Electronic Supplementary Information (ESI)

Cu(II)-Thiophene-2,5-Bis(amino-alcohol) mediated Asymmetric Aldol Reaction and Domino Knoevenagel Michael Cyclization: A New Highly Efficient Lewis acid Catalyst

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Electronic Supplementary Information (ESI)

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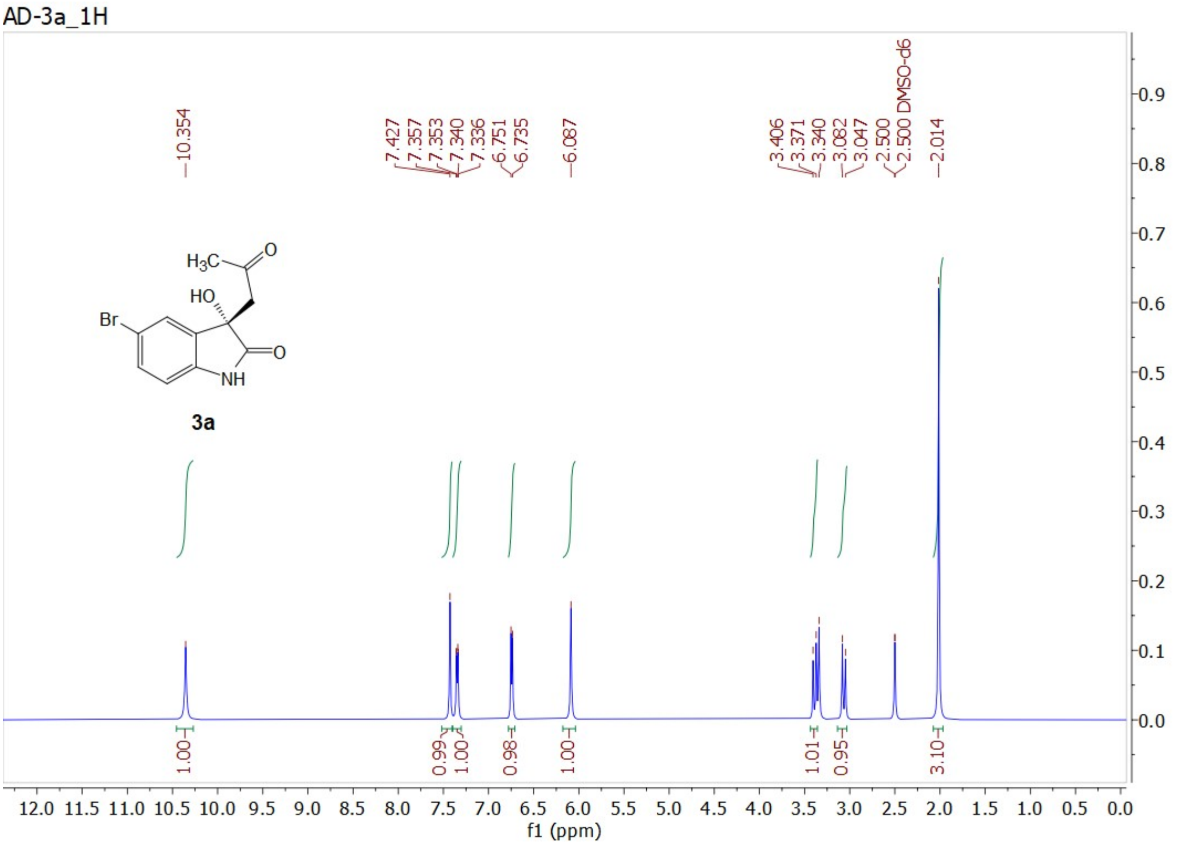
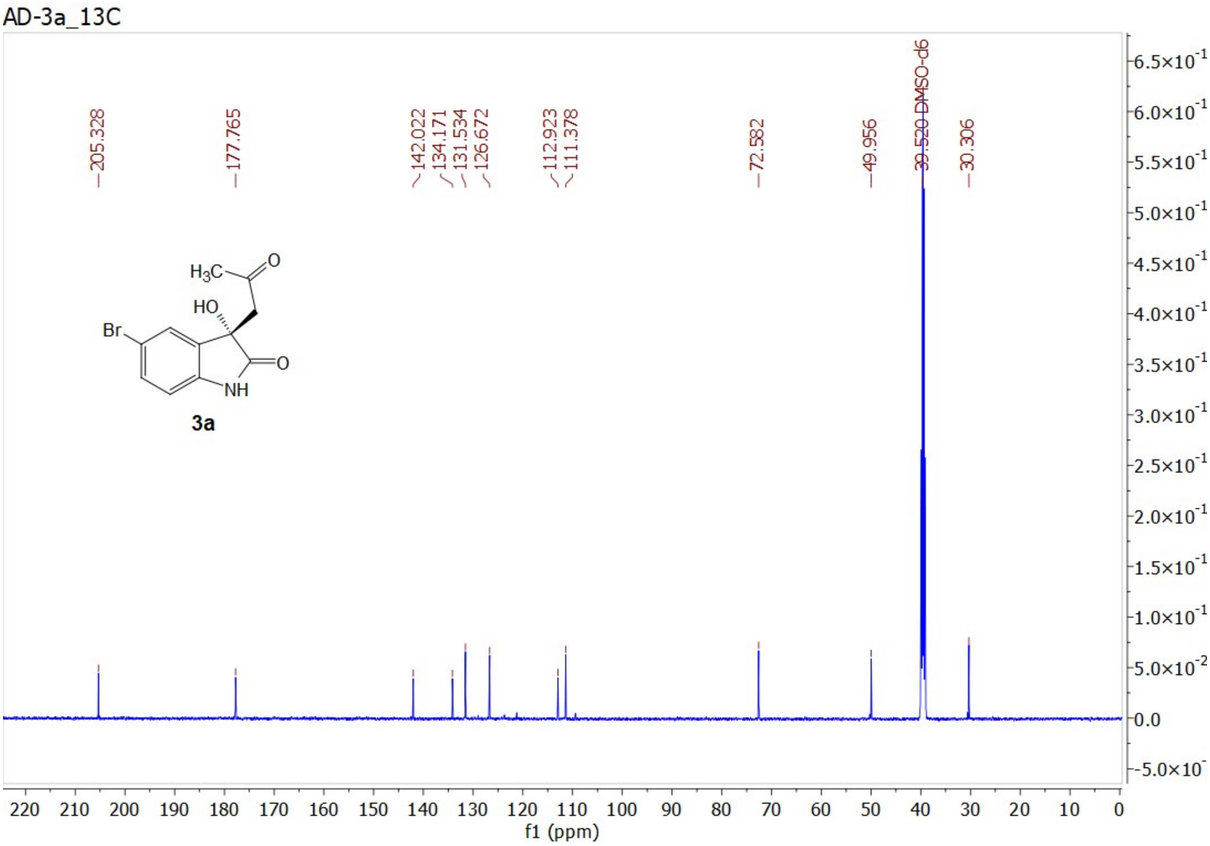
1.1.1 ^{1}H-NMR and ^{13}C-NMR of compound-3a





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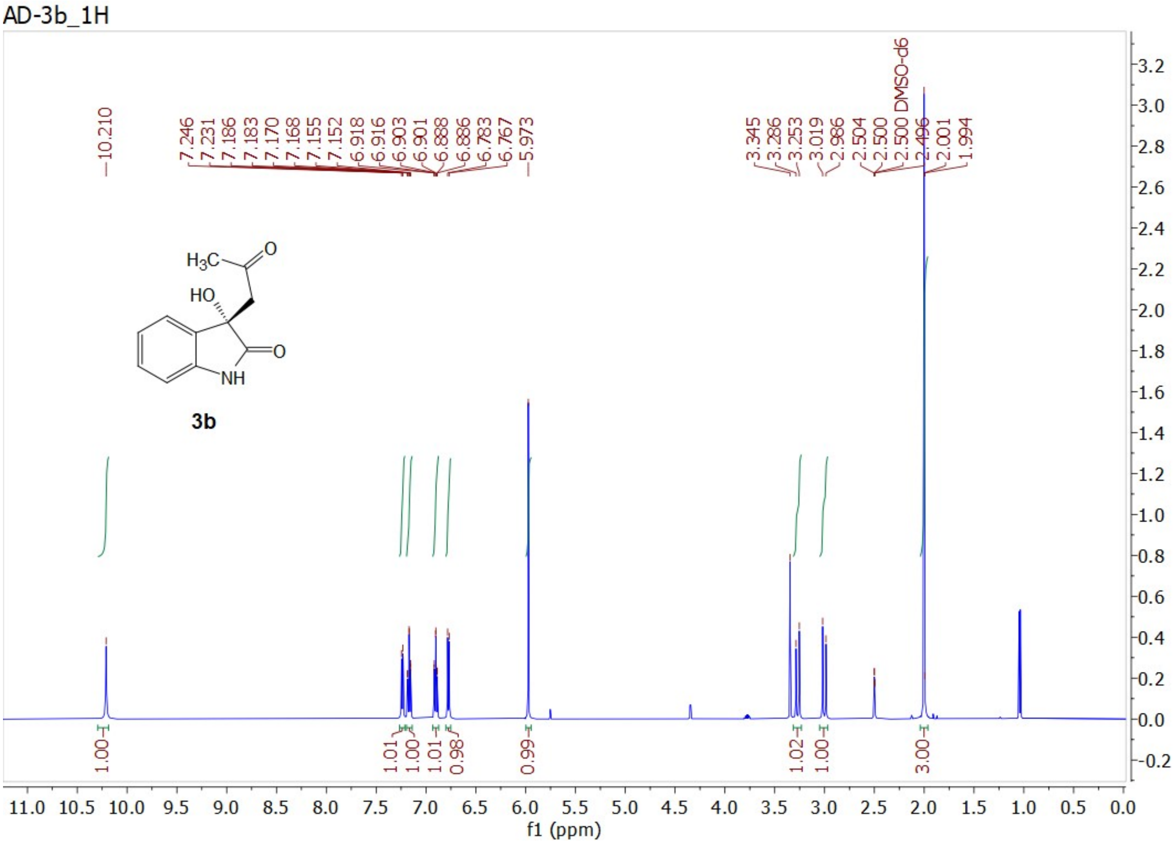

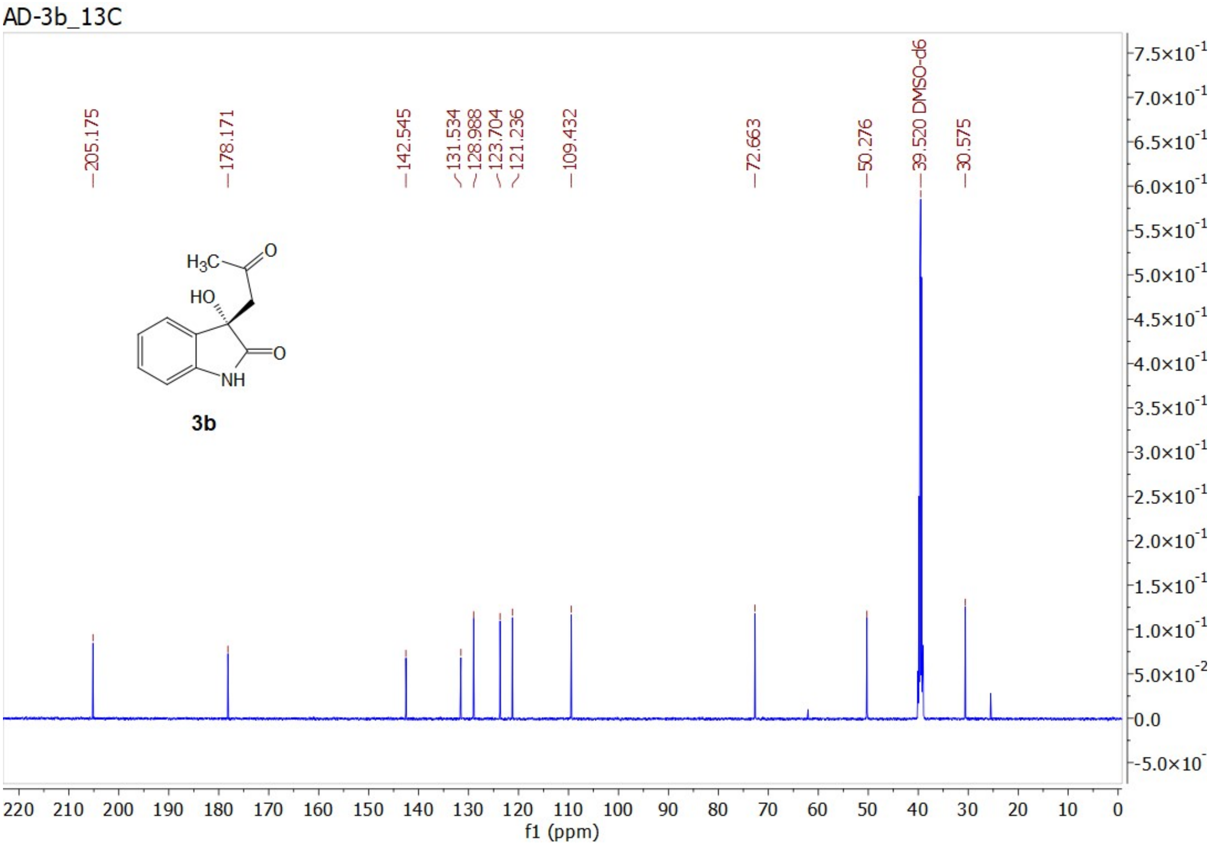

1.1.3 ^{1}H-NMR and ^{13}C-NMR for compound-3c

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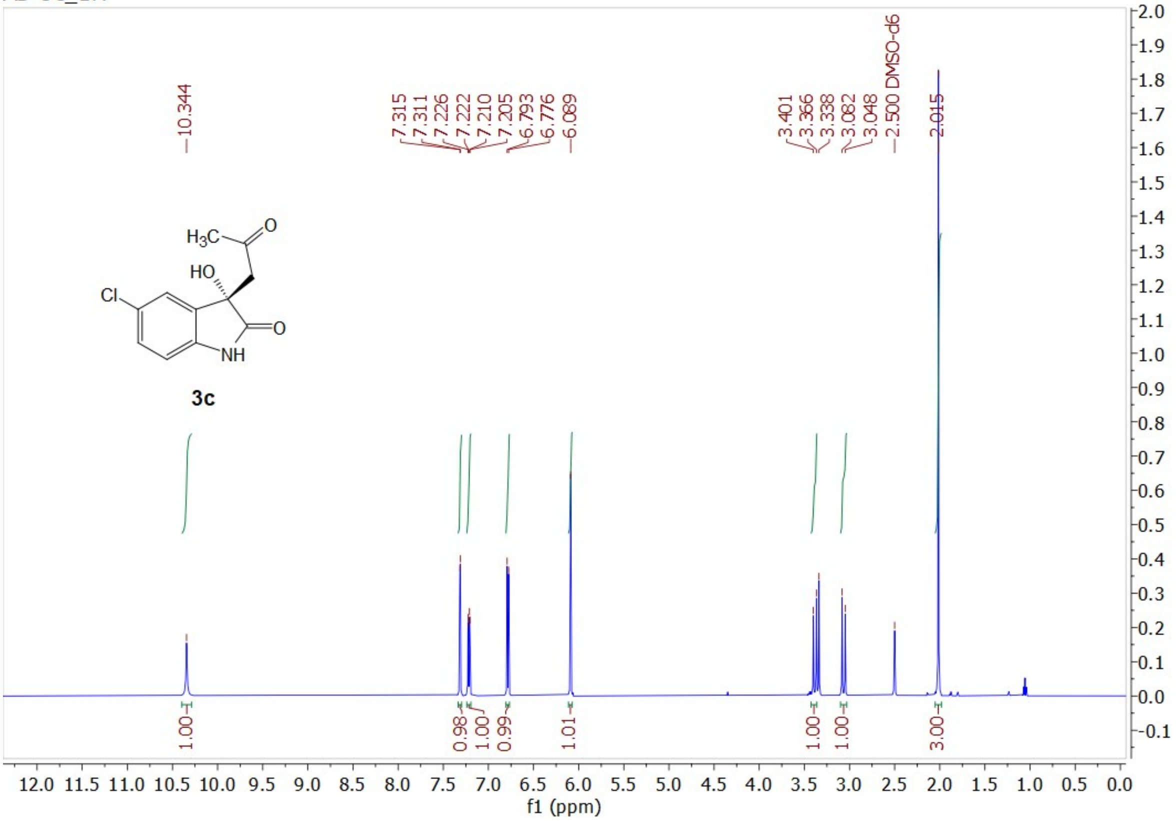

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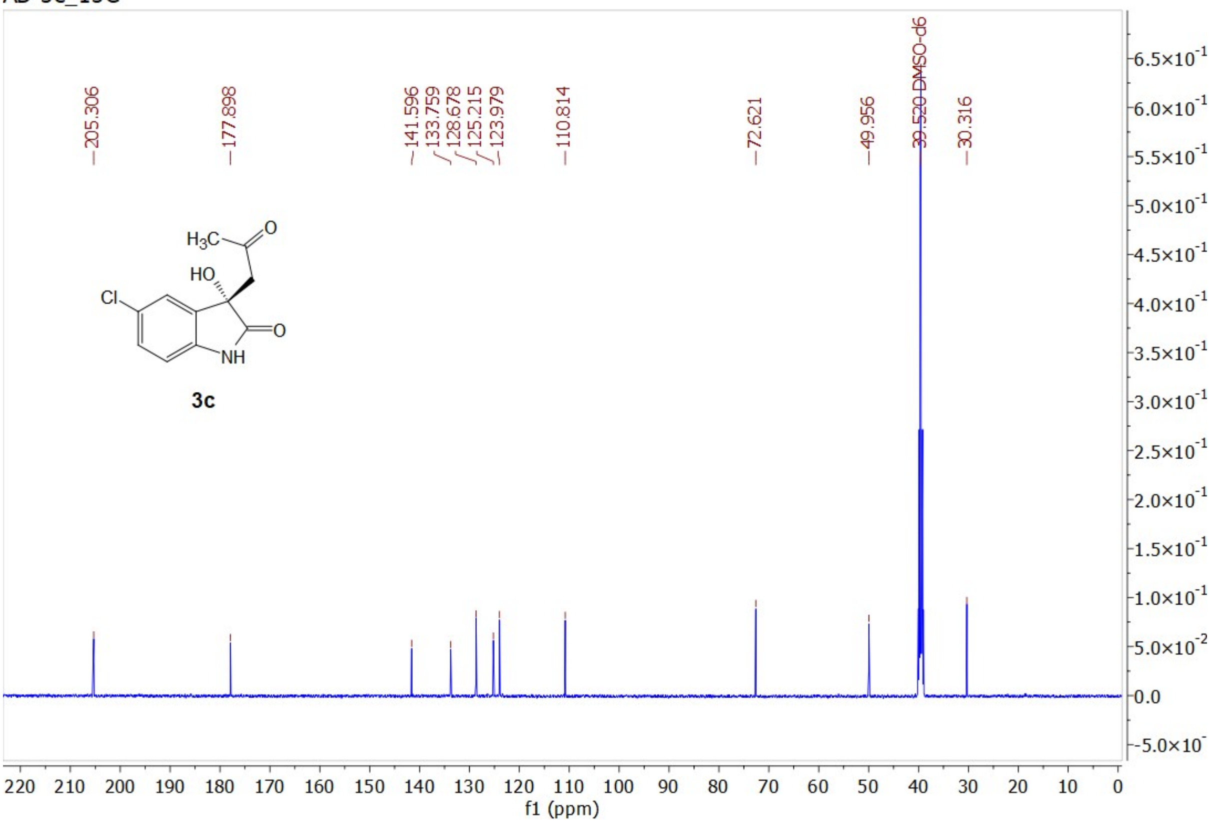

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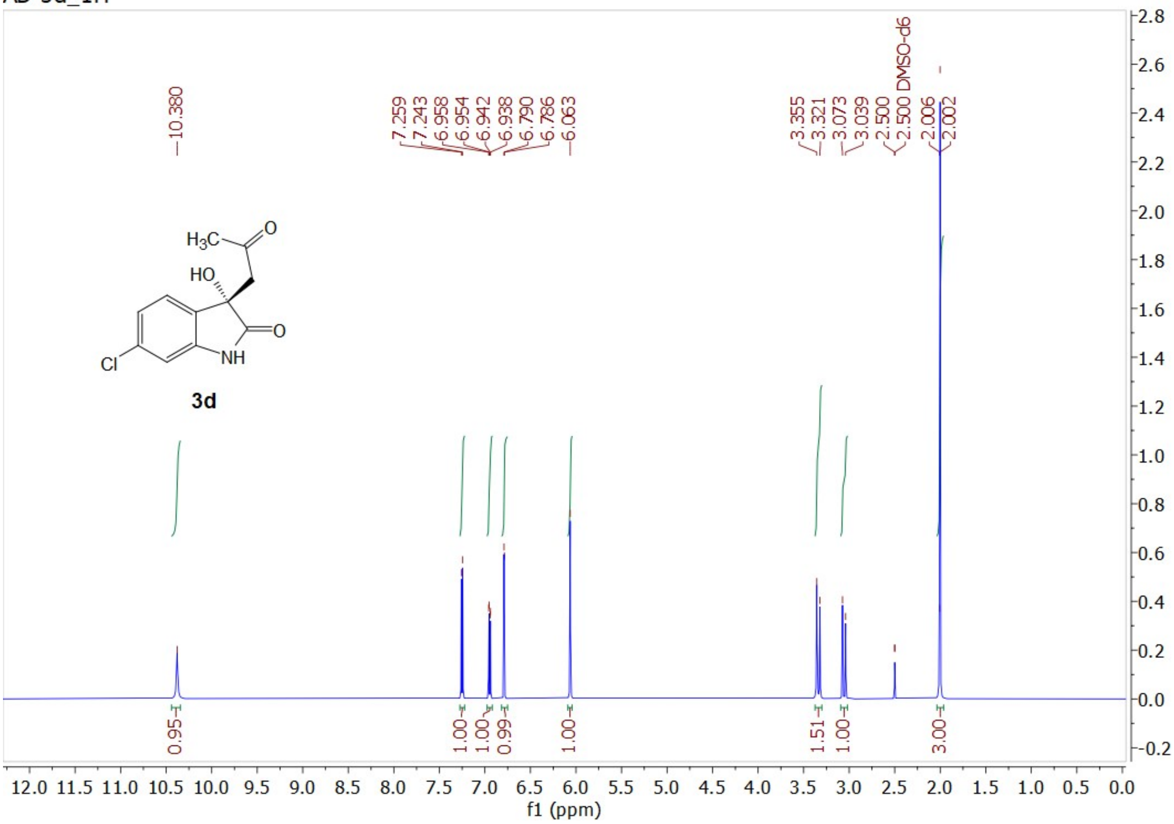

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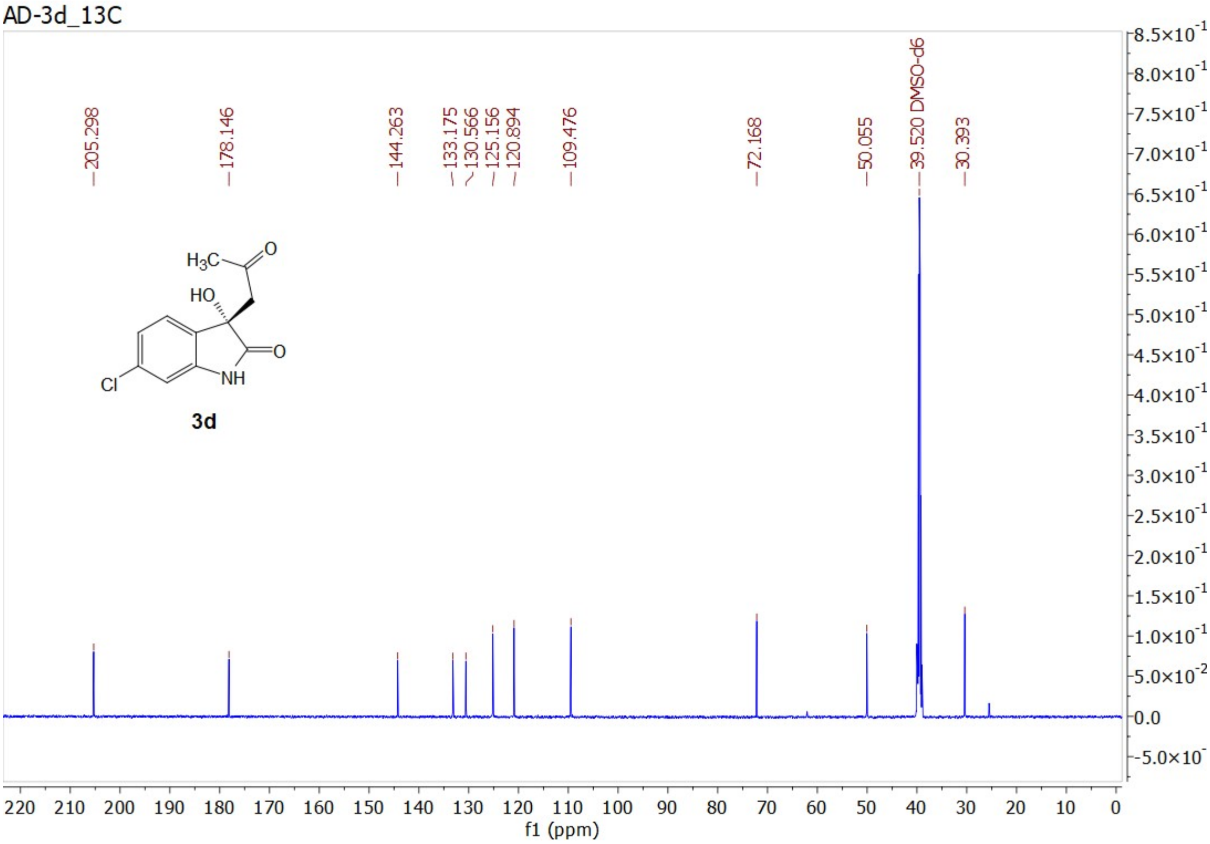

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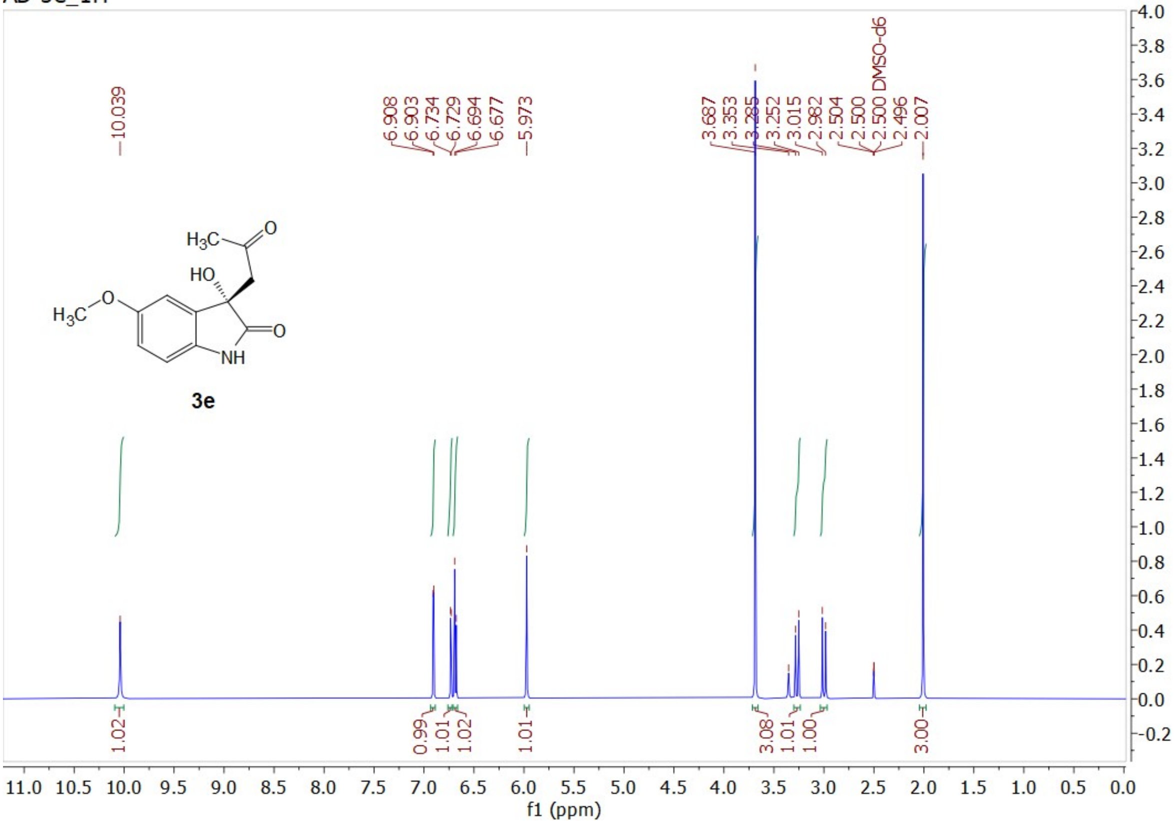

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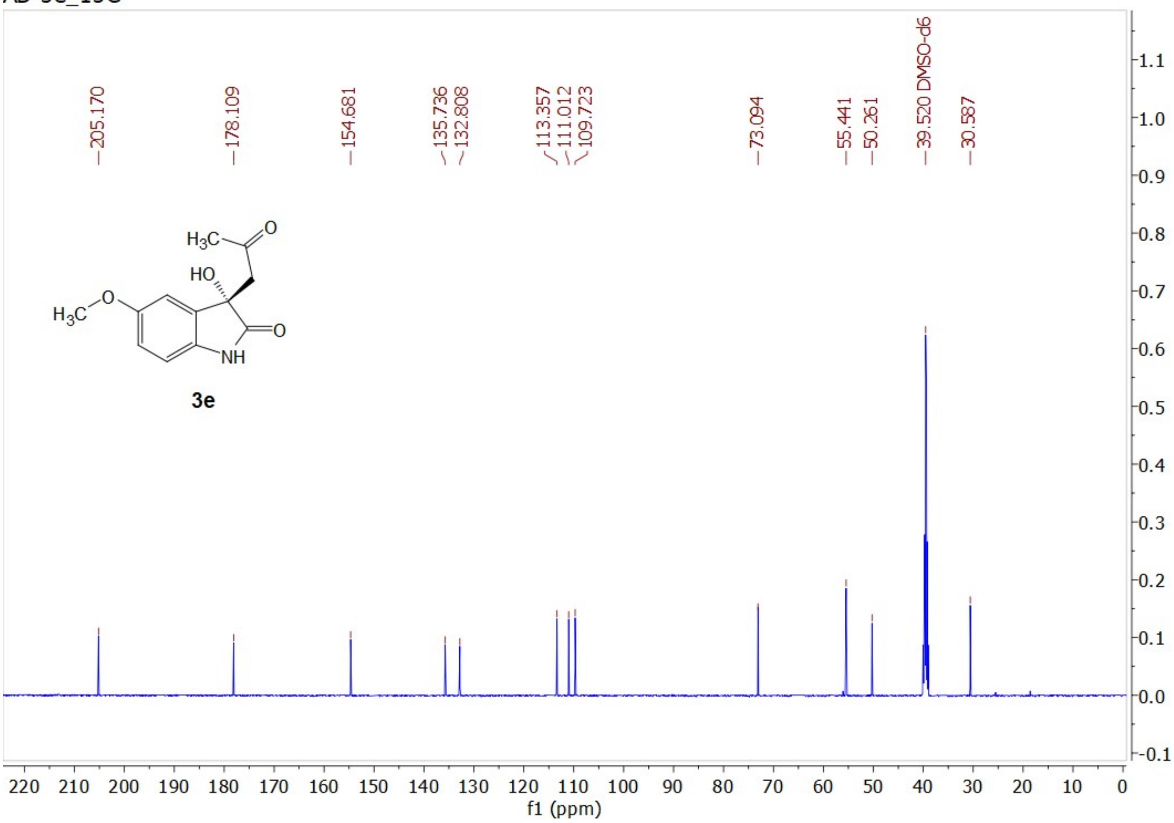

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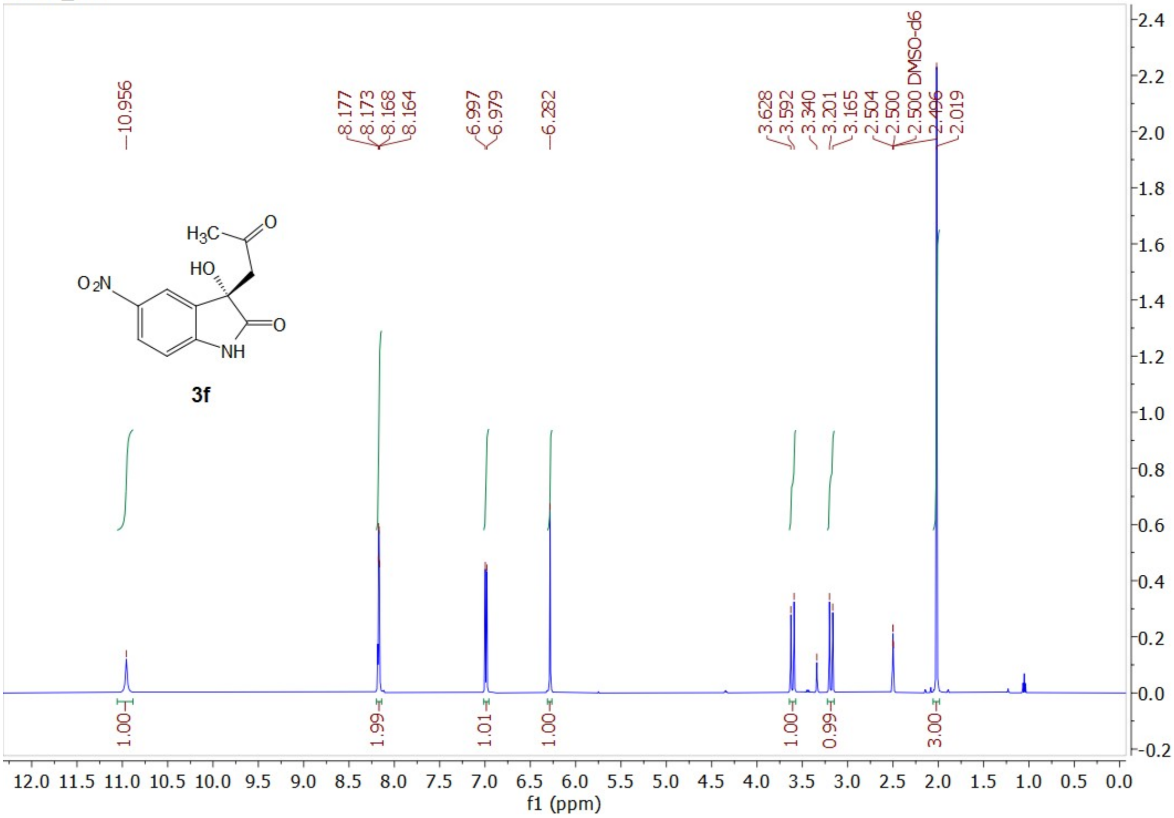

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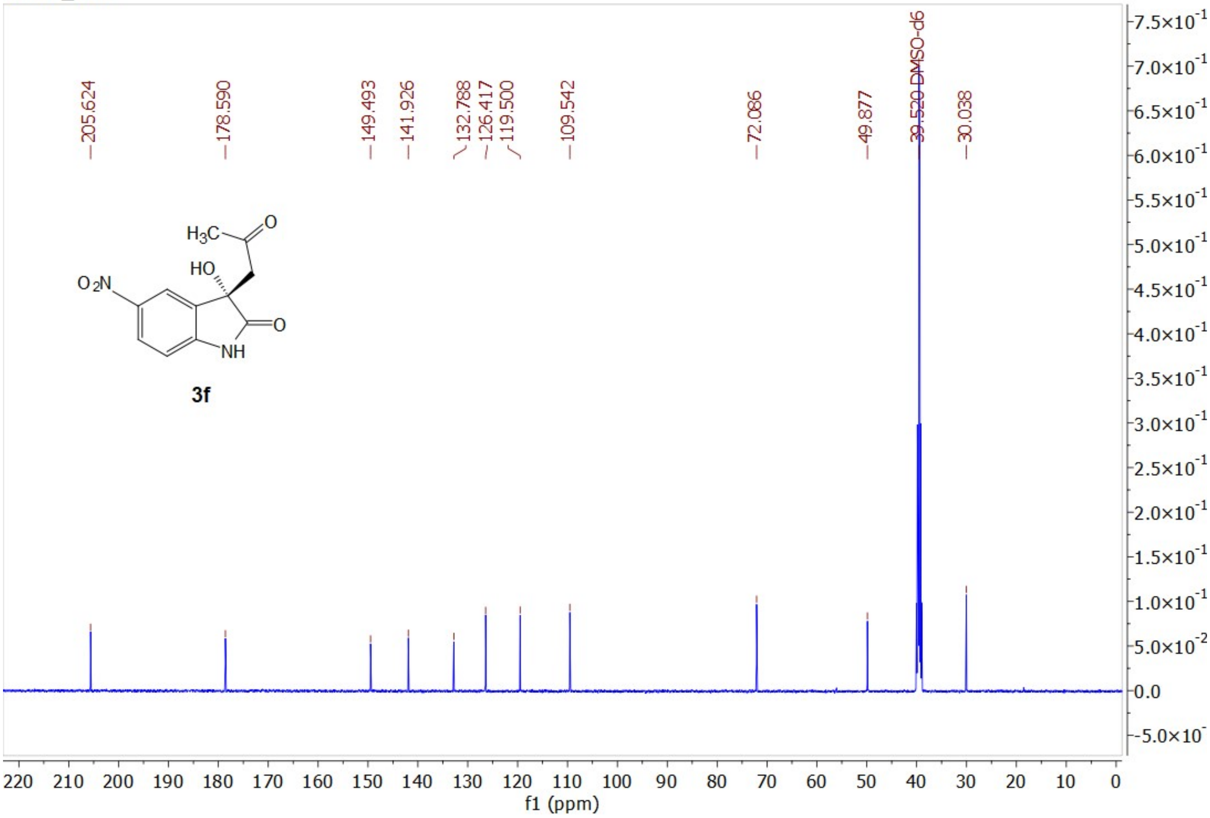

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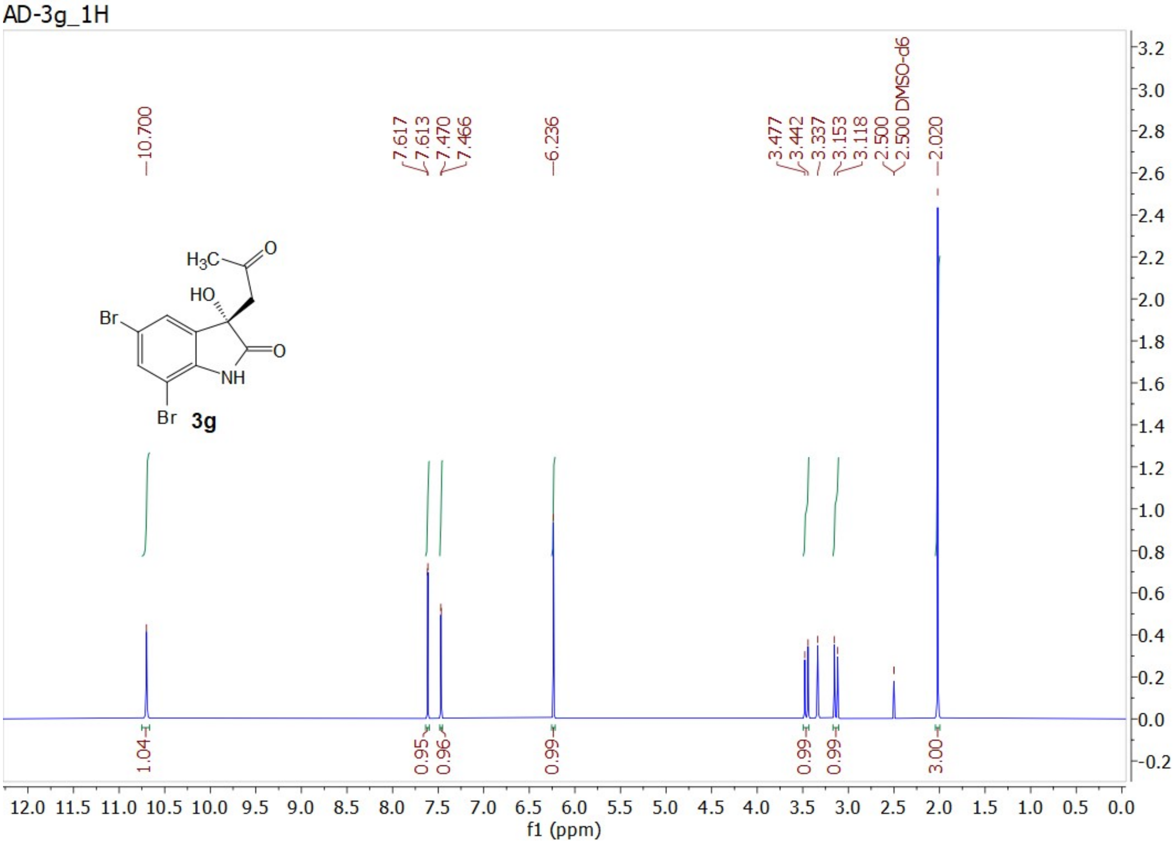

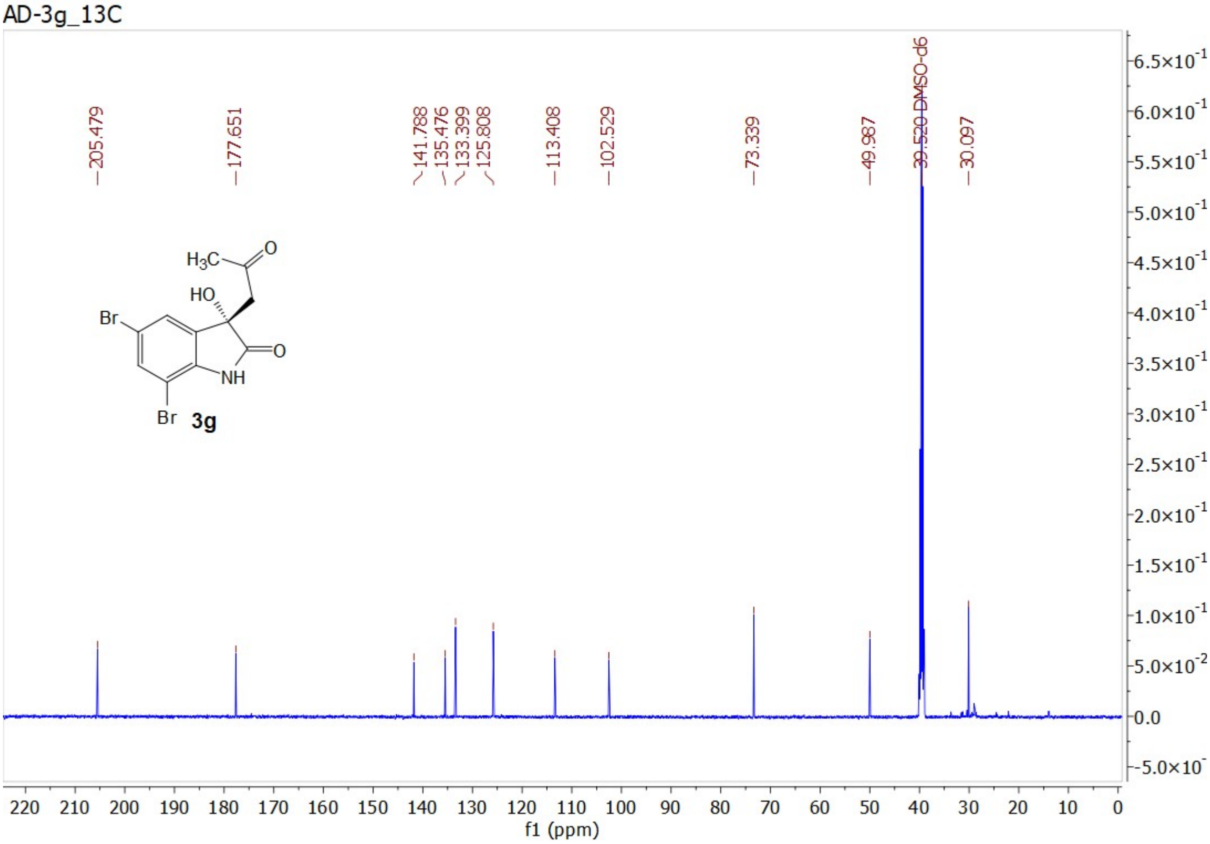

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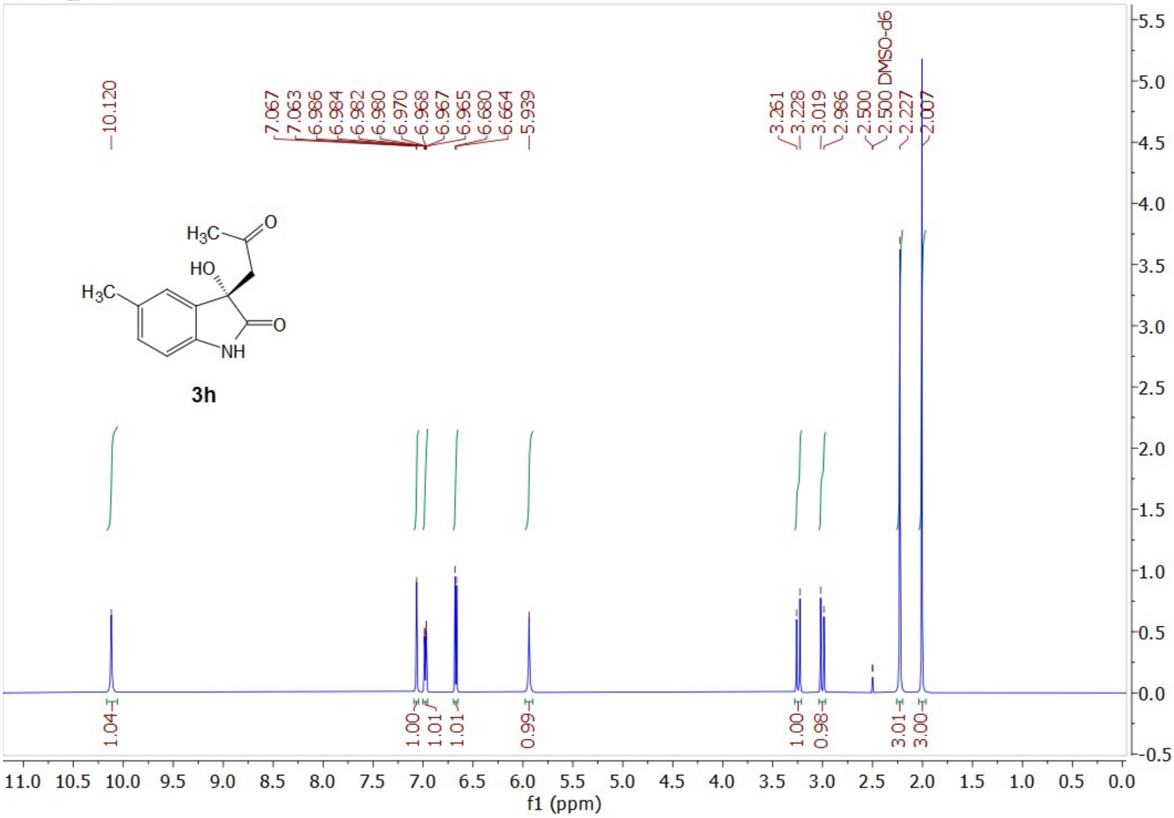

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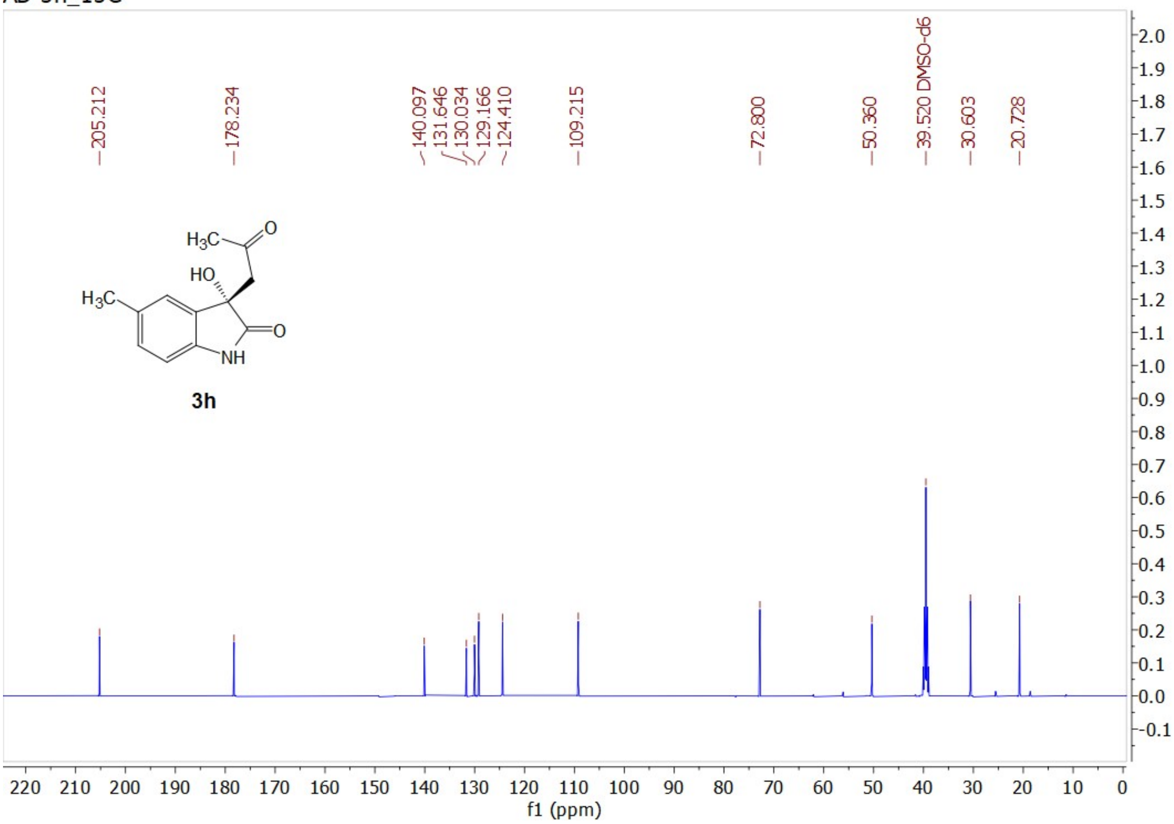
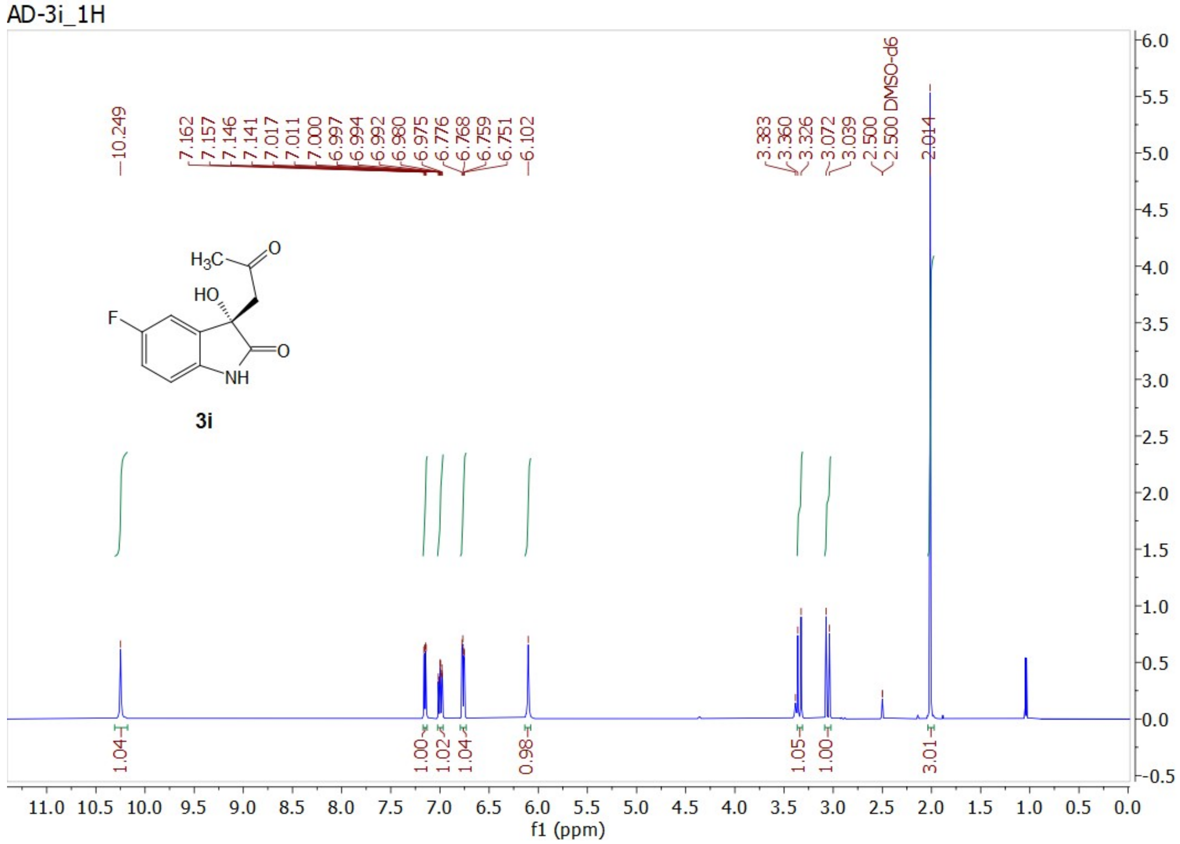
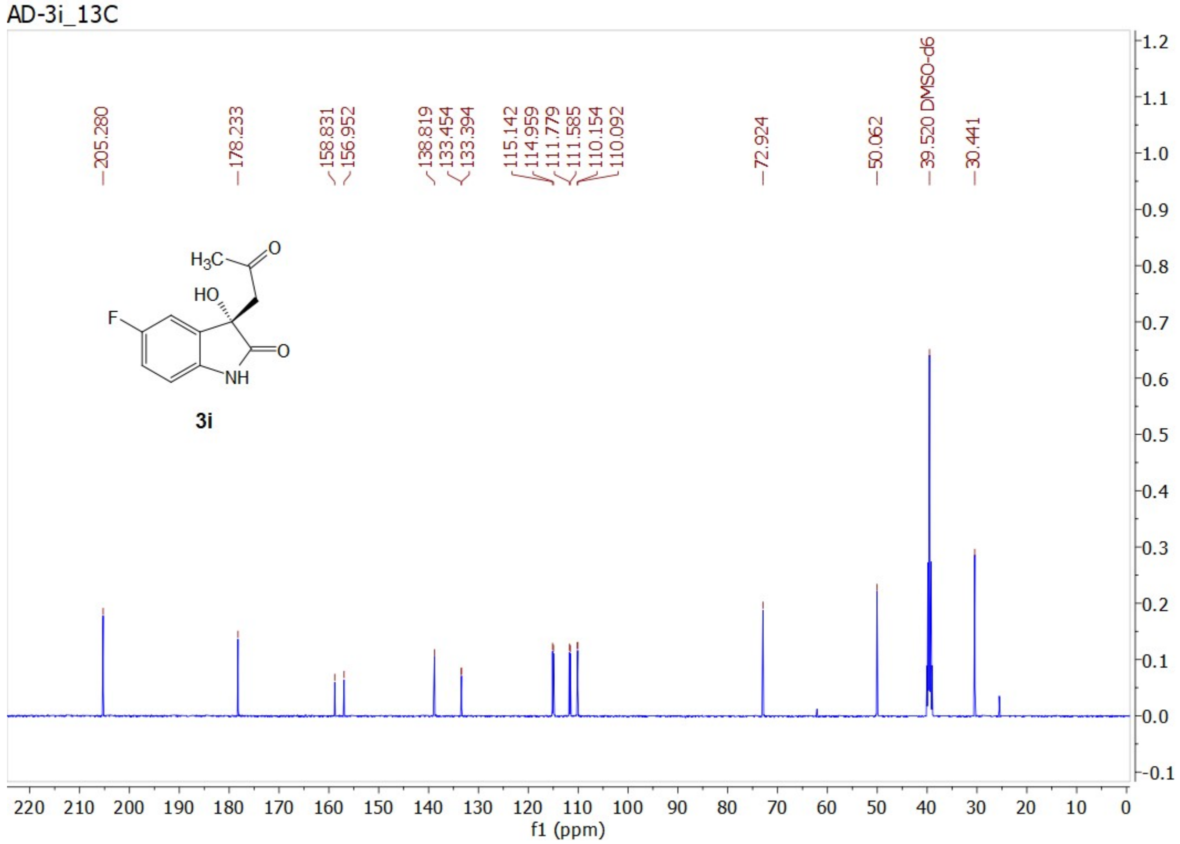


1.1.9 ^{1}H-NMR and ^{13}C-NMR for compound-3i





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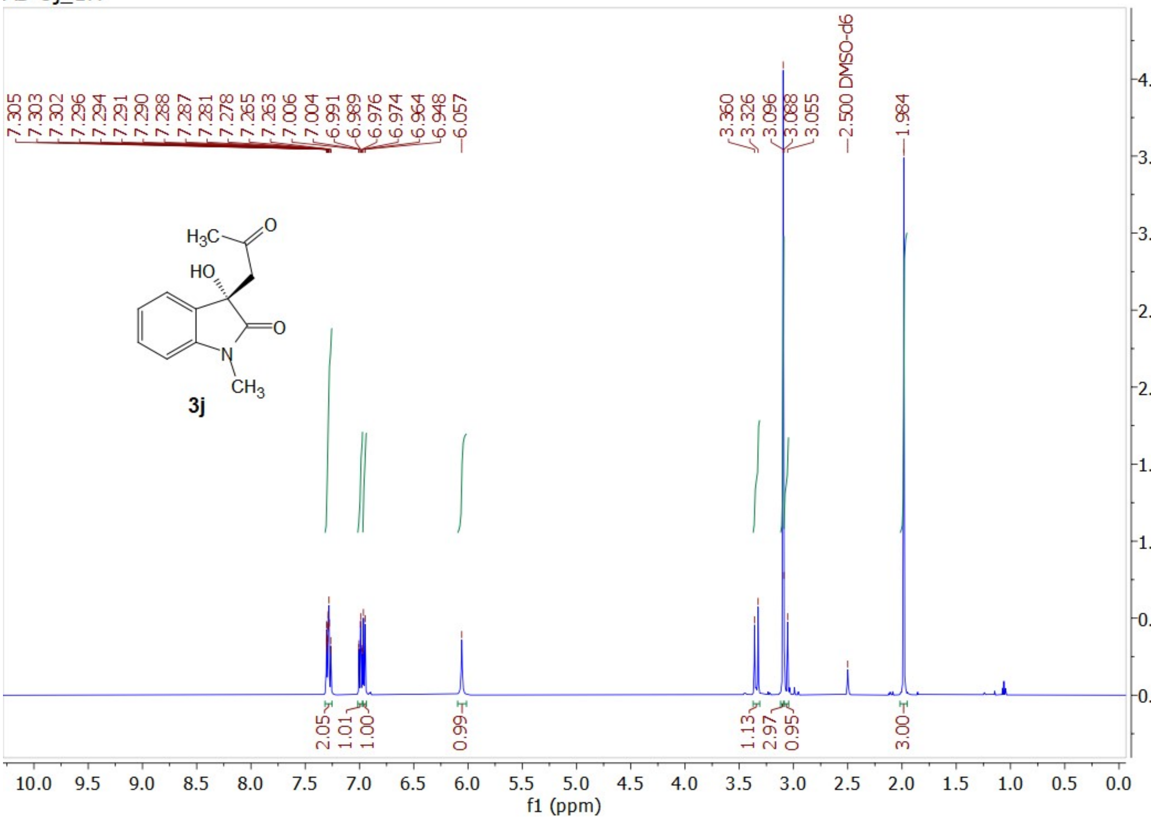

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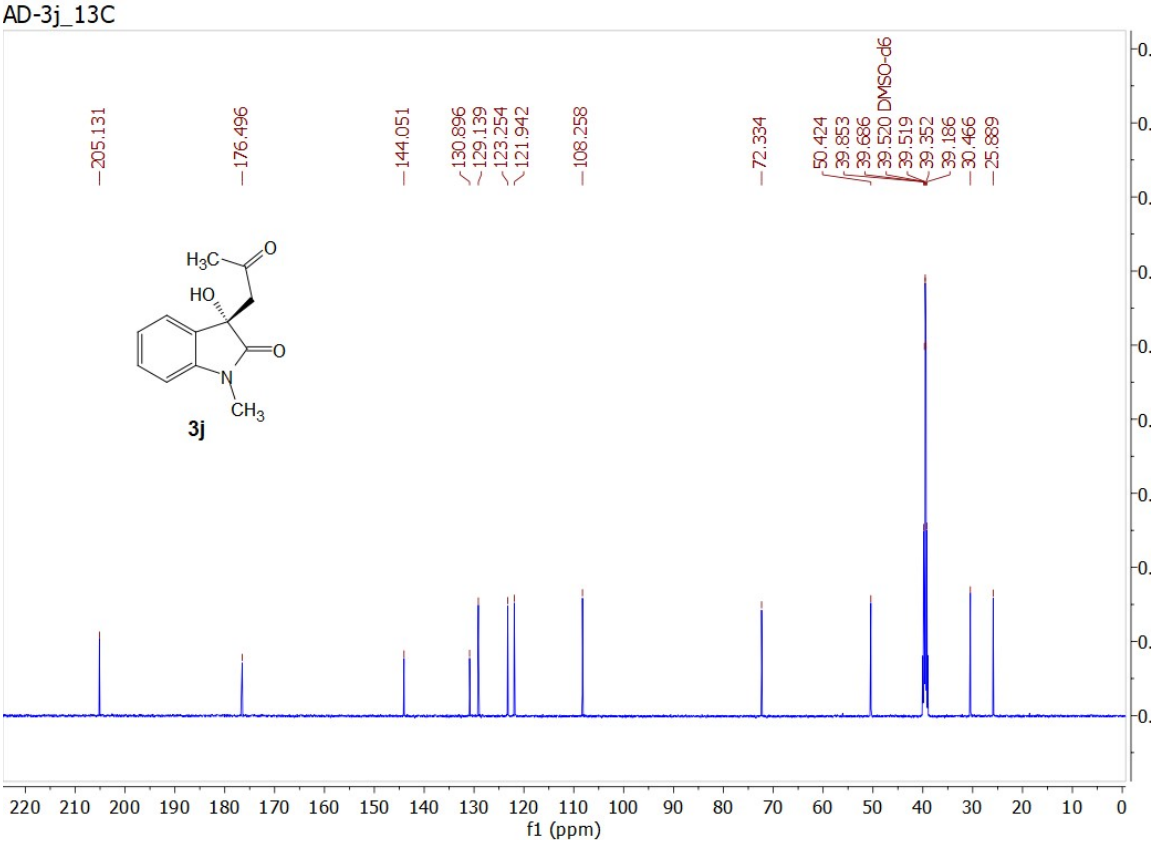

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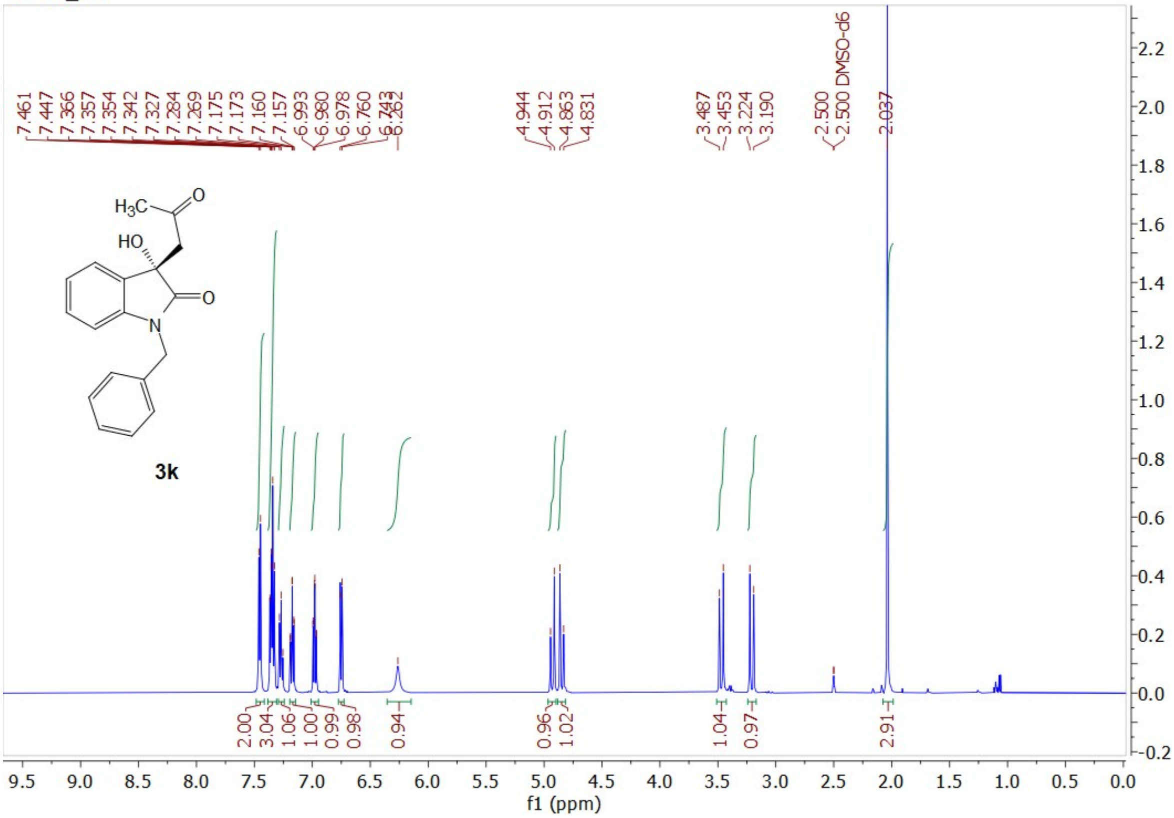

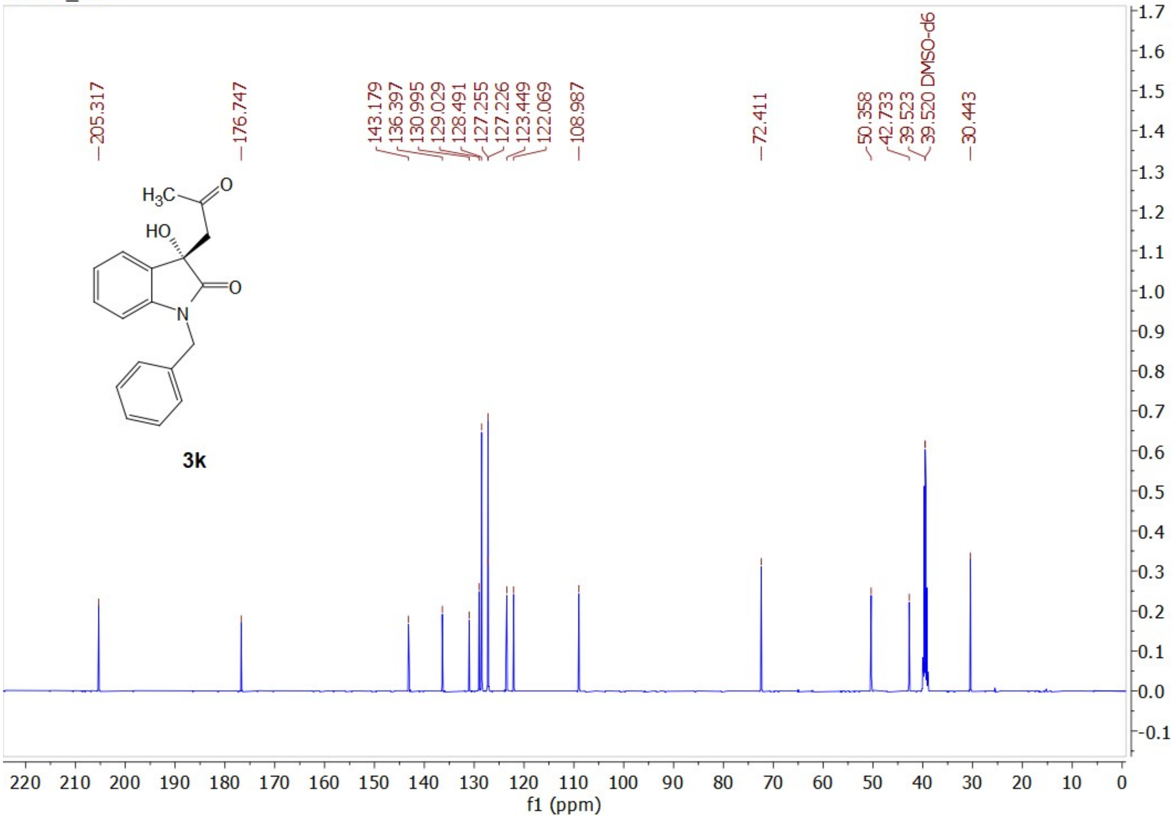

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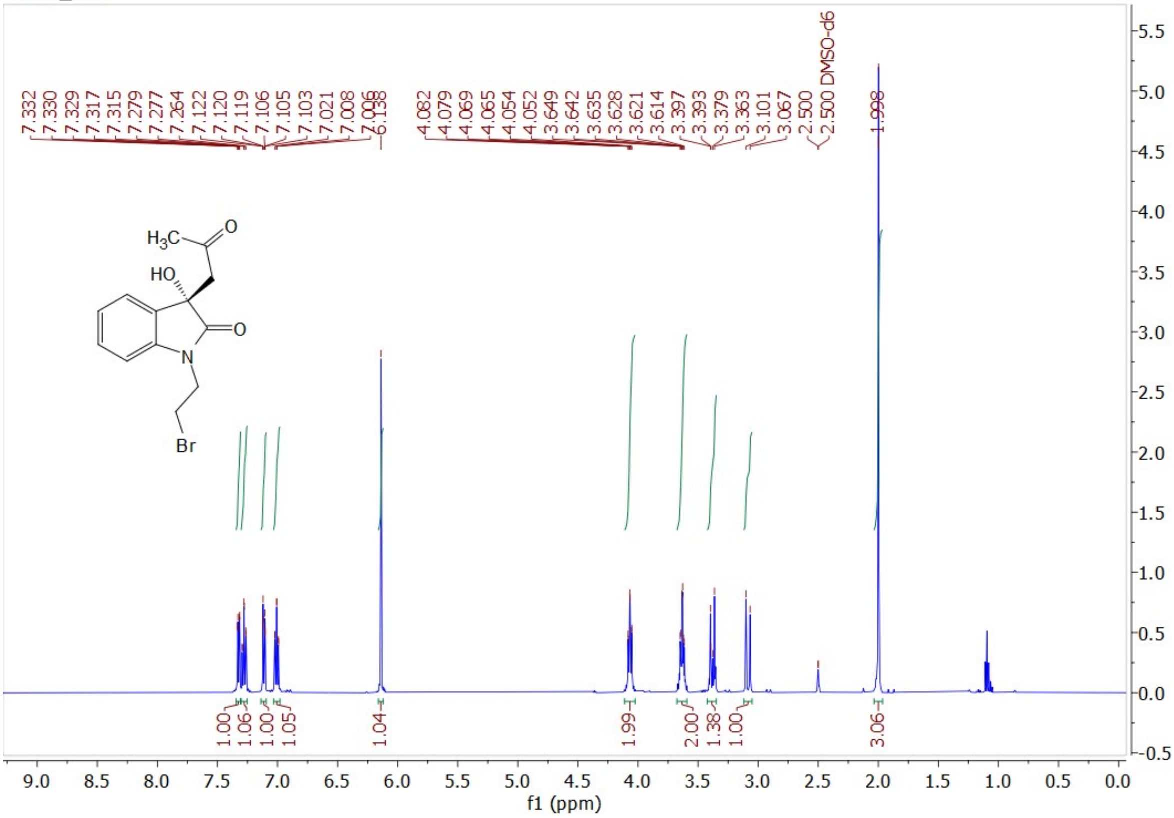

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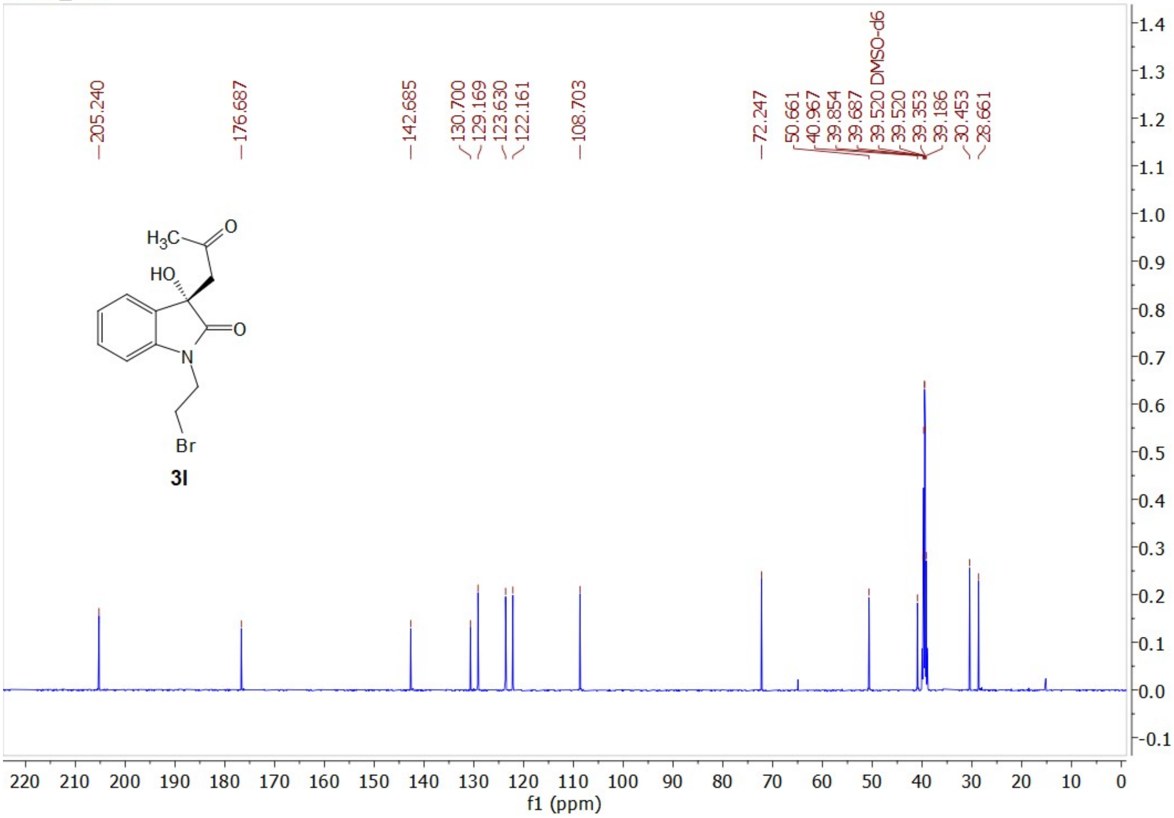

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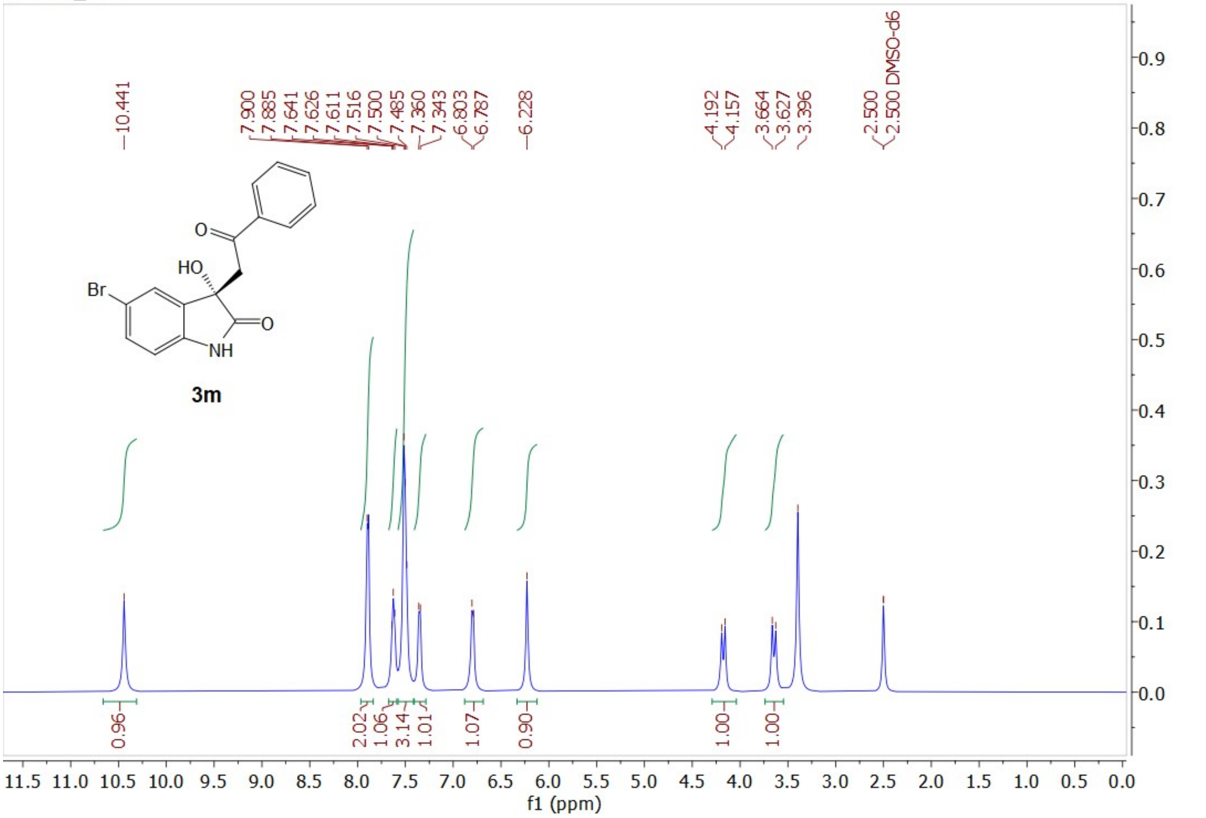
AD-3m_1H



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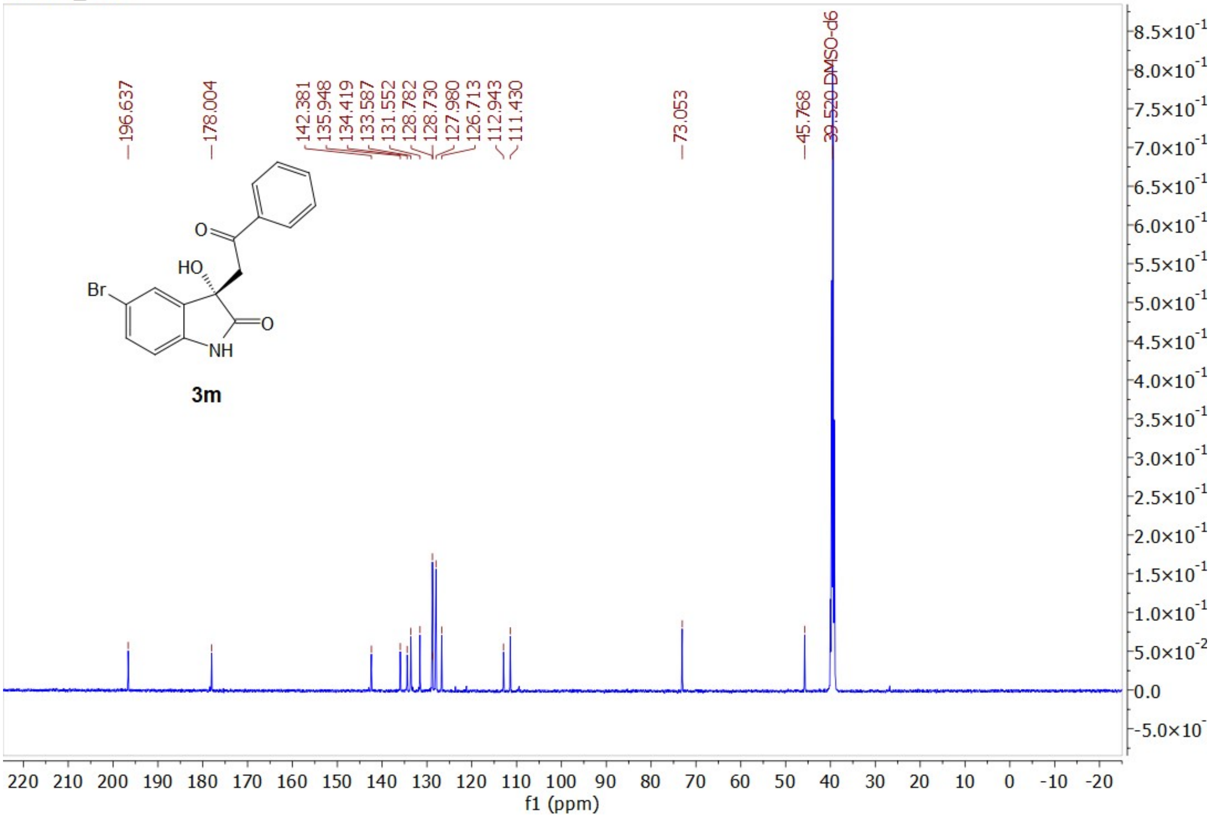

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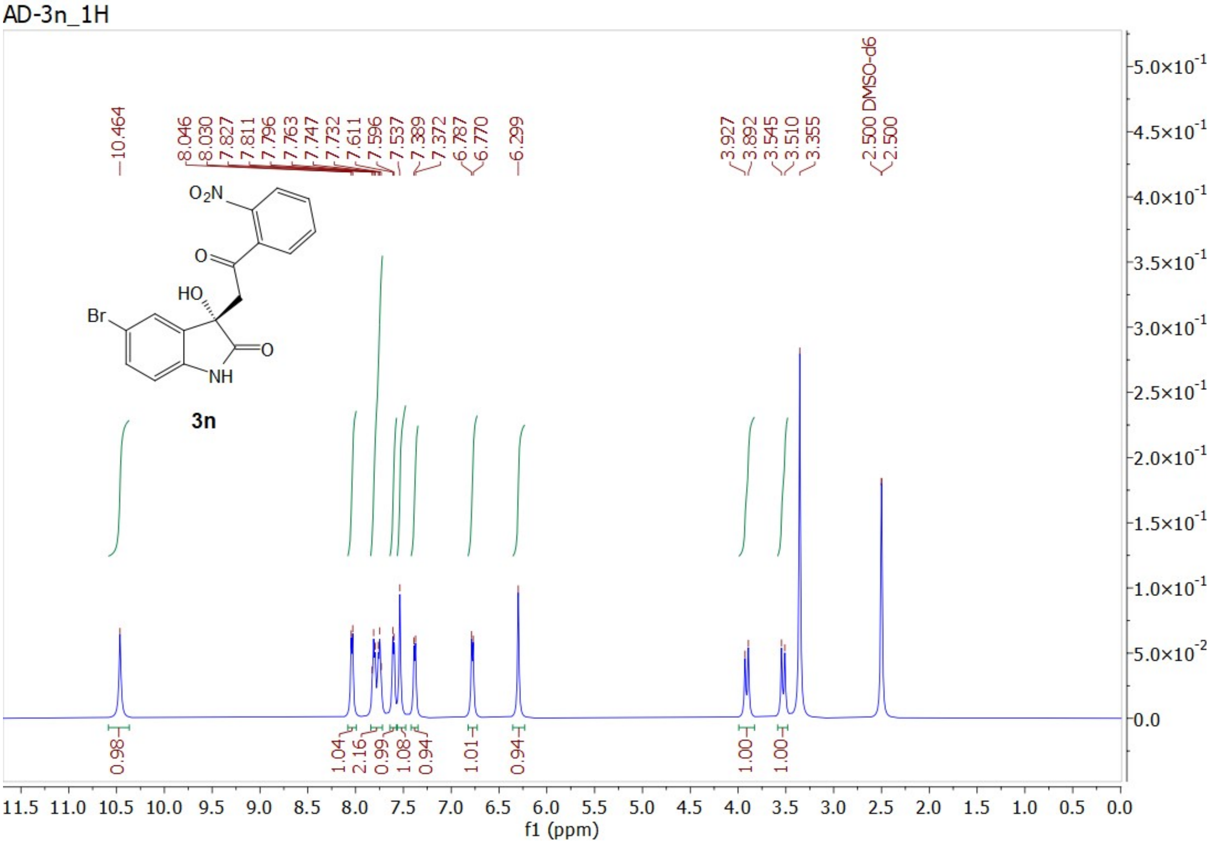

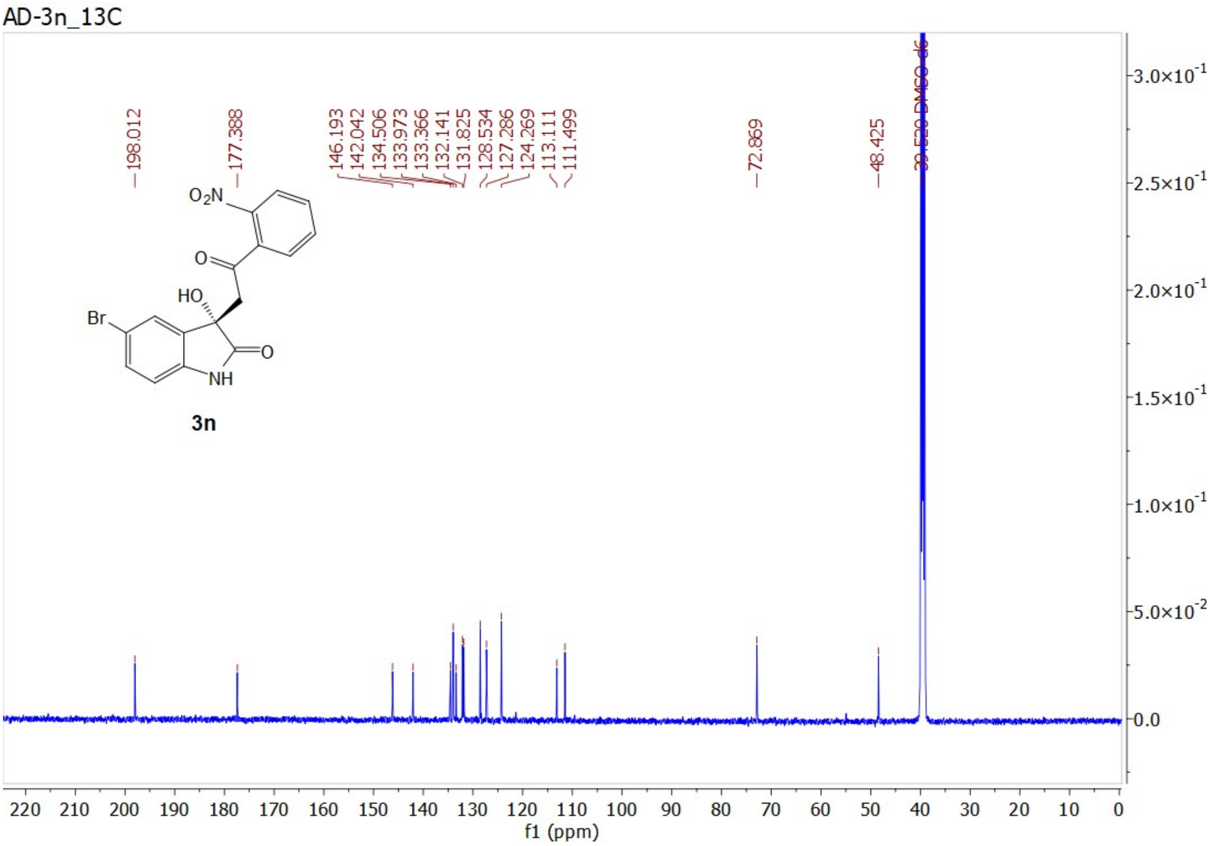

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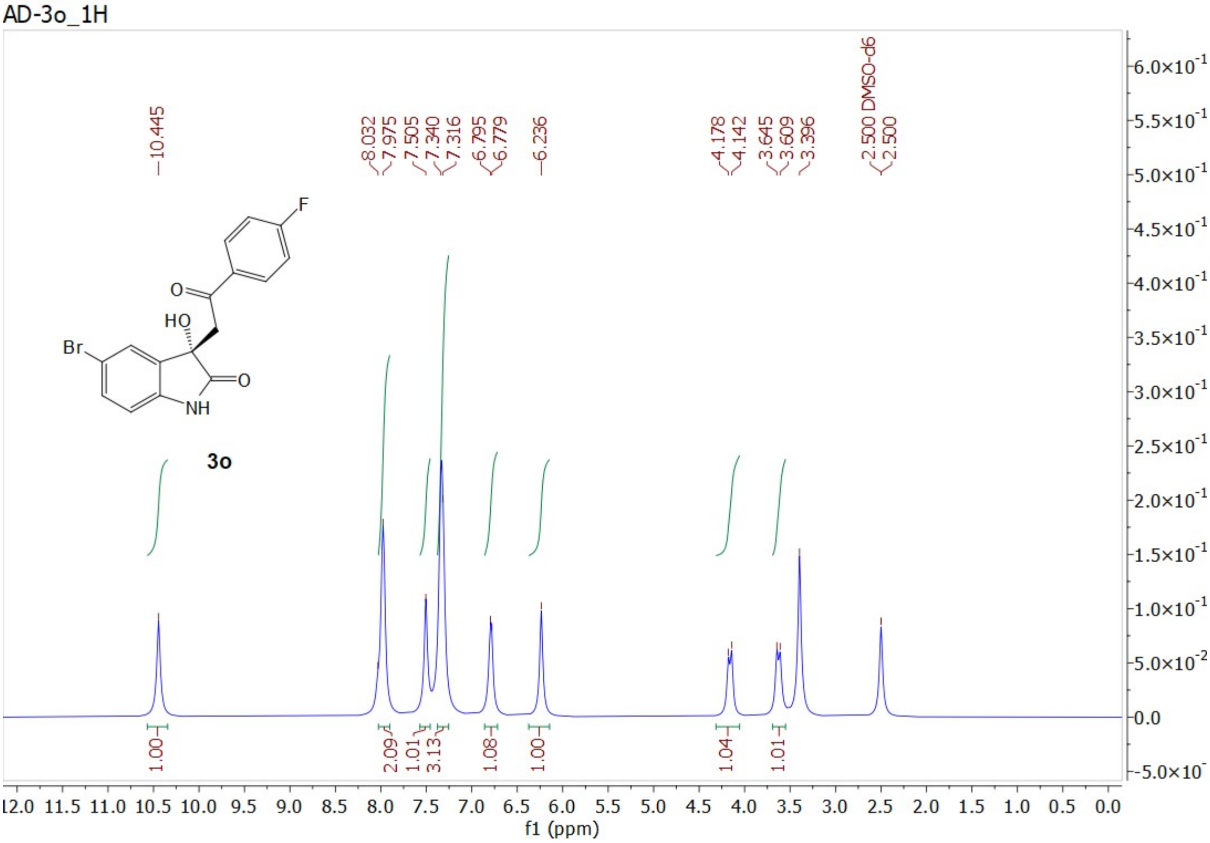
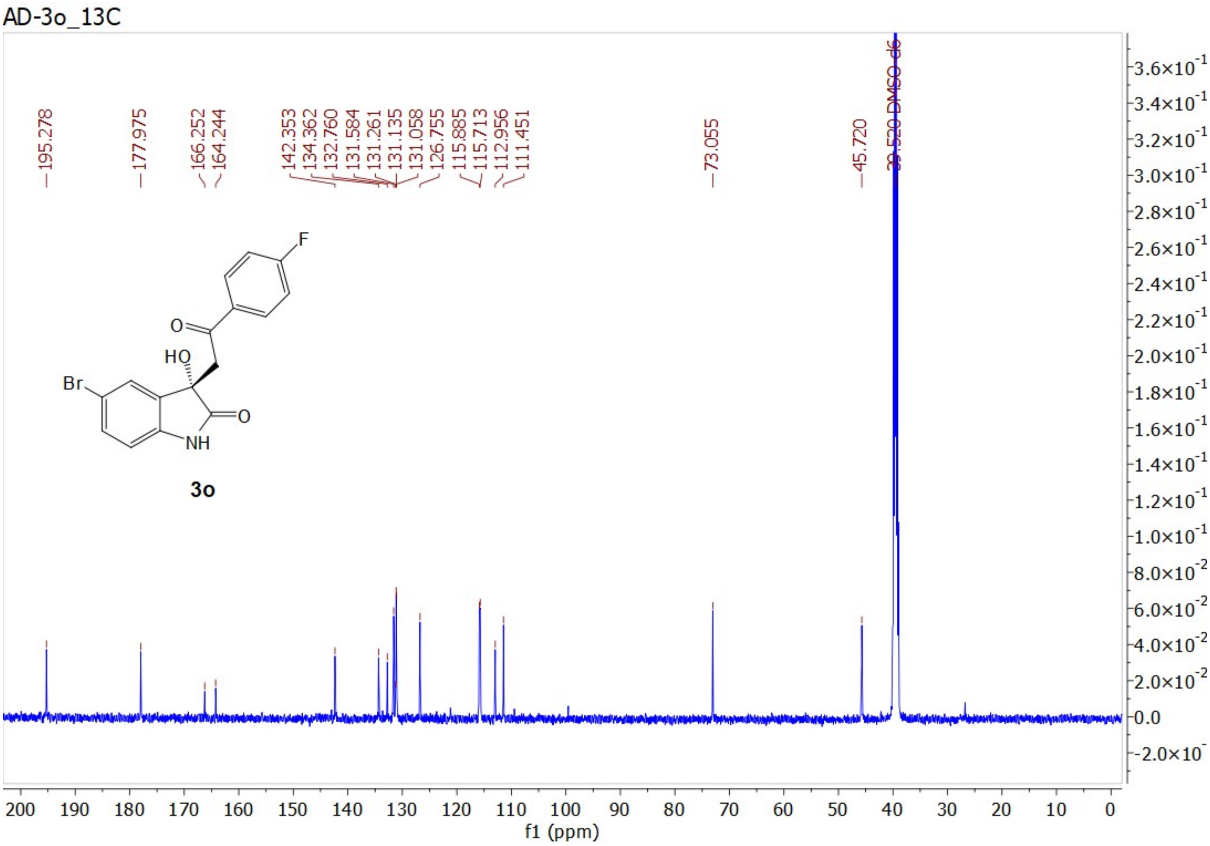
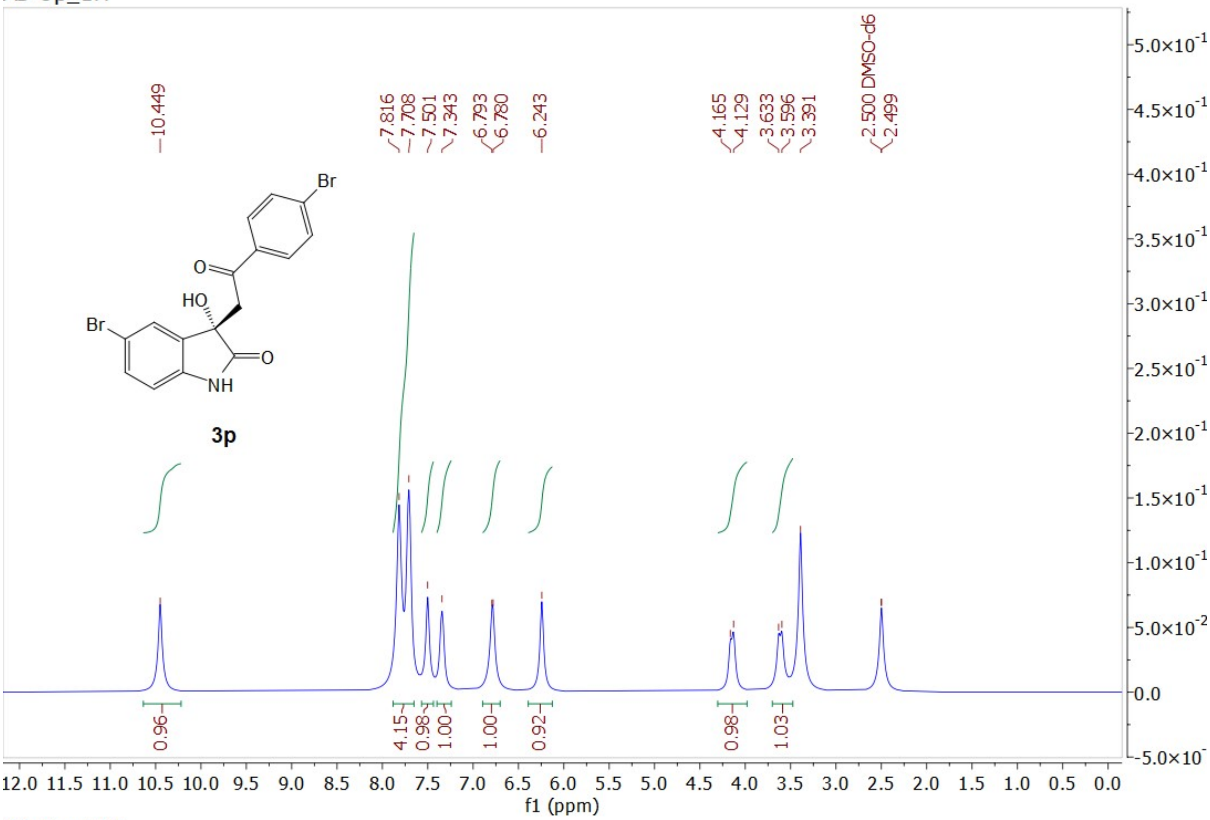


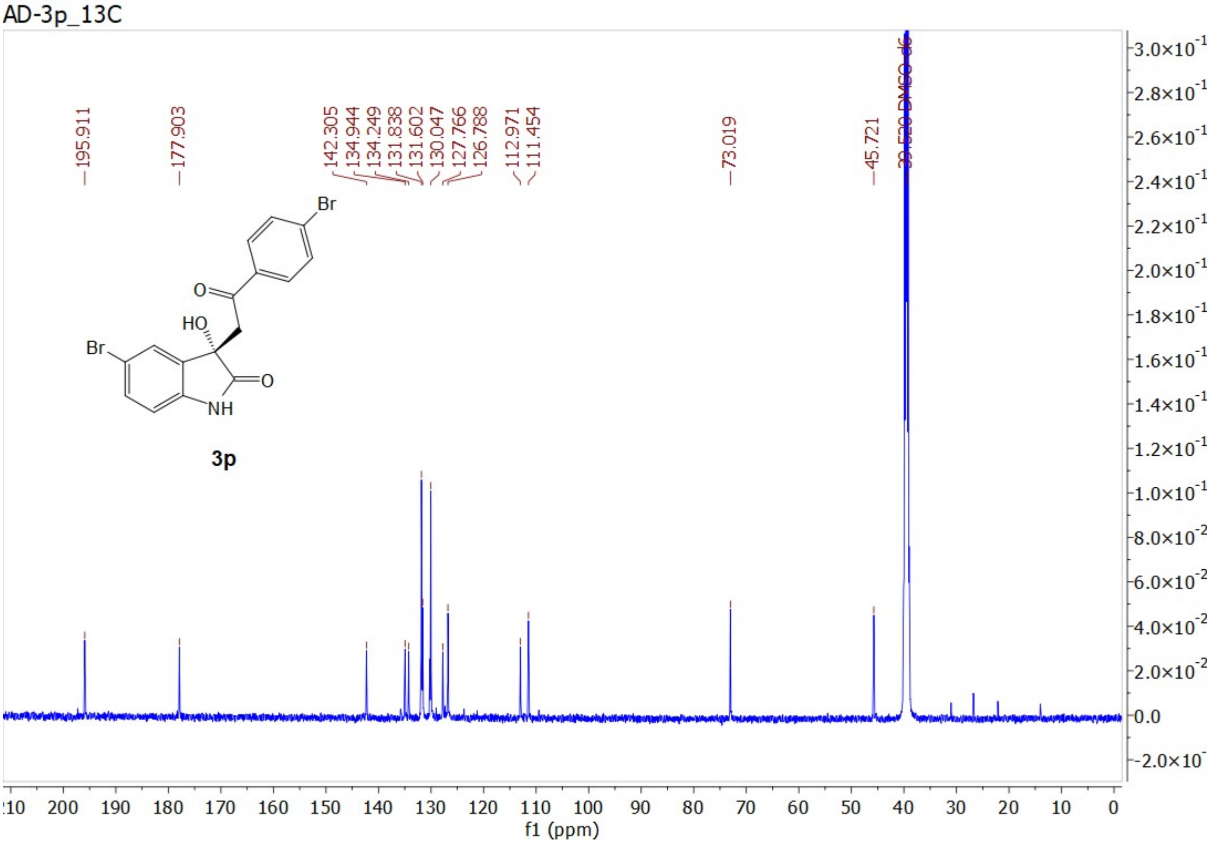


1.1.16 ^{1}H-NMR and ^{13}C-NMR for compound-3p

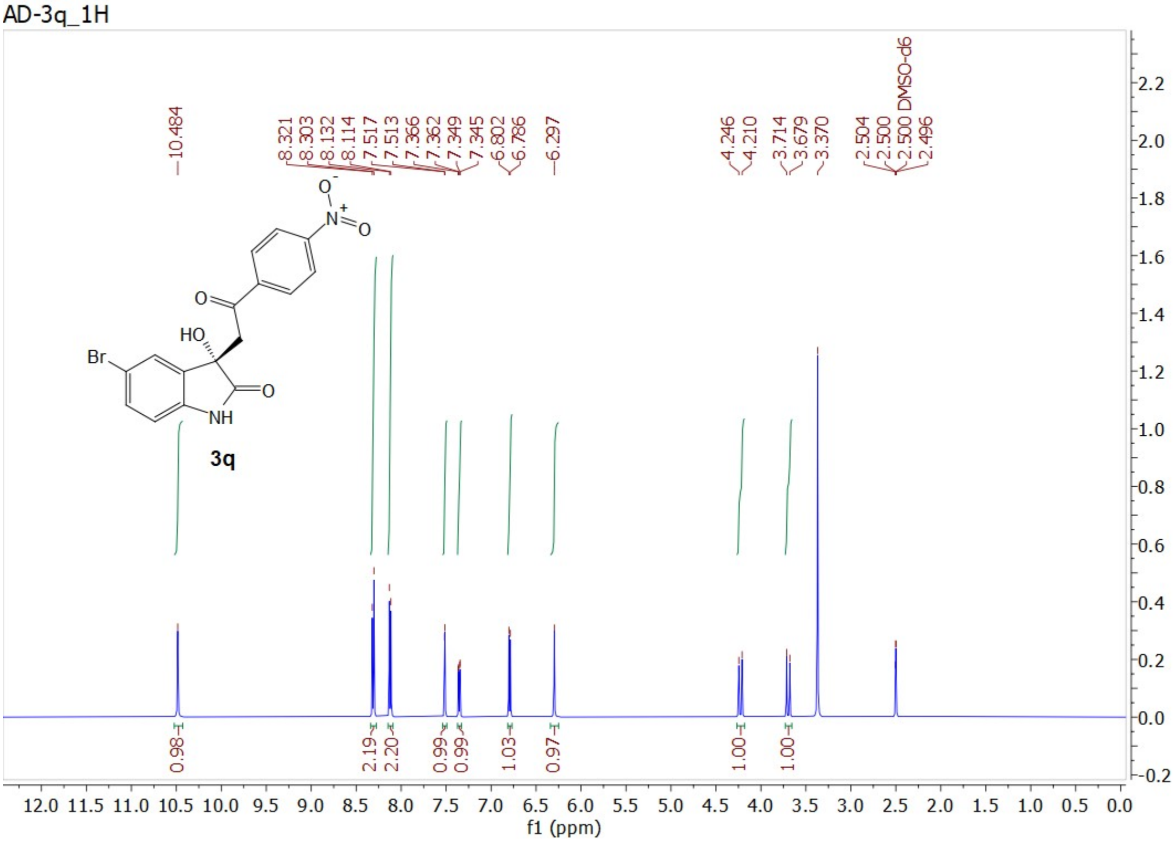
AD-3p_1H

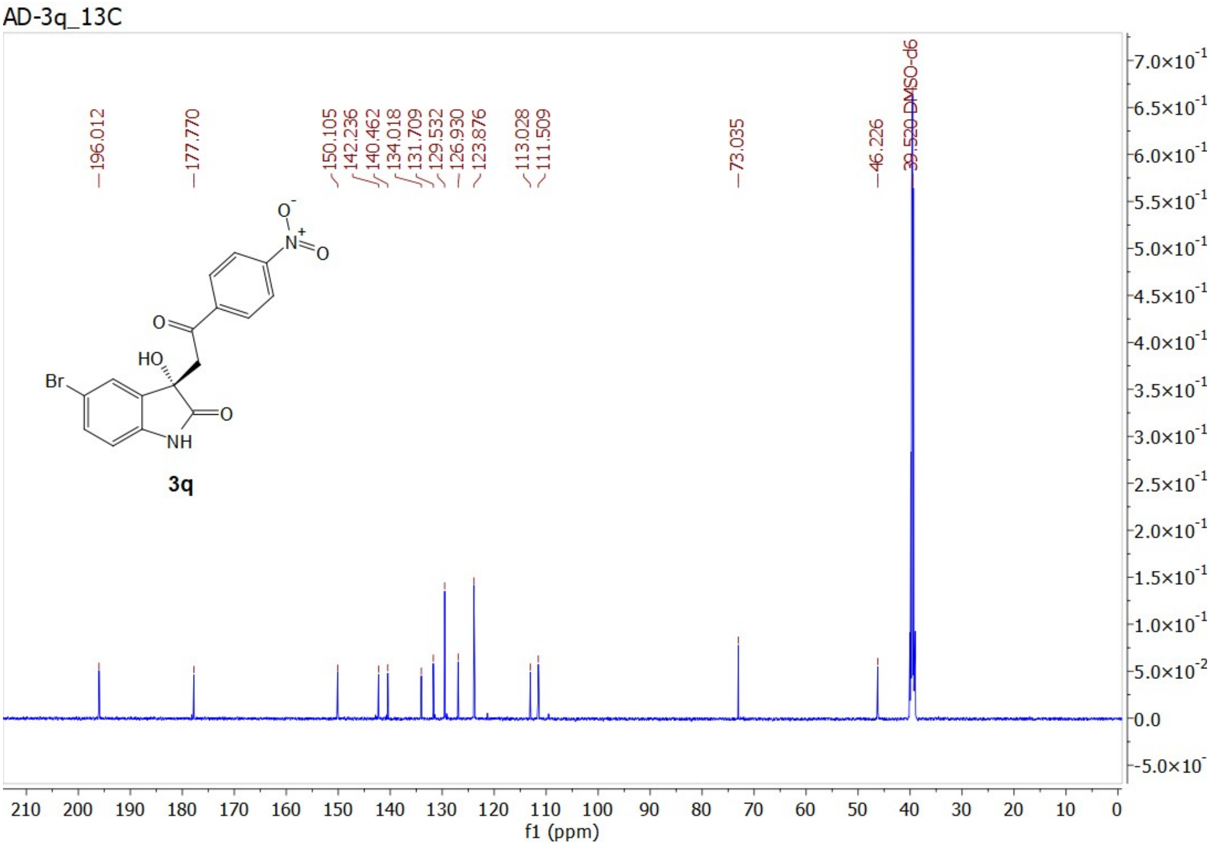


AD-3p_13C

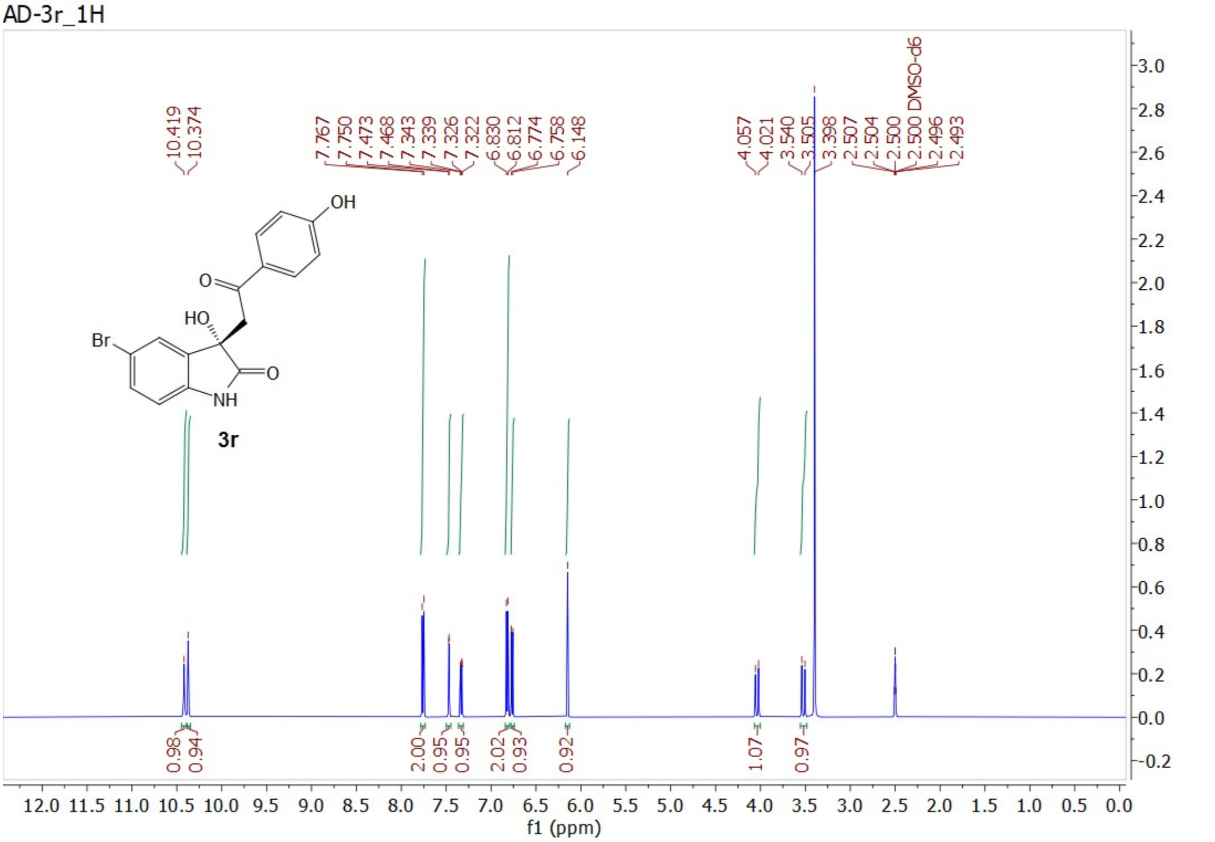


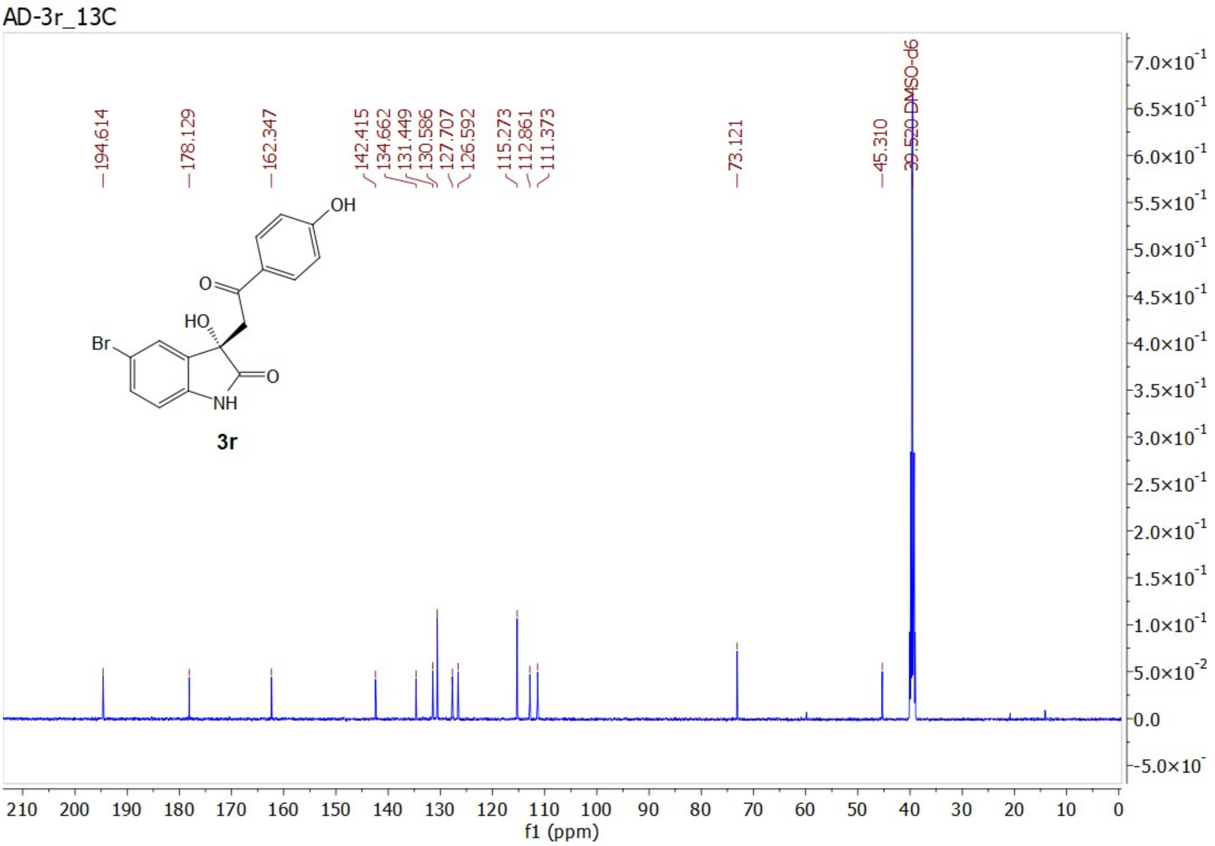
1.1.17 ^{1}H-NMR and ^{13}C-NMR for compound-3q





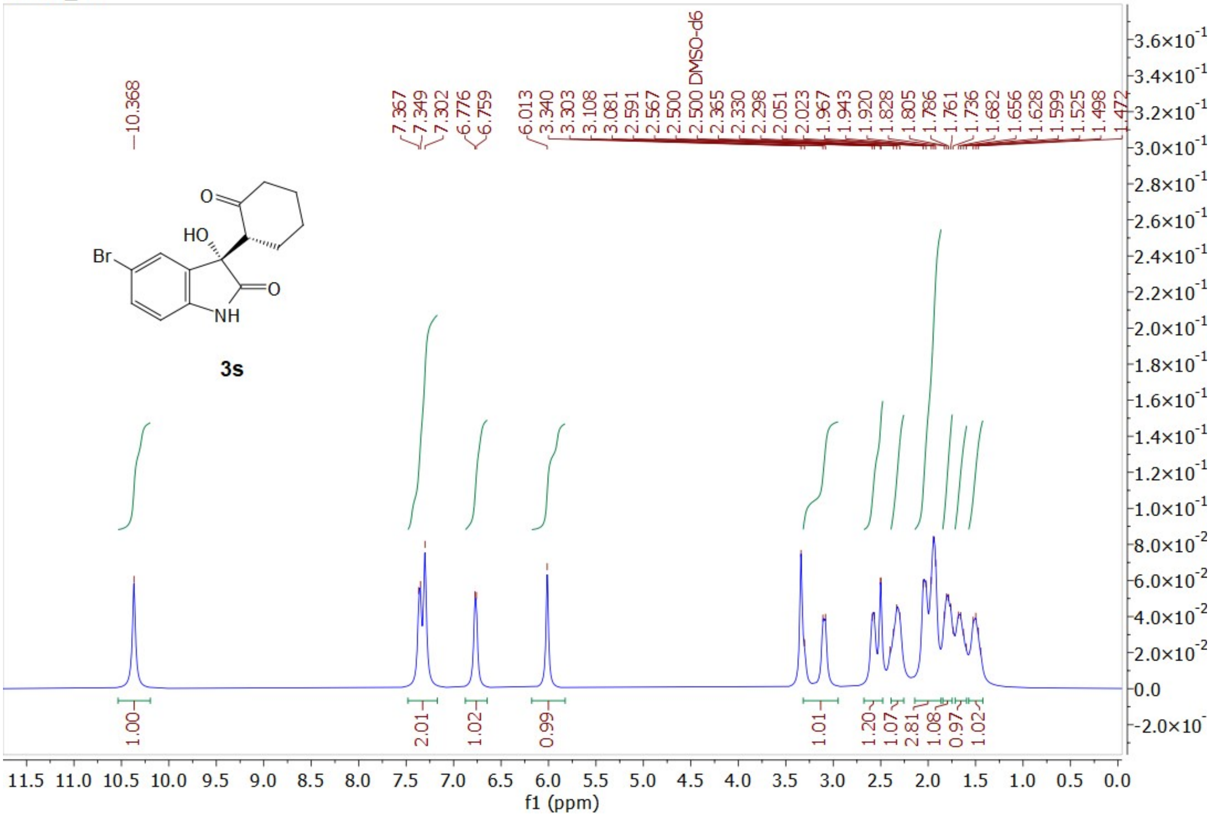
1.1.18 ^{1}H-NMR and ^{13}C-NMR for compound-3r



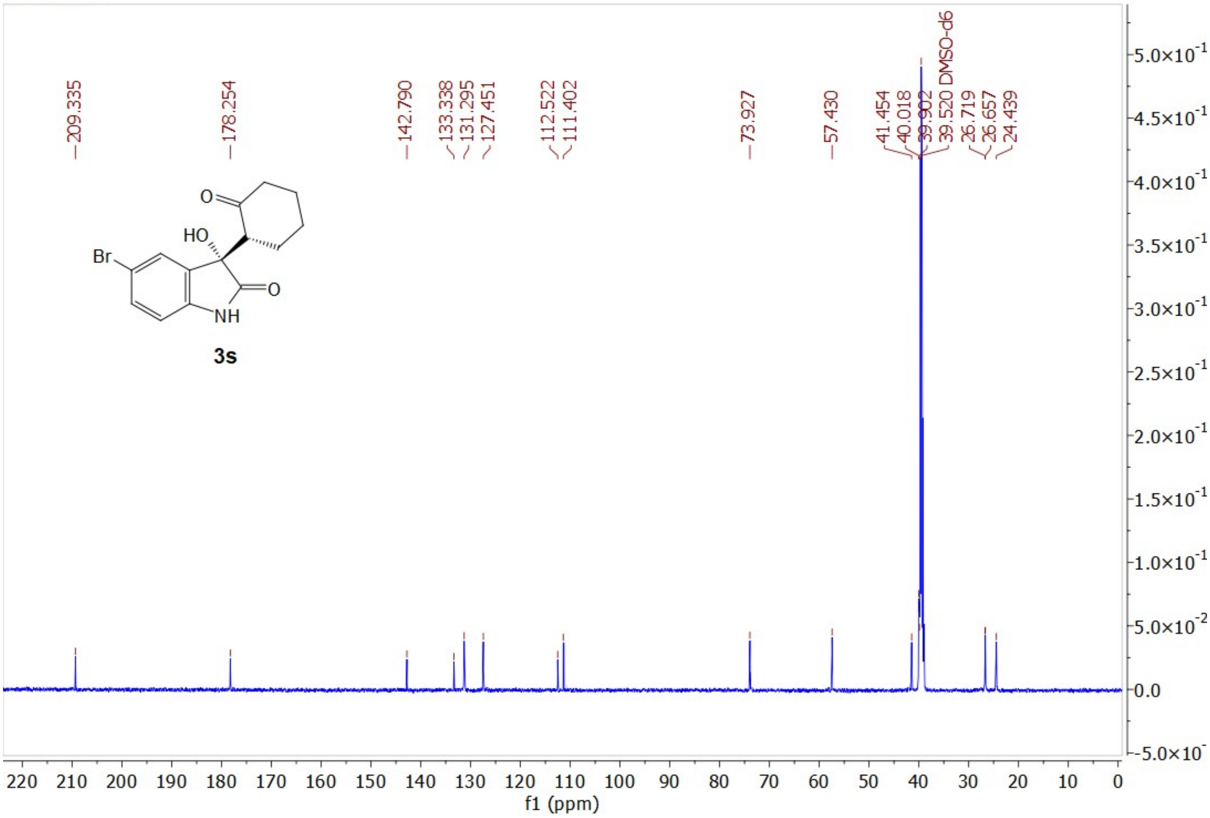


1.1.19 ^{1}H-NMR and ^{13}C-NMR for compound-3s

AD-3s_1H

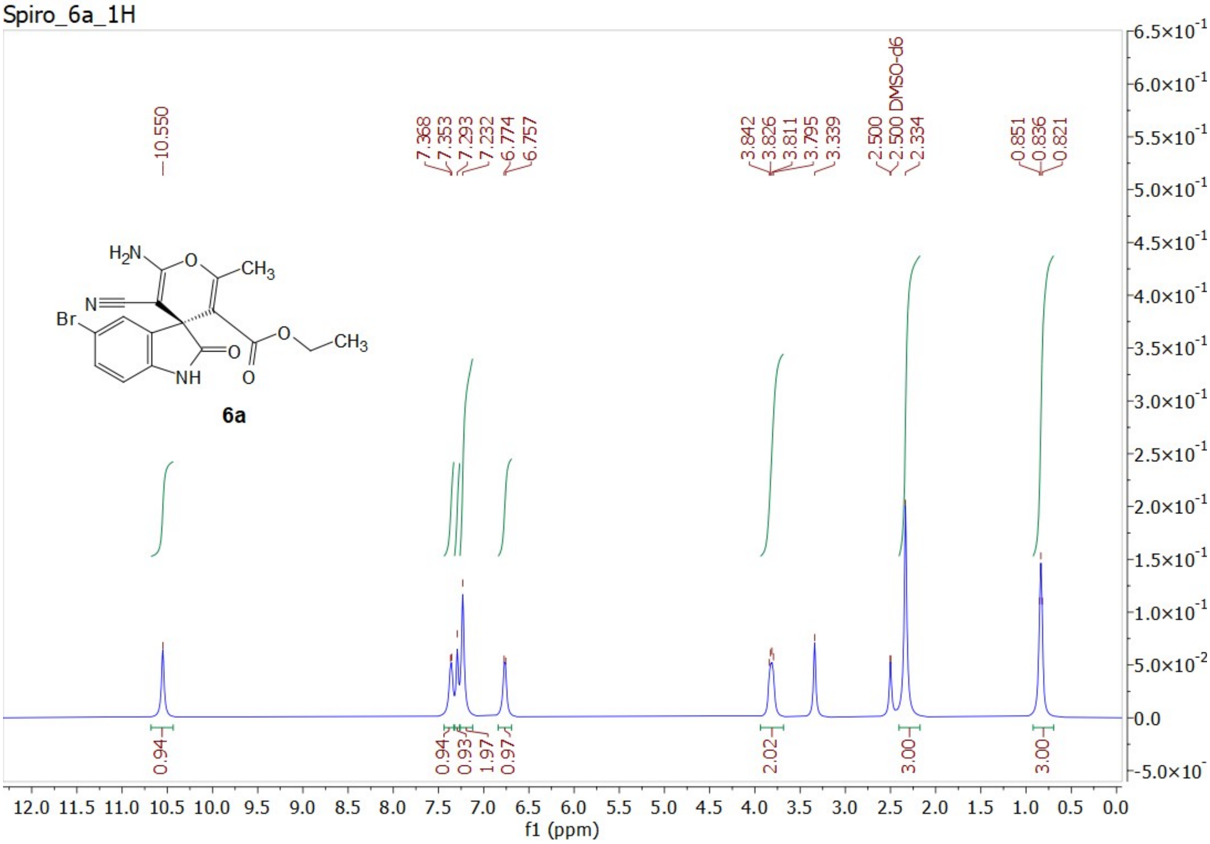


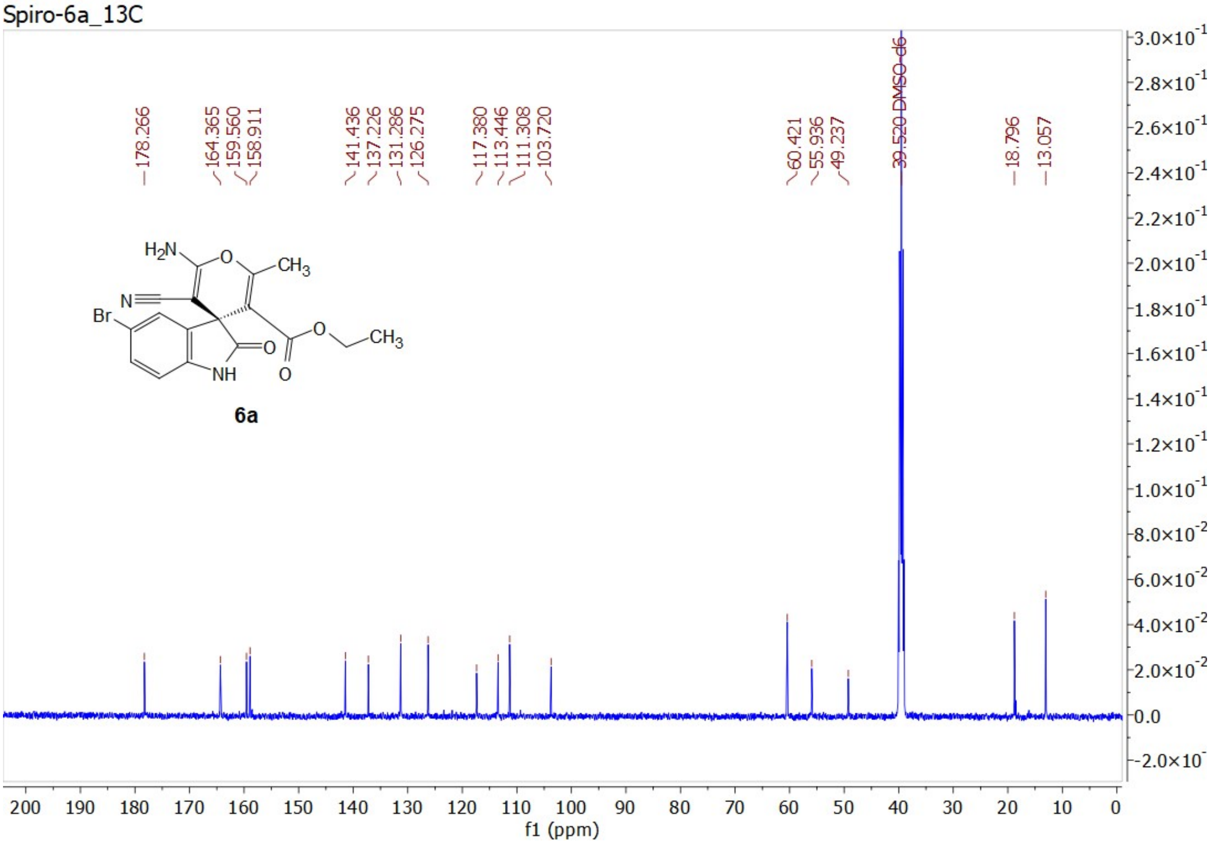
AD-3s_13C



1.2 NMR Spectra of spiro-compound 6a-l

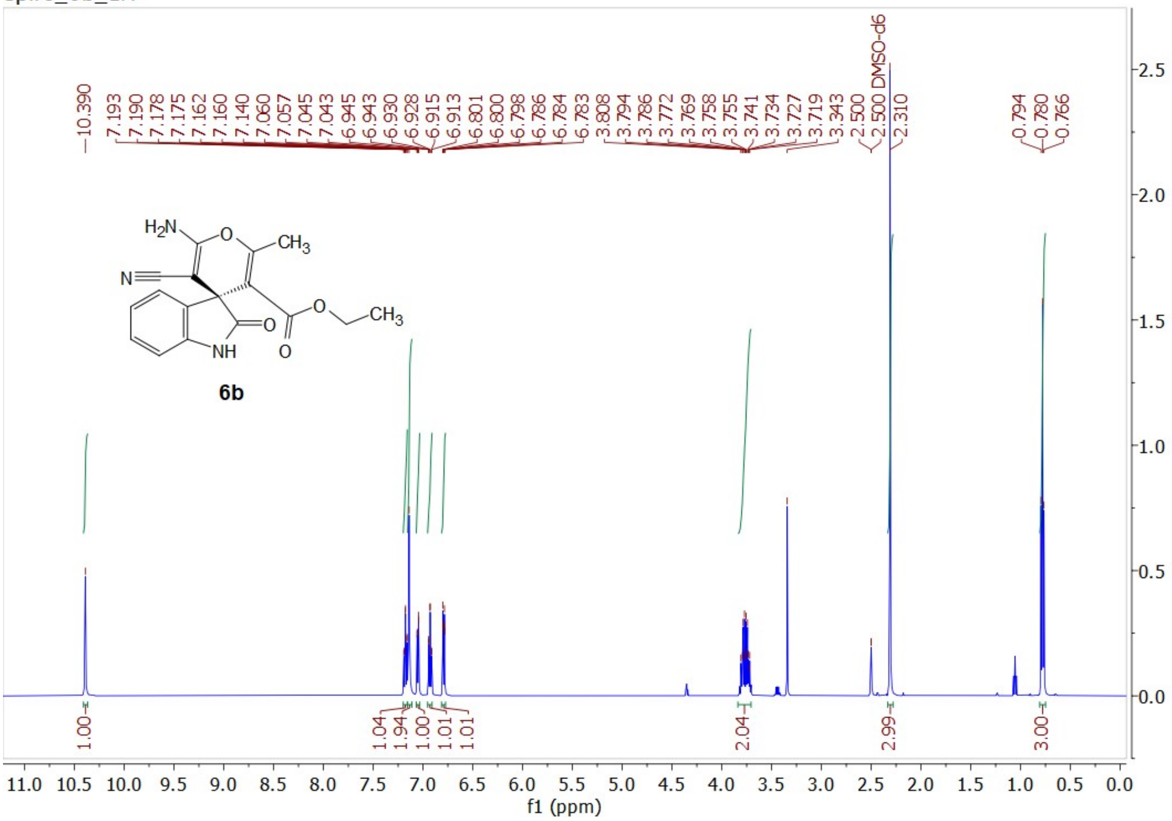
1.2.1 ^{1}H-NMR and ^{13}C-NMR for spiro-compound-6a



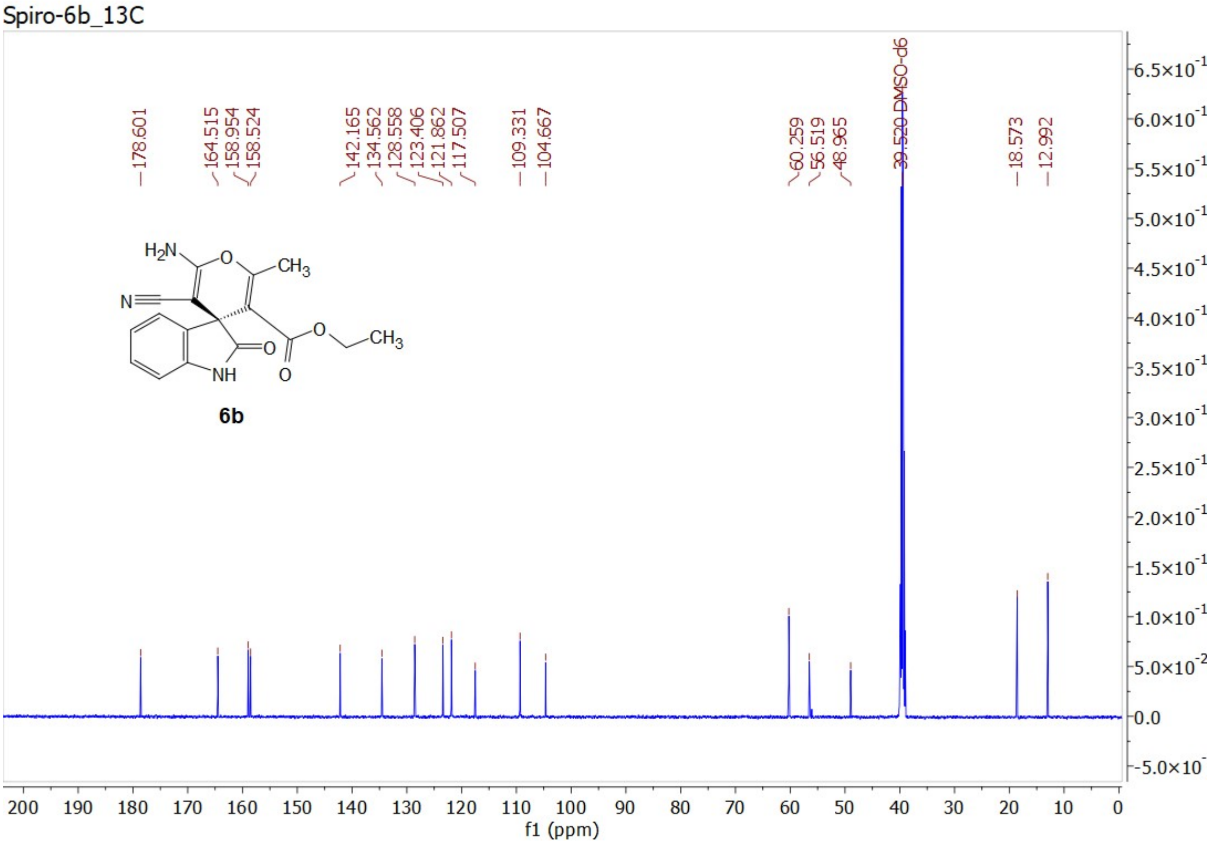


1.2.2 ^{1}H-NMR and ^{13}C-NMR for spiro-compound-6b

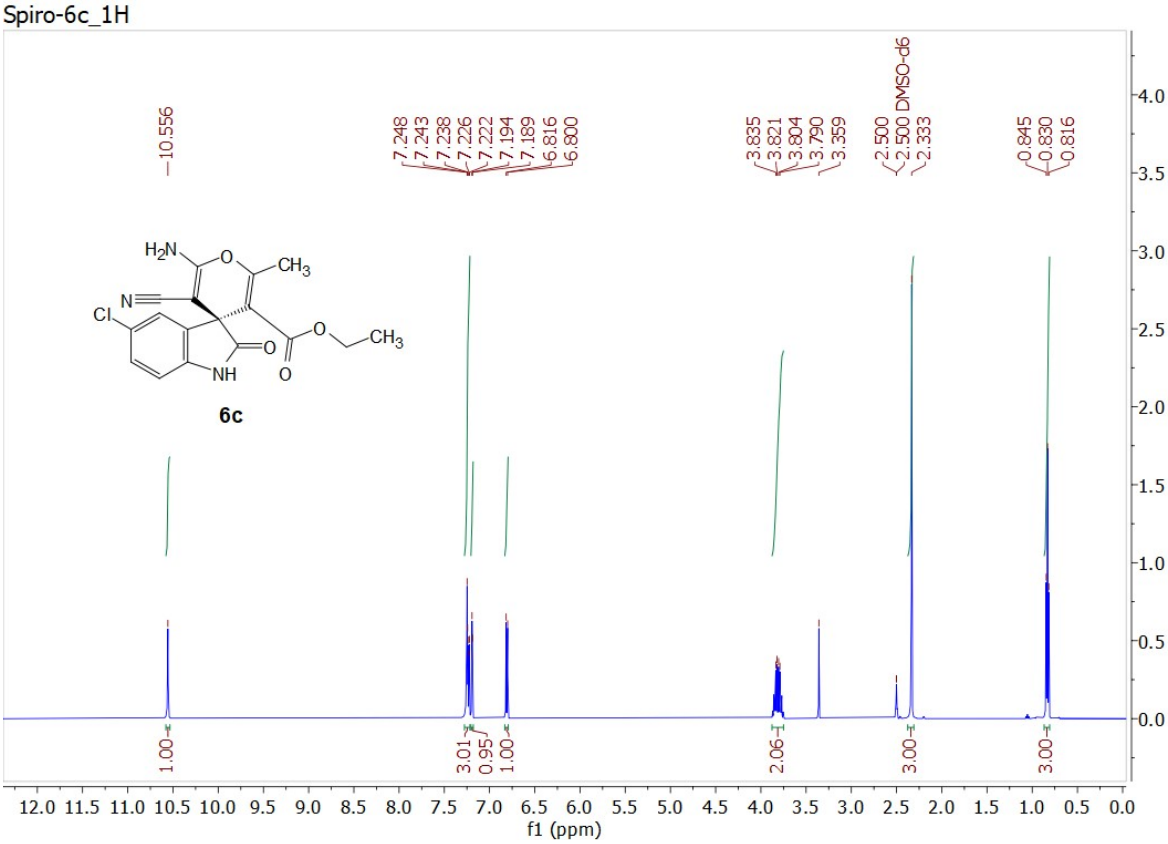
Spiro_6b_1H

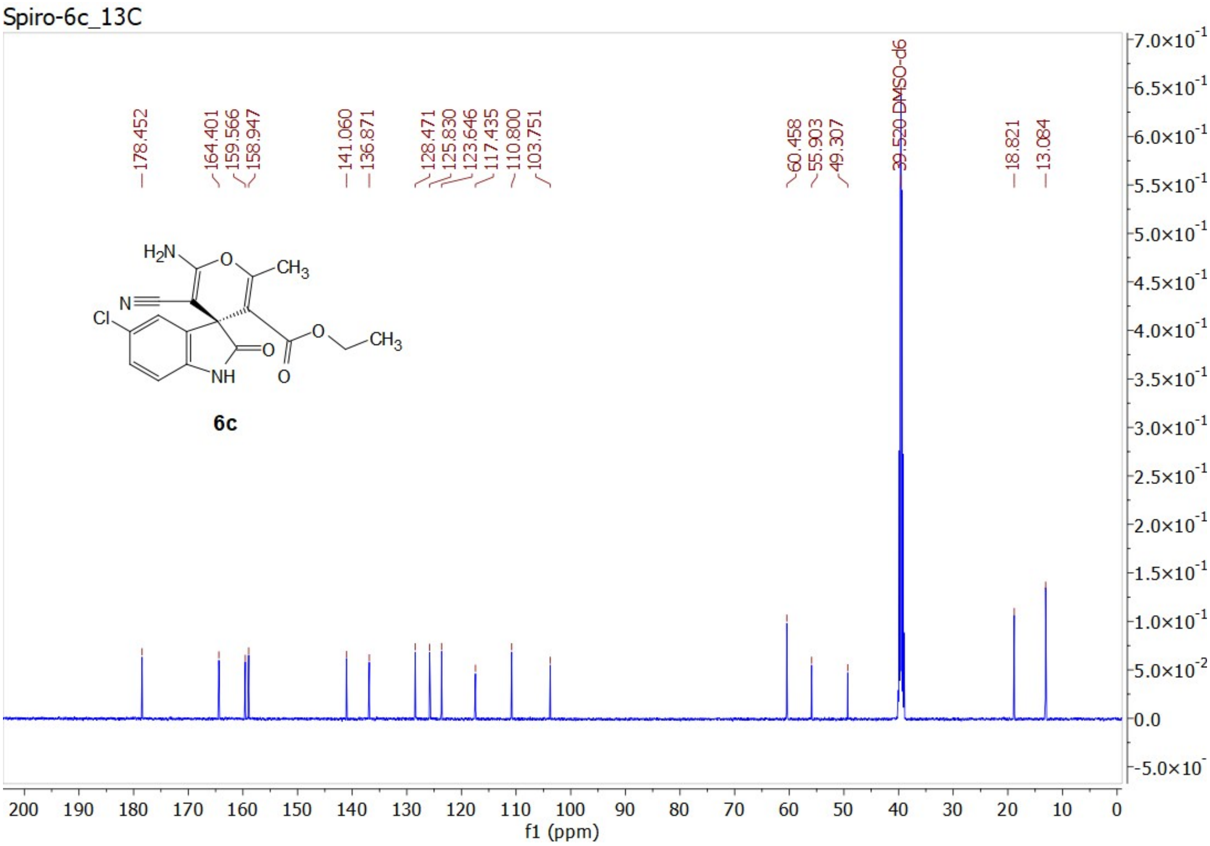


Spiro-6b_13C



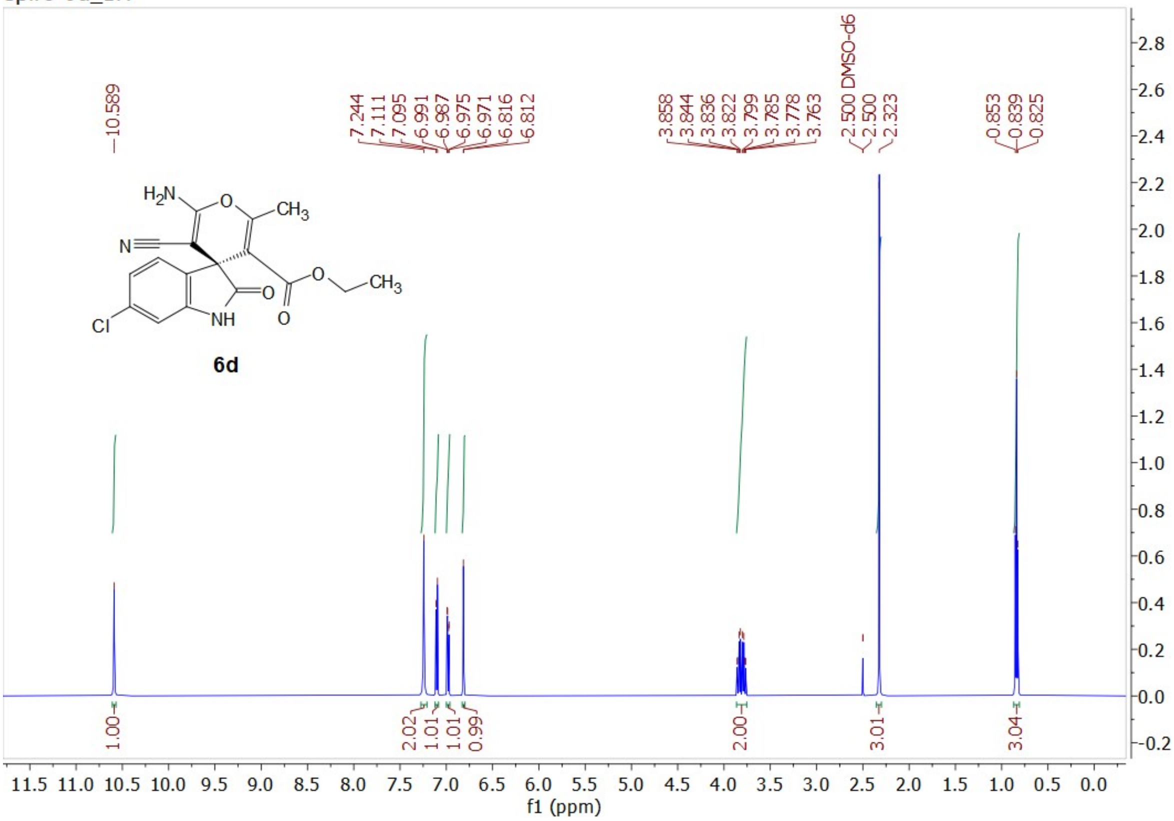
1.2.3 [1]H-NMR and [13]C-NMR for Spiro-compound-6c



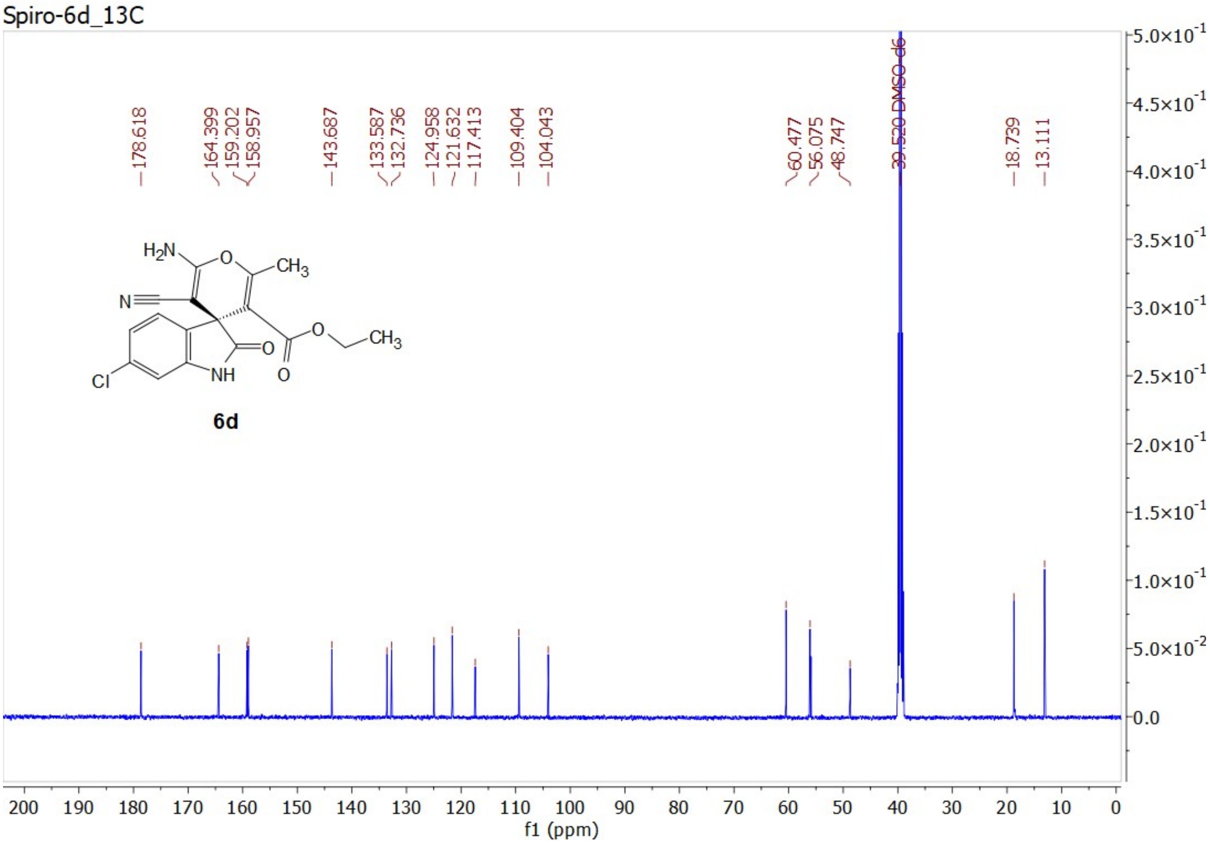


1.2.4 [1]H-NMR and [13]C-NMR for Spiro-compound-6d

Spiro-6d_1H

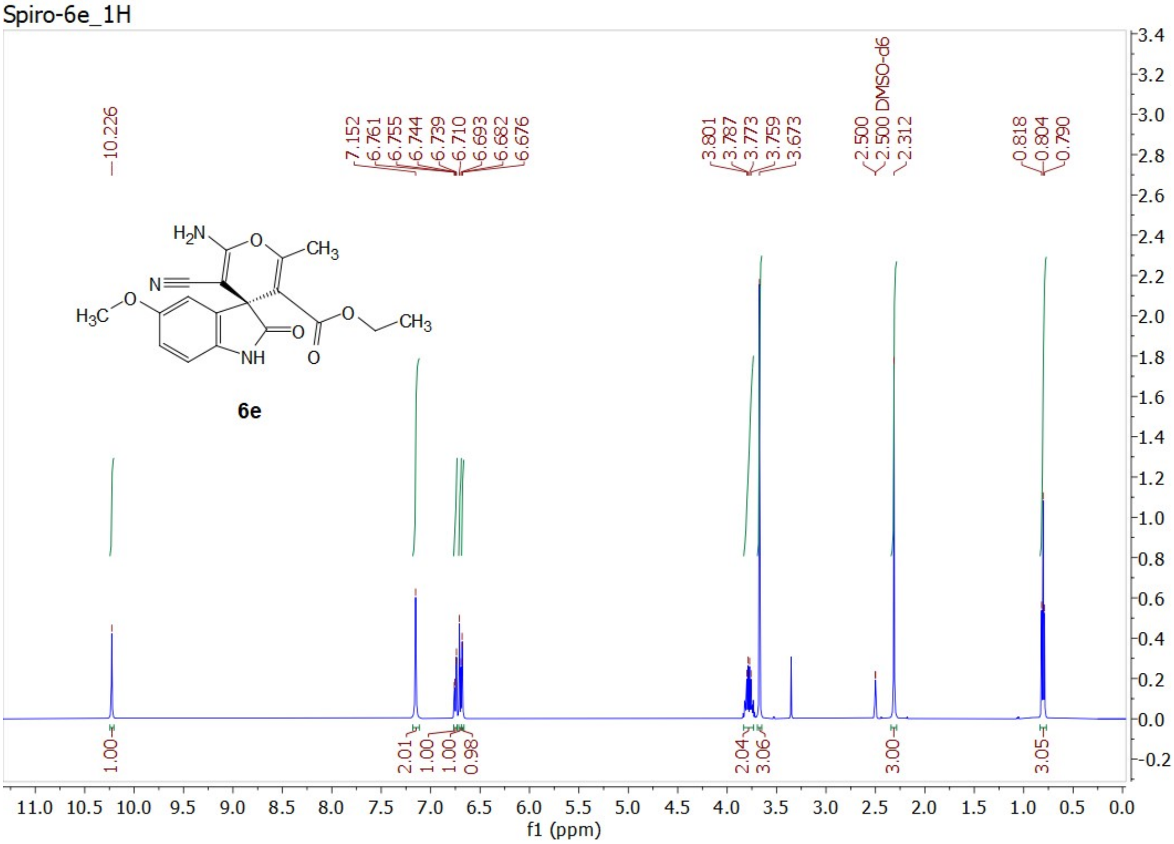


Spiro-6d_13C

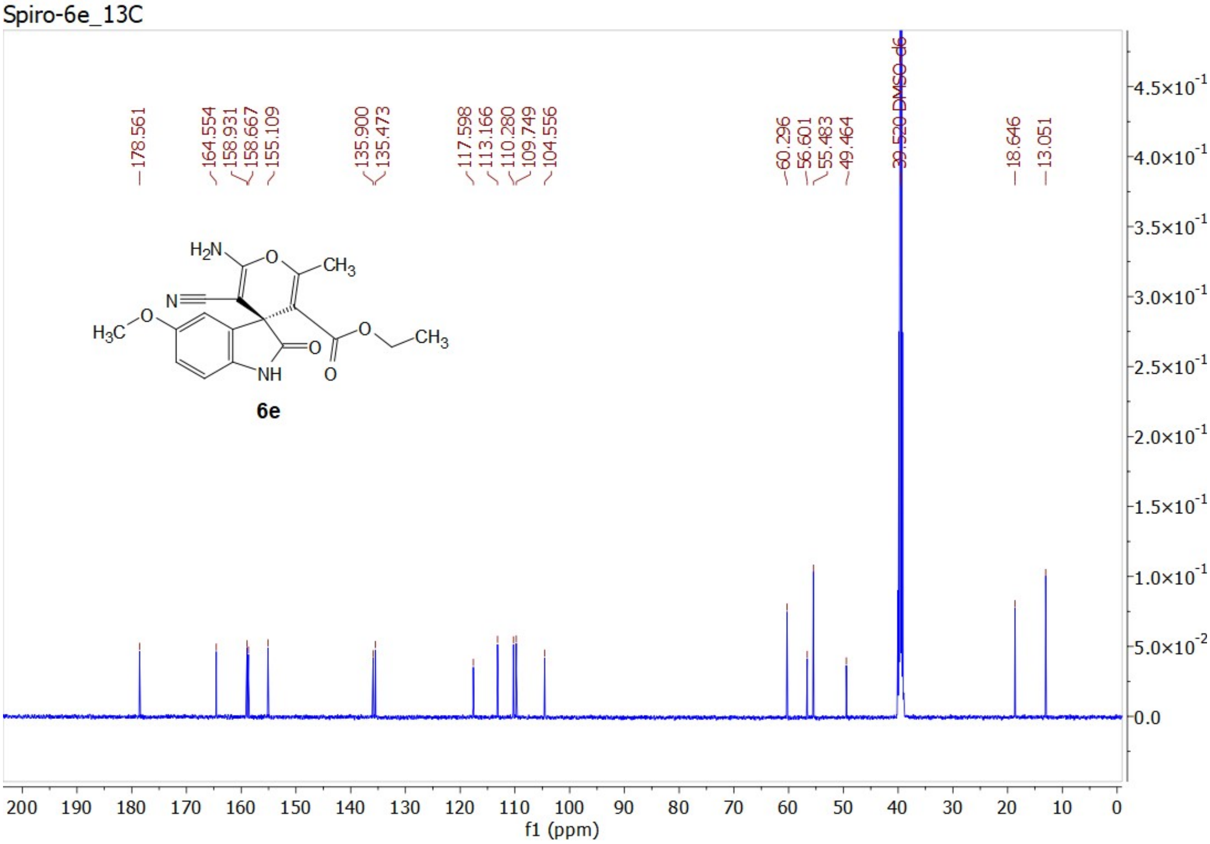


1.2.5 ^{1}H-NMR and ^{13}C-NMR for Spiro-compound-6e

Spiro-6e_1H



Spiro-6e_13C



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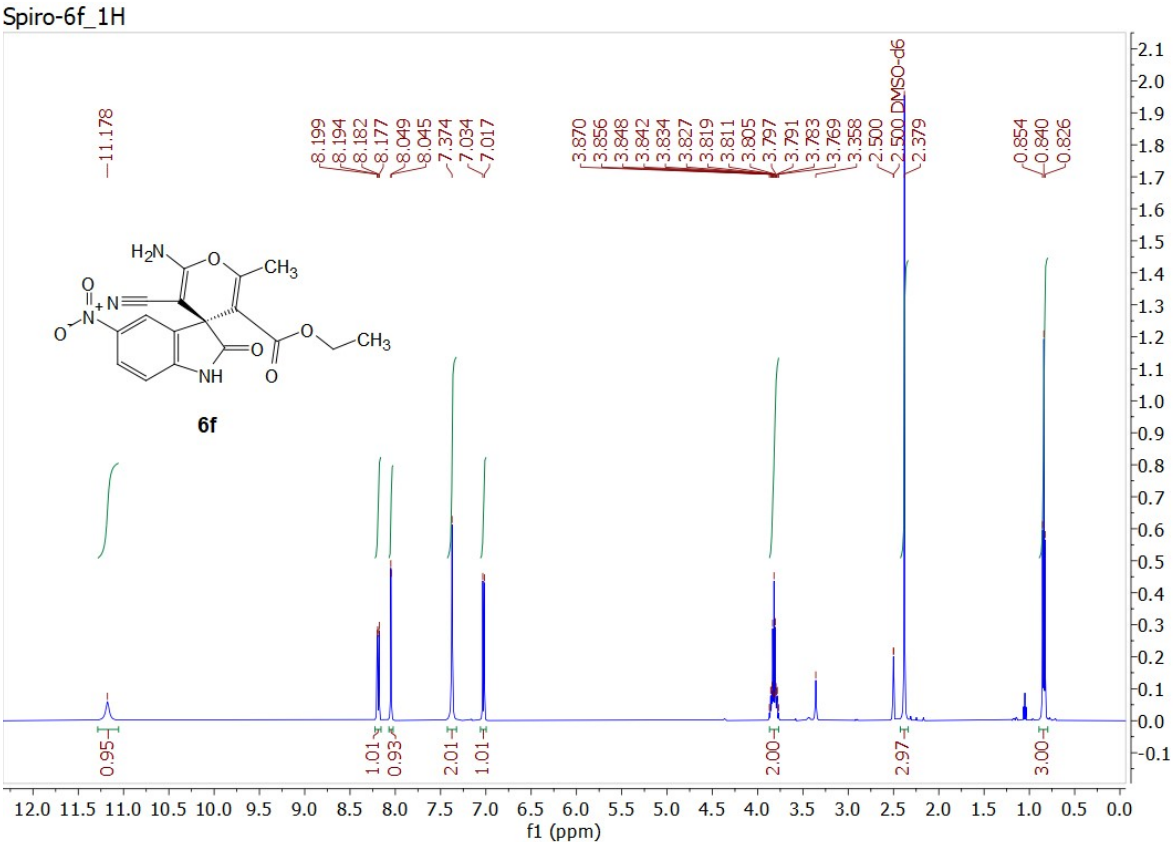

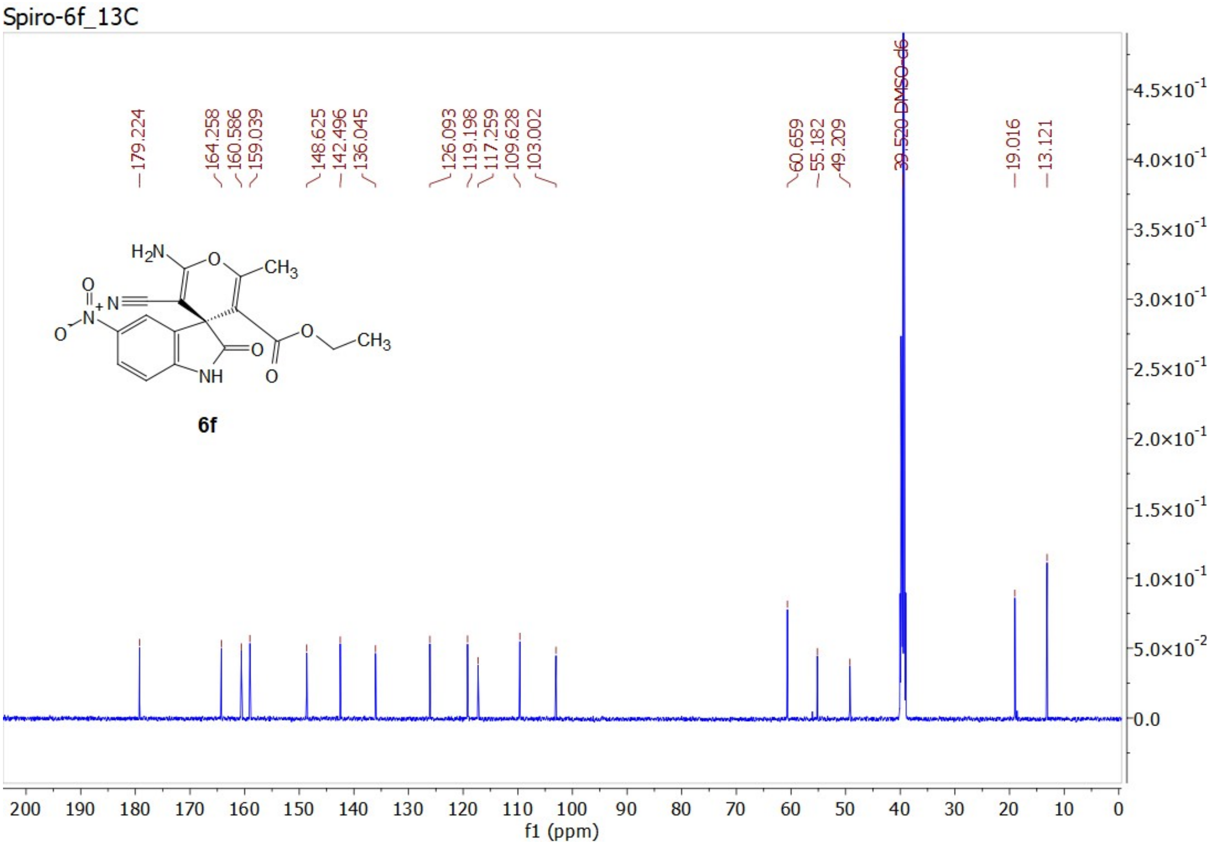

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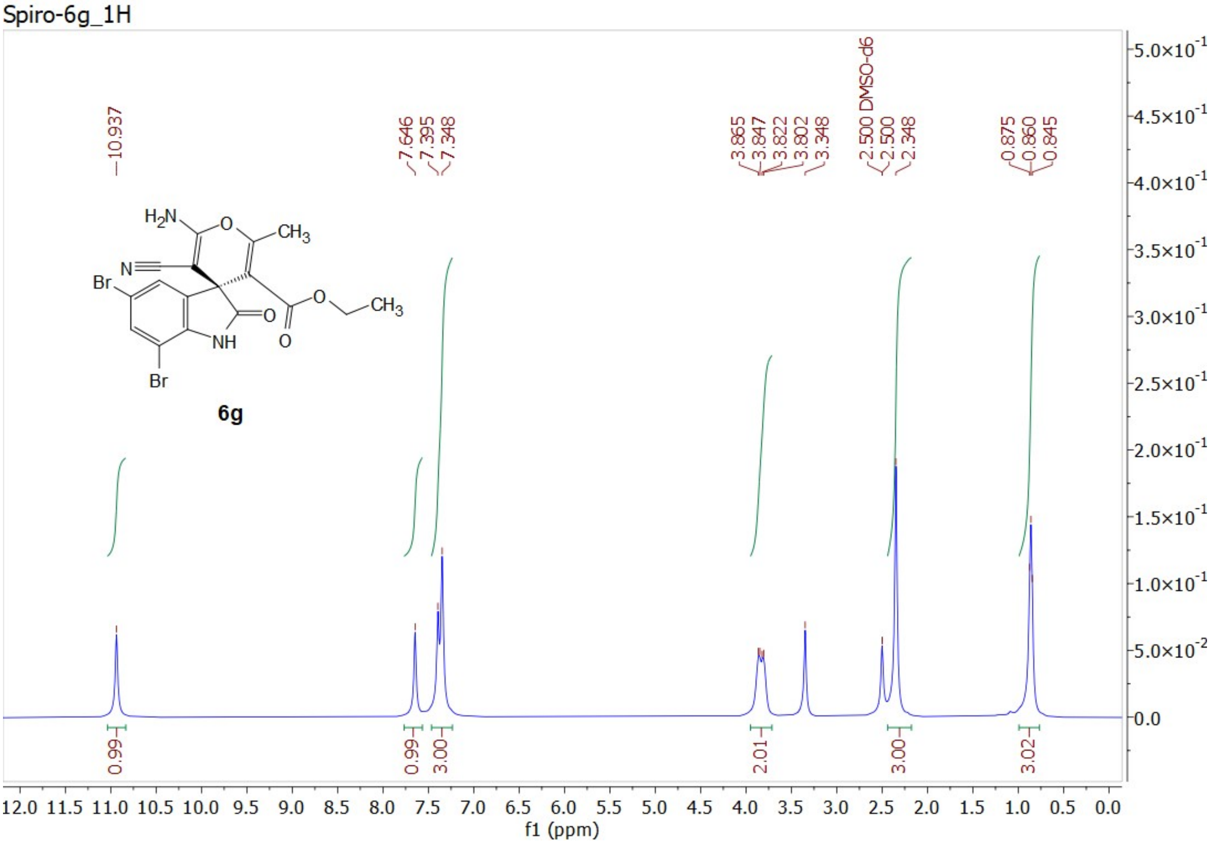
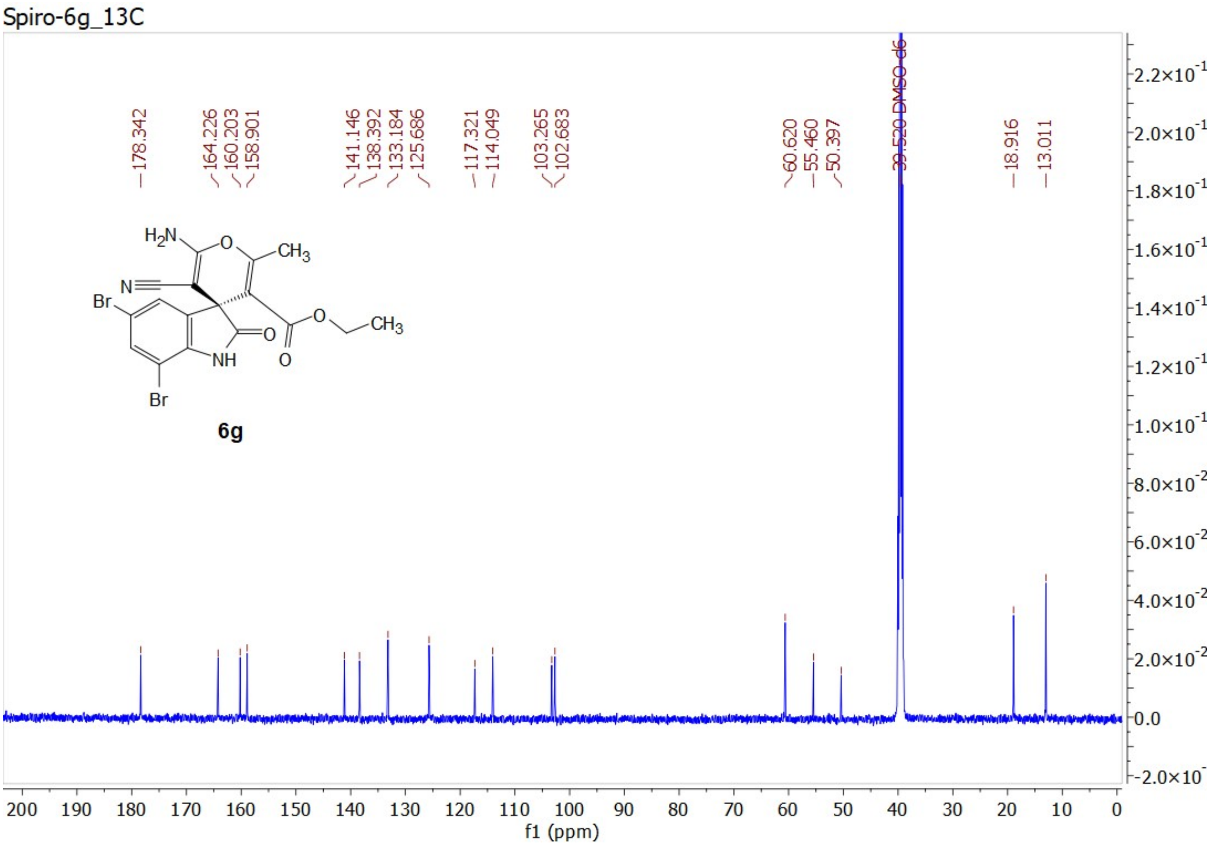
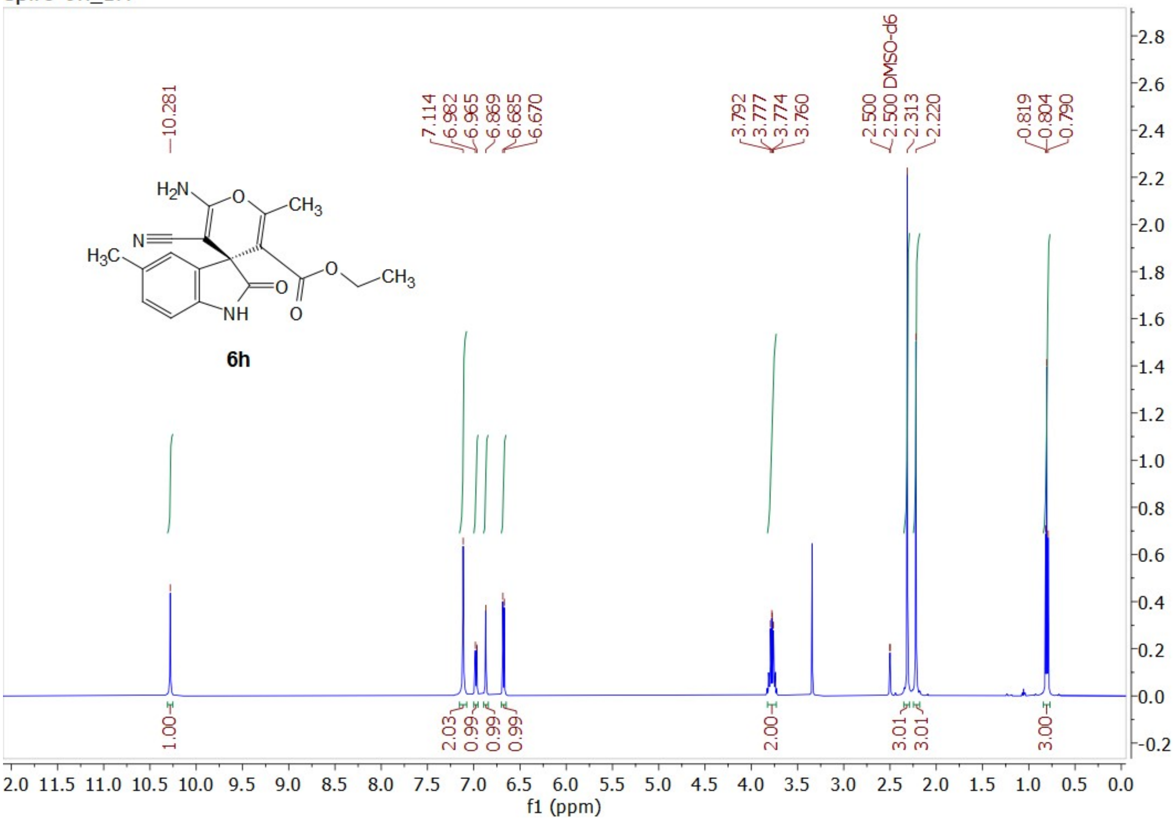


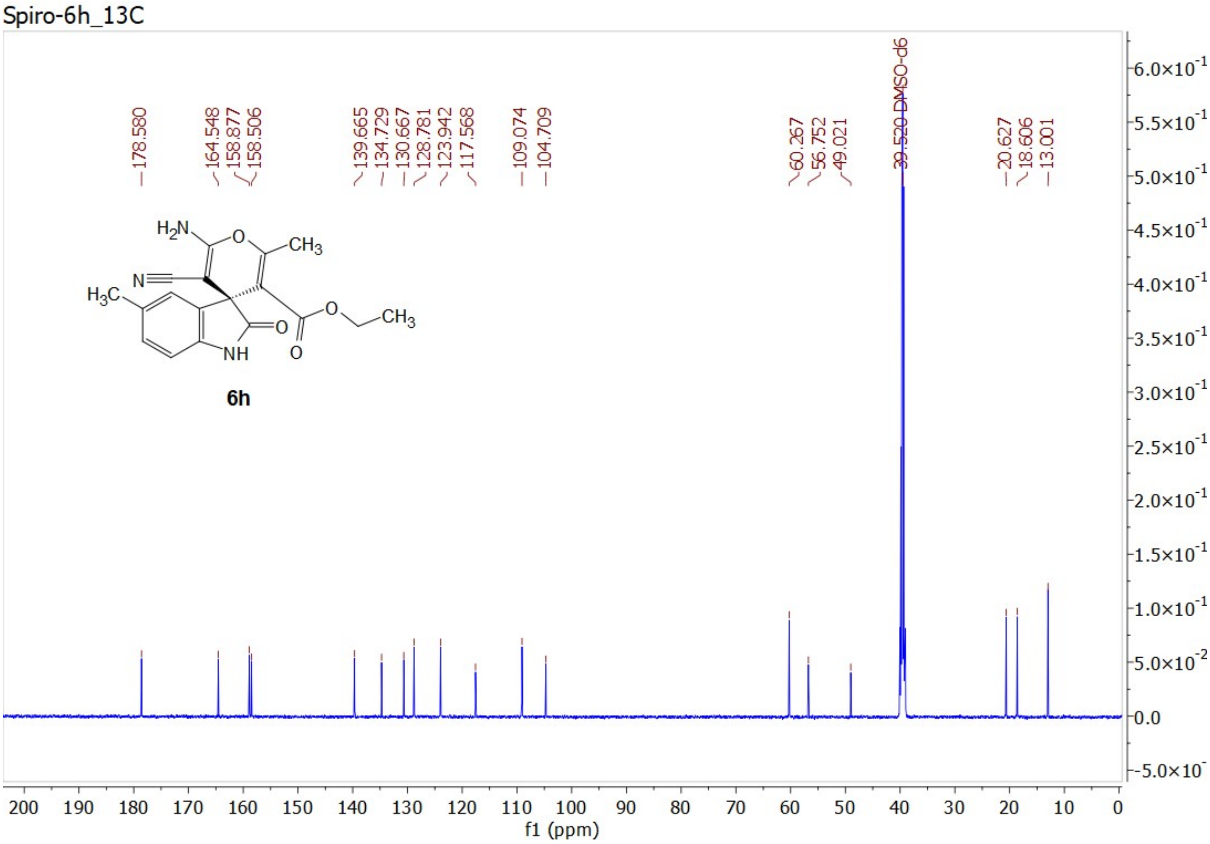


1.2.8 ^{1}H-NMR and ^{13}C-NMR for Spiro-compound-6h

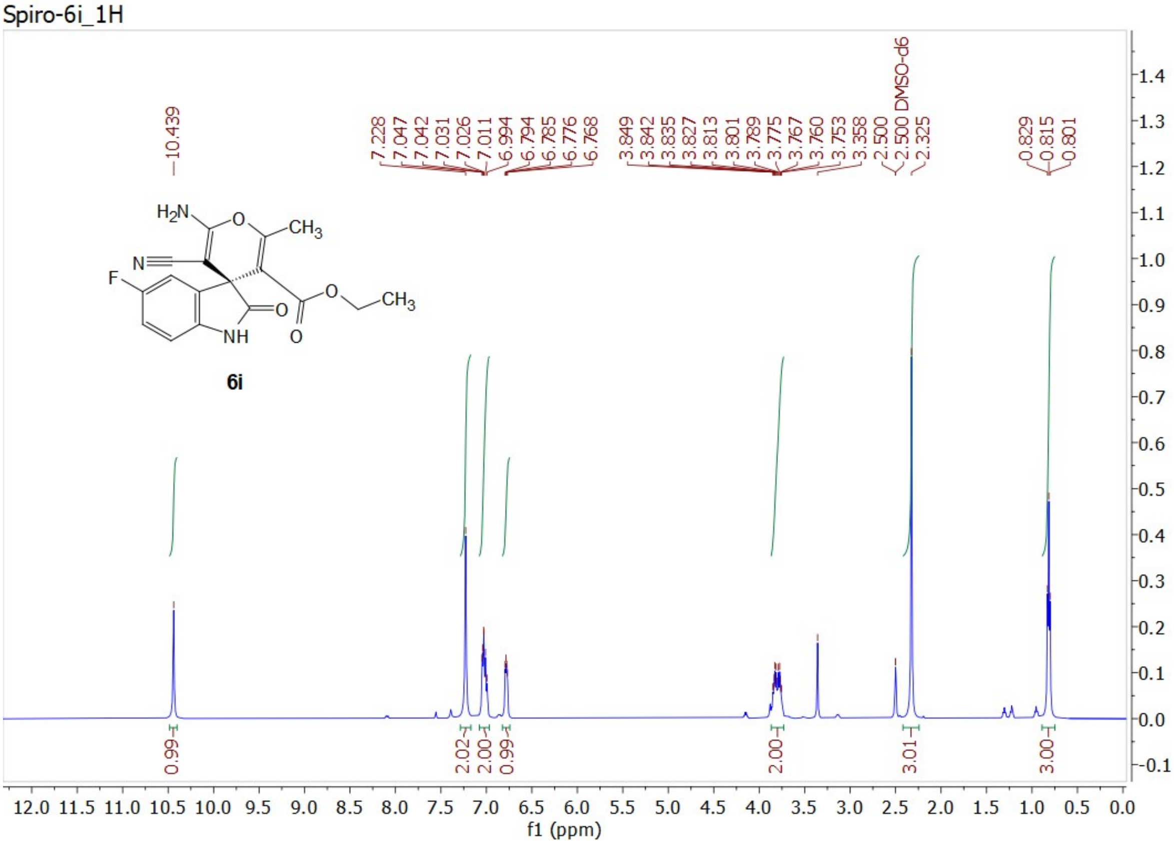
Spiro-6h_1H

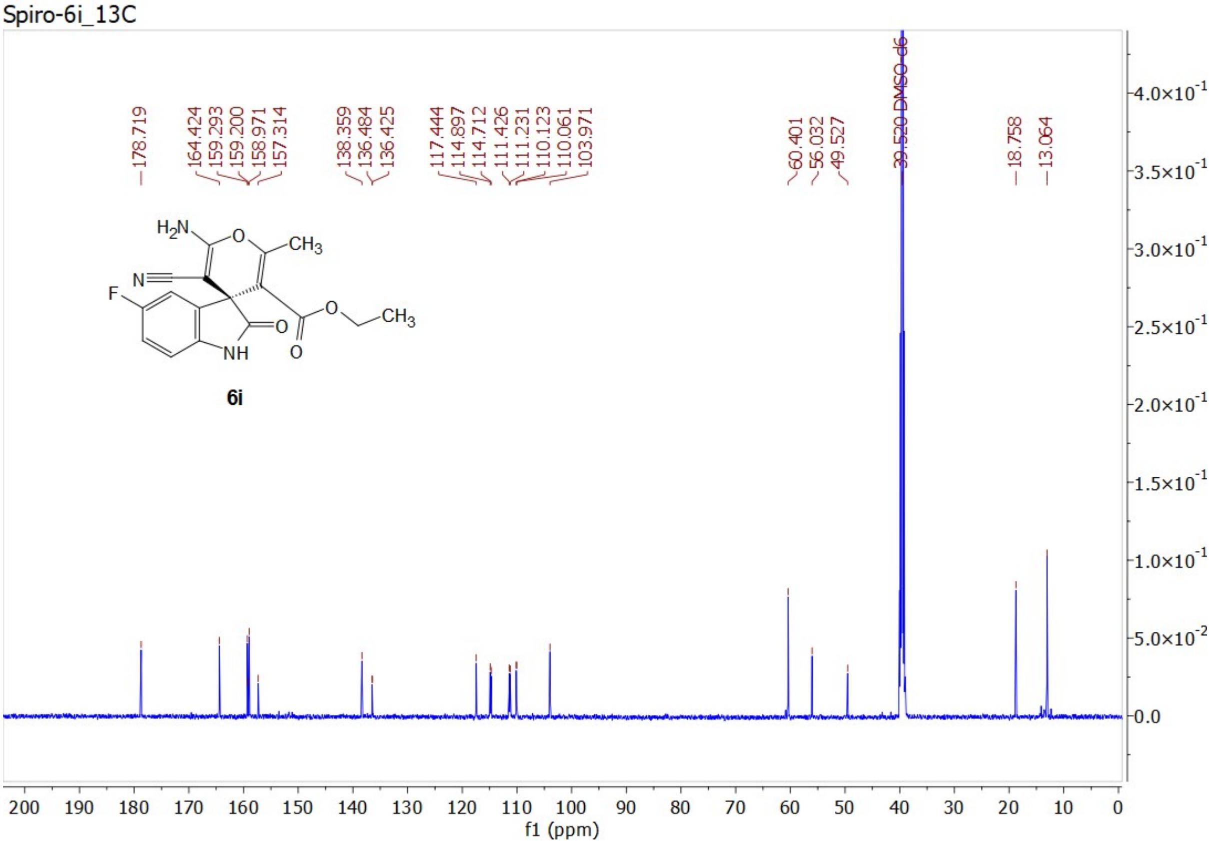


Spiro-6h_13C



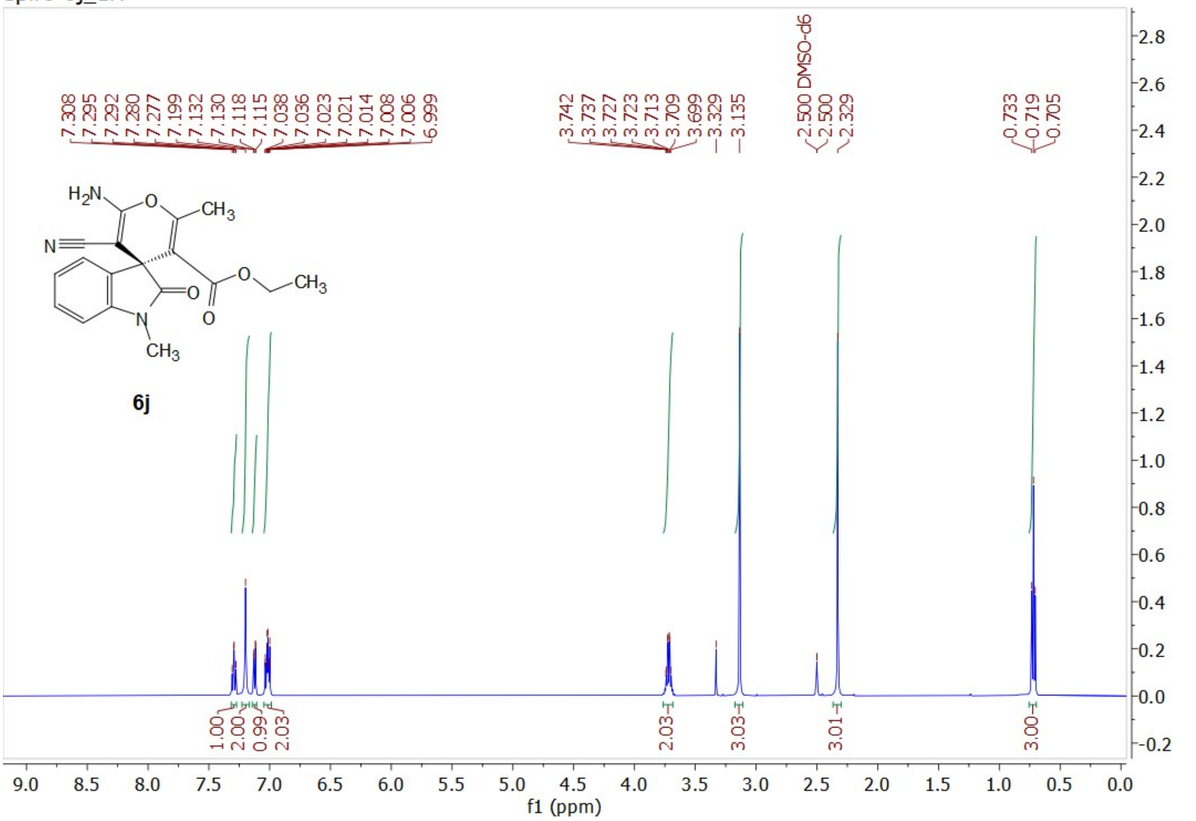
1.2.9 ^{1}H-NMR and ^{13}C-NMR for Spiro-compound-6i



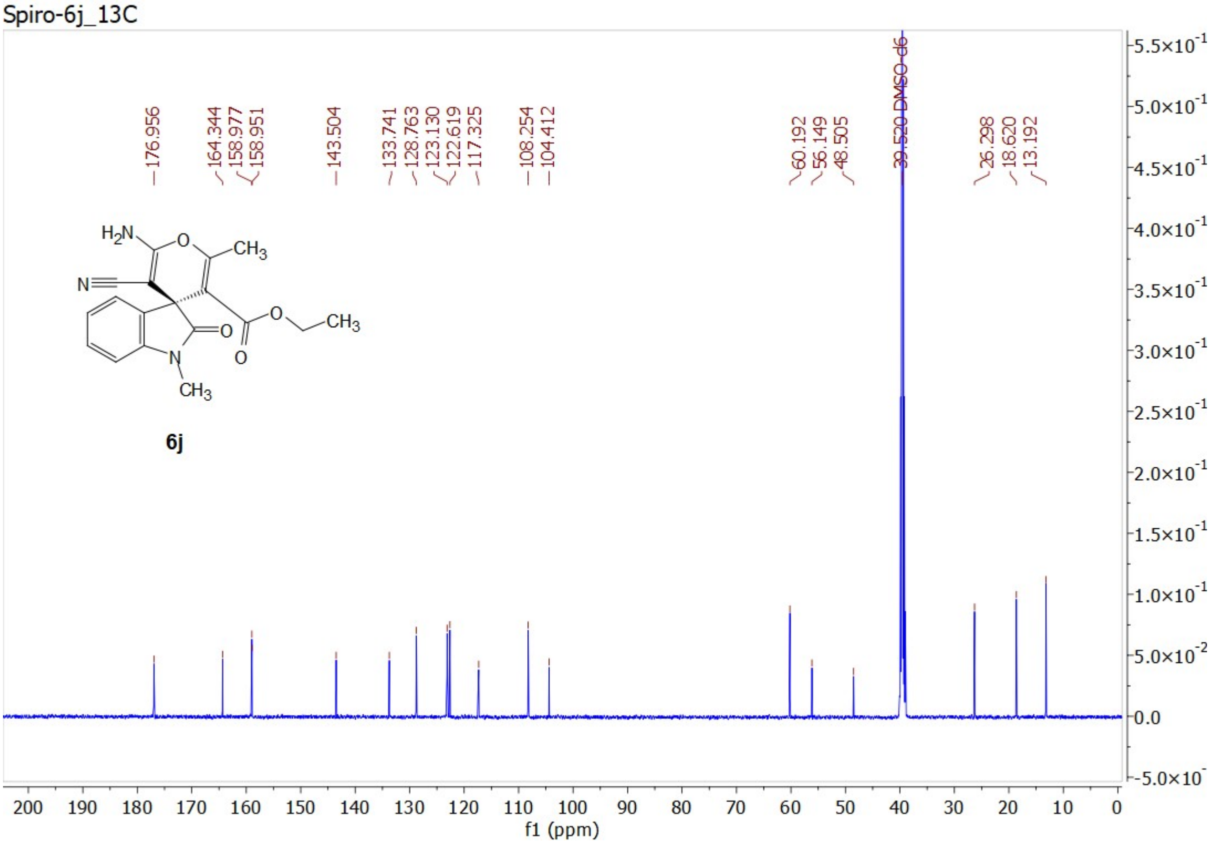


1.2.10 ^{1}H-NMR and ^{13}C-NMR for Spiro-compound-6j

Spiro-6j_1H

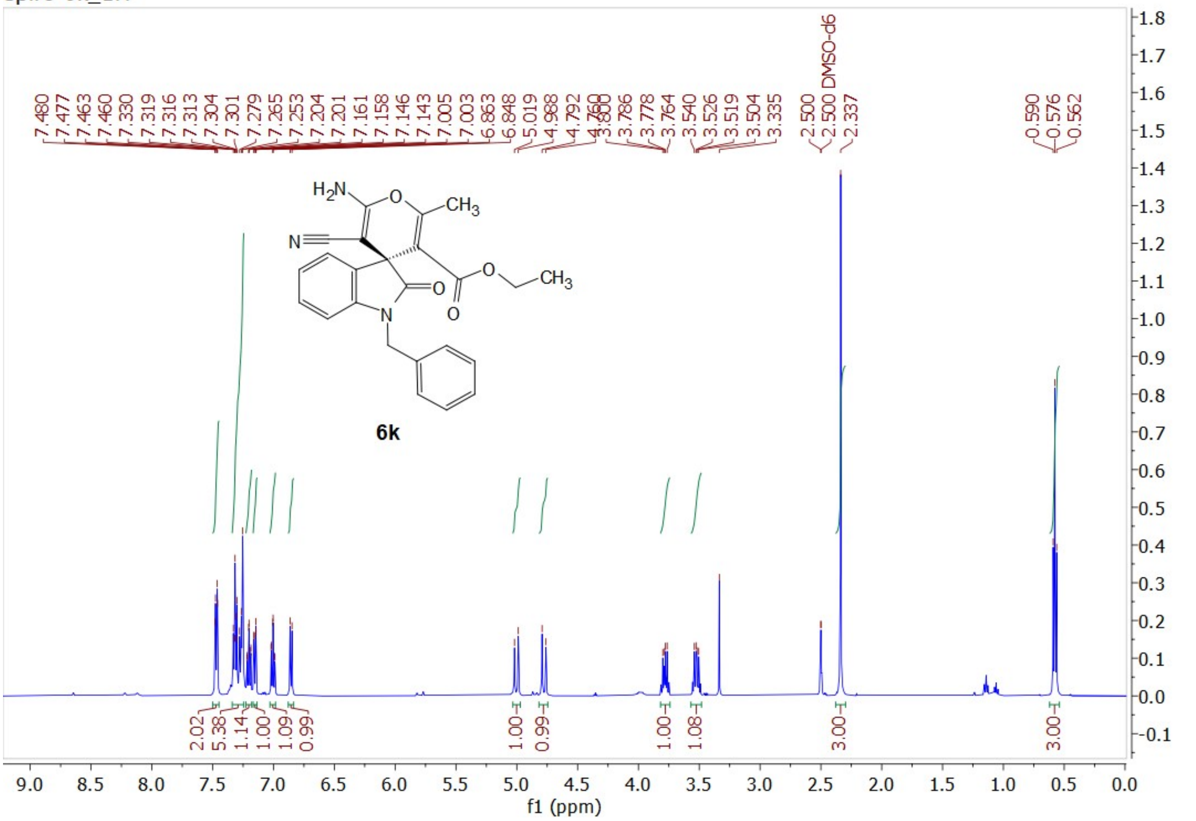


Spiro-6j_13C

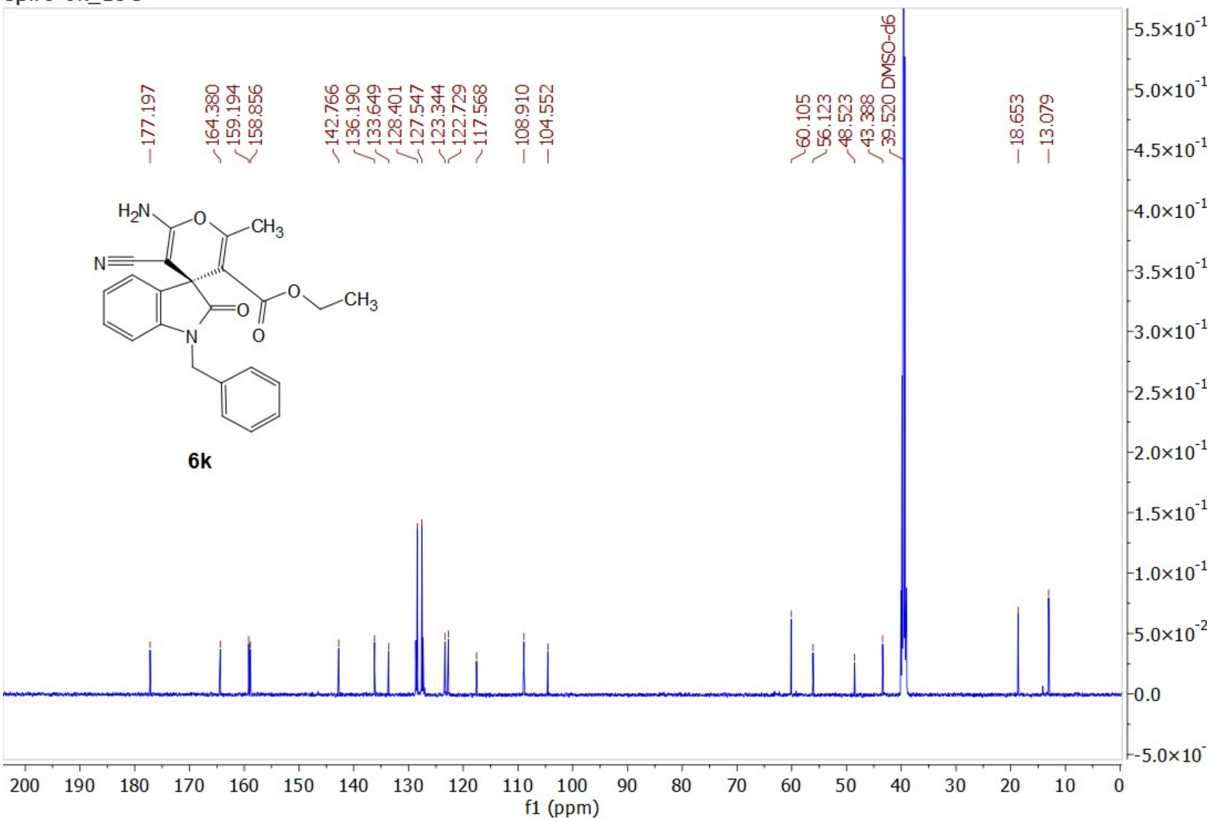


1.2.11 ^{1}H-NMR and ^{13}C-NMR for Spiro-compound-6k

Spiro-6k_1H



Spiro-6k_13C



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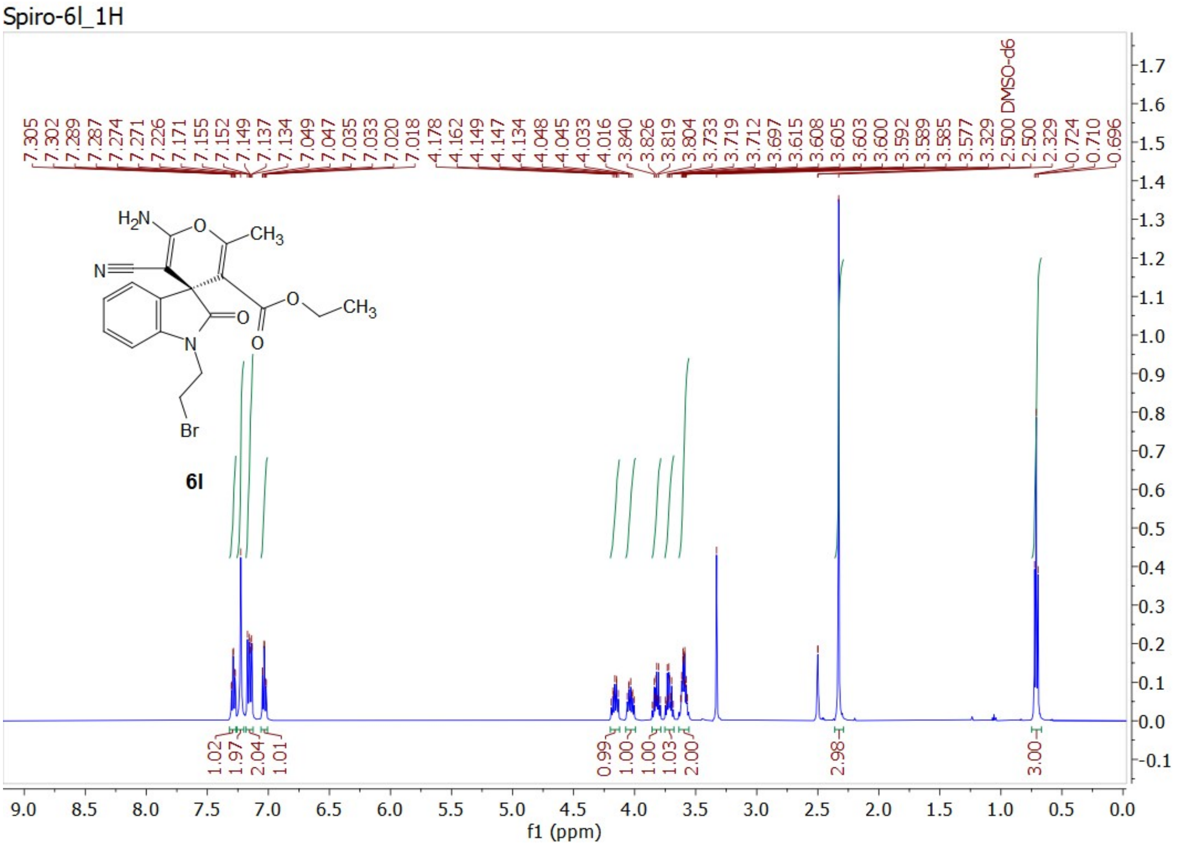
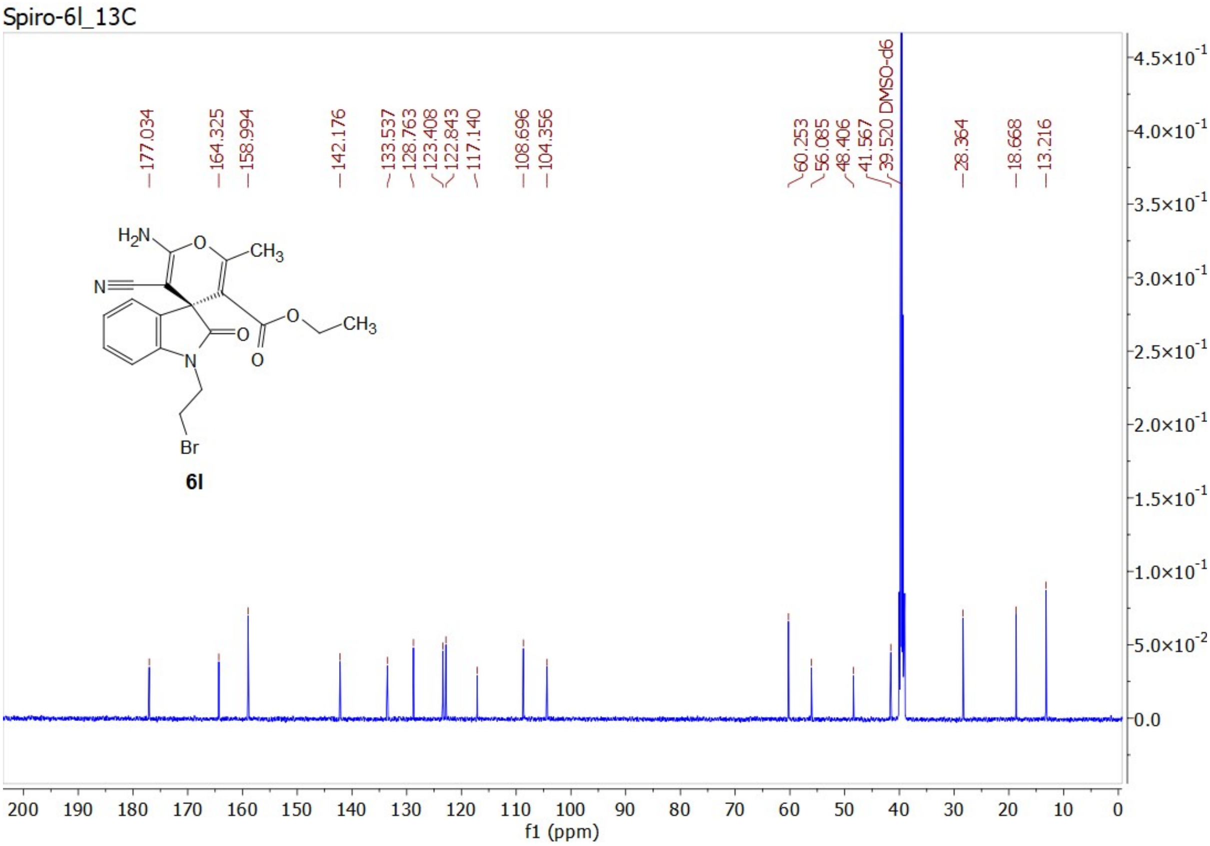
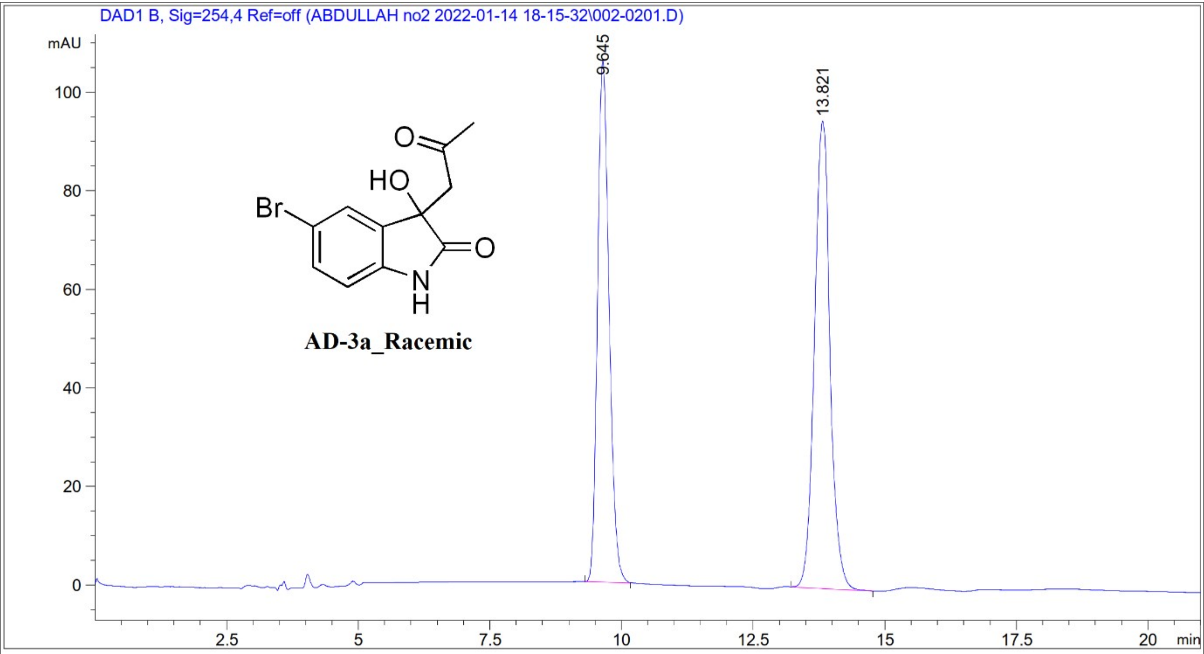




2. HPLC analysis

2.1 HPLC analysis of aldol product 3a-o

2.1.1 HPLC analysis of aldol product 3a



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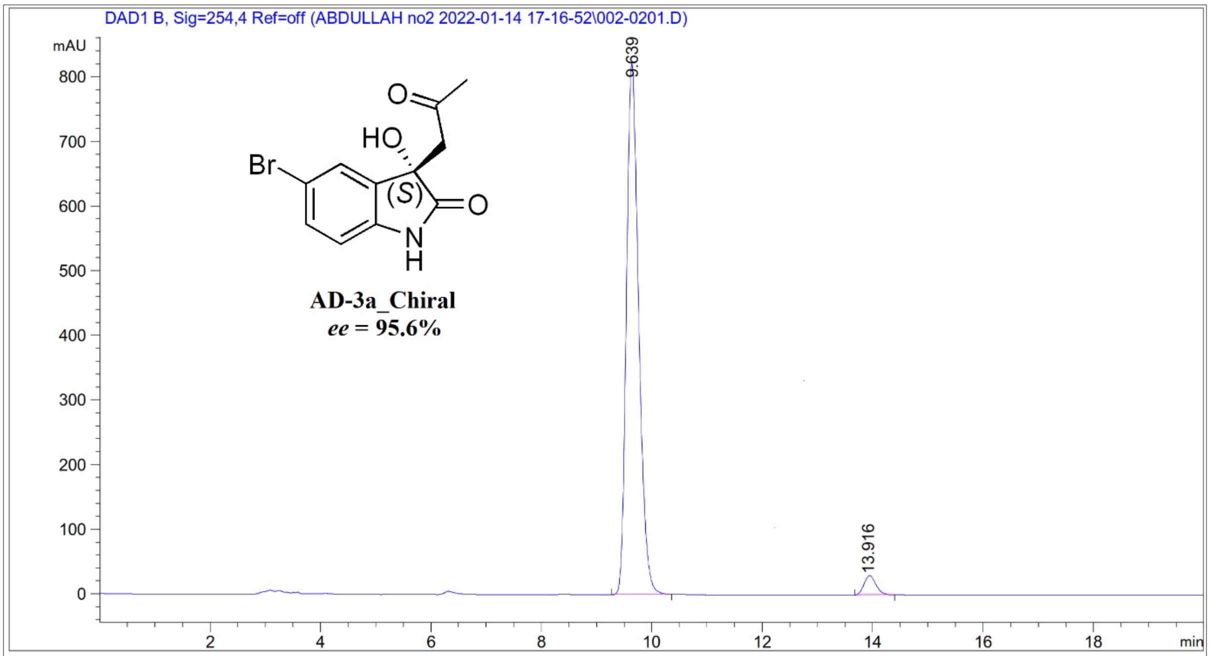
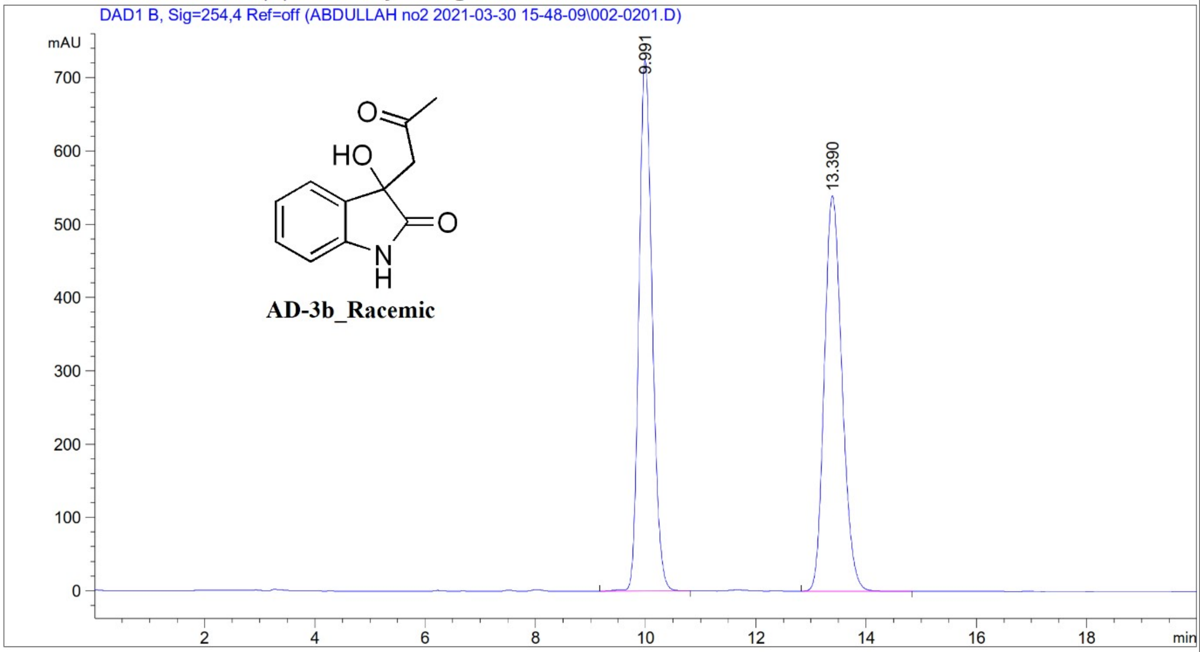
1	9.645	BB	0.3351	1703.59415	105.85462	50.0706
2	13.821	BB	0.3584	1698.75328	94.79651	49.9294

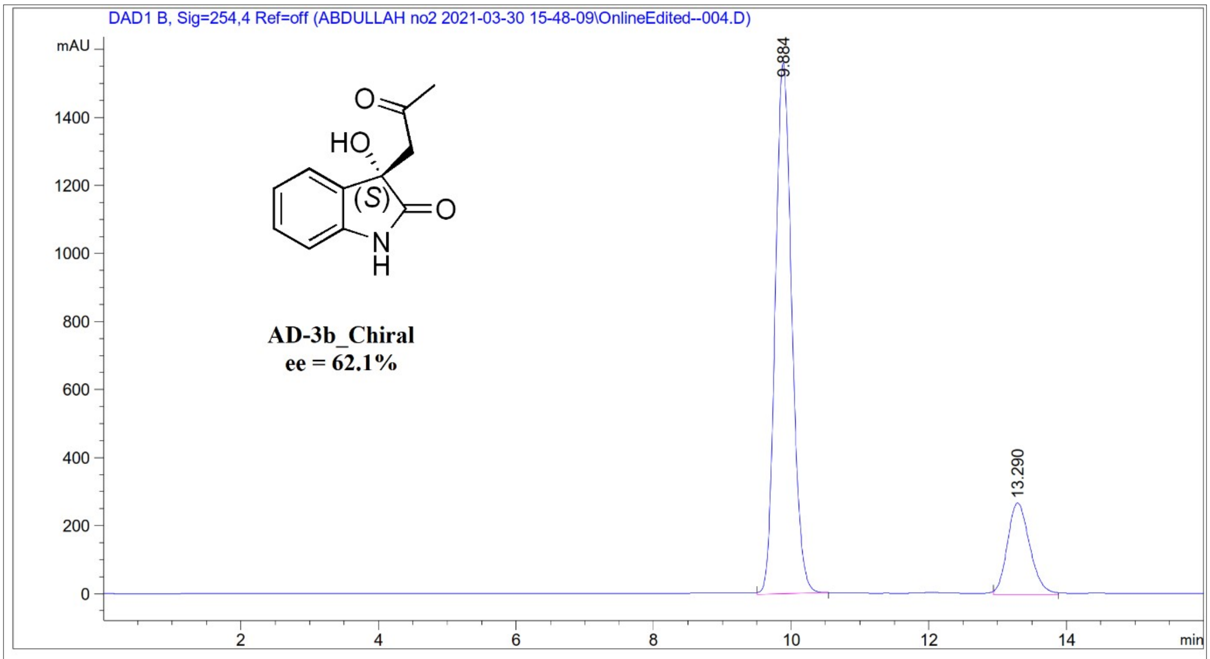


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.639	MM	0.8928	366.390187	820.76196	97.7932
2	13.916	MM	0.5513	8.2681245	29.99501	2.2068

2.1.2 HPLC analysis of aldol product 3b

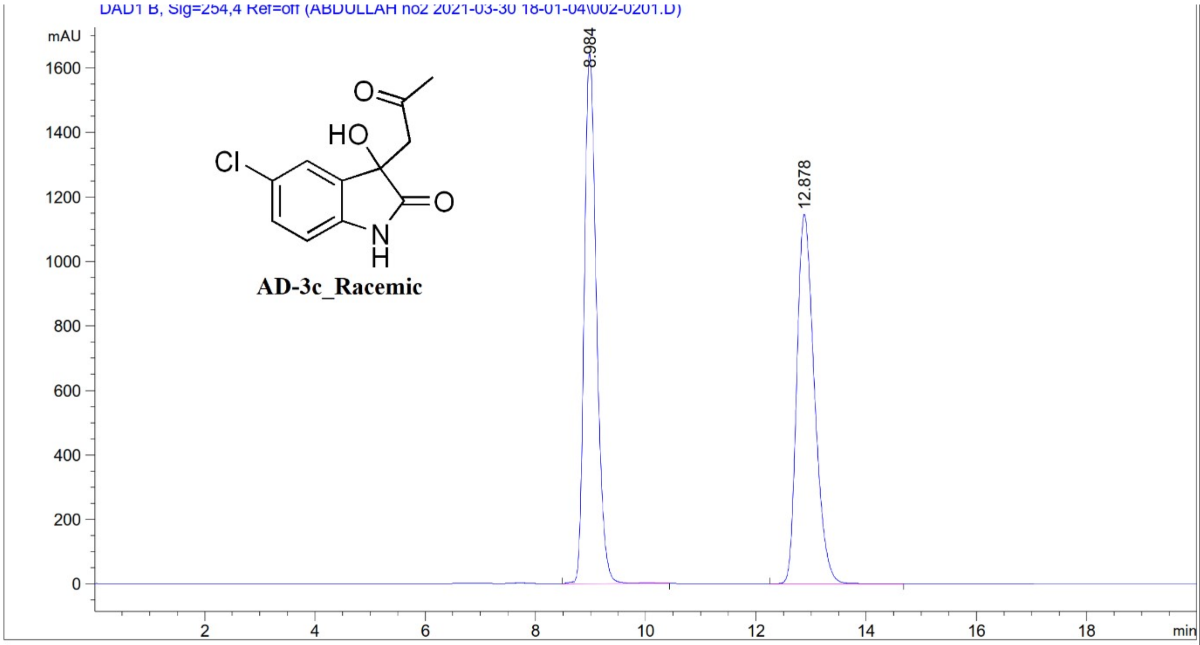


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.991	BB	0.2586	1.20829e4	724.57971	50.0515
2	13.390	BB	0.3466	1.20580e4	539.82800	49.9485

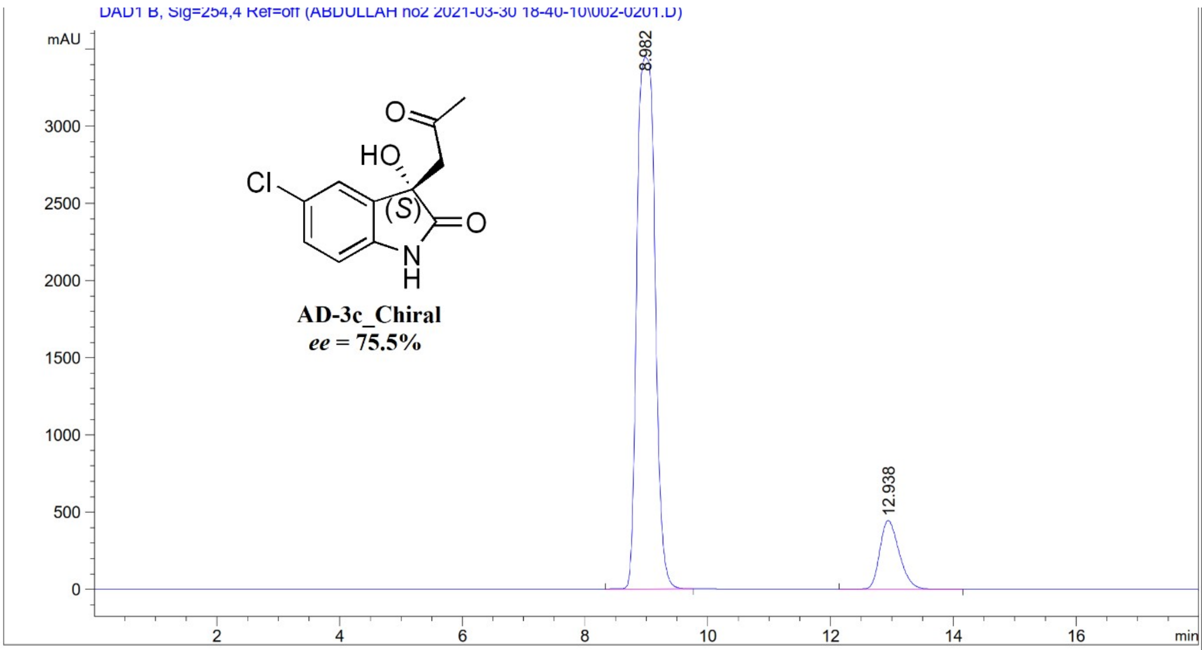


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.884	MM	0.2785	2.60754e4	1560.56116	81.0308
2	13.290	MM	0.3774	6104.23096	269.54108	18.9692

2.1.3 HPLC analysis of aldol product 3c

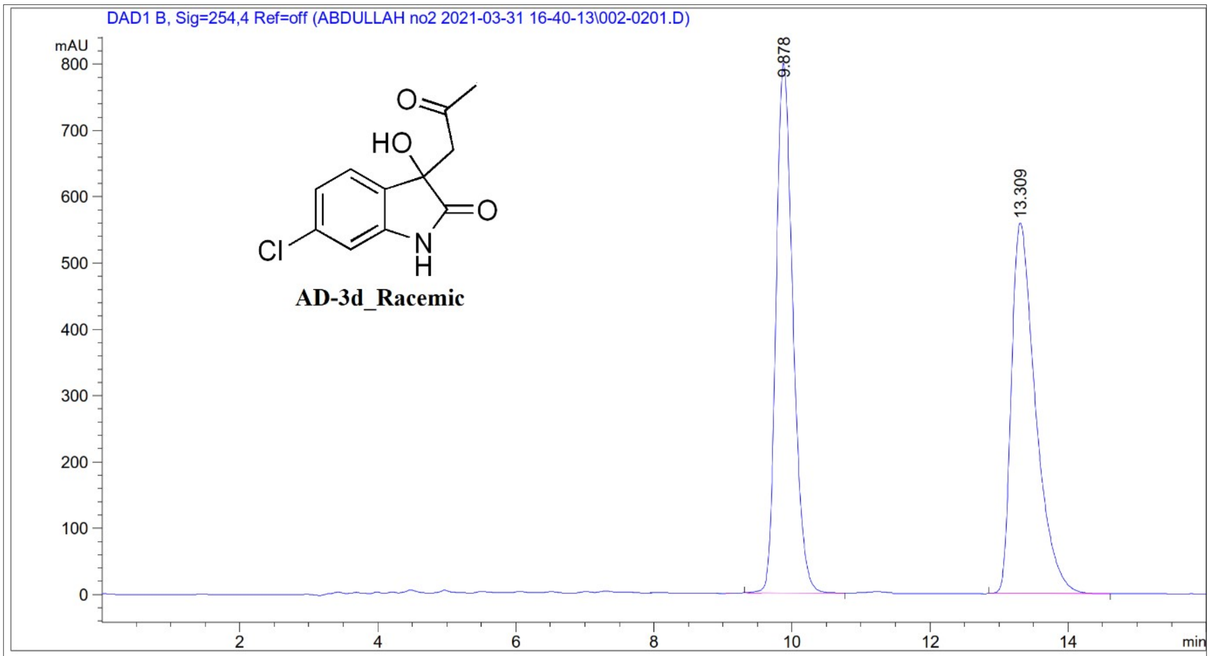


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.984	VV R	0.2368	2.57394e4	1643.65637	50.3374
2	12.878	BB	0.3426	2.53944e4	1145.51294	49.6626

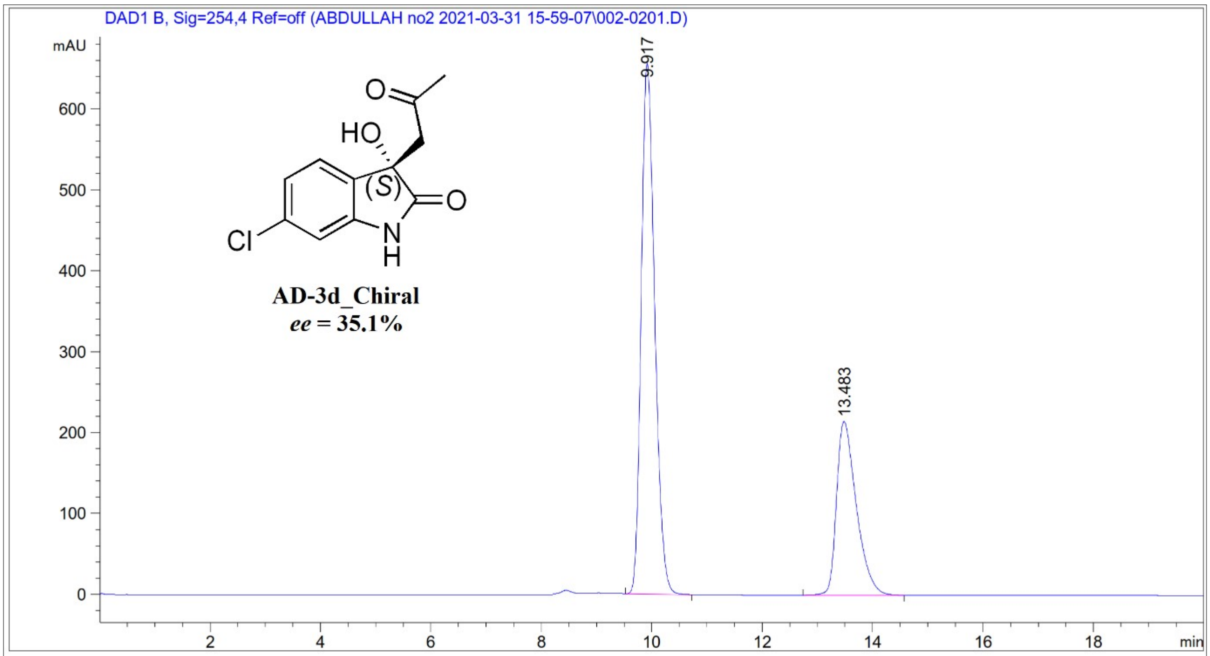


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.982	BB	0.2870	7.06271e4	3447.68164	87.7590
2	12.938	BB	0.3420	9851.34473	445.43735	12.2410

2.1.4 HPLC analysis of aldol product 3d

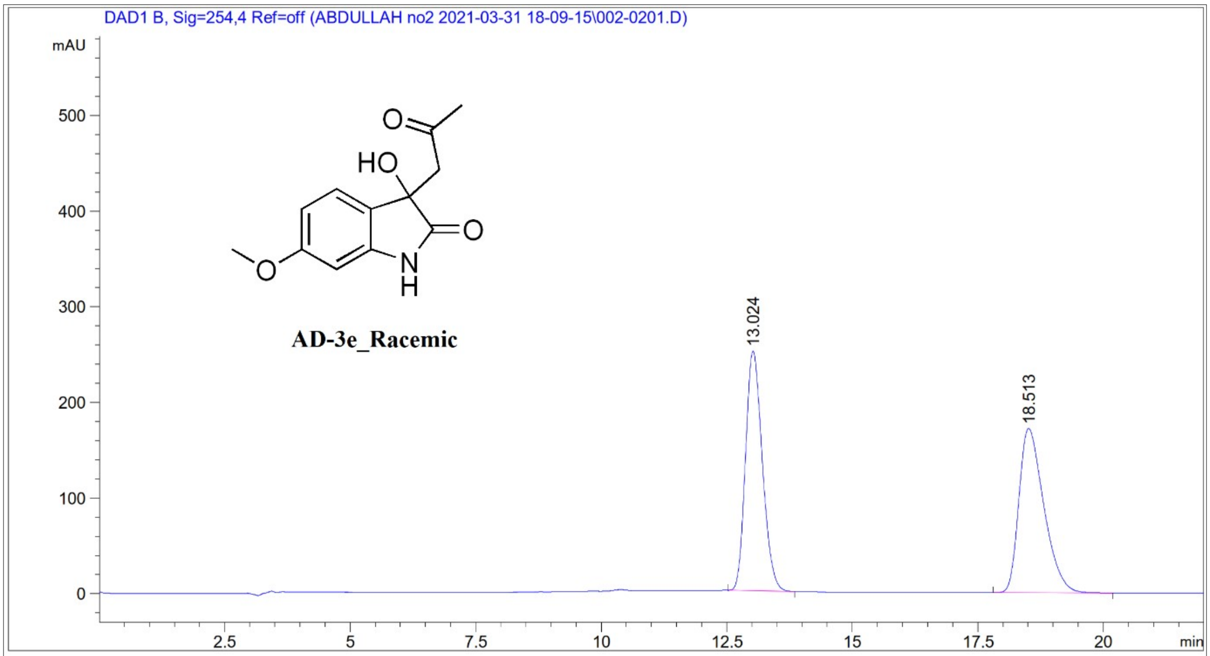


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.878	VB R	0.2621	1.38225e4	799.16718	50.4480
2	13.309	BB	0.3676	1.35770e4	558.57861	49.5520

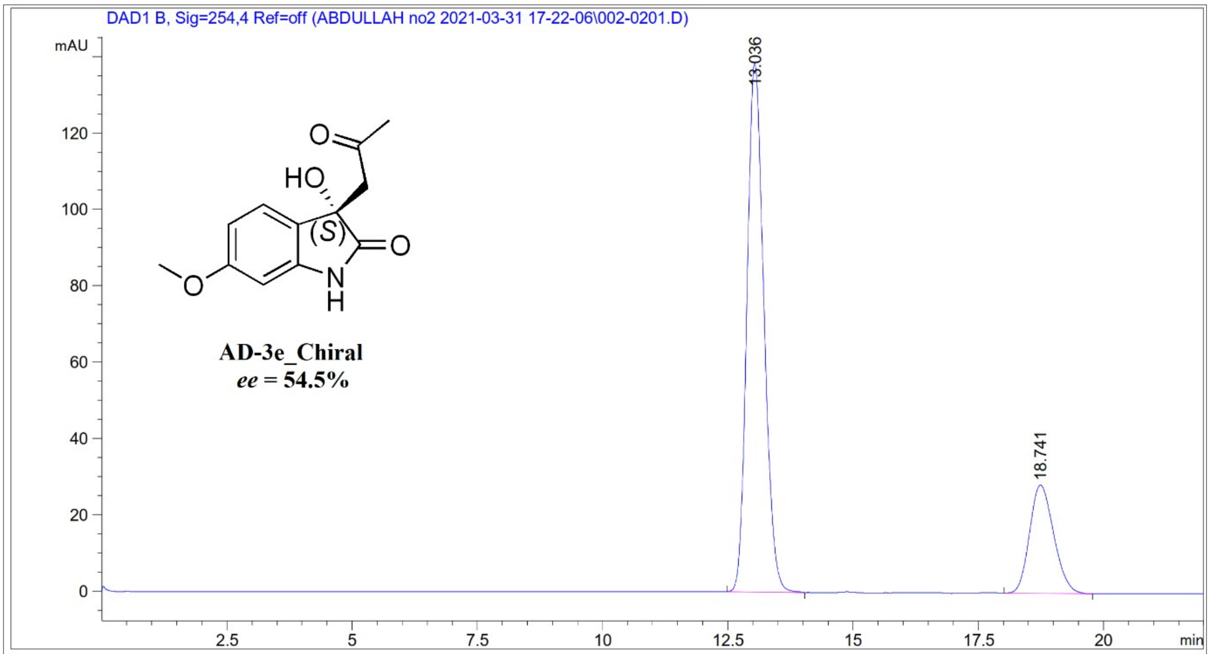


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.917	BB	0.2618	1.11234e4	656.19049	67.5298
2	13.483	BB	0.3766	5348.43848	214.75438	32.4702

2.1.5 HPLC analysis of aldol product 3e

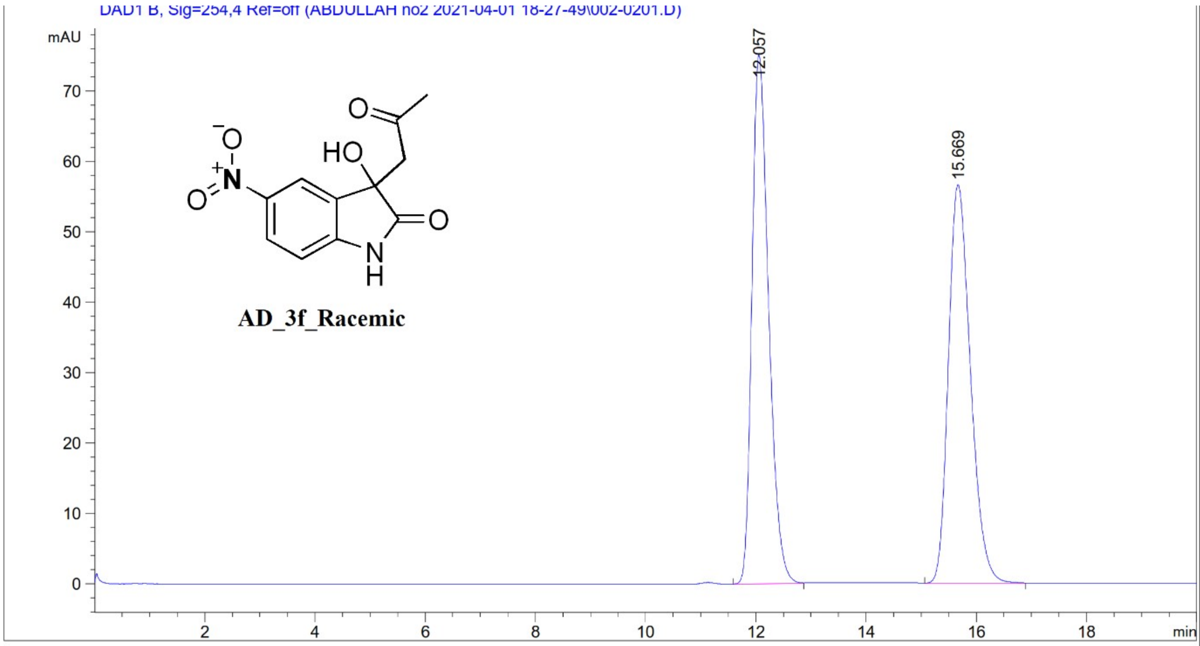


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.024	BB	0.3614	5826.97900	250.46043	49.5816
2	18.513	BB	0.5245	5925.32178	171.74911	50.4184

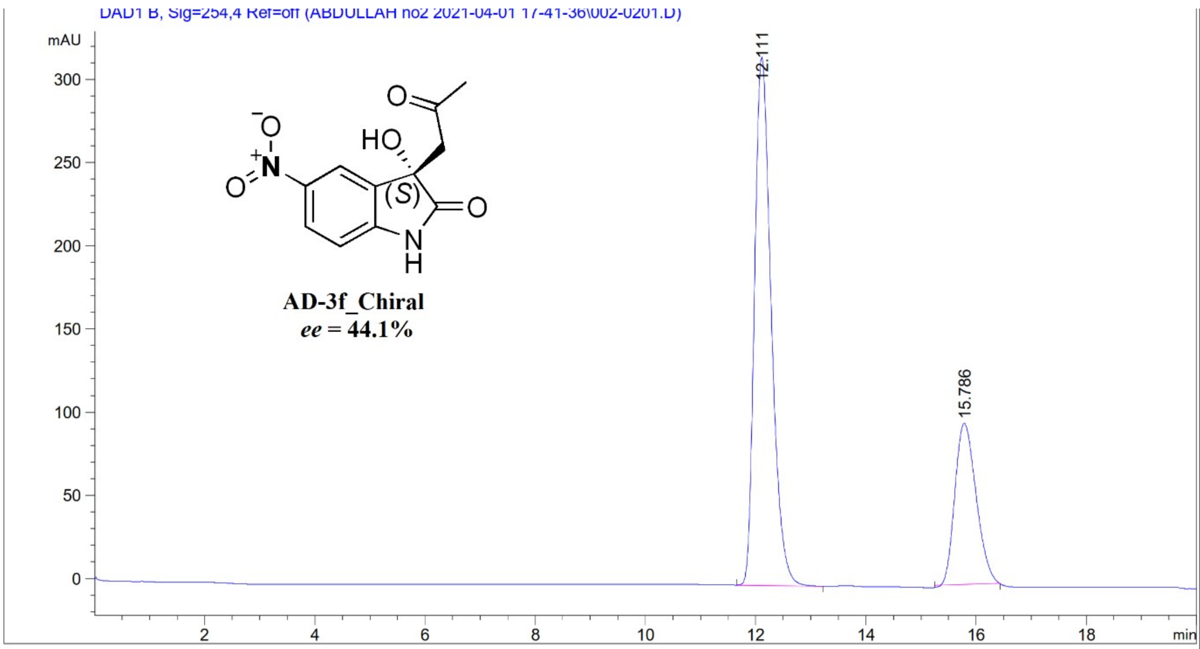


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.036	BB	0.3616	3247.21118	138.47548	77.2700
2	18.741	BB	0.5101	955.20935	28.42458	22.7300

2.1.6 HPLC analysis of aldol product 3f

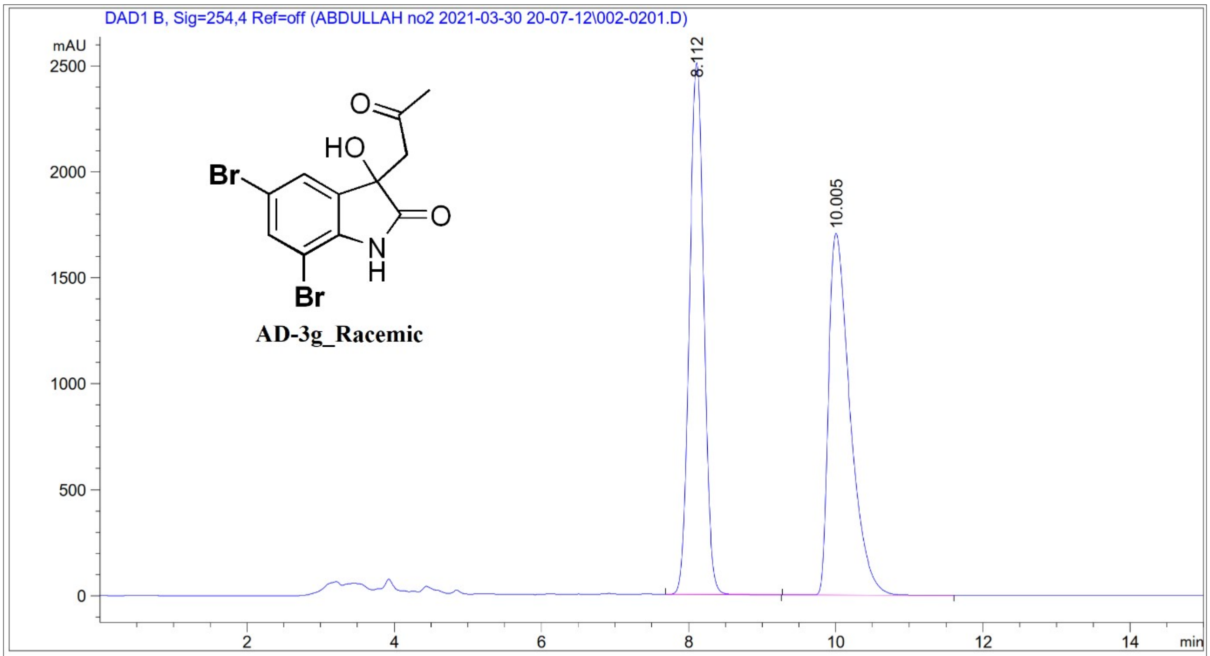


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.057	BB	0.3248	1577.82886	75.19449	50.0235
2	15.669	BB	0.4327	1576.34912	56.64012	49.9765

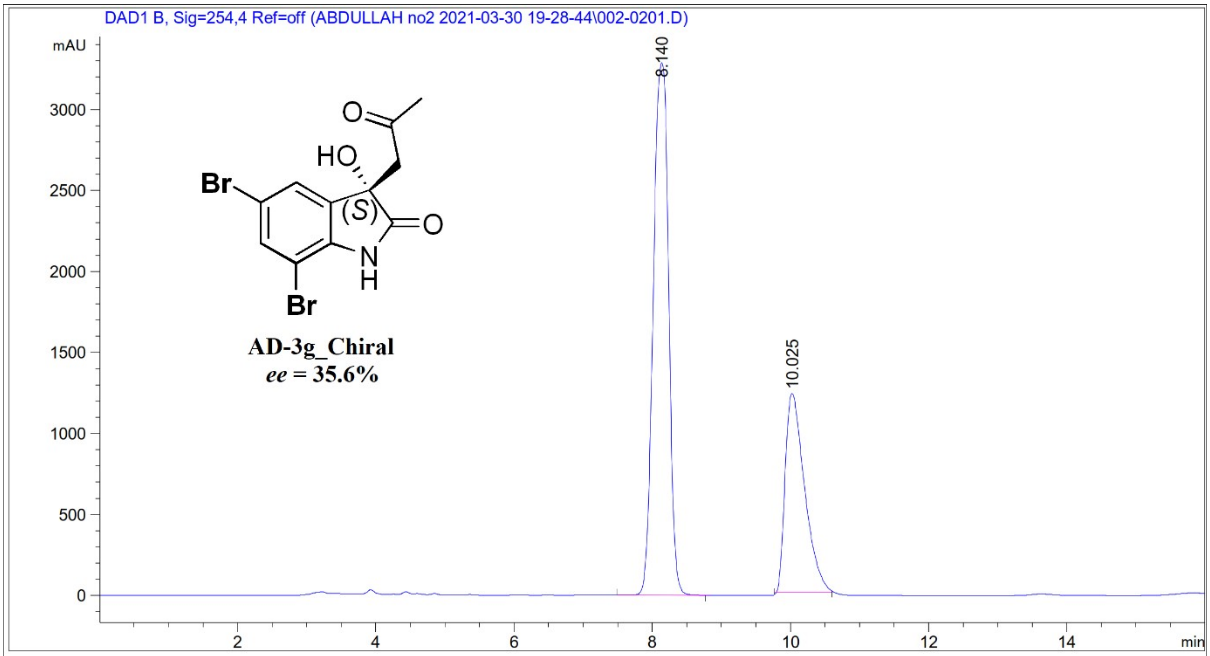


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.111	BB	0.3250	6663.52881	317.28882	72.0386
2	15.786	MM	0.4451	2586.40869	96.83911	27.9614

2.1.7 HPLC analysis of aldol product 3g

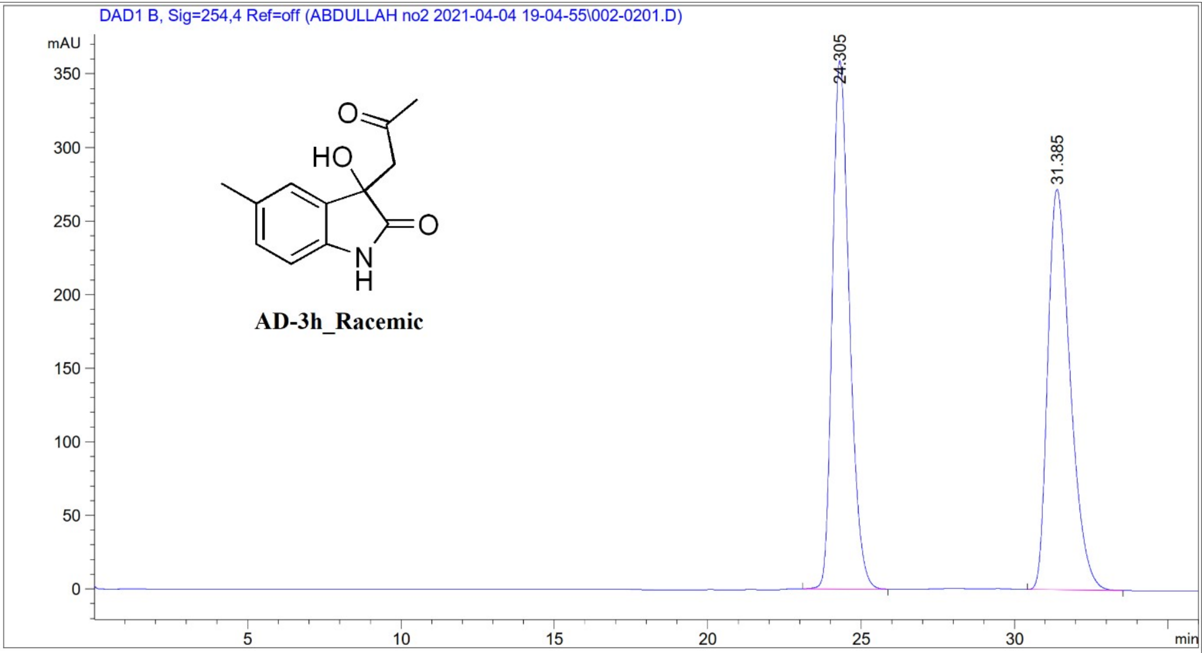


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.112	BB	0.2130	3.41794e4	2508.04443	49.5842
2	10.005	VB R	0.3093	3.47527e4	1707.64722	50.4158

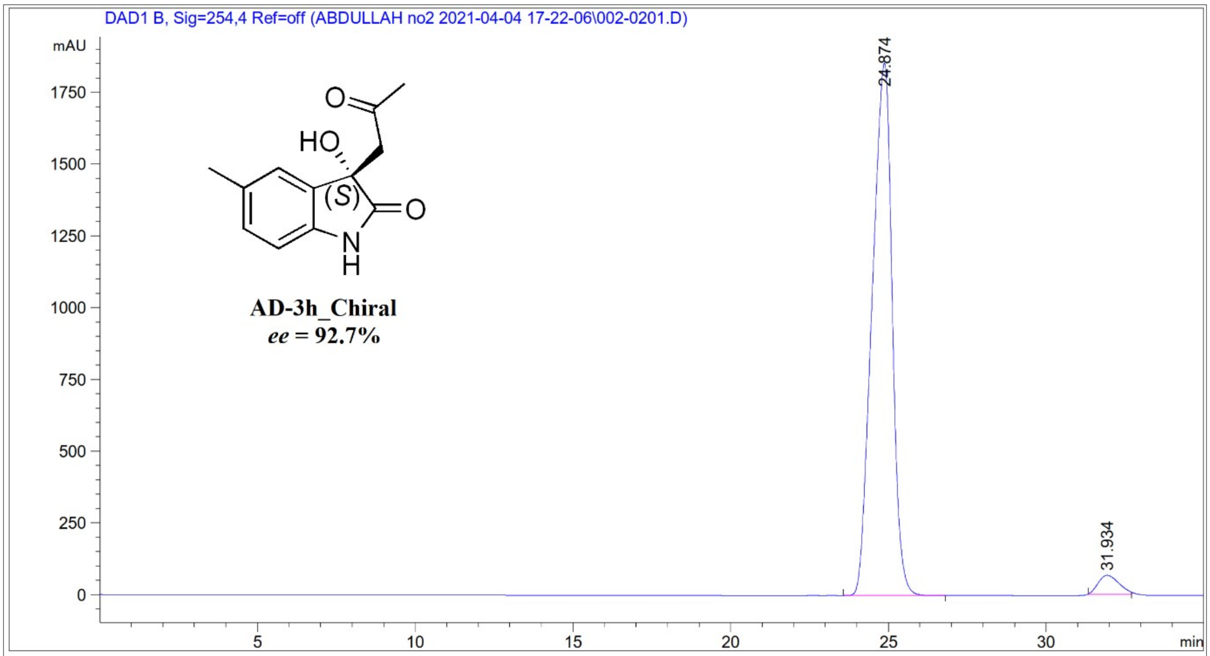


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.140	BB	0.2452	5.04676e4	3285.74951	67.7791
2	10.025	MM	0.3259	2.39913e4	1227.11060	32.2209

2.1.8 HPLC analysis of aldol product 3h

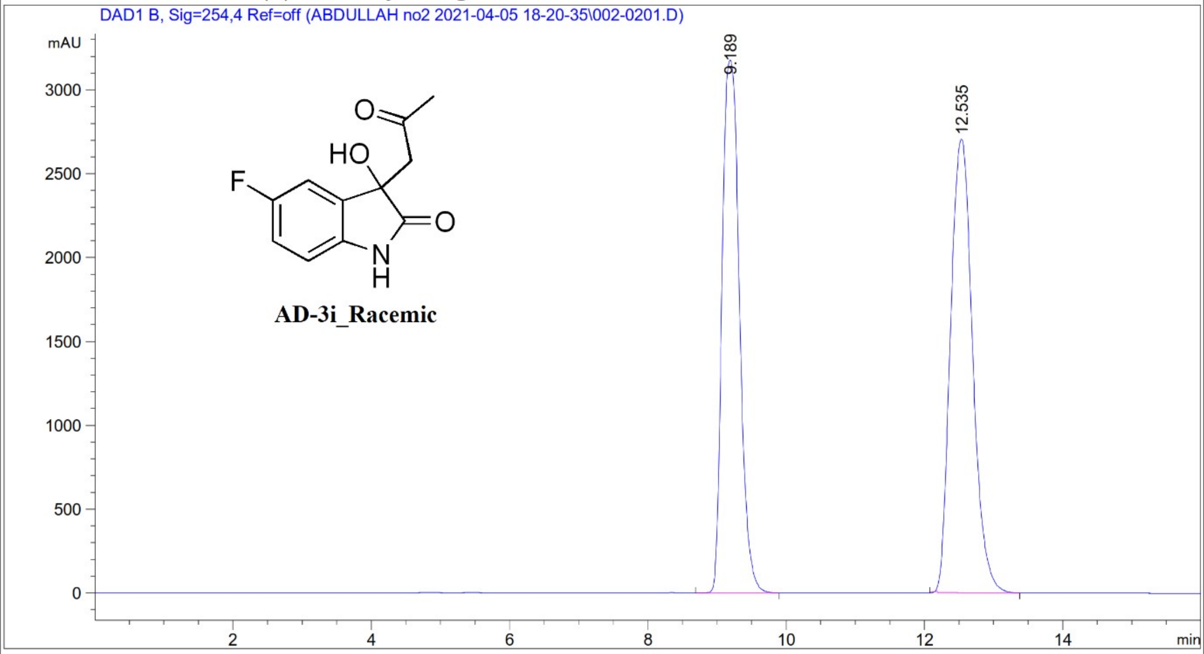


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.305	BB	0.6008	1.39336e4	359.02243	50.1281
2	31.385	BB	0.7783	1.38624e4	272.13998	49.8719

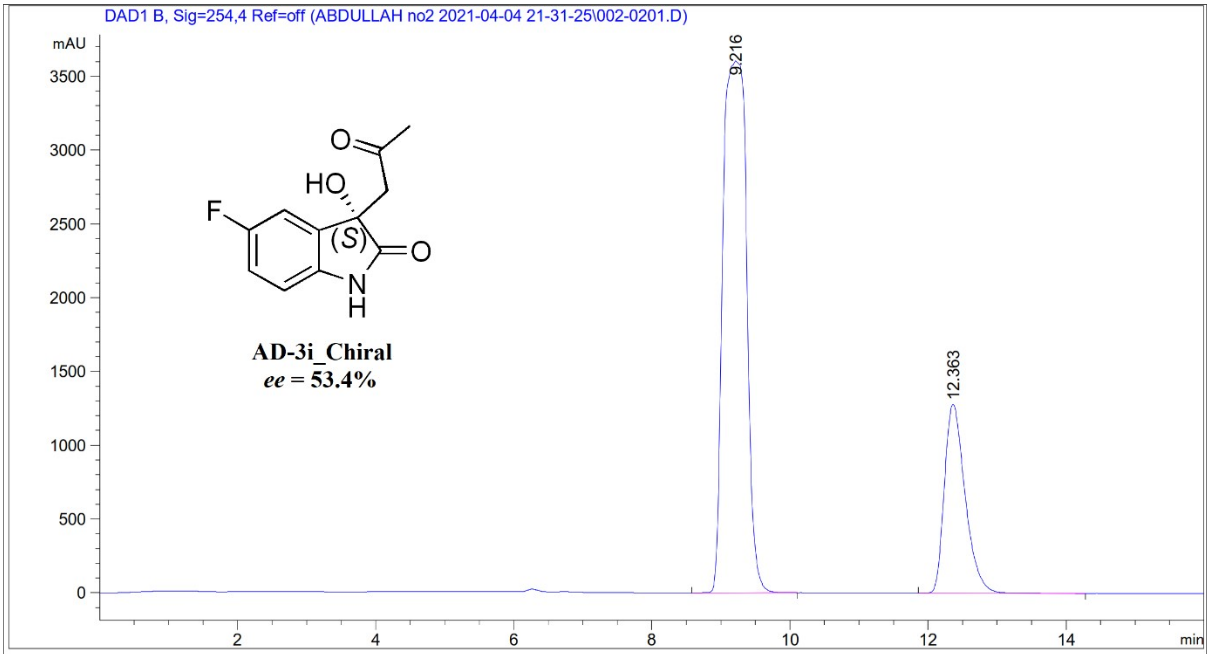


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.874	BB	0.7080	8.13830e4	1854.50378	96.3582
2	31.934	MM	0.7707	3075.83105	66.51230	3.6418

2.1.9 HPLC analysis of aldol product 3i

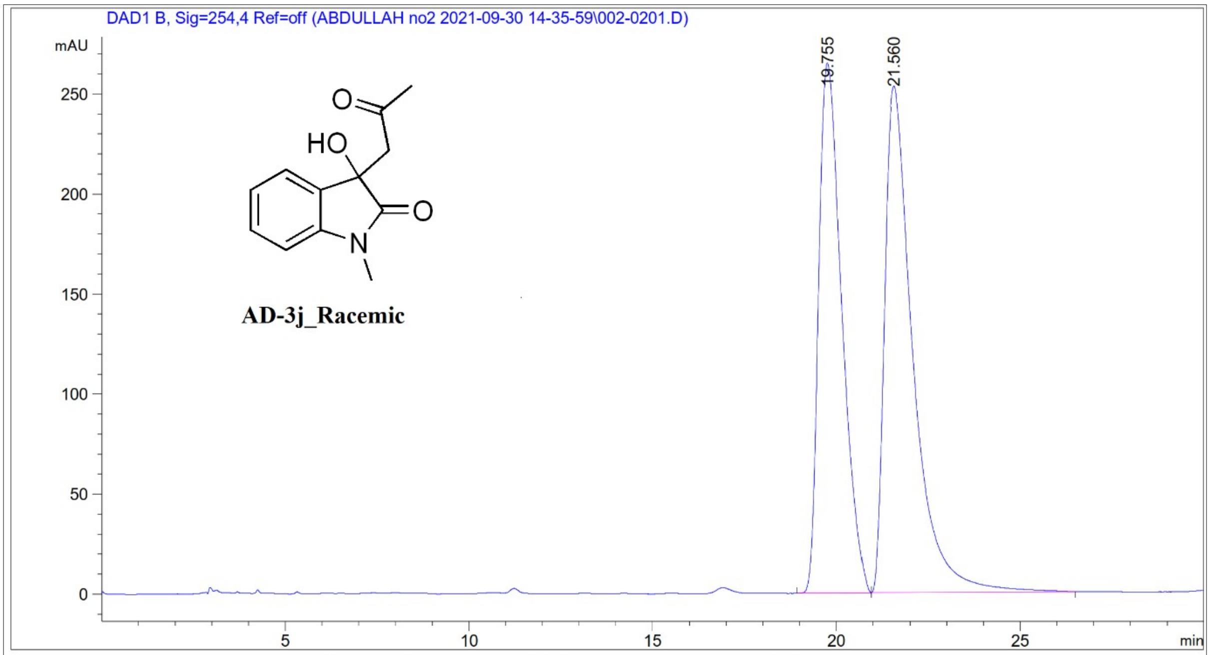


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.189	BB	0.2753	5.53588e4	3177.56543	48.4942
2	12.535	MM	0.3645	5.87966e4	2688.46362	51.5058

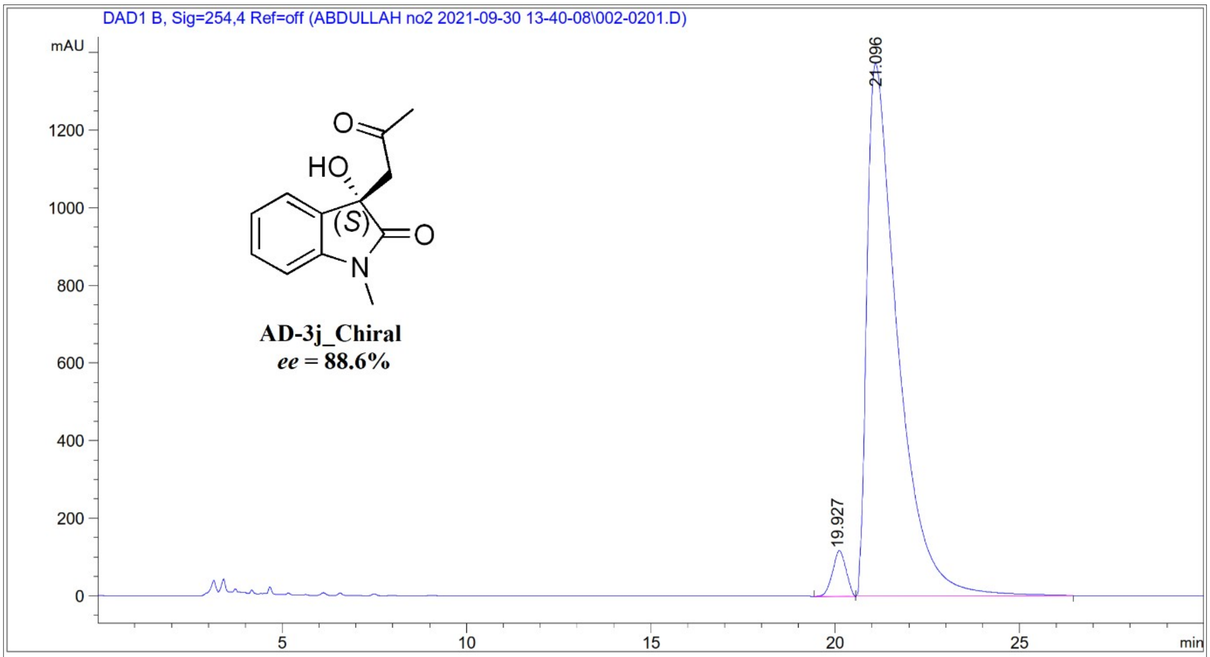


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.216	BB	0.2892	8.56764e4	3605.45386	76.1941
2	12.363	BB	0.3241	2.67686e4	1279.27124	23.8059

2.1.10 HPLC analysis of aldol product 3j

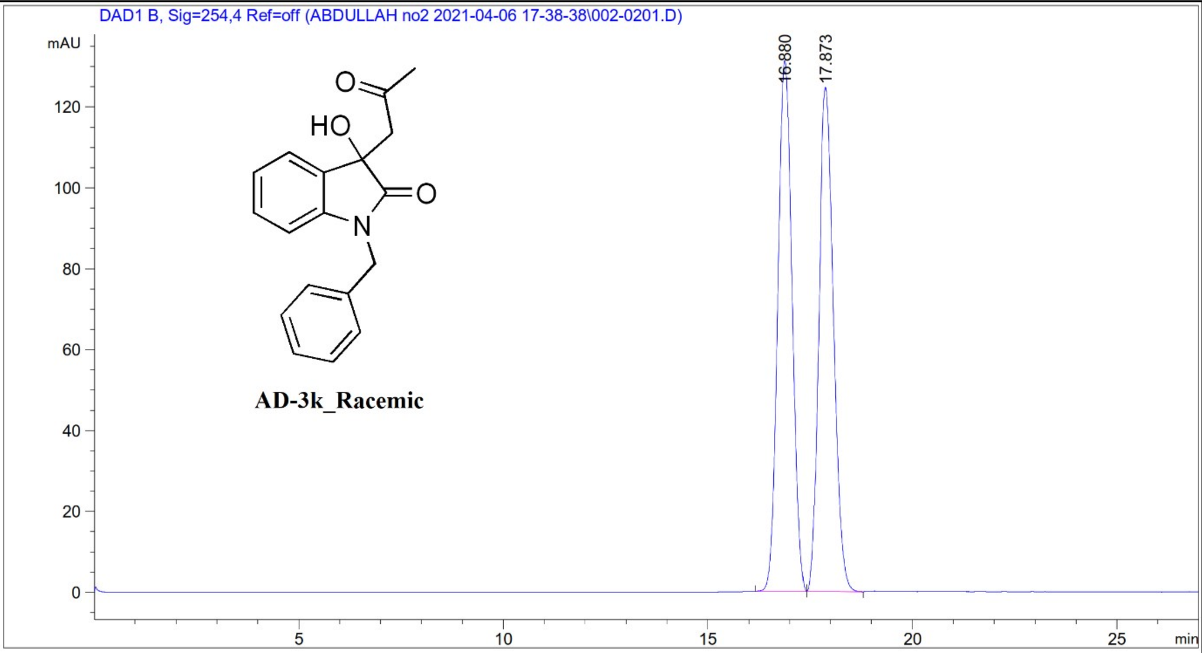


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.755	BV	0.7136	1.26207e4	265.02744	47.0325
2	21.560	VB	0.8247	1.42133e4	253.28951	52.9675

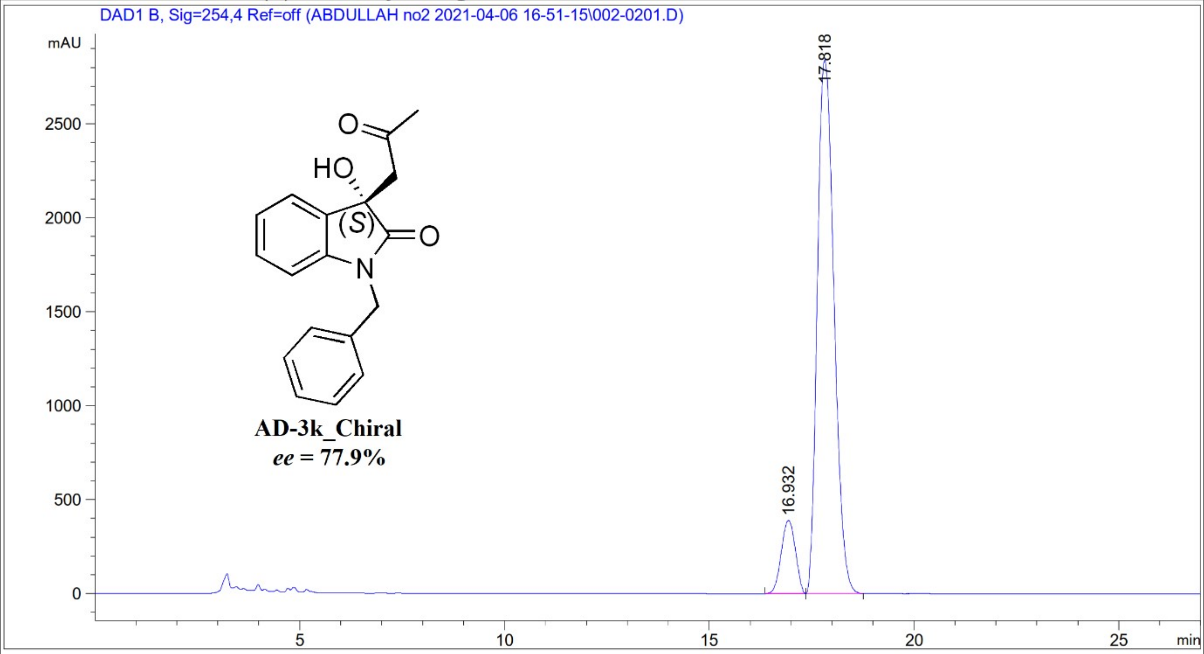


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.927	BV E	0.6351	4834.83154	118.22055	5.6933
2	21.096	VB R	0.8522	8.00868e4	1373.54846	94.3067

2.1.11 HPLC analysis of aldol product 3k

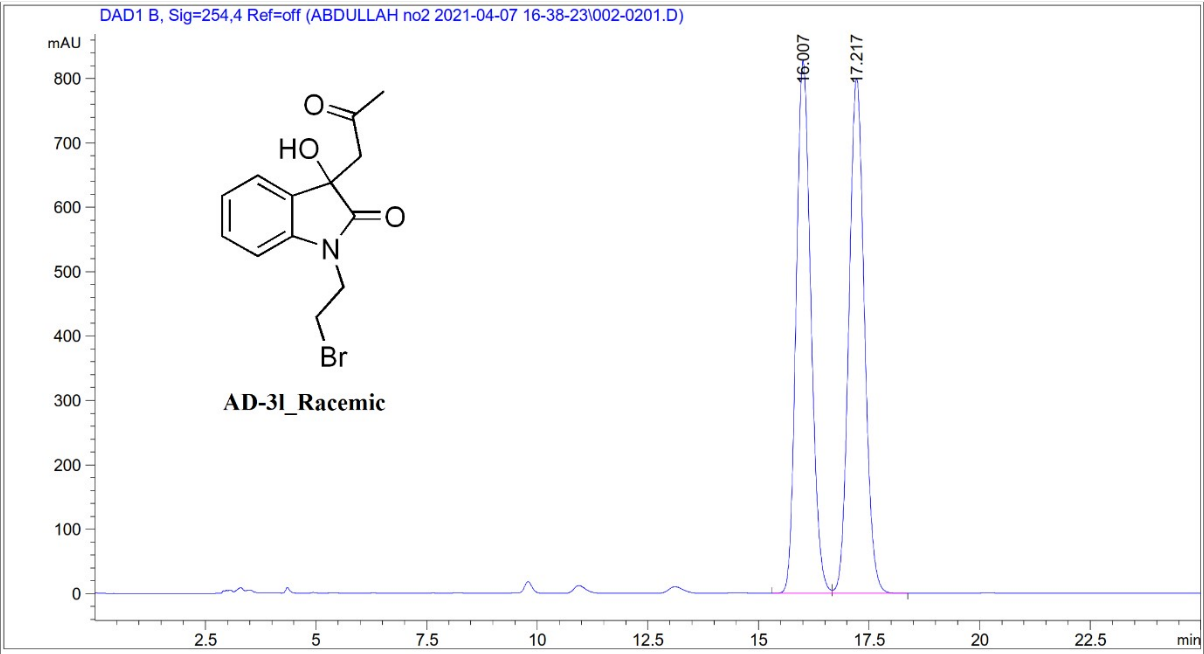


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.880	BV	0.3865	3268.51514	131.25287	49.8669
2	17.873	VB	0.4093	3285.96484	124.73854	50.1331

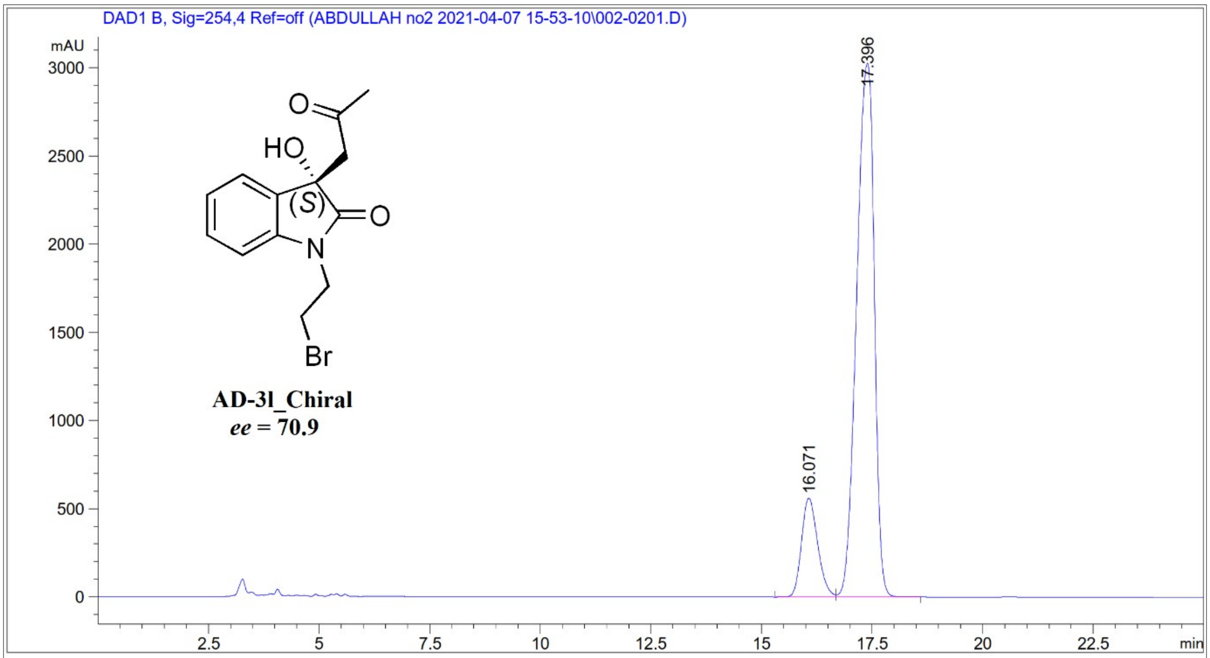


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.932	BV E	0.4030	1.00545e4	389.70786	11.0342
2	17.818	VB R	0.4471	8.10663e4	2839.71265	88.9658

2.1.12 HPLC analysis of aldol product 3l

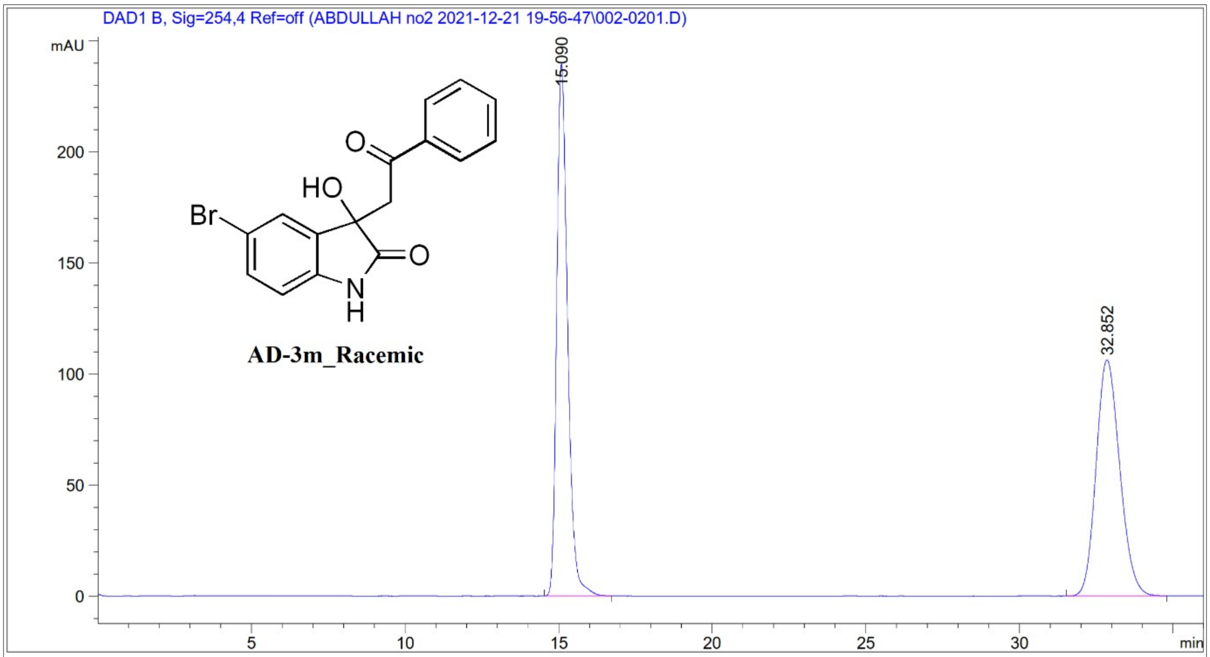


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.007	BV	0.3596	1.91294e4	827.71472	49.6179
2	17.217	VB	0.3772	1.94240e4	800.09637	50.3821

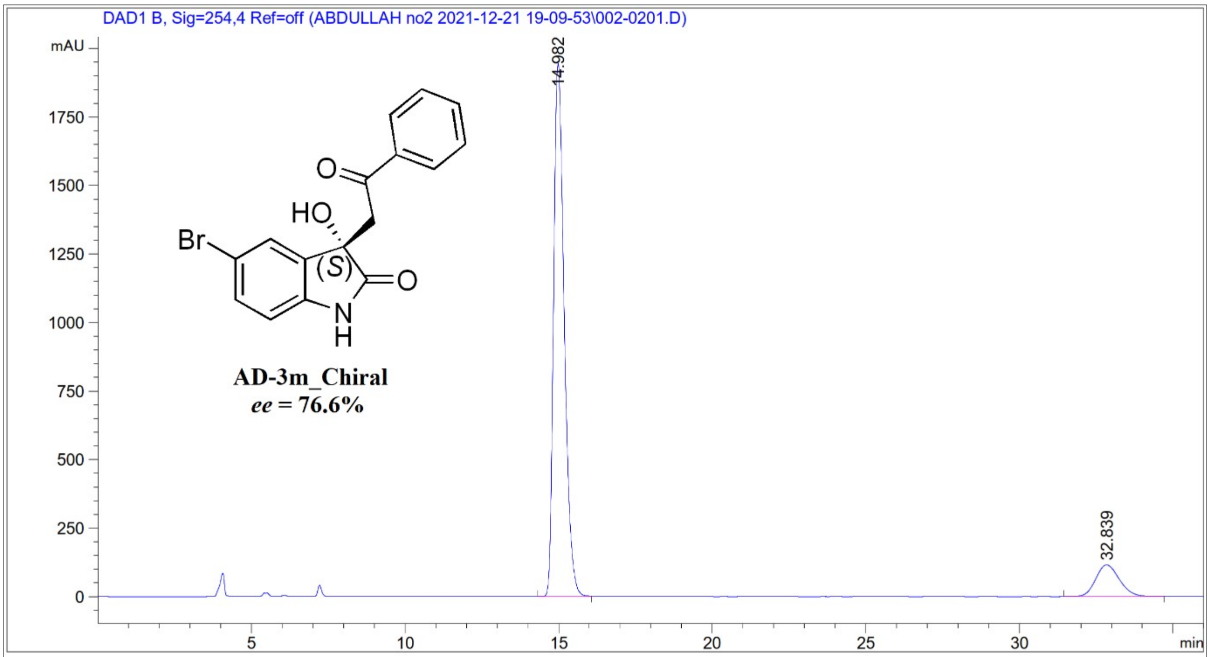


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.071	BV	0.3949	1.42838e4	561.33057	14.5532
2	17.396	VB	0.4393	8.38652e4	3026.50610	85.4468

2.1.13 HPLC analysis of aldol product 3m

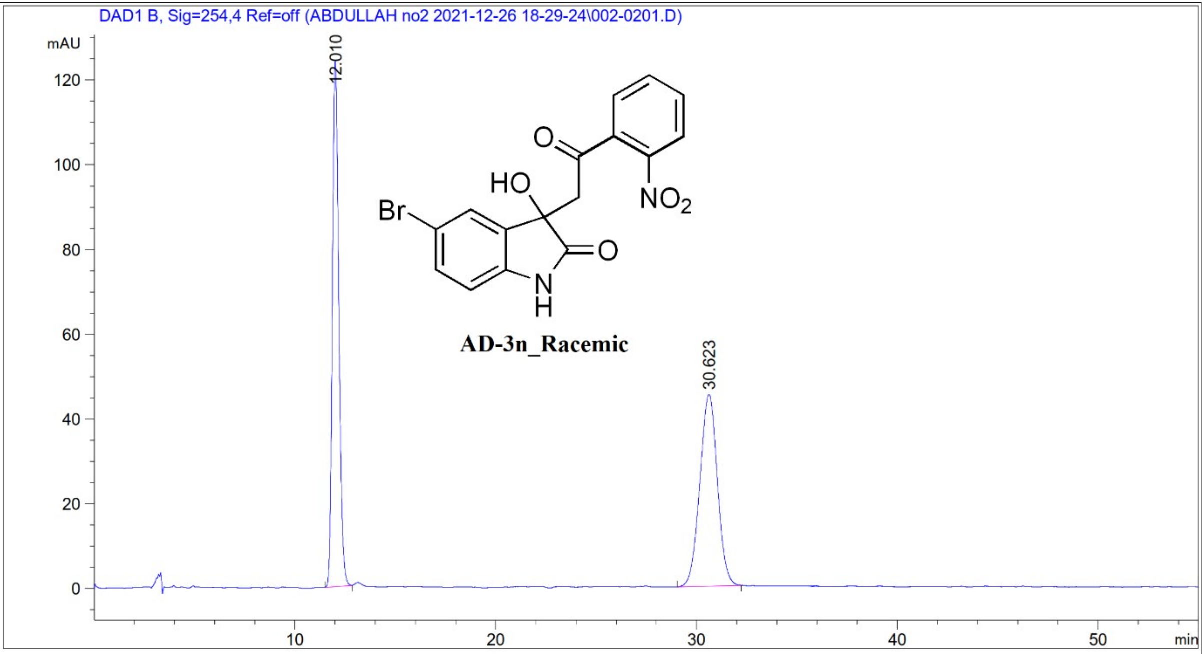


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.090	BB	0.3761	5881.06738	239.79439	50.7737
2	32.852	BB	0.8358	5701.82861	106.29649	49.2263

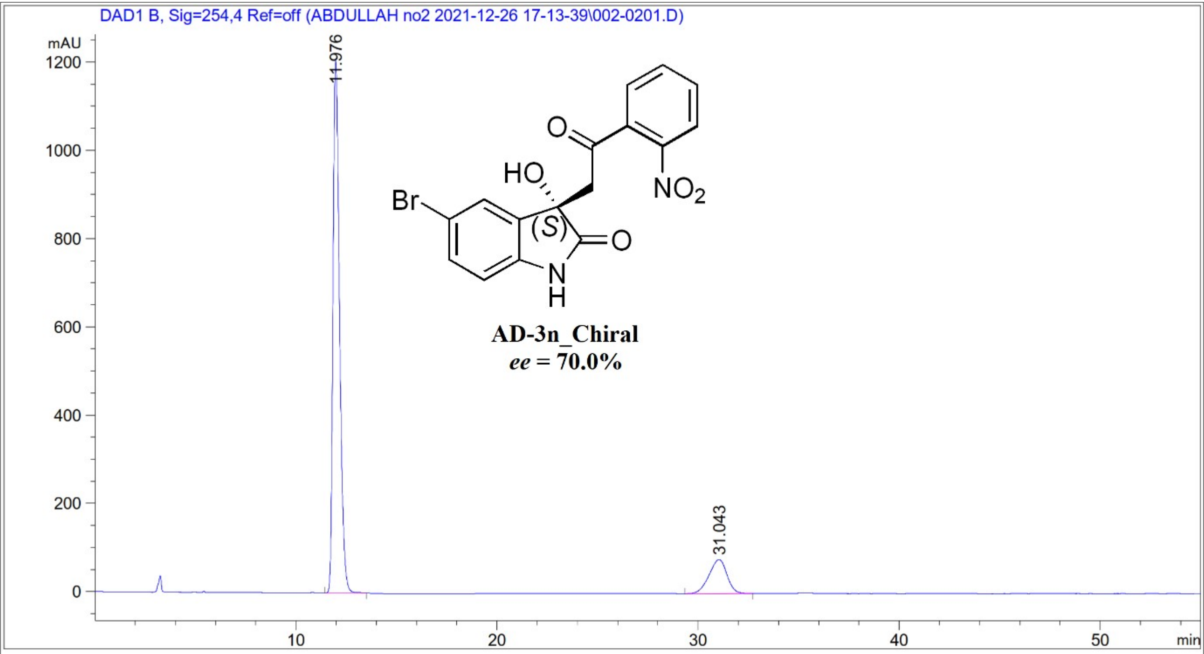


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.982	BB	0.3697	4.66397e4	1945.57837	88.2982
2	32.839	BB	0.8247	6180.93652	115.43489	11.7018

2.1.14 HPLC analysis of aldol product 3n

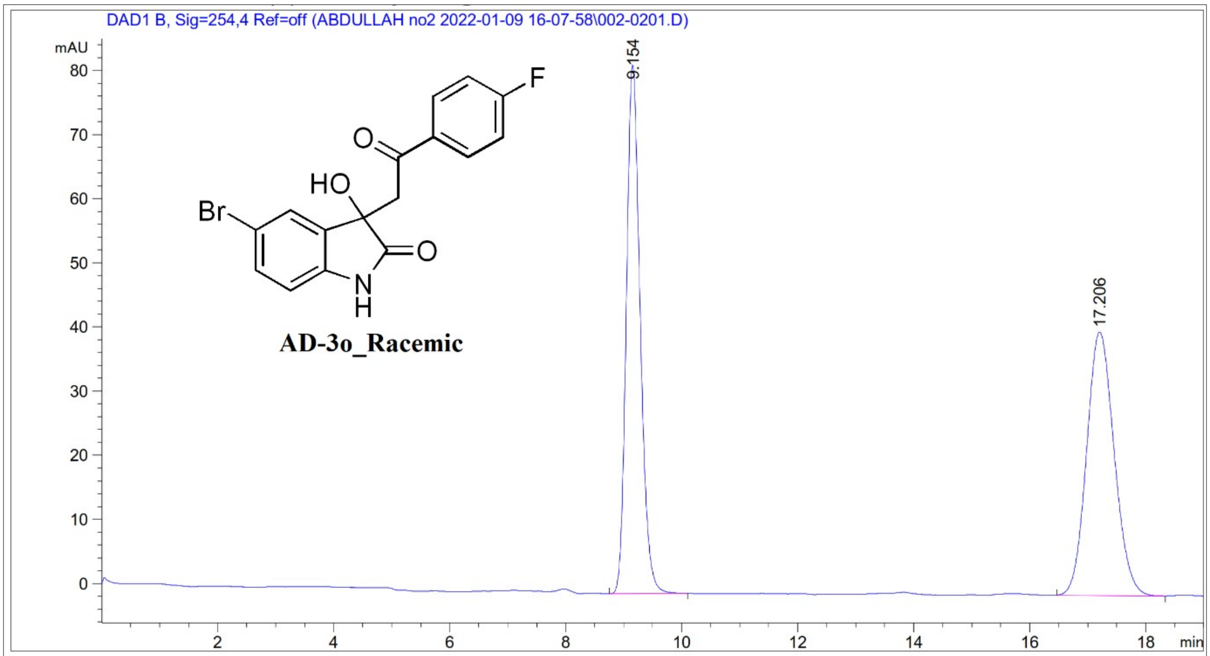


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.010	BB	0.3432	2758.73828	124.15577	50.0638
2	30.623	BB	0.9044	2751.70508	45.32007	49.9362

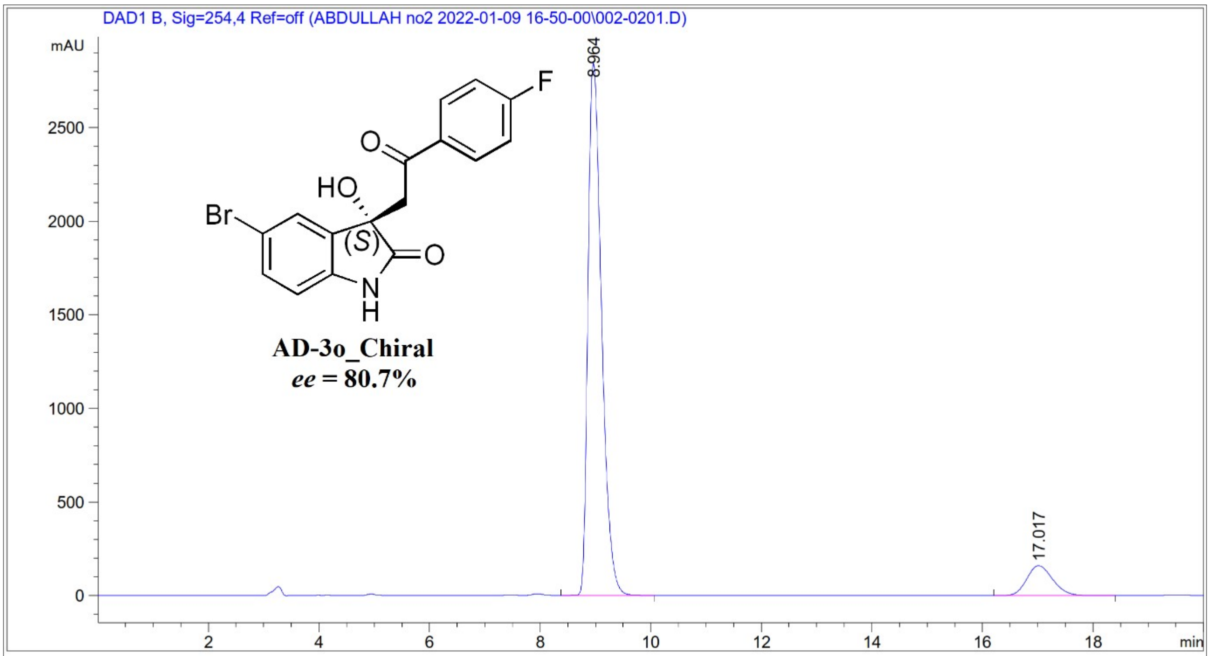


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.976	BB	0.3502	2.73173e4	1206.26953	85.0029
2	31.043	BB	0.9323	4819.59326	77.41415	14.9971

2.1.15 HPLC analysis of aldol product 3o

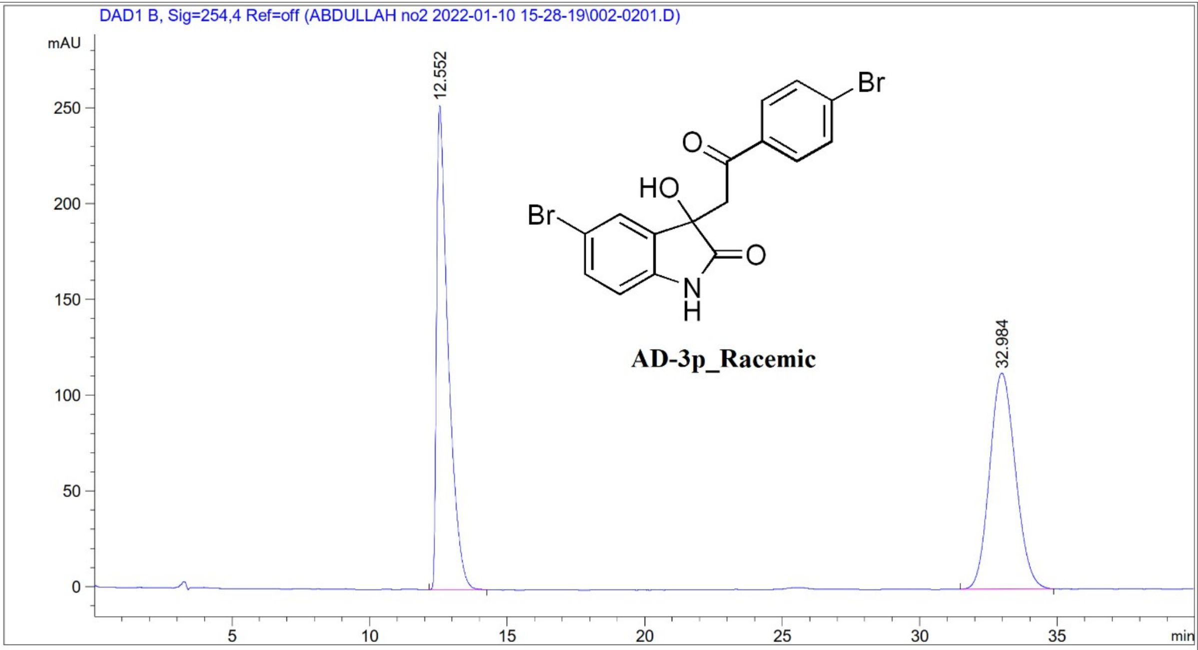


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.154	BB	0.2556	1368.07349	82.45618	50.2374
2	17.206	BB	0.5027	1355.14392	41.10032	49.7626

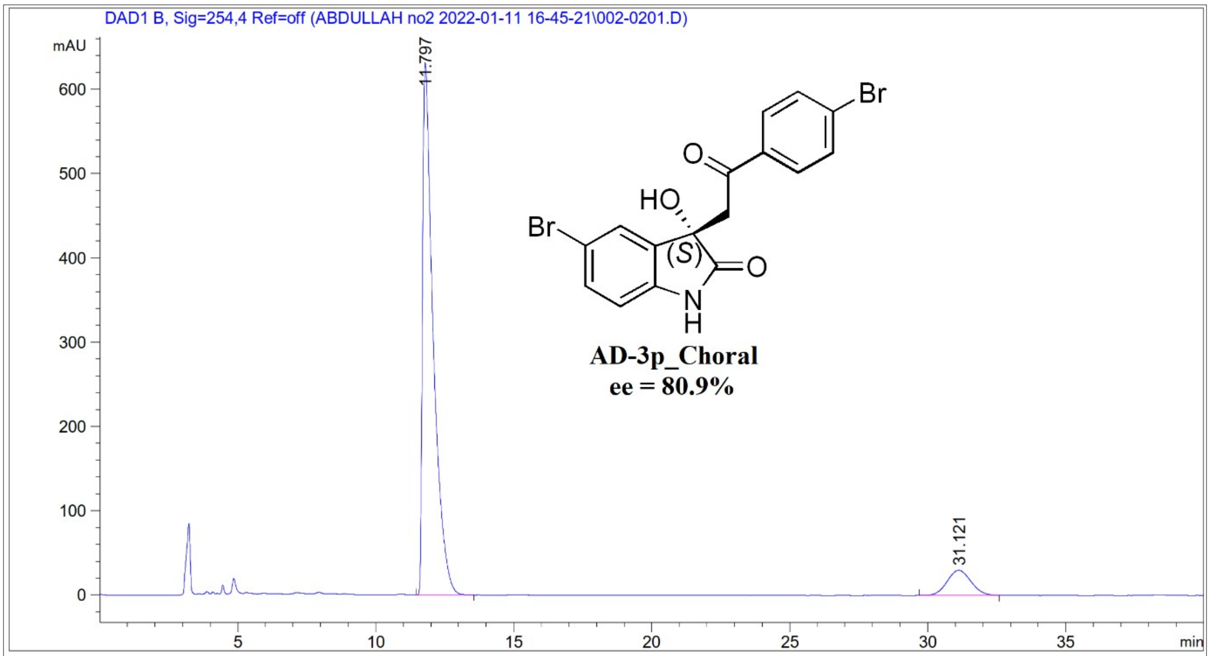


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.964	BB	0.2645	4.83746e4	2843.65649	90.2798
2	17.017	BB	0.5060	5208.35205	159.88551	9.7202

2.1.16 HPLC analysis of aldol product 3p

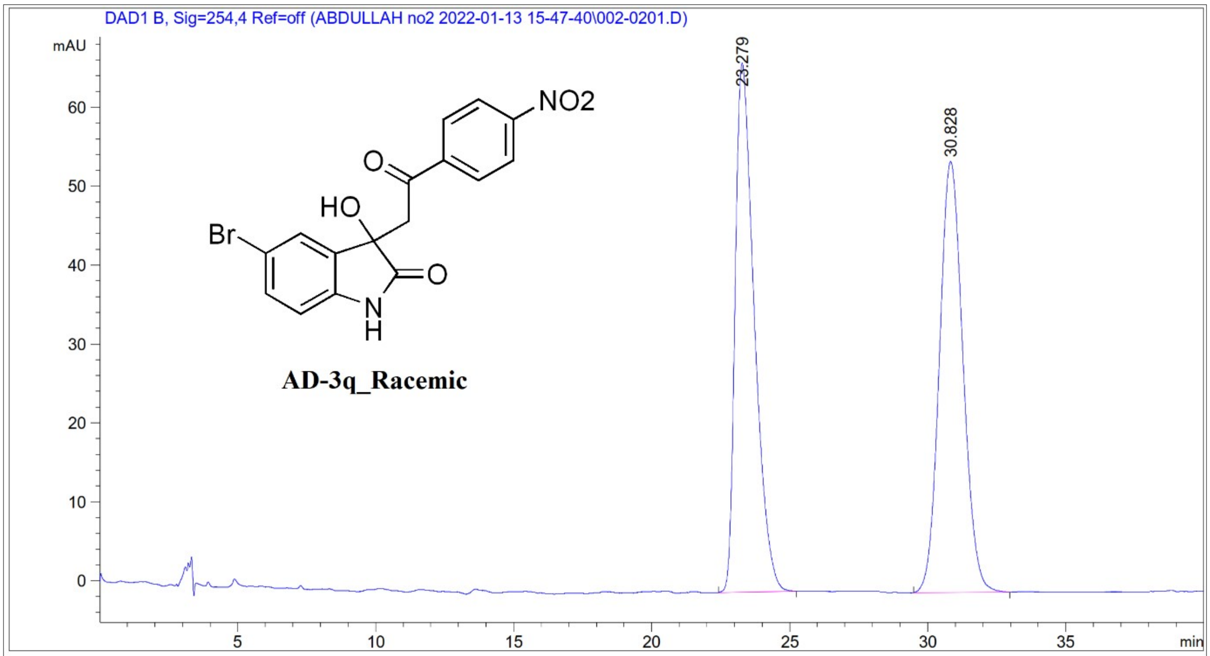


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.552	BB	0.4247	7426.74023	252.80455	50.1364
2	32.984	BB	0.9833	7386.33105	112.89825	49.8636

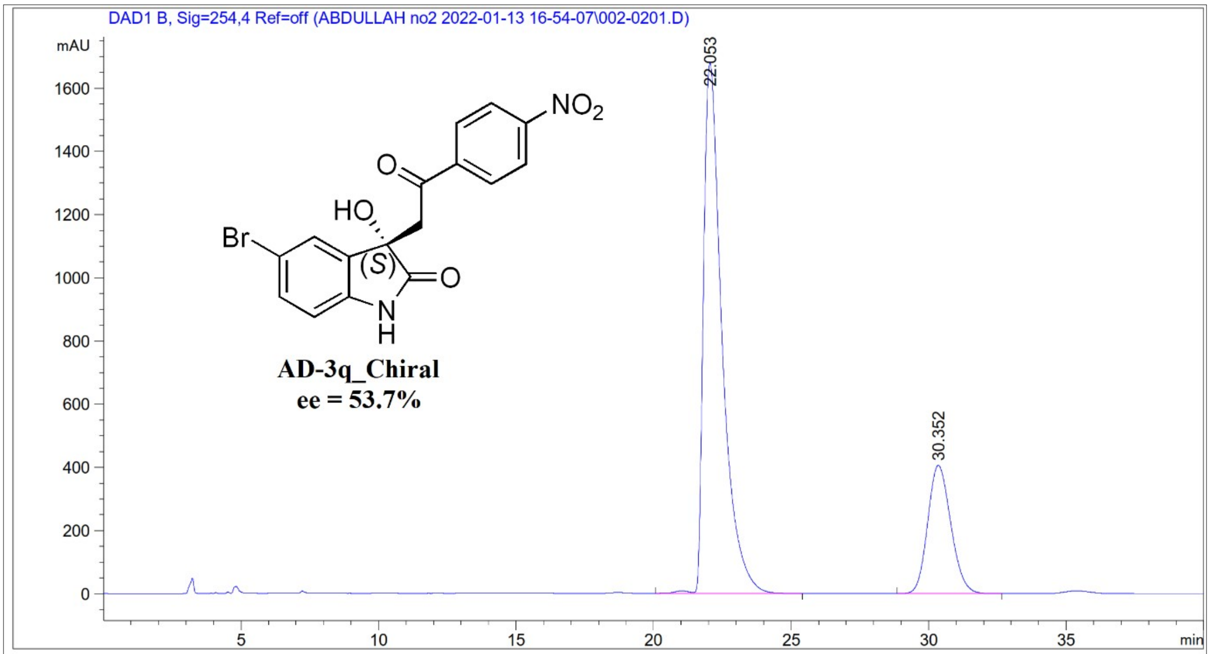


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.797	BB	0.3878	1.69676e4	630.90466	90.4413
2	31.121	BB	0.8332	1793.28992	29.83571	9.5587

2.1.17 HPLC analysis of aldol product 3q

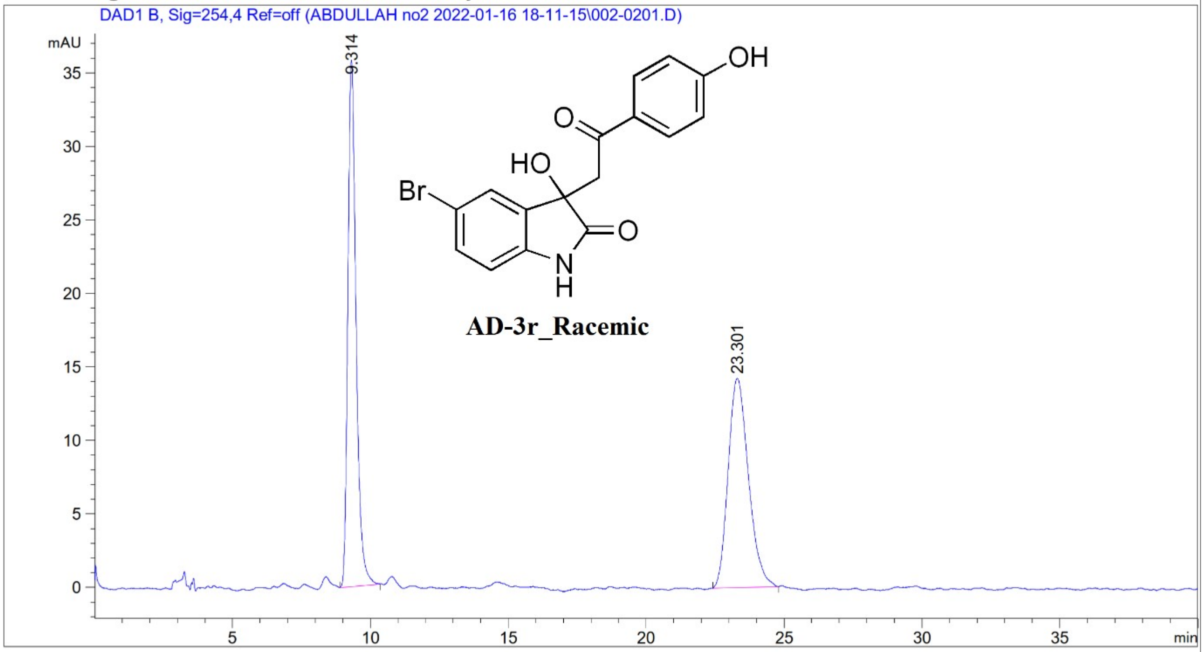


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.279	BB	0.7104	3220.98560	67.05275	49.8893
2	30.828	BB	0.8649	3235.28467	54.62811	50.1107

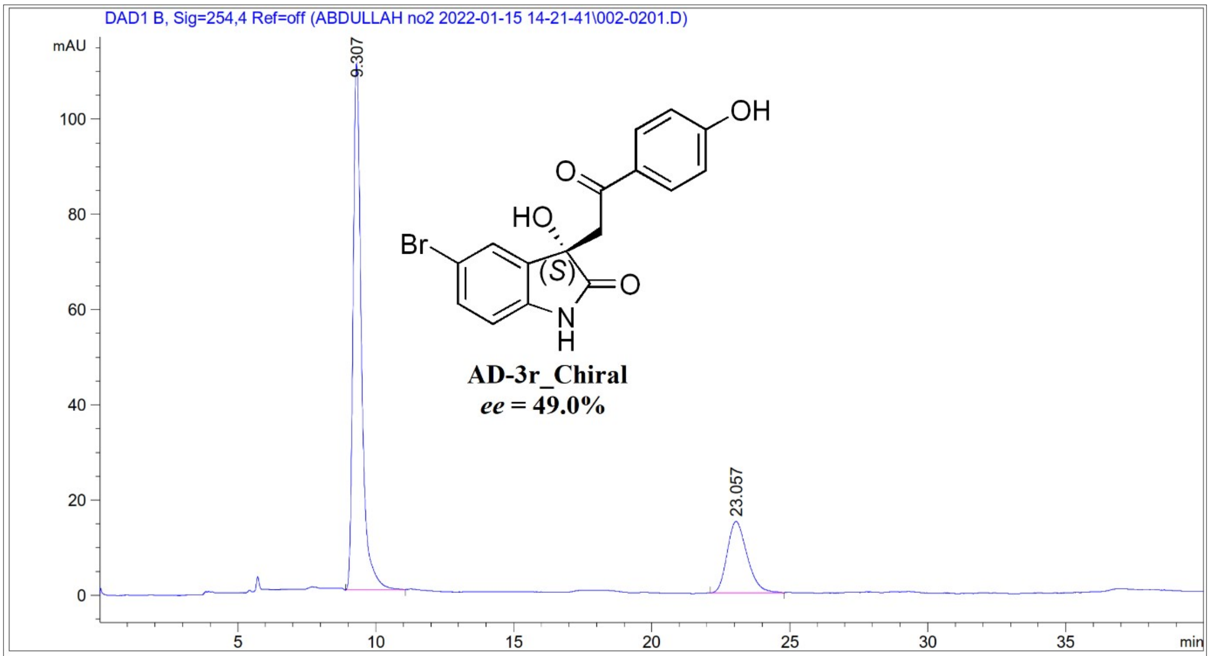


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.053	VB R	0.7072	7.86958e4	1678.40186	76.8404
2	30.352	BB	0.9080	2.37188e4	405.75281	23.1596

2.1.18 HPLC analysis of aldol product 3r

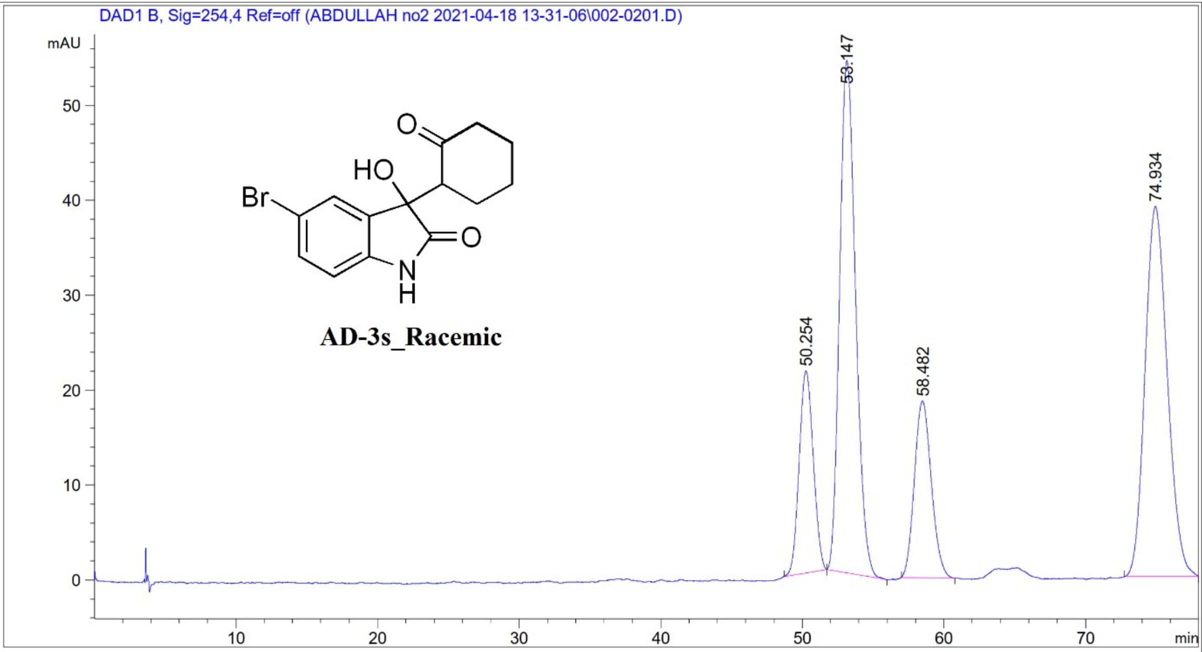


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.314	BB	0.3132	740.46729	35.81948	50.4224
2	23.301	BB	0.6929	728.06122	14.23102	49.5776

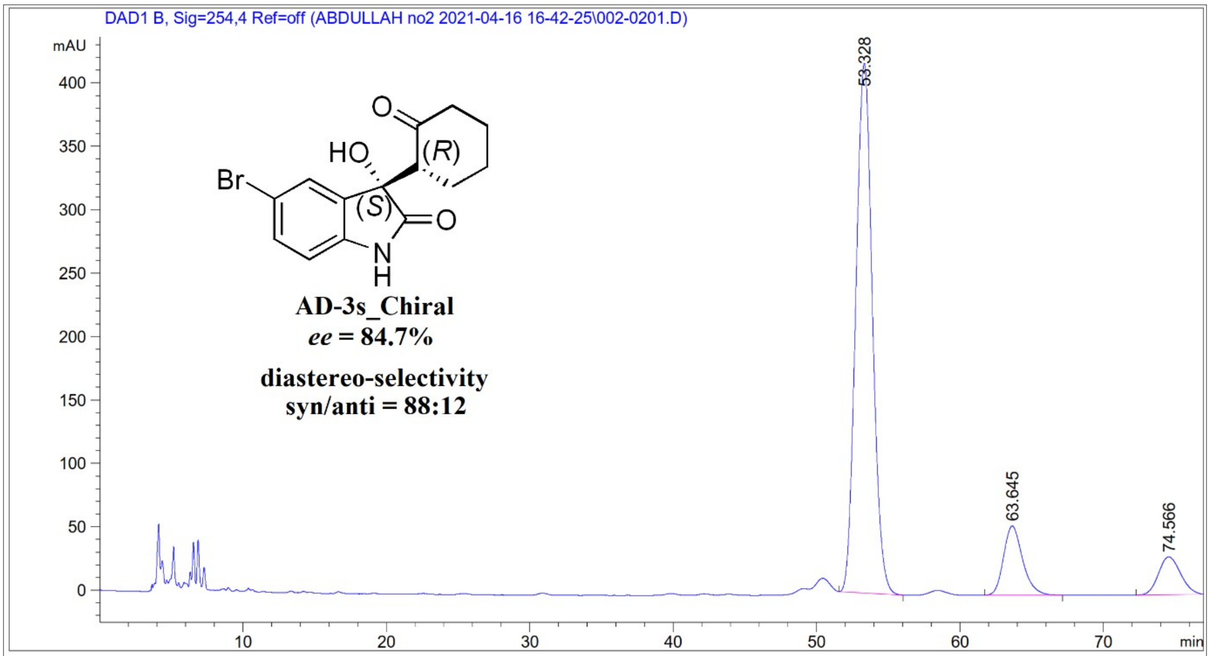


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.307	BB	0.3124	2267.67749	110.06409	75.0245
2	23.057	BB	0.6948	754.90320	15.01004	24.9755

2.1.19 HPLC analysis of aldol product 3s



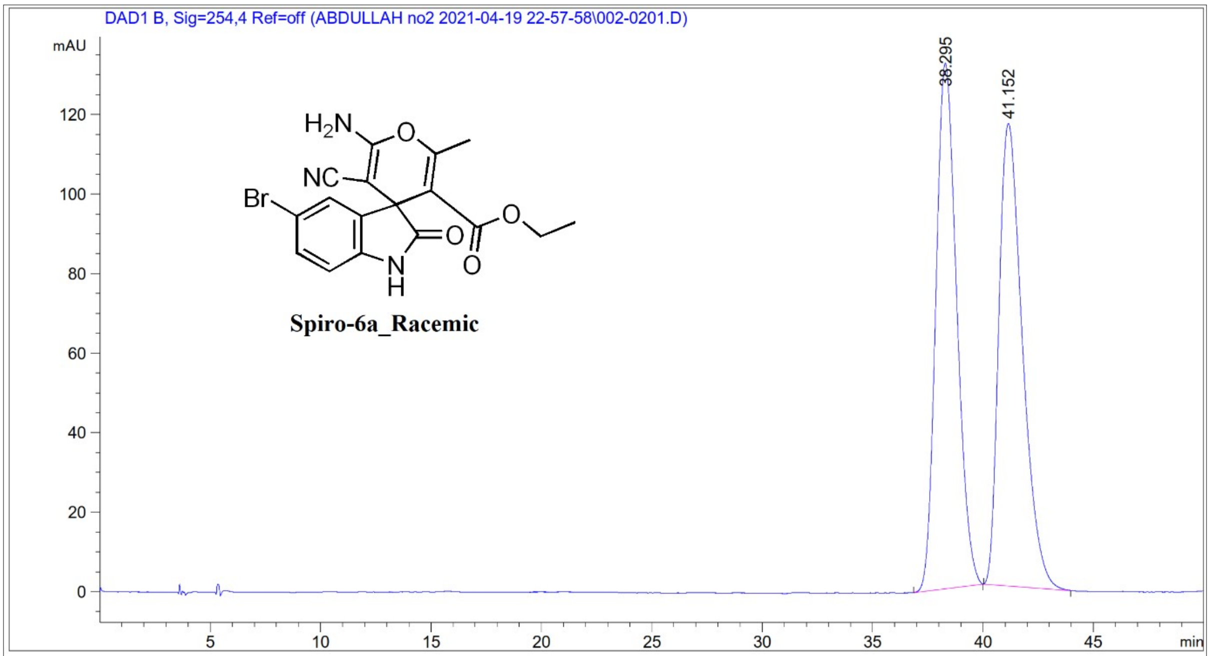
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	50.254	BB	0.9063	1471.26953	21.28909	12.7645
2	53.147	BB	1.1301	4229.72070	53.98263	36.6965
3	58.482	BB	0.9961	1555.46802	18.66837	13.4950
4	74.934	BBA	1.3513	4269.76367	38.99911	37.0439



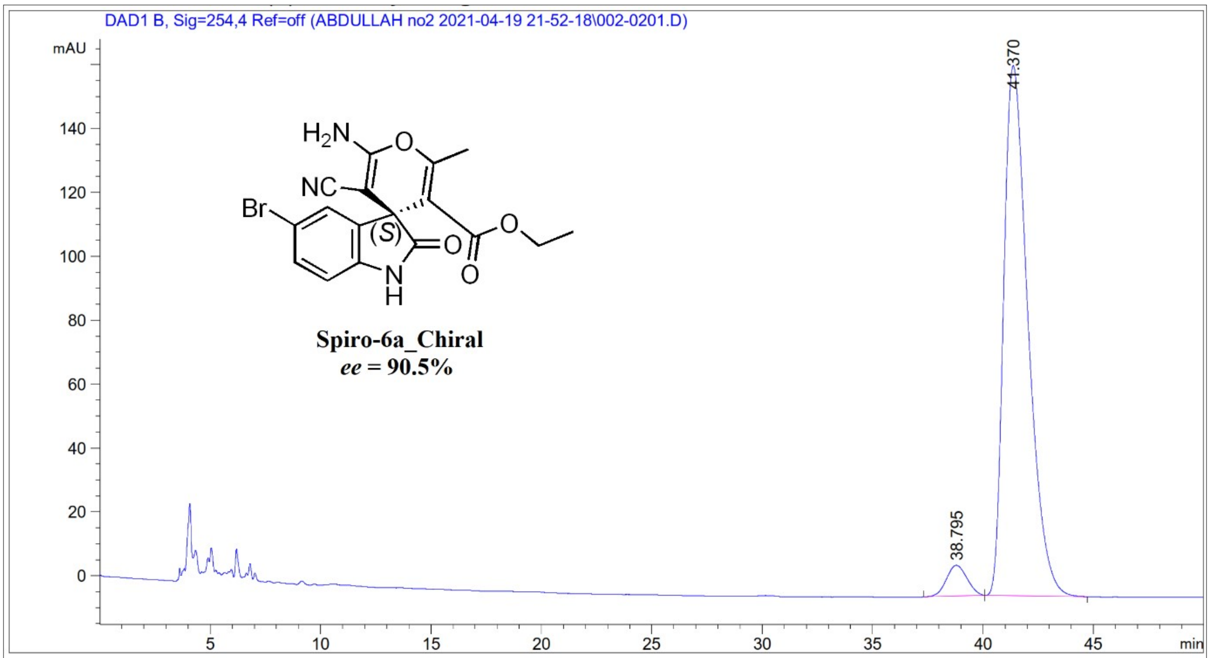
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	53.328	BB	1.2544	3.35290e4	417.75809	80.2248
2	63.645	BB	1.2836	5056.12646	54.63883	12.0978
3	74.566	BBA	1.2891	3208.71460	29.87341	7.6775

2.2 HPLC analysis of spiro-compound 6a-l

2.2.1 HPLC analysis of spiro-compound 6a

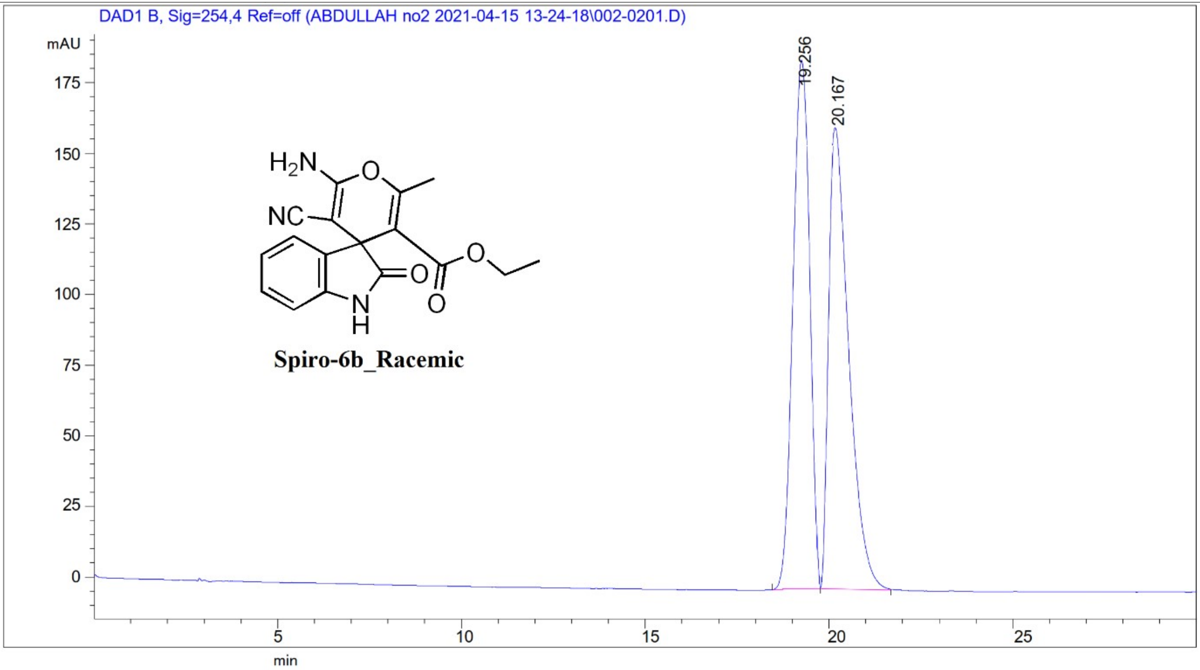


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.295	BB	1.0047	8663.94629	132.18552	50.0734
2	41.152	BB	1.1101	8638.55371	116.36103	49.9266

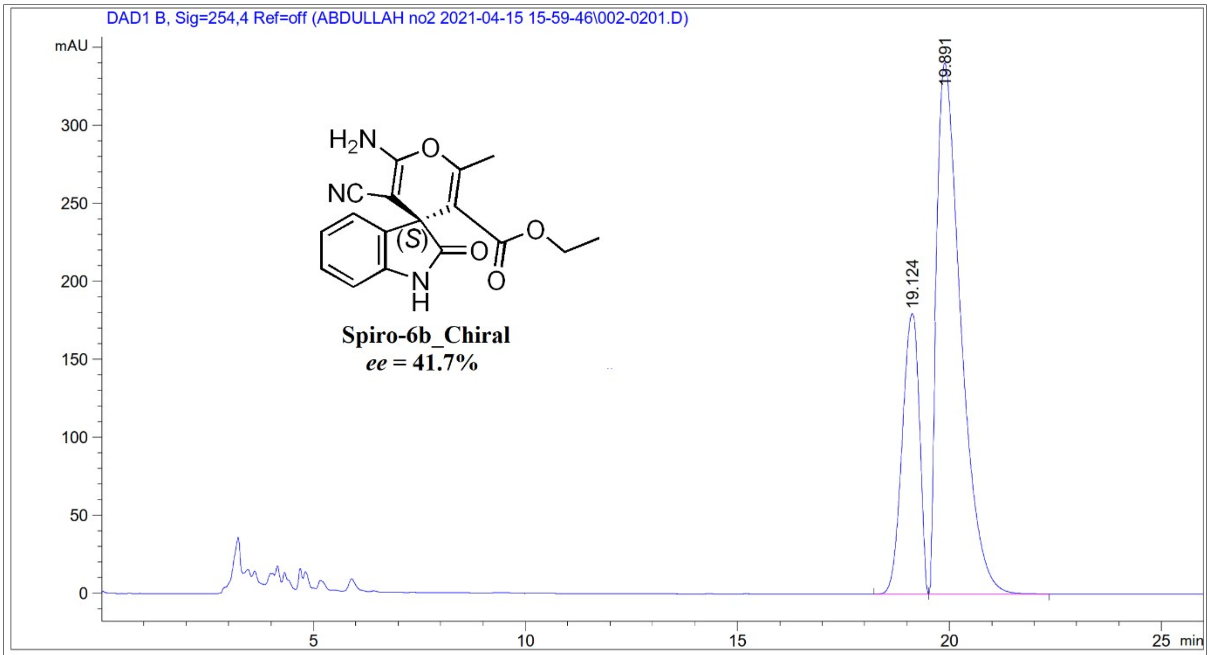


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.795	BB	0.7808	631.46246	9.68322	4.7458
2	41.370	BB	1.1688	1.26742e4	165.99530	95.2542

2.2.2 HPLC analysis of spiro-compound 6b

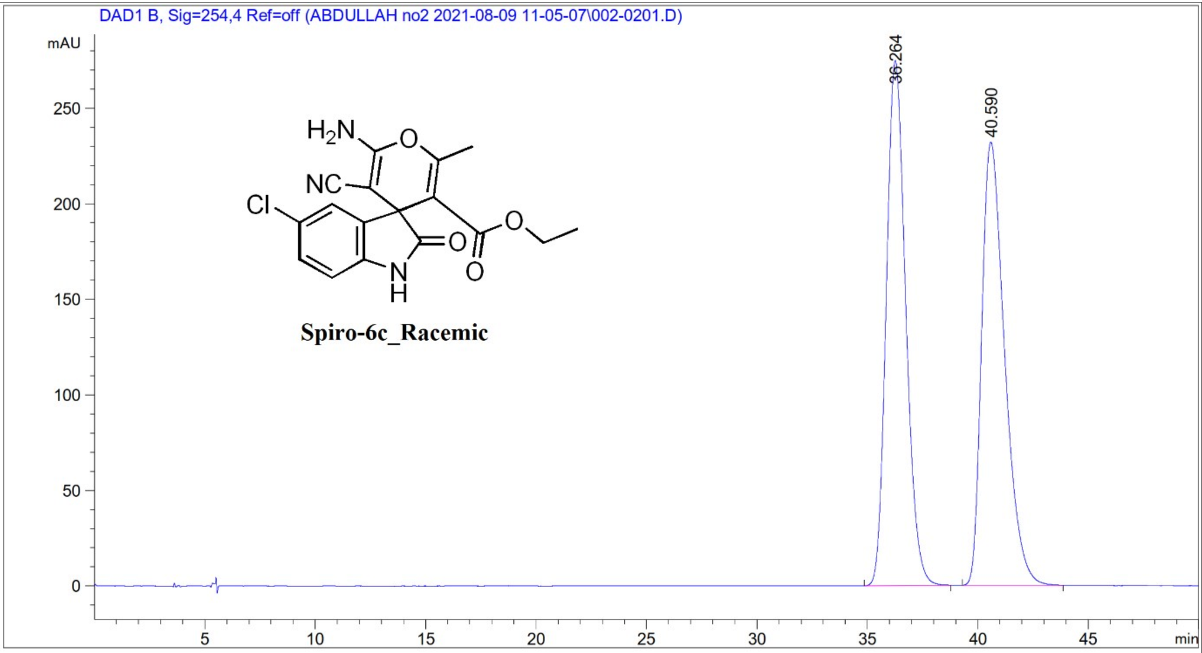


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.256	MF	0.5873	6629.49902	188.14458	49.8291
2	20.167	FM	0.6786	6674.96973	163.94574	50.1709

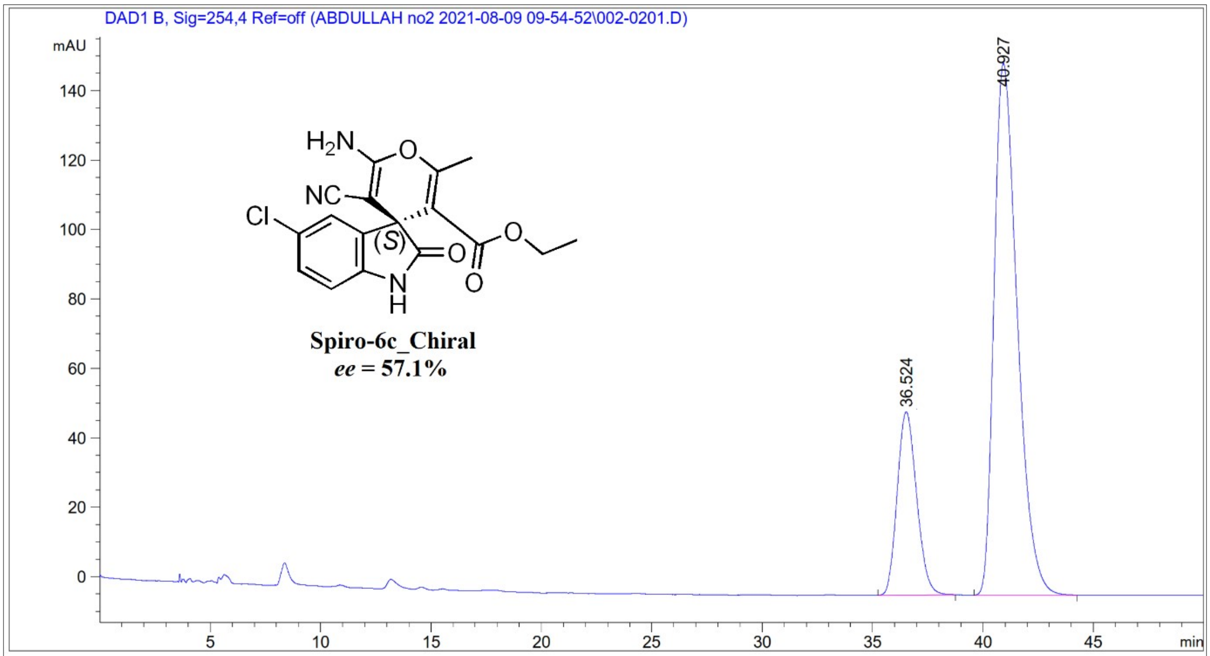


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.124	BV	0.5127	5841.40430	179.96642	29.1652
2	19.891	VB	0.6259	1.41872e4	340.67624	70.8348

2.2.3 HPLC analysis of spiro-compound 6c

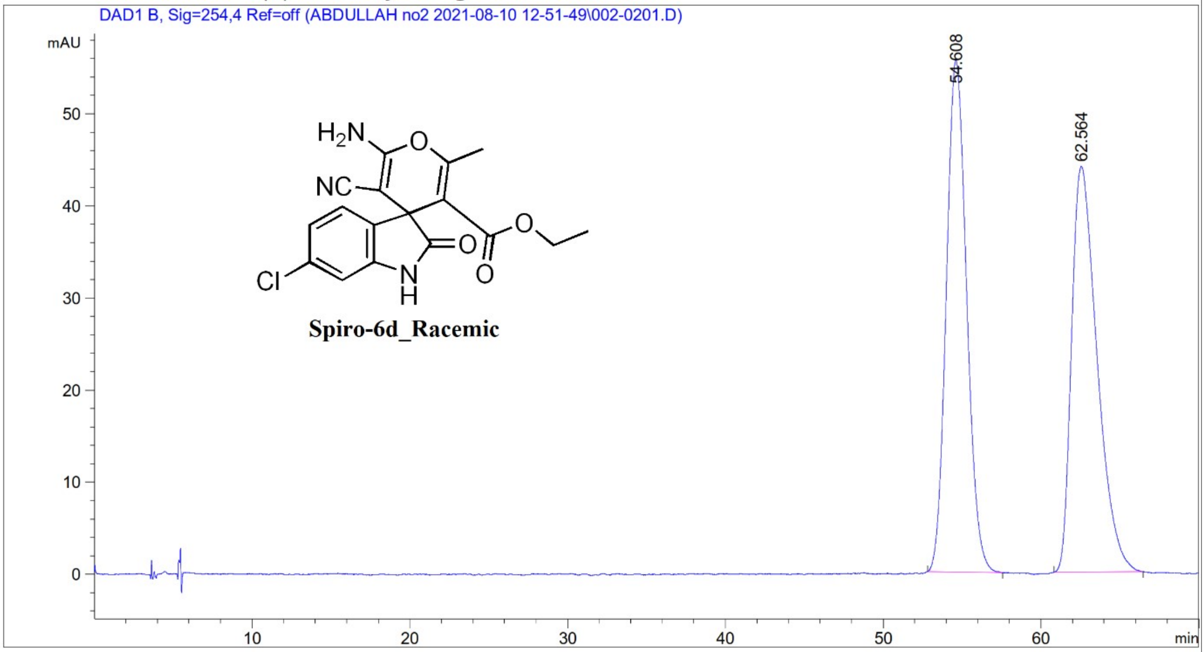


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	36.264	BB	0.9399	1.67605e4	274.68066	50.0264
2	40.590	BB	1.1062	1.67428e4	231.94525	49.9736

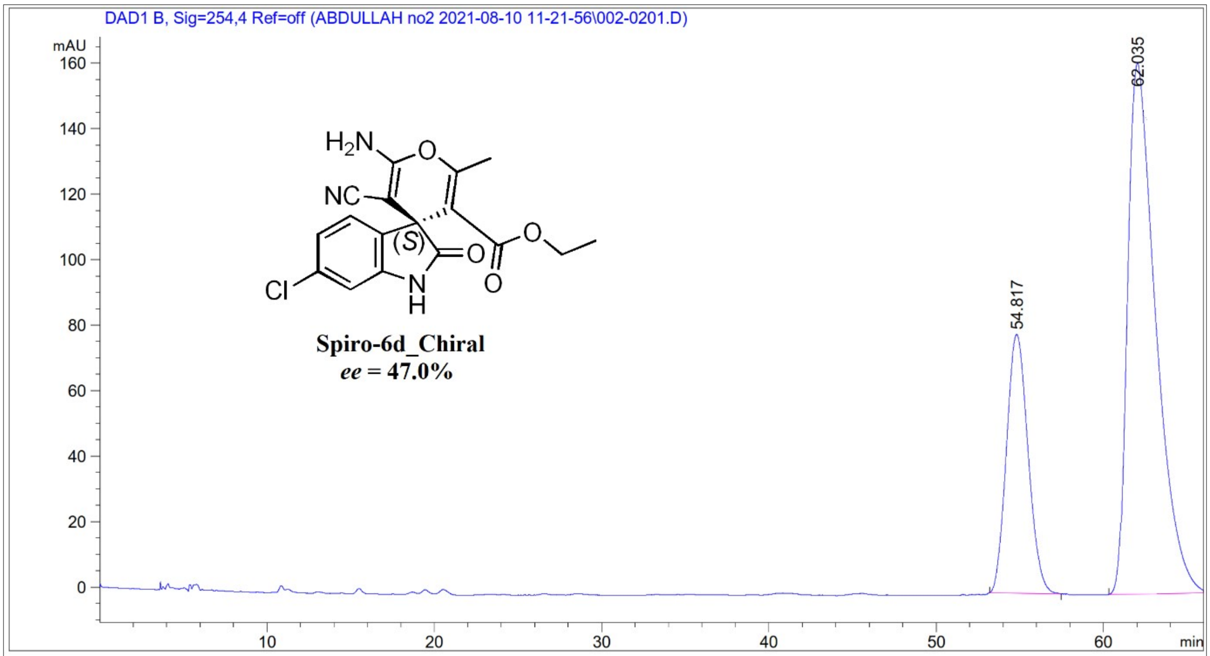


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	36.524	MM	0.9998	3109.81934	51.84235	21.4300
2	40.927	MM	1.2333	1.14017e4	154.07979	78.5700

2.2.4 HPLC analysis of spiro-compound 6d

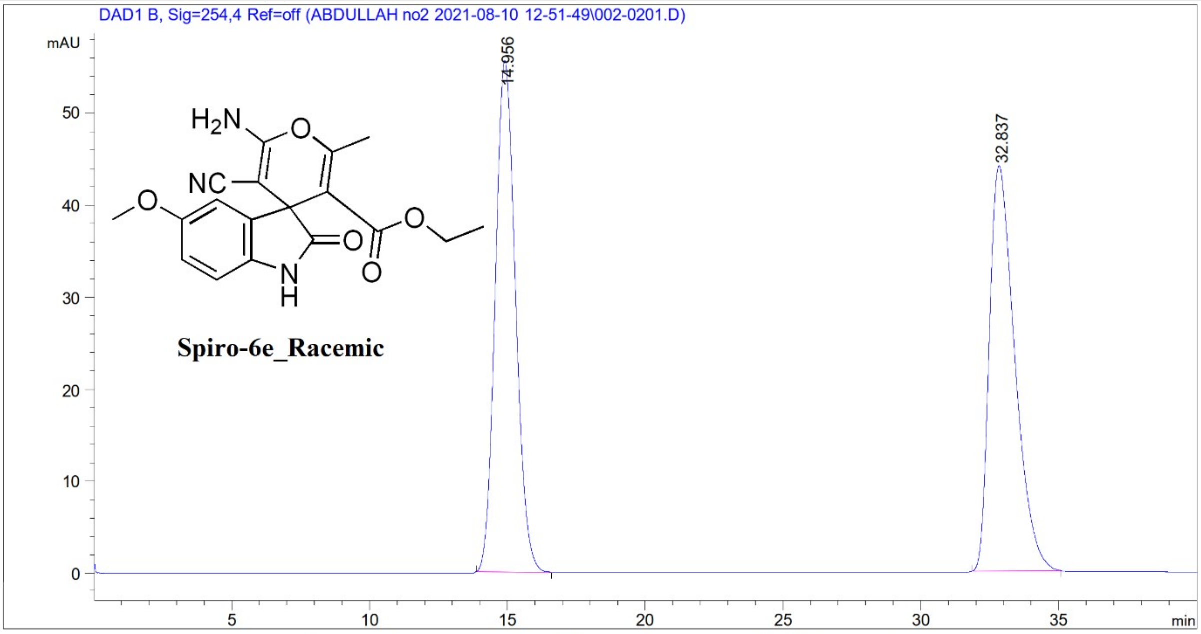


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	54.608	BB	1.2990	4970.16357	55.59554	50.0760
2	62.564	BB	1.4762	4955.07910	44.05468	49.9240

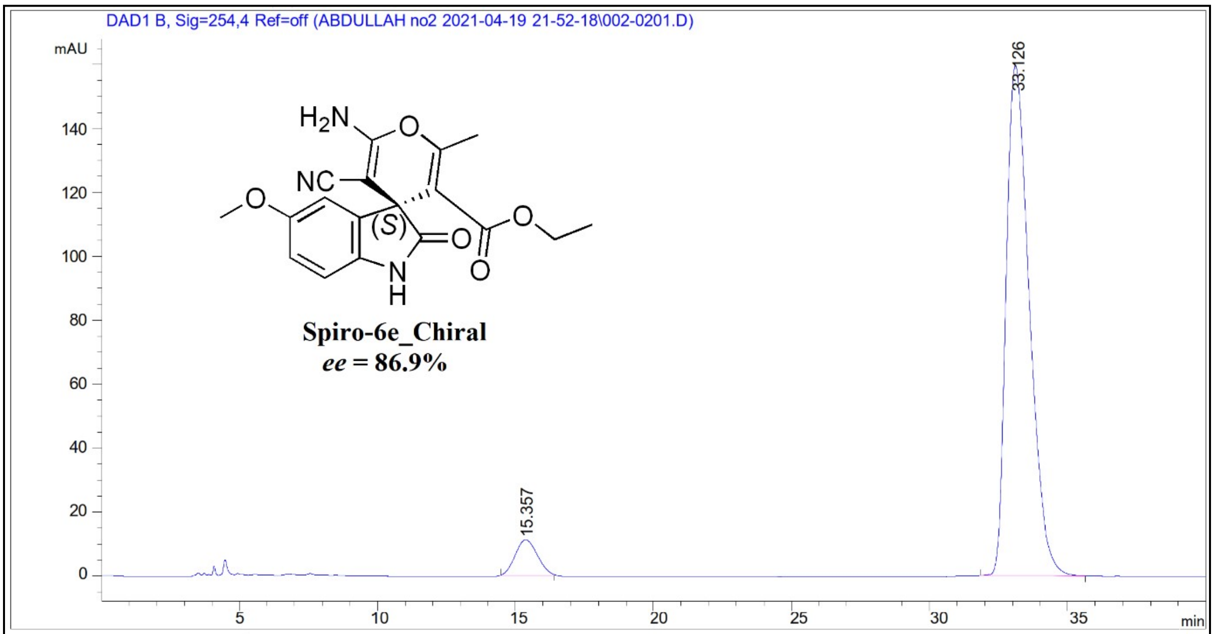


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	54.817	MM	1.4772	6956.40430	78.48705	26.5217
2	62.035	MM	1.9666	1.92727e4	163.33656	73.4783

2.2.5 HPLC analysis of spiro-compound 6e

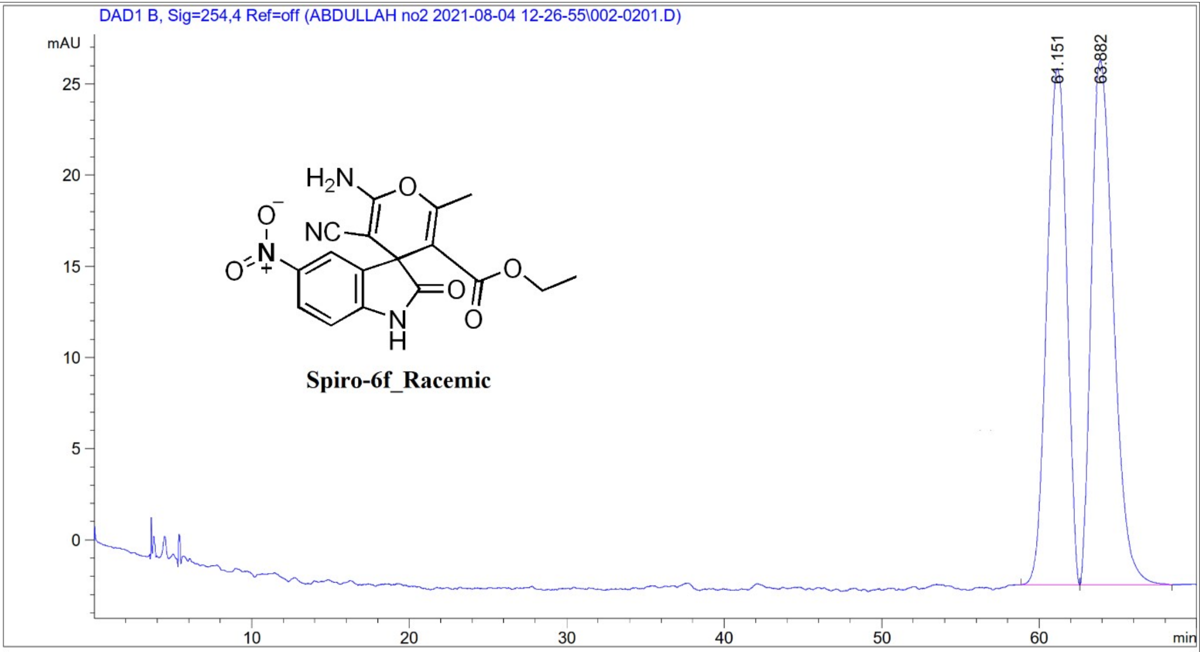


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.956	BB	0.7423	2840.09347	55.59498	50.0721
2	32.837	BB	0.8435	2831.47377	44.04793	49.9279

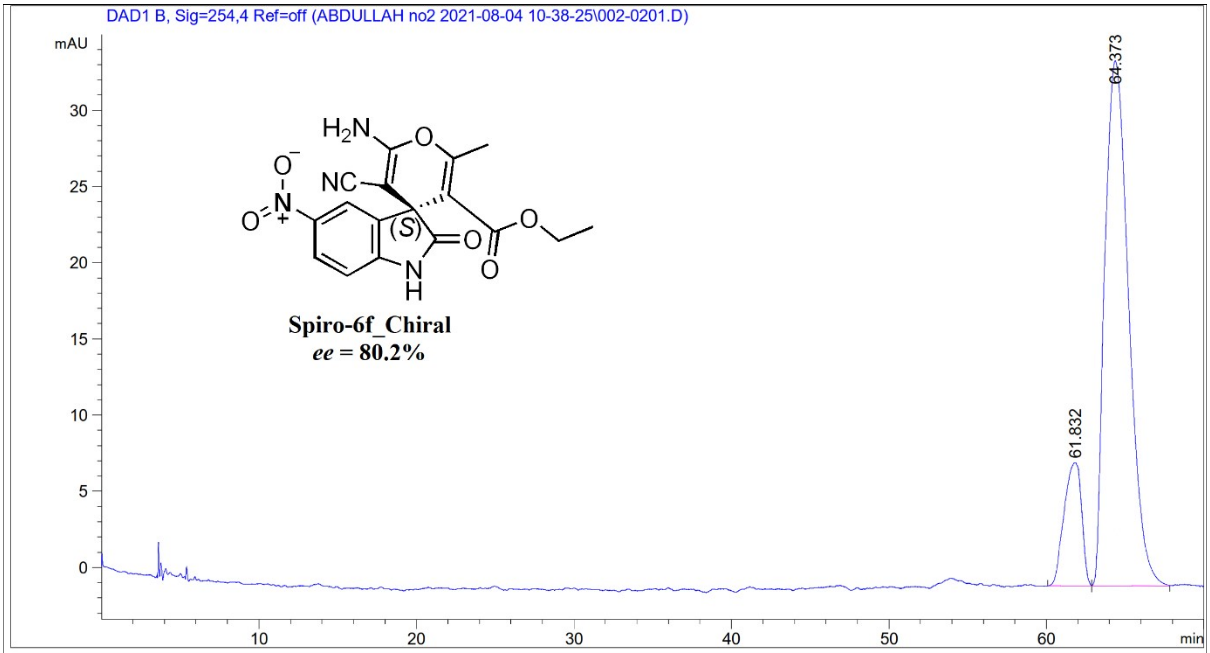


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.357	BB	0.8571	833.26538	11.83296	6.5391
2	33.126	BB	1.1428	1.19095e4	160.08523	93.4608

2.2.6 HPLC analysis of spiro-compound 6f

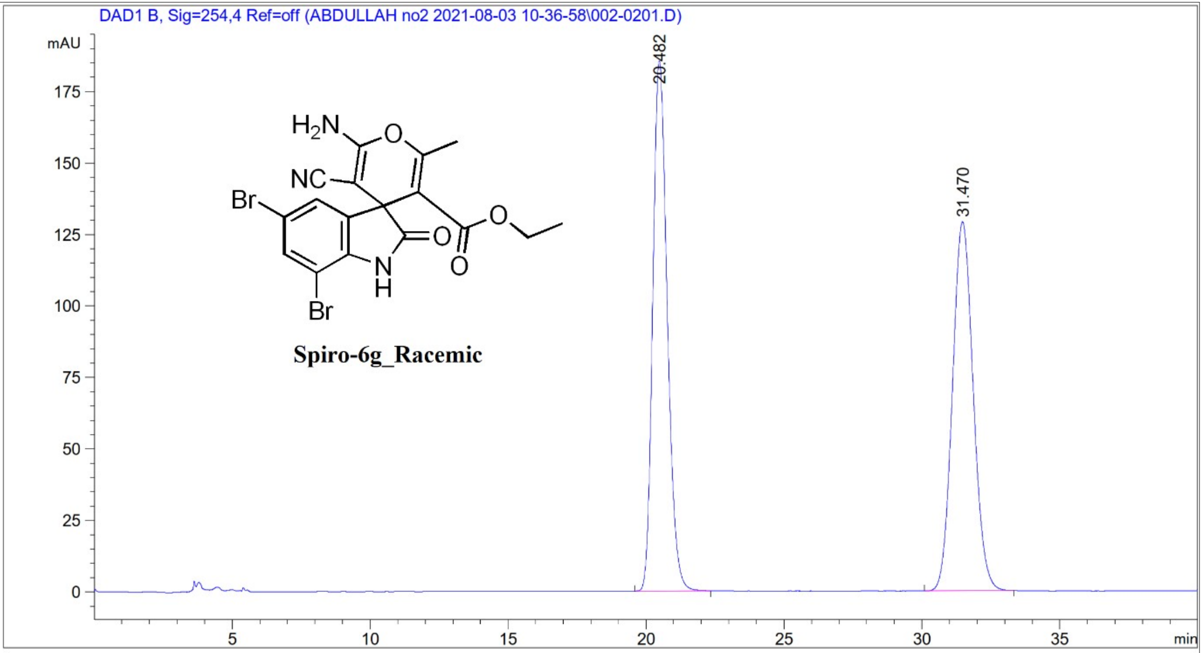


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	61.151	BB	1.0293	1943.06384	22.77639	49.1901
2	63.882	BB	1.0848	2007.04529	21.99085	50.8099

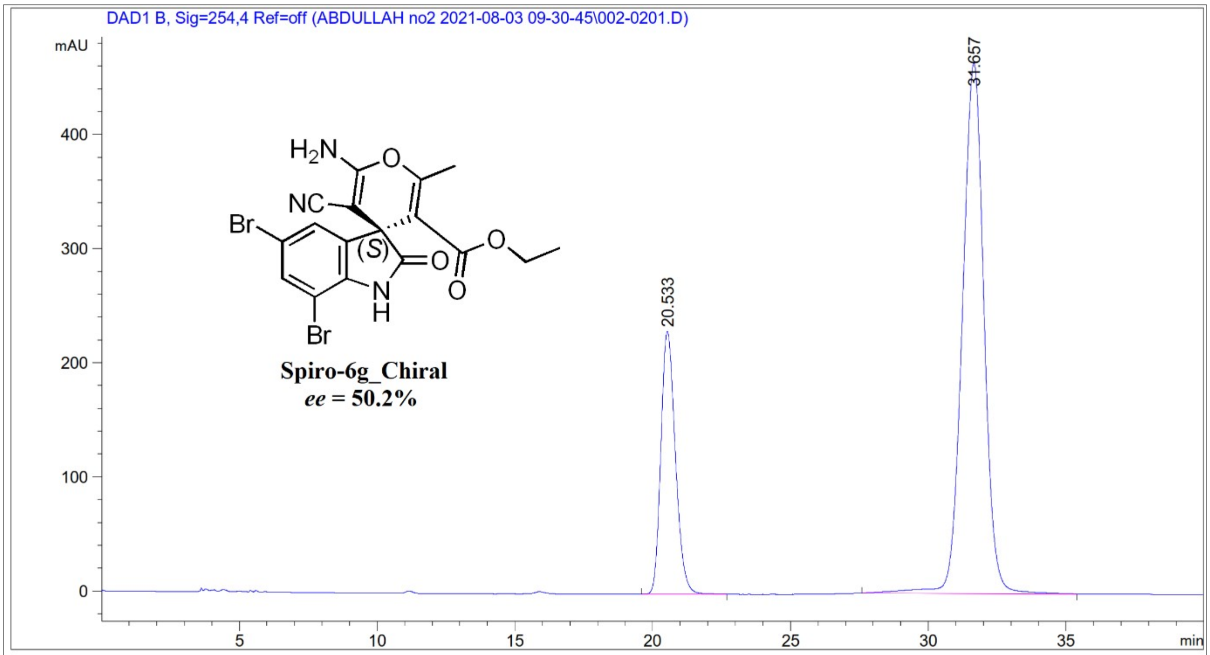


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	61.832	BB	0.8856	345.04288	4.57650	9.9129
2	64.373	BB	1.2031	3135.68823	30.97287	90.0871

2.2.7 HPLC analysis of spiro-compound 6g

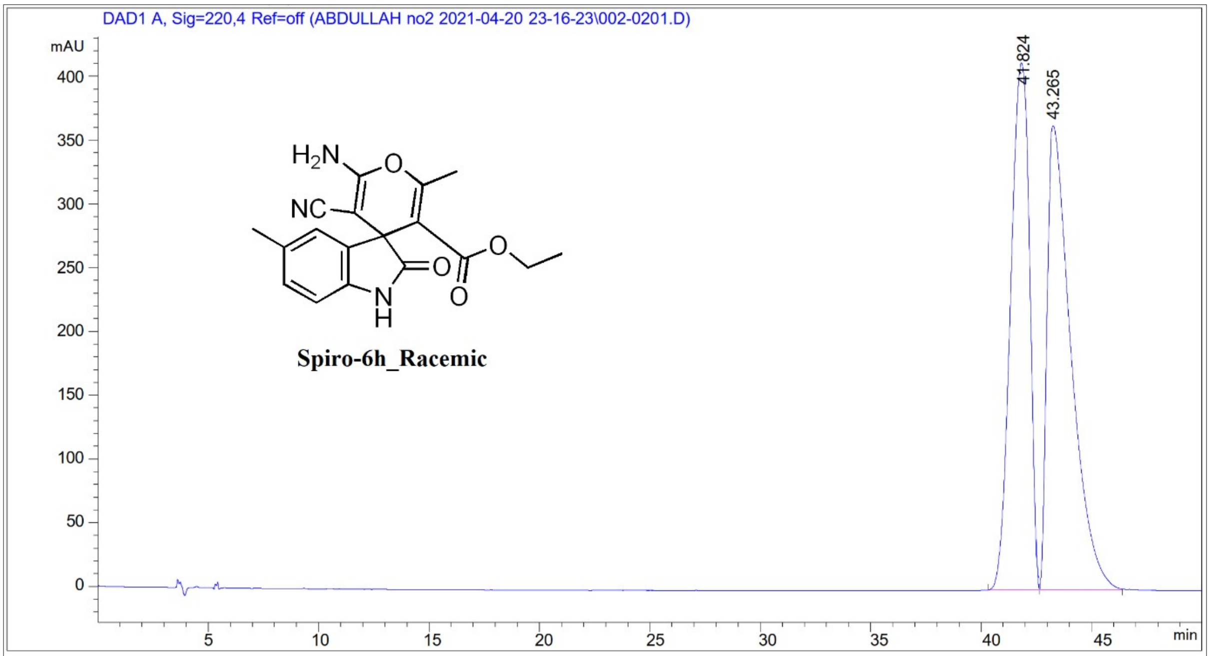


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.482	BB	0.5625	6749.83545	185.50739	50.0258
2	31.470	BB	0.8095	6742.87451	129.07594	49.9742

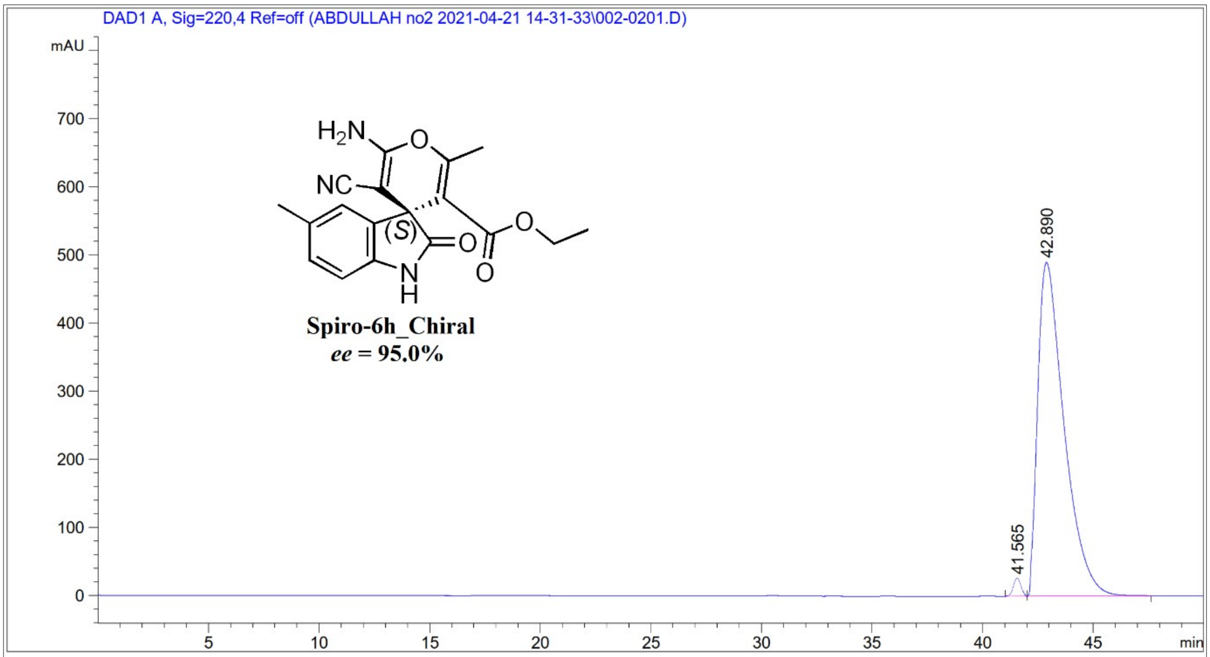


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.533	BB	0.5689	8431.41016	230.43633	24.9076
2	31.657	BB	0.8419	2.54194e4	464.90509	75.0924

2.2.8 HPLC analysis of spiro-compound 6h

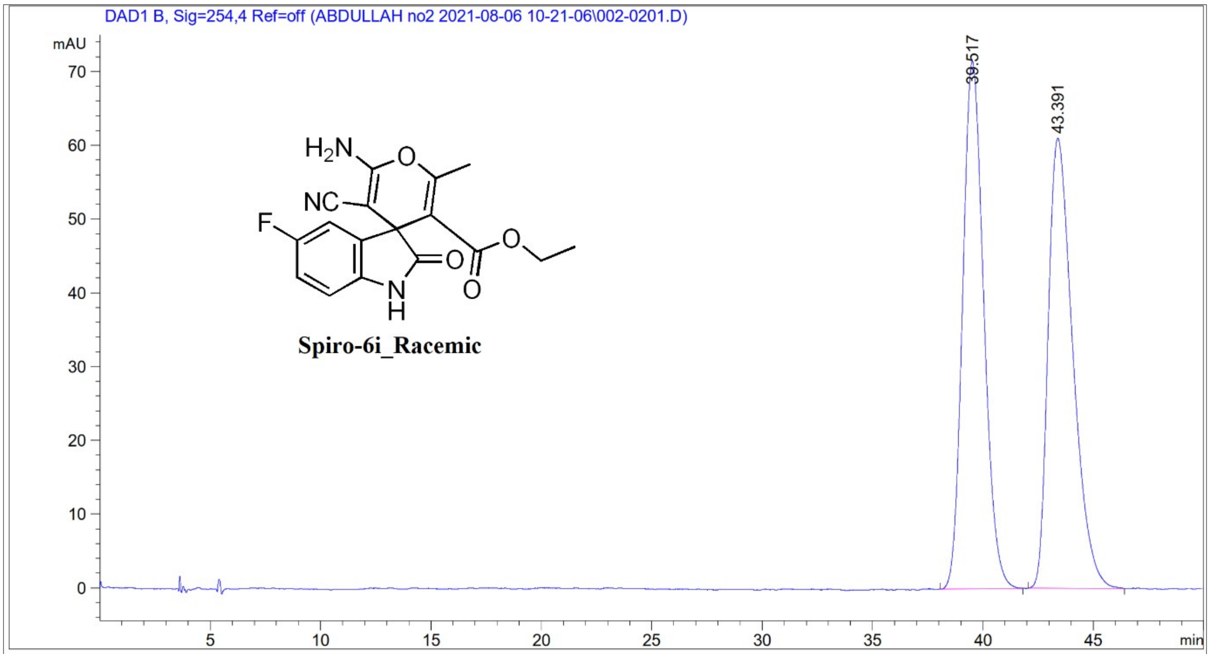


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	41.824	MF	1.1816	2.93858e4	414.49051	49.8536
2	43.265	FM	1.3525	2.95584e4	364.23413	50.1464

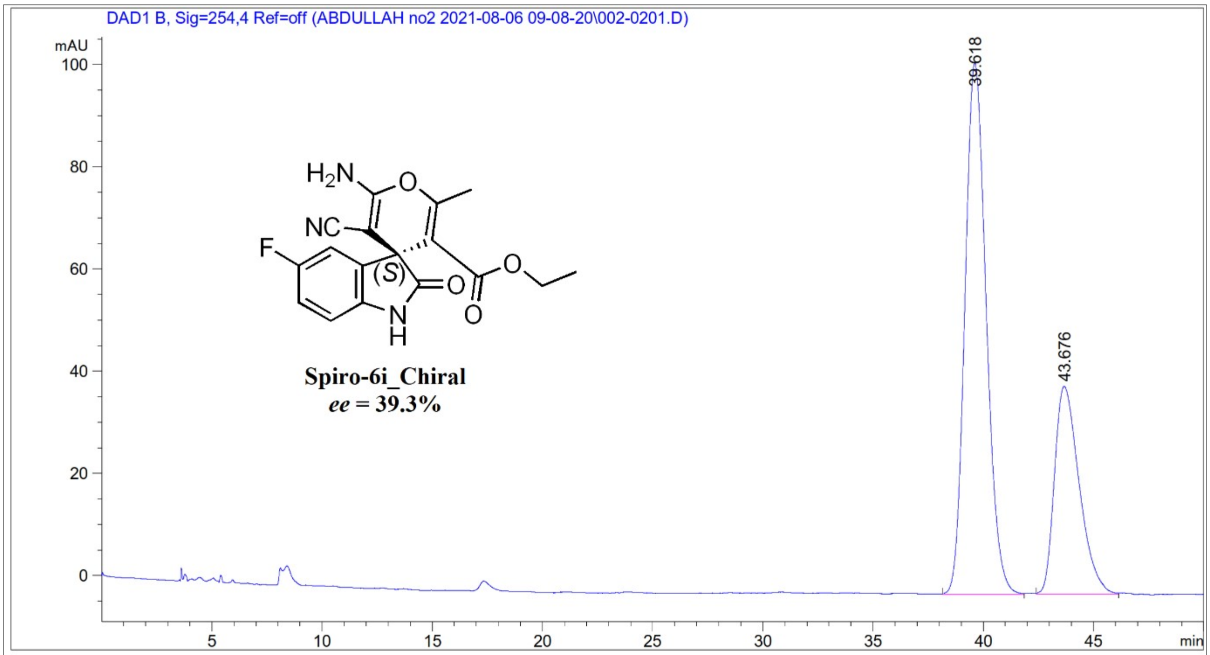


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	41.565	BV E	0.6721	1043.73694	18.73127	2.4859
2	42.890	VB R	1.2313	4.09423e4	489.77911	97.5141

2.2.9 HPLC analysis of spiro-compound 6i

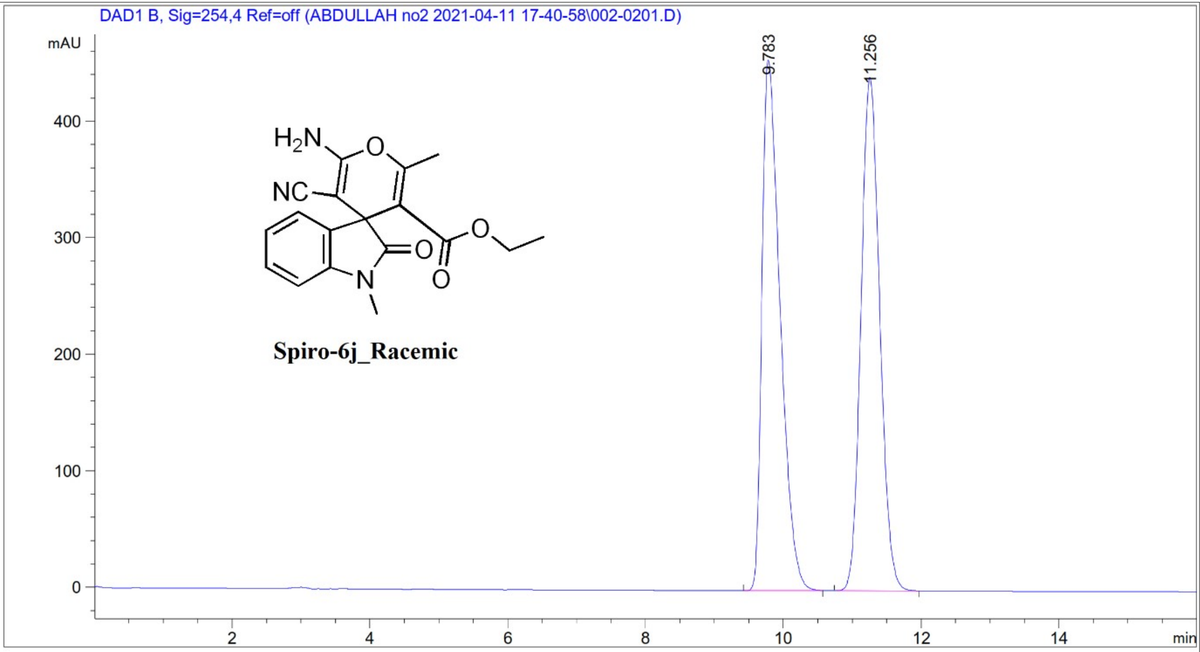


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	39.517	BB	0.9911	4753.42139	71.54505	50.0533
2	43.391	BB	1.1470	4743.30420	61.01168	49.9467

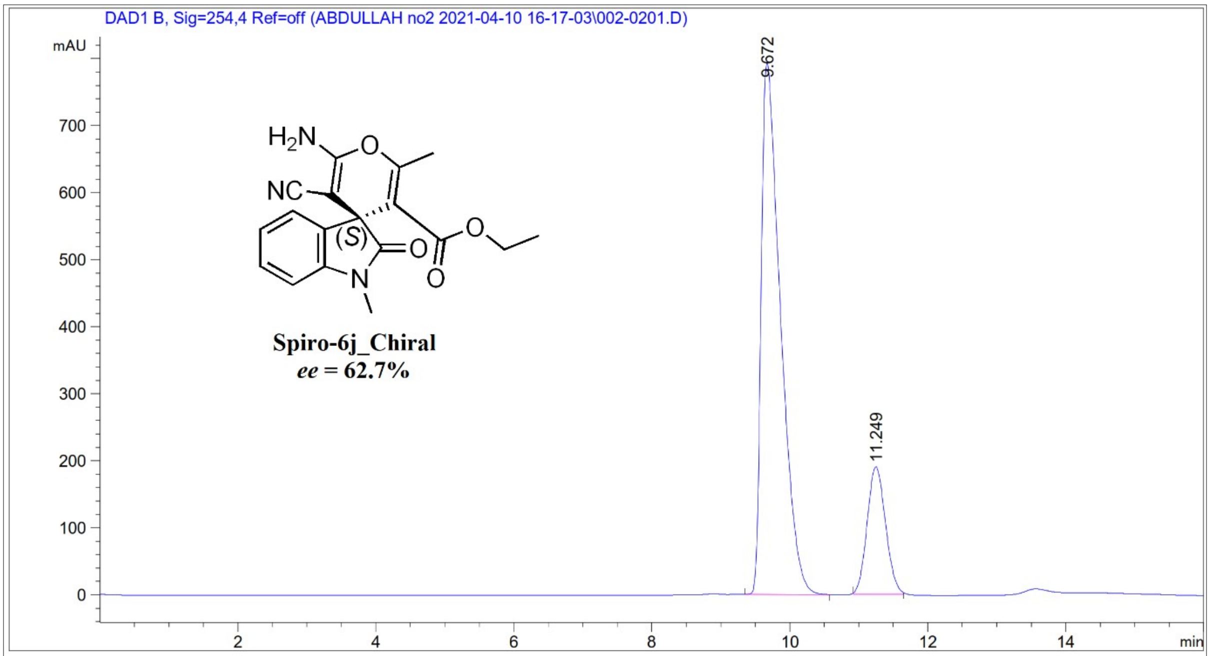


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	39.618	MM	1.1331	7088.00928	104.25698	69.6319
2	43.676	MM	1.2744	3091.25122	40.42716	30.3681

2.2.10 HPLC analysis of spiro-compound 6j

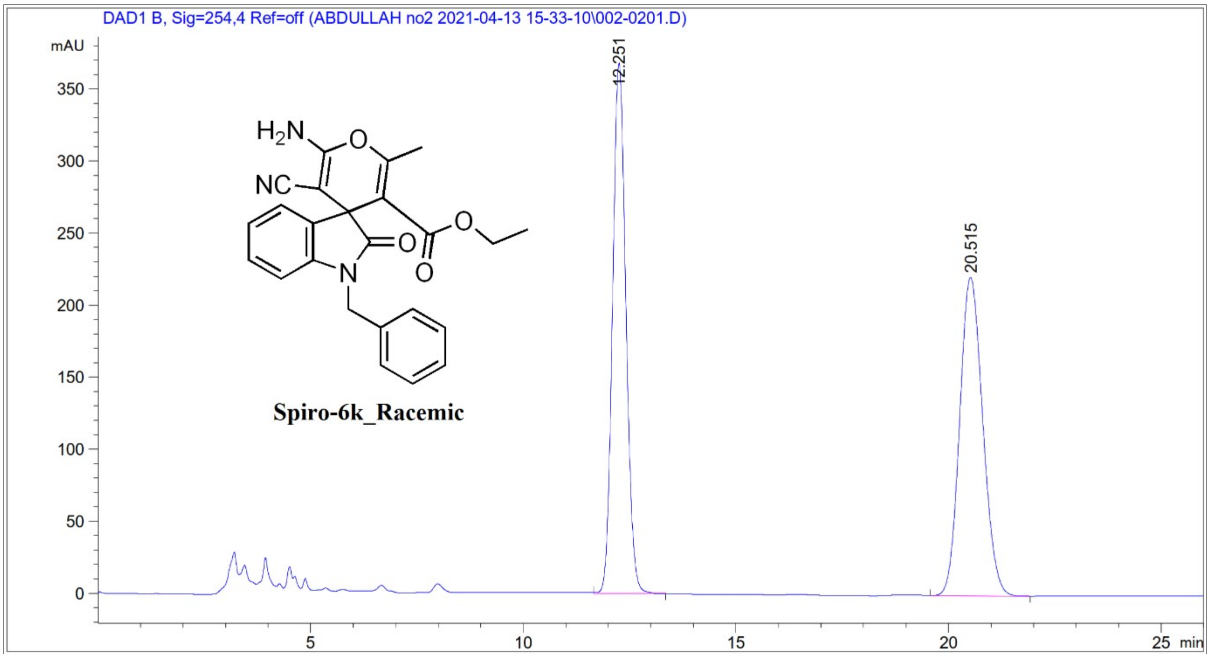


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.783	BB	0.2797	8342.37695	455.36761	50.0215
2	11.256	BB	0.2927	8335.21191	440.70712	49.9785

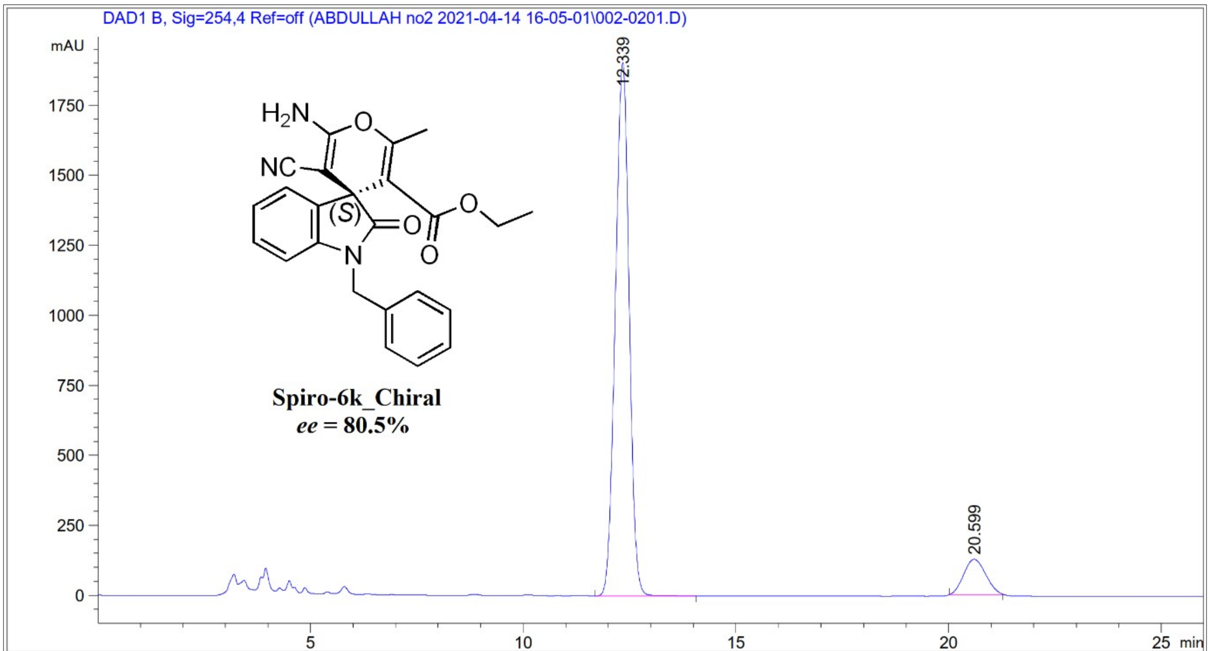


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.672	BB	0.2910	1.54218e4	792.74988	81.3544
2	11.249	MM	0.3096	3534.52539	190.26959	18.6456

2.2.11 HPLC analysis of spiro-compound 6k

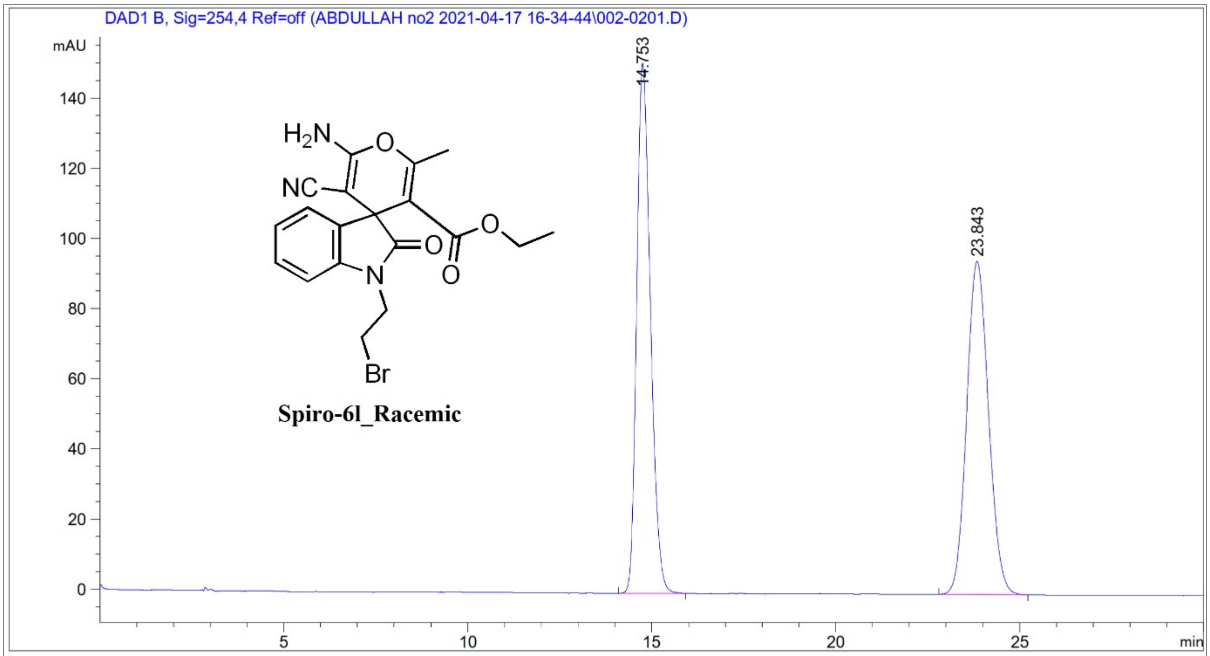


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.251	MM	0.3686	8146.11670	368.37732	49.4320
2	20.515	BB	0.5897	8333.32617	221.12598	50.5680

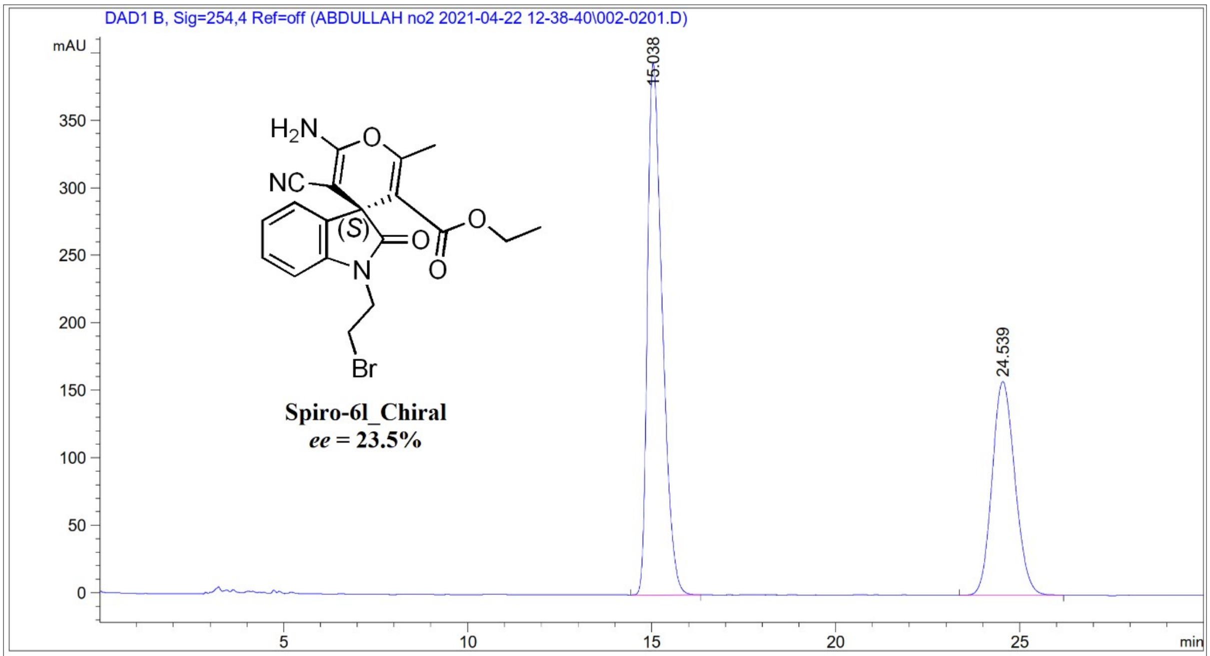


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.339	BB	0.3539	4.30293e4	1902.45837	90.2689
2	20.599	MM	0.6042	4638.60010	127.95790	9.7311

2.2.12 HPLC analysis of spiro-compound 6l



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.753	BB	0.4020	3912.61572	151.15005	49.9770
2	23.843	BB	0.6306	3916.21875	95.05820	50.0230



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.038	BB	0.4294	1.10548e4	393.95184	61.7228
2	24.539	BB	0.6729	6855.57178	158.40242	38.2772

3. X-Ray structure determinations

The crystal of Aldol product **3a** was immersed in cryo-oil, mounted in a loop, and measured at a temperature of 120 K. The X-ray diffraction data was collected on a Rigaku Oxford Diffraction Supernova diffractometer using Cu Kα radiation. The *CrysAlisPro*[1] software package was used for cell refinement and data reduction. A Multi-scan, absorption correction (*CrysAlisPro*[1]) was applied to the intensities before structure solution. Structure was solved by intrinsic phasing (*SHELXT*[2]) method. Structural refinement was carried out using *SHELXL*[3] software with *SHELXLE*[4] graphical user interface. The structure contained 0.167 molecules of H_2O per each organic molecule. The oxygen atom of H_2O was located on a two-fold rotation axis. The H_2O and NH hydrogen atoms were located from the difference Fourier map and refined isotropically. Other hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms, with C-H = 0.95 – 0.99 Å and U_{iso} = 1.2-1.5·U_{eq}(parent atom). The crystallographic details are summarized in Table **7**.

Table 7. Crystal Data.

	3f
CCDC	2143162
empirical formula	$C_{11}H_{10.33}N_2O_{5.17}$
fw	253.21
temp (K)	120(2)
λ(Å)	1.54184
cryst syst	Monoclinic
space group	*C2/c*
a (Å)	20.0082(4)
b (Å)	9.9146(2)
c (Å)	11.4233(2)
β (deg)	98.317(2)
V (Å^3)	2242.24(8)
Z	8
ρ_{calc} (Mg/m^3)	1.500
μ(Mo Kα) (mm^{-1})	1.039
No. reflns.	15099
Unique reflns.	2362
Completeness to θ=67.684°	100%
GOOF (F^2)	1.047
R_{int}	0.0278
R_1^{a} ($I \geq 2\sigma$)	0.0398

wR_2[b] ($I \geq 2\sigma$)	0.1038

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b] $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/ \Sigma[w(F_o^2)^2]\}^{1/2}$

References

1. *Journal*, Rikagu Oxford Diffraction, CrysAlisPro, Agilent Technologies inc., 2018, Yarnton, Oxfordshire, England

2. G. M. Sheldrick, *Acta Crystallogr. A*, 2015, **71**, 3-8.
3. G. M. Sheldrick, *Acta Crystallogr. C Struct. Chem.*, 2015, **71**, 3-8.
4. C. B. Hübschle, G. M. Sheldrick and B. Dittrich, *J. Appl. Crystallogr.*, 2011, **44**, 1281-1284.