

Electronic Supplementary Material (ESI) for RSC Advances.
This journal is © The Royal Society of Chemistry 2022

Supporting Information

Enantioselective Synthesis of α -Tetrasubstituted (3-Indoliziny1) (diaryl)- methanamines via Chiral Phosphoric Acid Catalysis

Jialing Zhong^a, Rihuang Pan^a, and Xufeng Lin^{*a}

^a*Department of Chemistry, Zhejiang University, Hangzhou 310027, China*

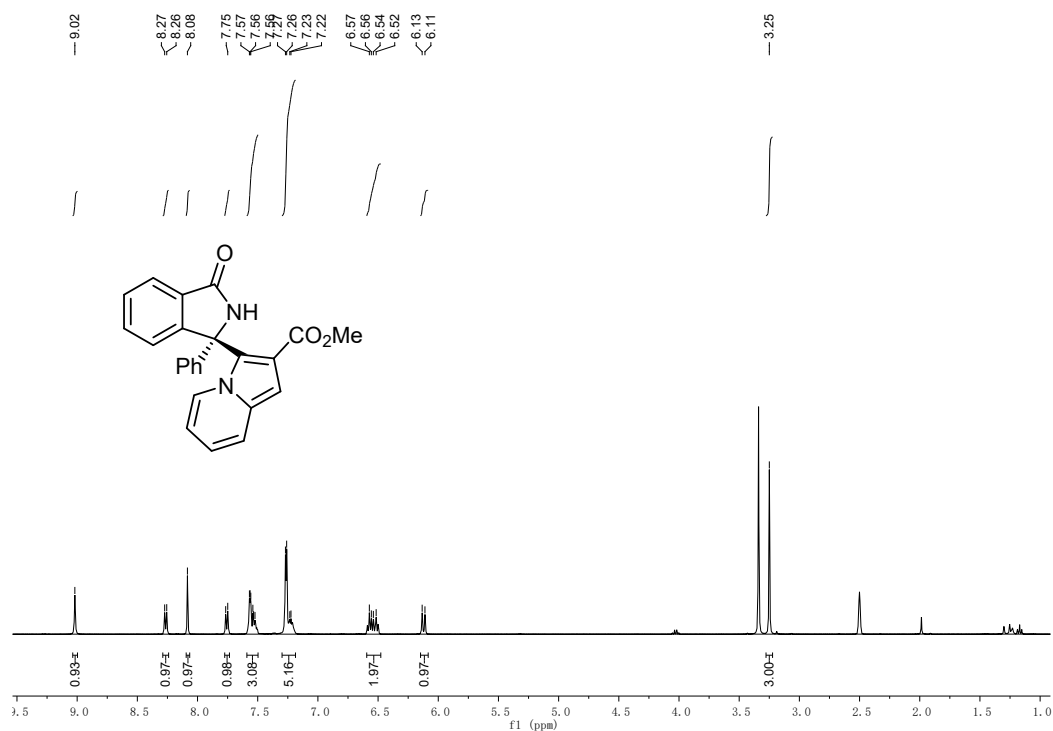
E-mail: lxfoke@zju.edu.cn

Table of Contents

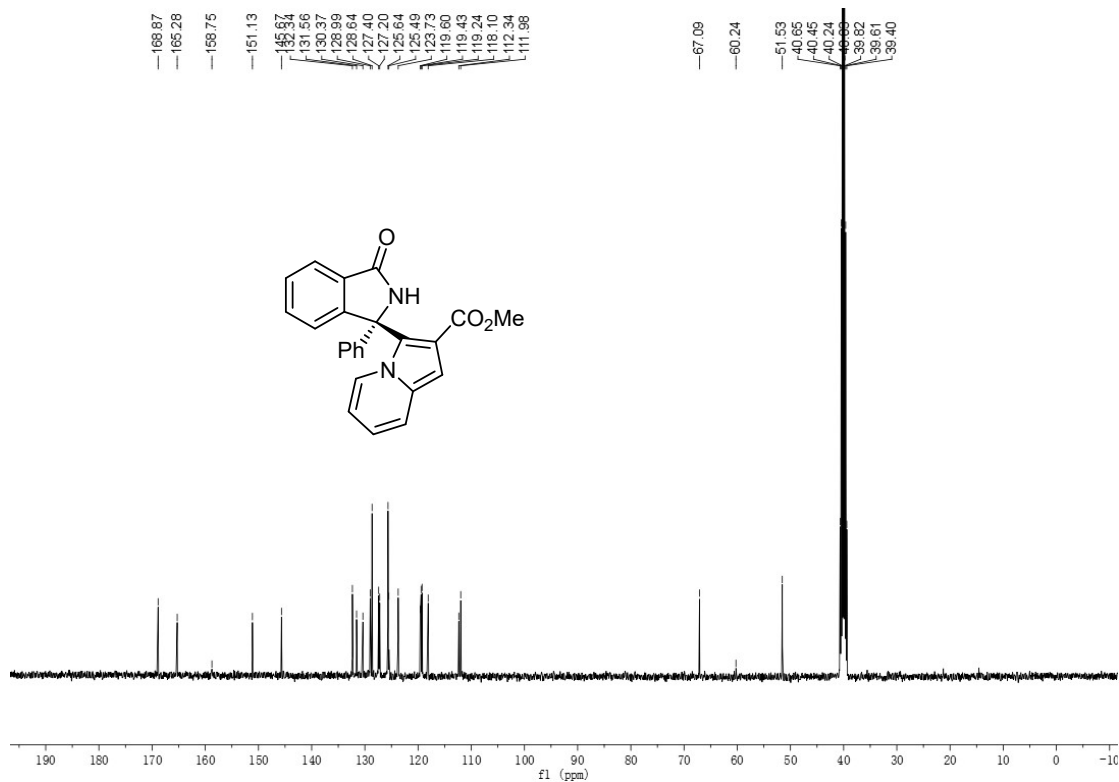
1. Copies of ¹ H NMR, ¹³ C NMR and ¹⁹ F NMR Spectra.....	S2
2. X-ray Single Crystal Data for Product (<i>R</i>)- 3p	S21
3. Copies of HPLC Spectra.....	S23

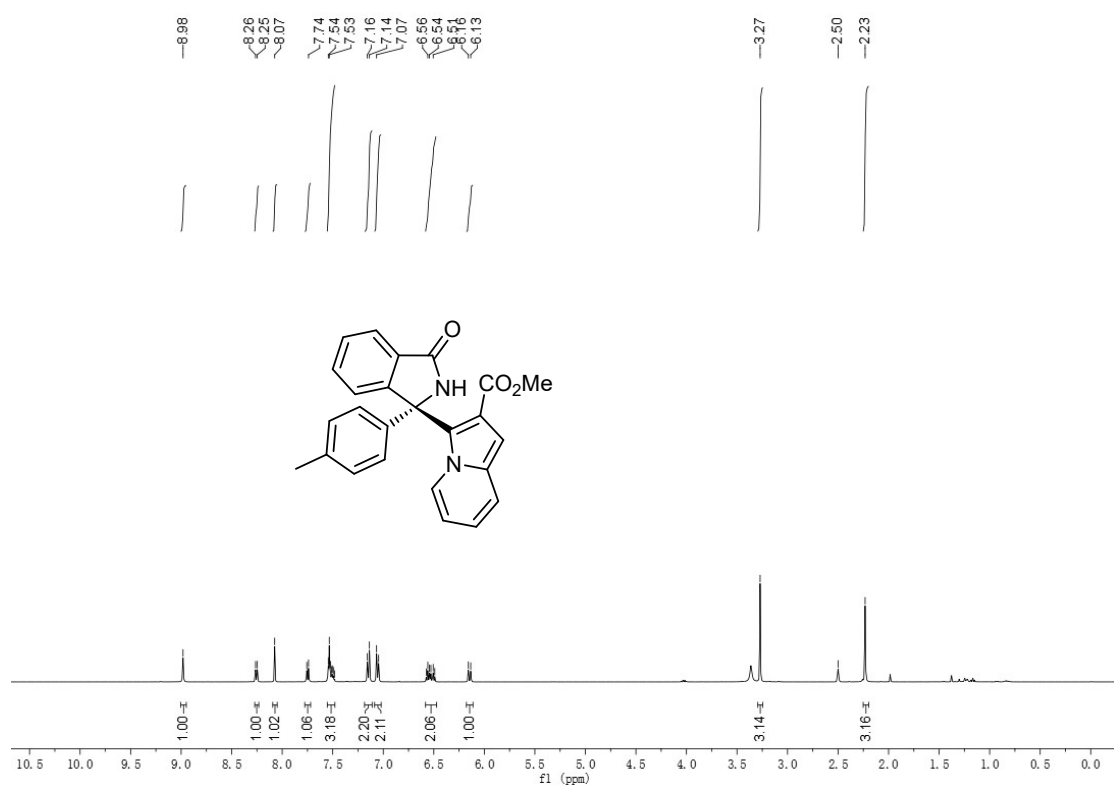
1. Copies of ^1H NMR, ^{13}C NMR and ^{19}F NMR Spectra

3a ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



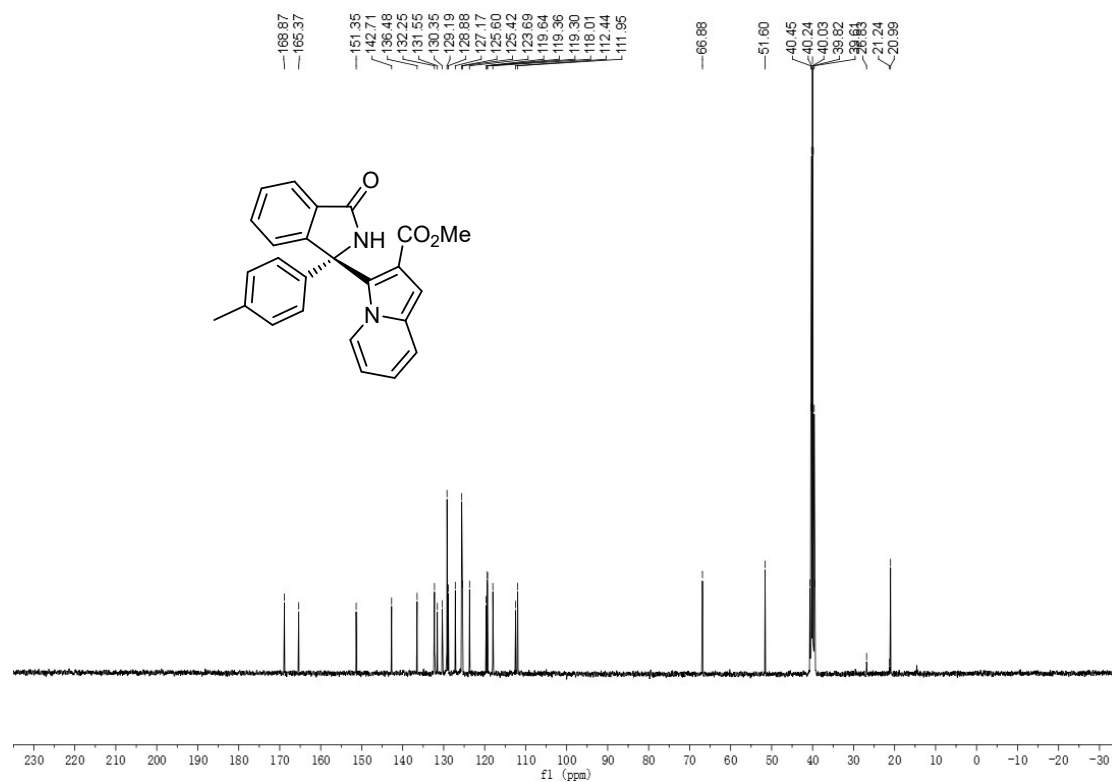
3a ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$)



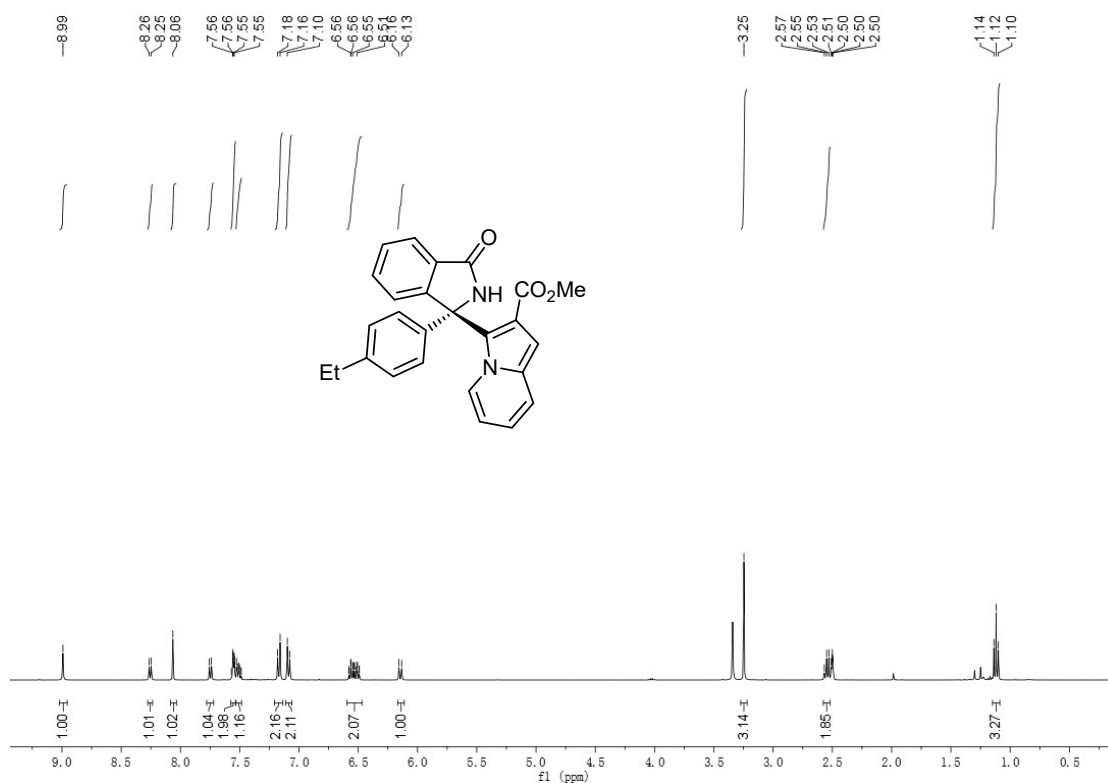


3b ¹H NMR (400 MHz, DMSO-*d*₆)

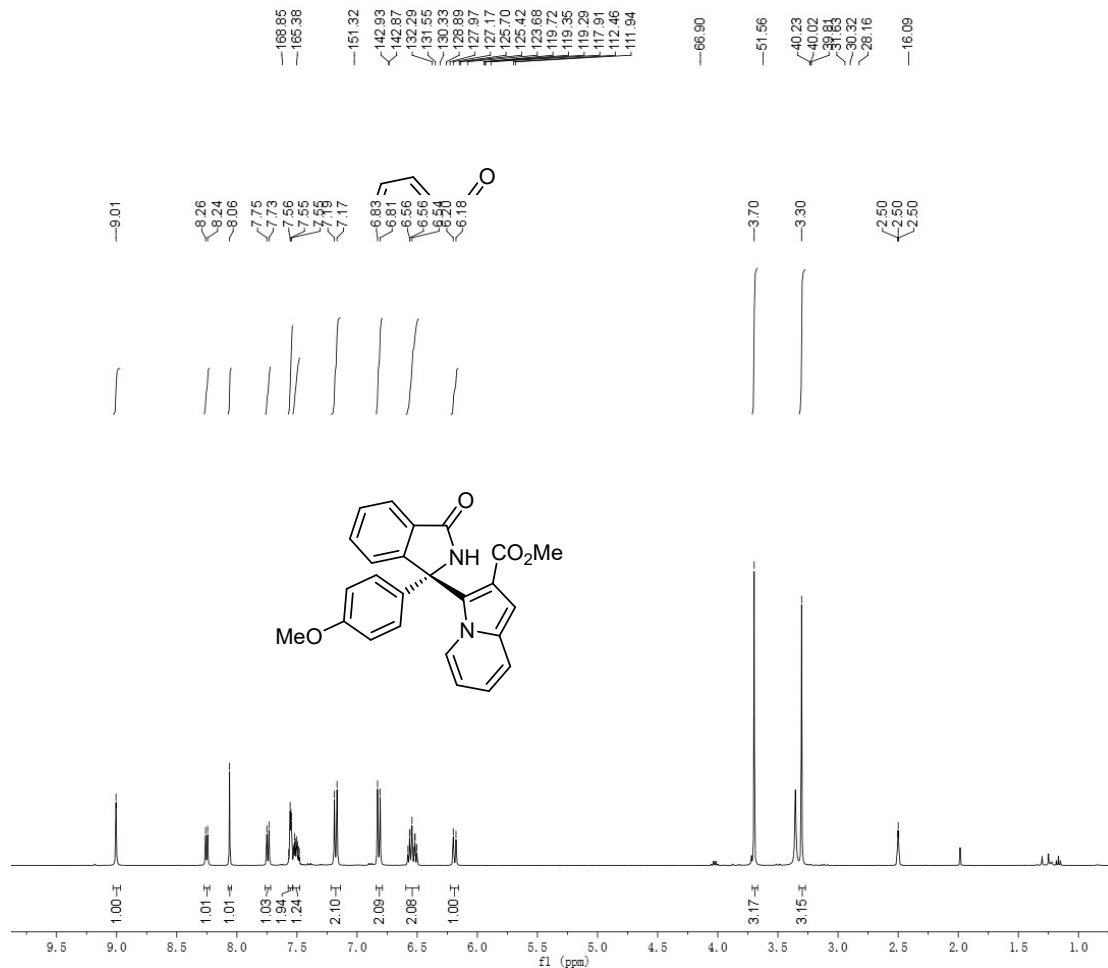
3b ¹³C NMR (101 MHz, DMSO-*d*₆)

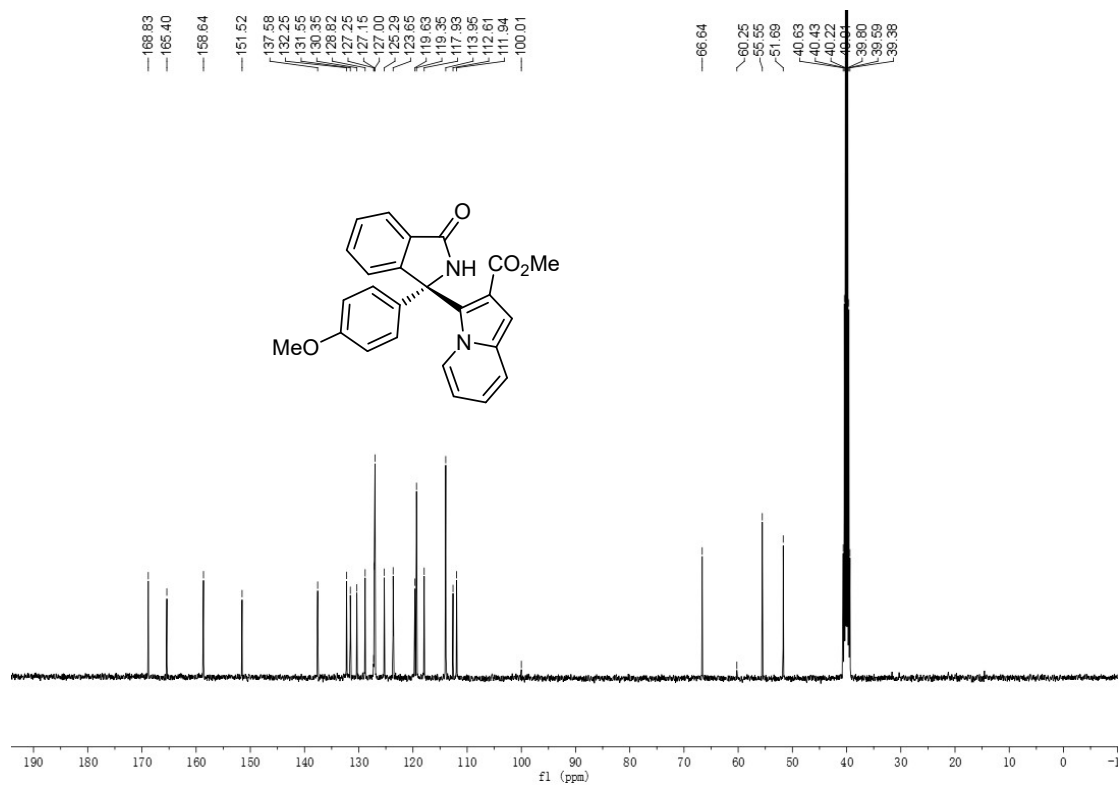


3c ^1H NMR (400 MHz, $\text{DMSO}-d_6$)

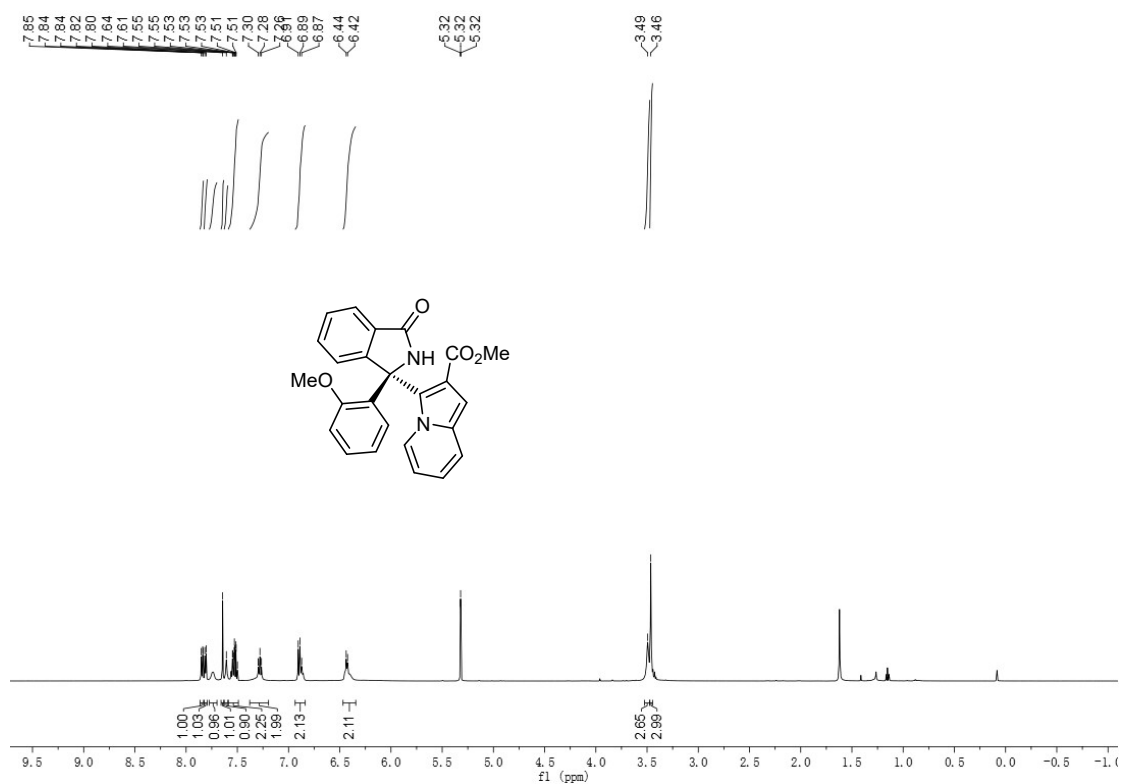


3c ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$)

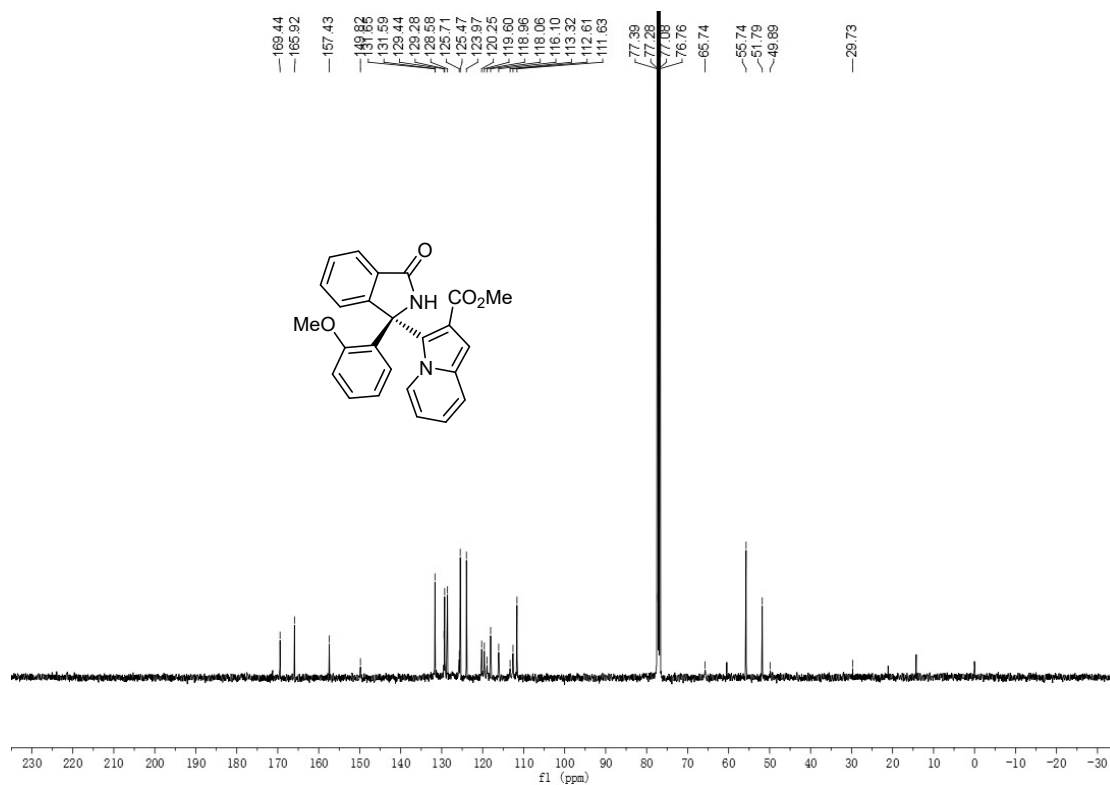




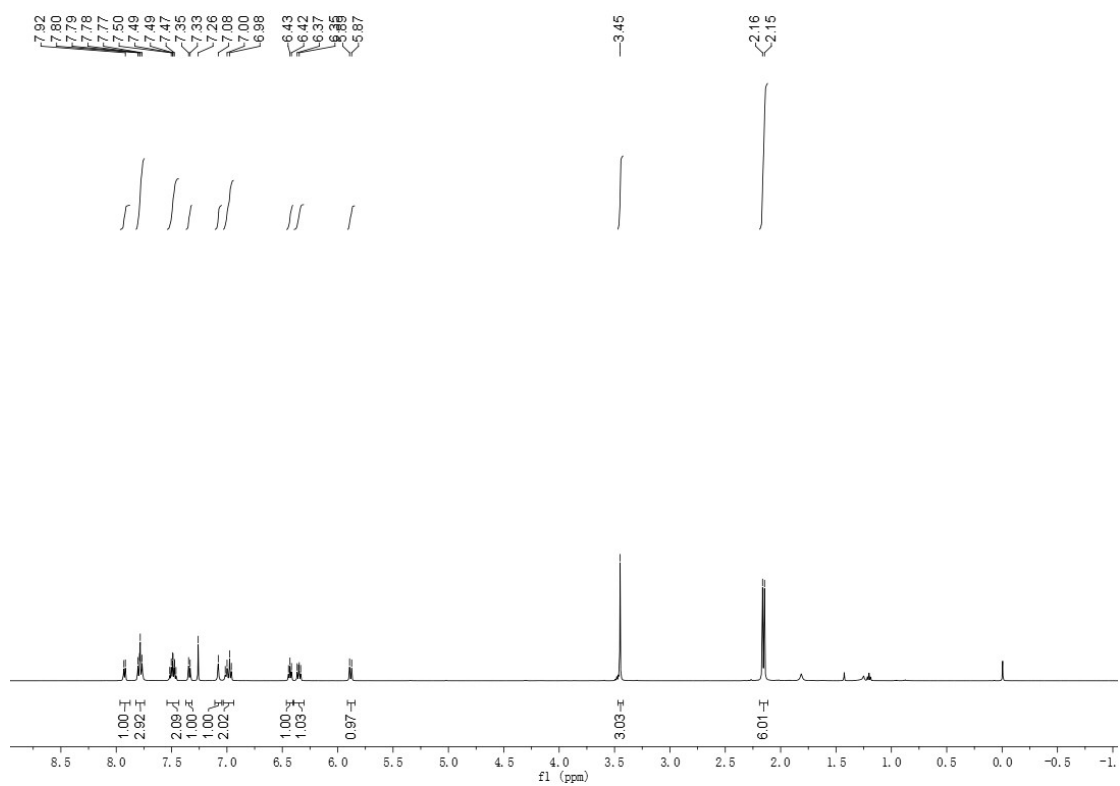
3d ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$)



3e ¹H NMR (500 MHz, CD₂Cl₂)

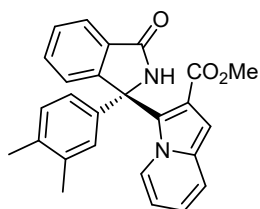
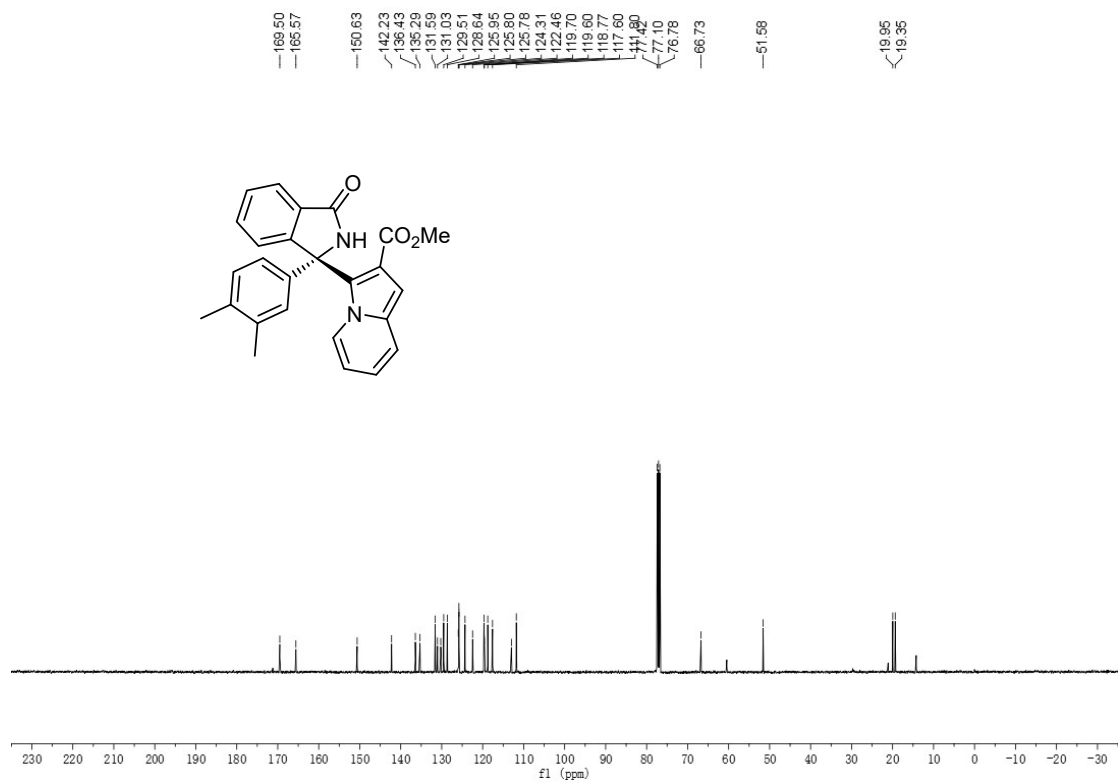


3e ¹³C NMR (101 MHz, CDCl₃-d₆)

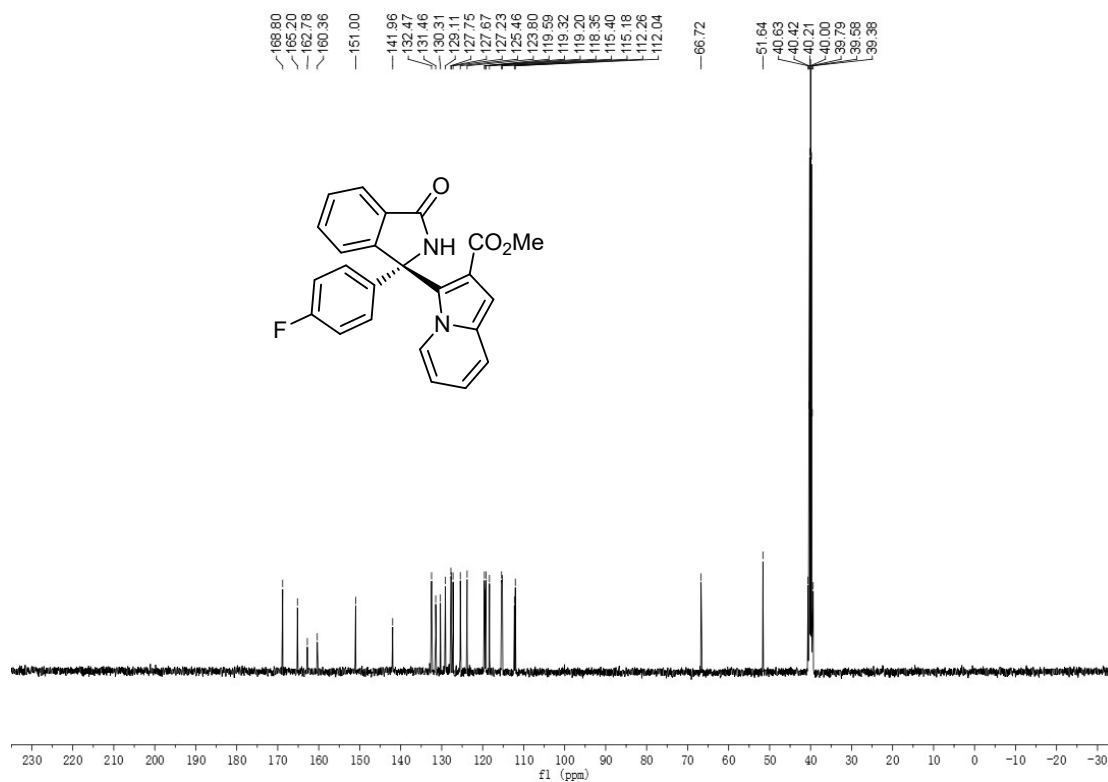
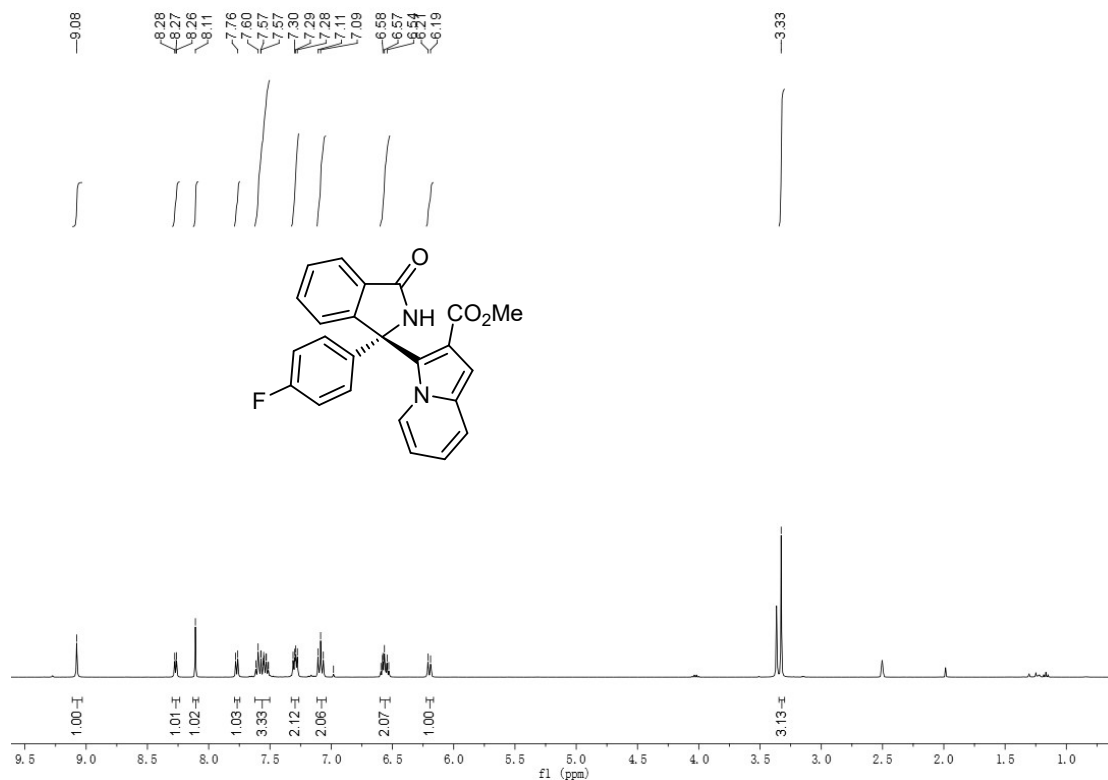


3f ¹H NMR (500 MHz, CDCl₃)

3f ¹³C NMR (101 MHz, CDCl₃)

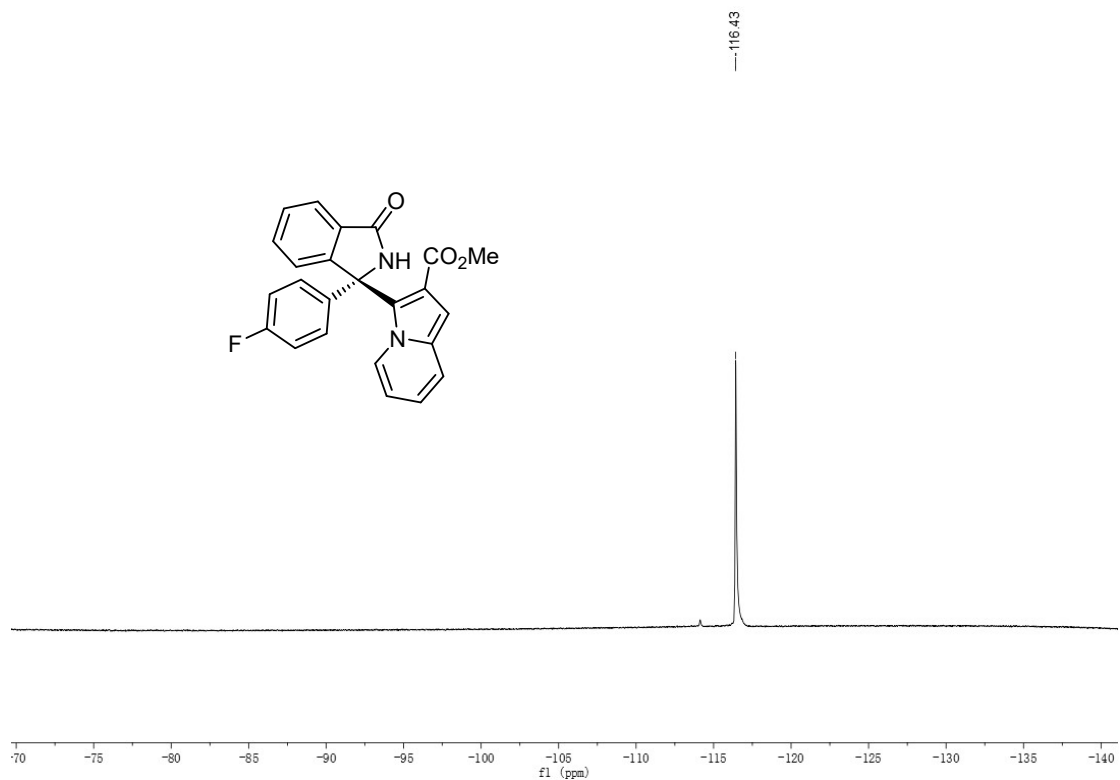


3g ^1H NMR (400 MHz, DMSO- d_6)

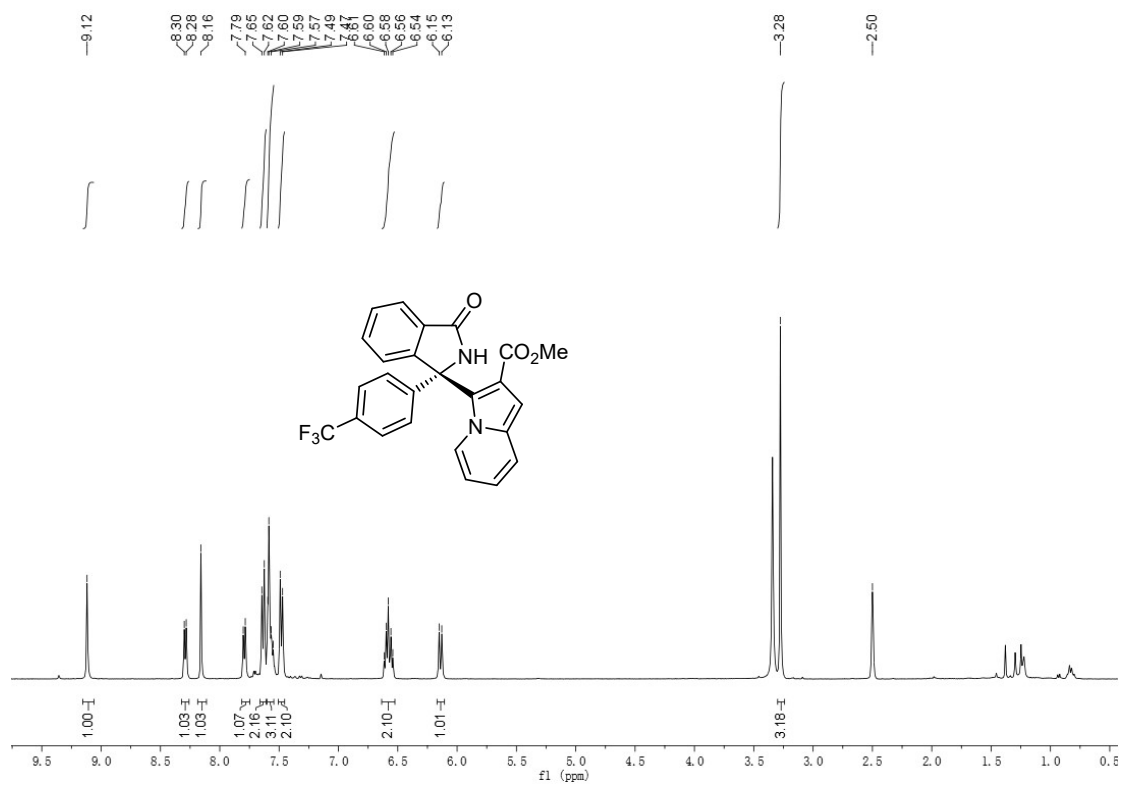


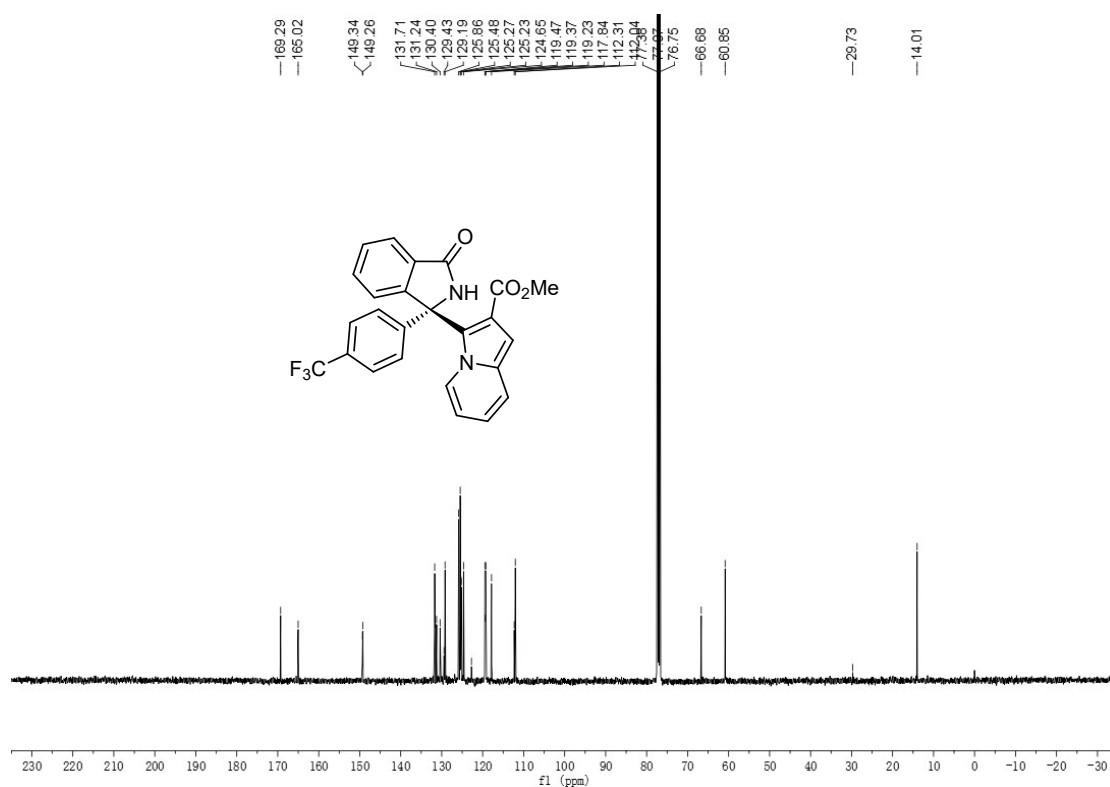
3g ^{13}C NMR (101 MHz, DMSO- d_6)

3g ^{19}F NMR (376 MHz, CDCl_3)



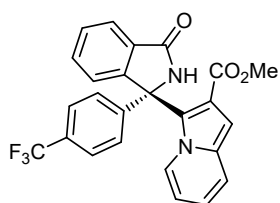
3h ^1H NMR (400 MHz, $\text{DMSO}-d_6$)

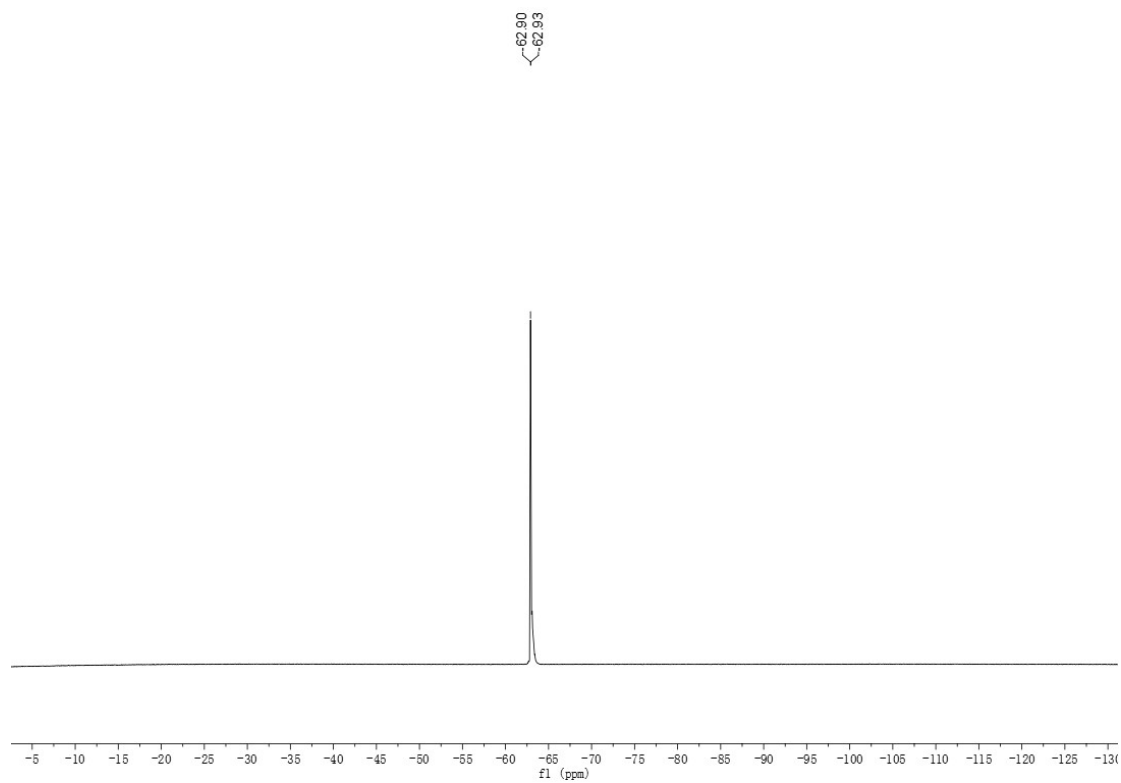




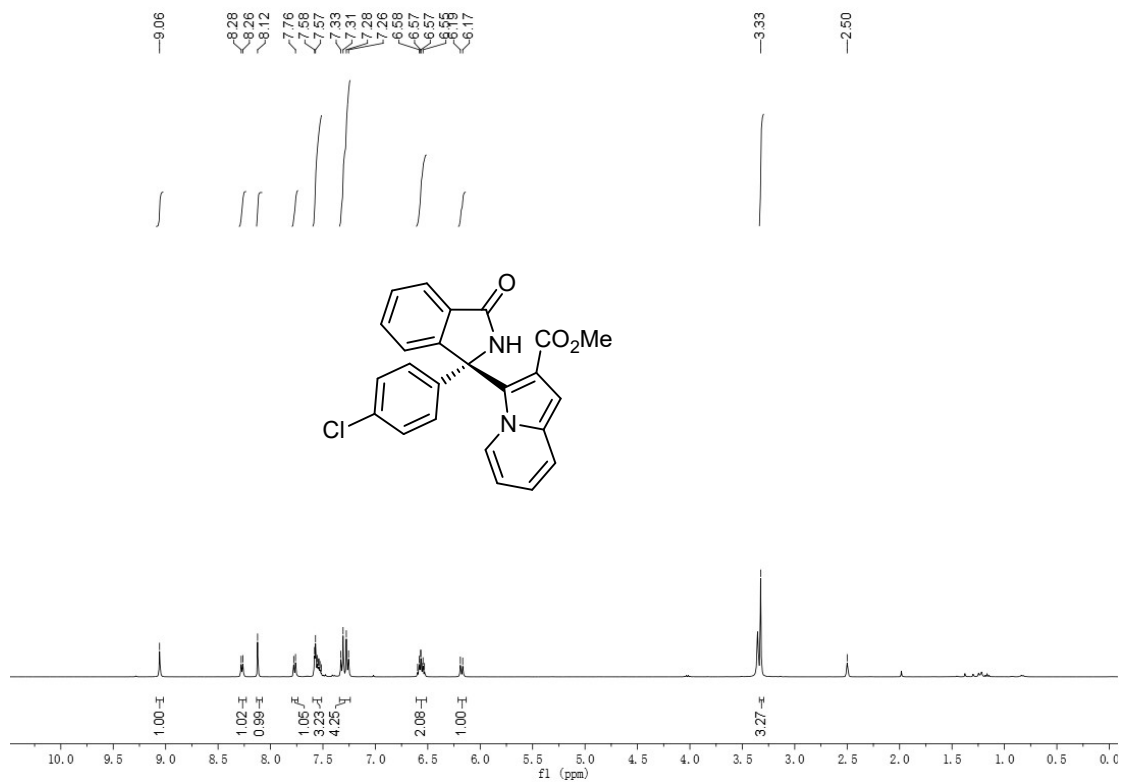
3h ¹³C NMR (101 MHz, CDCl₃)

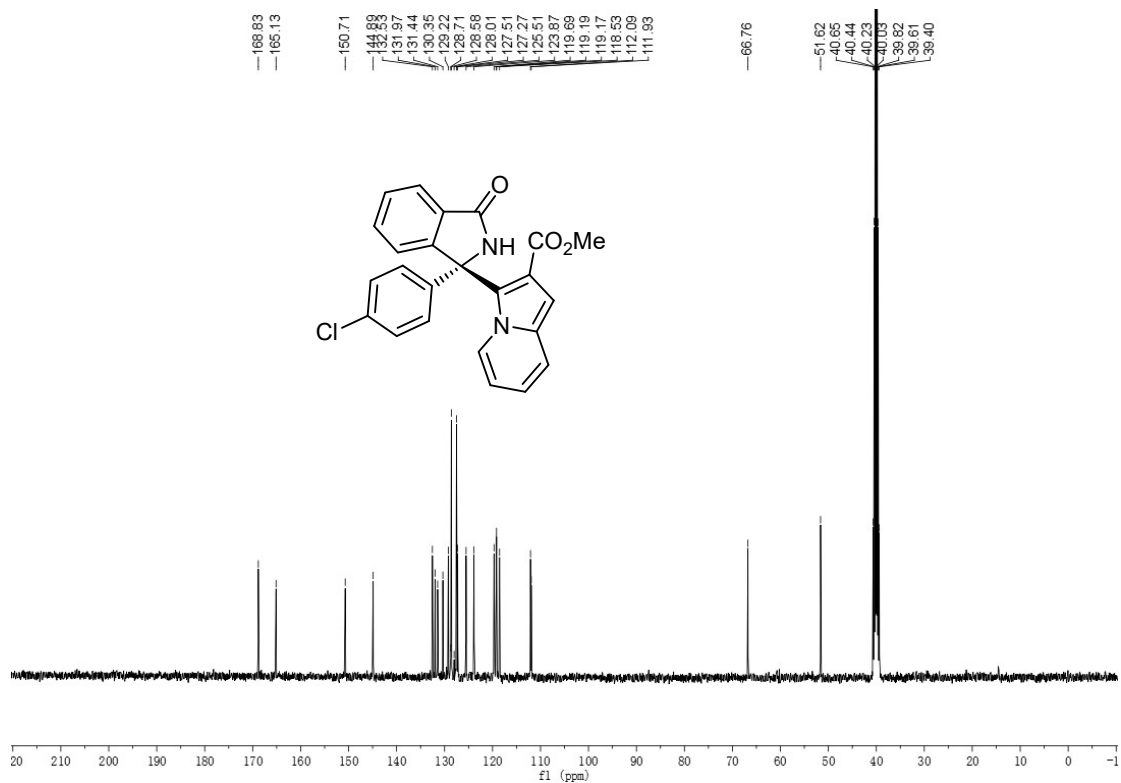
3h ¹⁹F NMR (376 MHz, CDCl₃)



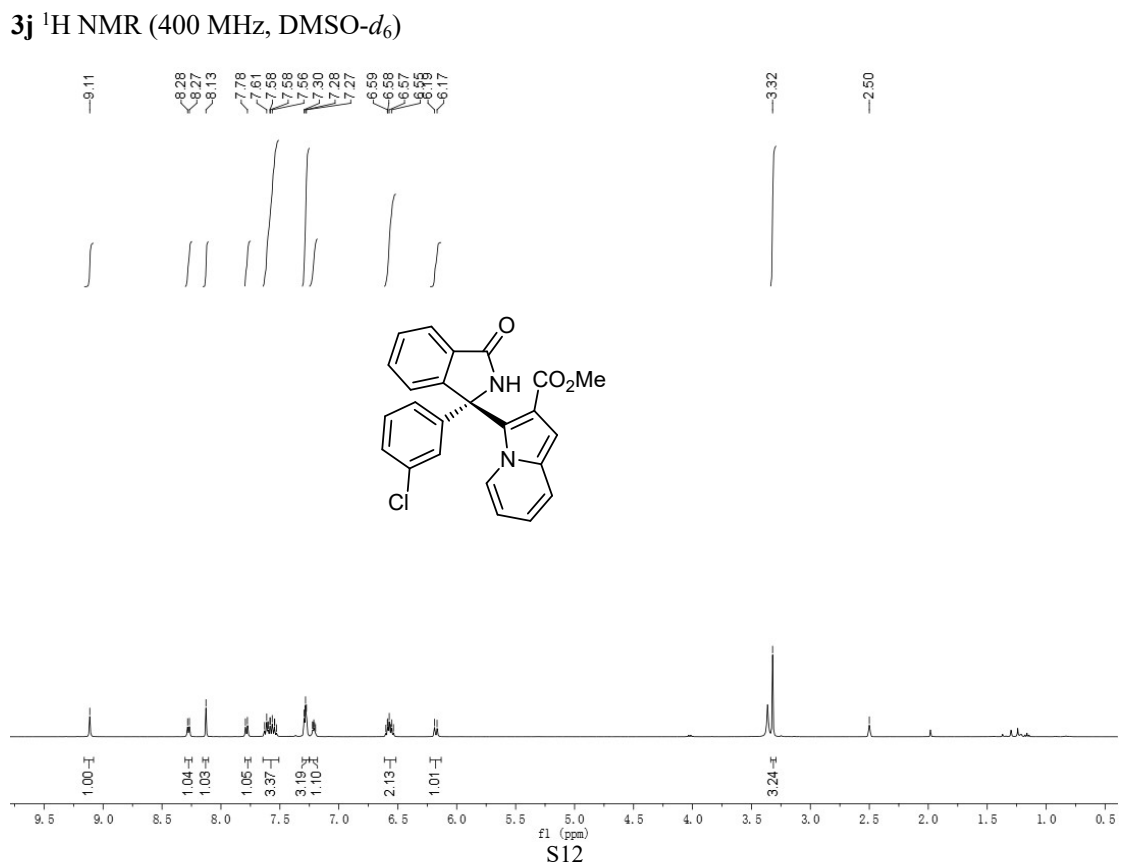


3i ^1H NMR (400 MHz, $\text{DMSO}-d_6$)



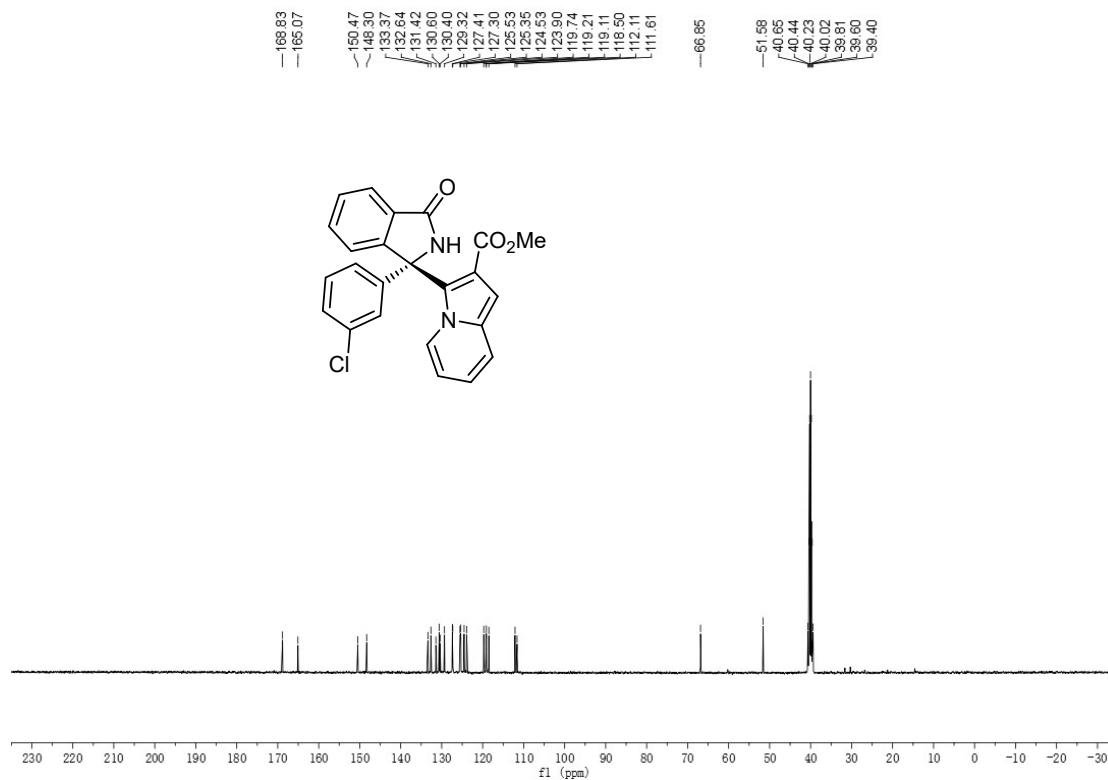


3i ¹³C NMR (101 MHz, DMSO-*d*₆)

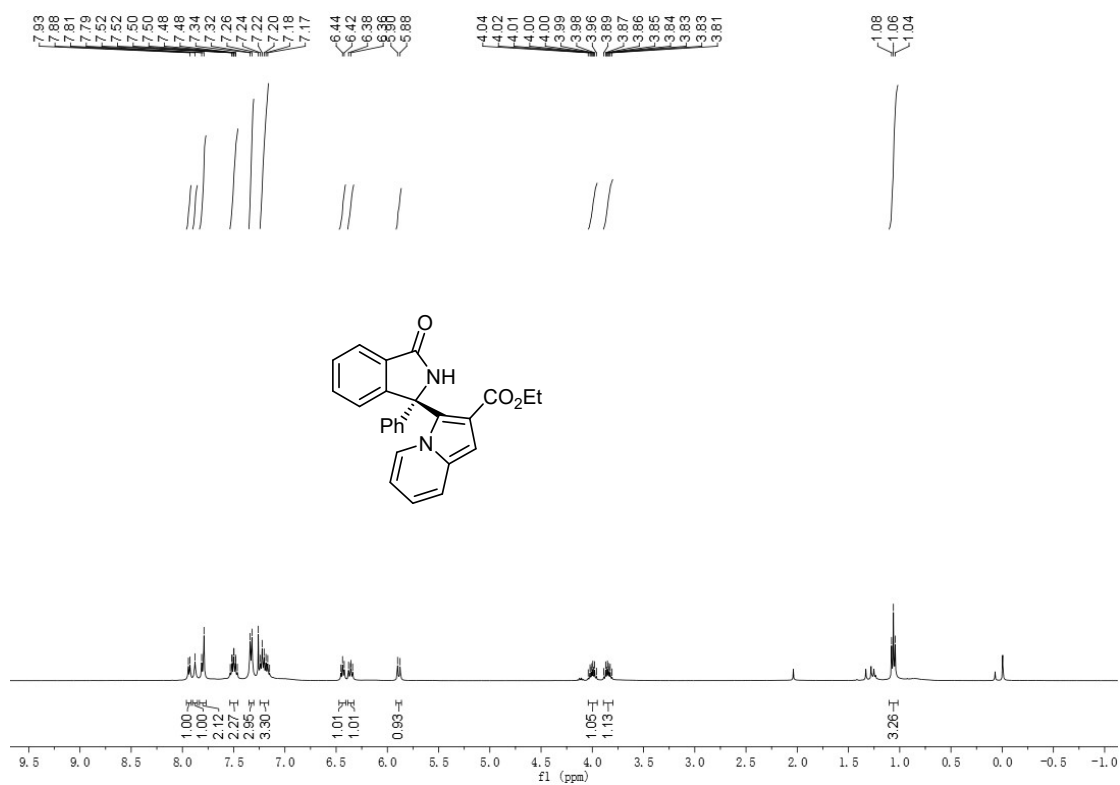


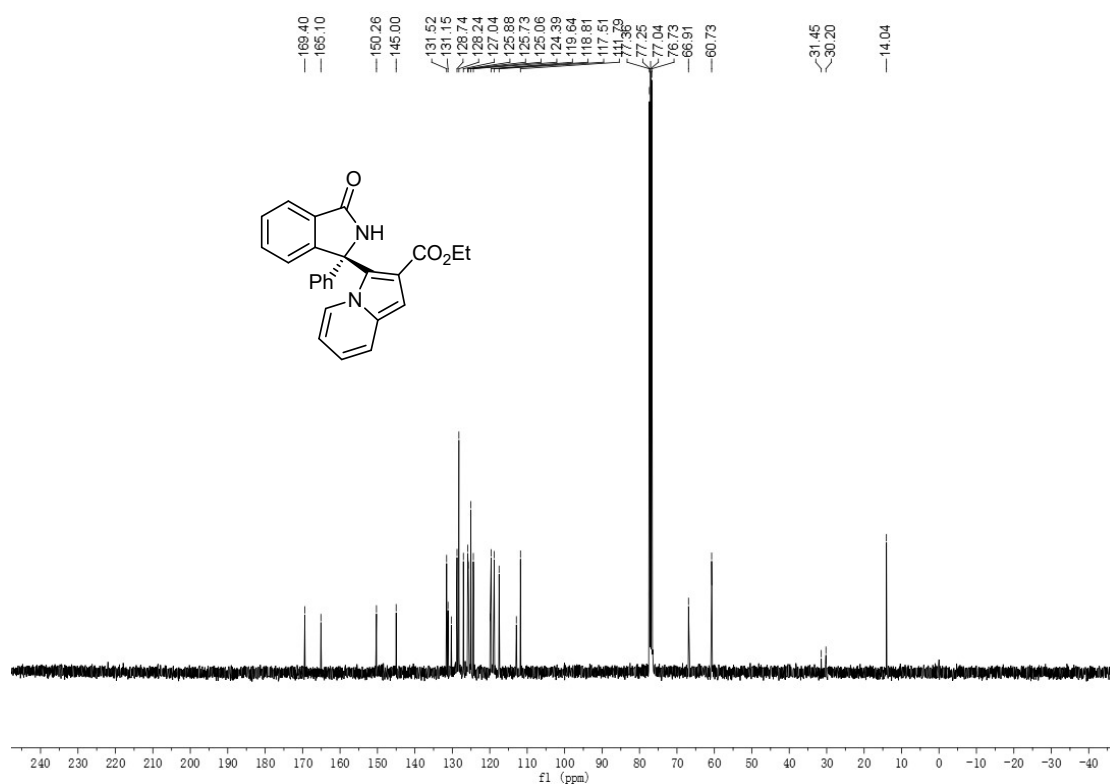
3j ¹H NMR (400 MHz, DMSO-*d*₆)

3j ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$)



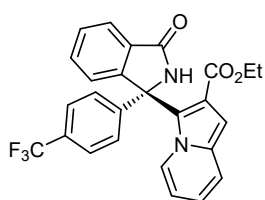
3k ^1H NMR (400 MHz, CDCl_3)

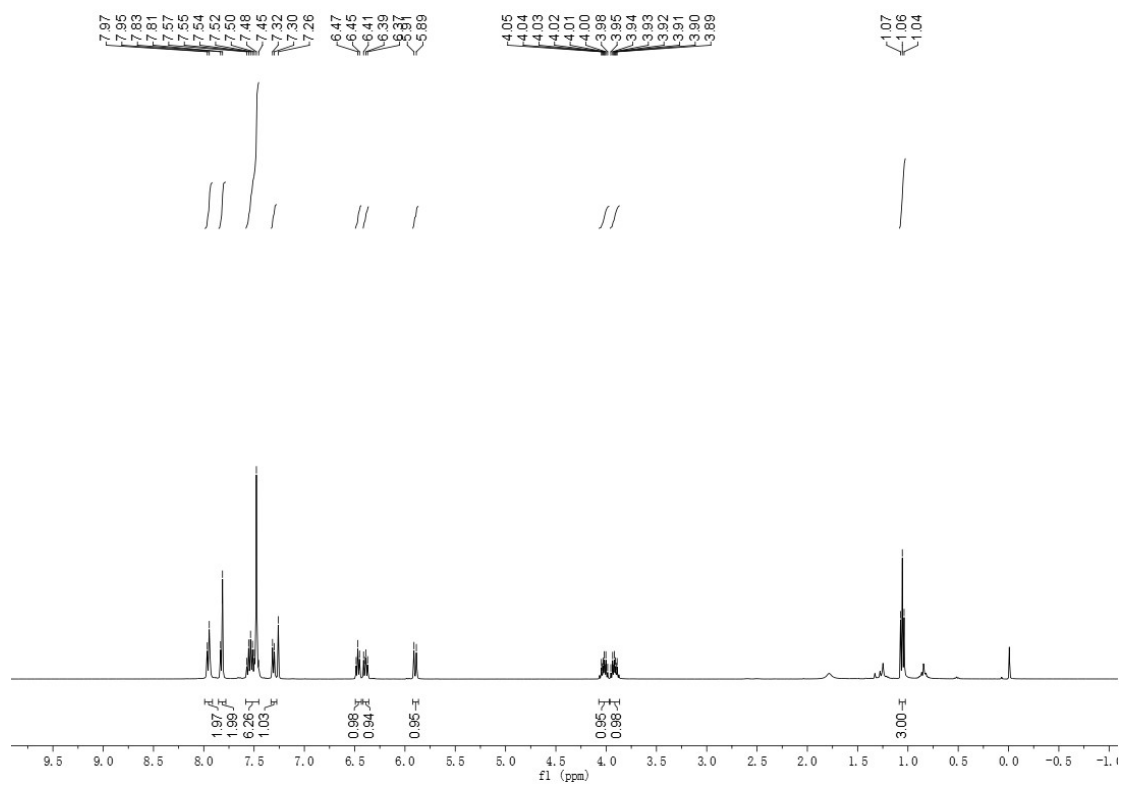




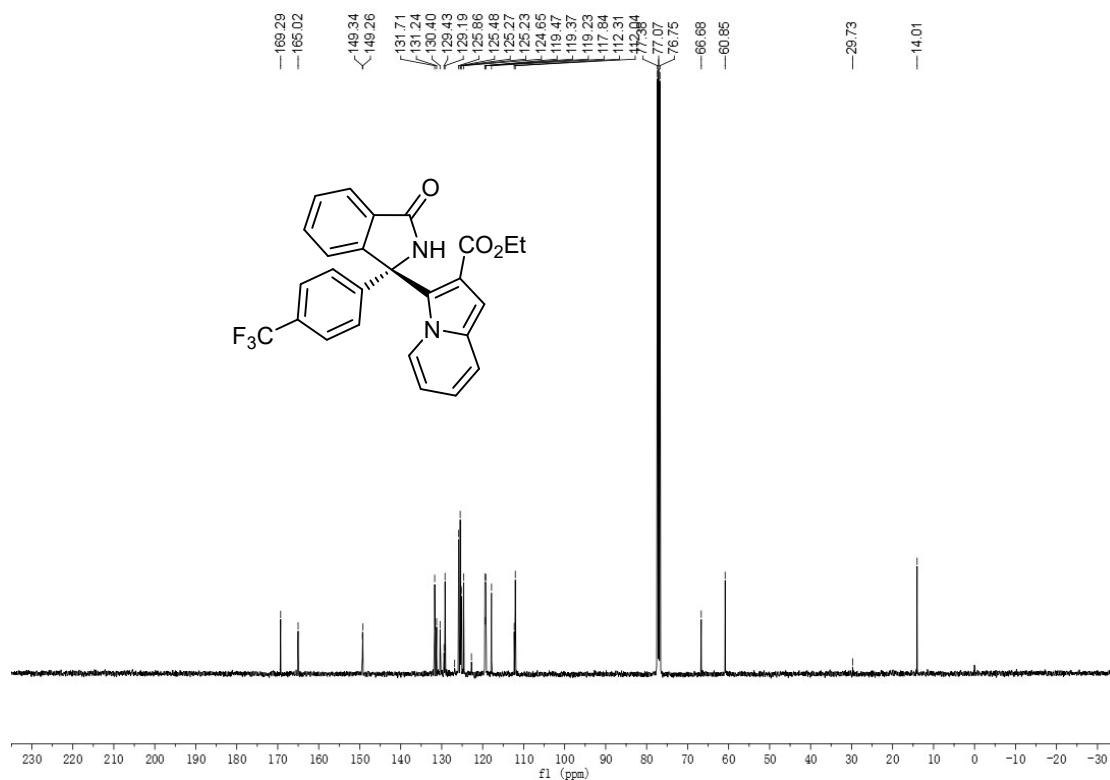
3k ¹³C NMR (101 MHz, CDCl₃)

3l ¹H NMR (400 MHz, CDCl₃)

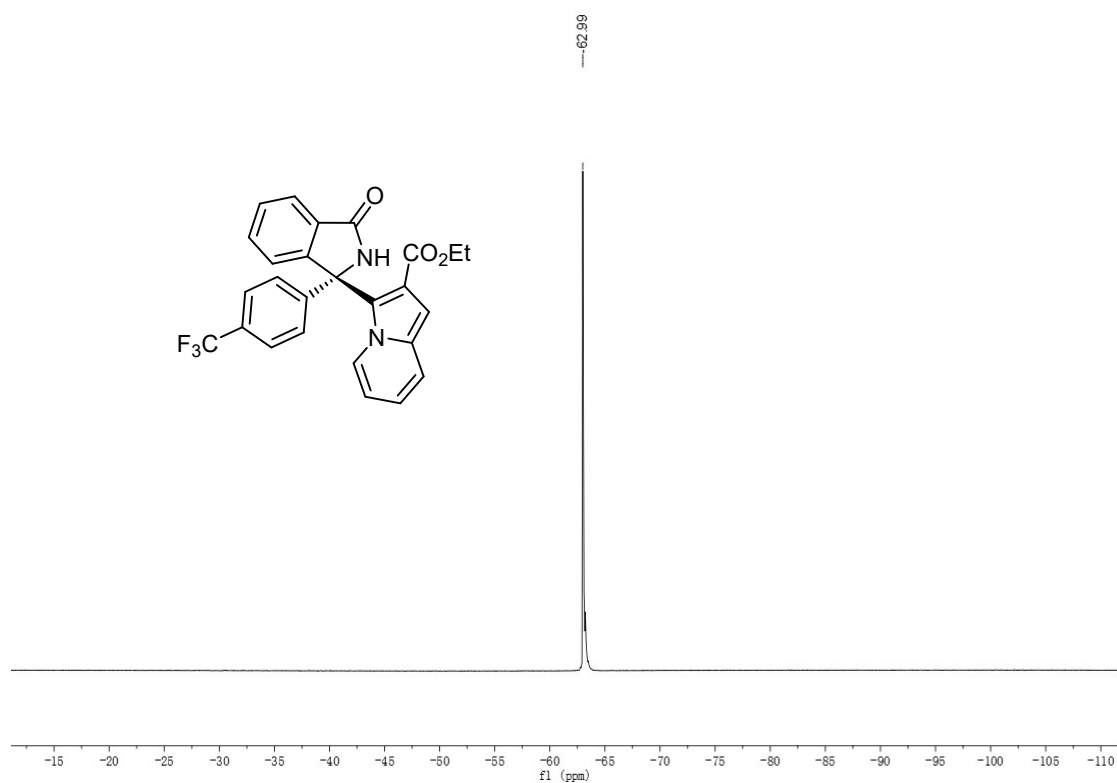




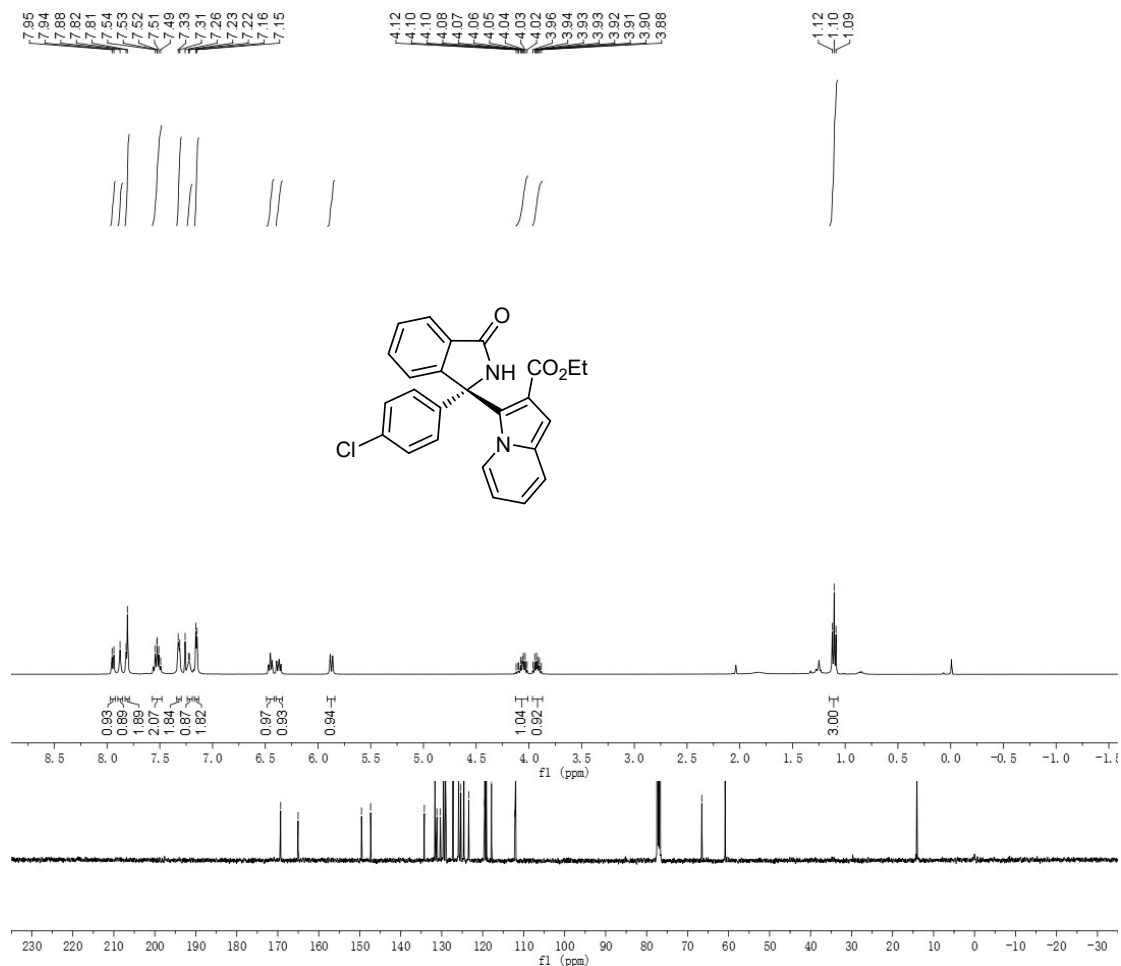
31 ¹³C NMR (101 MHz, CDCl₃)



3l ^{19}F NMR (376 MHz, CDCl_3)

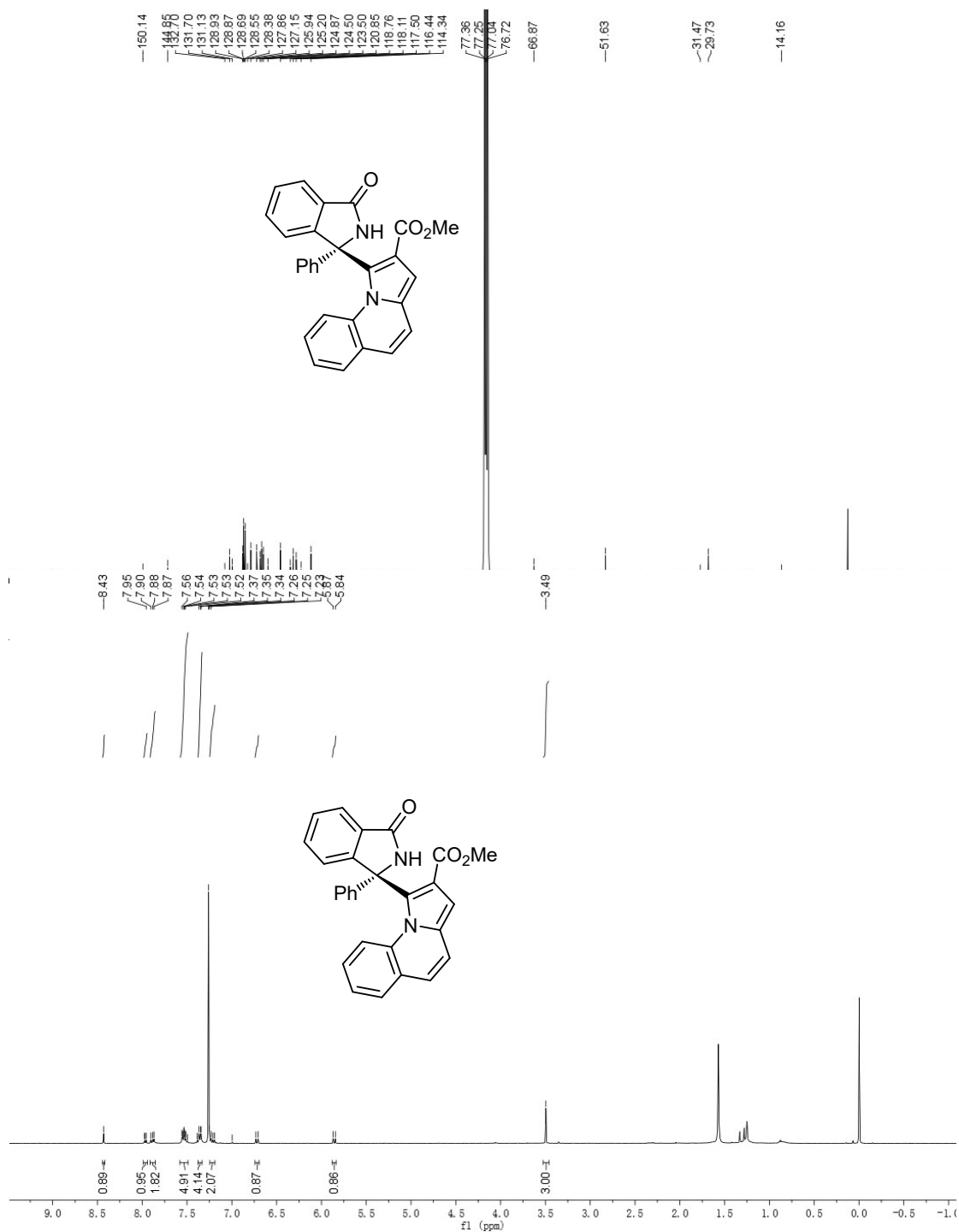


3m ^1H NMR (400 MHz, CDCl_3)

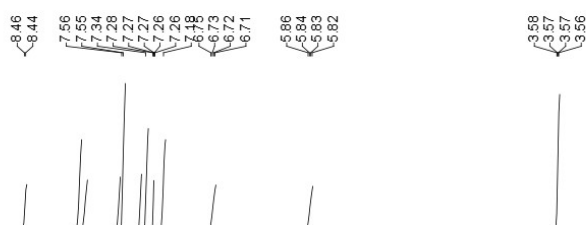


3n ^1H NMR (400 MHz, CDCl_3)

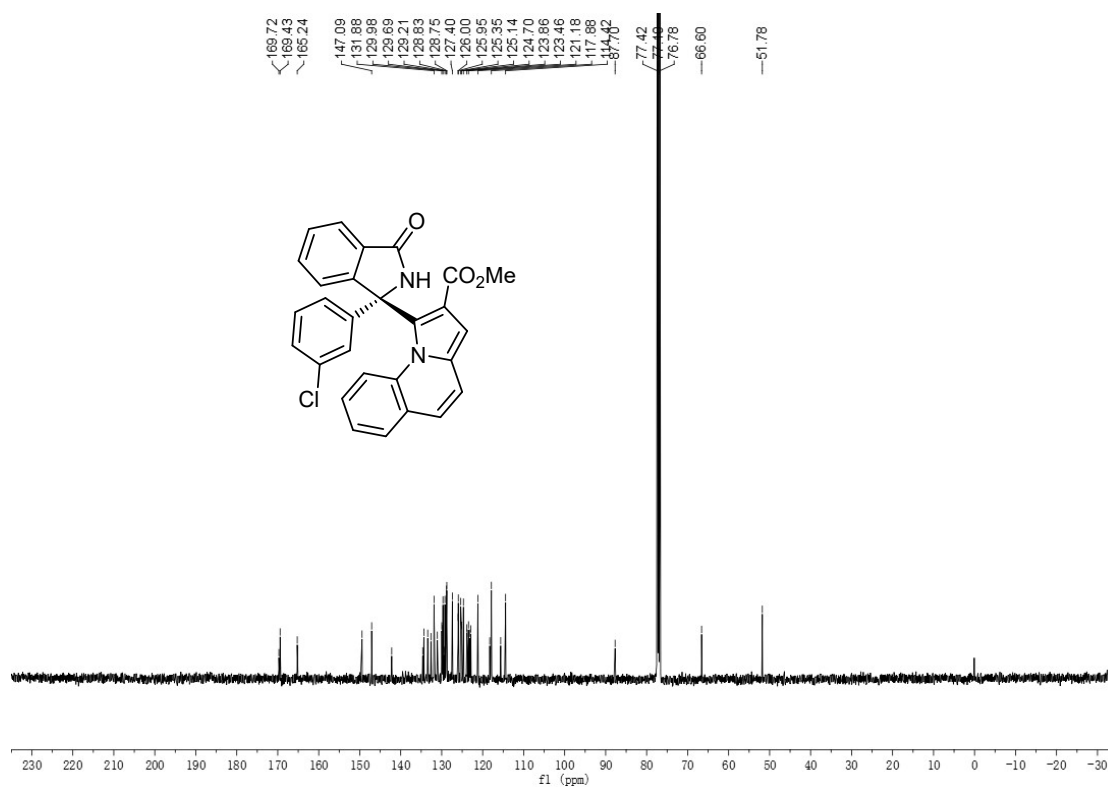
3n ^{13}C NMR (101 MHz, CDCl_3)



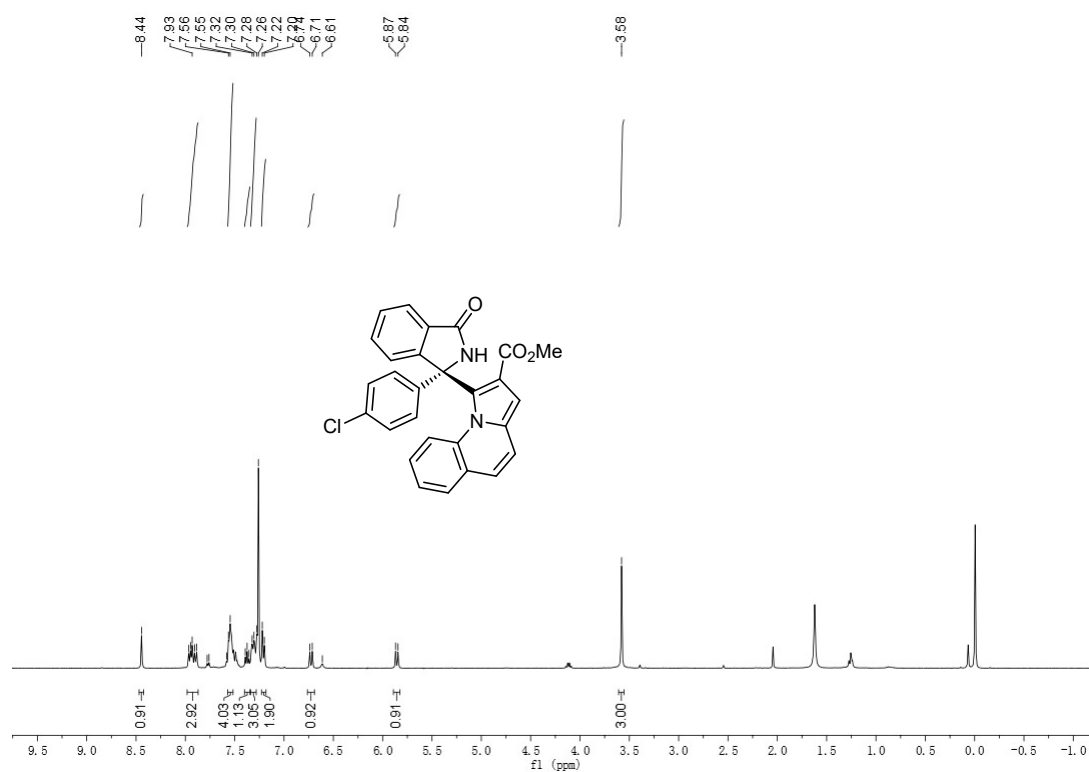
3o ^1H NMR (400 MHz, CDCl_3)



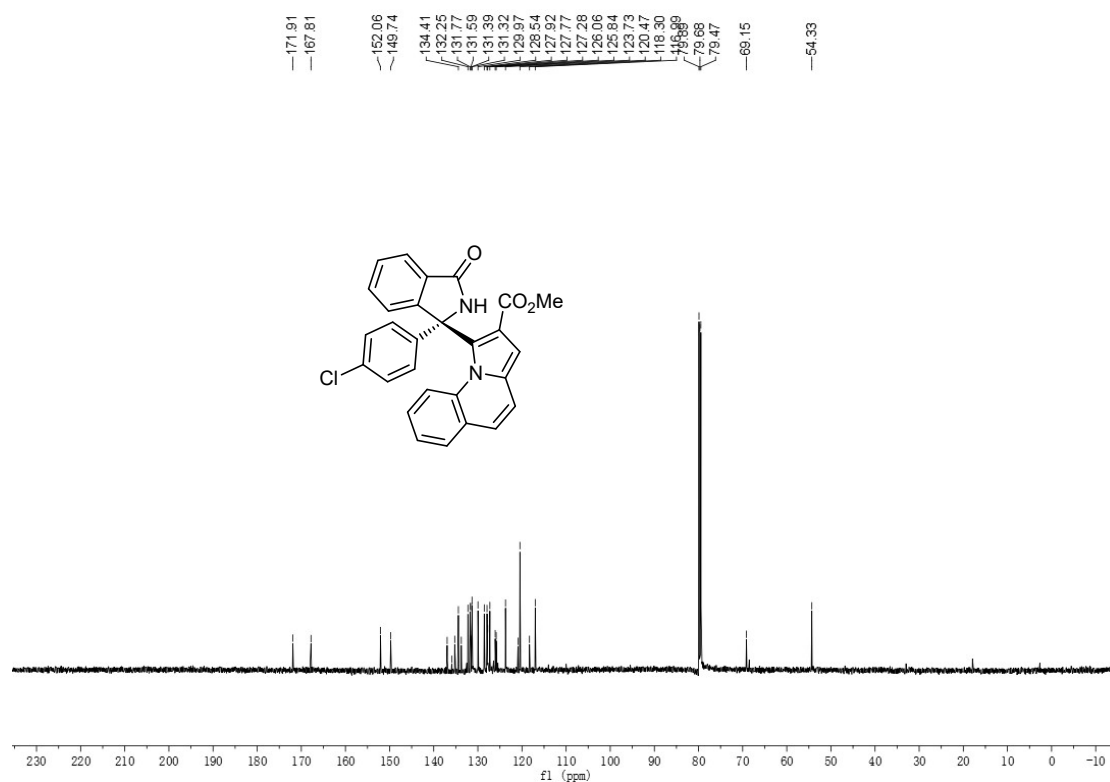
3o ^{13}C NMR (101 MHz, CDCl_3)



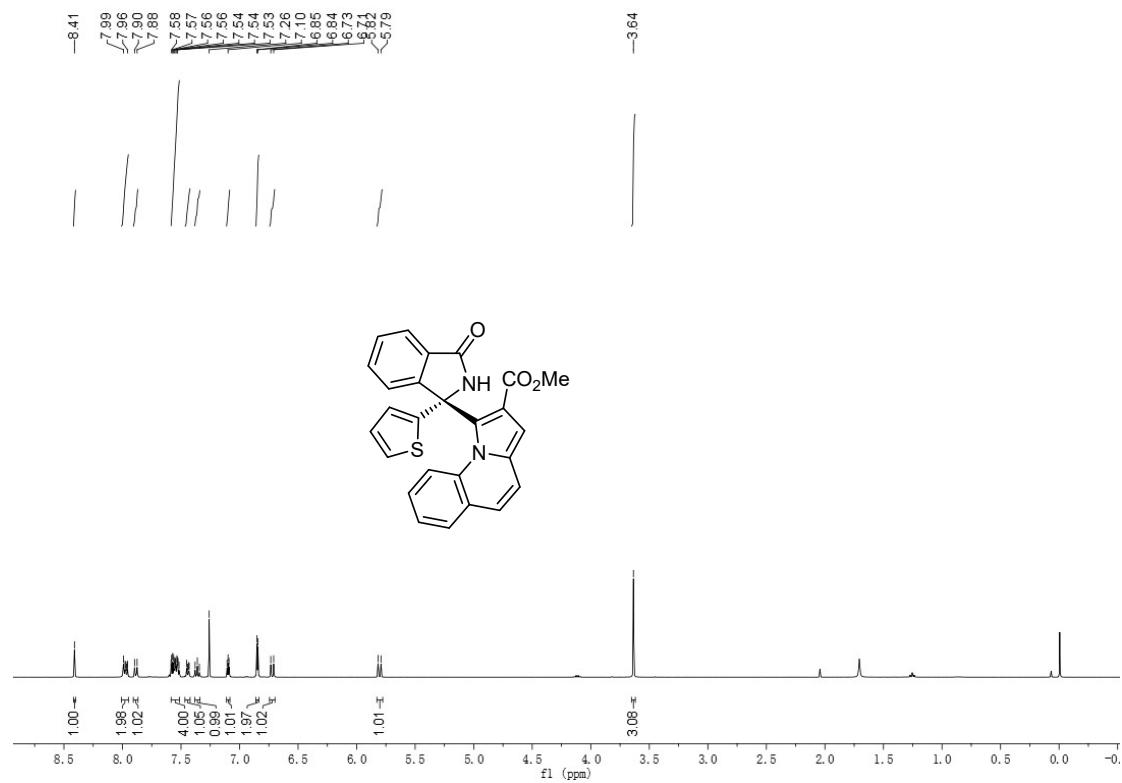
3p ^1H NMR (400 MHz, CDCl_3)



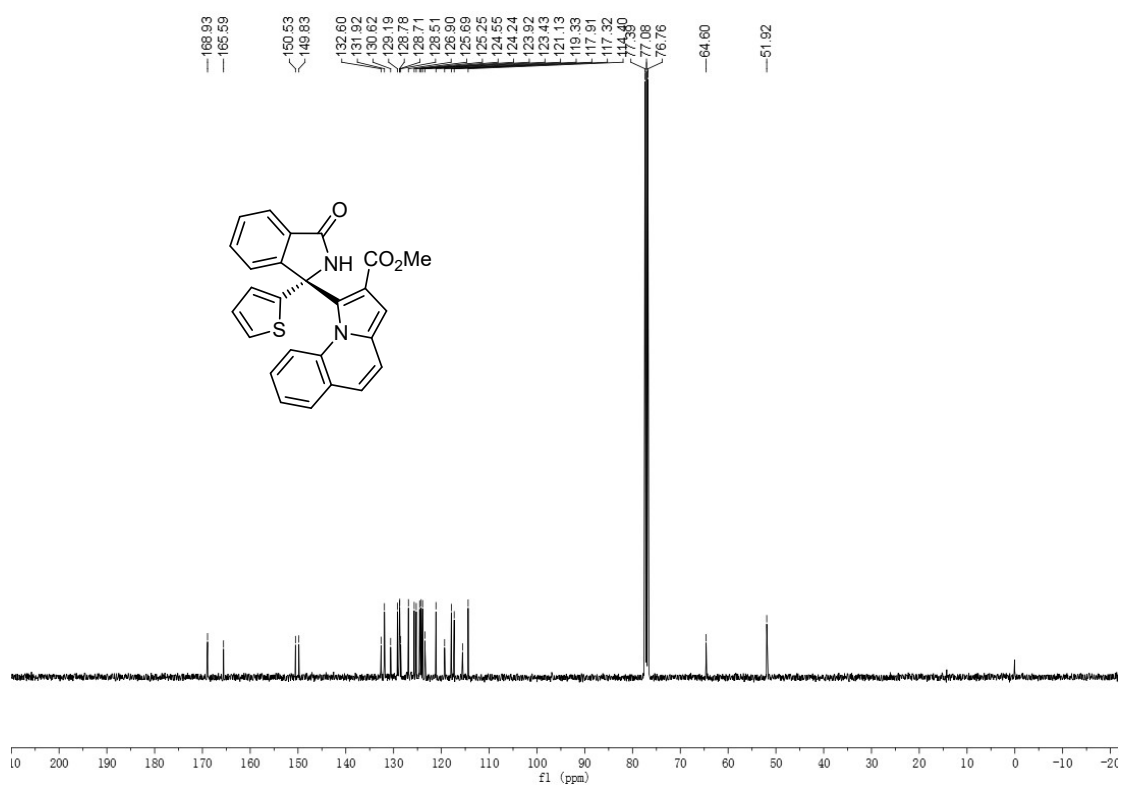
3p ^{13}C NMR (101 MHz, CDCl_3)



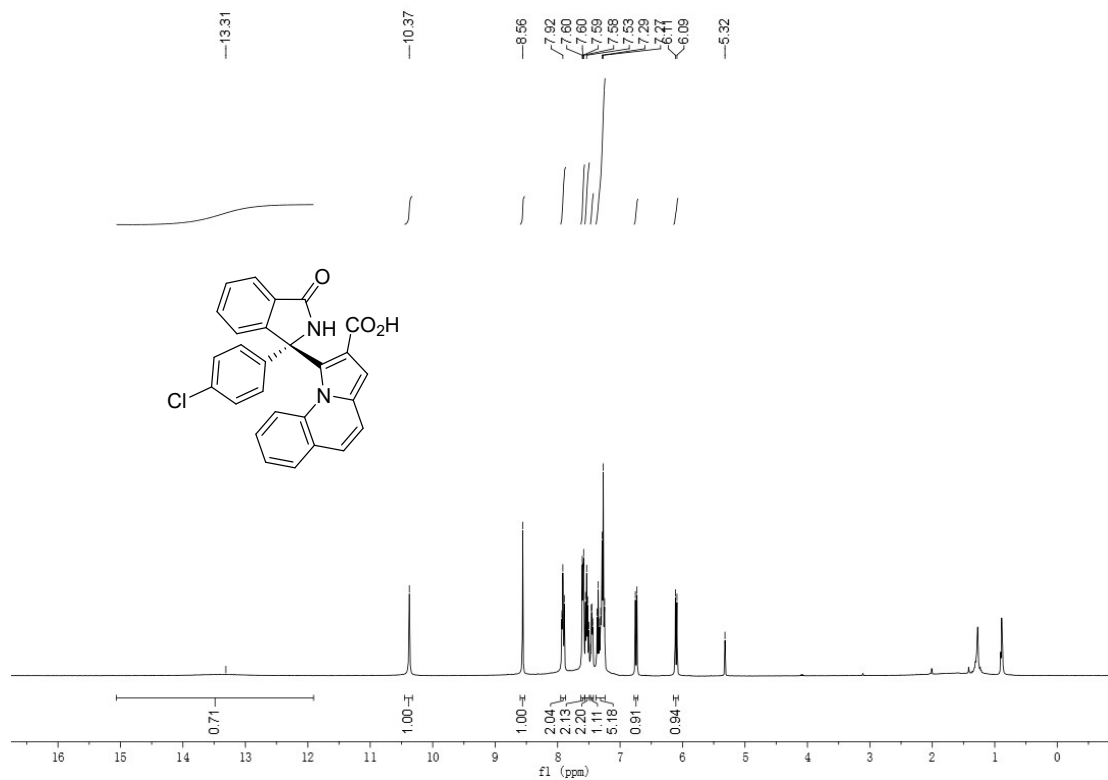
3q ^1H NMR (400 MHz, CDCl_3)



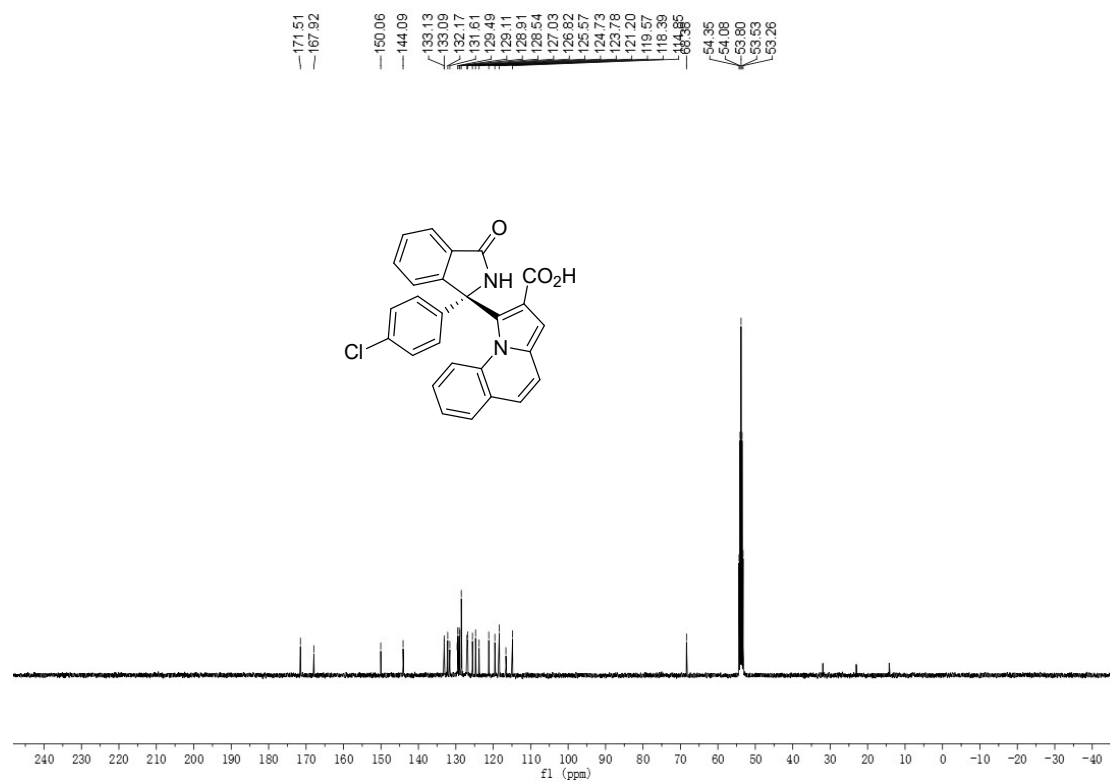
3q ^{13}C NMR (101 MHz, CDCl_3)



4 ^1H NMR (400 MHz, CD_2Cl_2)



4 ^{13}C NMR (101 MHz, CD_2Cl_2)



2. X-Ray Single Crystal Data for Product (*R*)-3p

Deposition Number CCDC 2167990

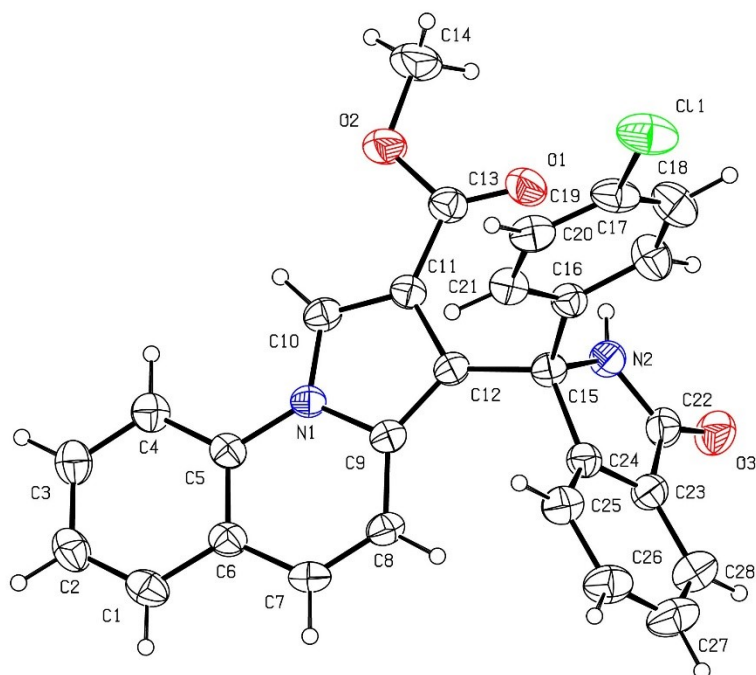
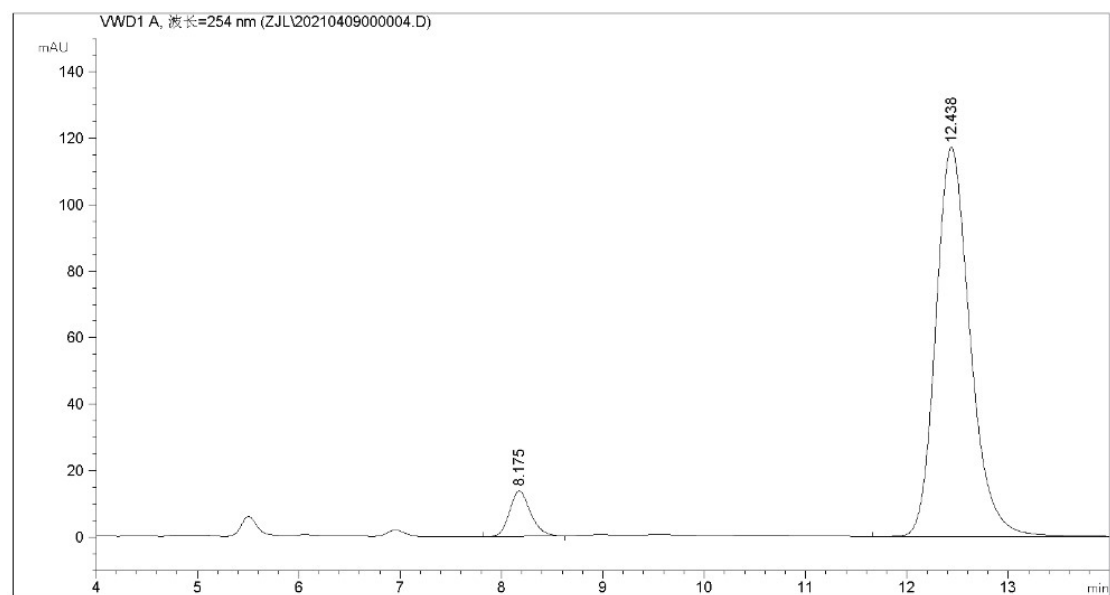
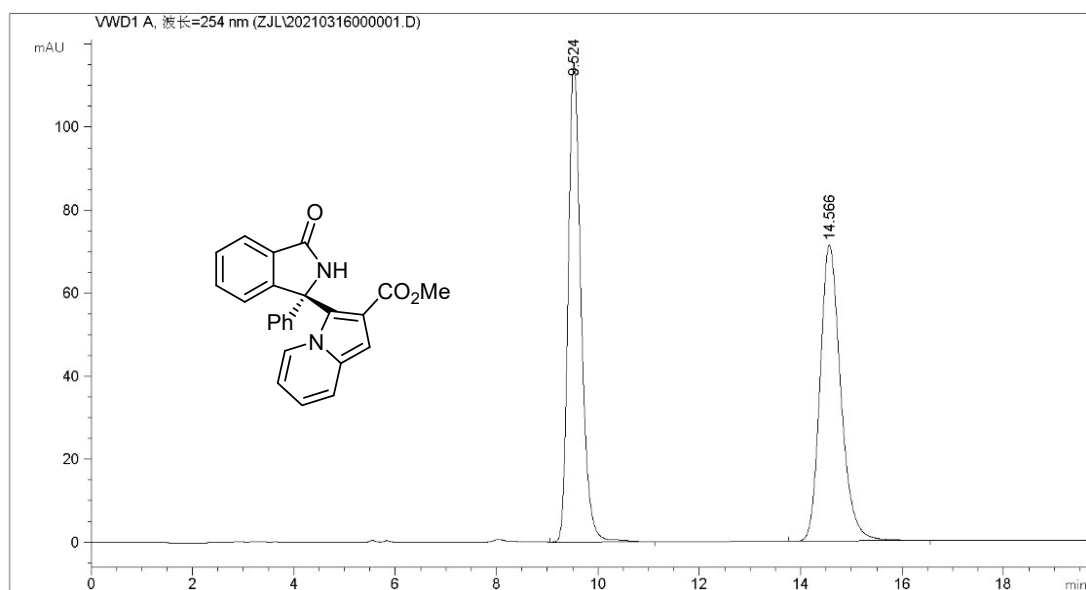


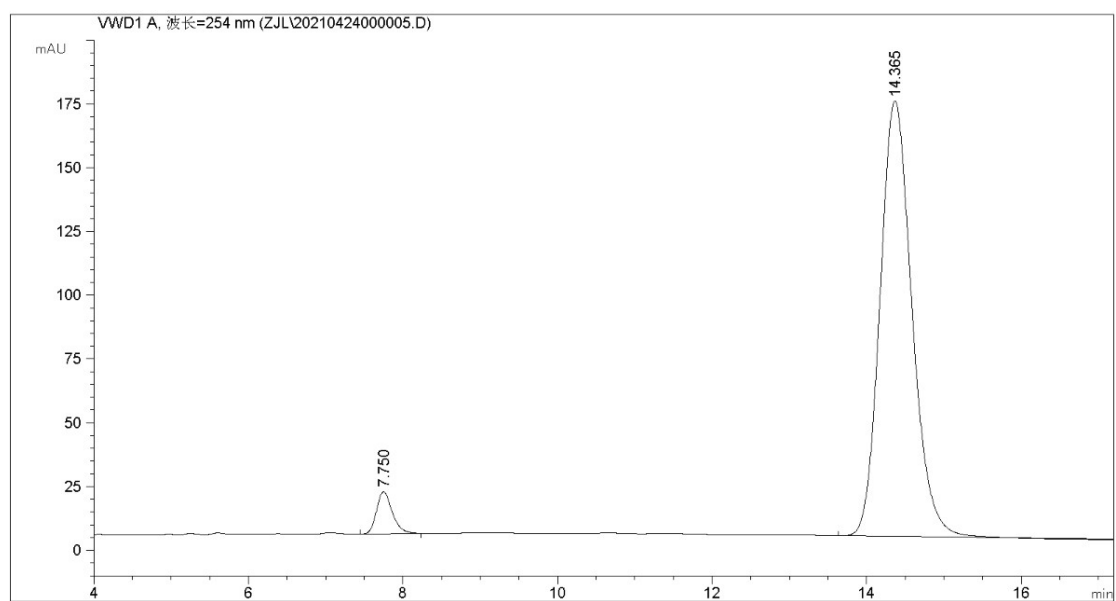
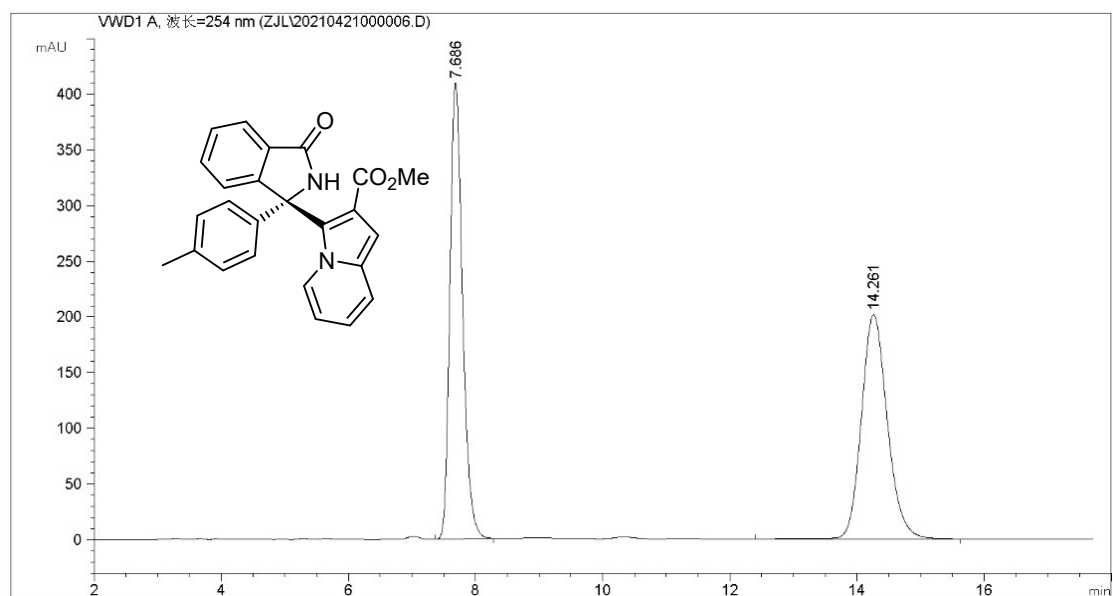
Table 1 Crystal data and structure refinement

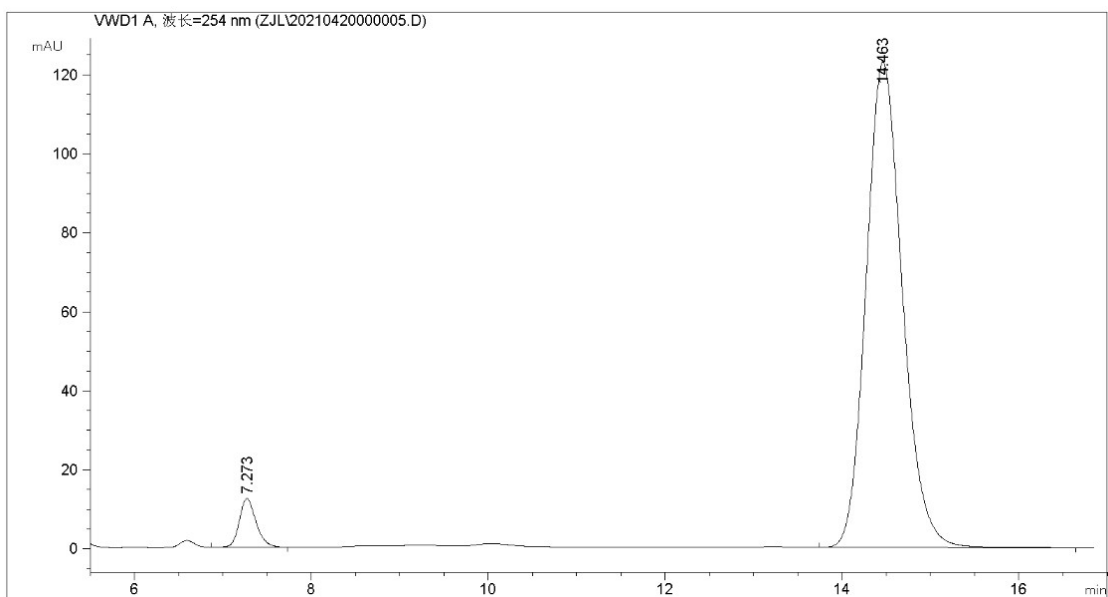
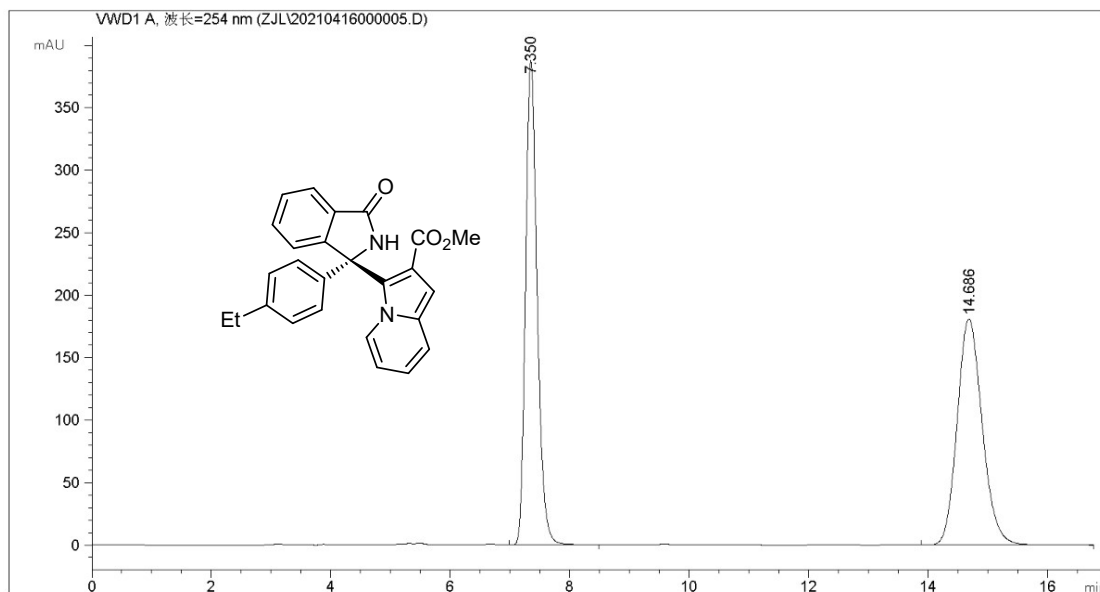
Empirical formula	C ₂₈ H ₂₃ Cl ₂ N ₂ O ₃
Formula weight	446.77
Temperature/K	296.15
Crystal system	monoclinic
Space group	C2
a/Å	14.5506(14)
b/Å	14.4941(4)
c/Å	24.9660(13)
α /°	90
β /°	125.773(8)
γ /°	90
Volume/Å ³	4467.8(8)
Z	4
$\rho_{\text{calc}}/\text{g cm}^{-3}$	1.333
μ/mm^{-1}	0.195
F(000)	3008.0
Crystal size/mm ³	0.48 × 0.19 × 0.15

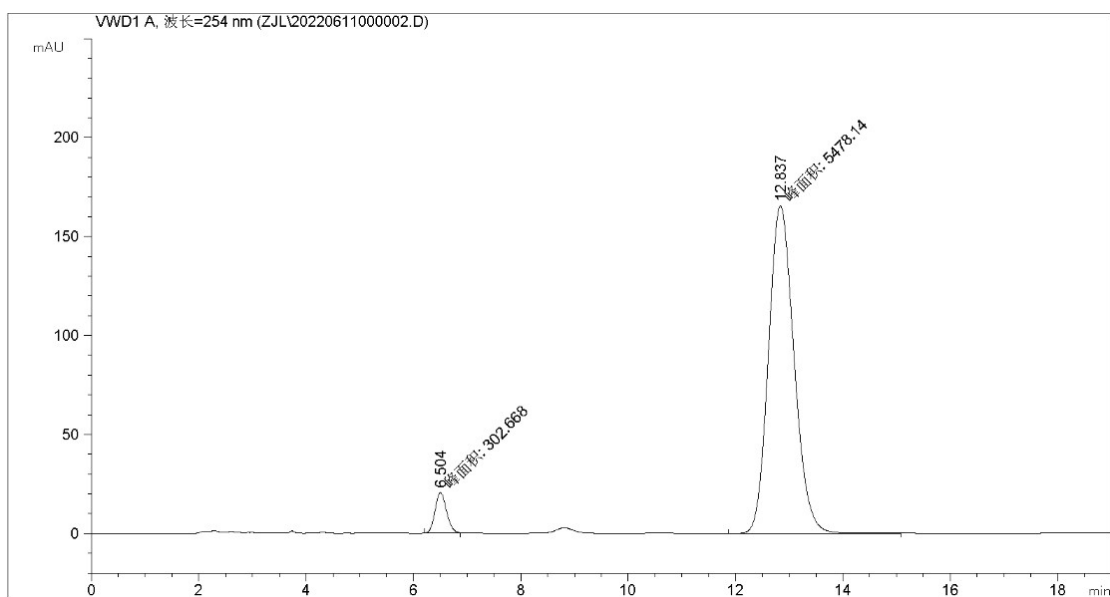
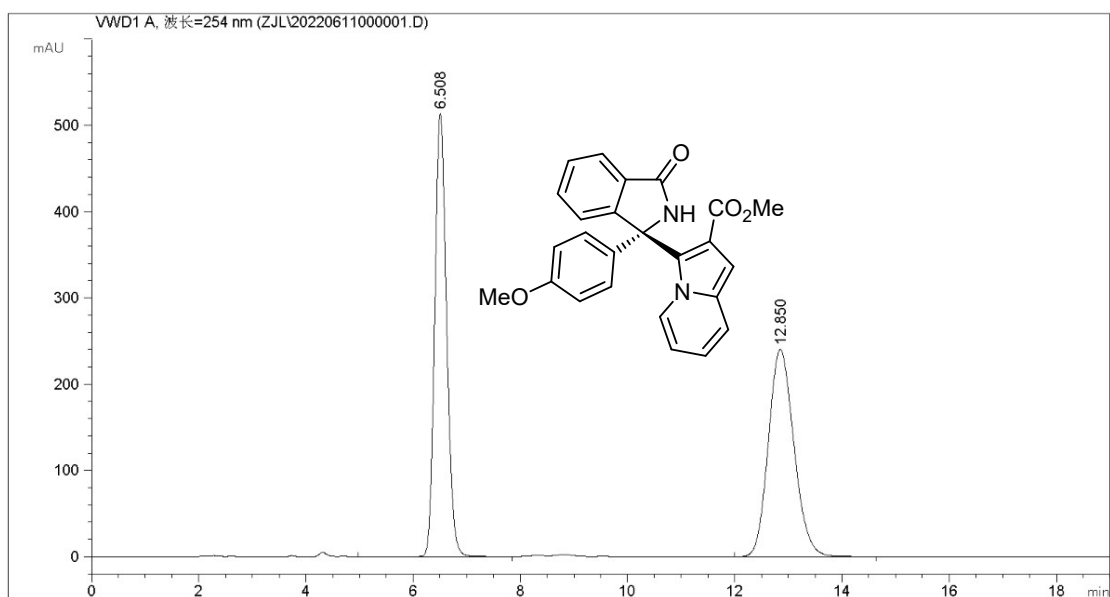
Radiation	MoK α ($\lambda = 0.71073$)
2 Θ range for data collection/ $^{\circ}$	3.698 to 58.786
Index ranges	$-32 \leq h \leq 33$, $-19 \leq k \leq 19$, $-34 \leq l \leq 33$
Reflections collected	63847
Independent reflections	17756 [$R_{\text{int}} = 0.0422$, $R_{\text{sigma}} = 0.0461$]
Data/restraints/parameters	17756/1/951
Goodness-of-fit on F^2	1.008
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0516$, $wR_2 = 0.1104$
Final R indexes [all data]	$R_1 = 0.0951$, $wR_2 = 0.1289$
Largest diff. peak/hole / e $\text{\AA}^{-3}$	0.32/-0.27
Flack parameter	-0.039(19)

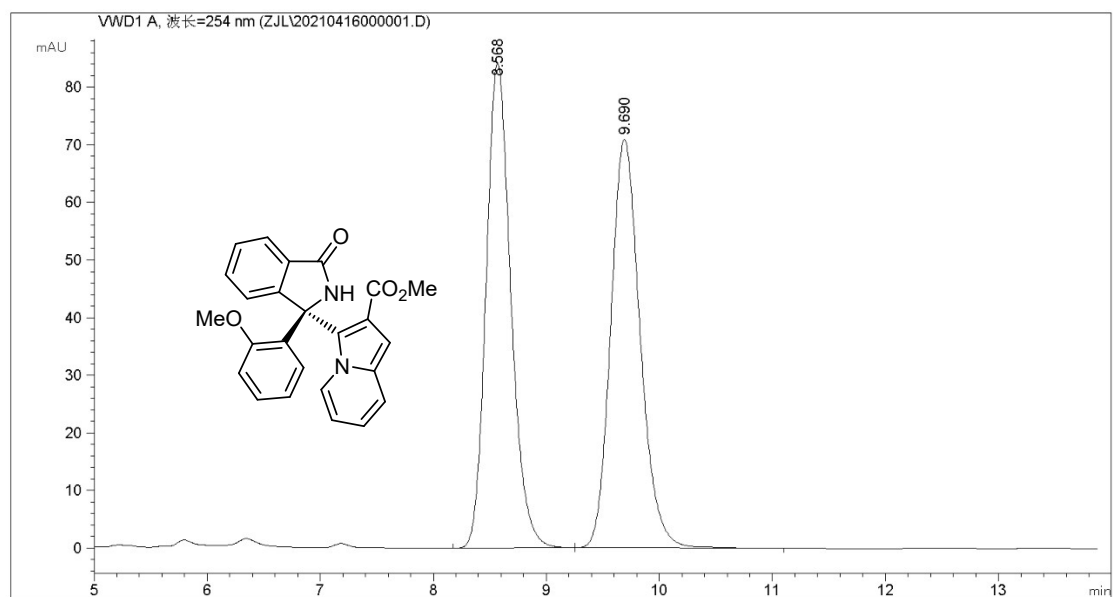
3. Copies of HPLC Spectra



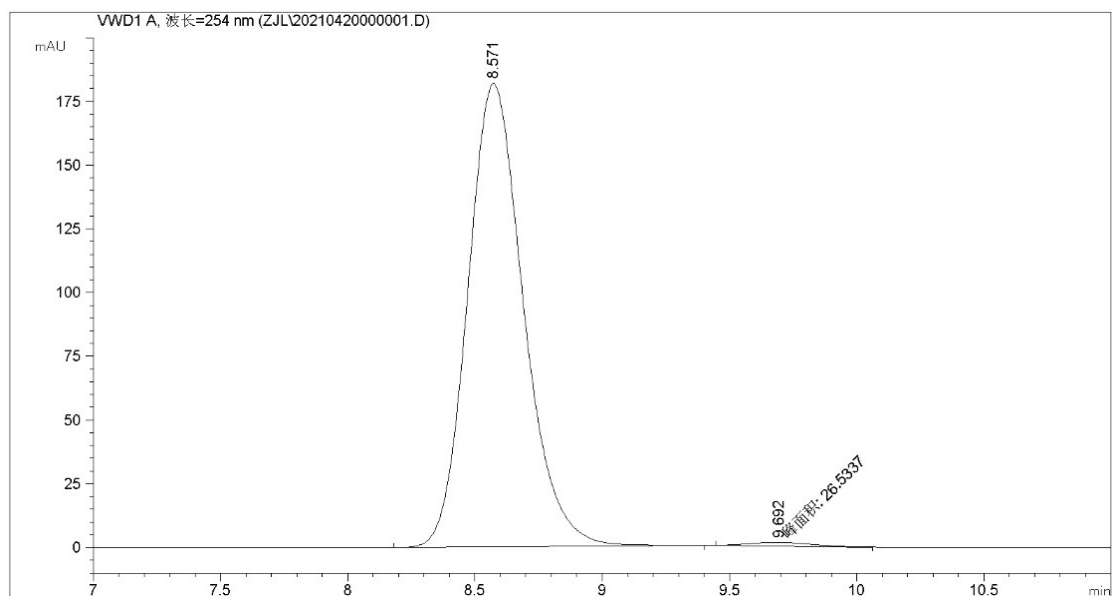




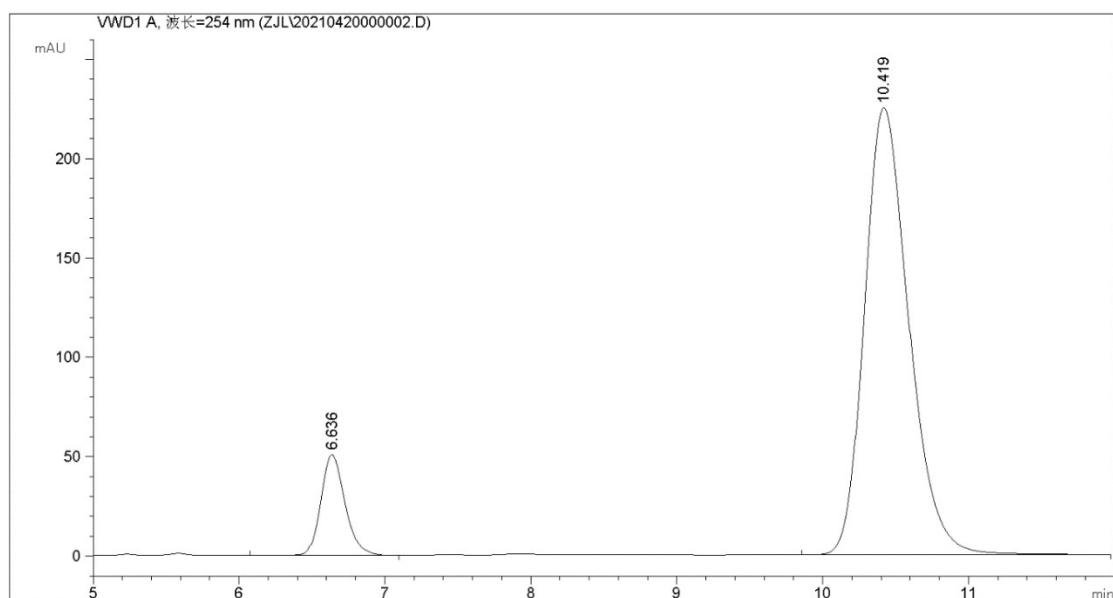
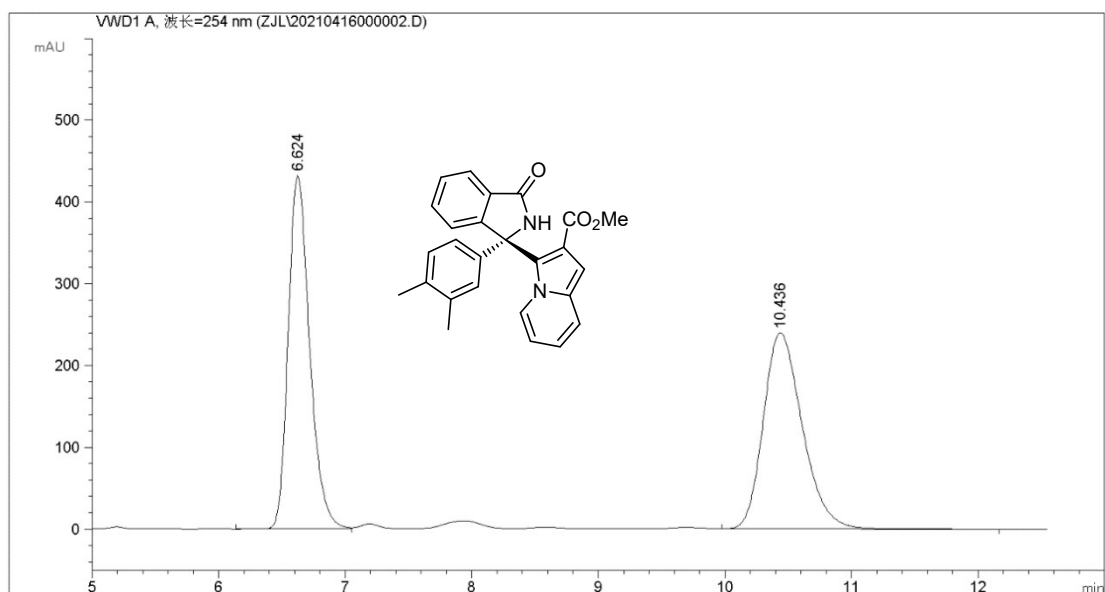


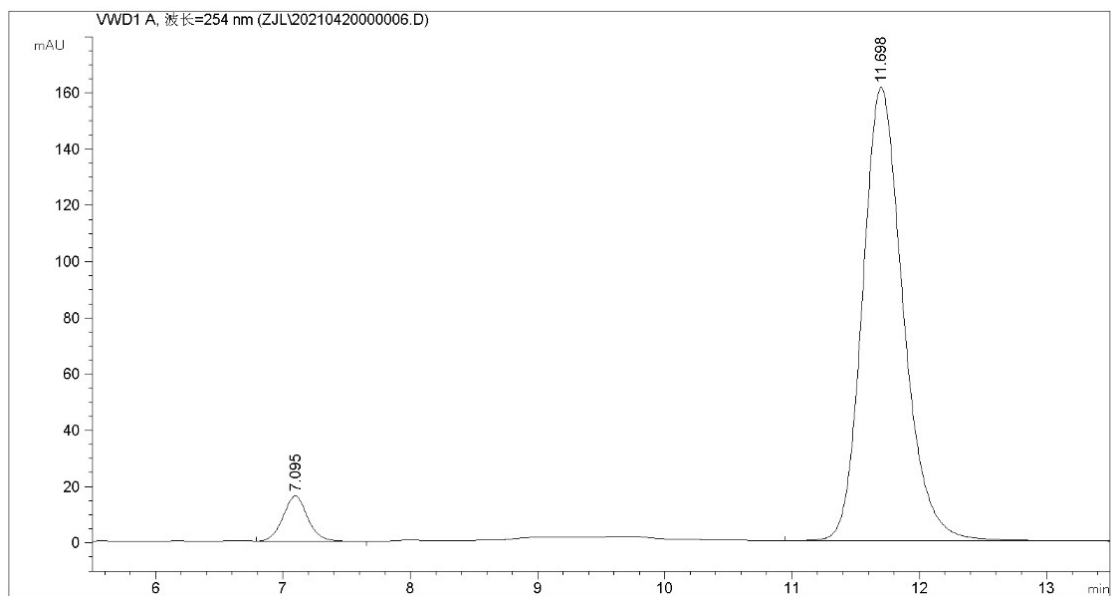
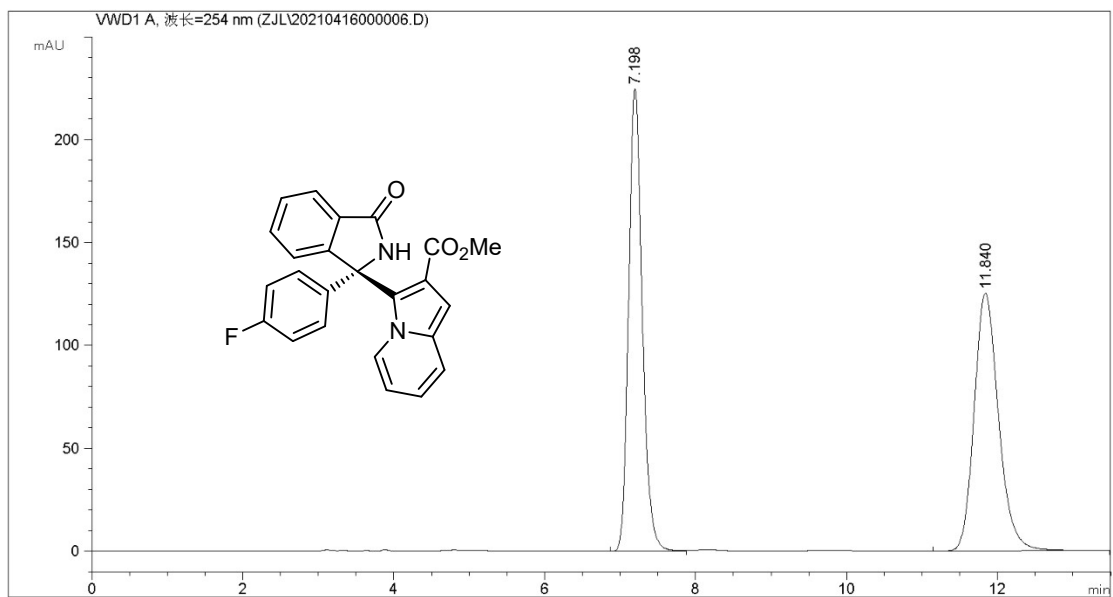


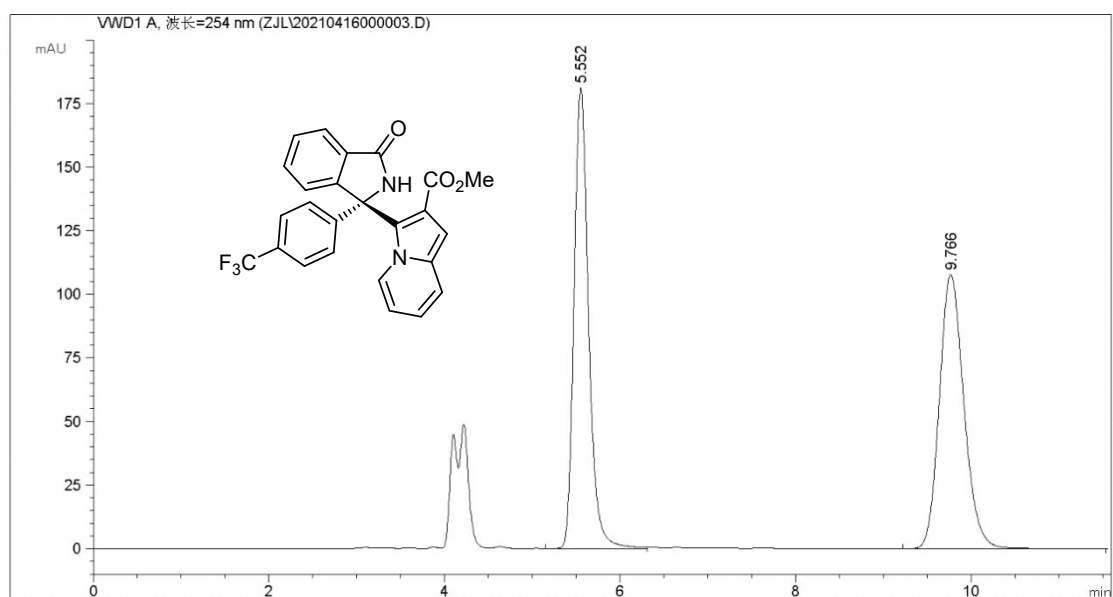
#	[min]		[min]	mAU	*s	[mAU]	%
1	8.568	BB	0.2337	1277.06982		84.20490	49.9232
2	9.690	BB	0.2777	1281.00098		70.91061	50.0768



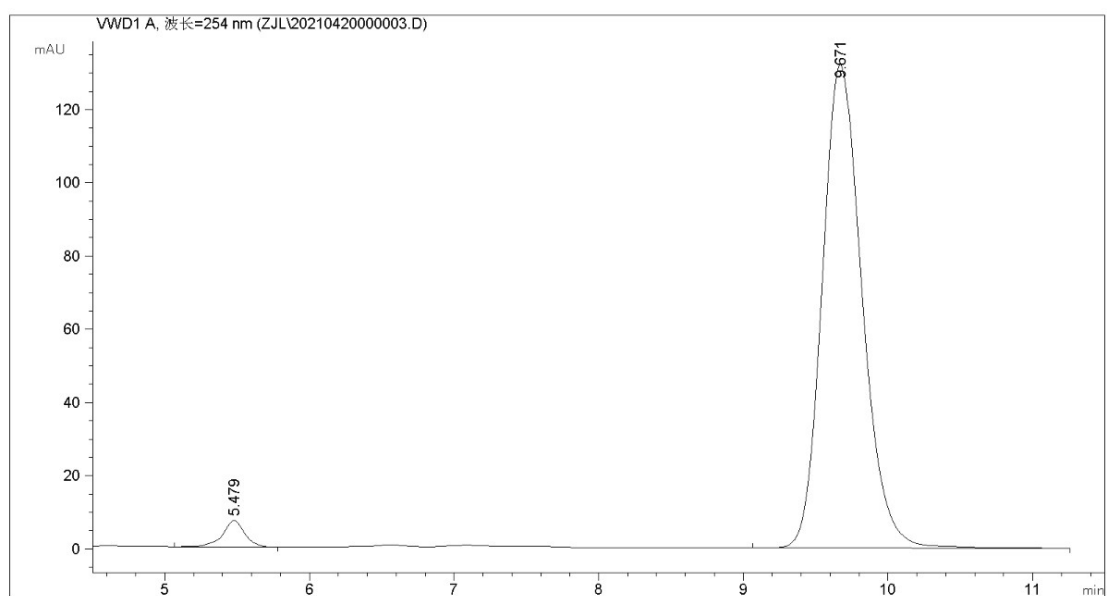
#	[min]		[min]	mAU	*s	[mAU]	%
1	8.571	BB	0.2342	2762.84277		181.69319	99.0488
2	9.692	MM	0.2969	26.53369		1.48951	0.9512



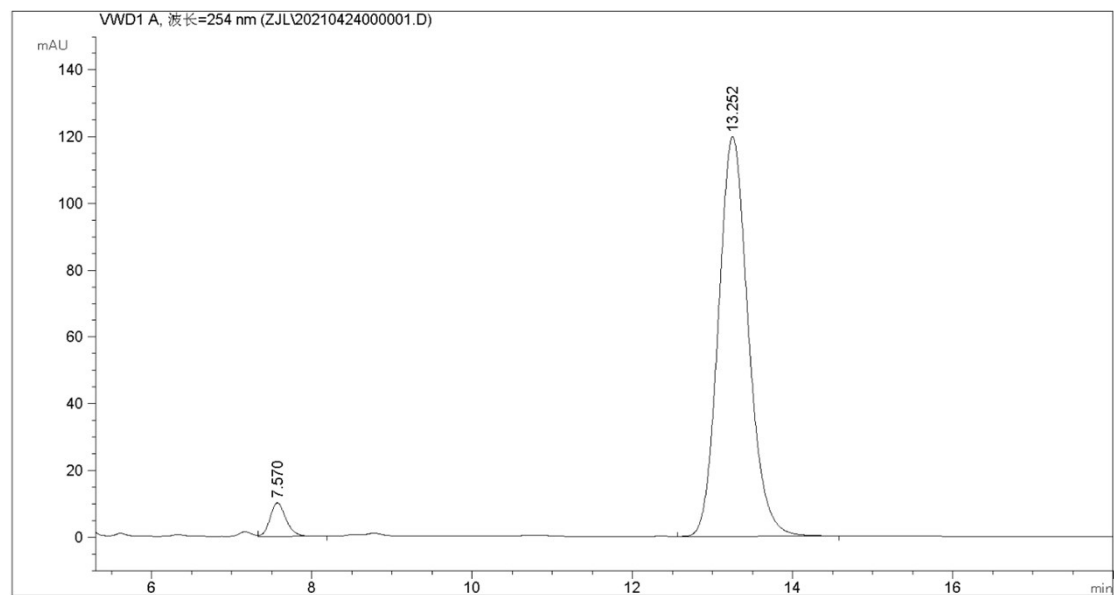
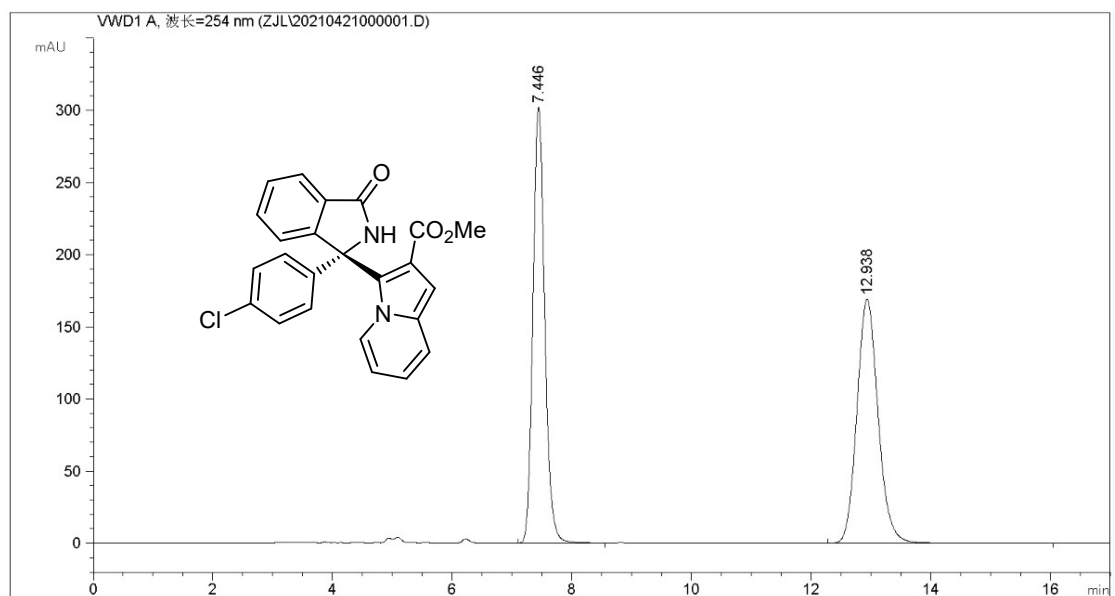


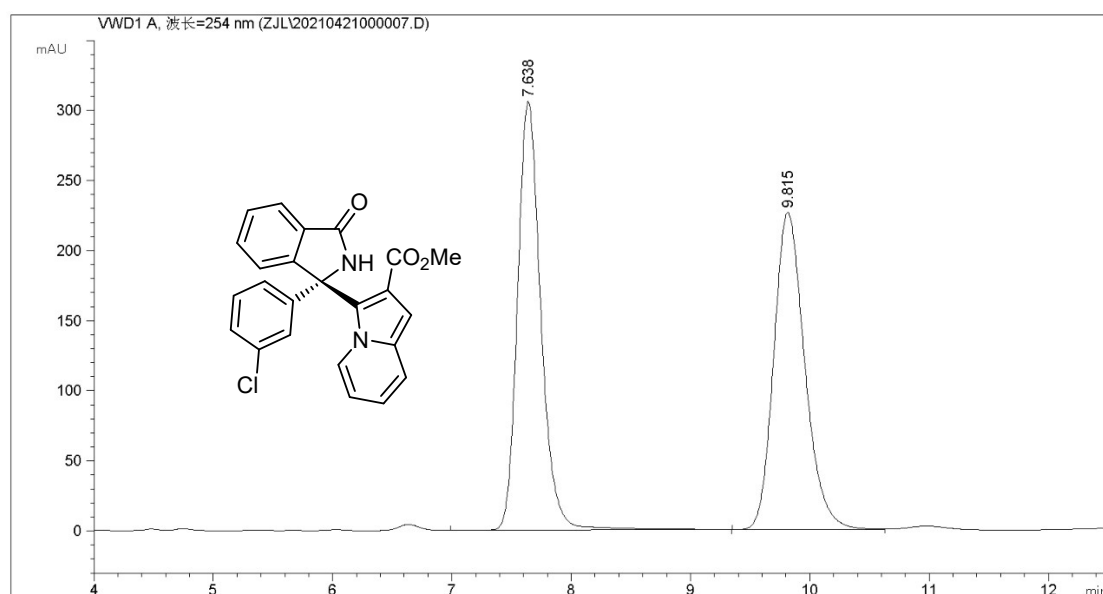


#	[min]		[min]	mAU	*s	[mAU]	%
1	5.552	BV	0.1776	2078.40649		181.06479	49.8345
2	9.766	BBA	0.2993	2092.21387		107.78885	50.1655

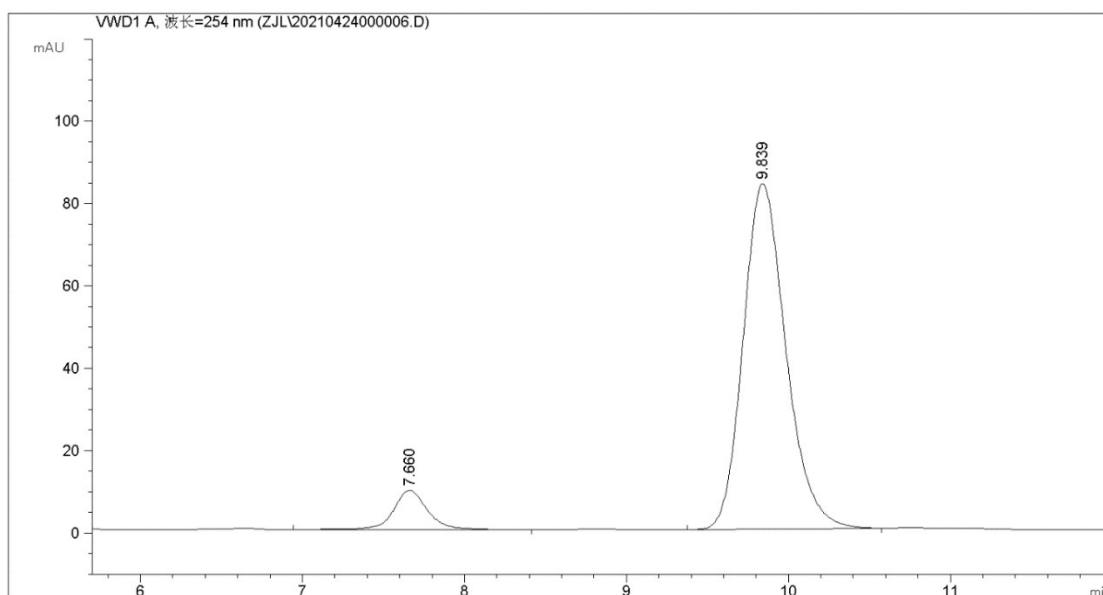


#	[min]		[min]	mAU	*s	[mAU]	%
1	5.479	BB	0.1613	79.69964		7.27233	3.0656
2	9.671	BBA	0.2927	2520.07837		131.98145	96.9344

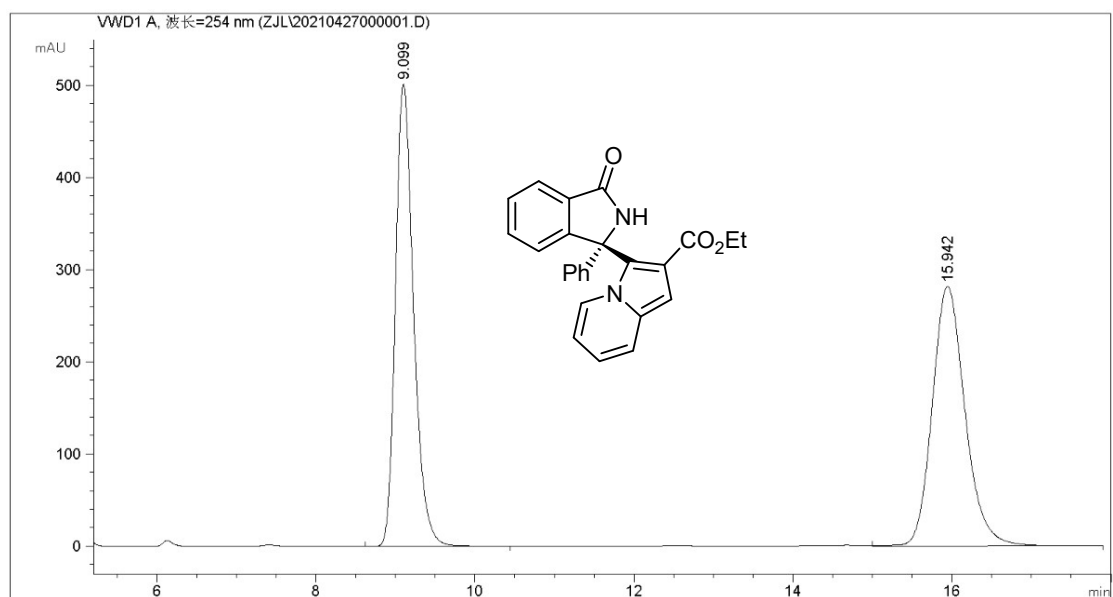




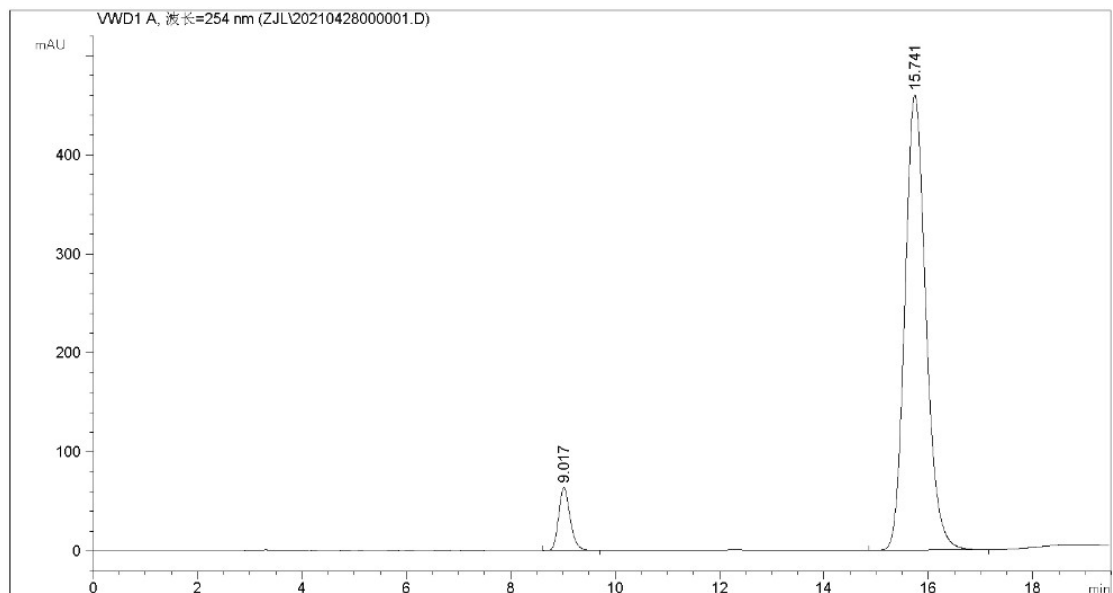
#	[min]		[min]	mAU	*s	[mAU]	%
1	7.638	BB	0.2081	4159.29834		305.87772	50.3478
2	9.815	BV	0.2784	4101.83203		226.35027	49.6522



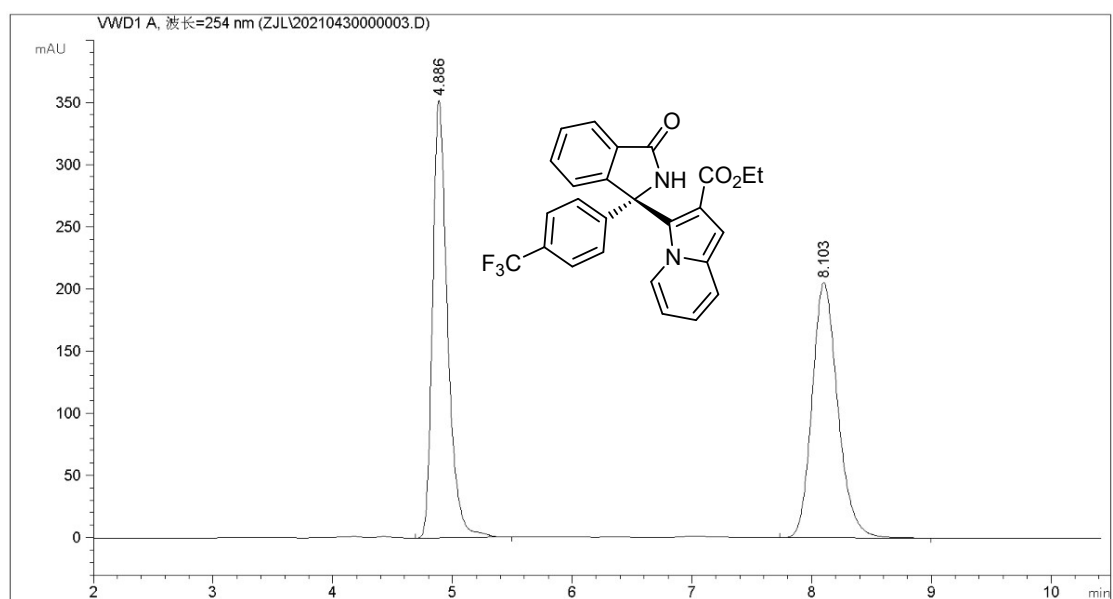
#	[min]		[min]	mAU	*s	[mAU]	%
1	7.660	BB	0.2232	143.88104		9.58637	8.5500
2	9.839	BB	0.2825	1538.93970		83.86980	91.4500



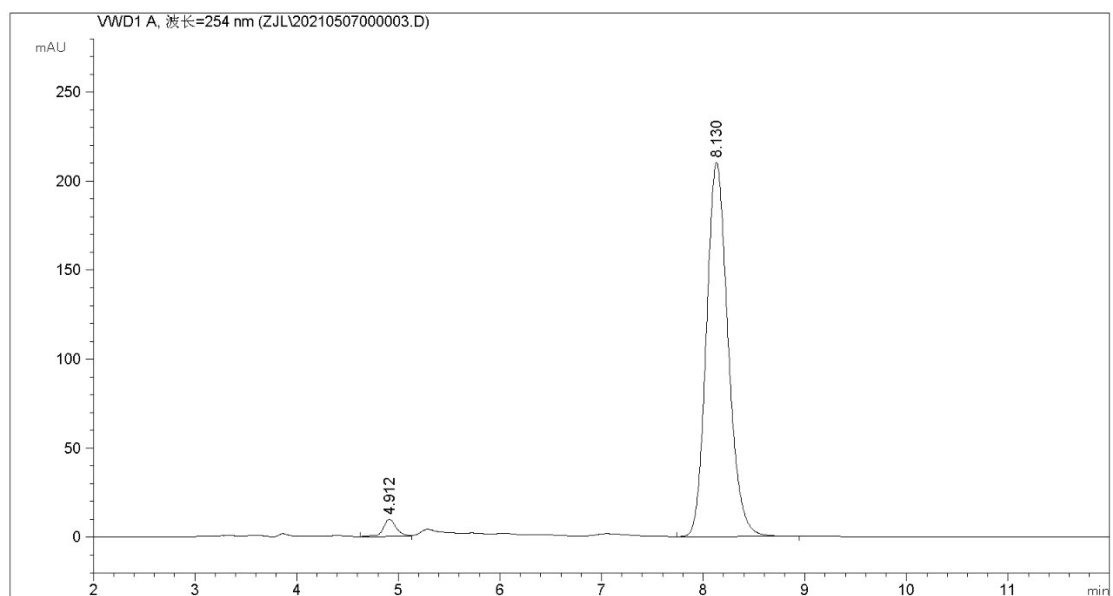
#	[min]		[min]	mAU	*s	[mAU]	%
1	9.099	BB	0.2429	7932.20752		501.04297	49.5839
2	15.942	VBA	0.4404	8065.34717		281.62671	50.4161



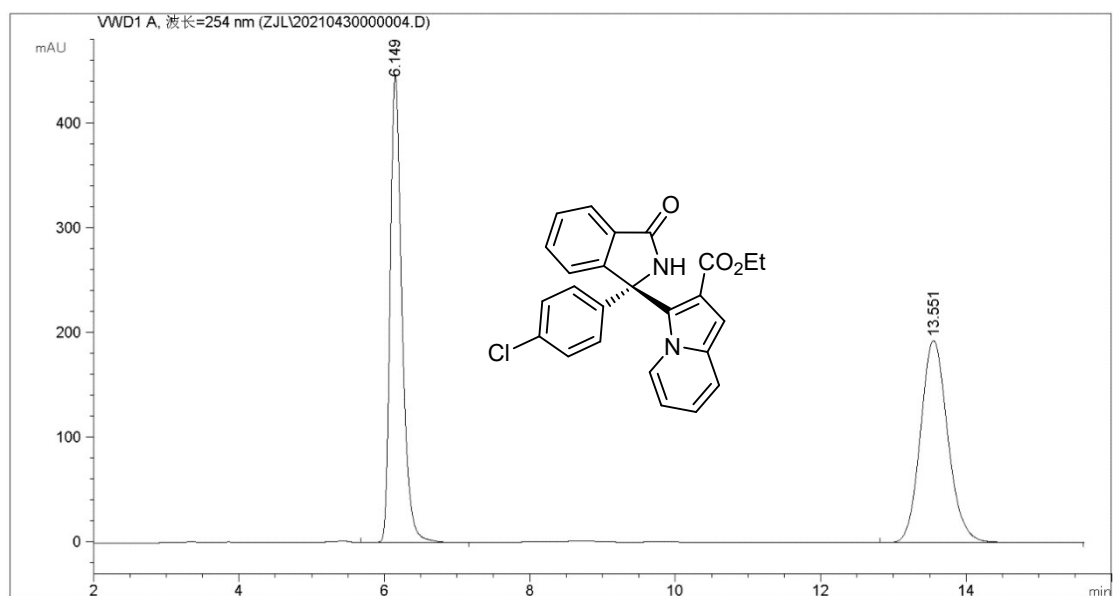
#	[min]		[min]	mAU	*s	[mAU]	%
1	9.017	BB	0.2362	977.97791		63.58527	7.0860
2	15.741	BB	0.4307	1.28236e4		459.14005	92.9140



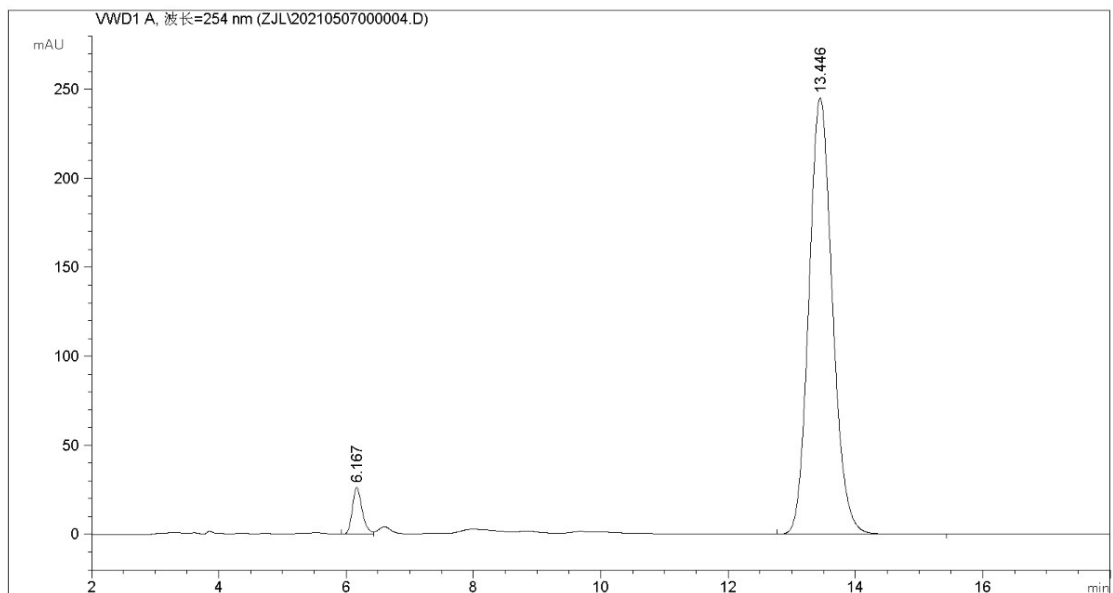
#	[min]		[min]	mAU	*s	[mAU]	%
1	4.886	BB	0.1306	3056.93433		351.46359	49.9732
2	8.103	VB	0.2295	3060.21631		204.99005	50.0268



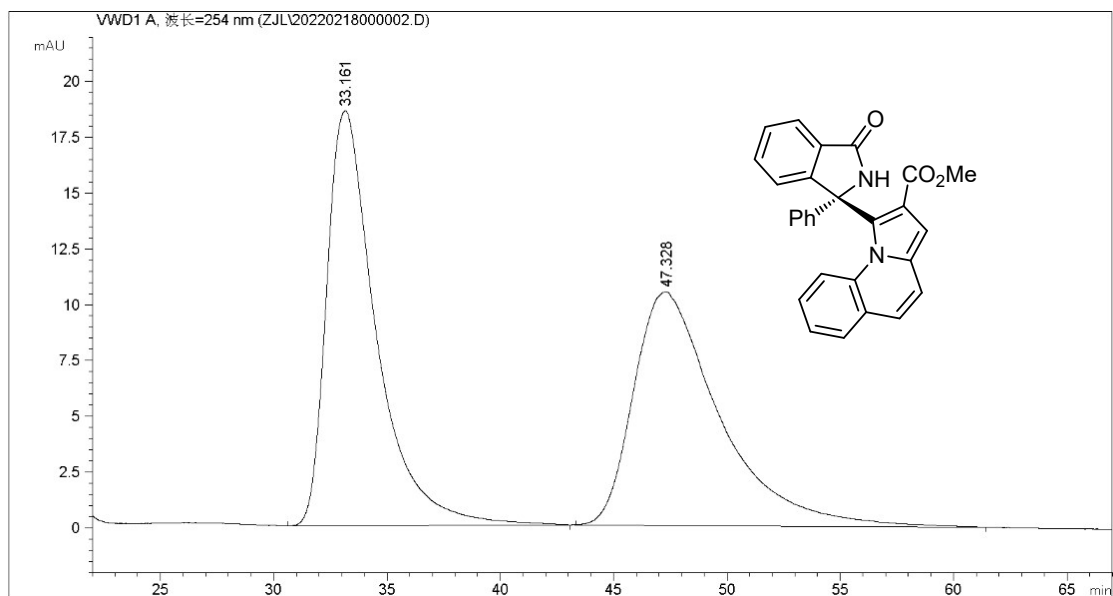
#	[min]		[min]	mAU	*s	[mAU]	%
1	4.912	BV	0.1318	83.21060		9.58670	2.6059
2	8.130	BB	0.2266	3109.95288		210.05093	97.3941



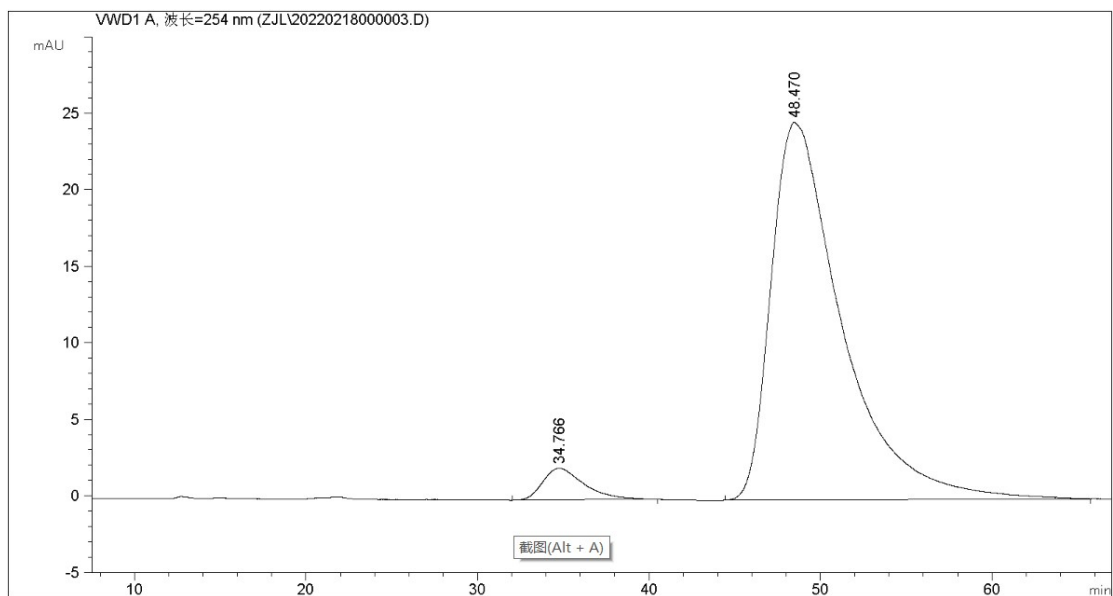
#	[min]		[min]	mAU	*s	[mAU]	%
1	6.149	VB	0.1699	4986.59082		445.57922	49.7901
2	13.551	BBA	0.4033	5028.63525		192.83023	50.2099



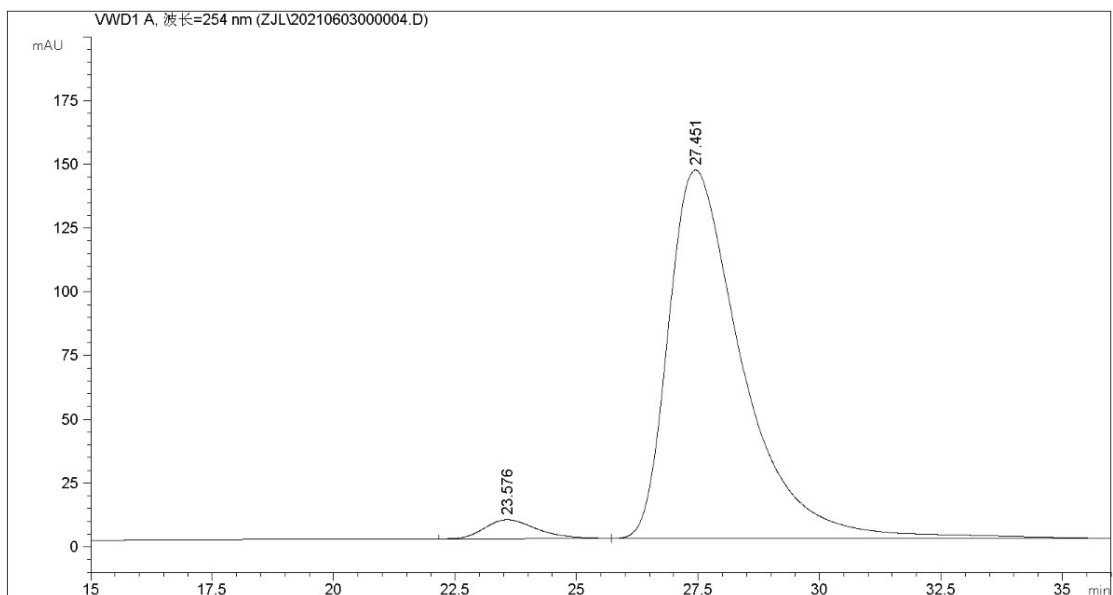
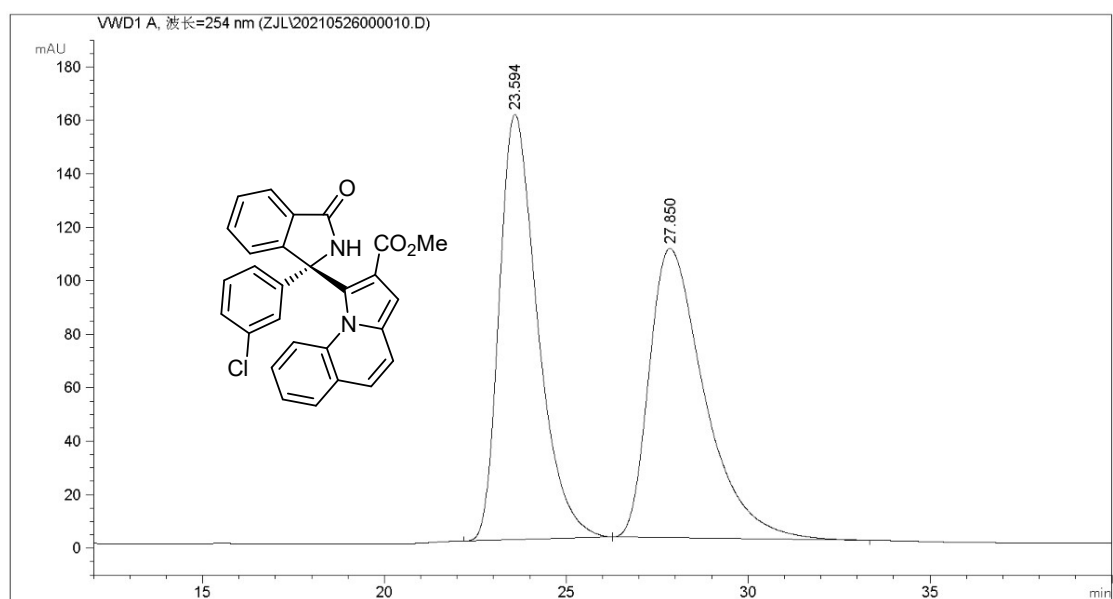
#	[min]		[min]	mAU	*s	[mAU]	%
1	6.167	BV	0.1626	279.25687		26.12478	4.2521
2	13.446	BB	0.3968	6288.31006		245.14624	95.7479

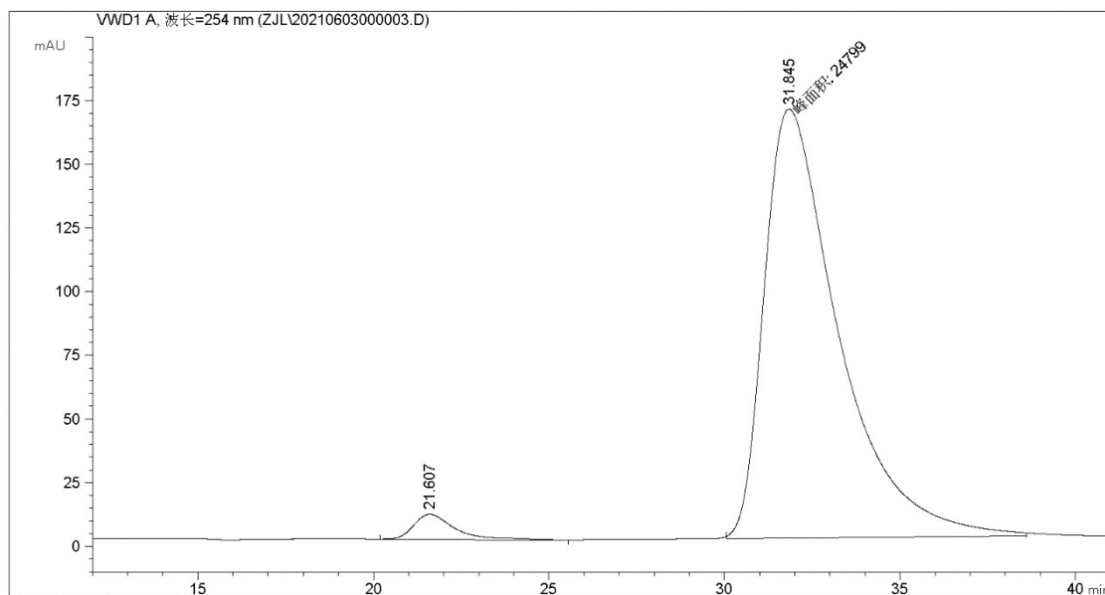
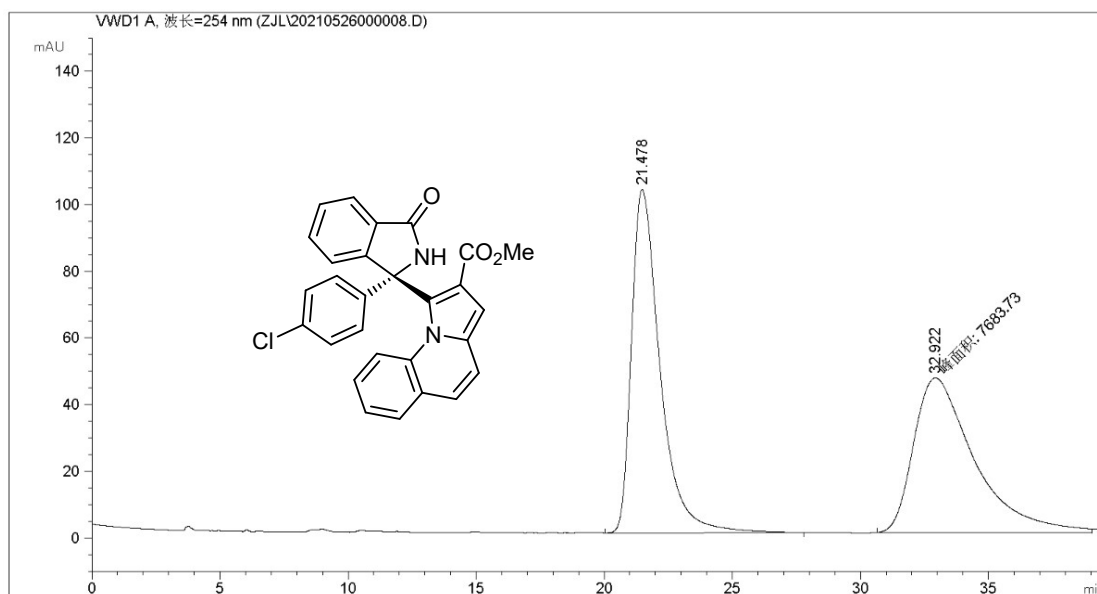


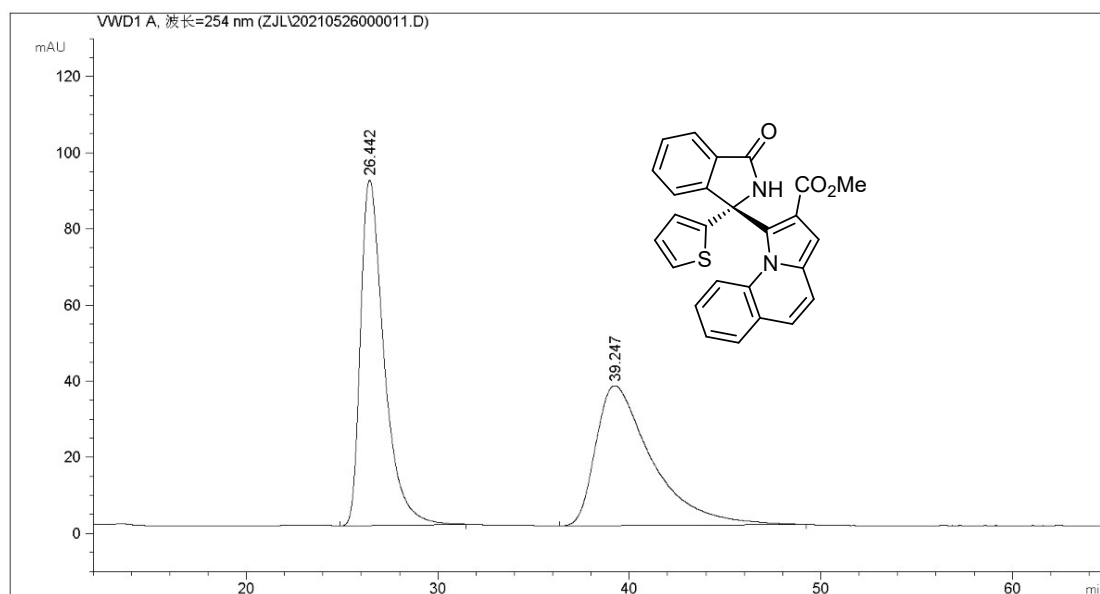
#	[min]		[min]	mAU	*s	[mAU]	%
1	33.161	BB	2.1936	2830.71313		18.61337	50.5632
2	47.328	BB	3.1382	2767.65625		10.47377	49.4368



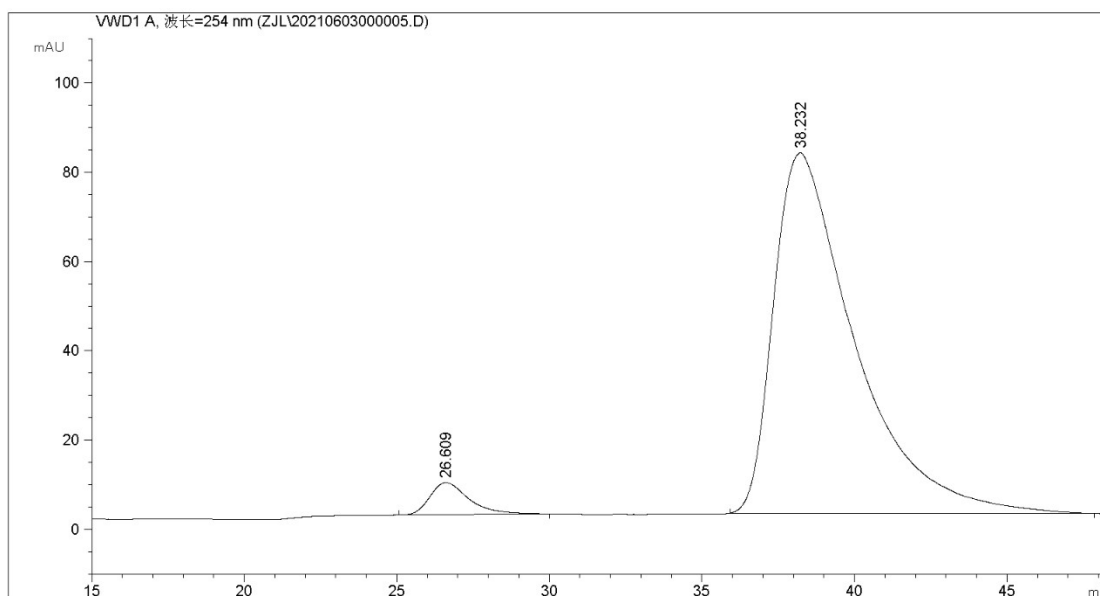
#	[min]		[min]	mAU	*s	[mAU]	%
1	34.766	BB	1.9806	348.20053		2.06934	4.6804
2	48.470	BB	3.7377	7091.32373		24.67168	95.3196







#	[min]	[min]	mAU	*s	[mAU]	%
1	26.442	BB	1.2877	7738.06934	90.72737	50.8038
2	39.247	BB	2.3954	7493.19922	36.71794	49.1962



#	[min]	[min]	mAU	*s	[mAU]	%
1	26.609	BB	1.1658	640.45844	7.18032	4.1116
2	38.232	BB	2.3671	1.49362e4	80.76825	95.8884

