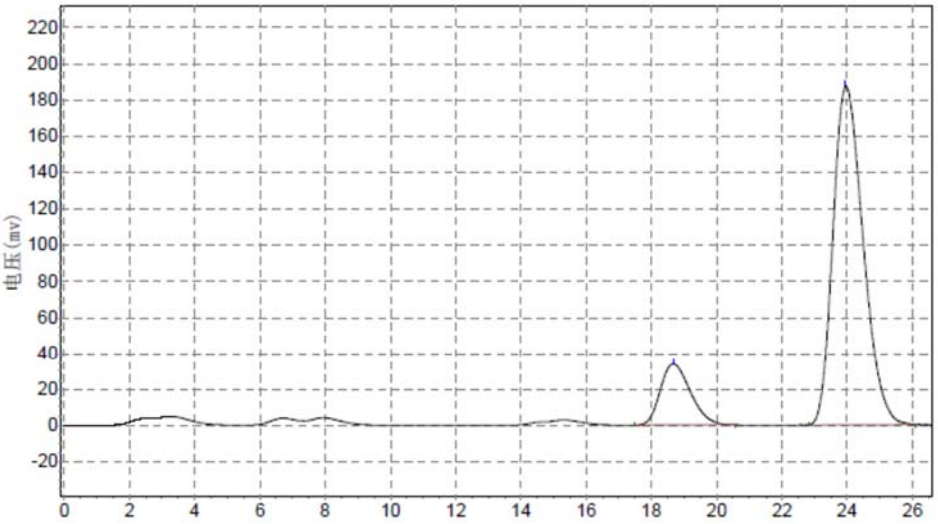


Racemic



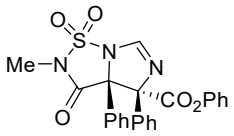
峰号	保留时间	峰高	峰面积	含量
1	18.462	124097.680	7995624.500	49.3522
2	23.813	127880.063	8205511.500	50.6478
总计		251977.742	16201136.000	100.0000

Chiral

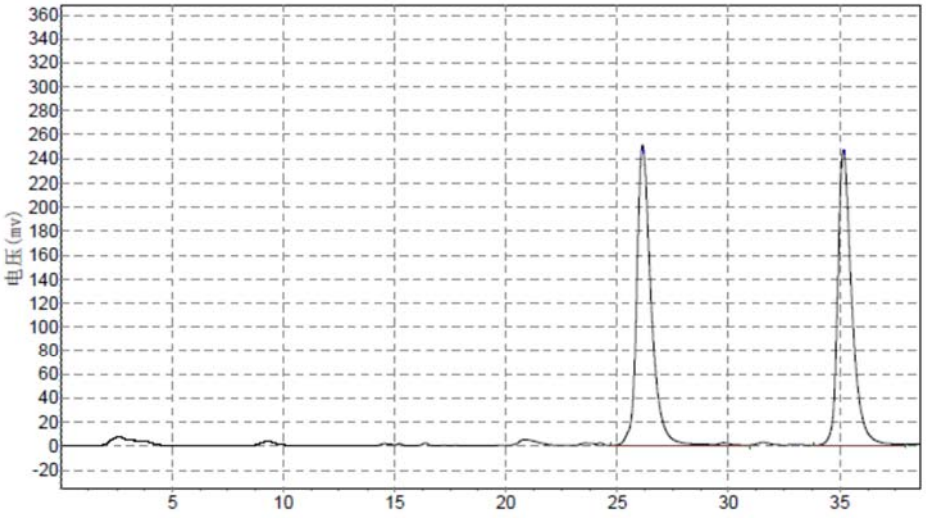


峰号	保留时间	峰高	峰面积	含量
1	18.667	33791.516	2157245.250	15.1998
2	23.950	187331.813	12035369.000	84.8002
总计		221123.328	14192614.250	100.0000

4m (Table 3, entry 13)

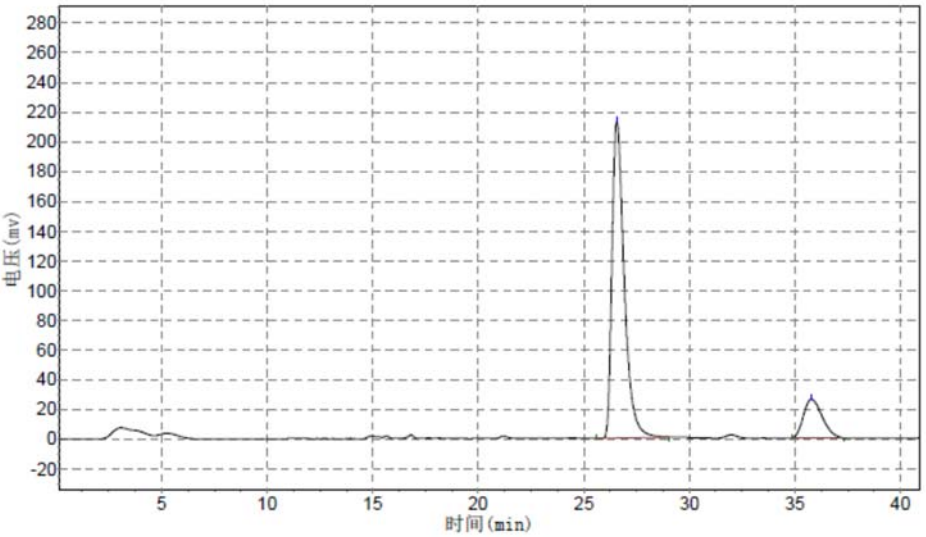


Racemic



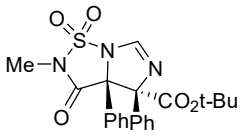
峰号	保留时间	峰高	峰面积	含量
1	26.198	248855.313	11002000.000	50.3223
2	35.198	245548.234	10861061.000	49.6777
总计		494403.547	21863061.000	100.0000

Chiral



峰号	保留时间	峰高	峰面积	含量
1	26.563	212963.469	8595660.000	84.3171
2	35.795	25964.457	1598787.875	15.6829
总计		238927.926	10194447.875	100.0000

4n (Table 3, entry 14)



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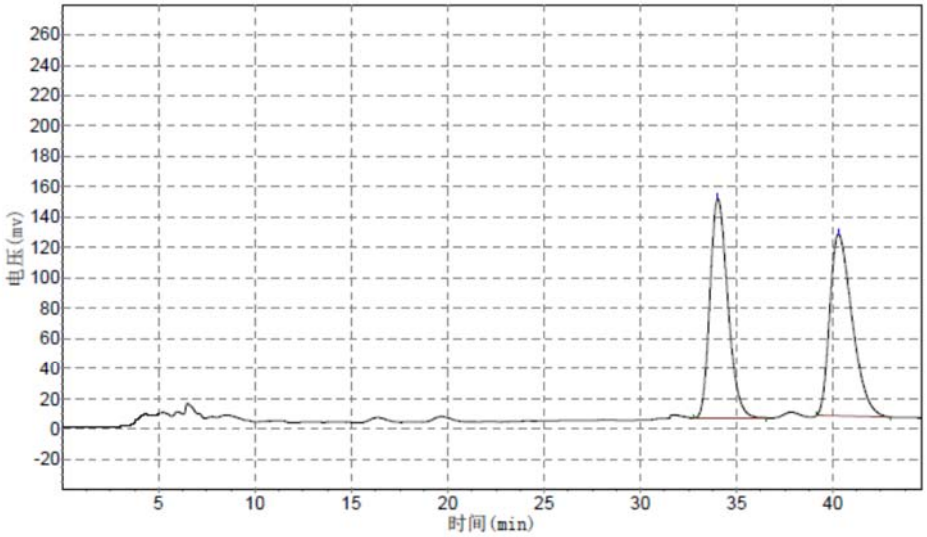
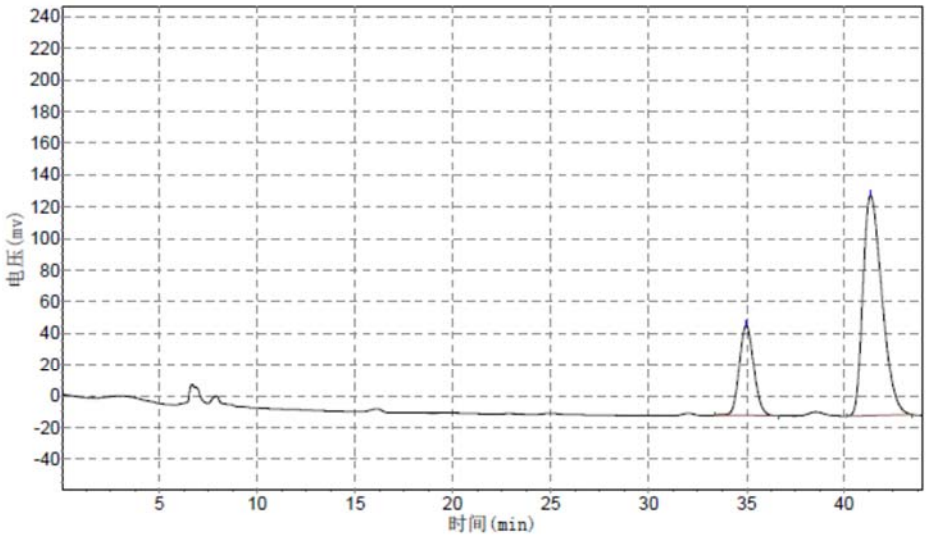


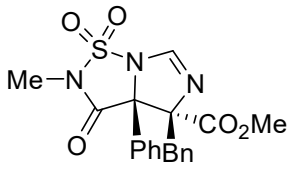
峰号	保留时间	峰高	峰面积	含量
1	34.032	144875.781	9333938.000	48.9971
2	40.302	120222.414	9716056.000	51.0029
总计		265098.195	19049994.000	100.0000

Chiral



峰号	保留时间	峰高	峰面积	含量
1	34.972	57511.621	2967145.500	23.8994
2	41.333	139755.313	9447989.000	76.1006
总计		197266.934	12415134.500	100.0000

4o (Table 3, entry 15)



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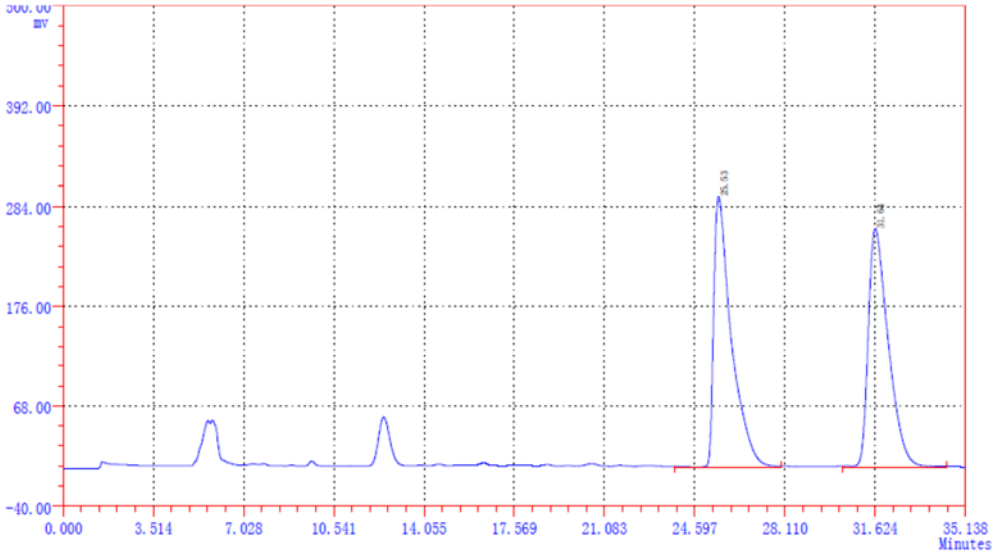
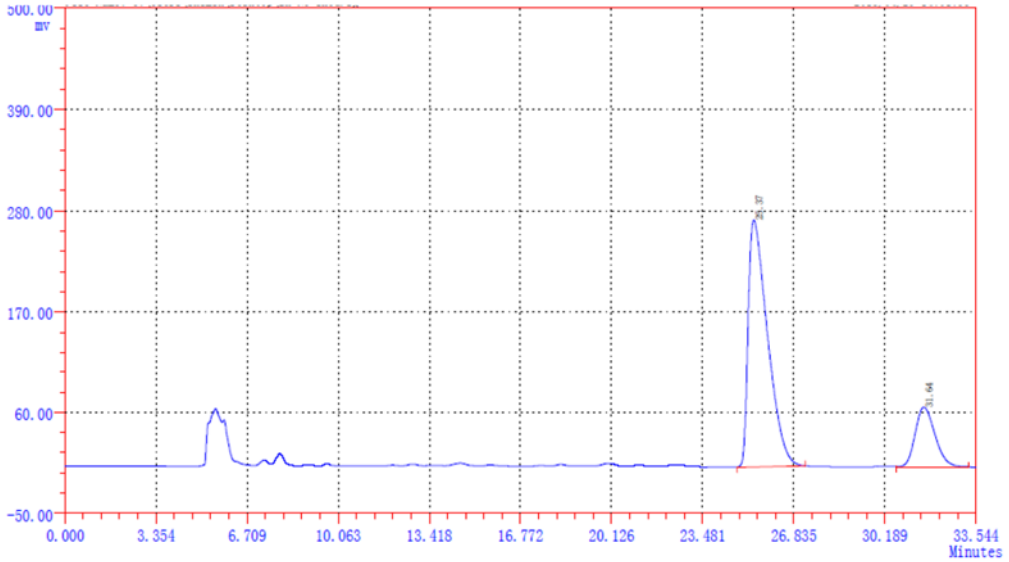


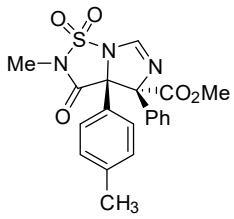
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		25.532	292500	13955732.1	49.8906	2.45	5707
2		31.637	257851	14016961.2	50.1094	1.64	6751
	Σ:		550351	27972693.3	100.0000		

Chiral

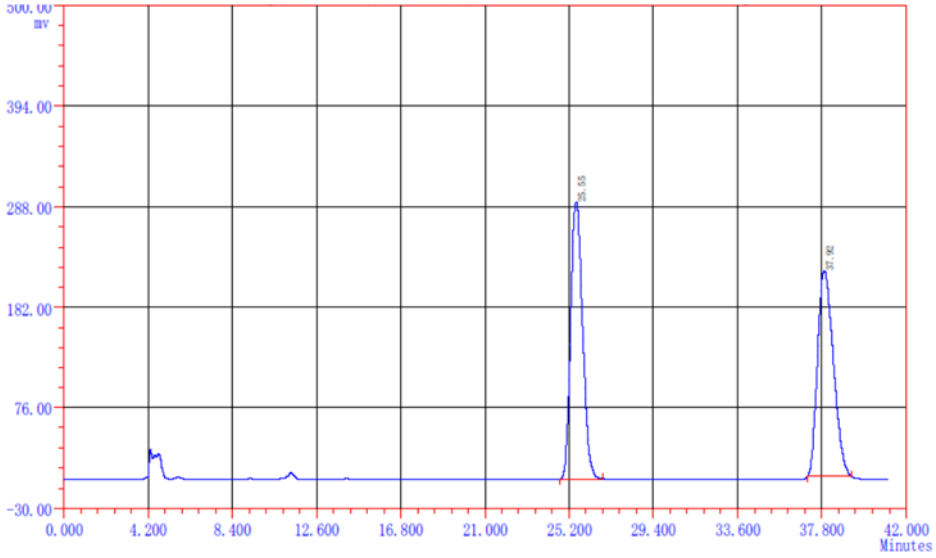


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		25.372	269420	13395945.8	80.0109	2.09	5190
2		31.642	65304	3346701.6	19.9891	1.22	7598
	Σ:		334724	16742647.4	100.0000		

4p (Table 4, entry 1)

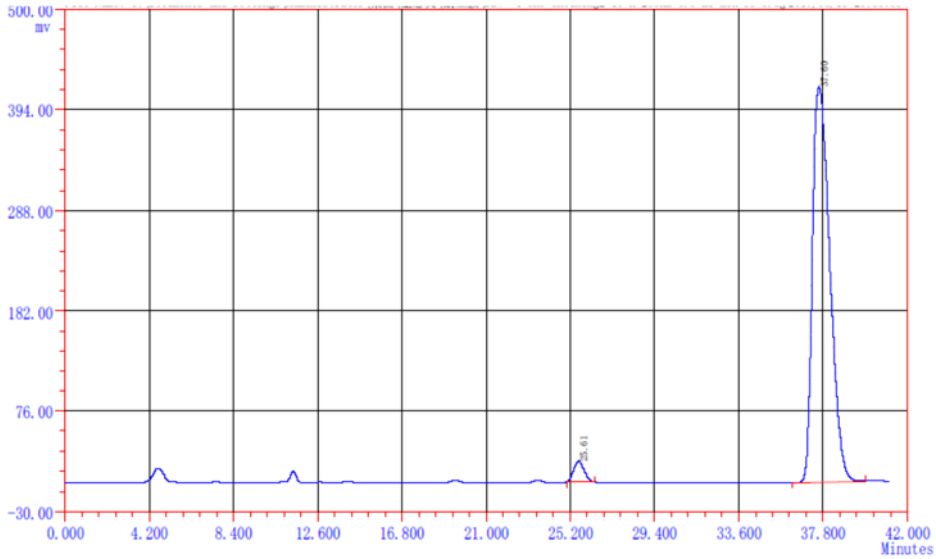


Racemic



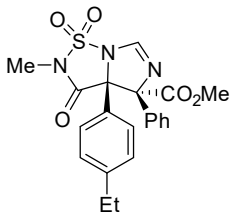
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		25.547	292537	12038171.3	50.1839	1.17	7681
2		37.922	217633	11949948.4	49.8161	1.27	9506
	Σ:		510170	23988119.7	100.0000		

Chiral

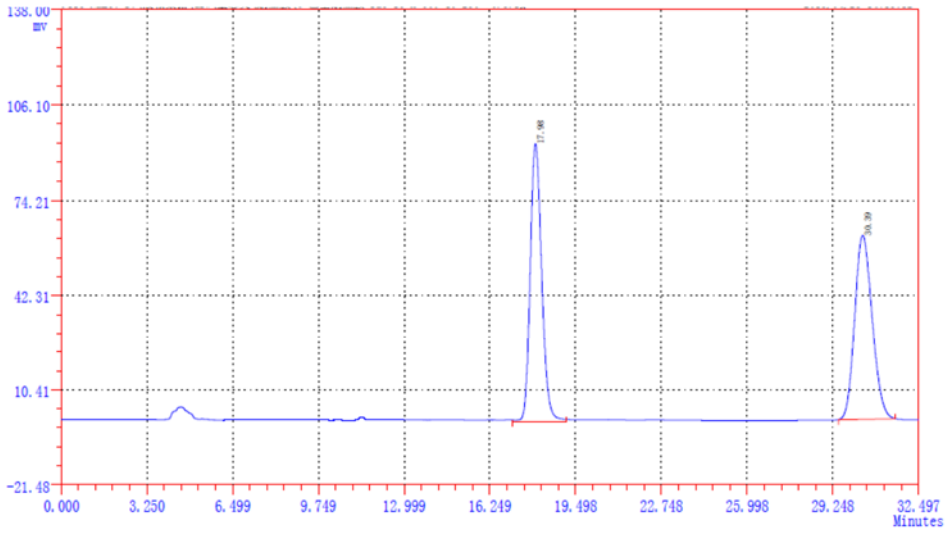


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		25.613	21730	806112.9	3.1067	1.10	9501
2		37.598	417607	25141662.1	96.8933	1.48	7773
	Σ:		439337	25947775.0	100.0000		

4q (Table 4, entry 2)

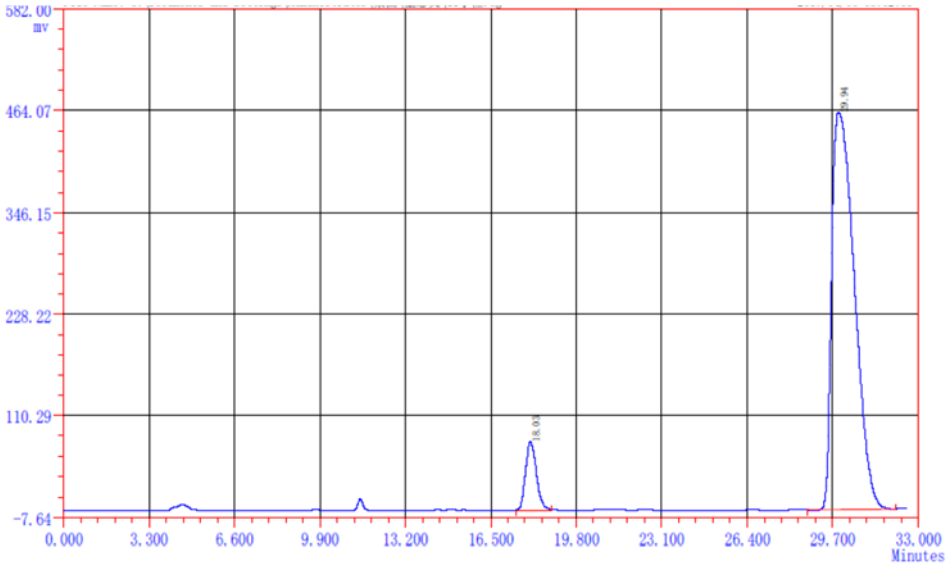


Racemic



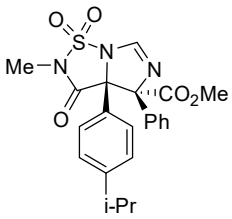
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
2		17.983	93373	2846038.0	49.8058	1.17	6938
2		30.387	62102	2868227.1	50.1942	1.17	8627
	Σ:		155475	5714265.1	100.0000		

Chiral

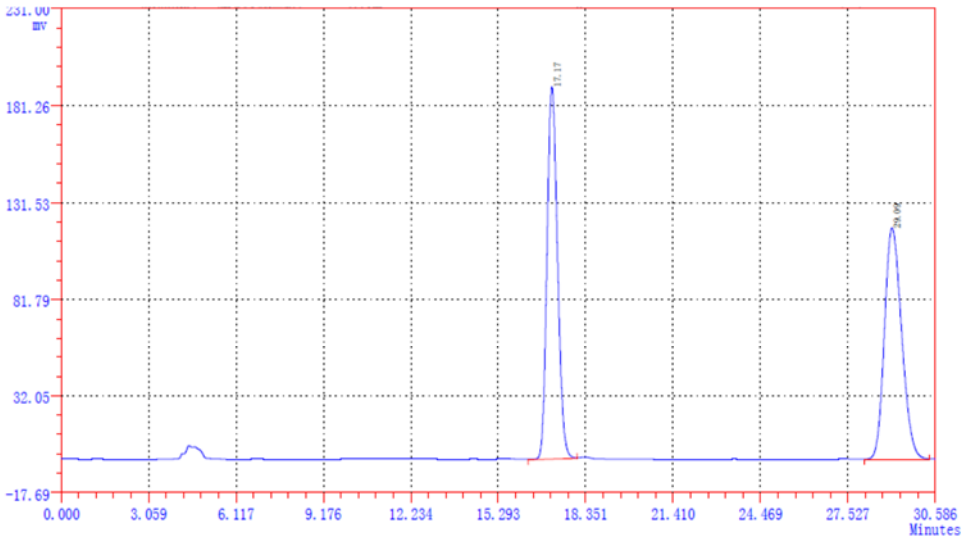


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
2	组份 2	18.032	80187	2362900.0	7.7754	1.19	7463
2		29.937	461344	28026445.4	92.2246	1.84	4840
	Σ:		541531	30389345.5	100.0000		

4r (Table 4, entry 3)

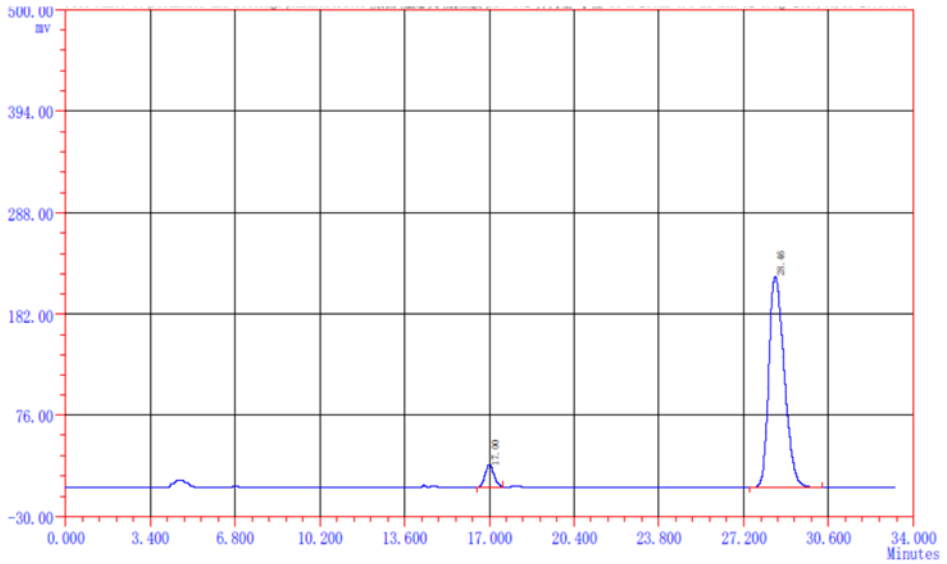


Racemic



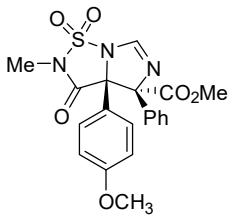
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		17.173	191411	4875615.5	49.2996	1.15	9060
2		29.087	119366	5014154.6	50.7004	1.19	9556
Σ:			310777	9889770.2	100.0000		

Chiral

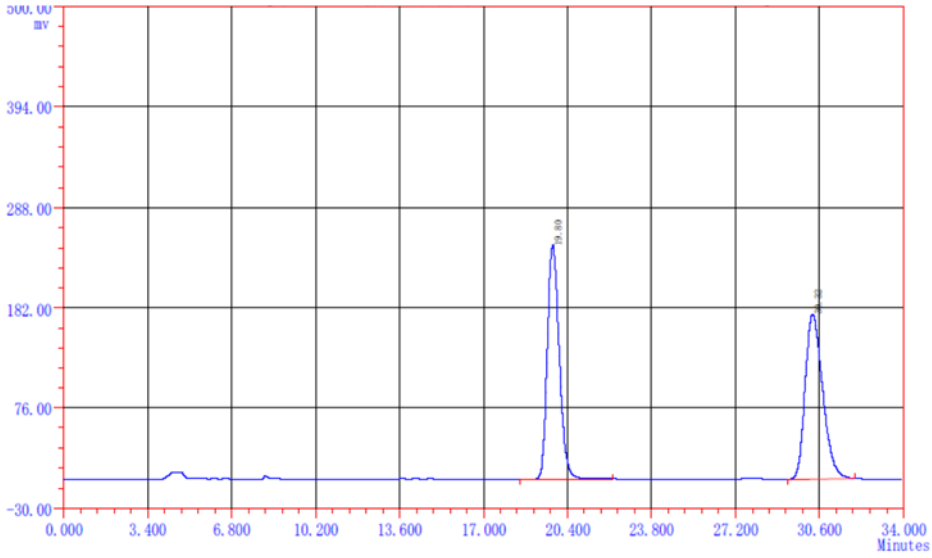


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		17.000	22898	564827.5	5.5971	1.10	9466
2		28.463	221732	9526588.8	94.4029	1.31	8747
Σ:			244630	10091416.4	100.0000		

4s (Table 4, entry 4)

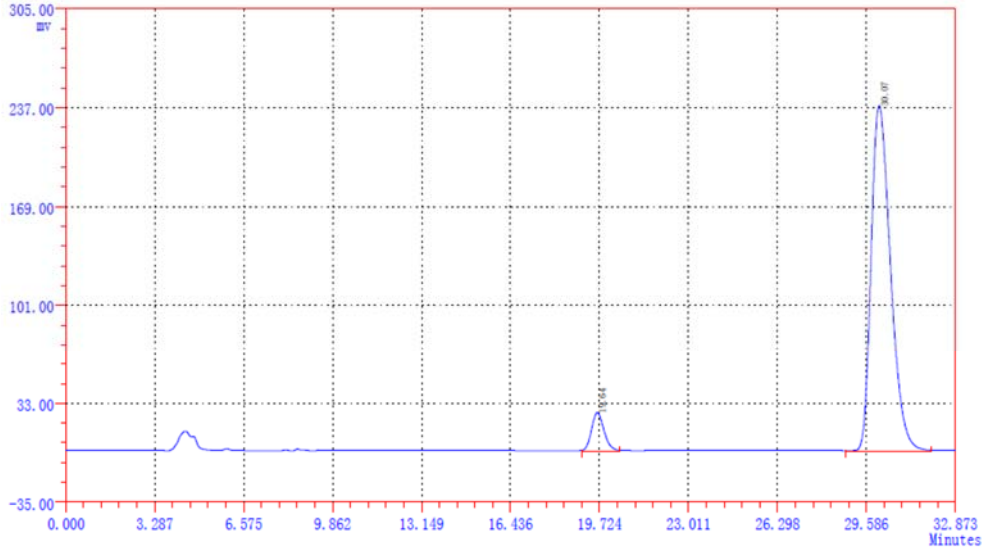


Racemic



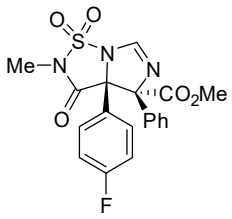
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
4	组份 4	19.797	248321	8451252.5	49.5194	1.22	6744
2		30.323	174863	8615295.2	50.4806	1.30	7550
	Σ:		423184	17066547.7	100.0000		

Chiral

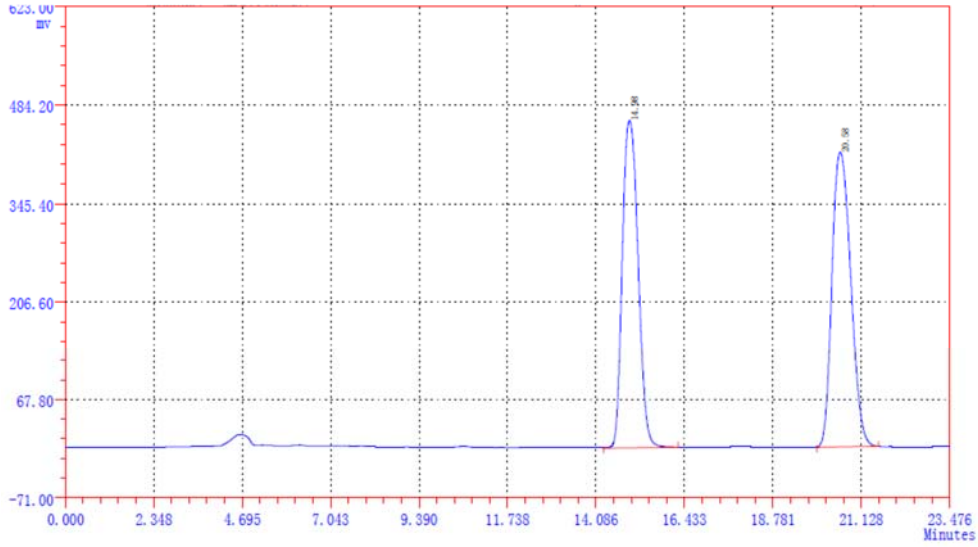


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
4		19.643	26903	904539.2	7.0130	1.23	6803
2		30.068	238727	11993436.8	92.9870	1.35	7139
	Σ:		265630	12897976.0	100.0000		

4t (Table 4, entry 5)

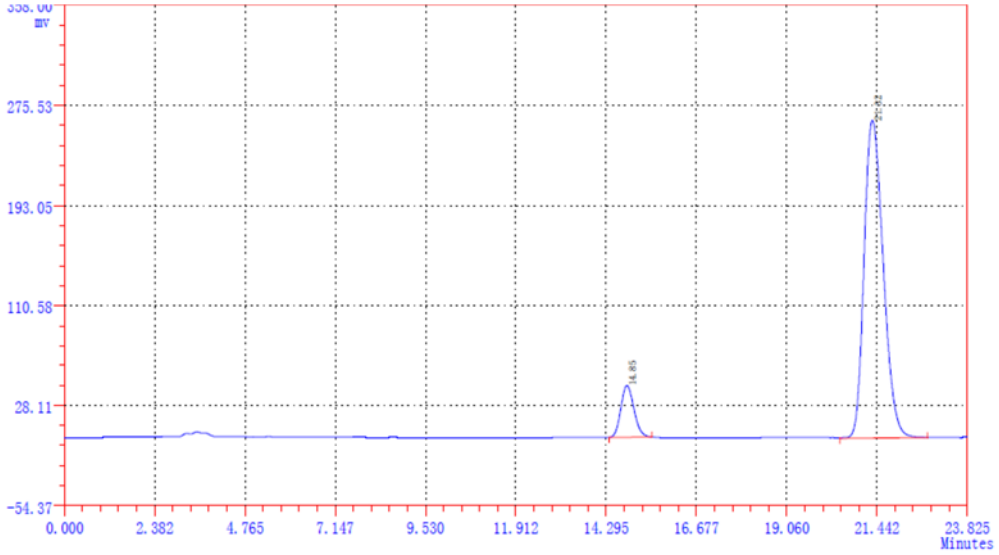


Racemic



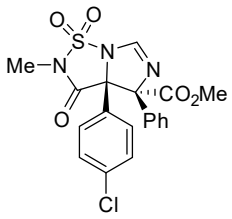
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		14.980	462899	13499179.1	48.2902	1.18	5259
4		20.577	416780	14455076.5	51.7098	1.22	7015
Σ:			879679	27954255.5	100.0000		

Chiral

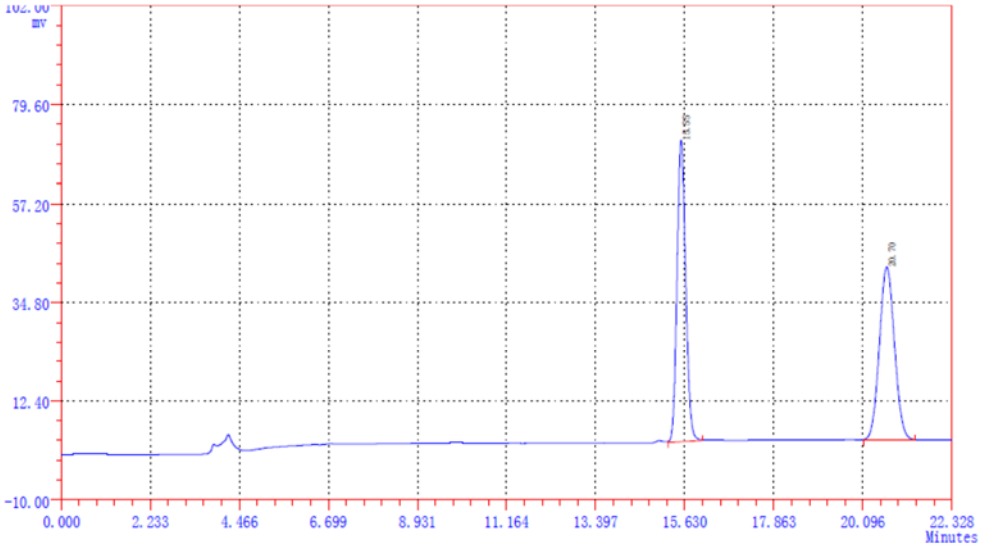


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		14.850	42482	1026694.3	9.9999	1.15	7525
4		21.325	261923	9240335.2	90.0001	1.26	7282
Σ:			304405	10267029.4	100.0000		

4u (Table 4, entry 6)

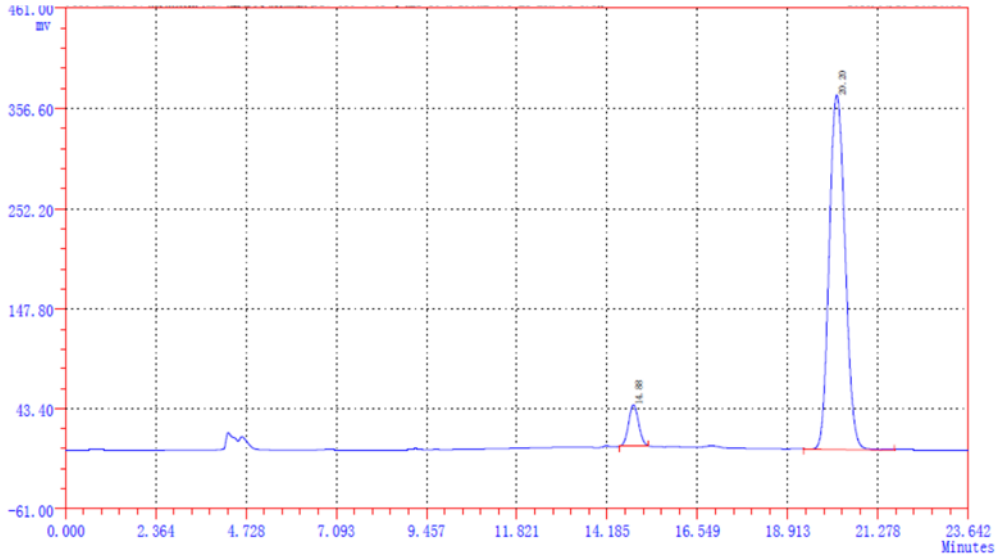


Racemic



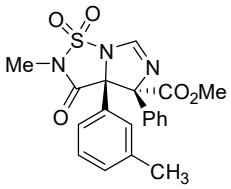
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		15.547	68371	1033505.4	49.6183	1.12	21082
4		20.702	39568	1049405.0	50.3817	1.06	12143
Σ:			107939	2082910.4	100.0000		

Chiral

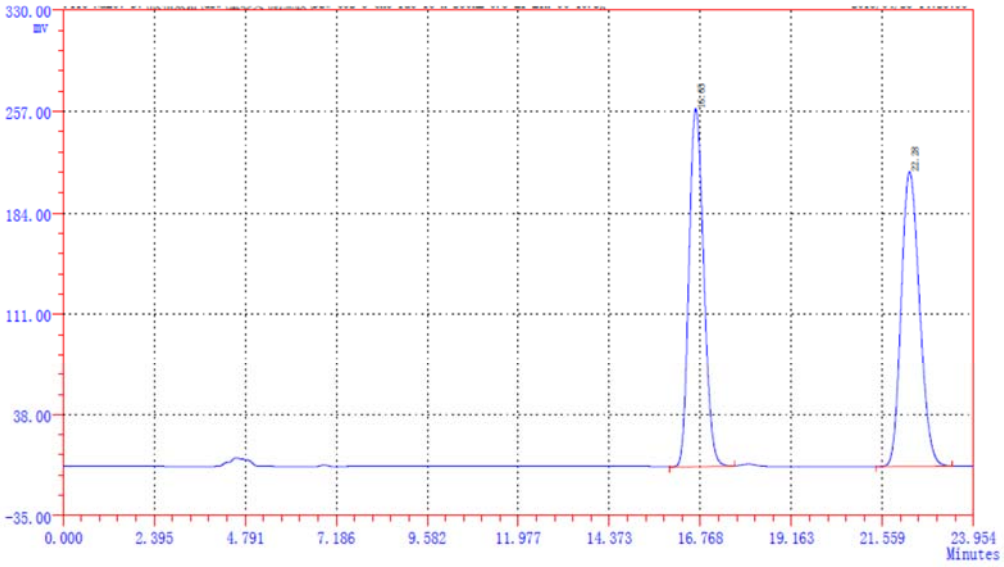


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		14.880	42948	784920.7	6.8195	1.06	13212
4		20.203	369903	10724987.4	93.1805	1.11	9677
Σ:			412851	11509908.1	100.0000		

4v (Table 4, entry 7)

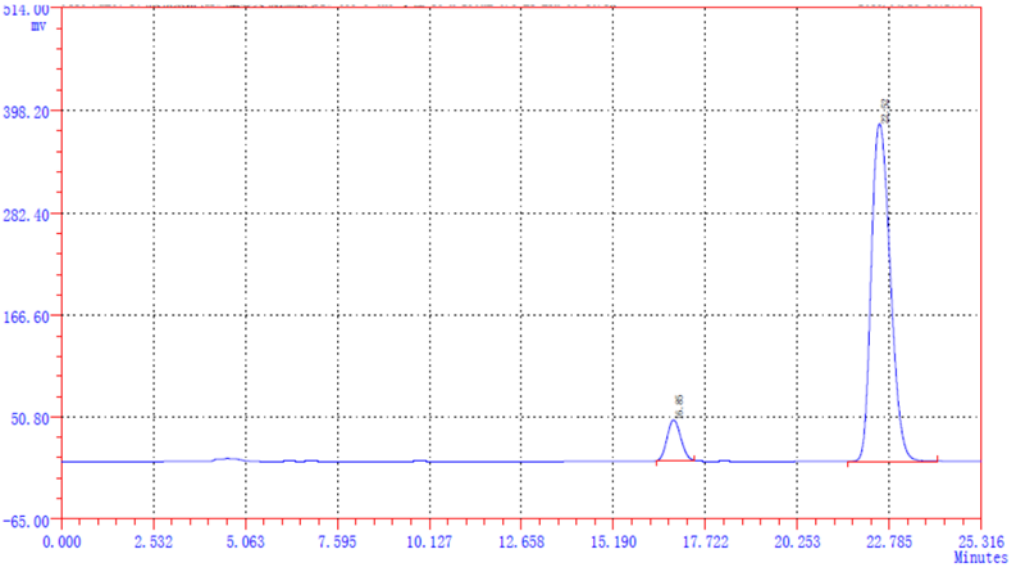


Racemic



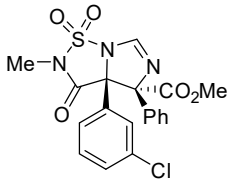
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		16.653	258869	7018923.3	49.5261	1.16	7519
2		22.282	212790	7153237.7	50.4739	1.17	8756
	Σ:		471659	14172161.0	100.0000		

Chiral

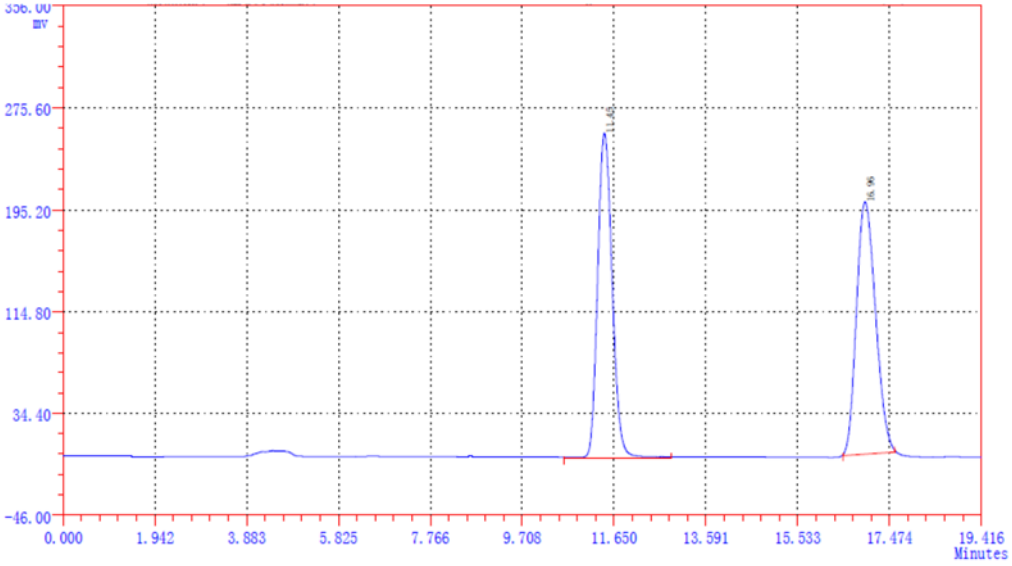


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		16.850	46442	1223946.8	8.0453	1.12	8147
2		22.522	382876	13989329.9	91.9547	1.25	7573
	Σ:		429318	15213276.7	100.0000		

4w (Table 4, entry 8)

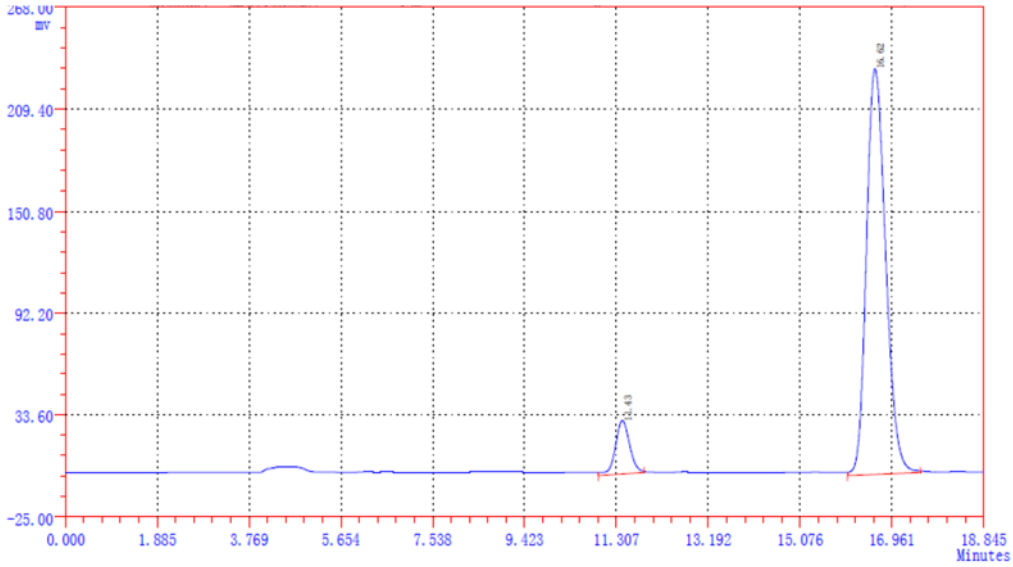


Racemic



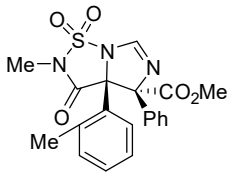
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		11.448	257274	5598270.7	50.0496	1.16	5517
2		16.962	199254	5587180.0	49.9504	1.21	7293
	Σ:		456528	11185450.7	100.0000		

Chiral



ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		11.433	30405	622253.1	8.8539	1.17	6221
2		16.618	233003	6405783.1	91.1461	1.22	7282
	Σ:		263408	7028036.2	100.0000		

4x (Table 4, entry 9)



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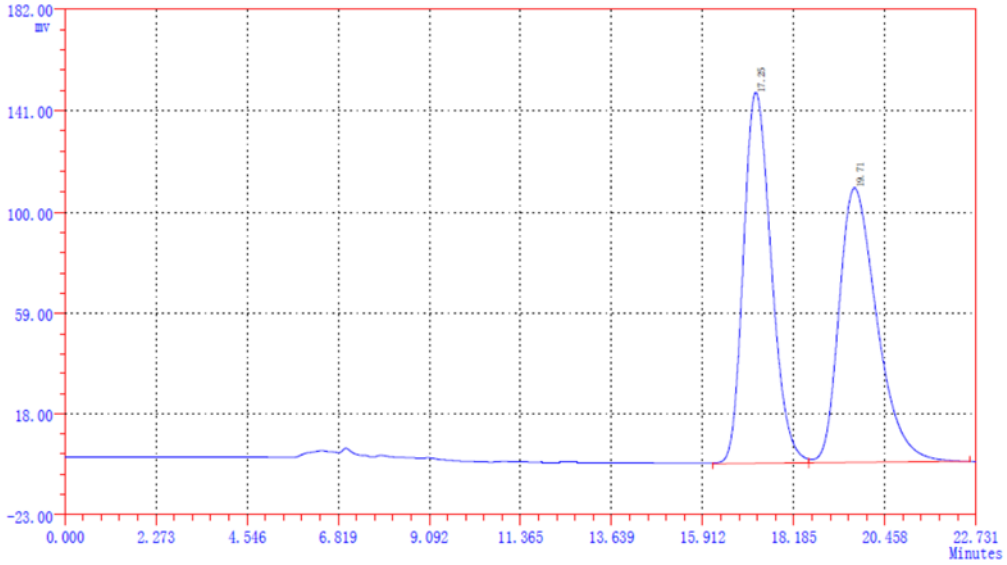
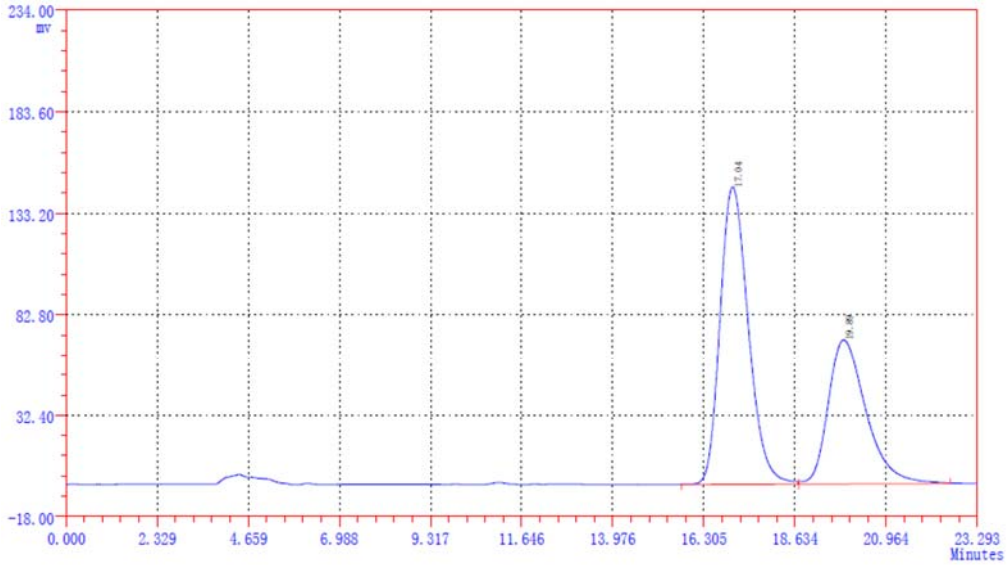


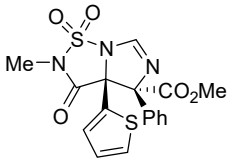
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
2		17.247	150535	7295440.0	49.8202	1.23	2524
4		19.712	111624	7348100.6	50.1798	1.42	1787
	Σ:		262159	14643540.6	100.0000		

Chiral



ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		17.043	148357	7457956.0	60.6656	1.25	2291
4		19.890	72196	4835590.8	39.3344	1.41	1758
	Σ:		220553	12293546.8	100.0000		

4y (Table 4, entry 10)



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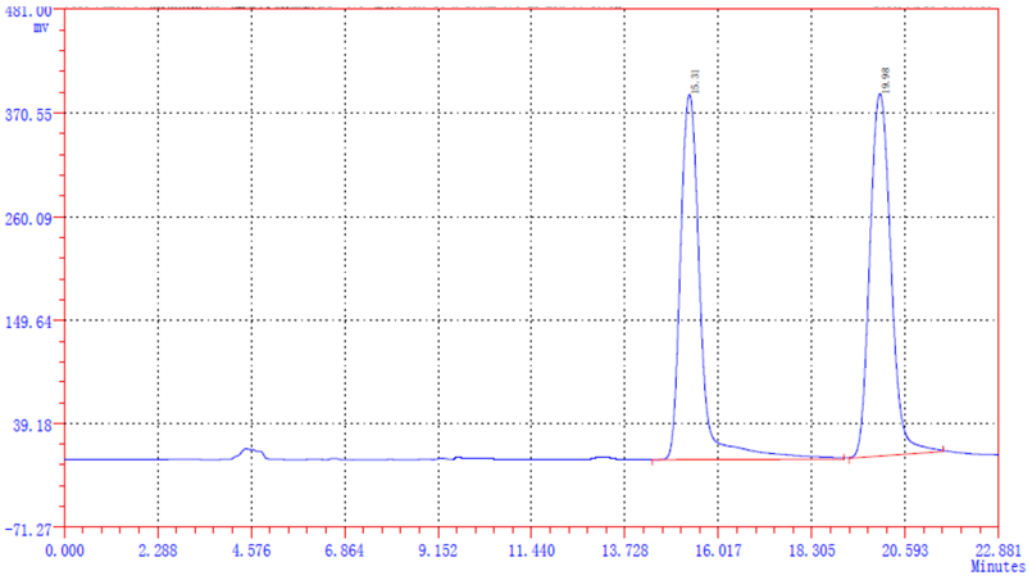
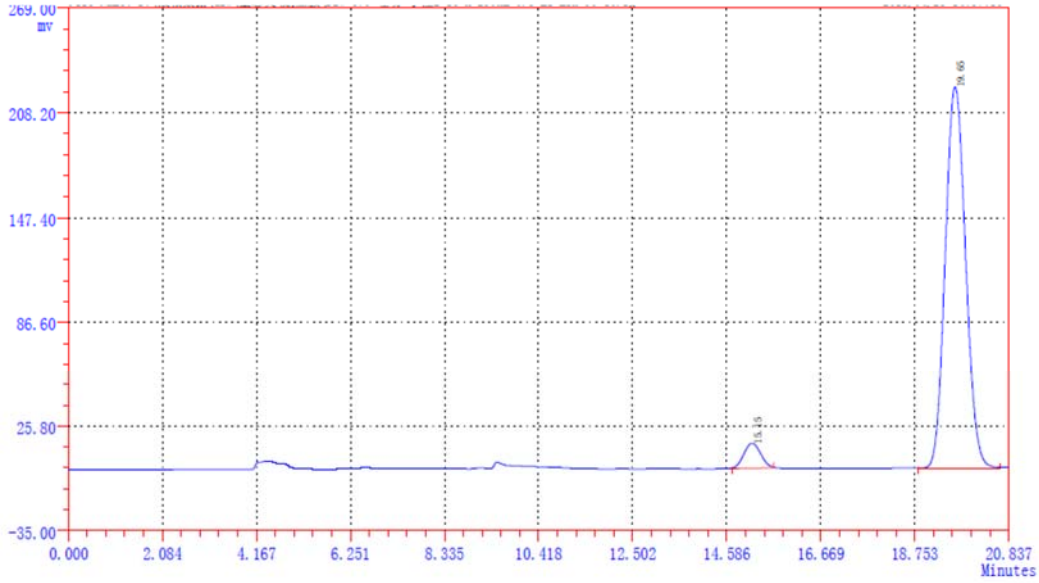


ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		15.310	389751	13378384.7	49.1117	1.25	3965
4		19.978	386422	13862349.0	50.8883	1.18	6181
	Σ:		776173	27240733.8	100.0000		

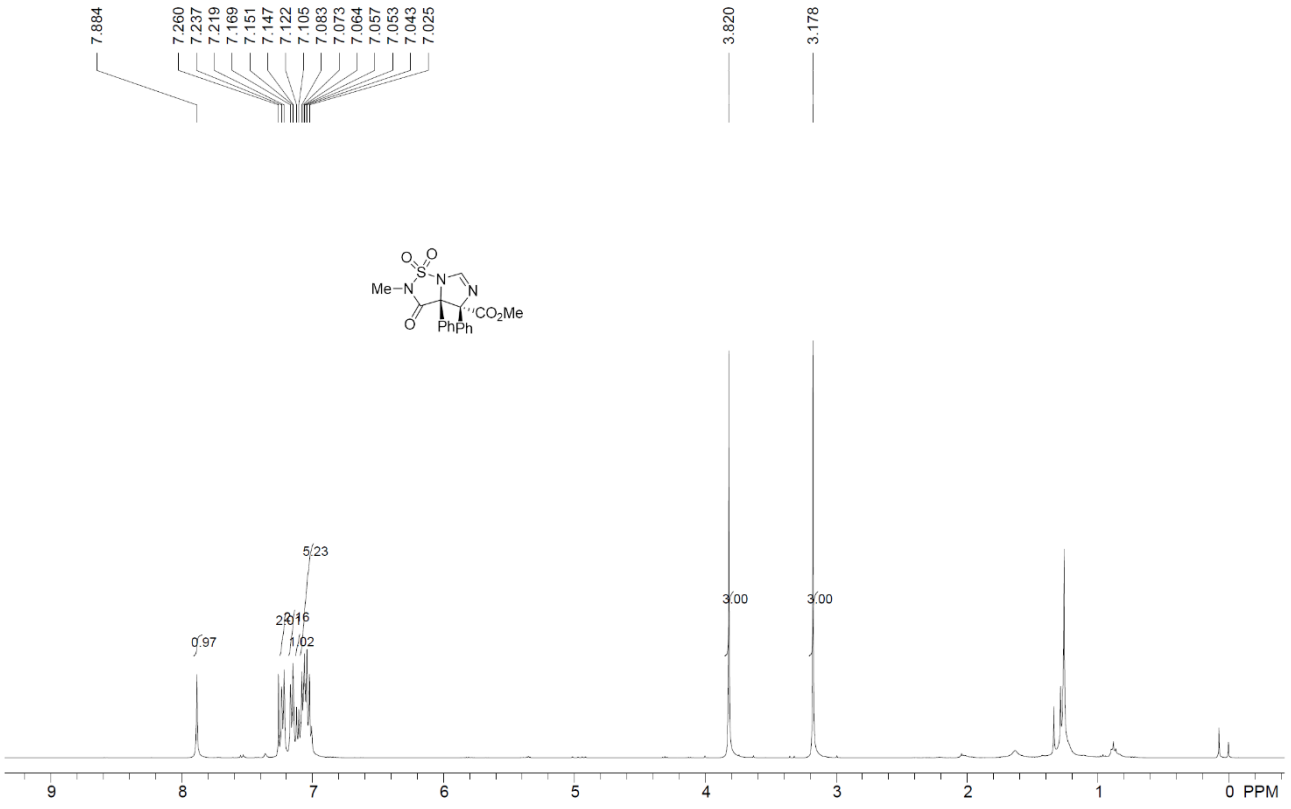
Chiral



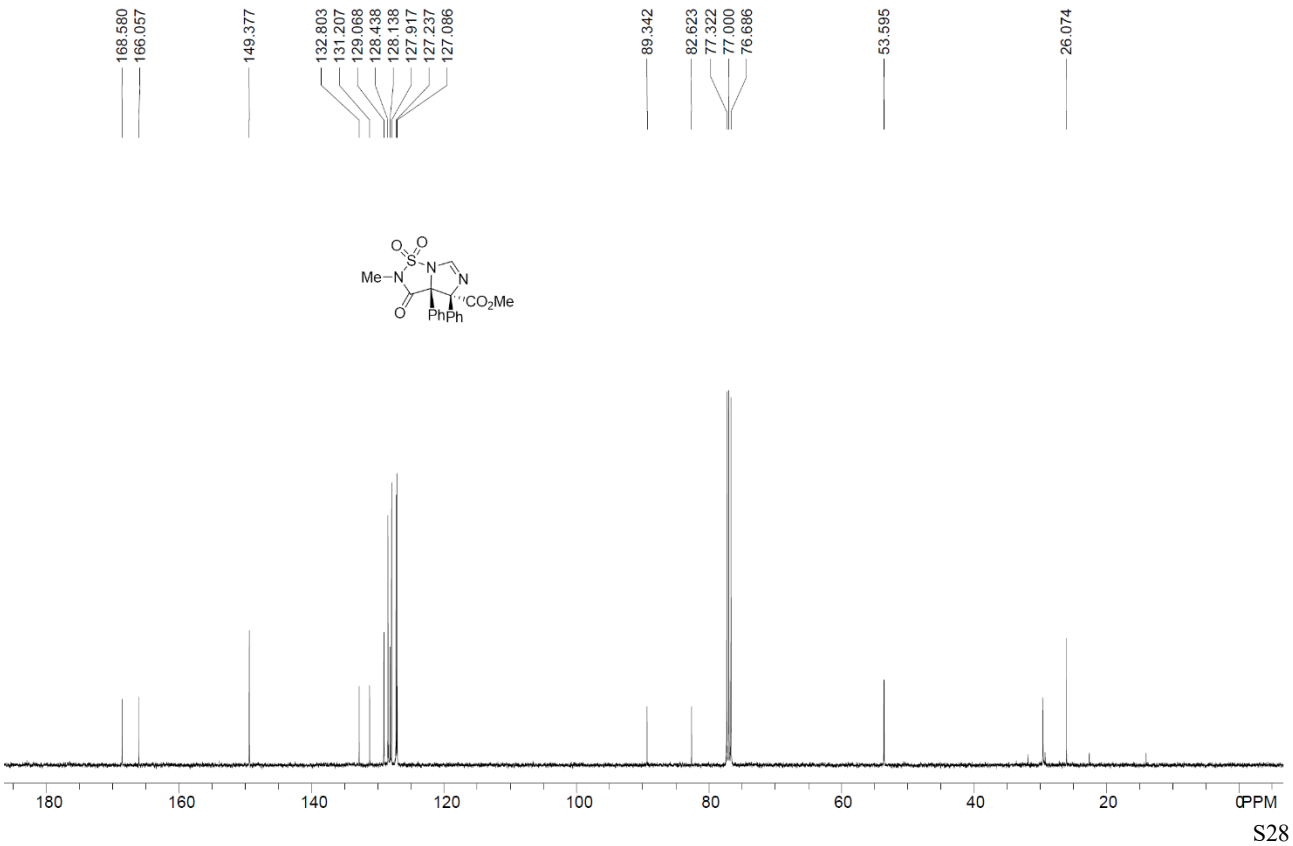
ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		15.153	14512	377728.0	5.1368	1.08	6755
2		19.648	222043	6975709.3	94.8632	1.10	7796
	Σ:		236555	7353437.3	100.0000		

3. Copies of NMR spectra for the compounds 4

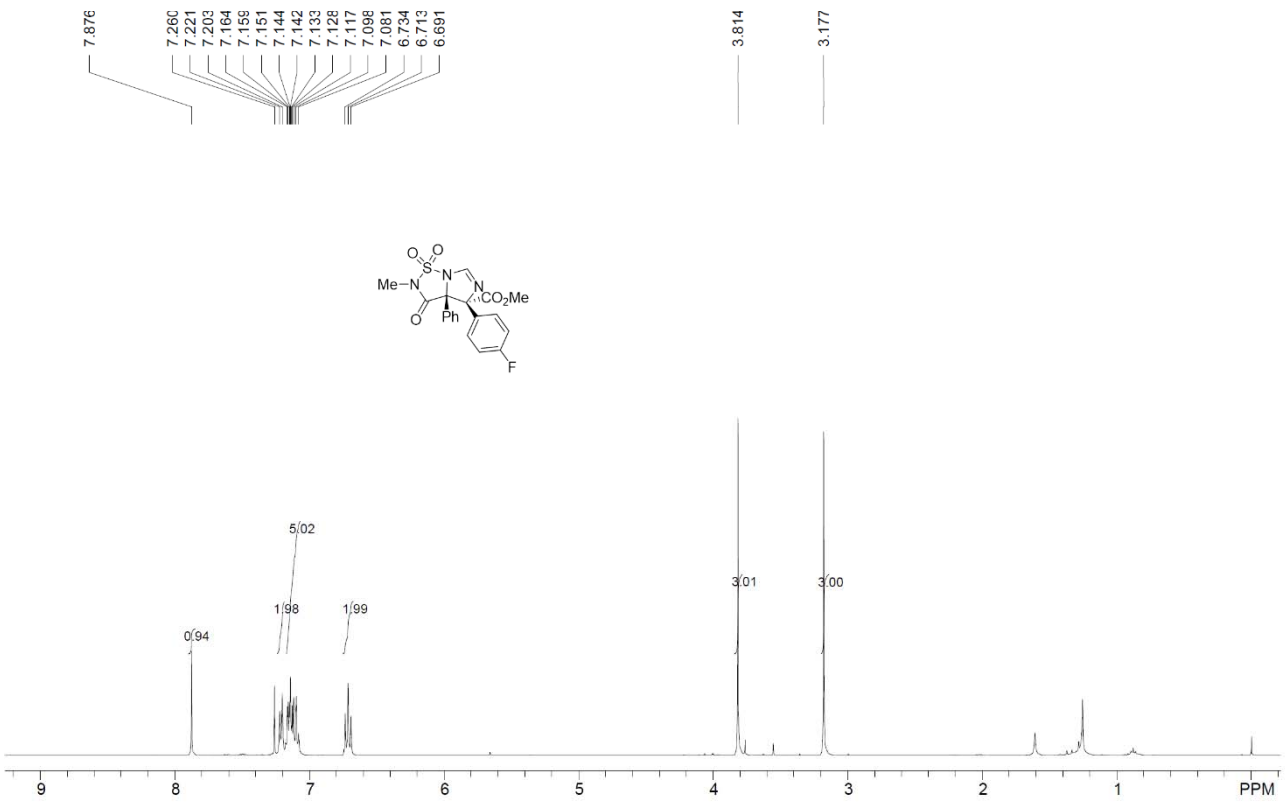
^{1}H NMR of **4a**



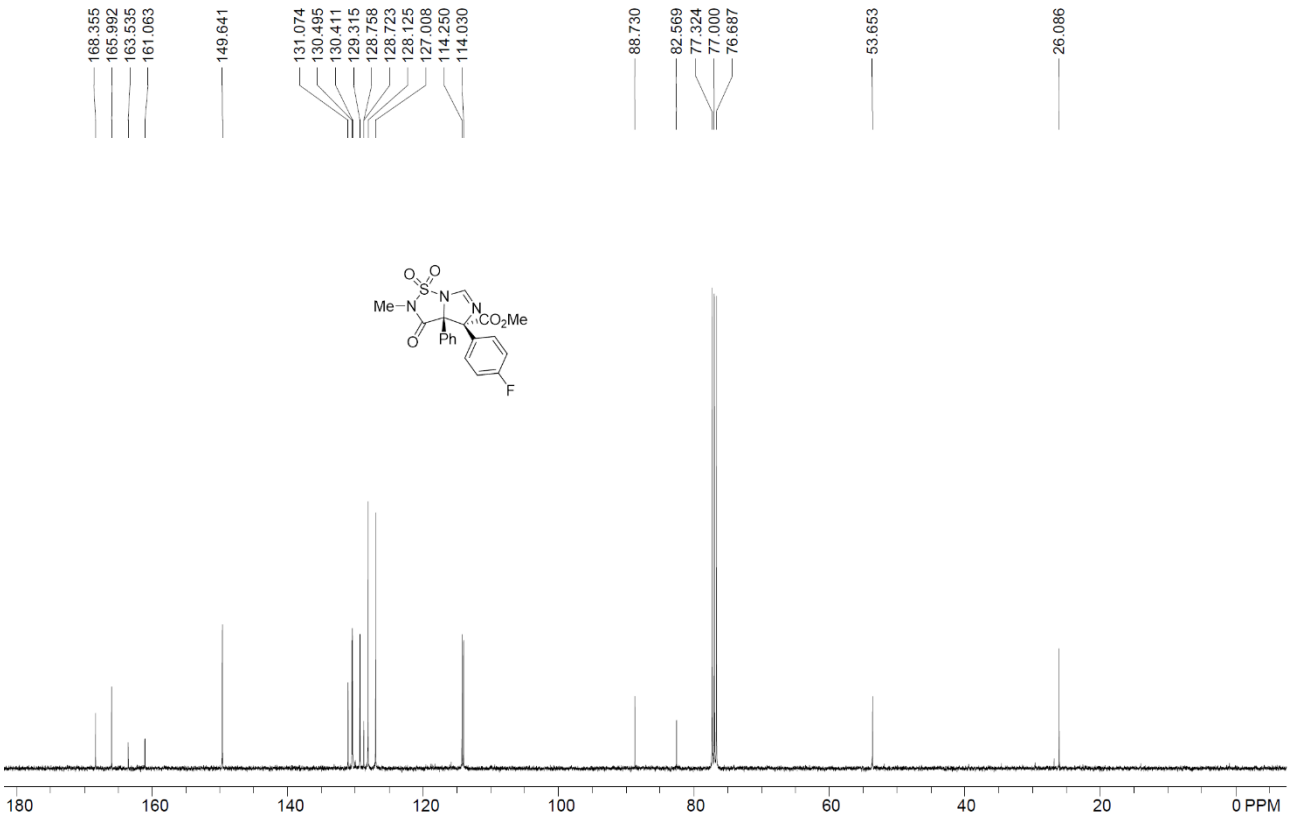
^{13}C NMR of **4a**



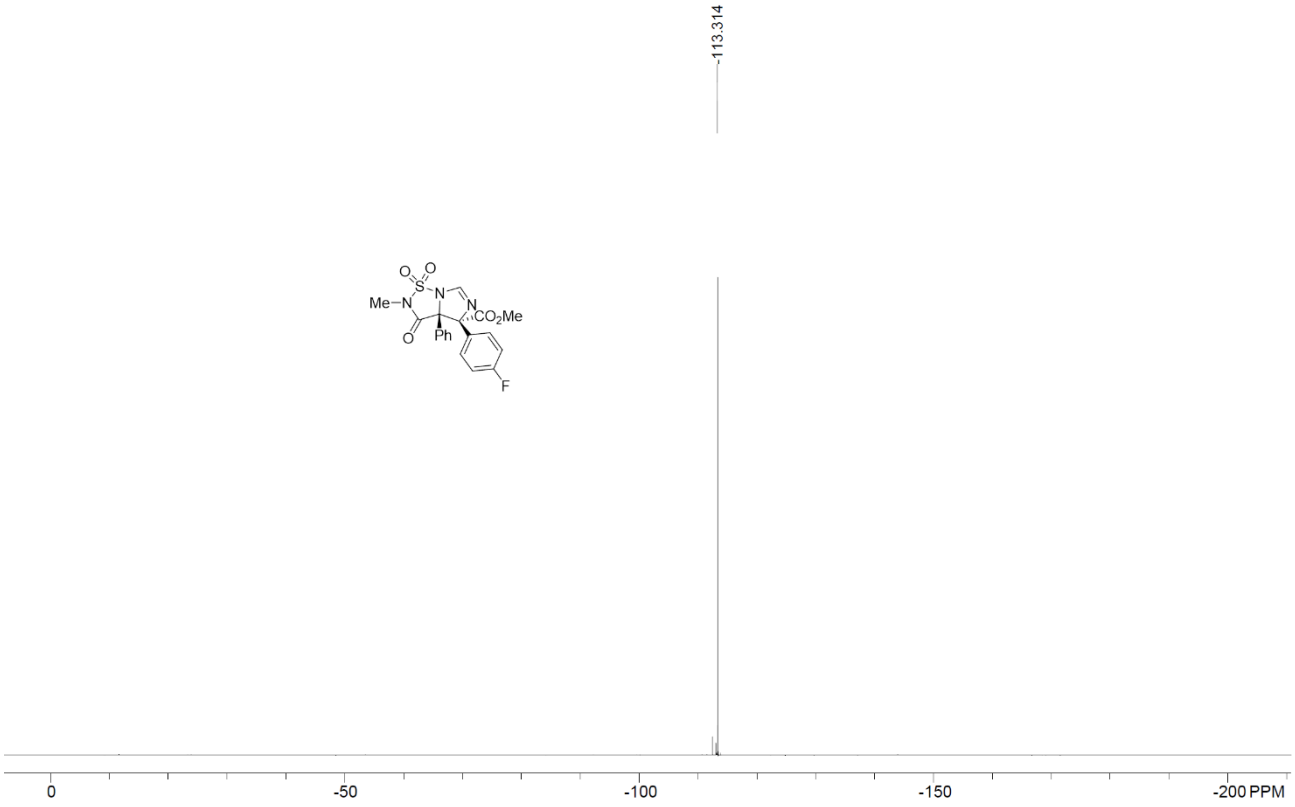
^{1}H NMR of **4b**



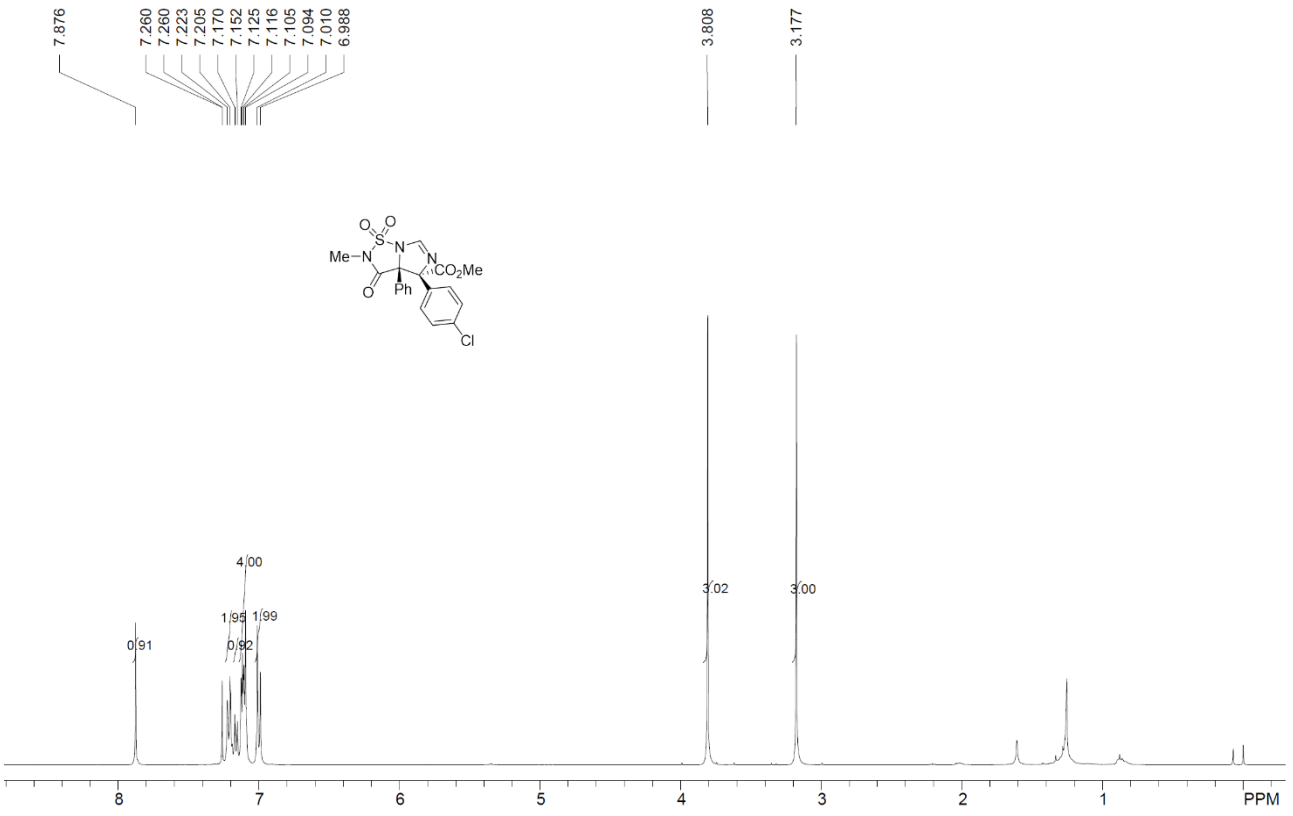
^{13}C NMR of **4b**



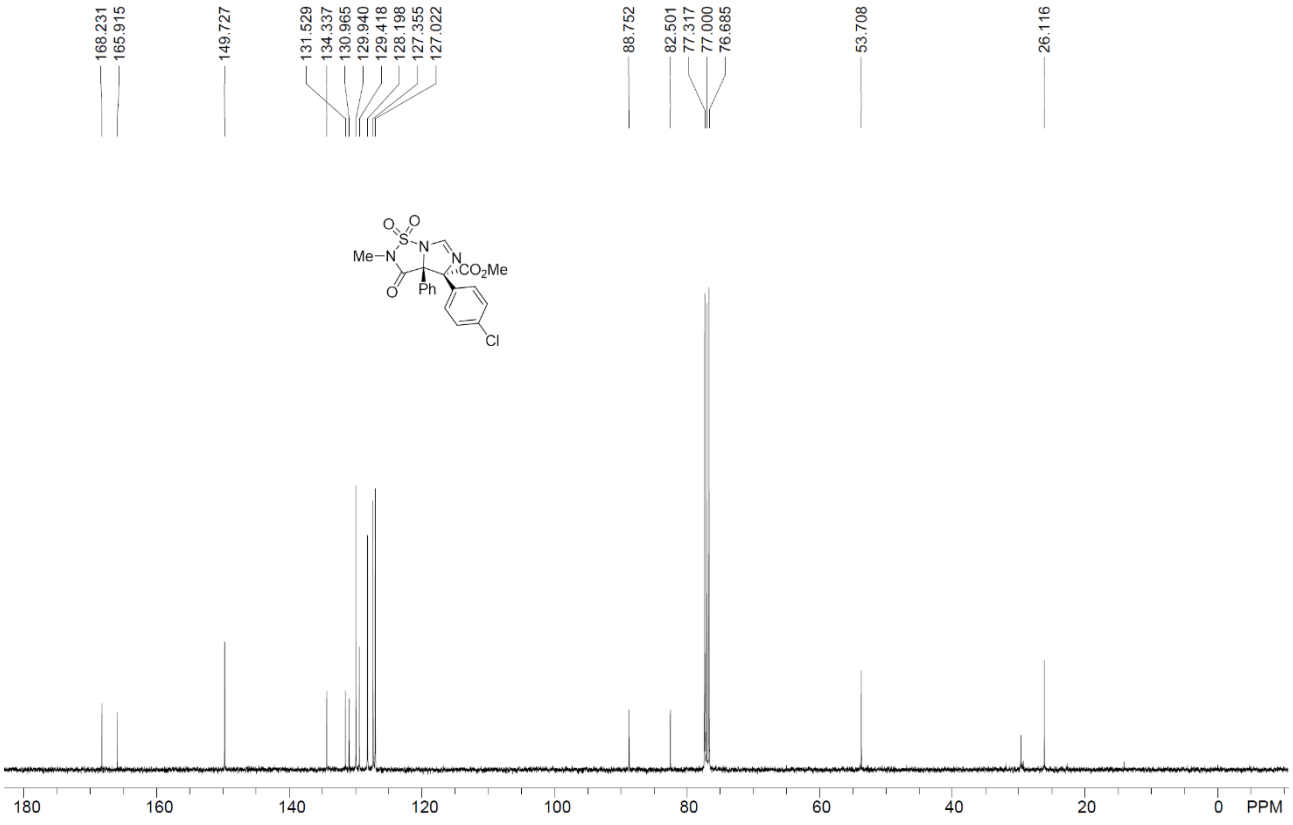
^{19}F NMR of **4b**



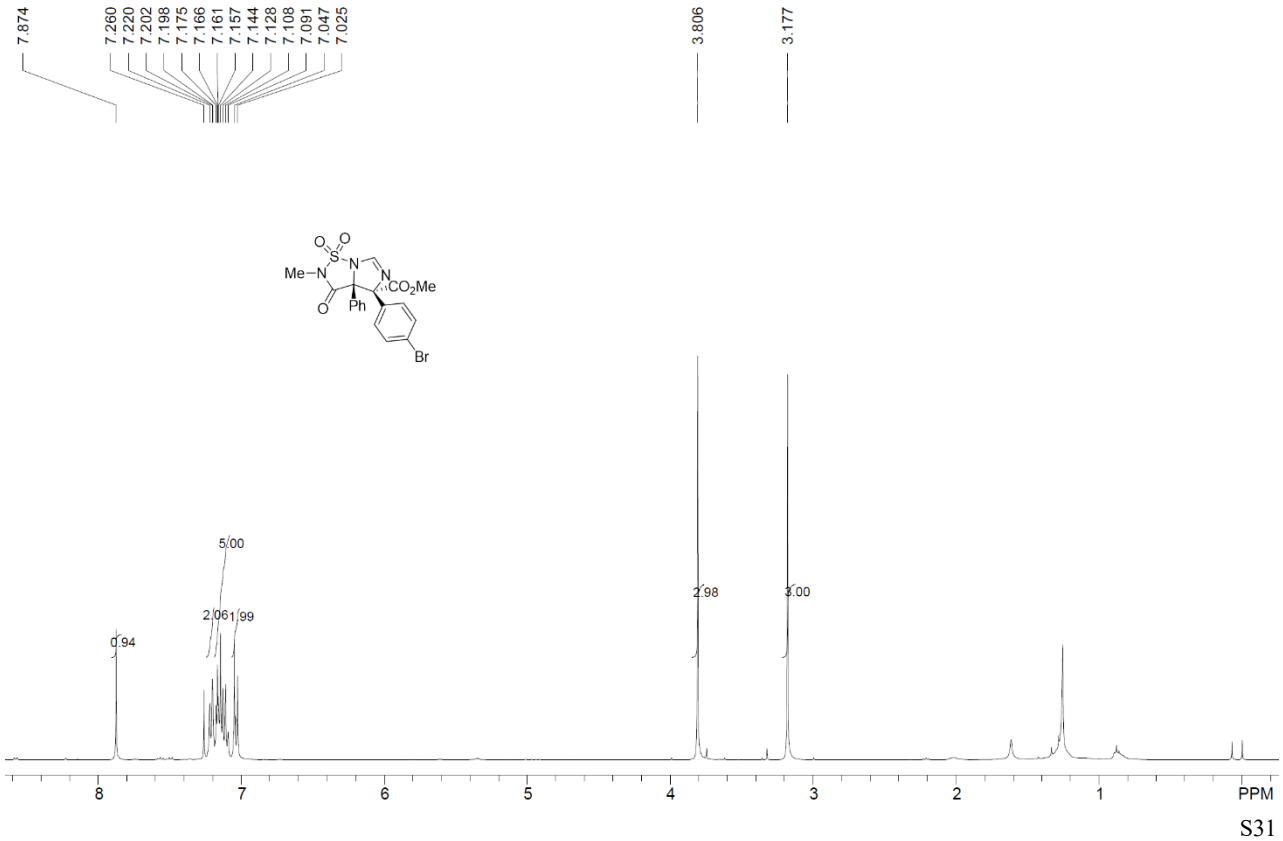
^{1}H NMR of **4c**



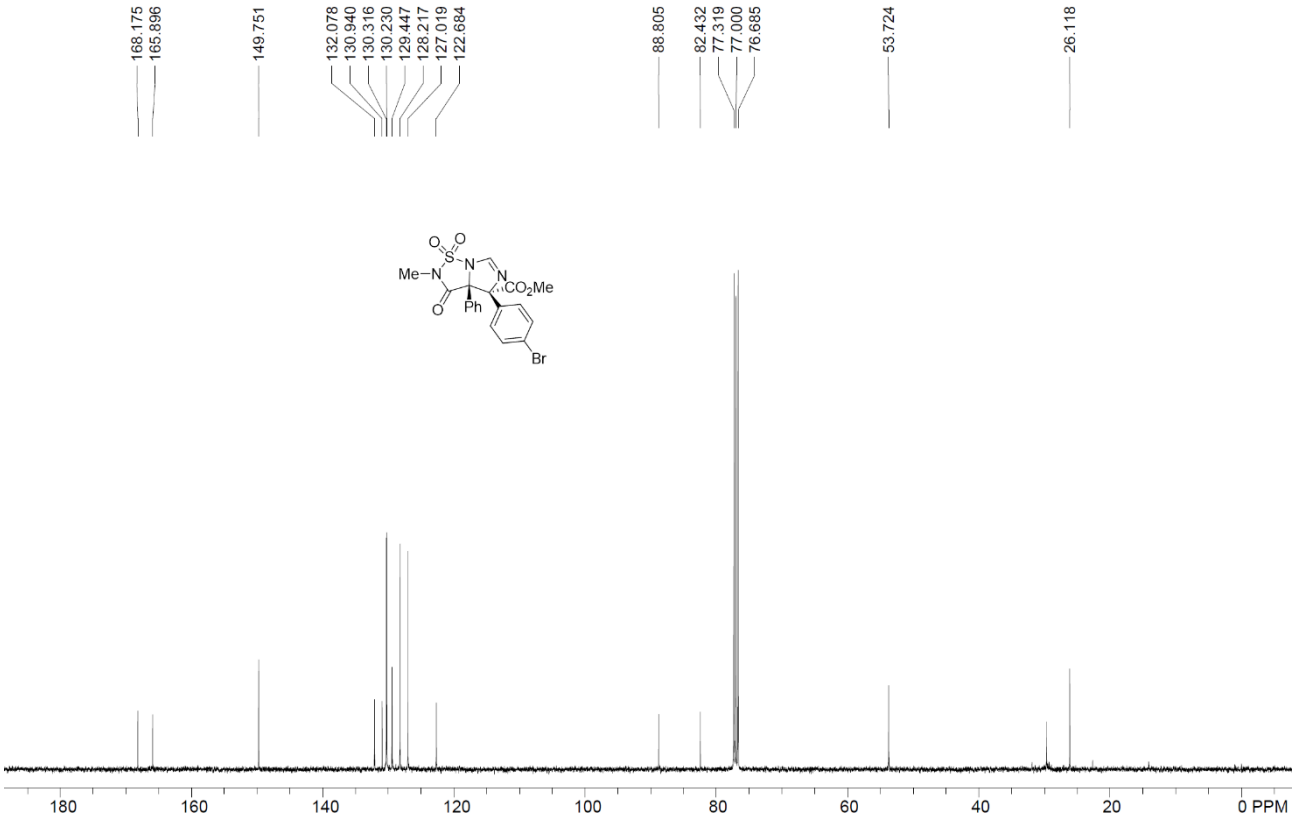
^{13}C NMR of **4c**



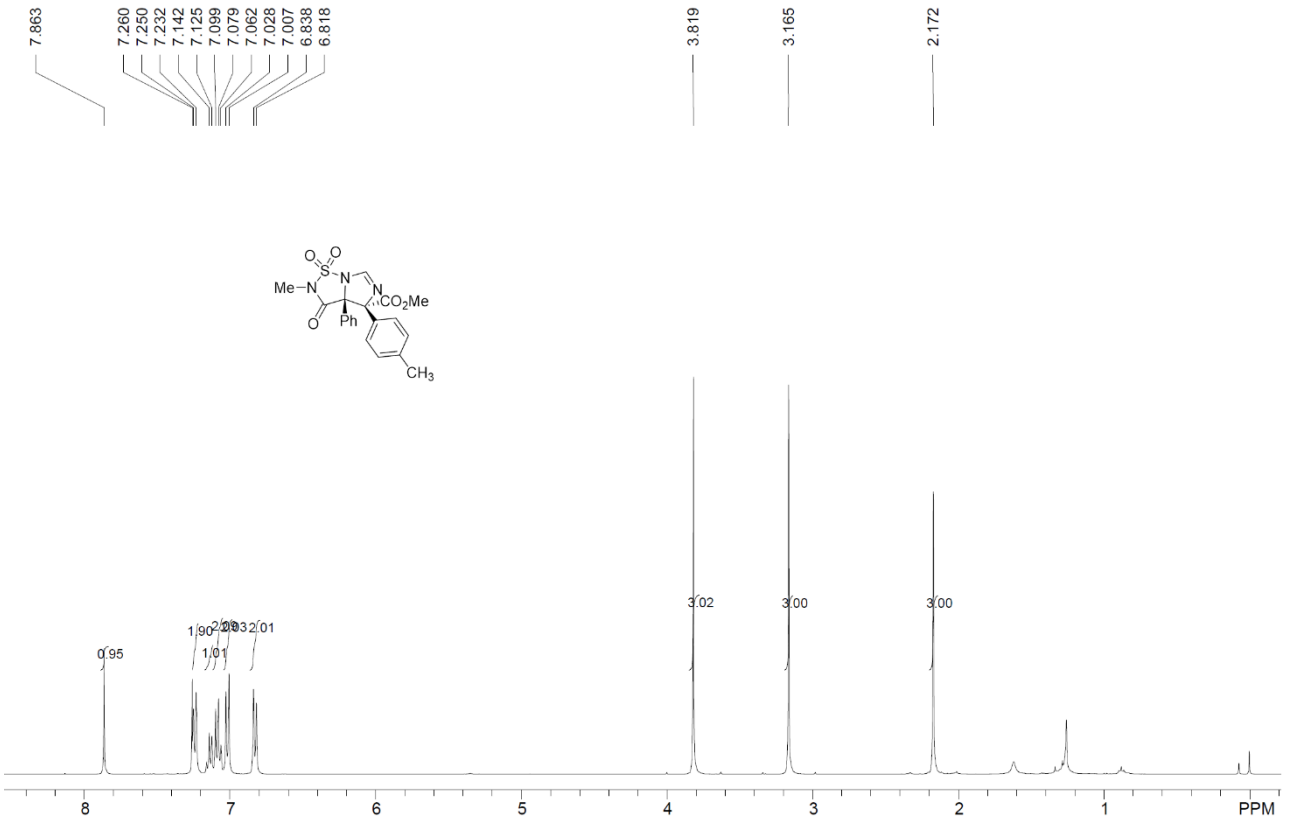
^{1}H NMR of **4d**



^{13}C NMR of **4d**



^{1}H NMR of **4e**



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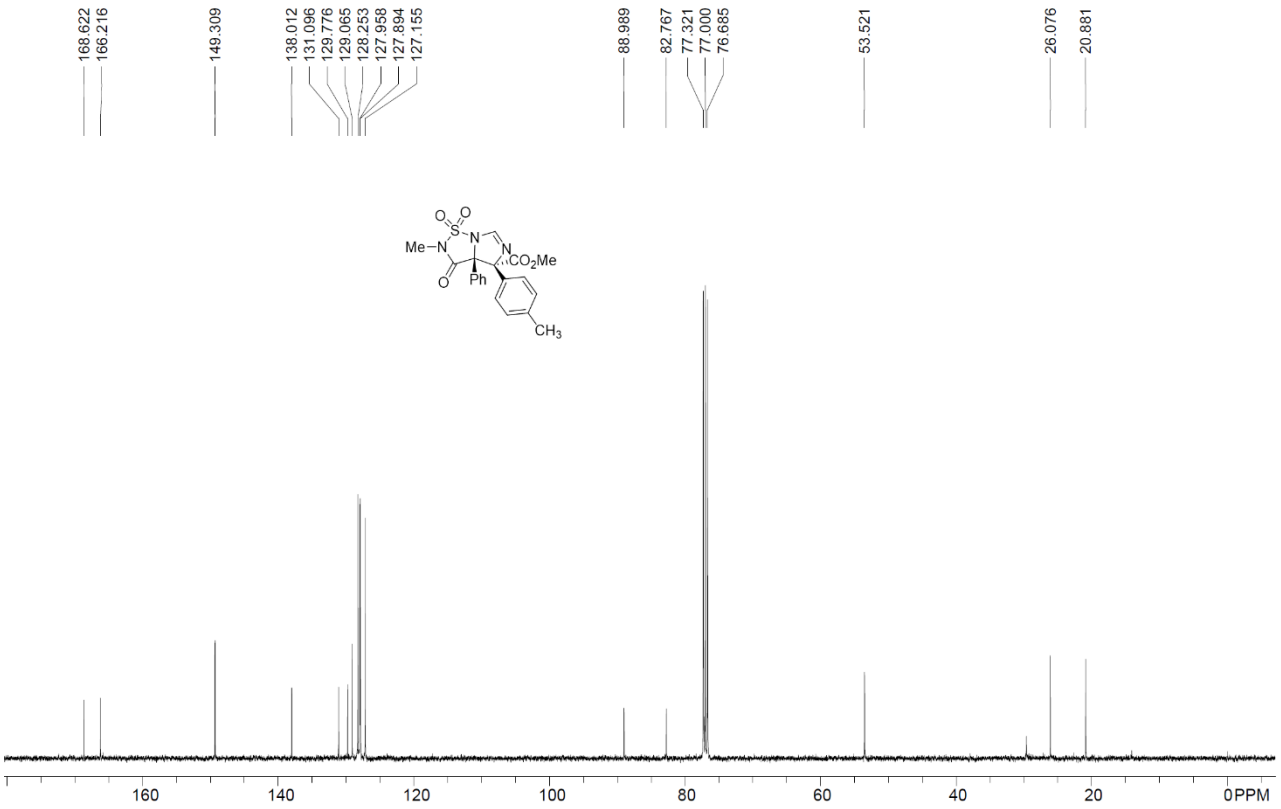
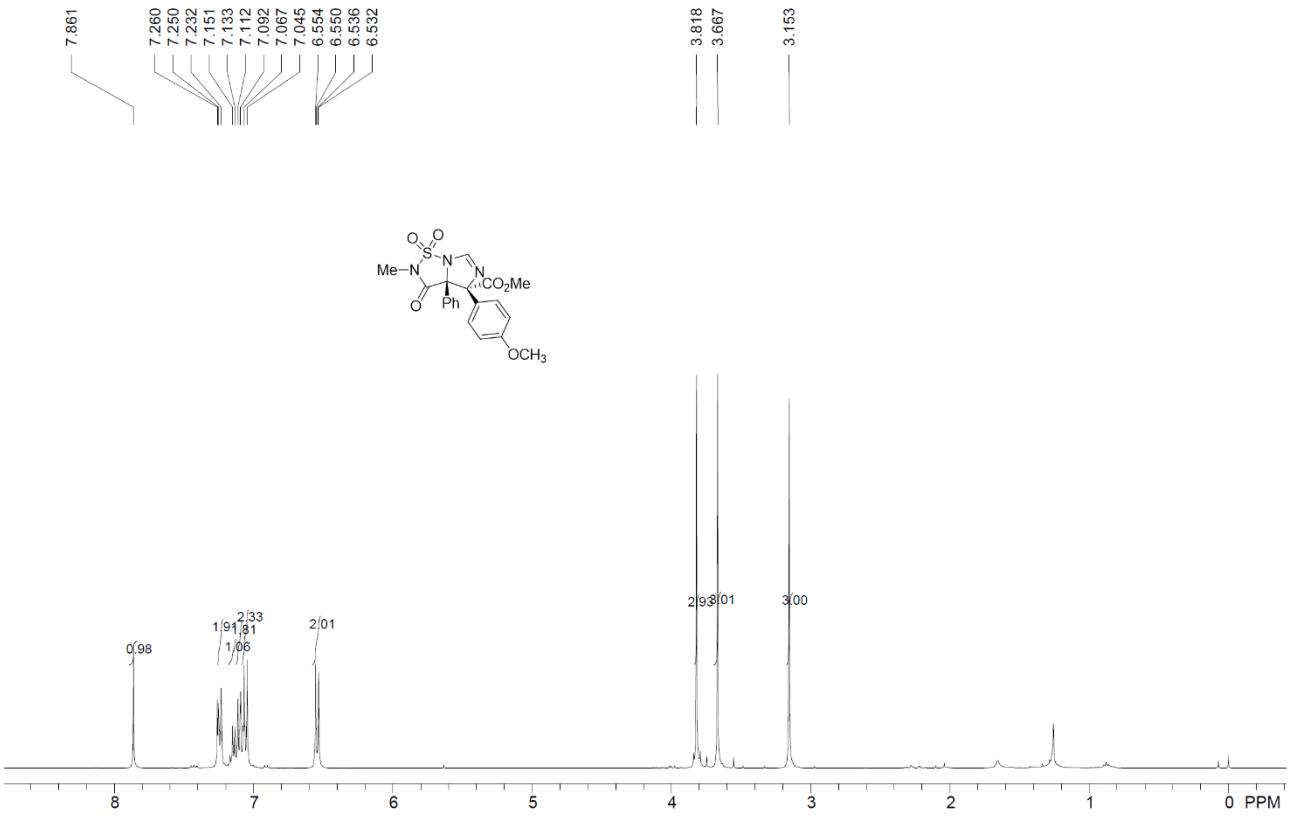
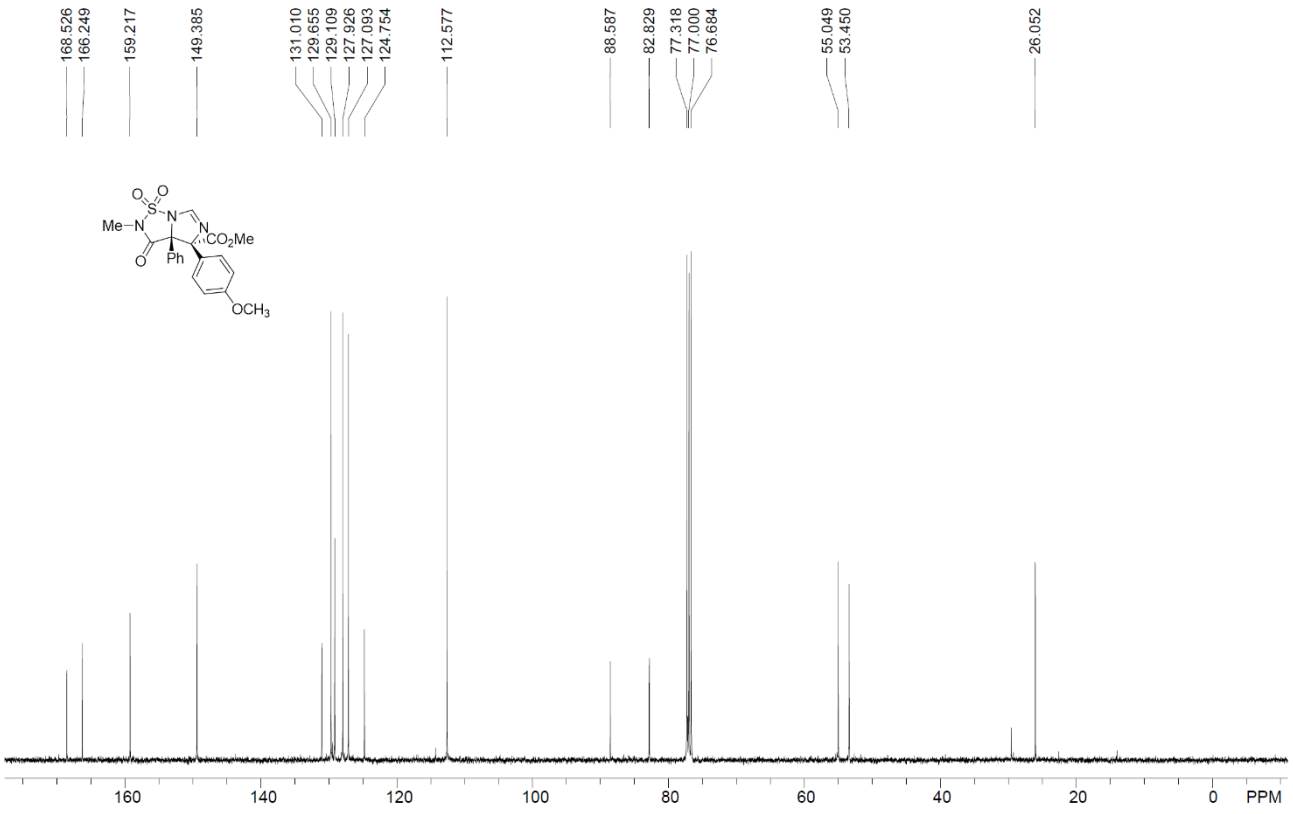


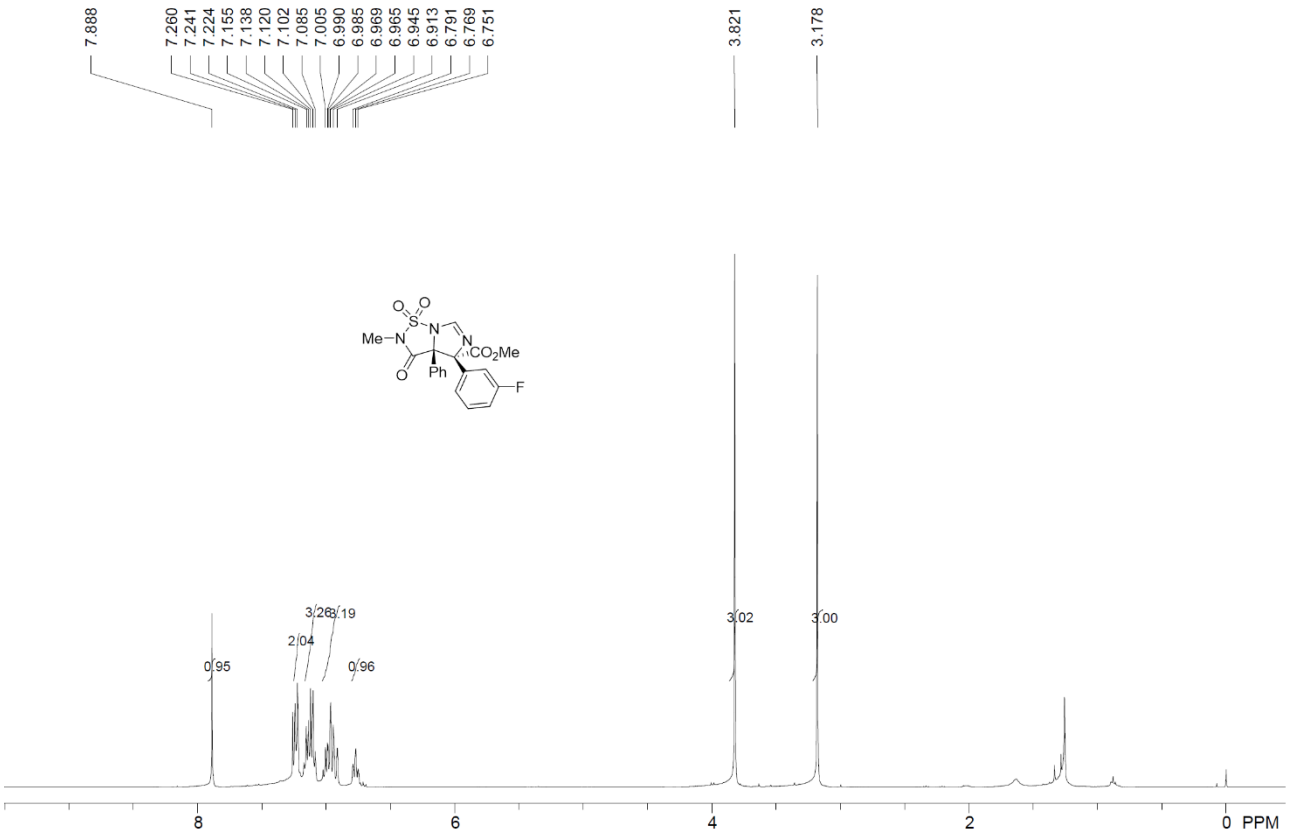
^{1}H NMR of **4f**



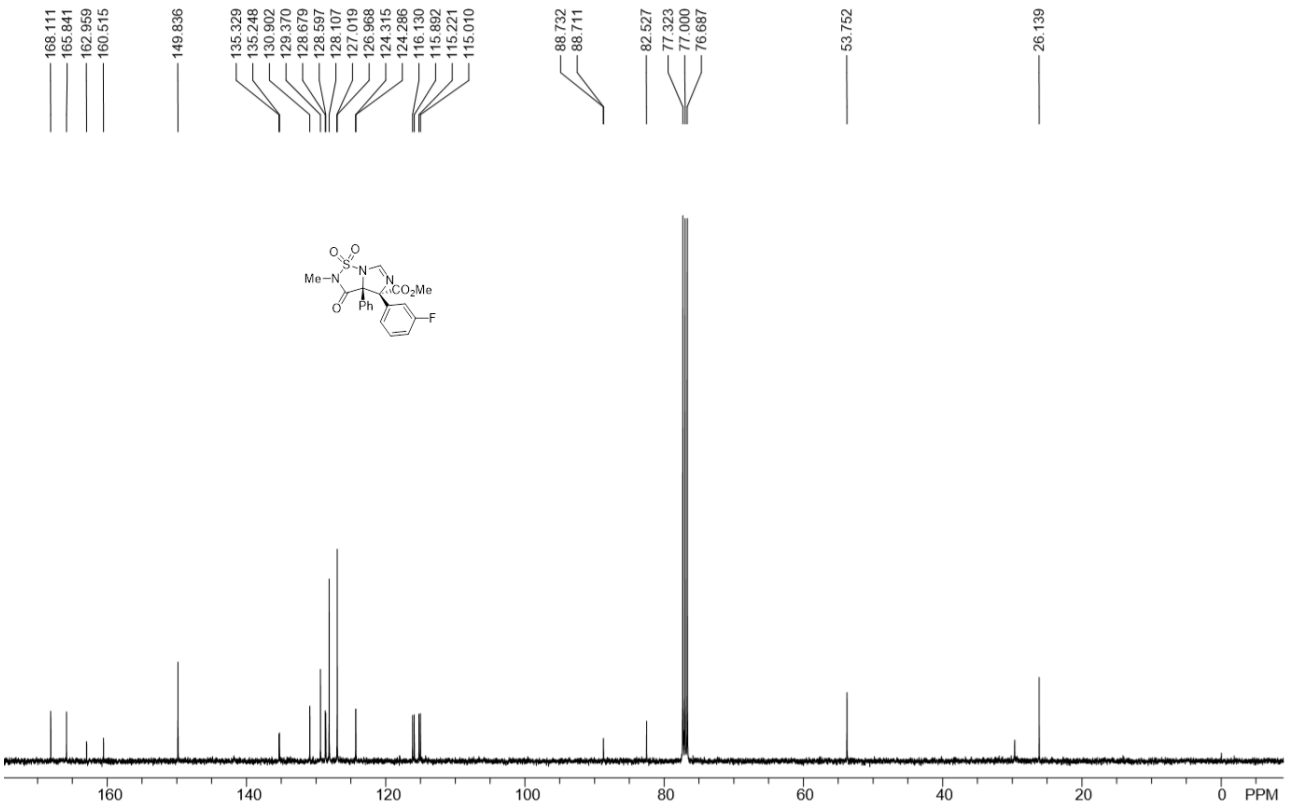
^{13}C NMR of **4f**



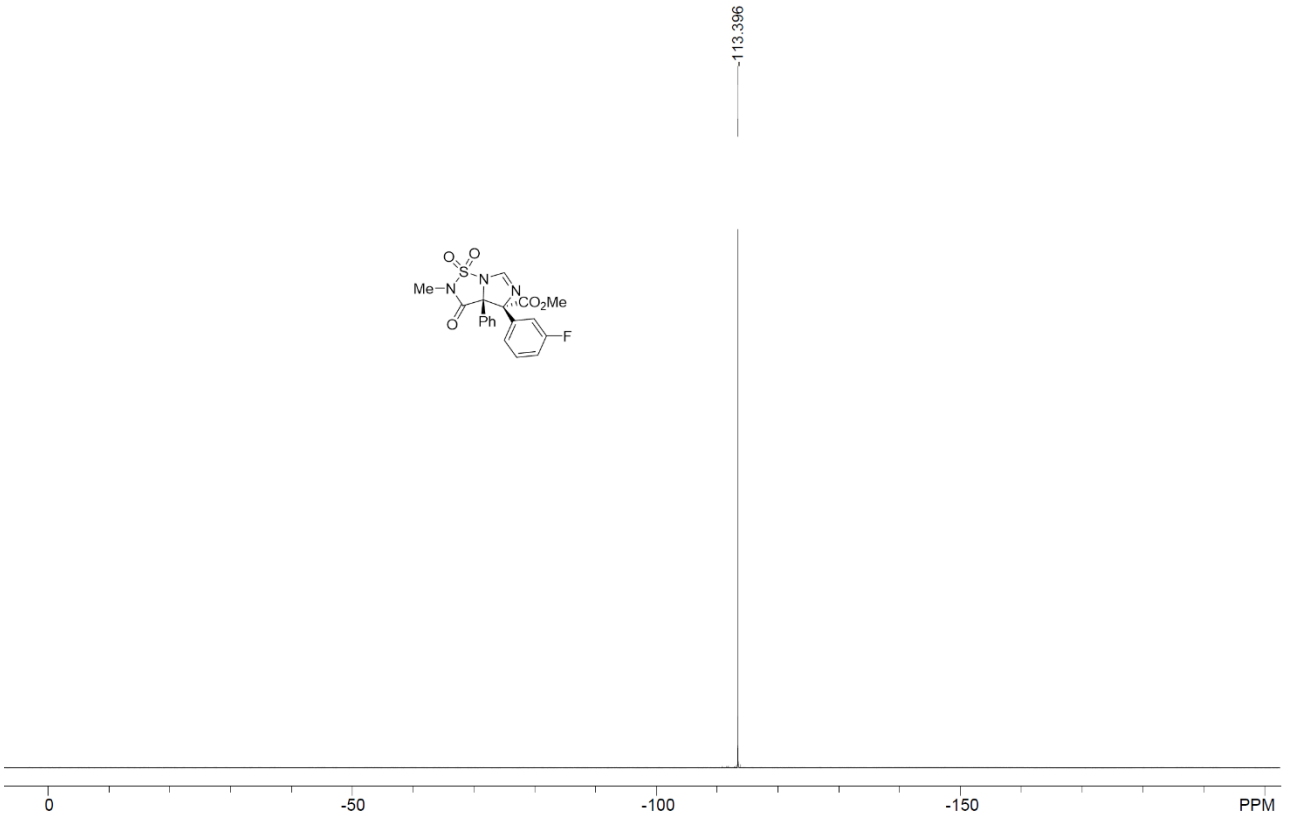
^{1}H NMR of **4g**



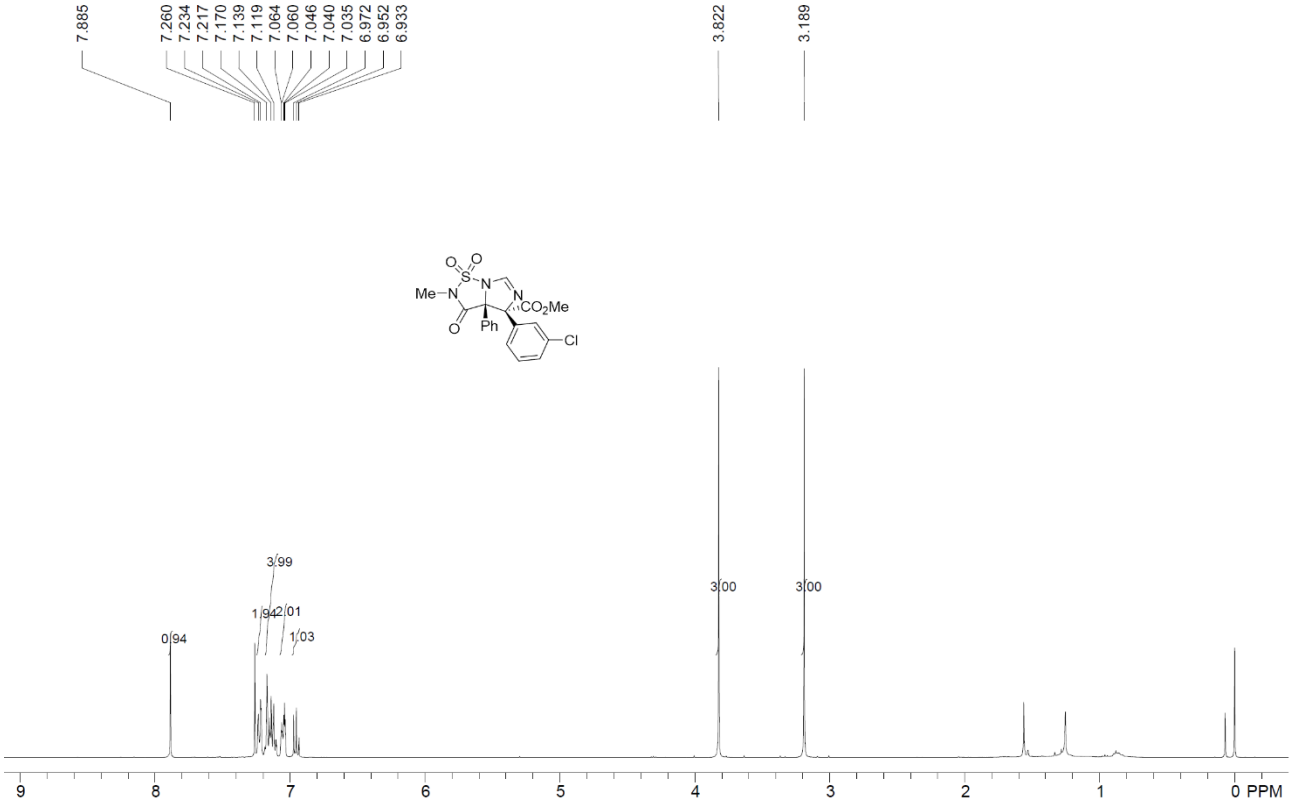
^{13}C NMR of **4g**



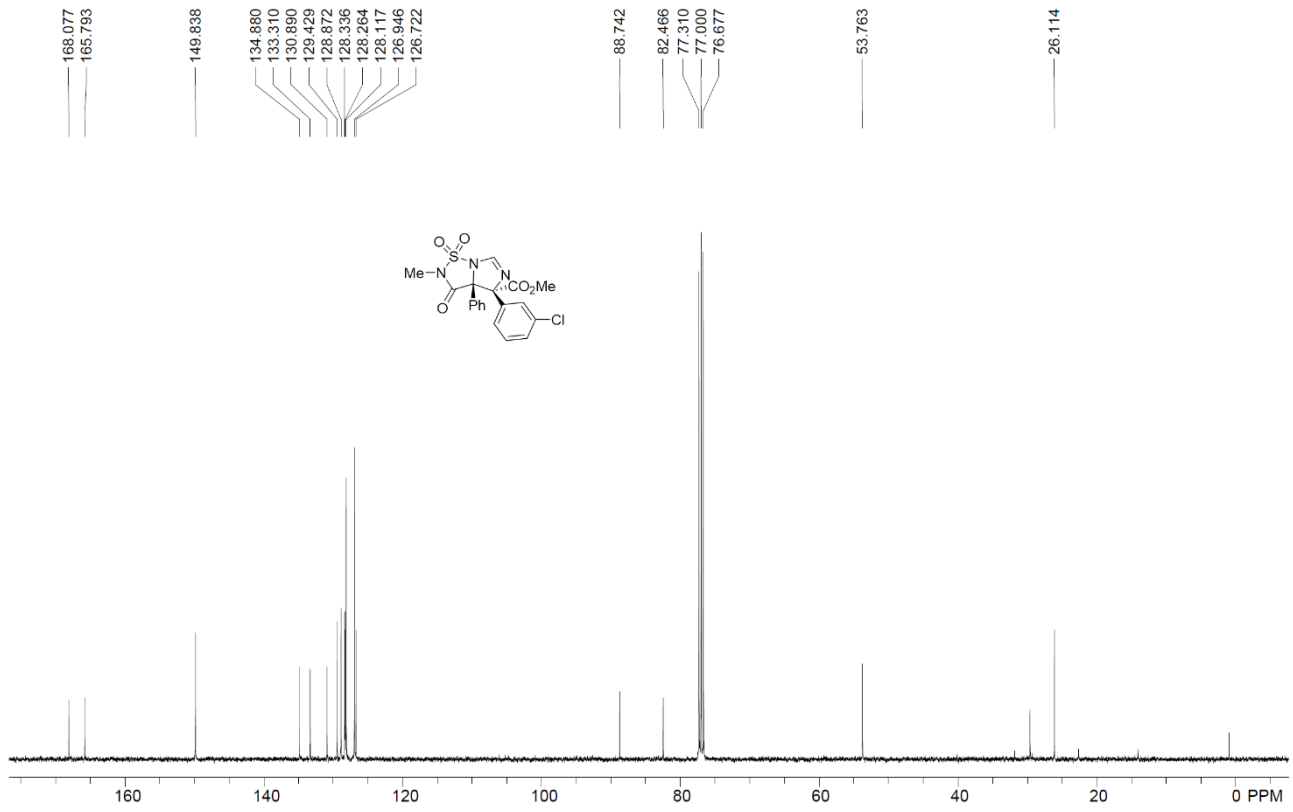
^{19}F NMR of **4g**



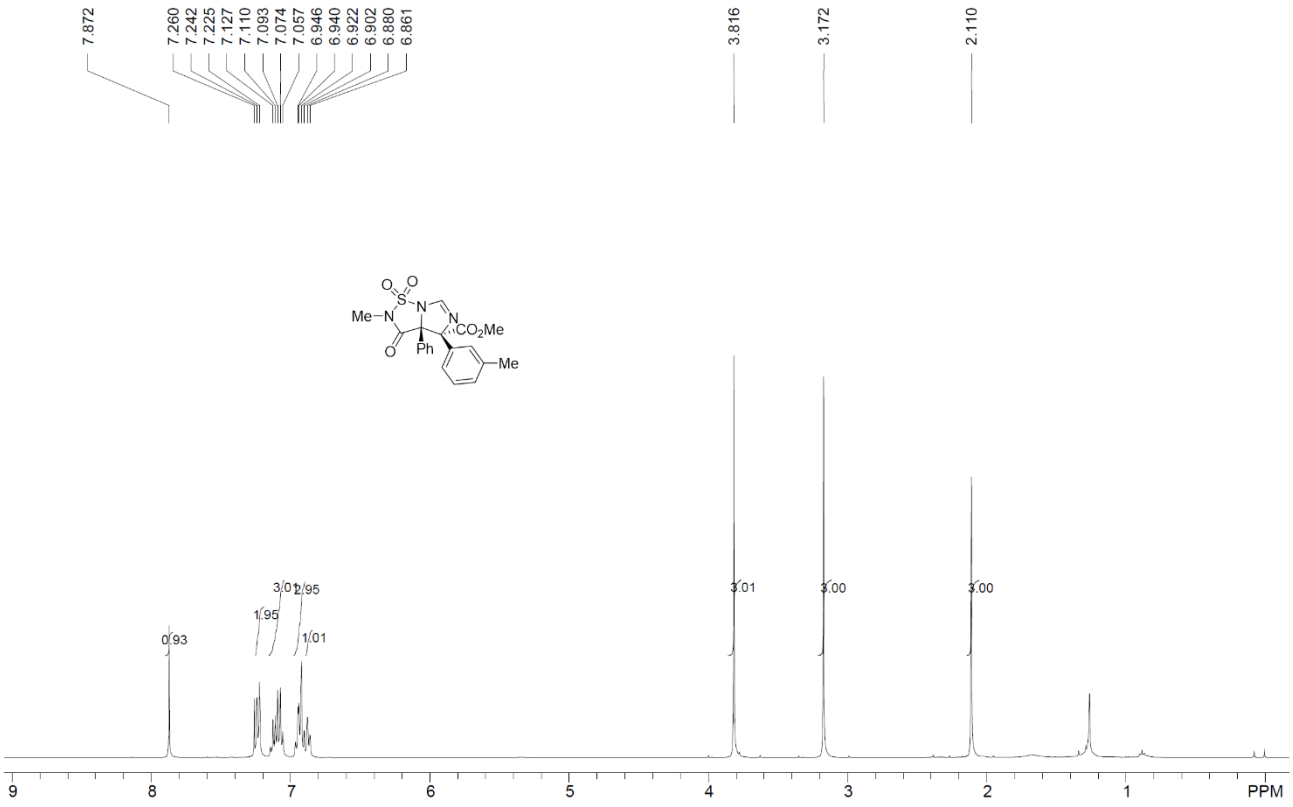
^{1}H NMR of **4h**



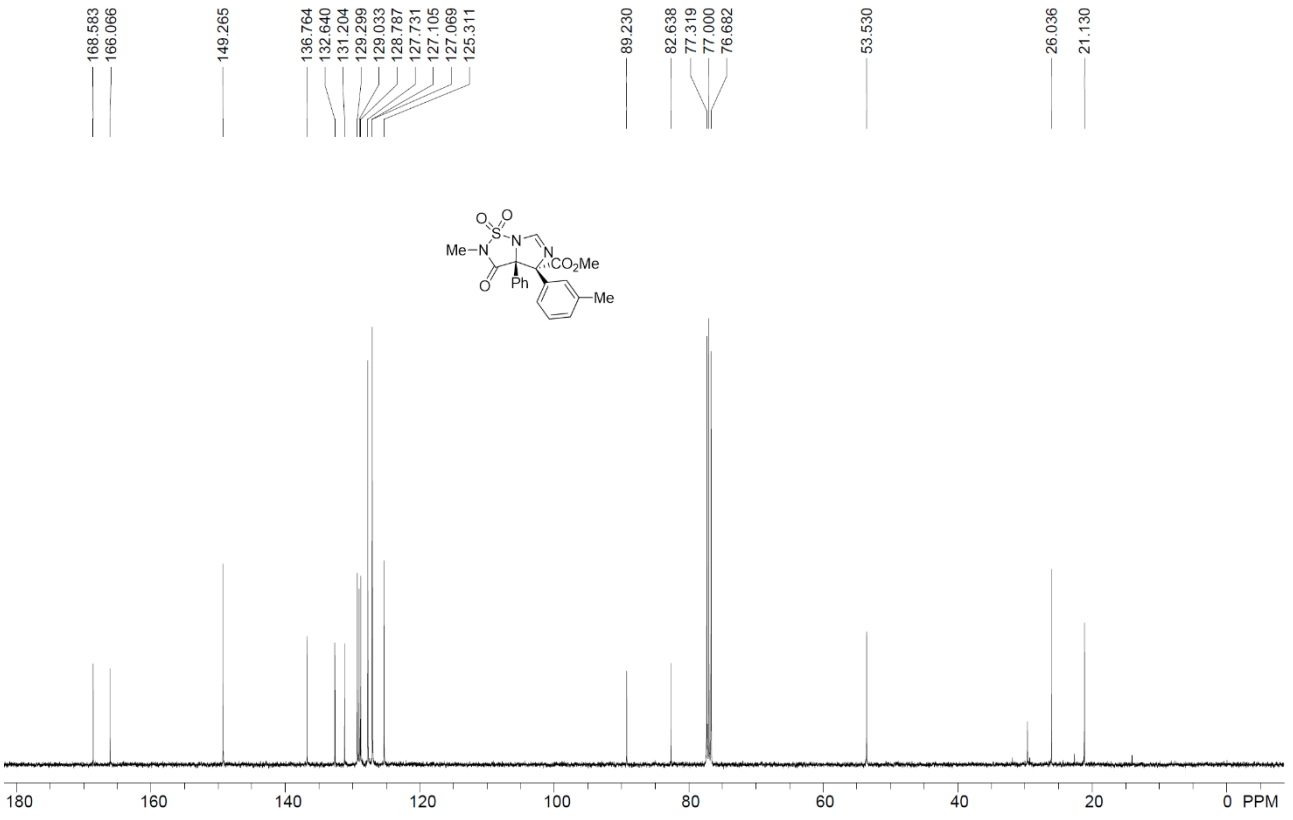
^{13}C NMR of **4h**



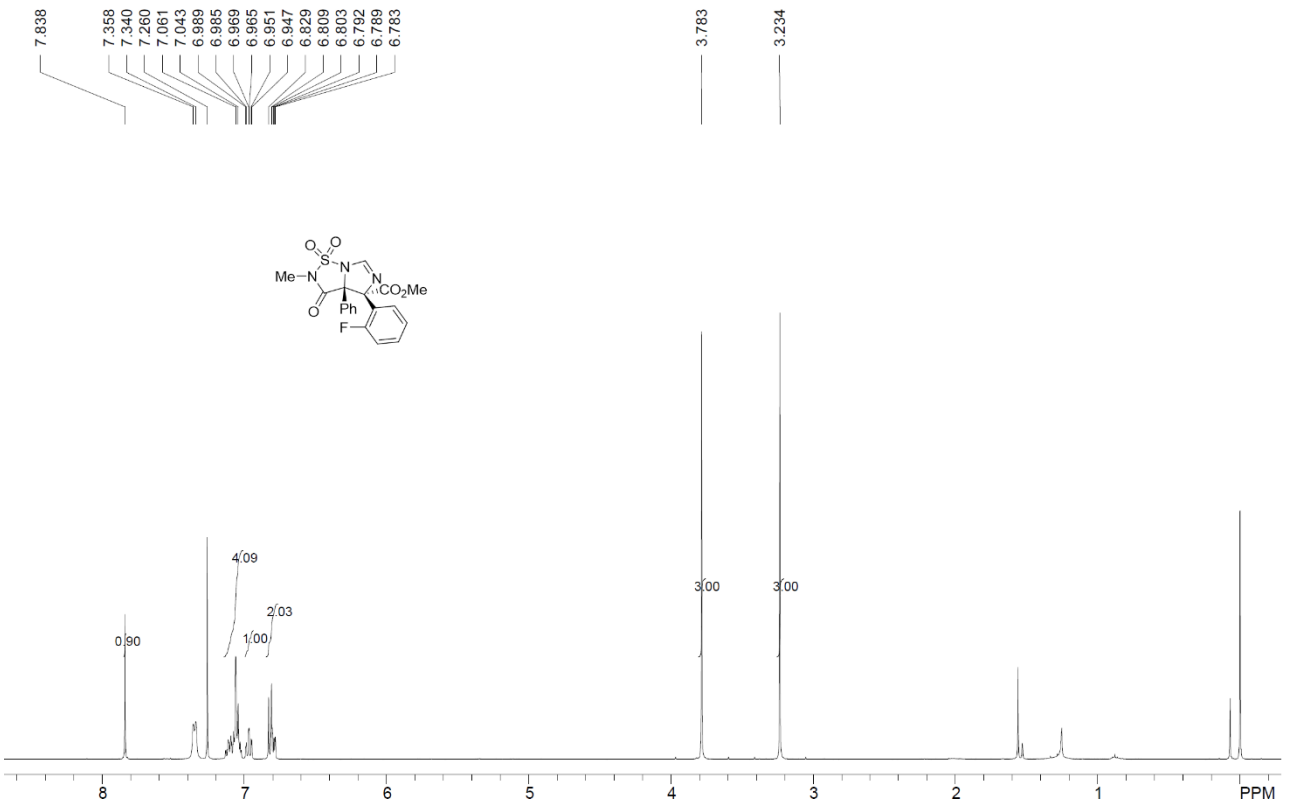
^{1}H NMR of **4i**



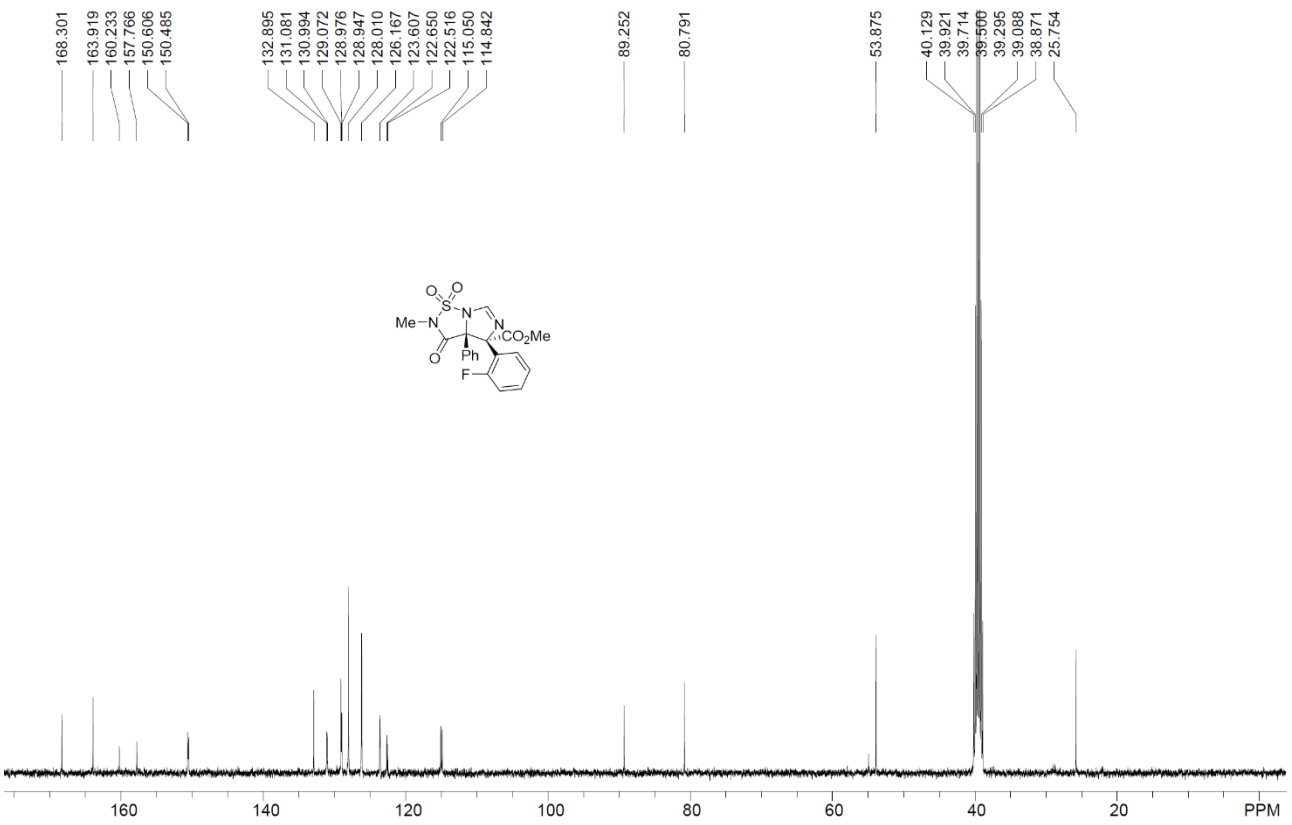
^{13}C NMR of **4i**



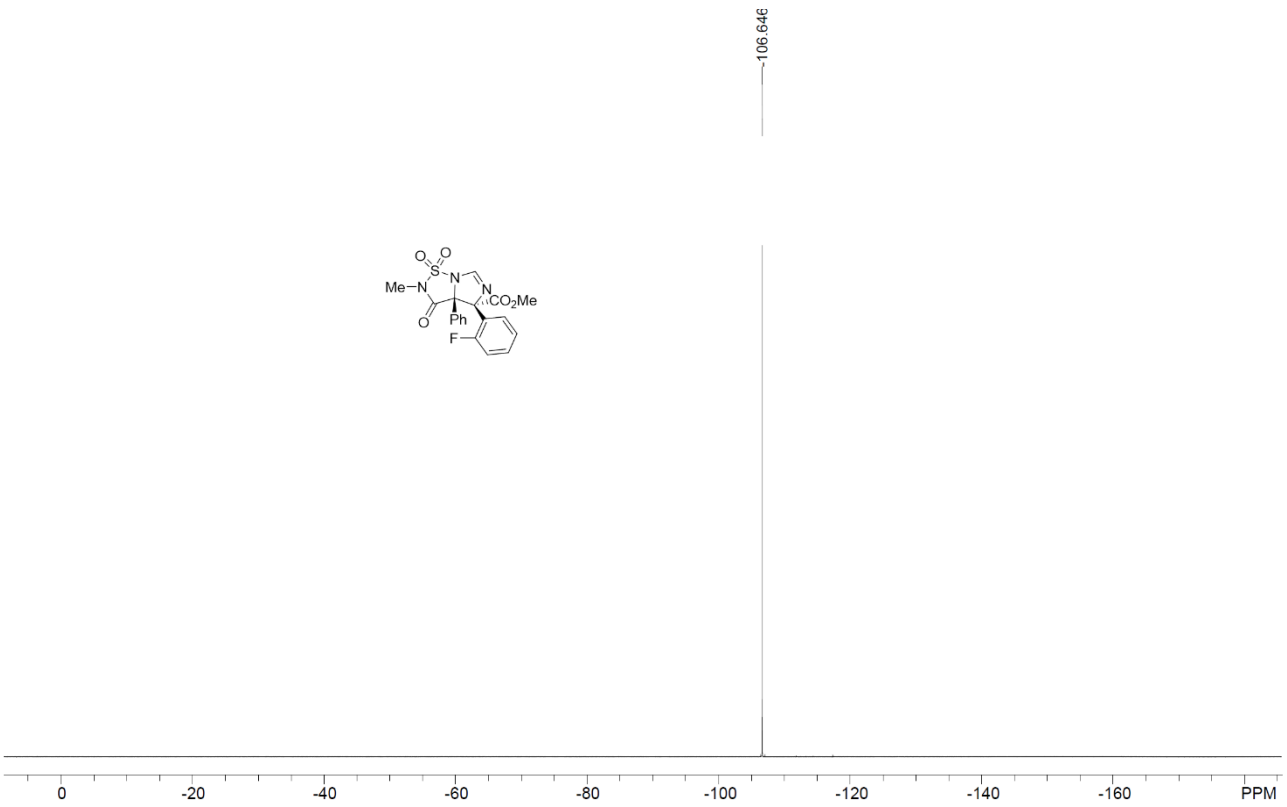
^{1}H NMR of **4j**



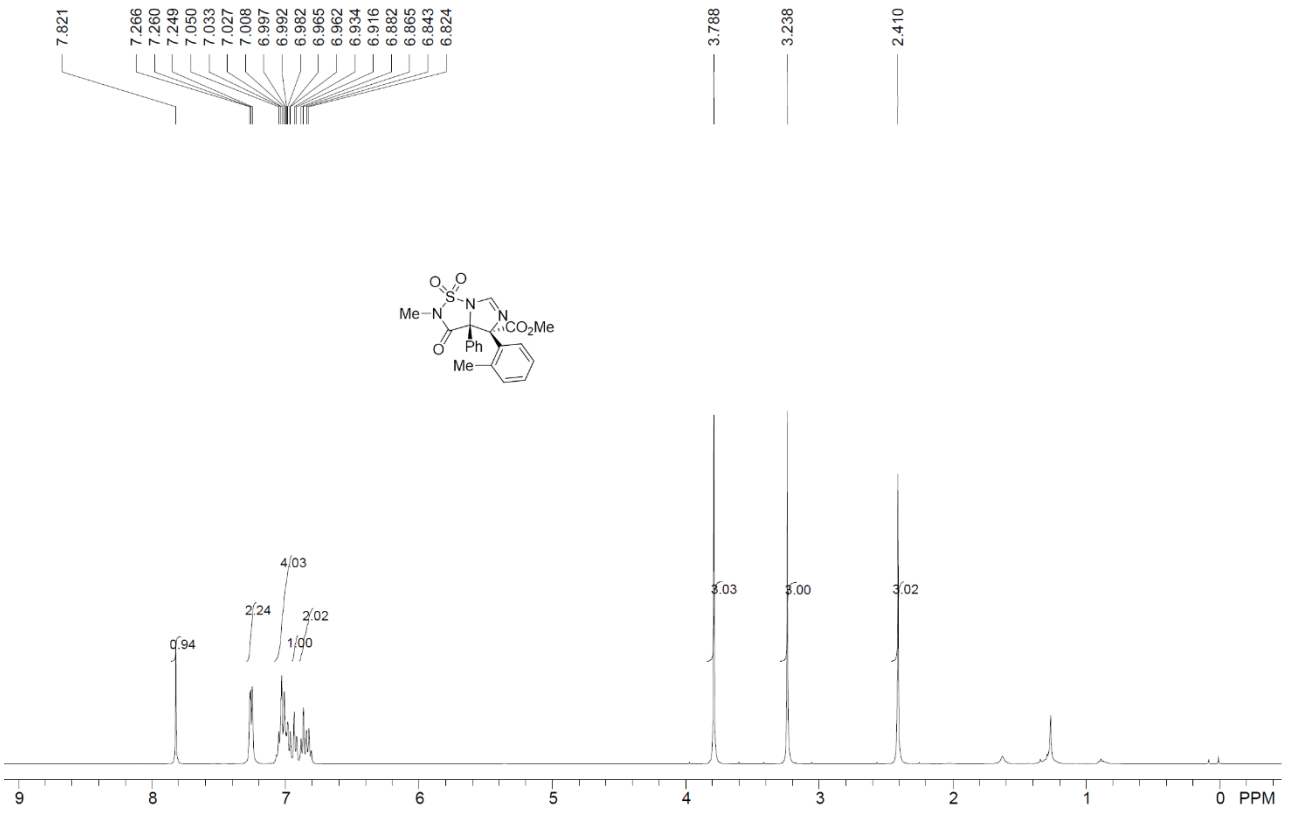
^{13}C NMR of **4j**



^{19}F NMR of **4j**



^{1}H NMR of **4k**



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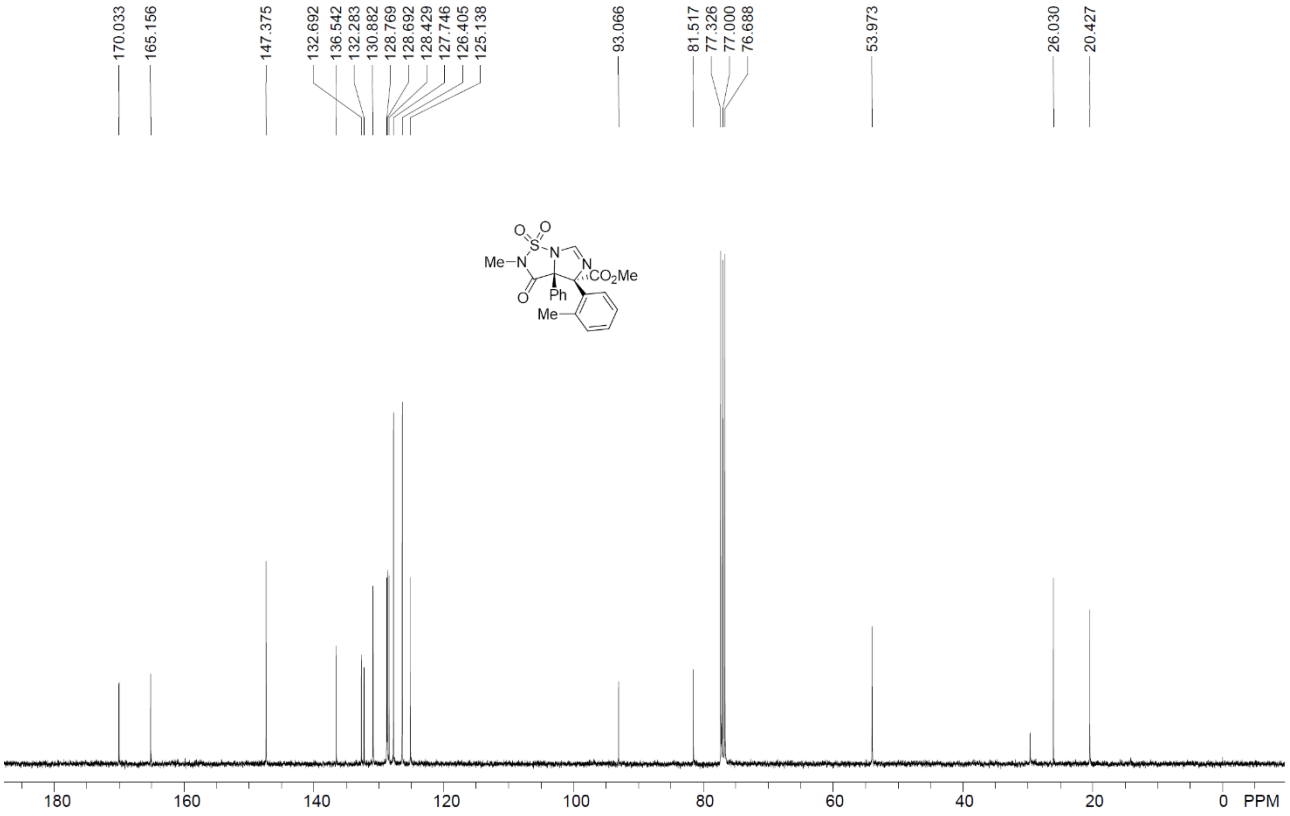
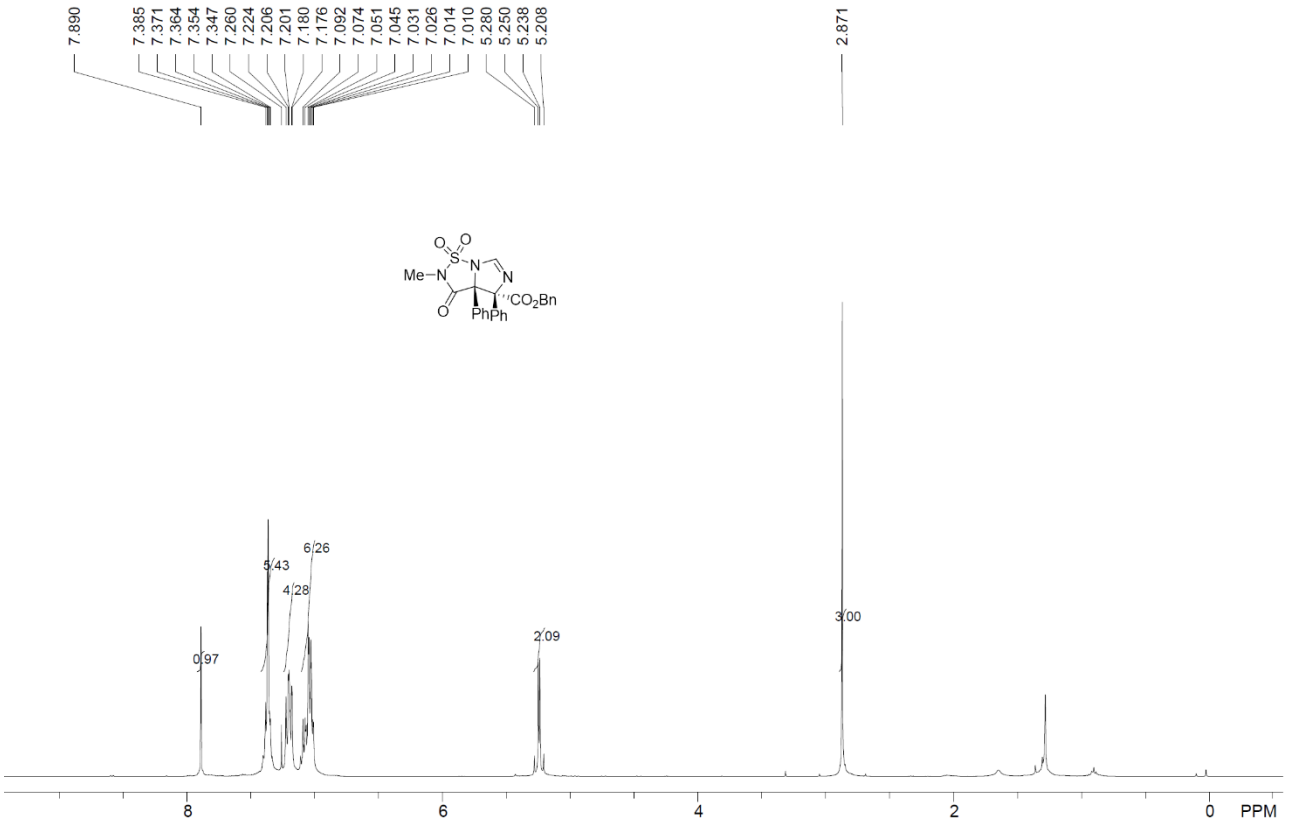
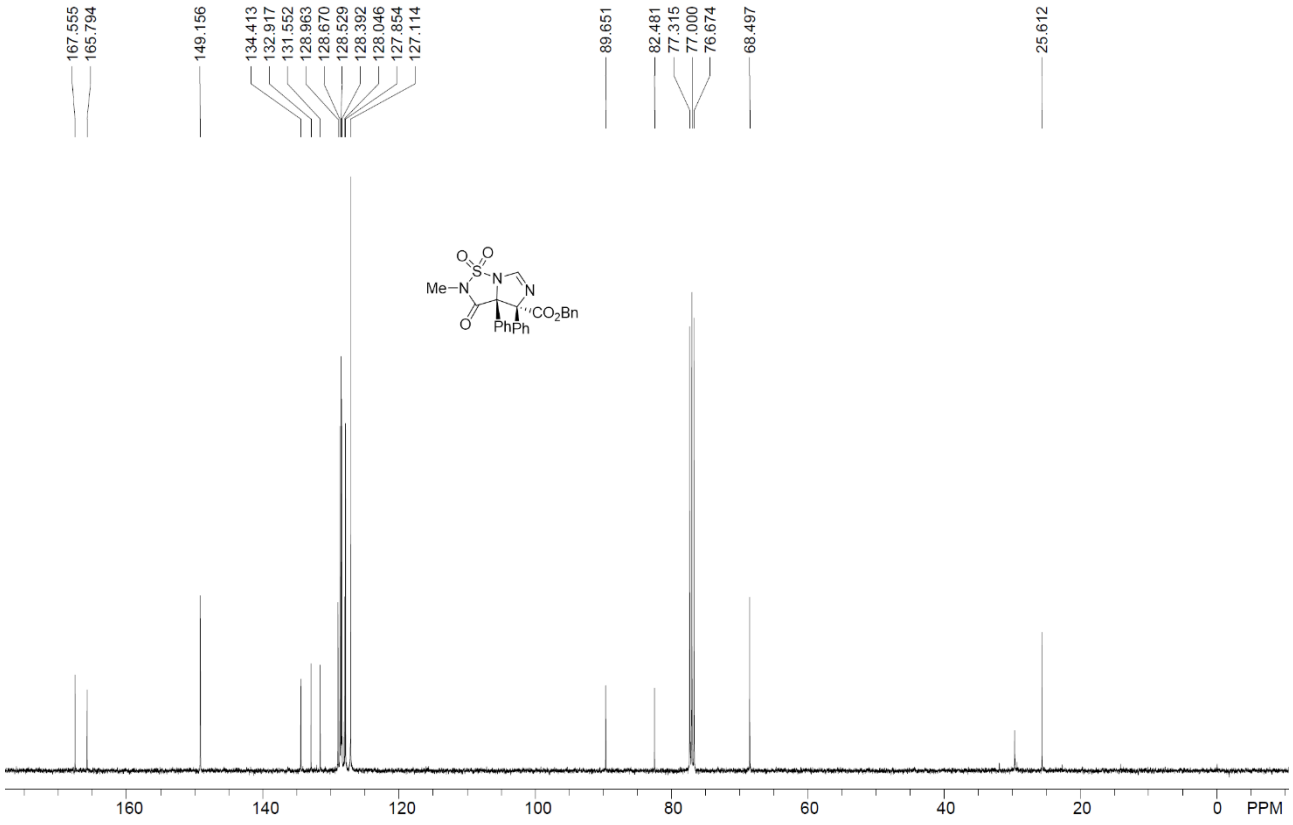


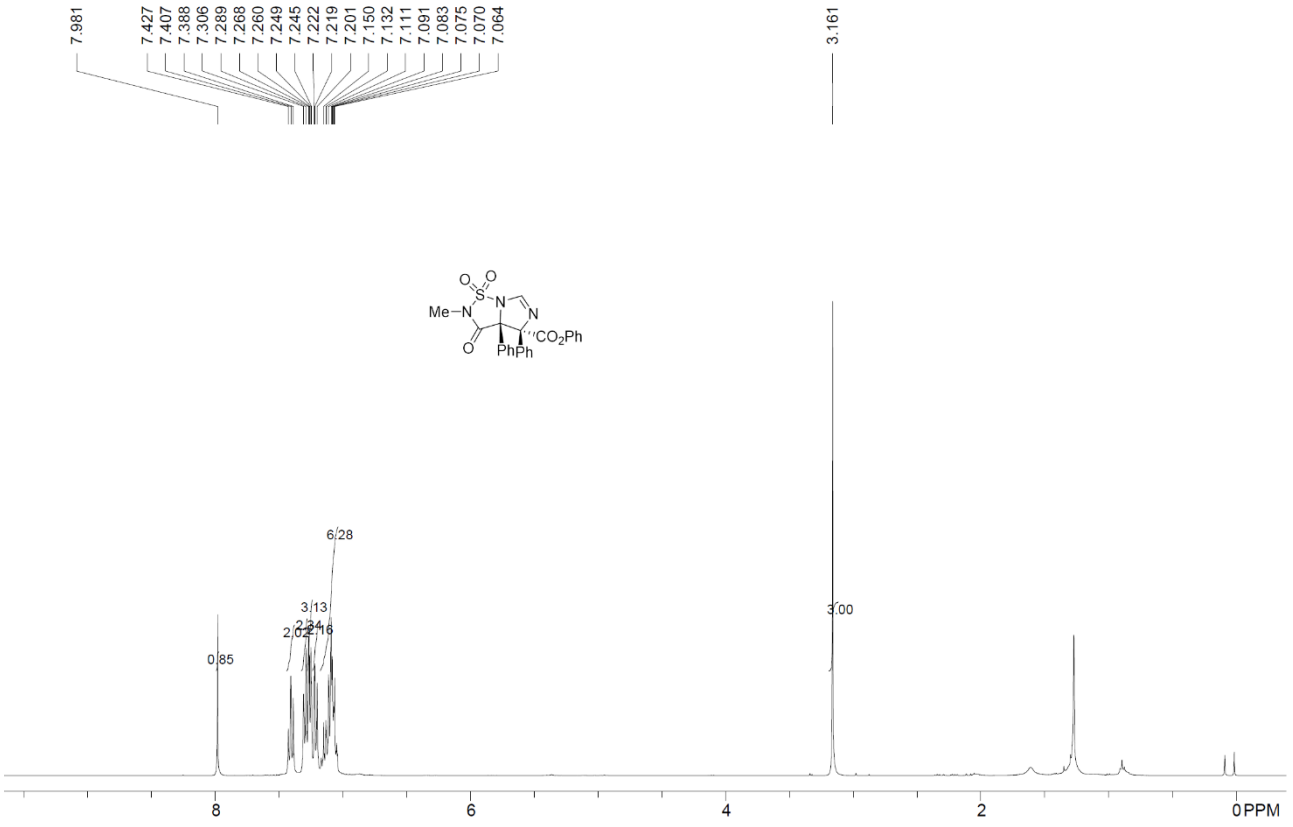
^{1}H NMR of **4l**



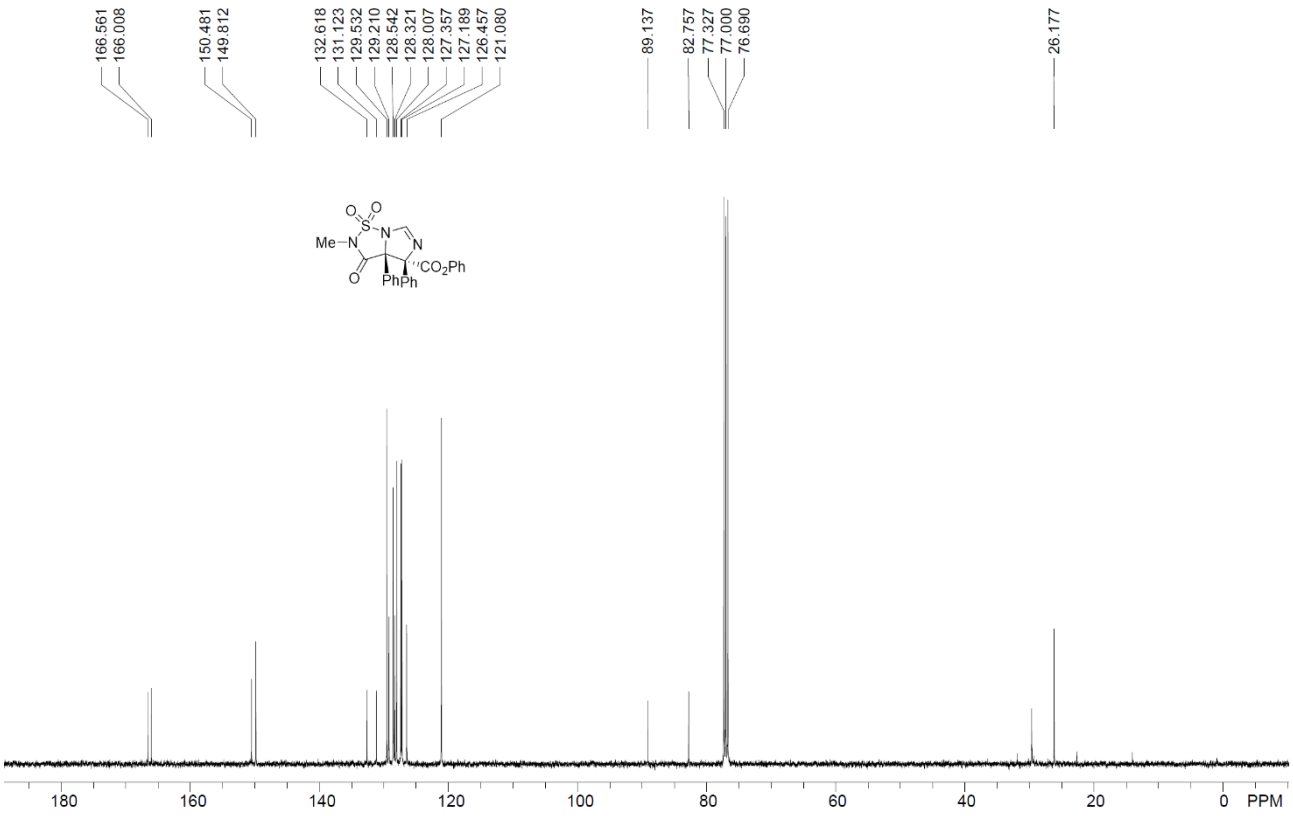
^{13}C NMR of **4l**



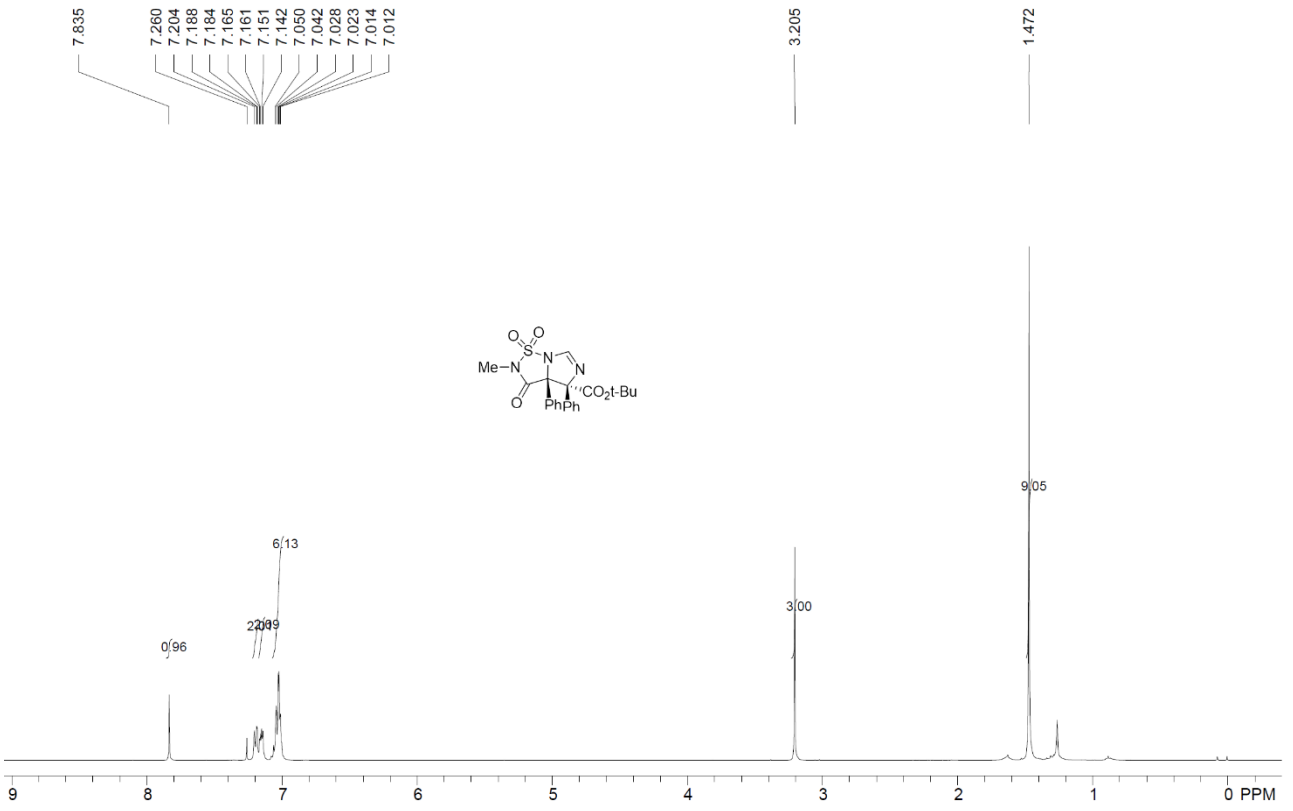
^{1}H NMR of **4m**



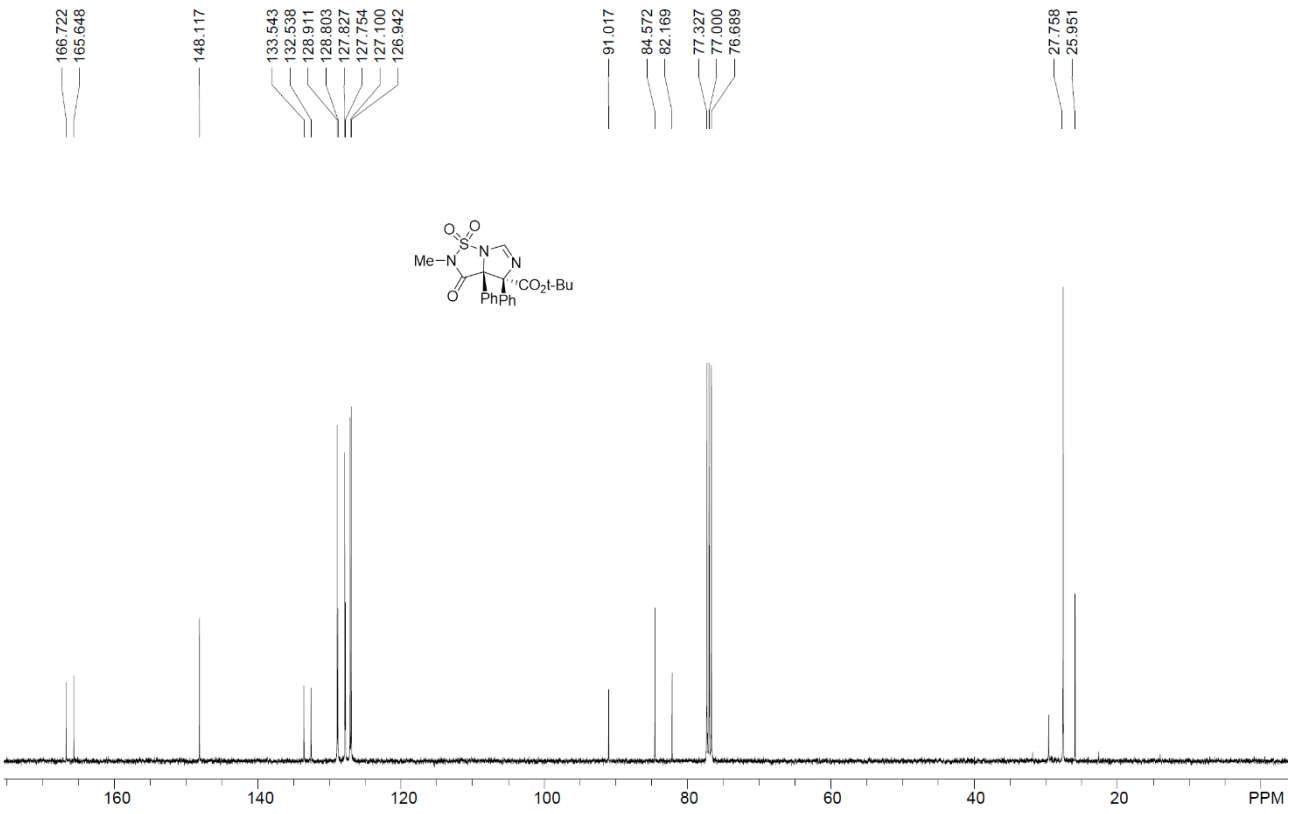
^{13}C NMR of **4m**



^{1}H NMR of **4n**



^{13}C NMR of **4n**



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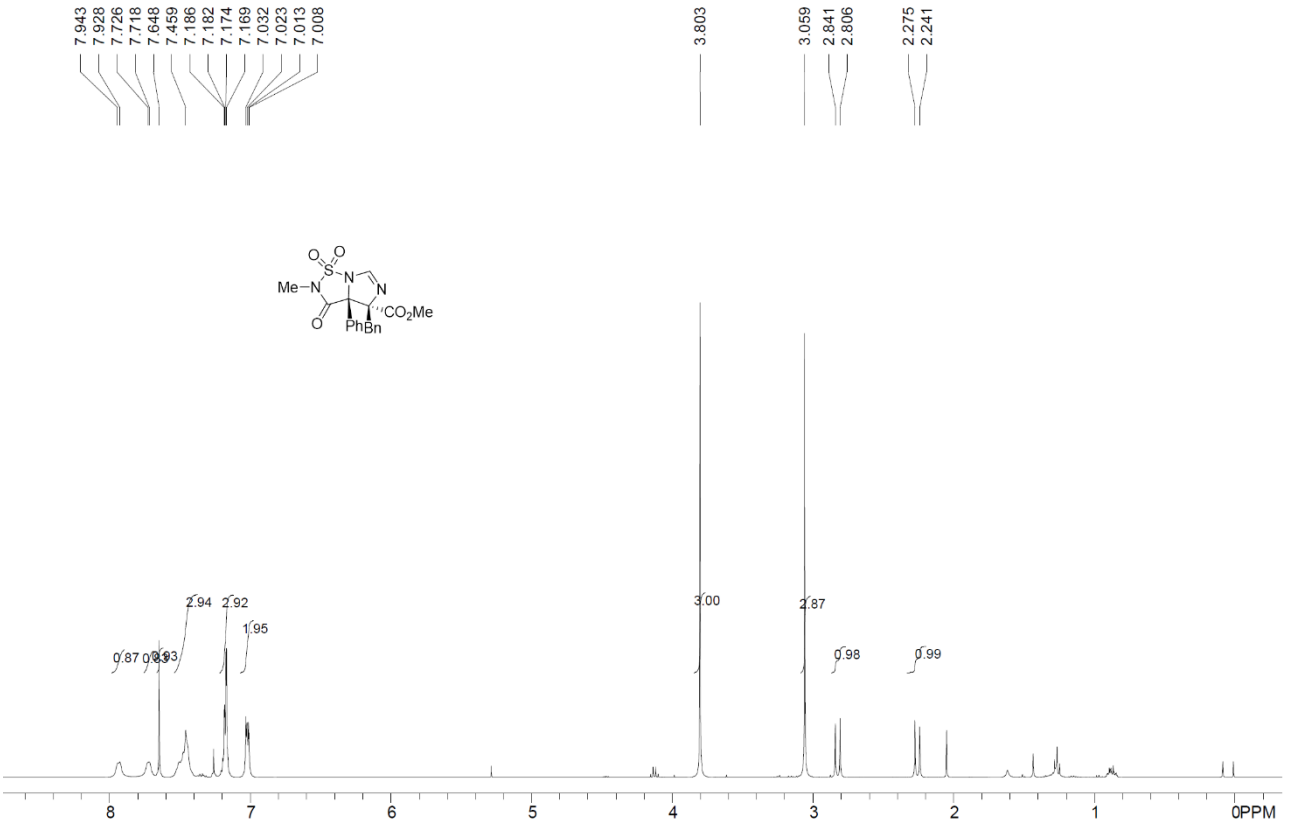

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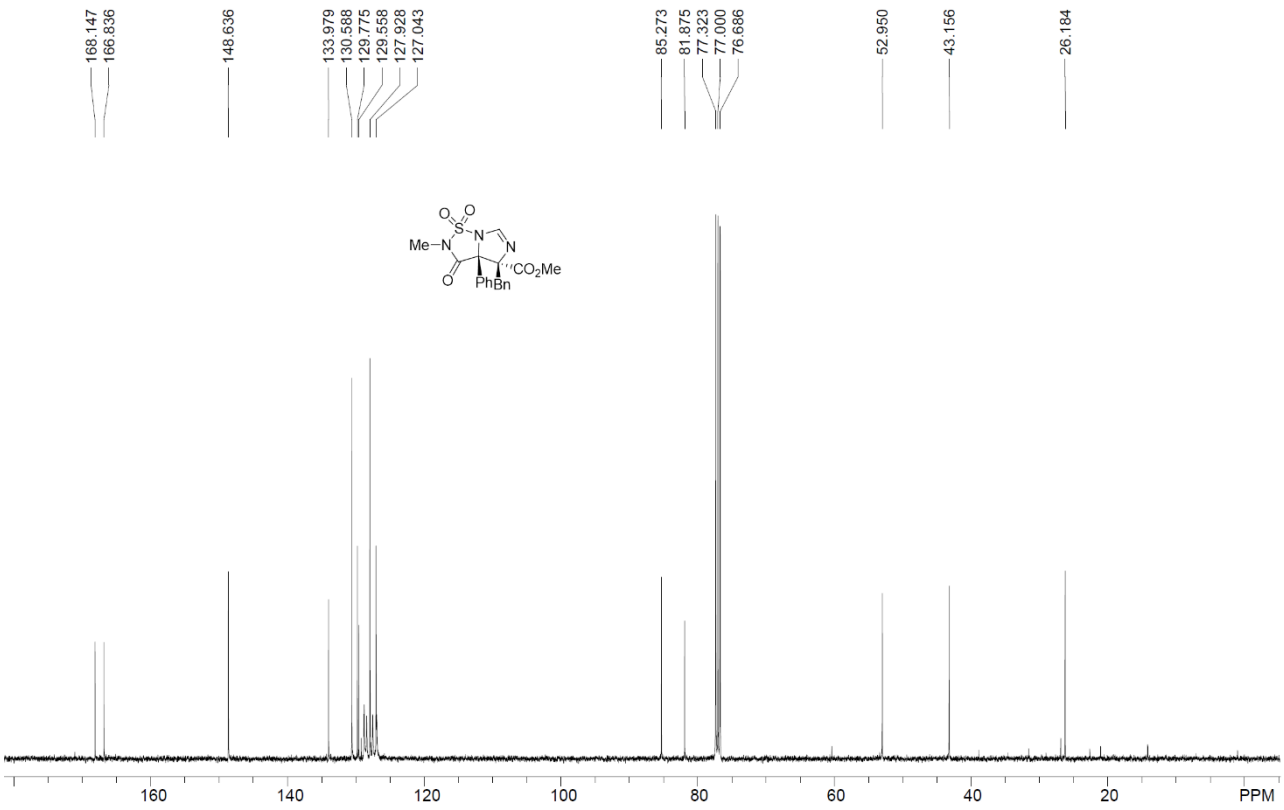
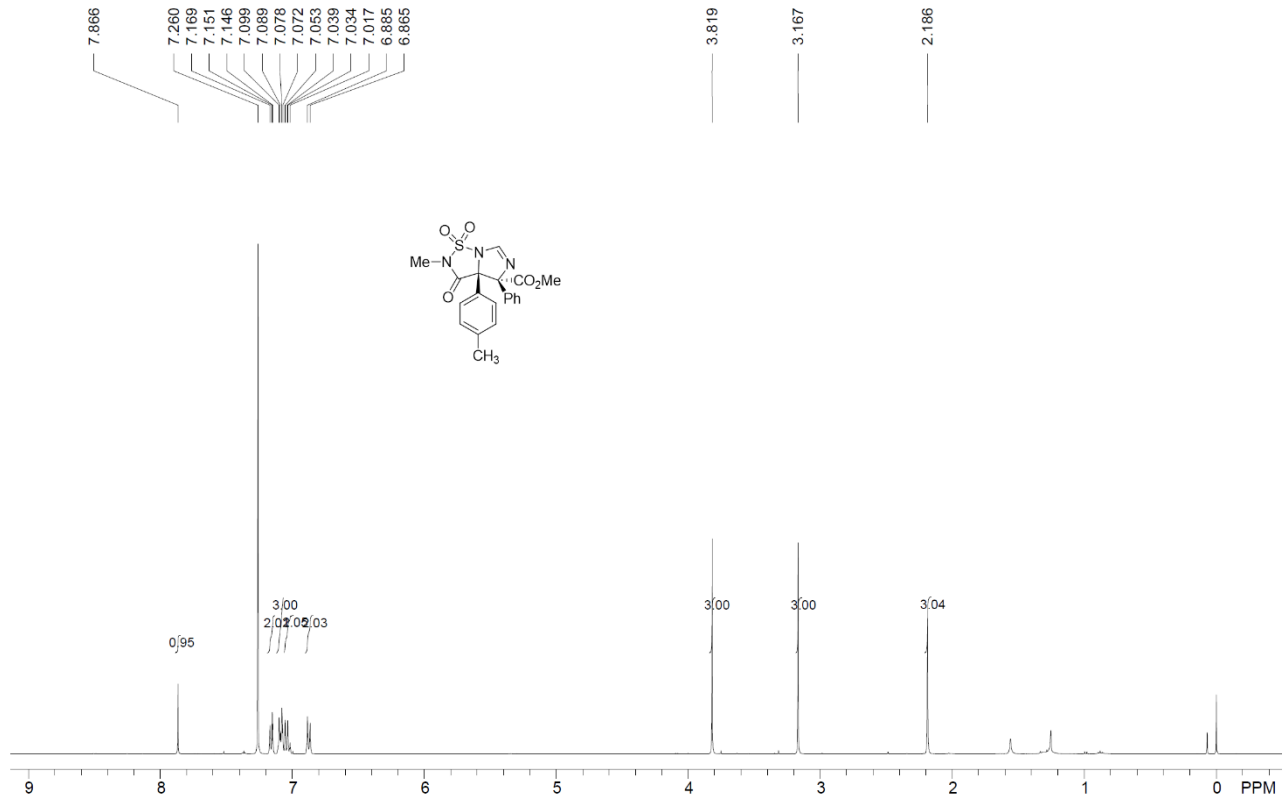
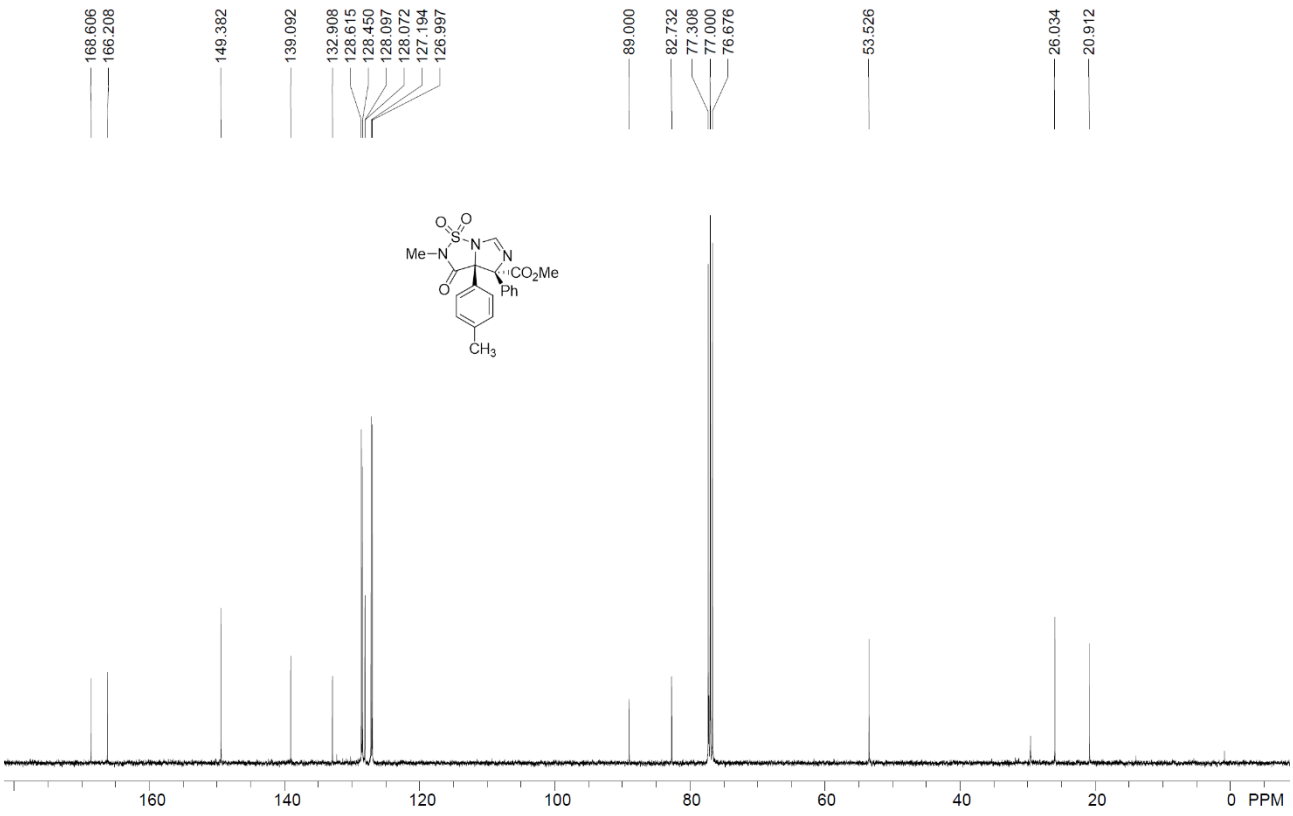


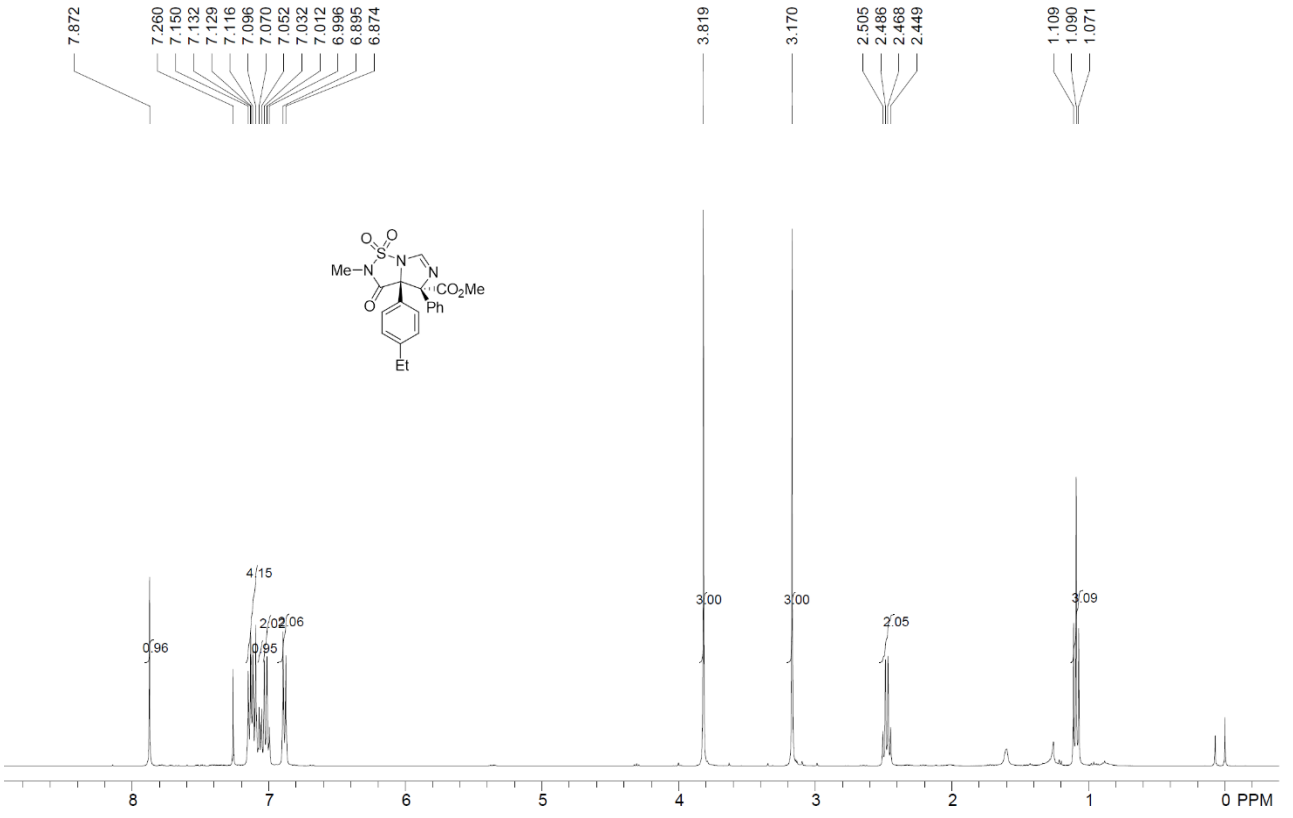
^{1}H NMR of **4p**



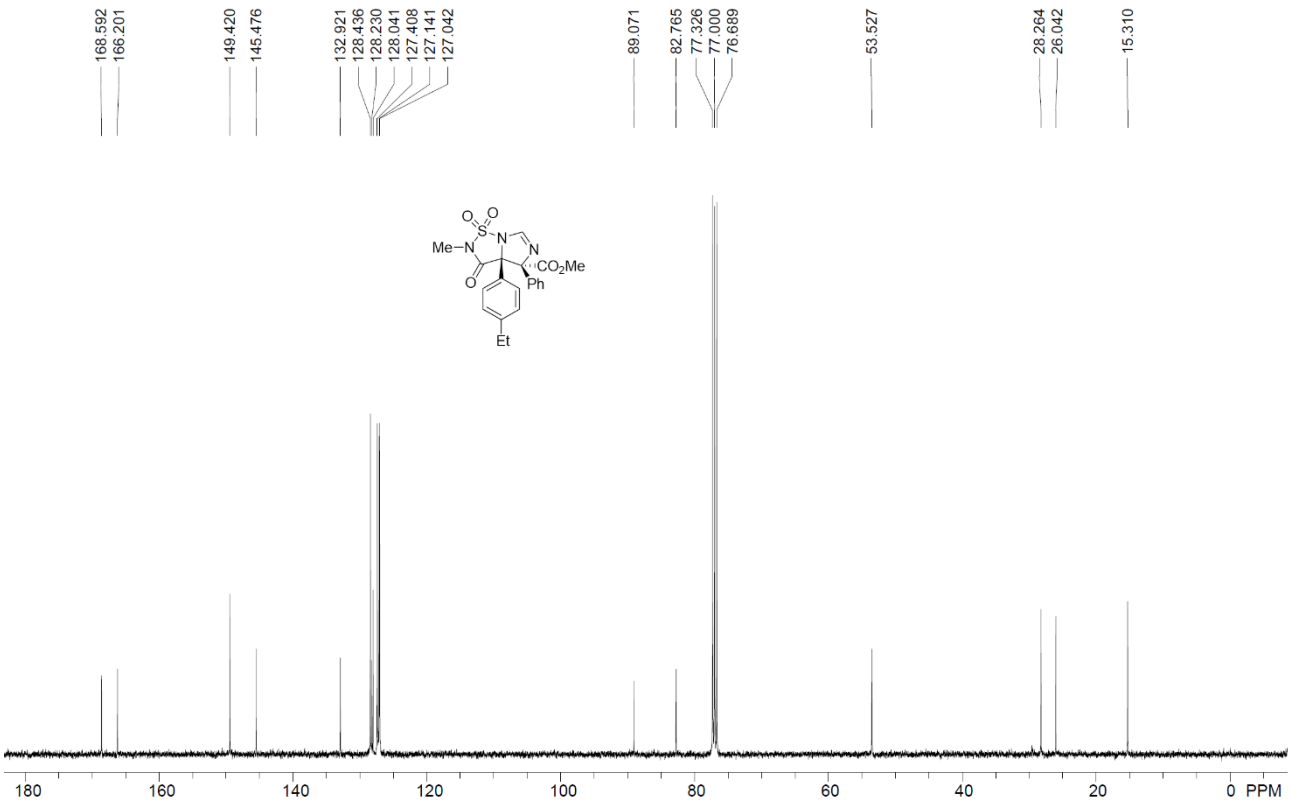
^{13}C NMR of **4p**



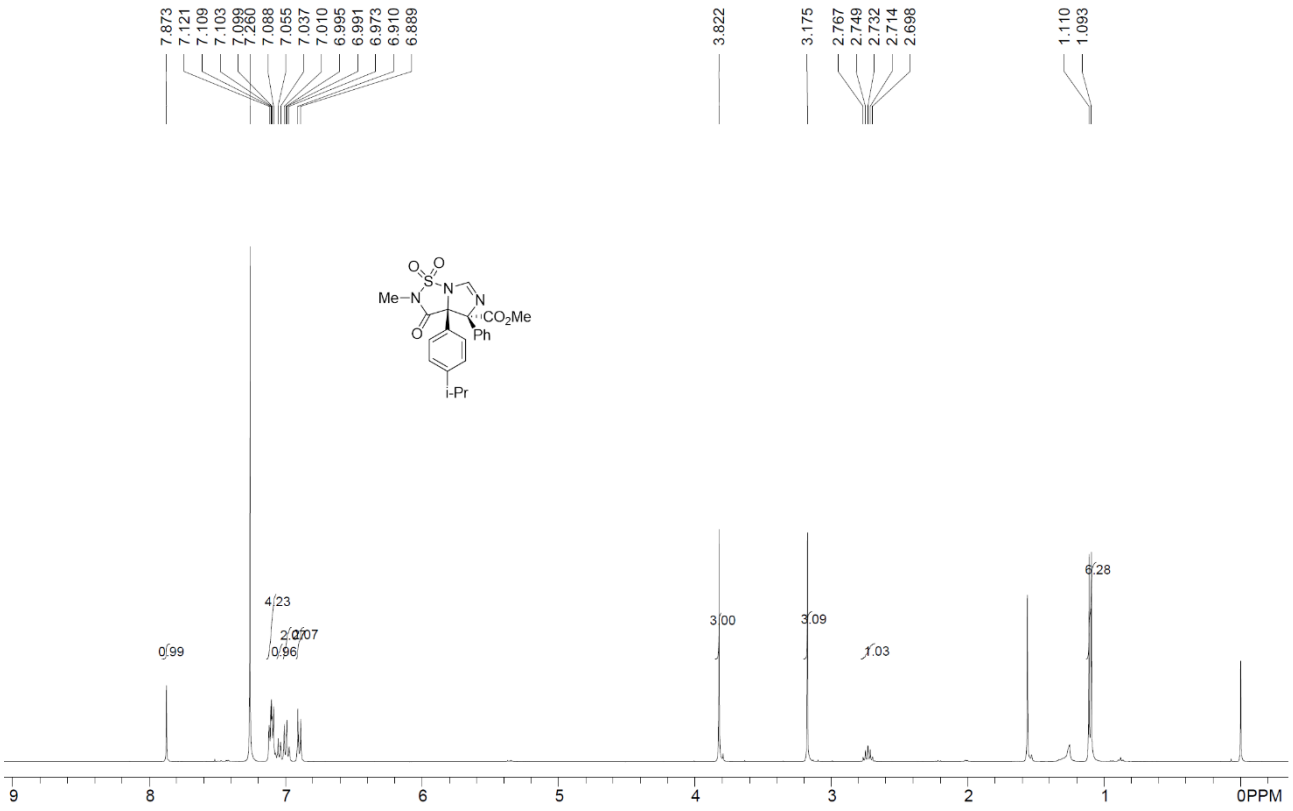
^{1}H NMR of **4q**



^{13}C NMR of **4q**



^{1}H NMR of **4r**



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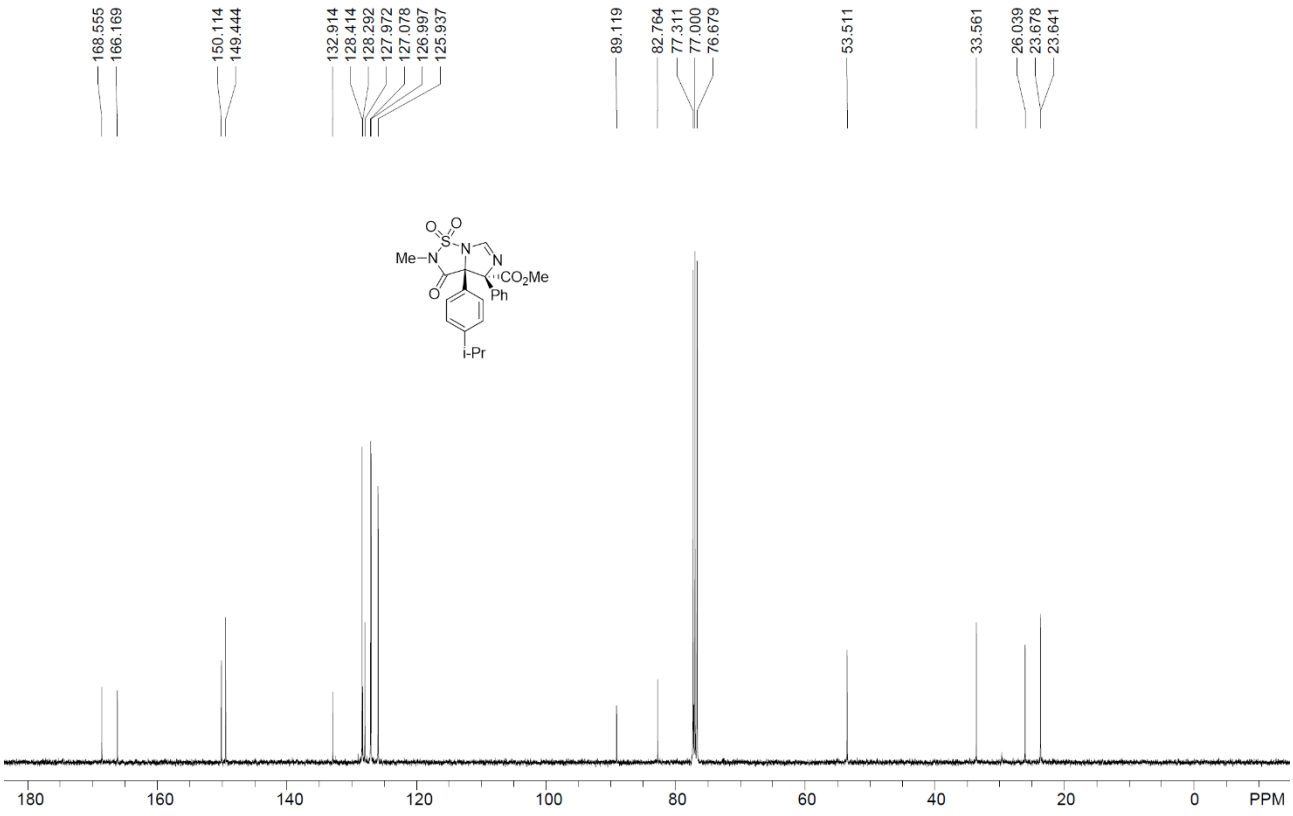

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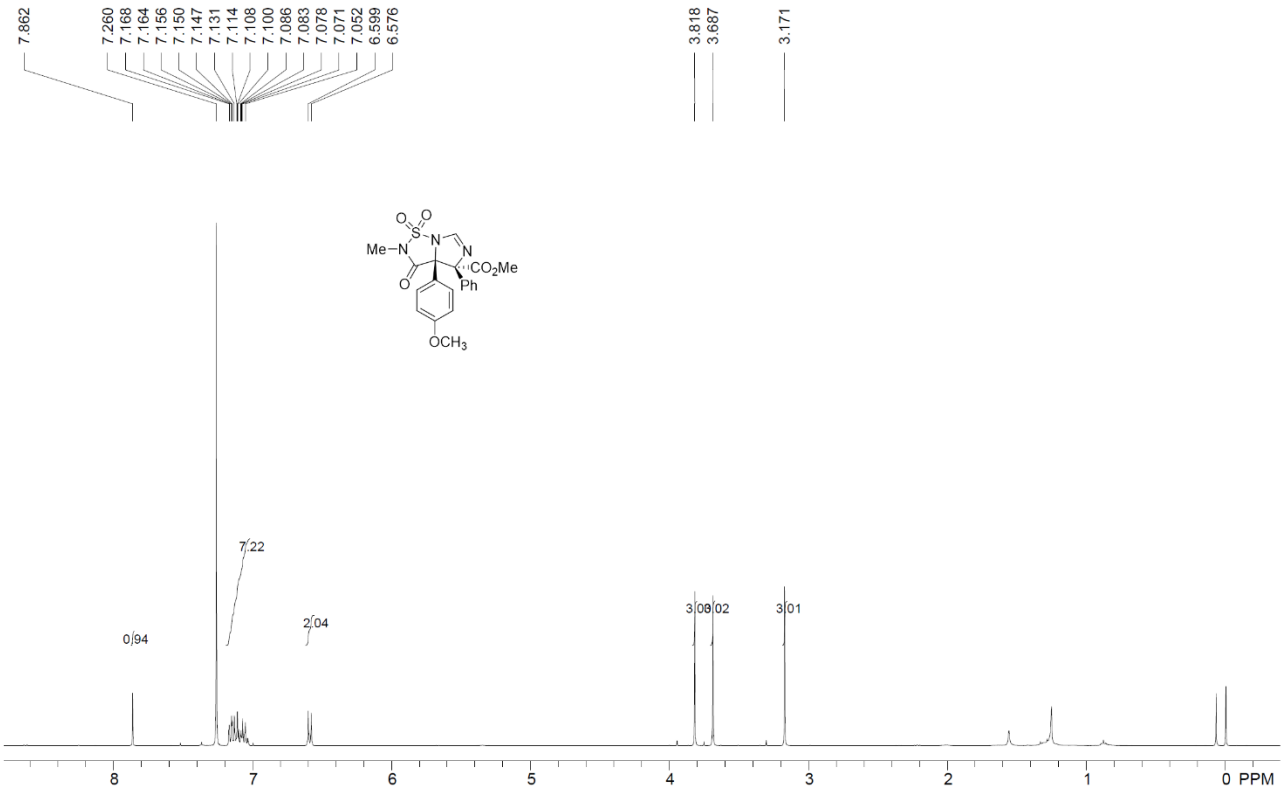
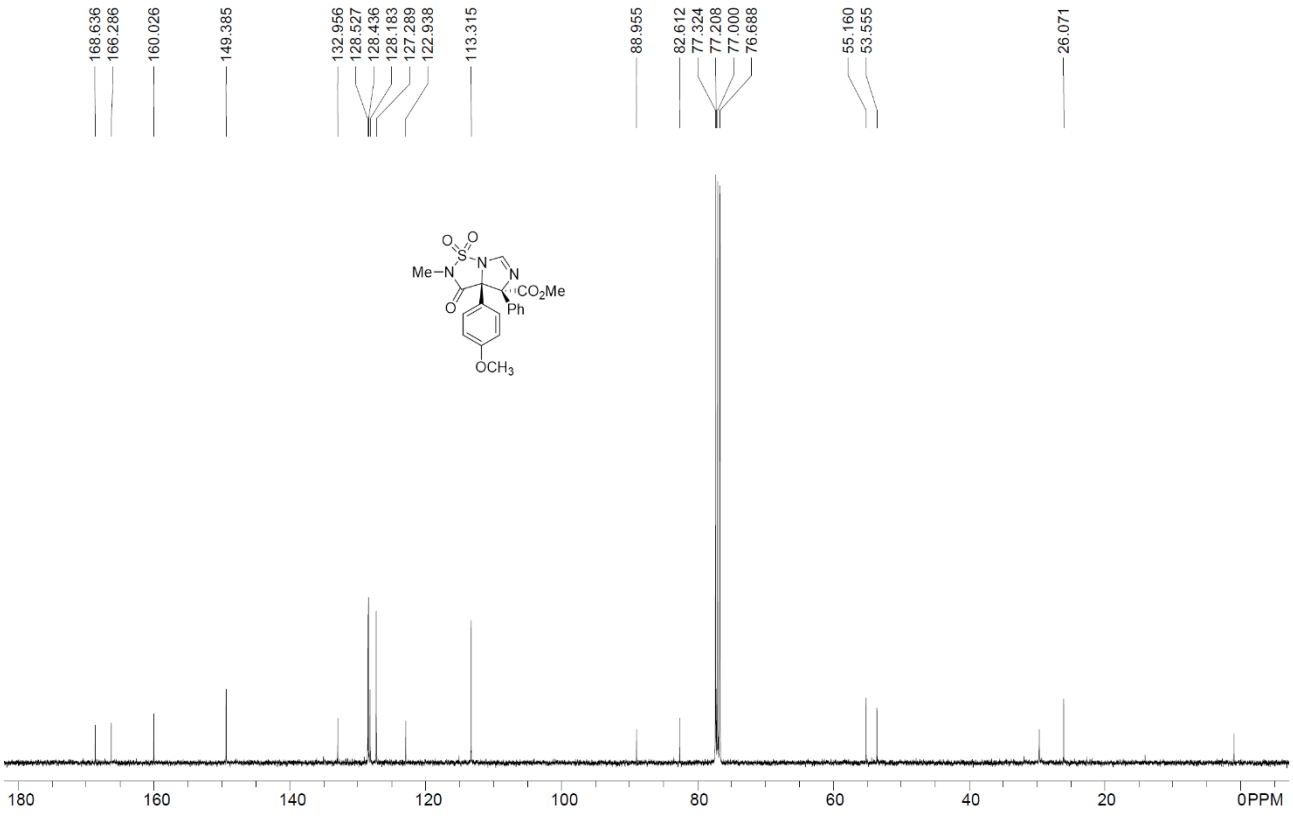
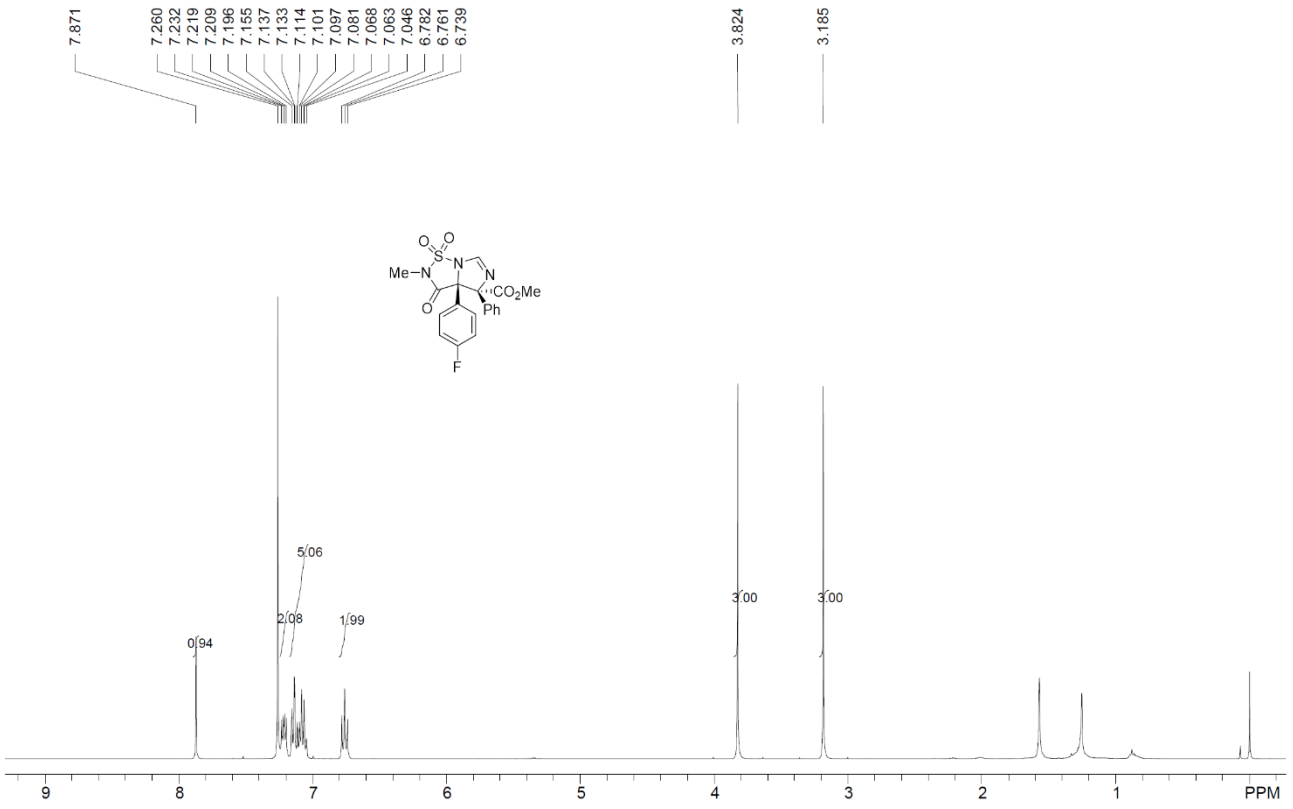


^{13}C NMR of **4s**



^{1}H NMR of **4t**



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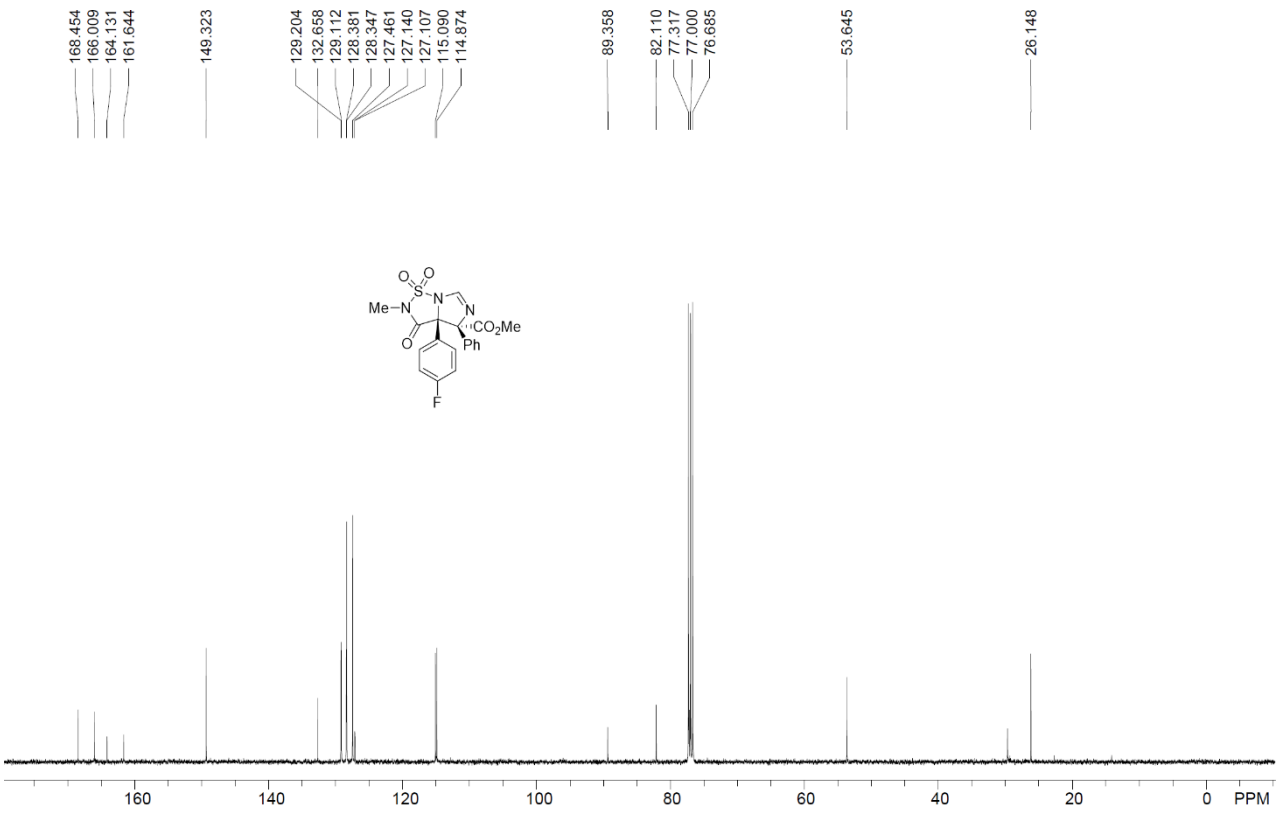
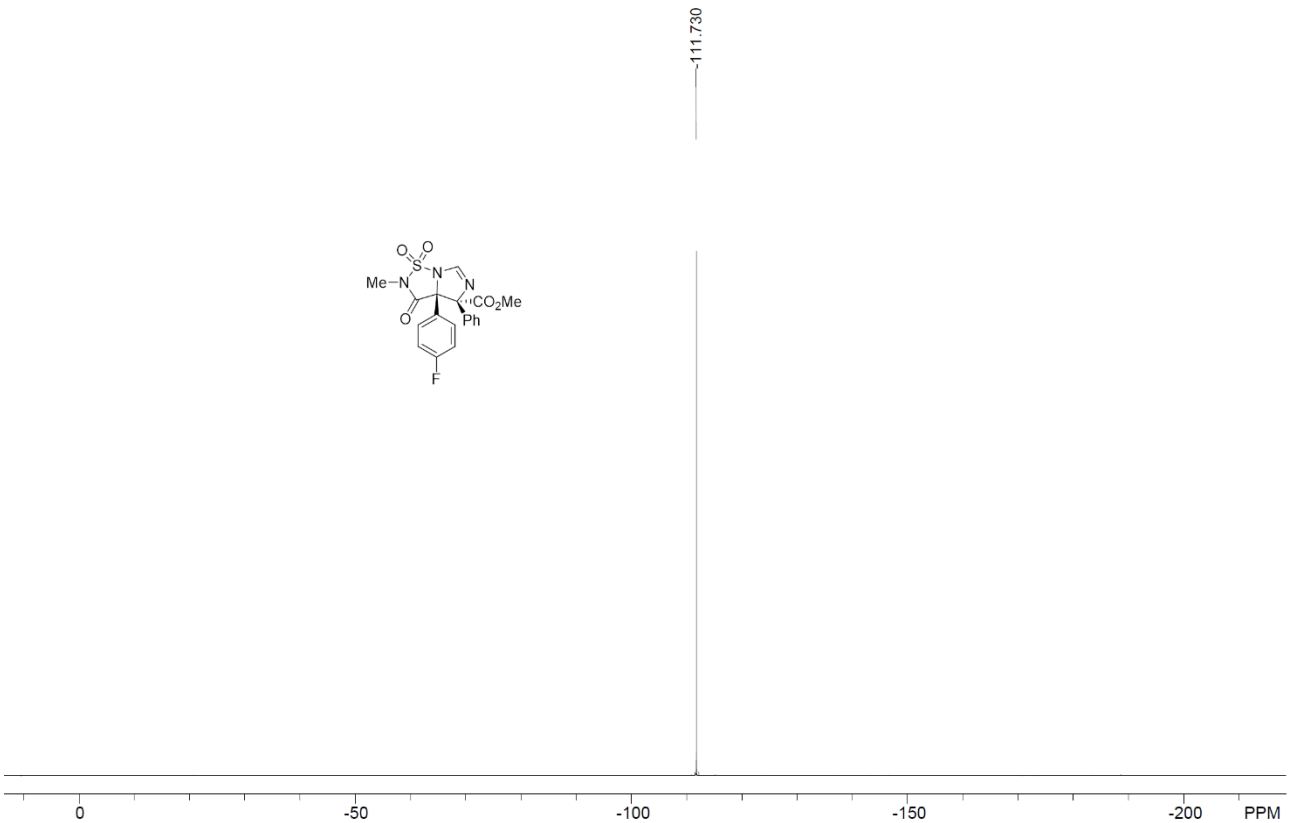
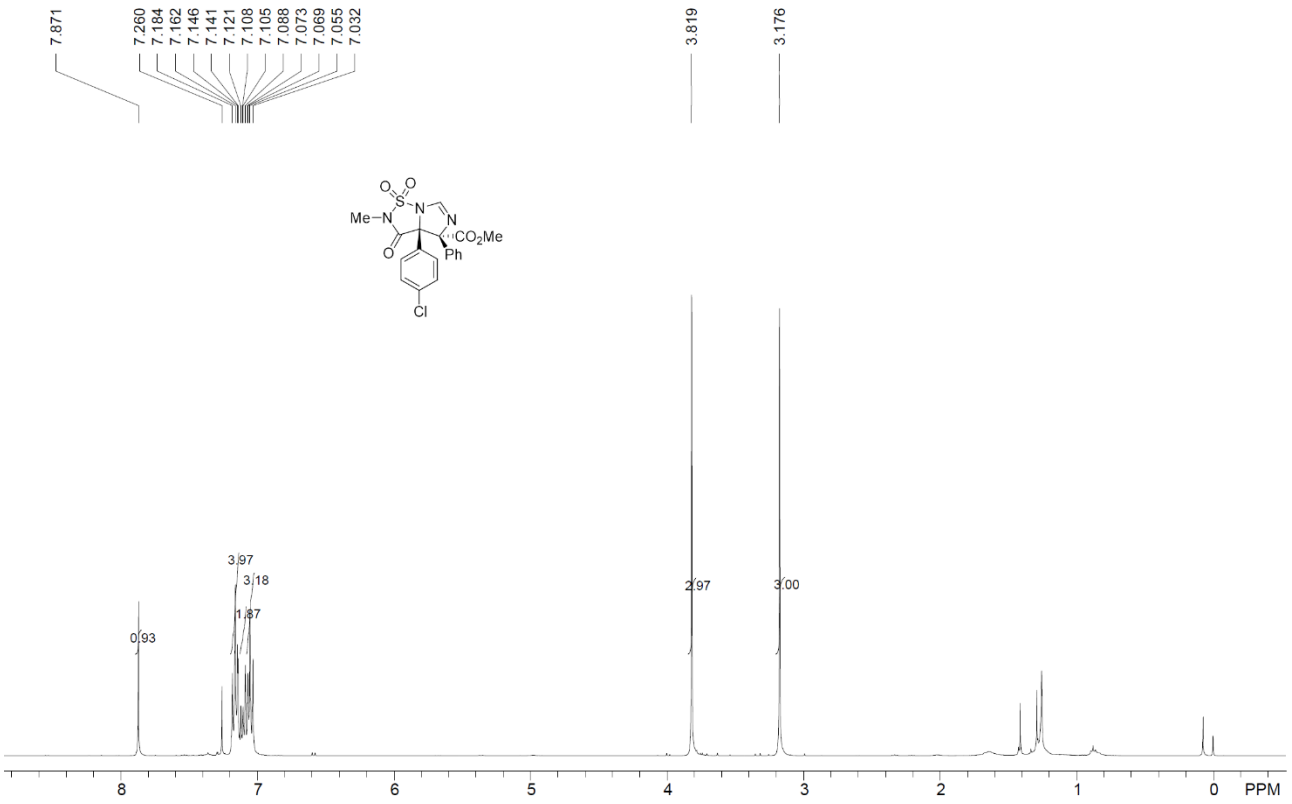


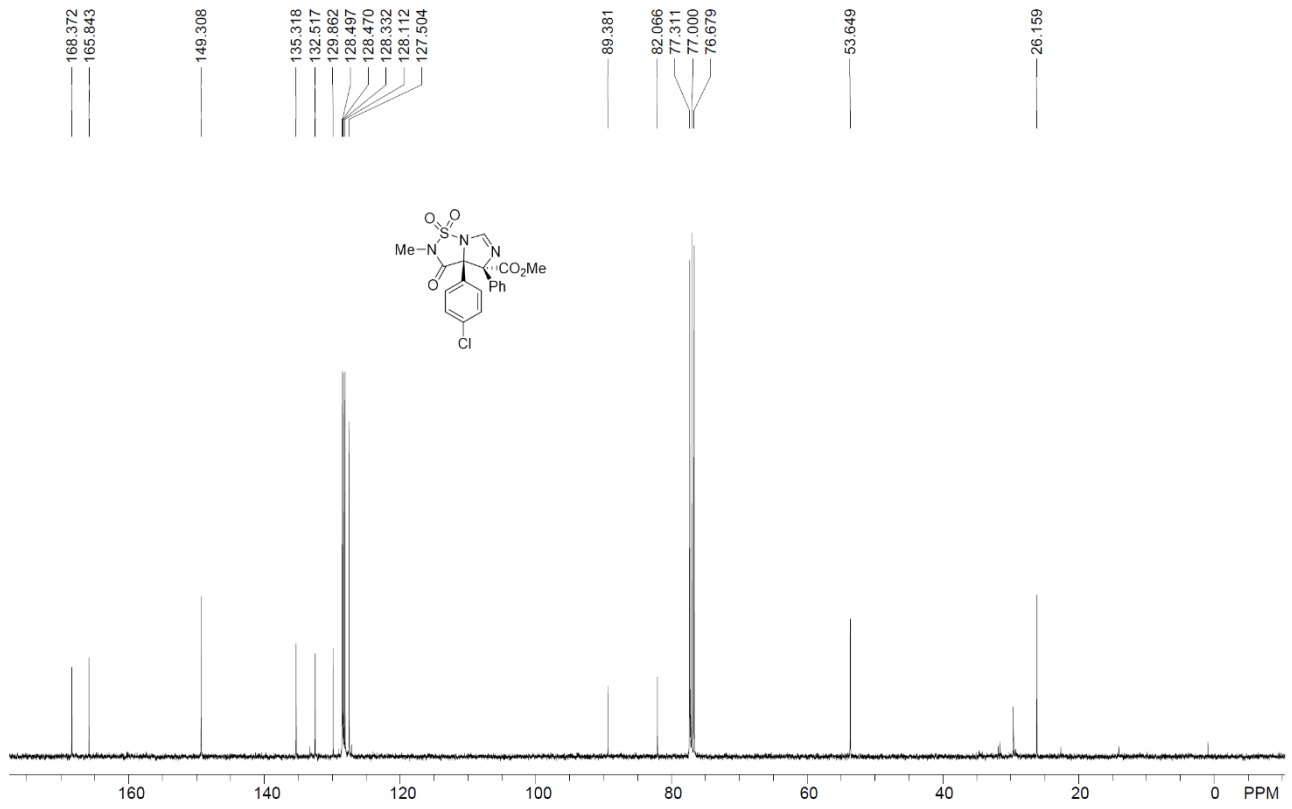
^{19}F NMR of **4t**



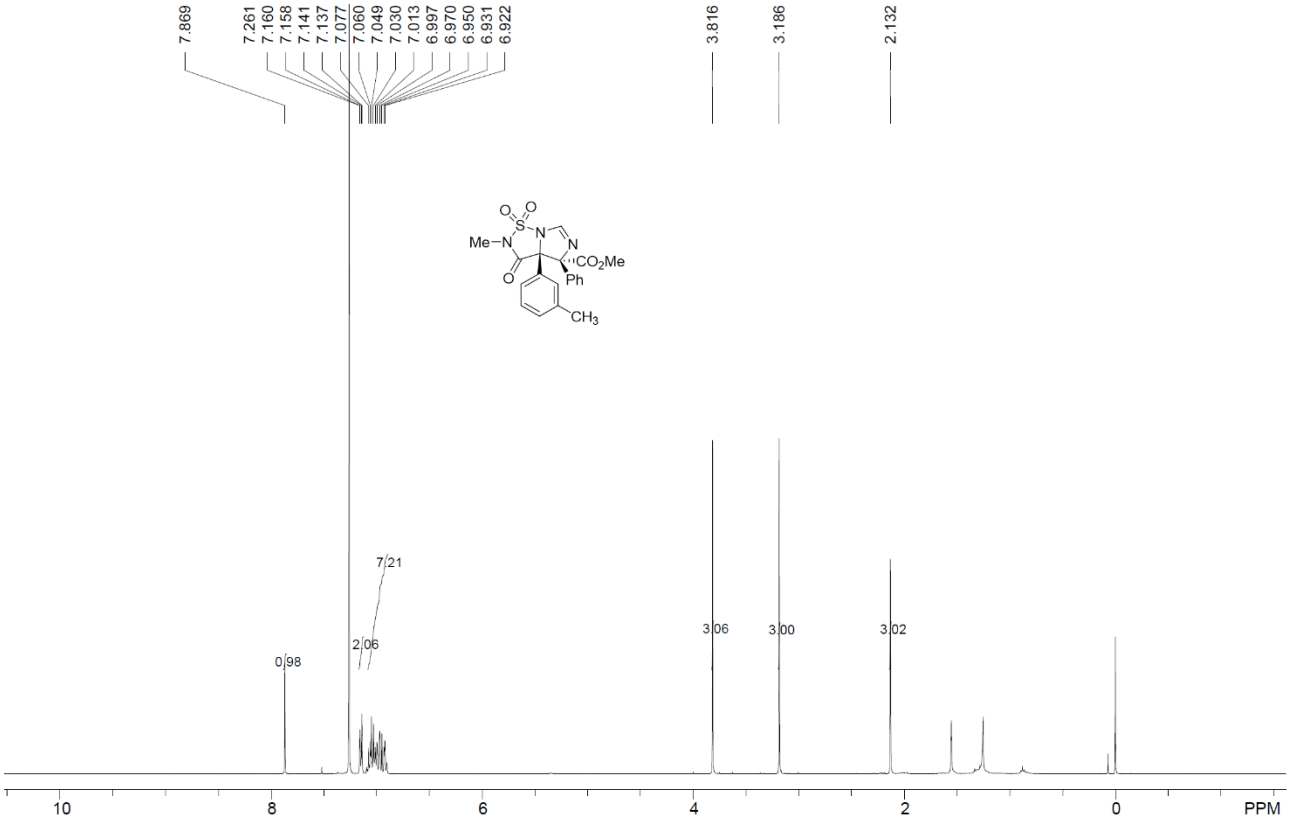
^{1}H NMR of **4u**



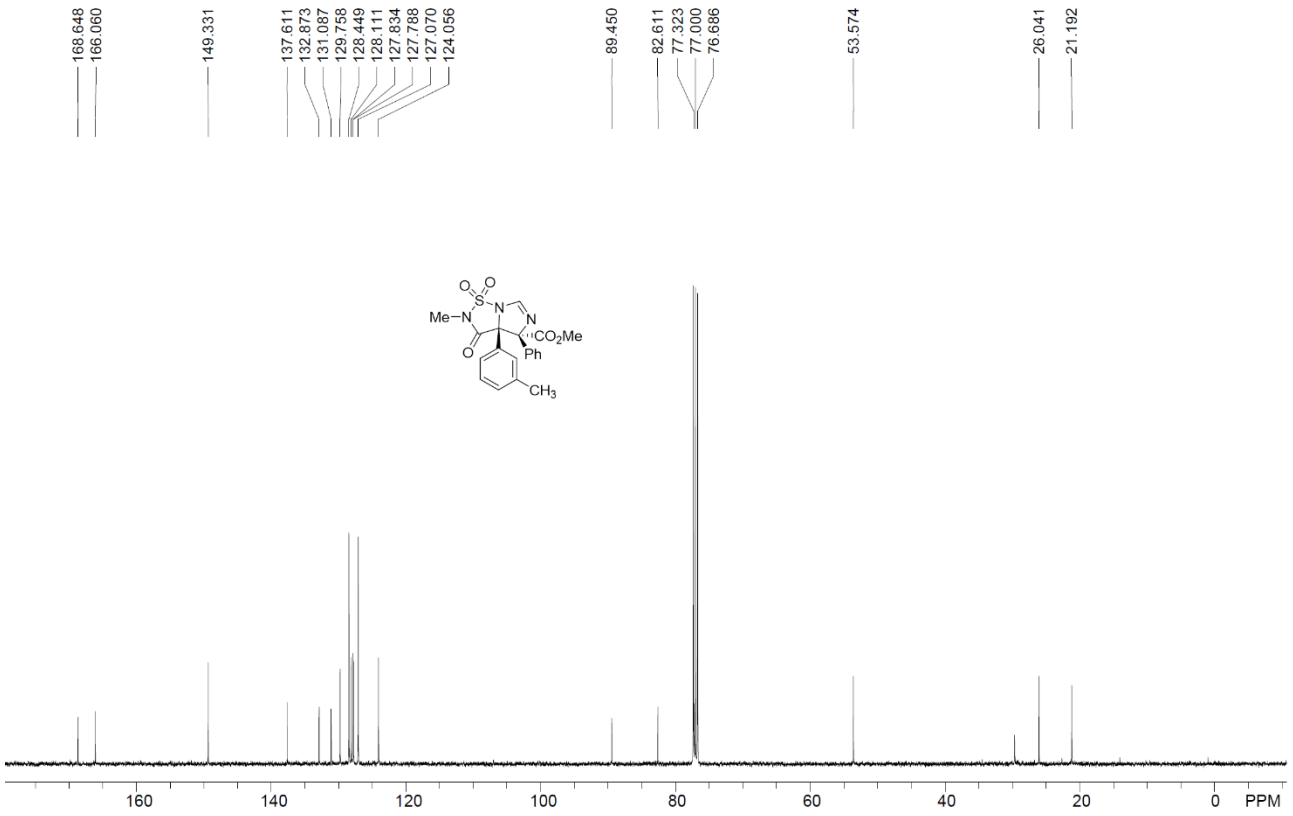
^{13}C NMR of **4u**



^{1}H NMR of **4v**



^{13}C NMR of **4v**



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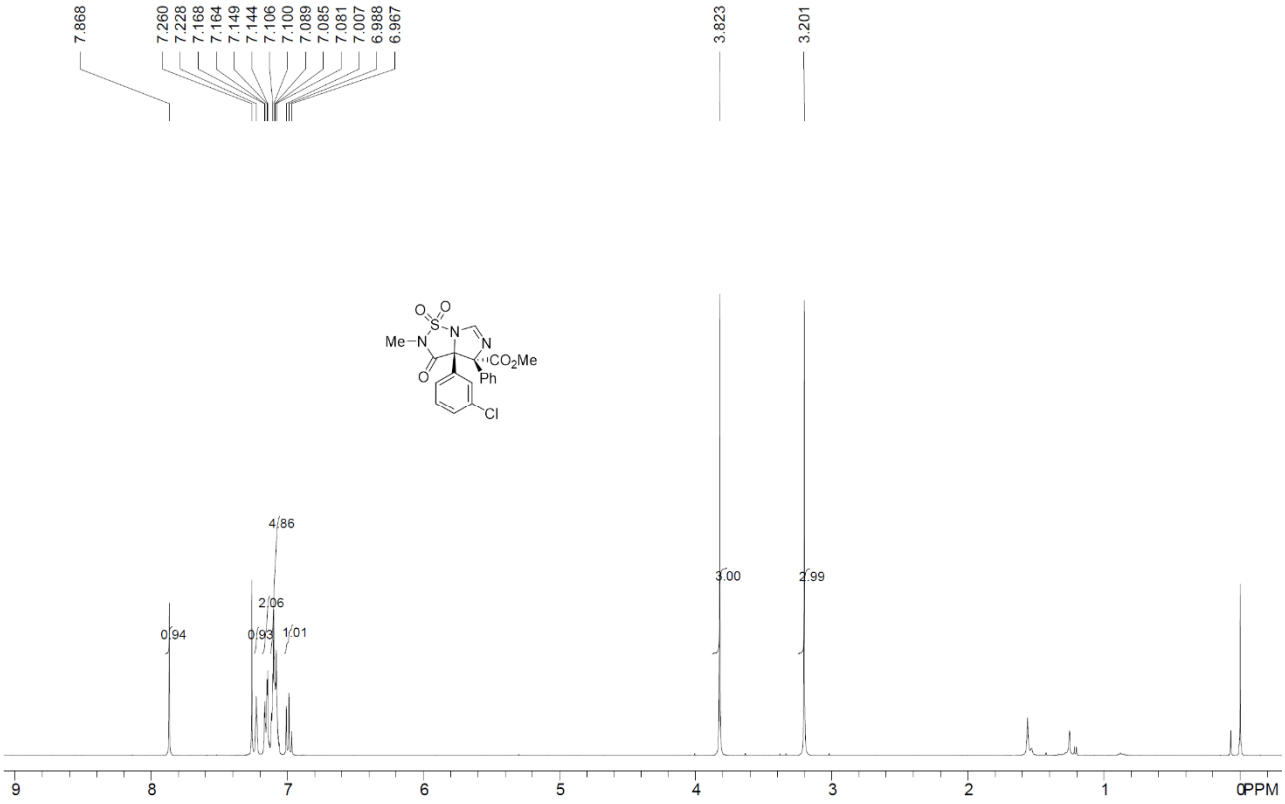
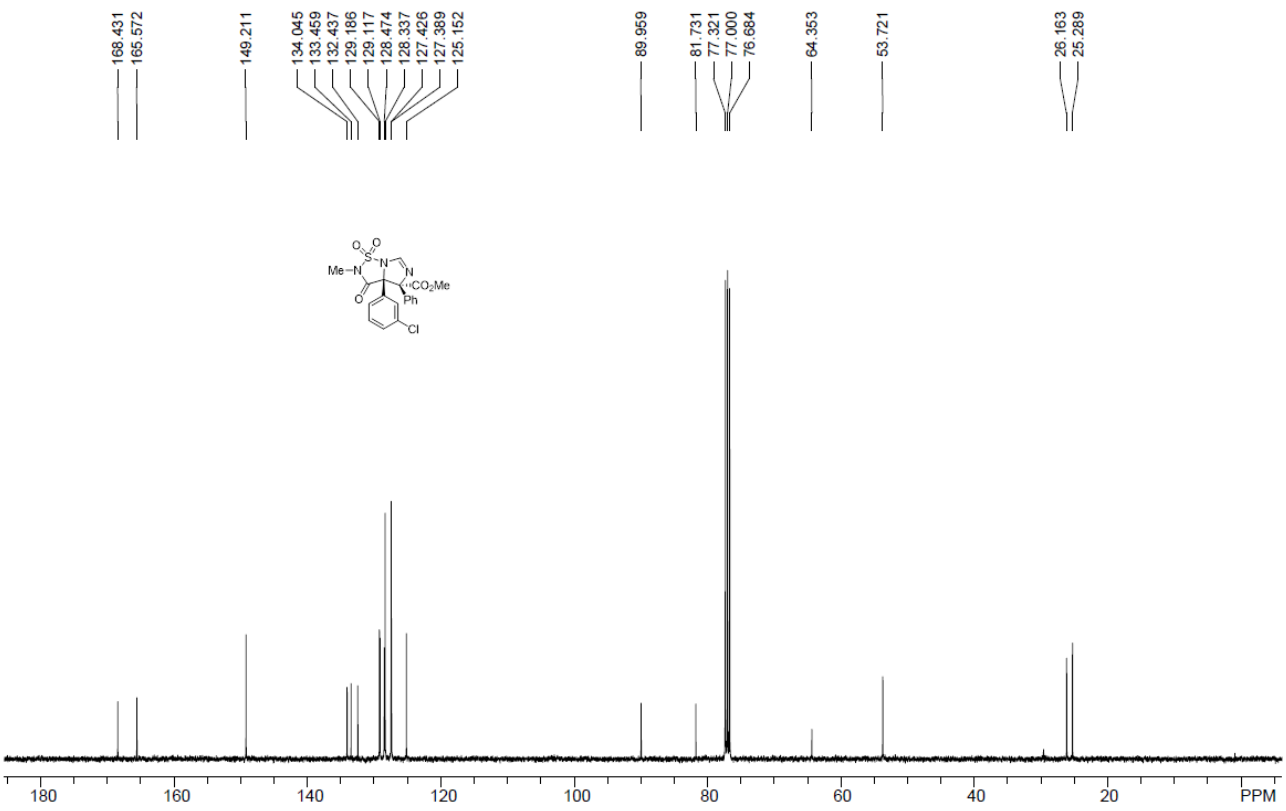
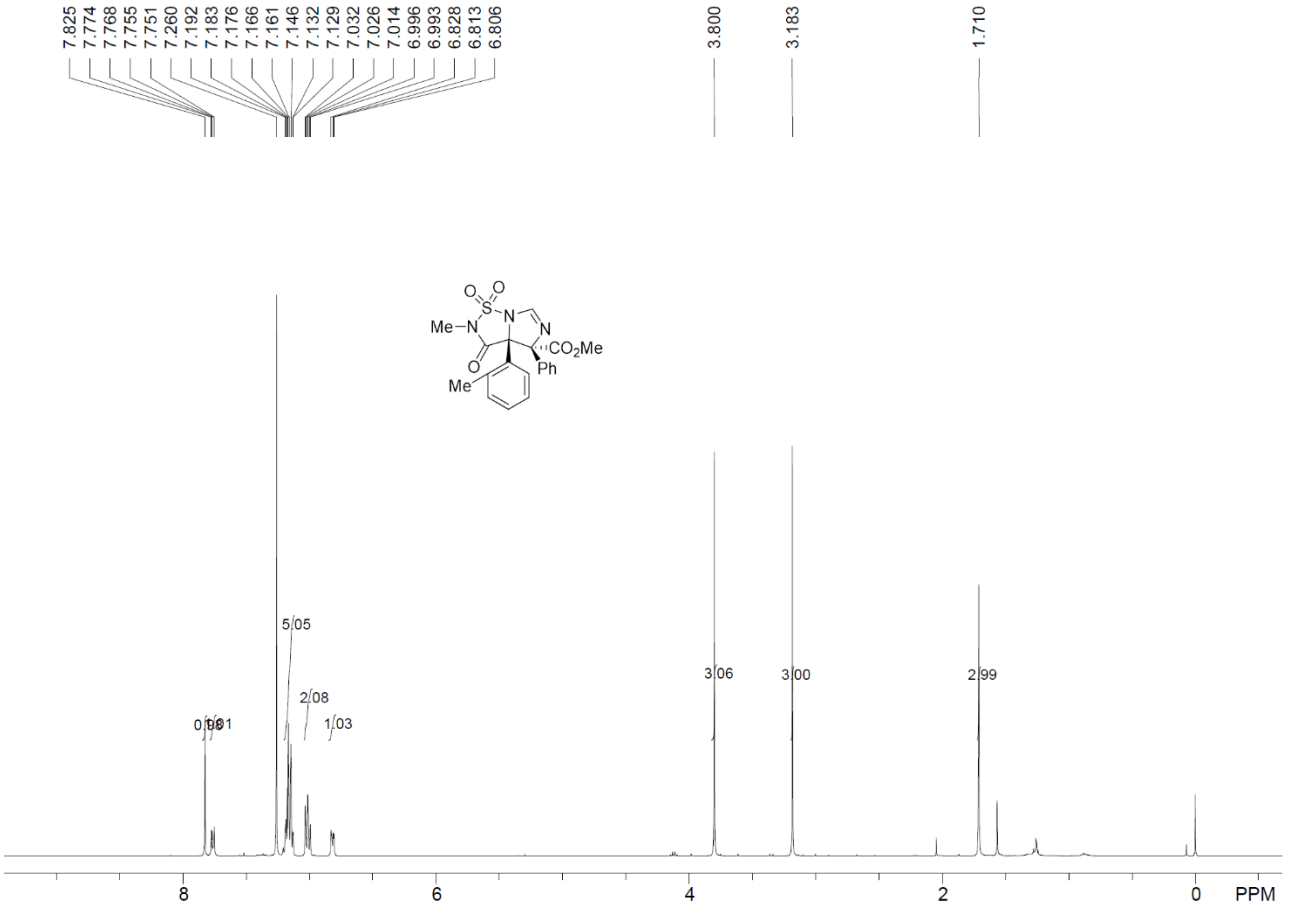


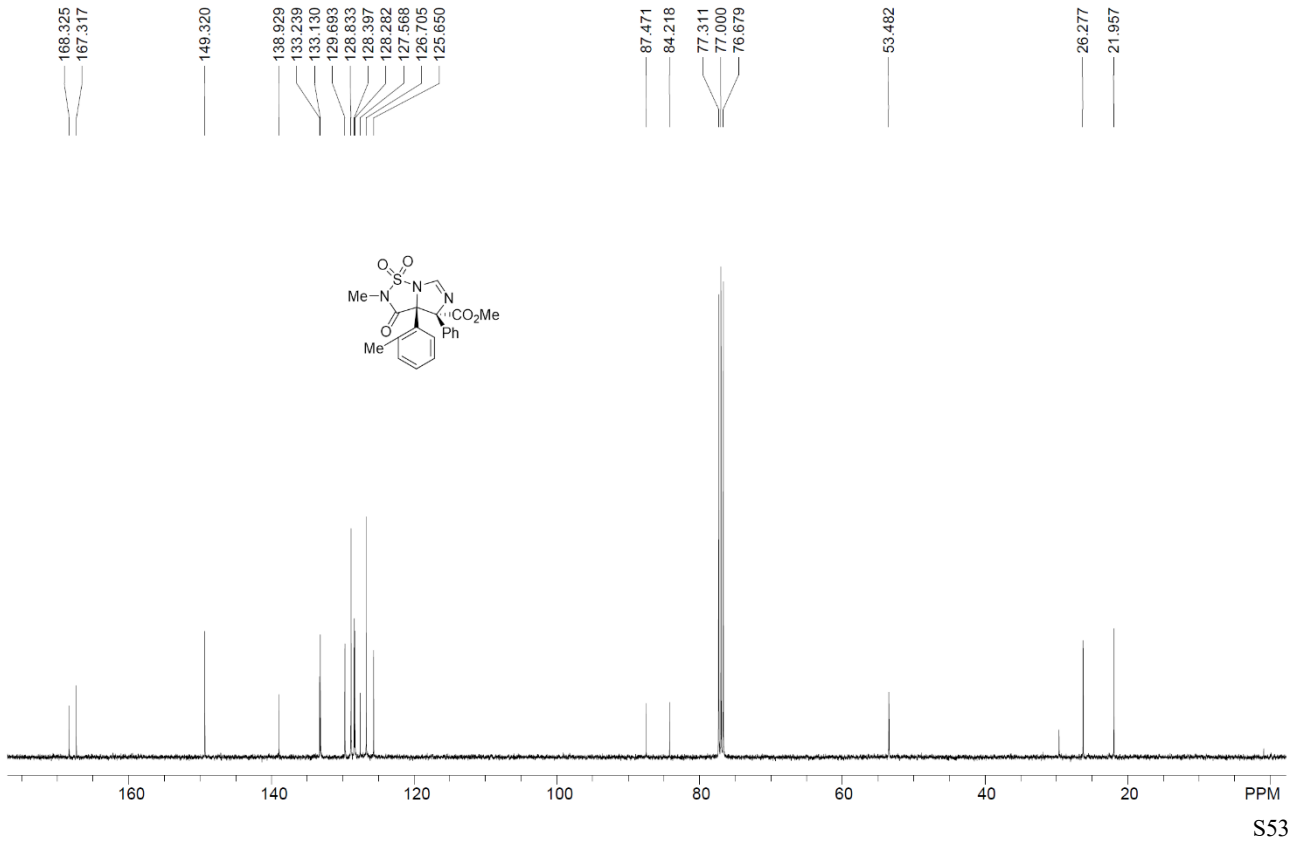
^{13}C NMR of **4w**



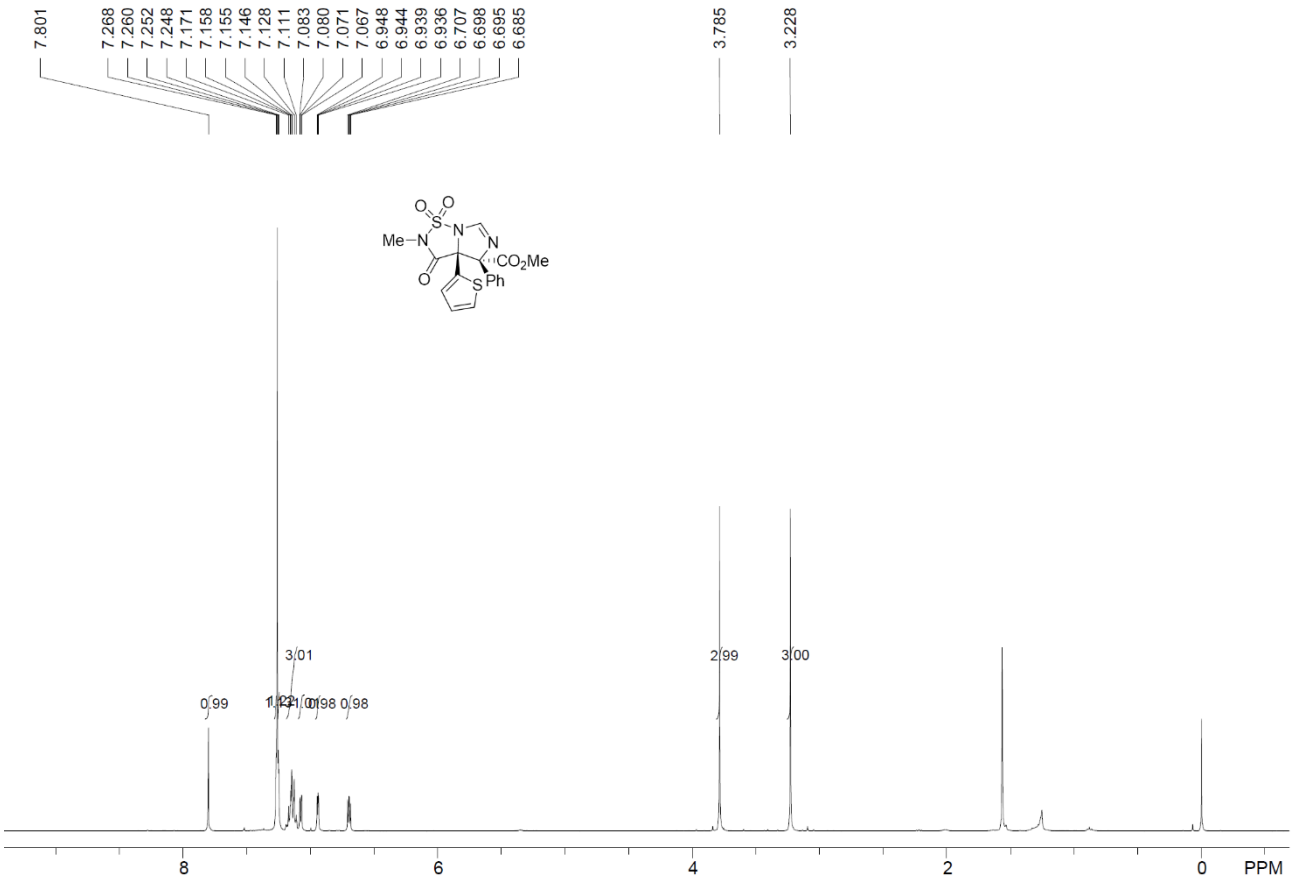
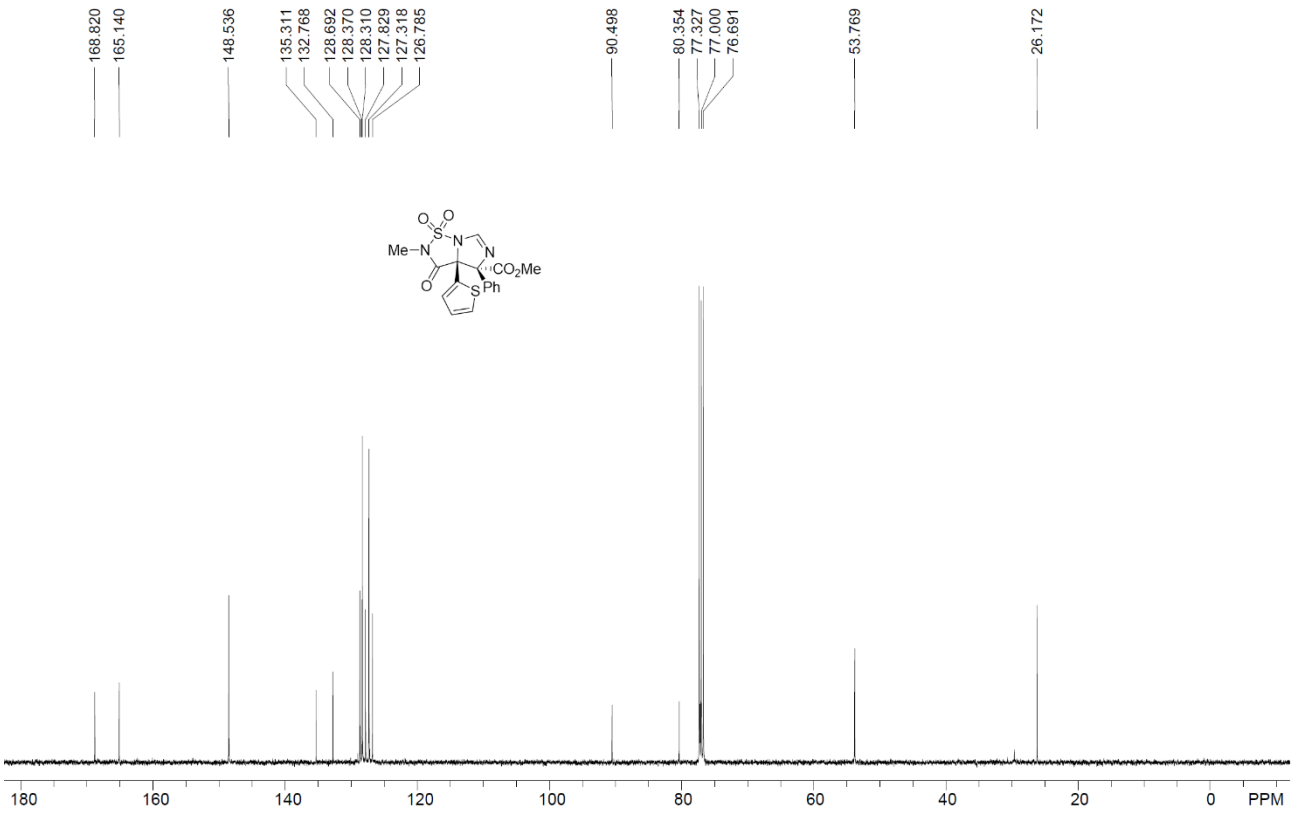
^{1}H NMR of **4x**



^{13}C NMR of **4x**



^{1}H NMR of **4y**



^{13}C NMR of **4y**