

Supporting Information

Chiral (phosphine)-(imidazoline) PCN pincer palladium(II) complexes: synthesis and application in asymmetric hydroporphosphination of 2-alkenoylpyridines with diphenylphosphine

Jun-Jian Huang, Xiao-Qi Zhang, Jing-Jing Yang, Jun-Fang Gong* and Mao-Ping Song*

*College of Chemistry, Green Catalysis Center, Zhengzhou University, Zhengzhou 450001,
China*

E-mail: gongjf@zzu.edu.cn (J.-F. Gong) or mpsong@zzu.edu.cn (M.-P. Song).

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X-ray diffraction studies of complexes **3c, **3e**, **3i**, and **3k**.**

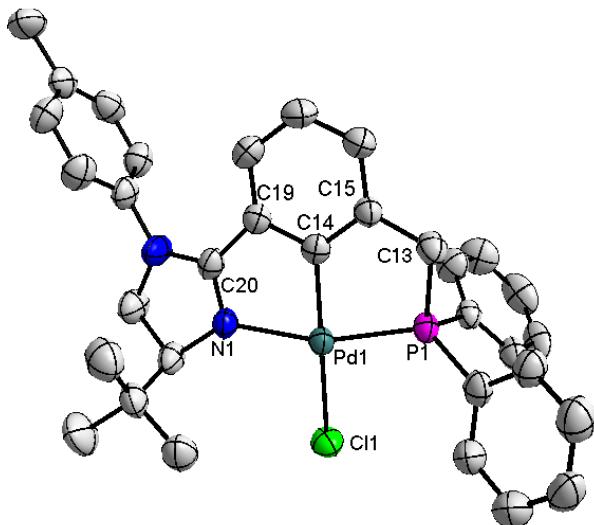


Figure S1. Molecular structure of complex **3c** with ellipsoids drawn at the 50% probability level (hydrogen atoms are omitted for clarity). Selected bond lengths (\AA) and angles (deg): Pd(1)–C(14) 1.980(5), Pd(1)–P(1) 2.2308(12), Pd(1)–N(1) 2.112(4), Pd(1)–Cl(1) 2.3964(14); C(14)–Pd(1)–P(1) 82.30(15), C(14)–Pd(1)–N(1) 79.73(18), C(14)–Pd(1)–Cl(1) 172.70(15), N(1)–Pd(1)–P(1) 162.01(12), P(1)–Pd(1)–Cl(1) 99.26(5), N(1)–Pd(1)–Cl(1) 98.47(12).

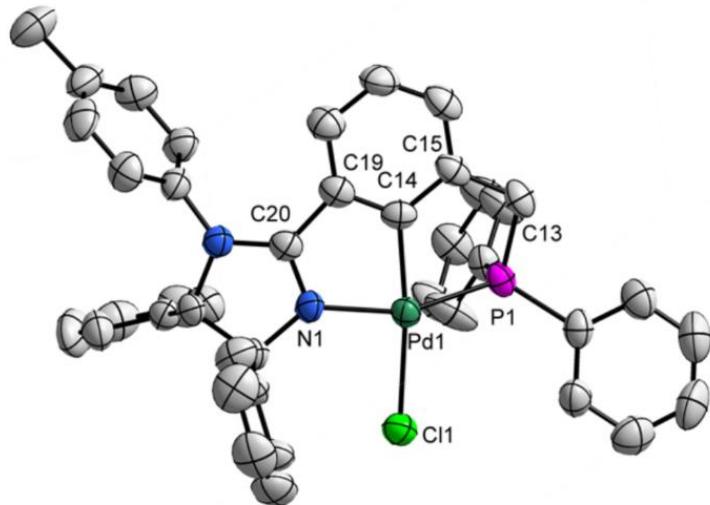


Figure S2. Molecular structure of complex **3e** with ellipsoids drawn at the 50% probability level (hydrogen atoms are omitted for clarity; one of the two independent molecules shown). Selected bond lengths (\AA) and angles (deg): Pd(1)–C(14) 1.985(8), Pd(1)–P(1) 2.217(2), Pd(1)–N(1) 2.065(6), Pd(1)–Cl(1) 2.391(2); C(14)–Pd(1)–P(1) 81.1(3), C(14)–Pd(1)–N(1) 79.5(3), C(14)–Pd(1)–Cl(1) 173.0(3), N(1)–Pd(1)–P(1) 159.06(19), P(1)–Pd(1)–Cl(1) 104.10(8), N(1)–Pd(1)–Cl(1) 94.7(2).

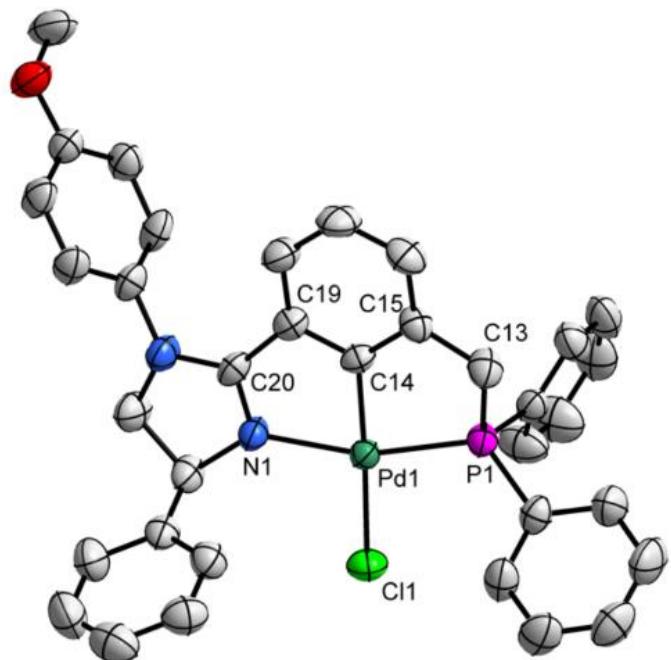


Figure S3. Molecular structure of complex **3i** with ellipsoids drawn at the 50% probability level (hydrogen atoms are omitted for clarity). Pd(1)–C(14) 1.9859(19), Pd(1)–P(1) 2.2175(13), Pd(1)–N(1) 2.082(4), Pd(1)–Cl(1) 2.3942(12); C(14)–Pd(1)–P(1) 82.12(9), C(14)–Pd(1)–N(1) 79.59(15), C(14)–Pd(1)–Cl(1) 174.37(9), N(1)–Pd(1)–P(1) 161.59(12), P(1)–Pd(1)–Cl(1) 101.02(5), N(1)–Pd(1)–Cl(1) 97.04(12).

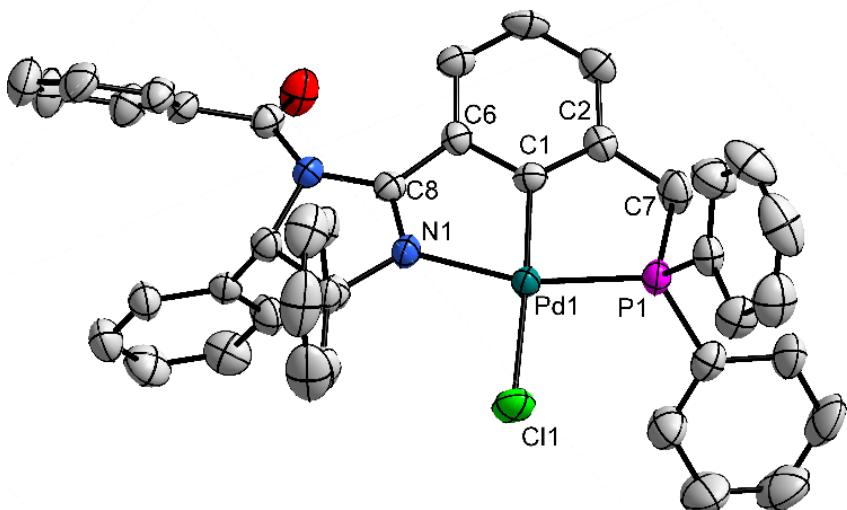
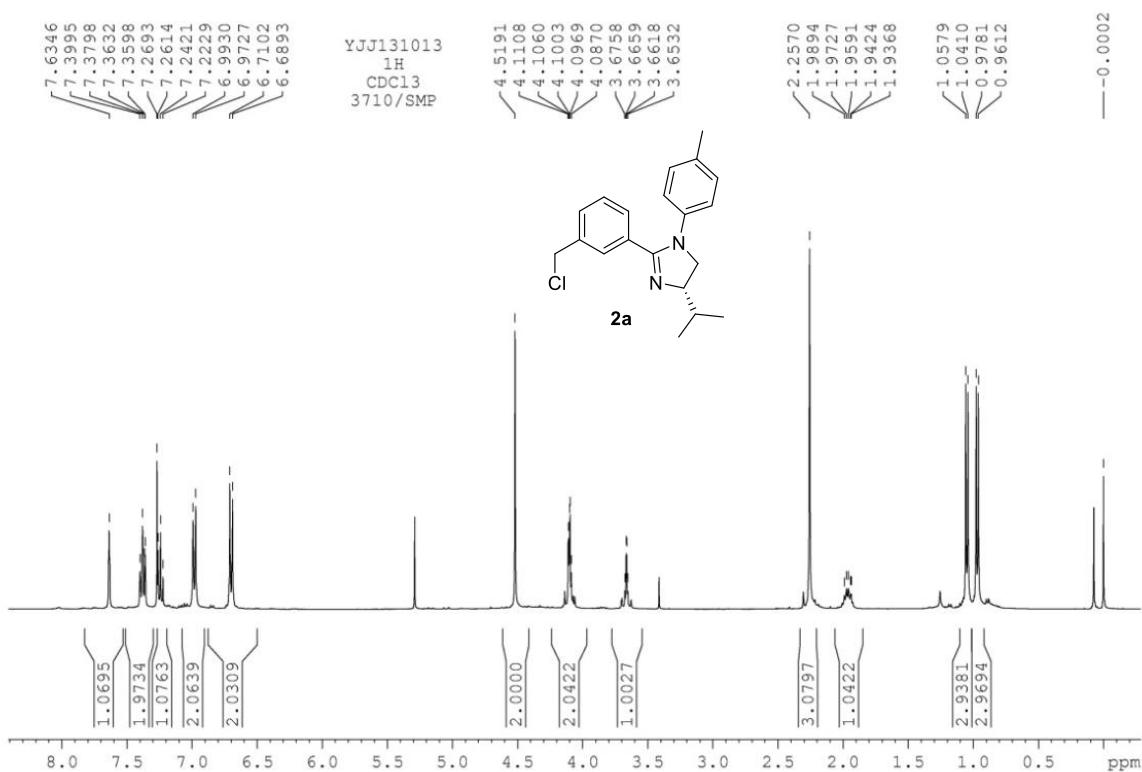


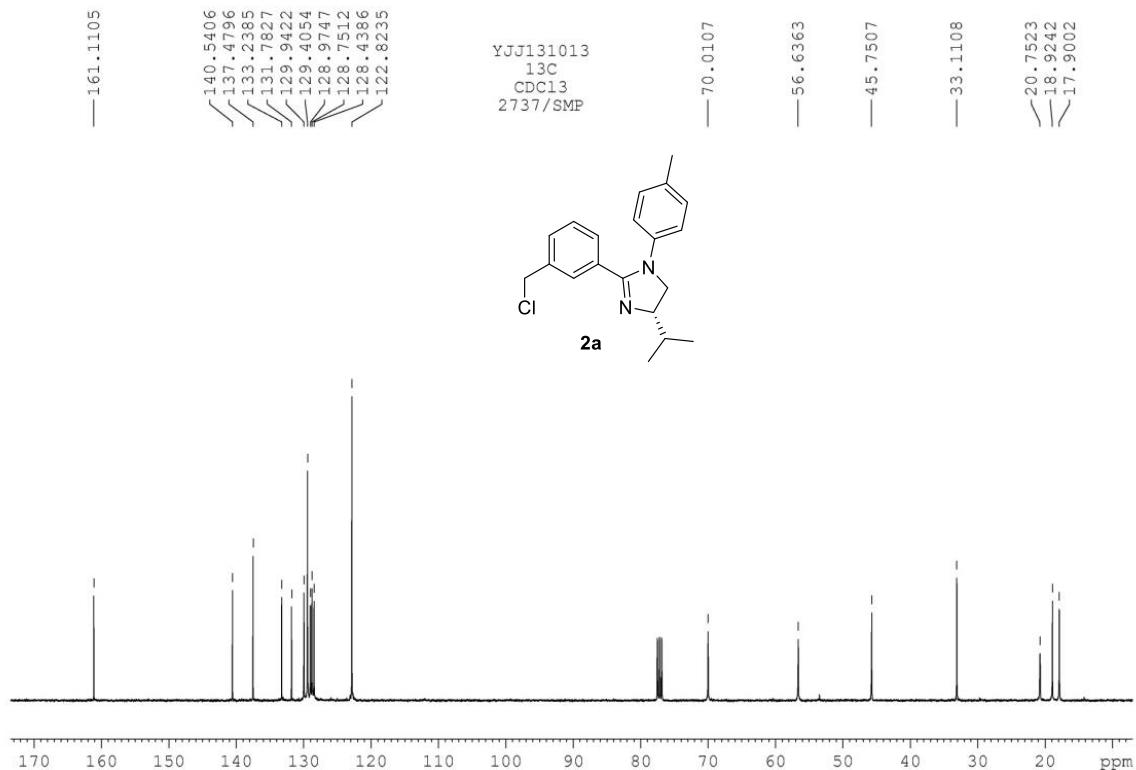
Figure S4. Molecular structure of complex **3k** with ellipsoids drawn at the 50% probability level. (hydrogen atoms are omitted for clarity) Selected bond lengths (\AA) and angles (deg): Pd(1)–C(1) 1.987(5), Pd(1)–P(1) 2.2159(14), Pd(1)–N(1) 2.082(4), Pd(1)–Cl(1) 2.3701(15); C(1)–Pd(1)–P(1) 83.25(16), C(1)–Pd(1)–N(1) 79.8(2), C(1)–Pd(1)–Cl(1) 176.90(16), N(1)–Pd(1)–P(1) 162.82(13), P(1)–Pd(1)–Cl(1) 99.84(5), N(1)–Pd(1)–Cl(1) 97.09(13).

Table S1. Summary of crystal structure determination for 3c, 3e, 3i and 3k.

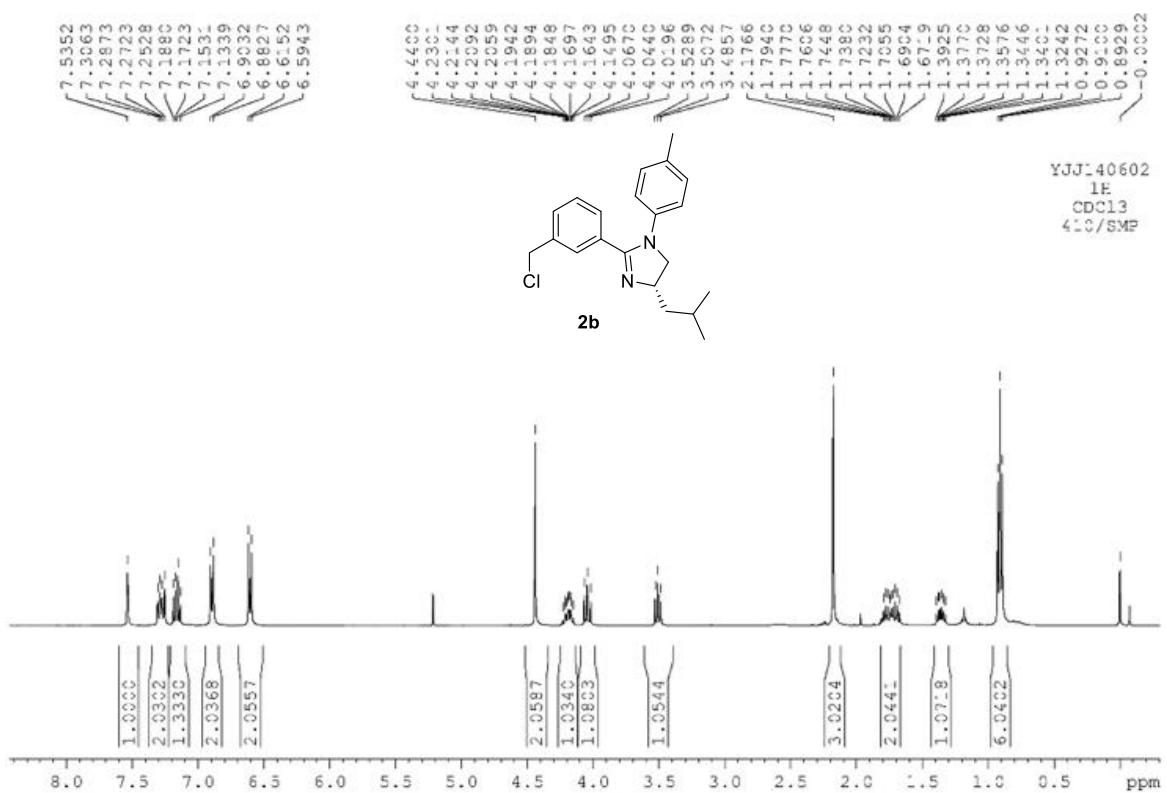
	3c	3e	3i	3k
formula	C ₃₃ H ₃₄ ClN ₂ PPd	C ₄₁ H ₃₄ ClN ₂ PPd	C ₃₅ H ₃₀ ClN ₂ OPPd	C ₄₁ H ₃₂ ClN ₂ OPPd
Mr	631.44	727.52	667.43	741.50
Temp[K]	291.15	293(2)	293(2)	293(2)
wavelength [Å]	0.71073	1.54184	1.54184	1.54184
Cryst system	monoclinic	monoclinic	orthorhombic	orthorhombic
Space group	P2 ₁	P2 ₁	P2 ₁ 2 ₁ 2 ₁	P2 ₁ 2 ₁ 2 ₁
a/Å	10.2862(5)	12.41683(19)	9.20754(8)	9.34262(12)
b/Å	12.1285(4)	23.3274(3)	14.47998(12)	14.76131(19)
c/Å	12.6954(5)	12.73072(19)	22.6523(2)	24.6818(3)
α/°	90	90	90	90
β/°	111.540(5)	113.1047(17)	90	90
γ/°	90	90	90	90
V [Å ³]	1473.22(11)	3391.70(9)	3020.12(5)	3403.85(8)
Z	2	4	4	4
ρ _{calc} g/cm ³	1.423	1.425	1.468	1.447
μ/mm ⁻¹	0.799	5.823	6.508	5.838
F(000)	648.0	1488.0	1360.0	1512.0
Crystal size/mm ³	0.22 × 0.2 × 0.18	0.13 × 0.1 × 0.08	0.13 × 0.1 × 0.09	0.23 × 0.171 × 0.119
θ range [deg]	6.39 to 52.728	7.55 to 141.9	7.246 to 134.144	7.164 to 134.15
Index ranges	-12 ≤ h ≤ 12 -15 ≤ k ≤ 13 -15 ≤ l ≤ 12	-15 ≤ h ≤ 14 -27 ≤ k ≤ 28 -15 ≤ l ≤ 15	11 ≤ h ≤ 9 -17 ≤ k ≤ 17 27 ≤ l ≤ 26	-10 ≤ h ≤ 11 -11 ≤ k ≤ 17 -29 ≤ l ≤ 25
no. of data collected	6698	32816	22452	13142
no. of unique data	5022	12231	5374	6055
Final R indexes	R ₁ = 0.0324	R ₁ = 0.0452	R ₁ = 0.0279	R ₁ = 0.0320
[I>=2σ (I)]	wR ₂ = 0.0575	wR ₂ = 0.1138	wR ₂ = 0.0694	wR ₂ = 0.0684
Final R indexes [all data]	R ₁ = 0.0380	R ₁ = 0.0491	R ₁ = 0.0302	R ₁ = 0.0382
peak/hole [e Å ⁻³]	0.55/-0.29	0.86/-0.58	0.33/-0.35	0.30/-0.41
Flack parameter	-0.03(2)	-0.034(7)	-0.030(5)	-0.024(6)
CCDC number	2154126	2154135	2154138	2154139



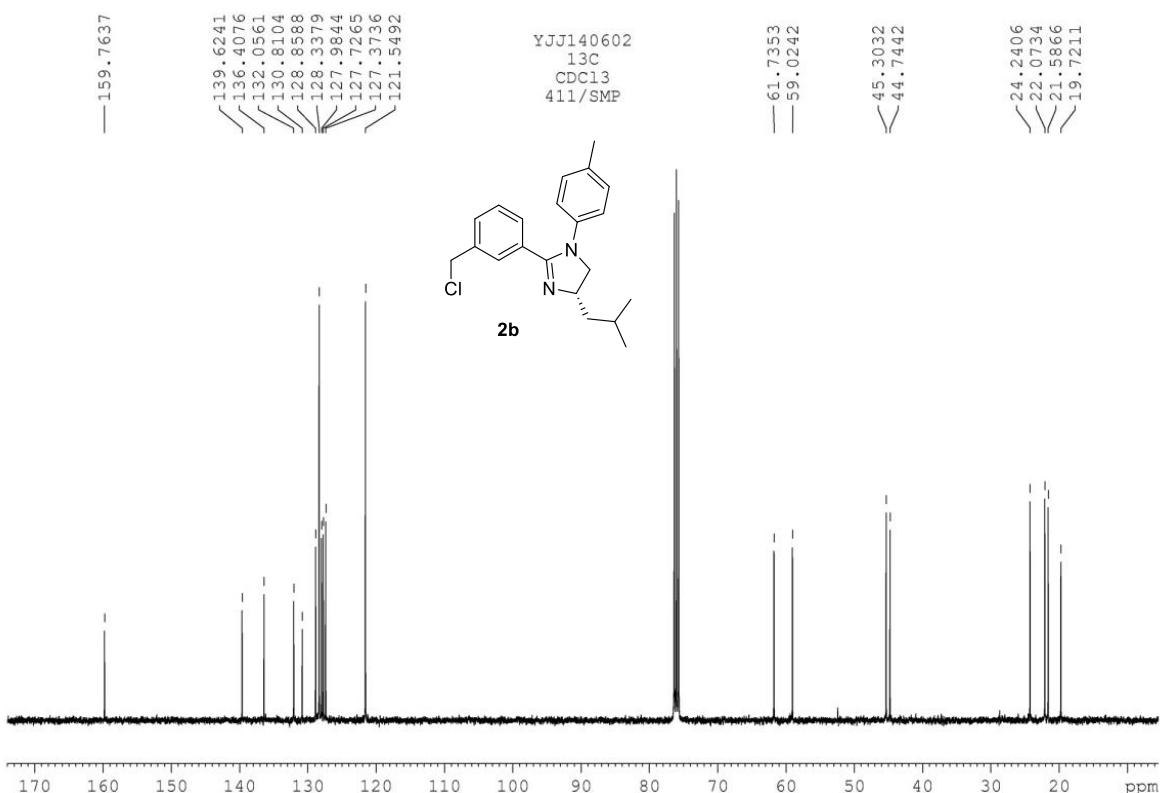
¹H NMR spectrum of **2a** (400 MHz, CDCl₃)



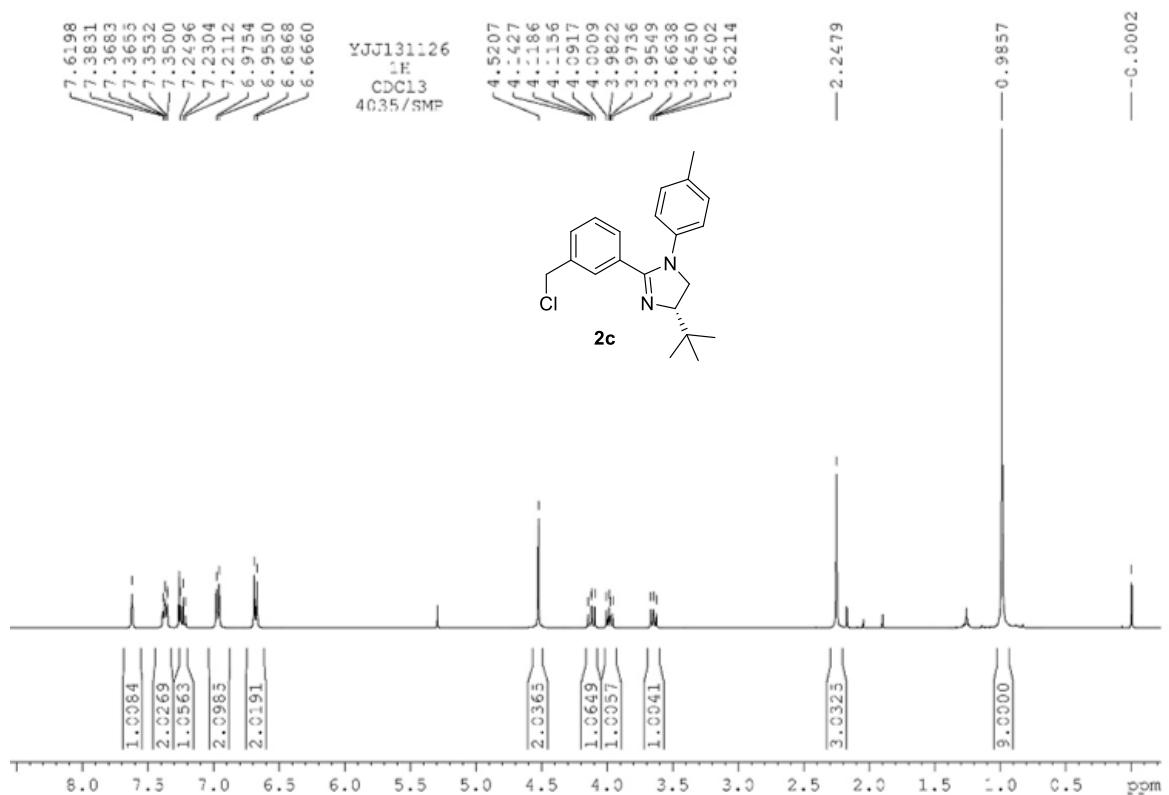
¹³C{¹H} NMR spectrum of **2a** (100 MHz, CDCl₃)



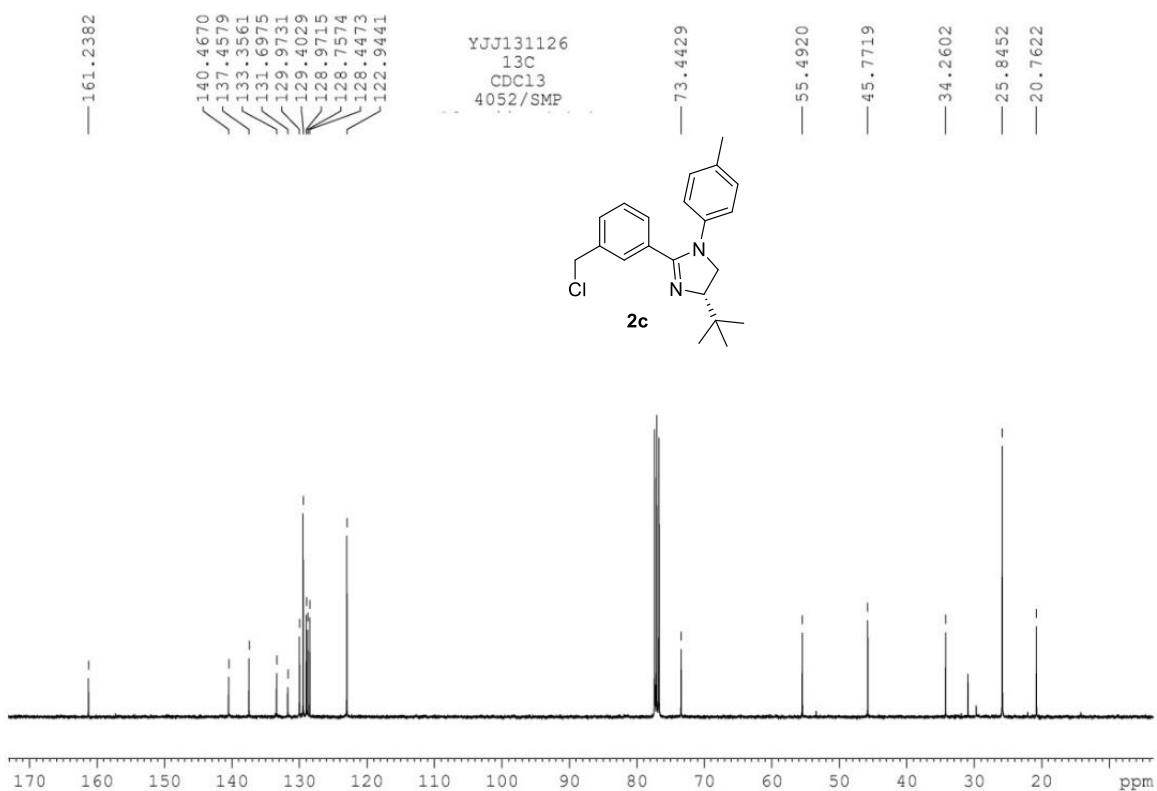
¹H NMR spectrum of **2b** (400 MHz, CDCl₃)



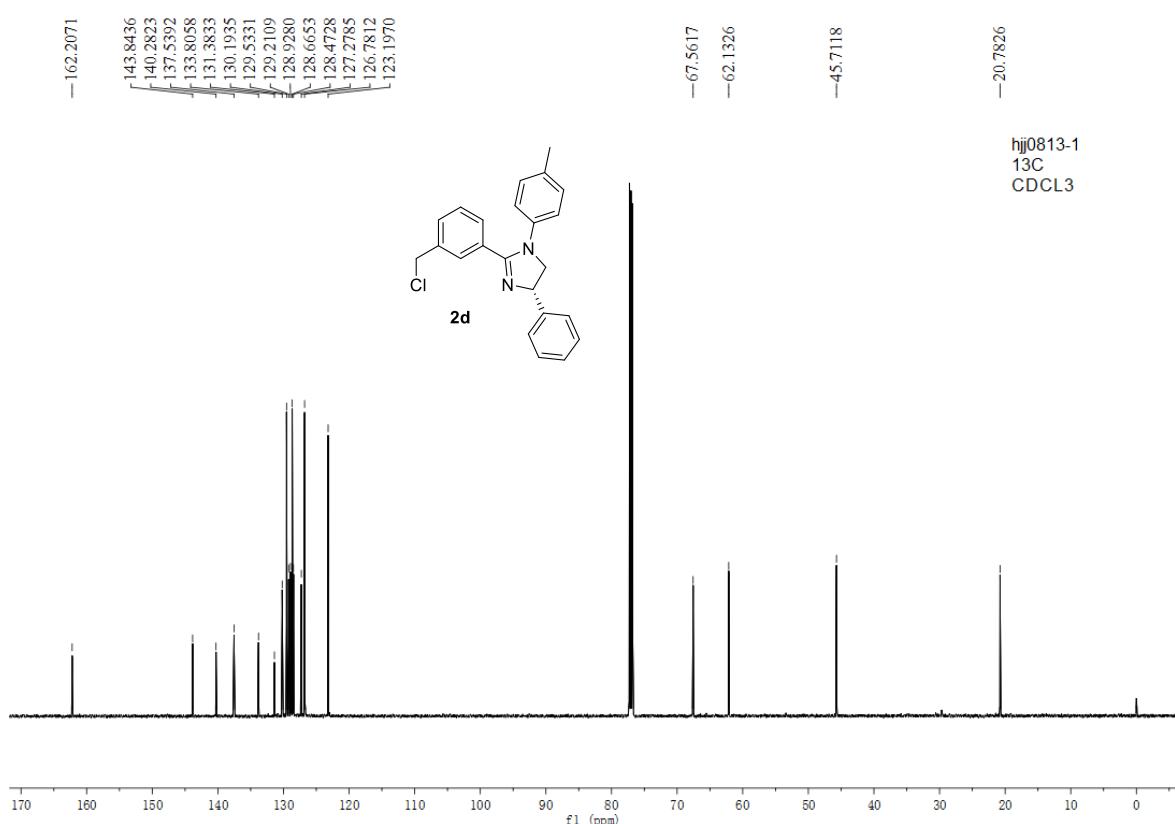
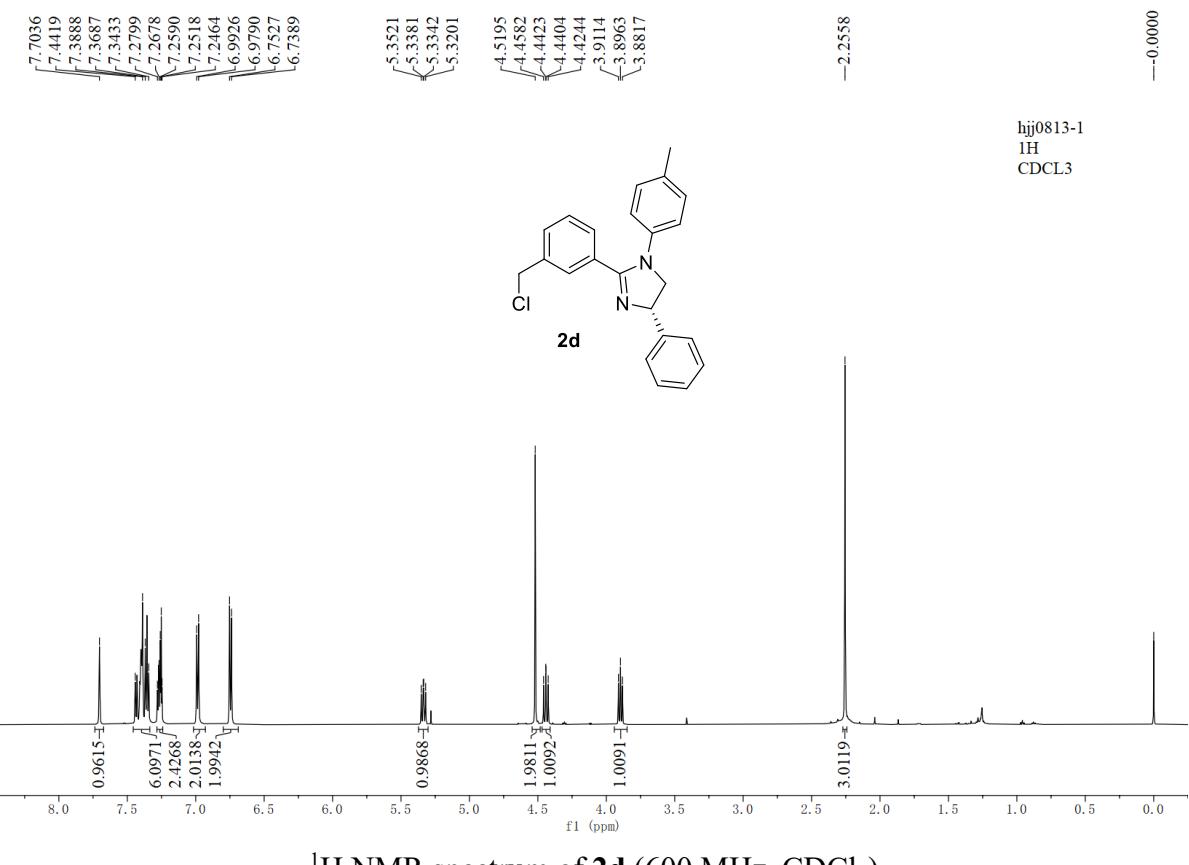
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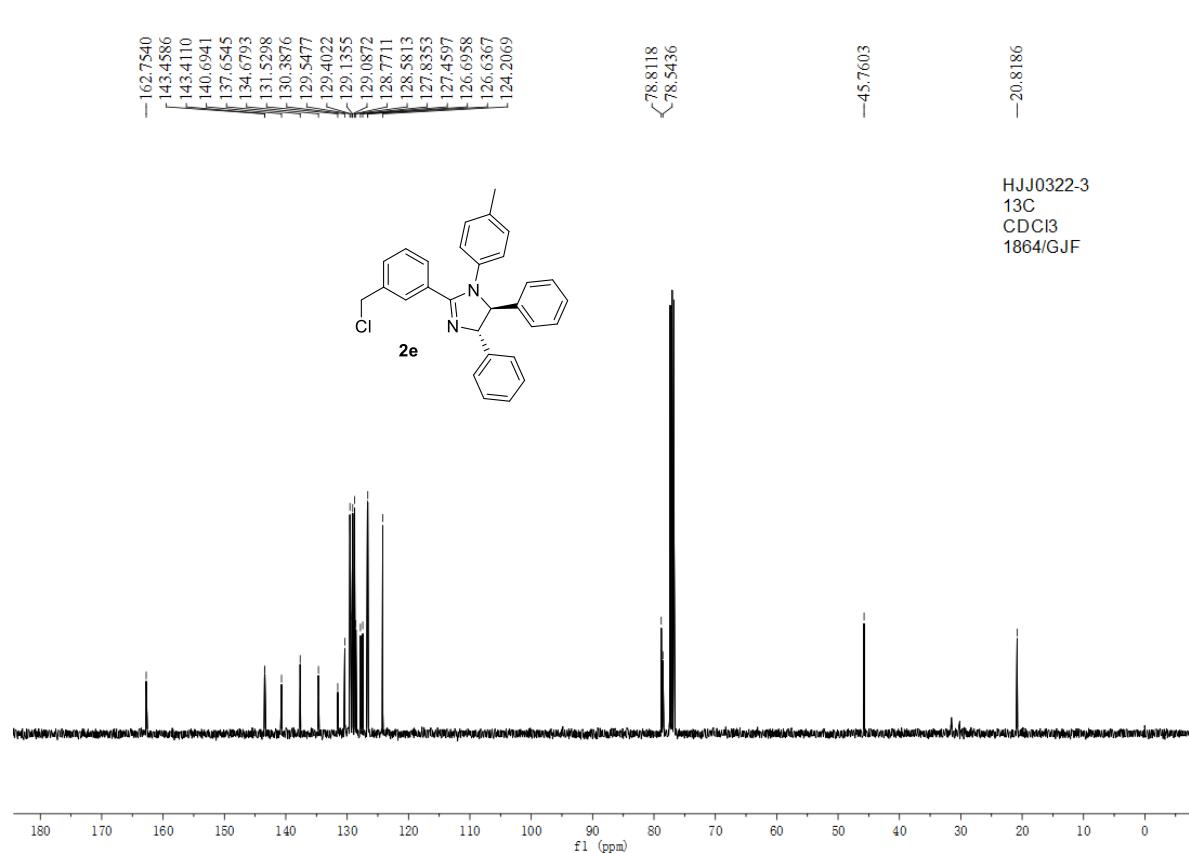
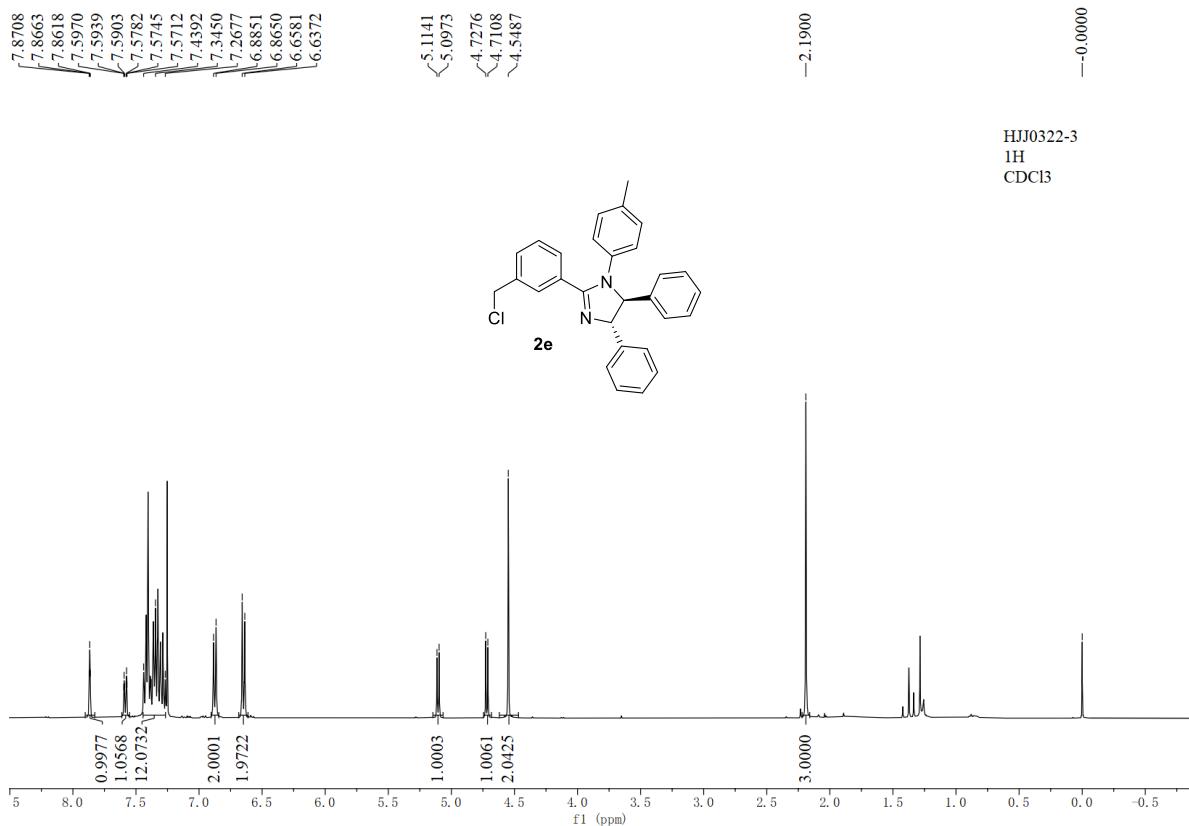


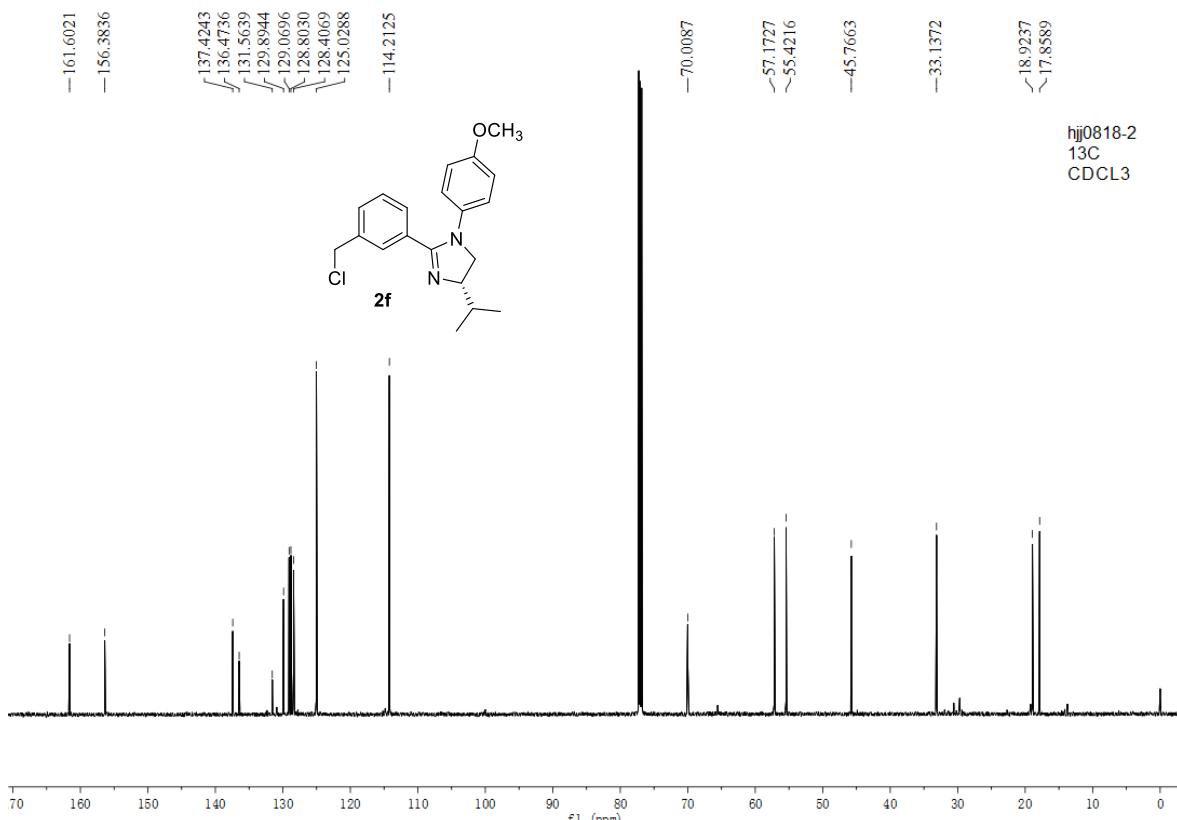
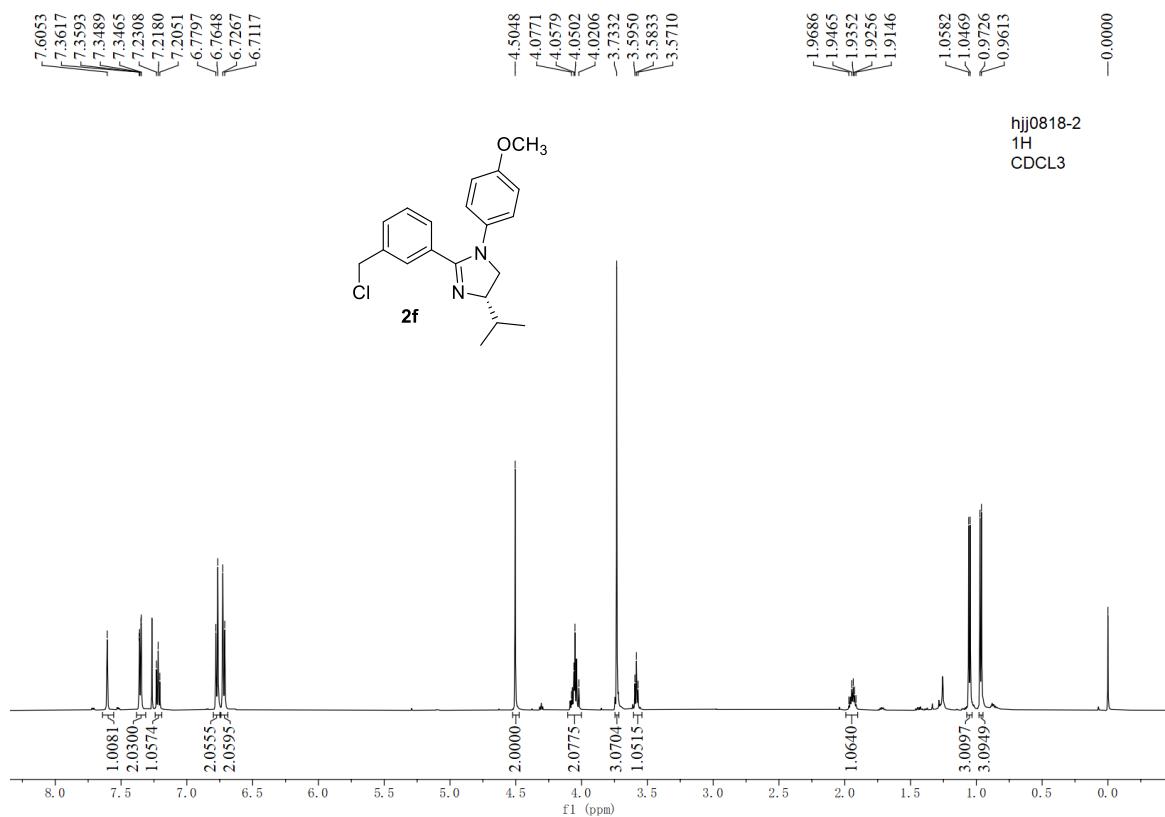
¹H NMR spectrum of **2c** (400 MHz, CDCl₃)

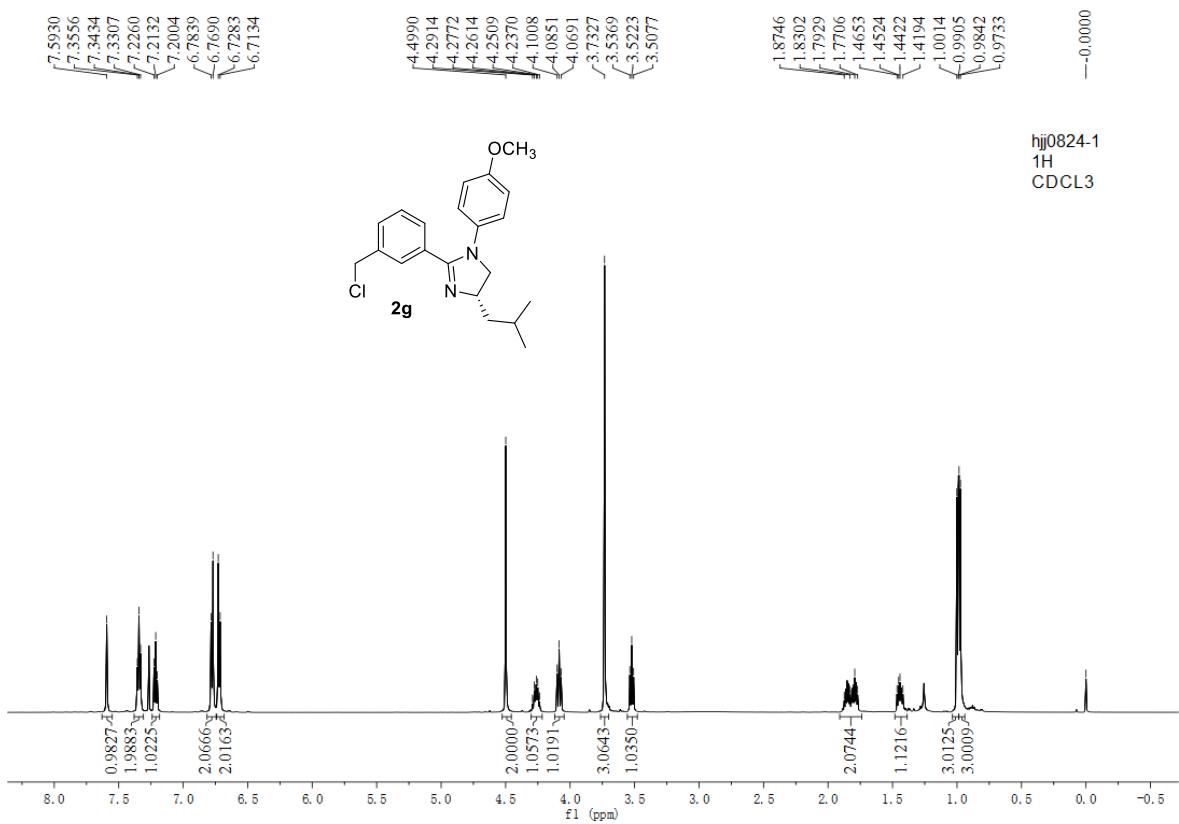


¹³C{¹H} NMR spectrum of **2c** (100 MHz, CDCl₃)

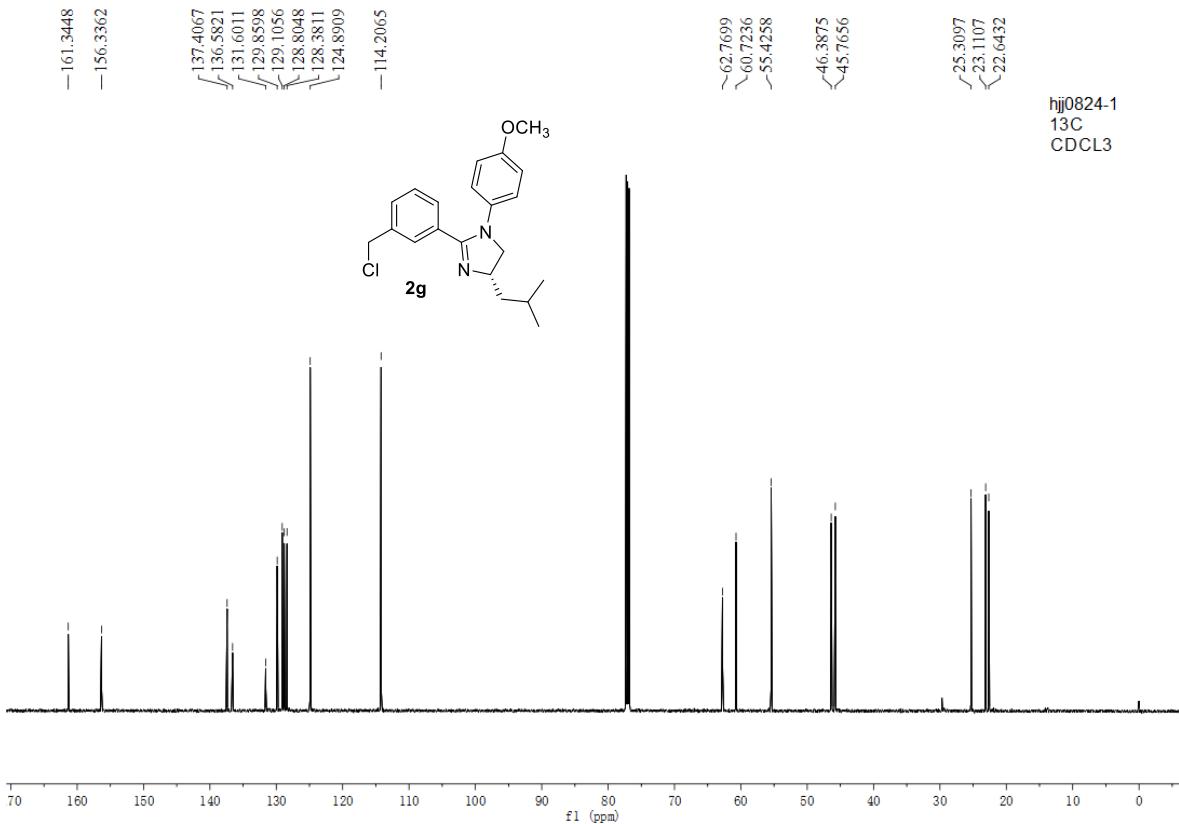




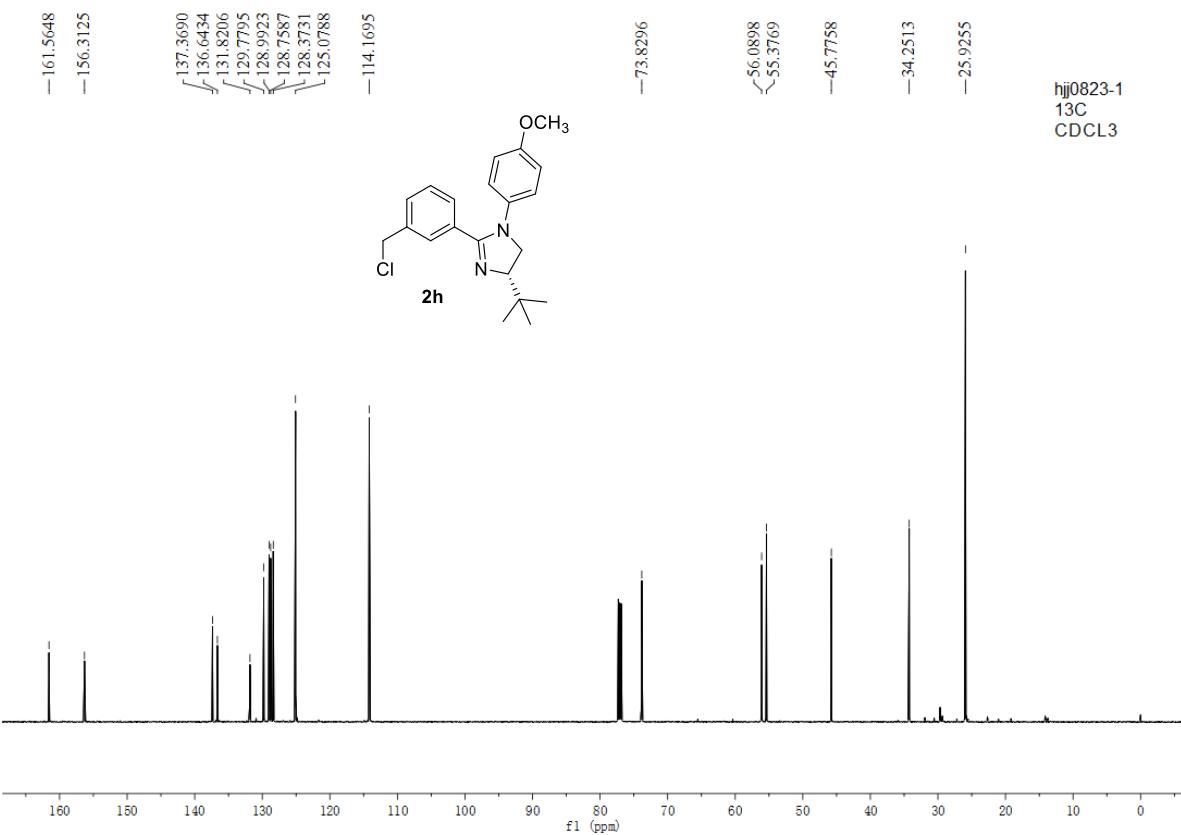
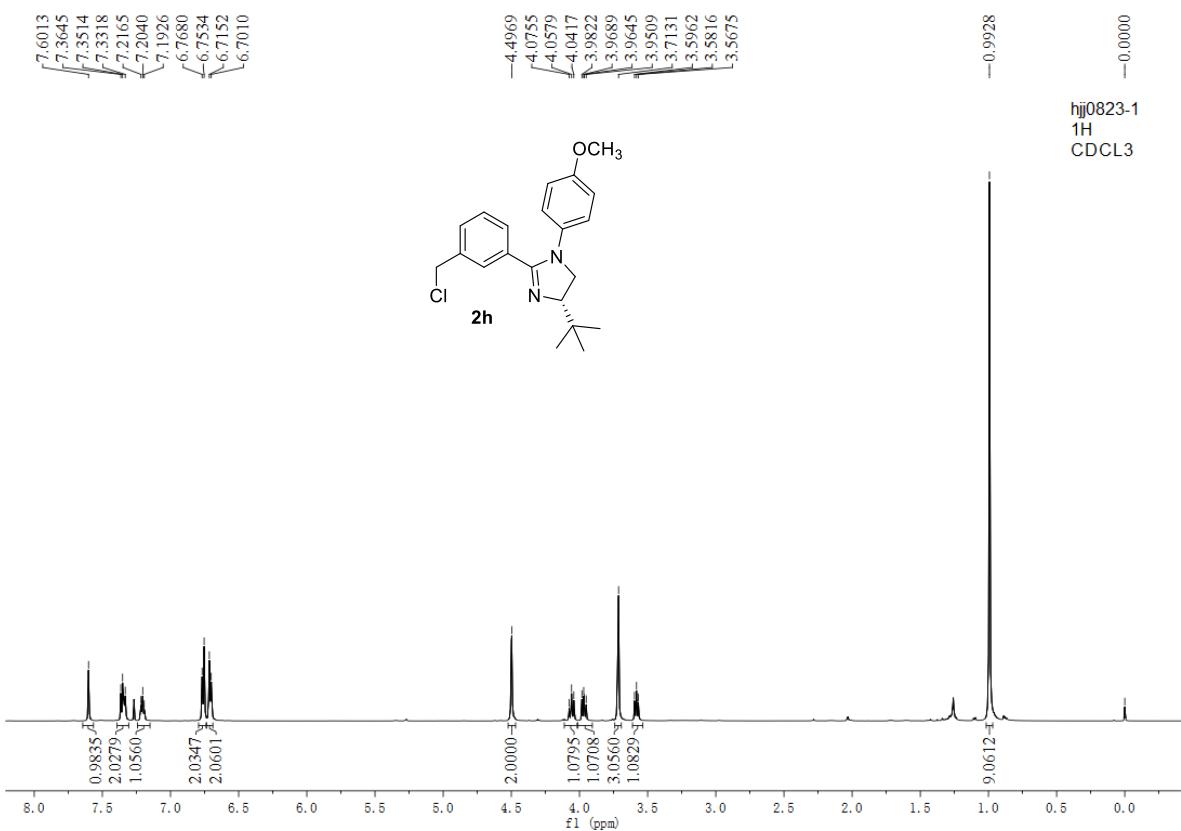




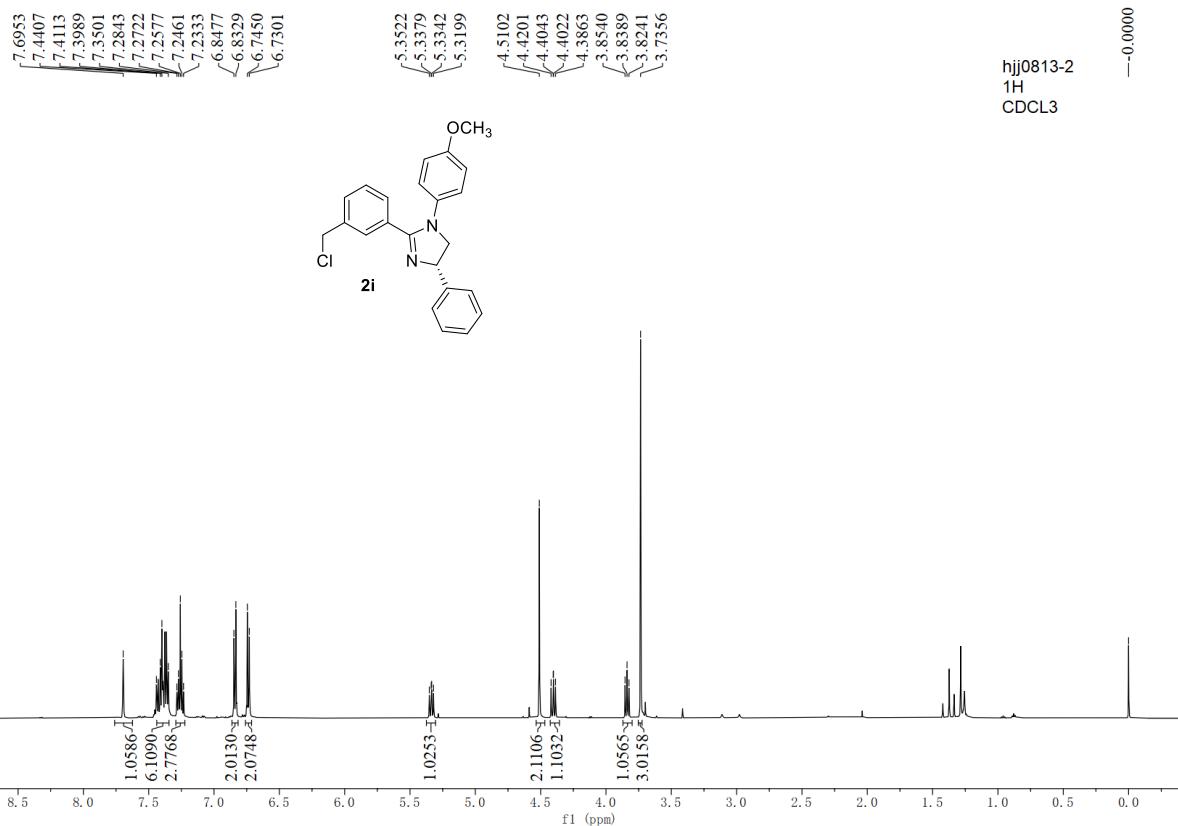
¹H NMR spectrum of **2g** (600 MHz, CDCl₃)



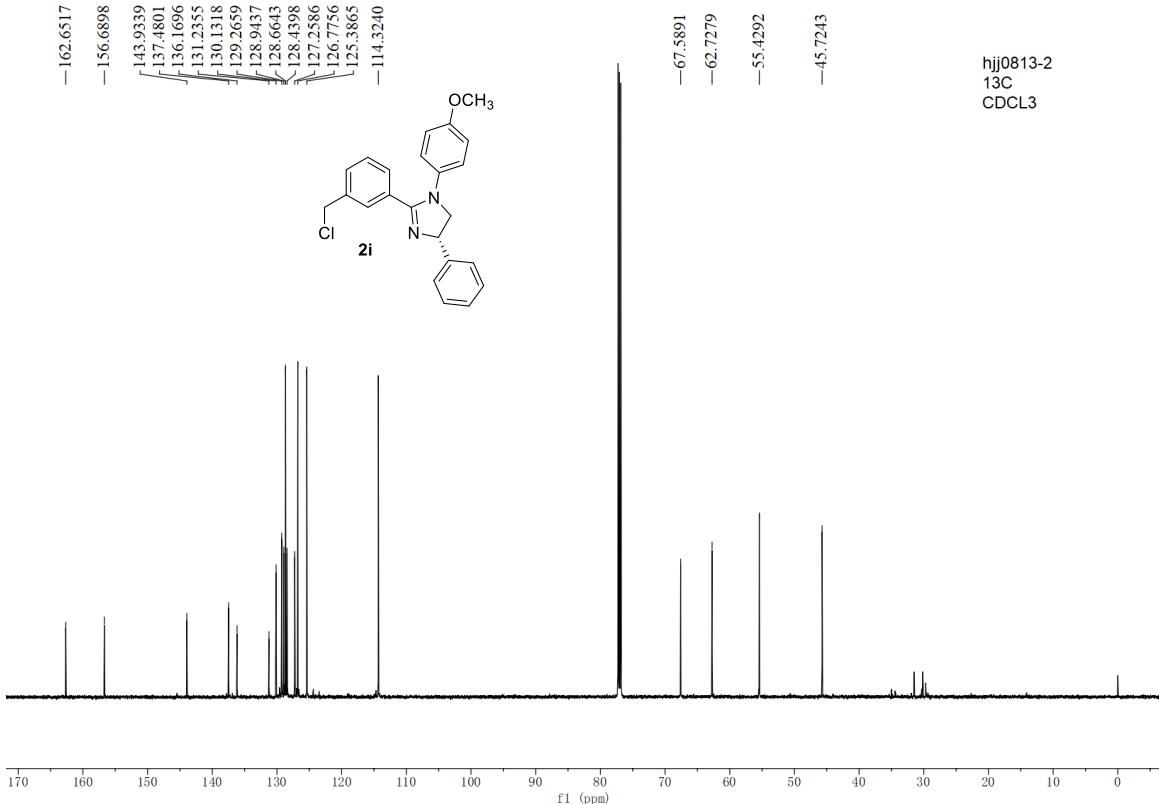
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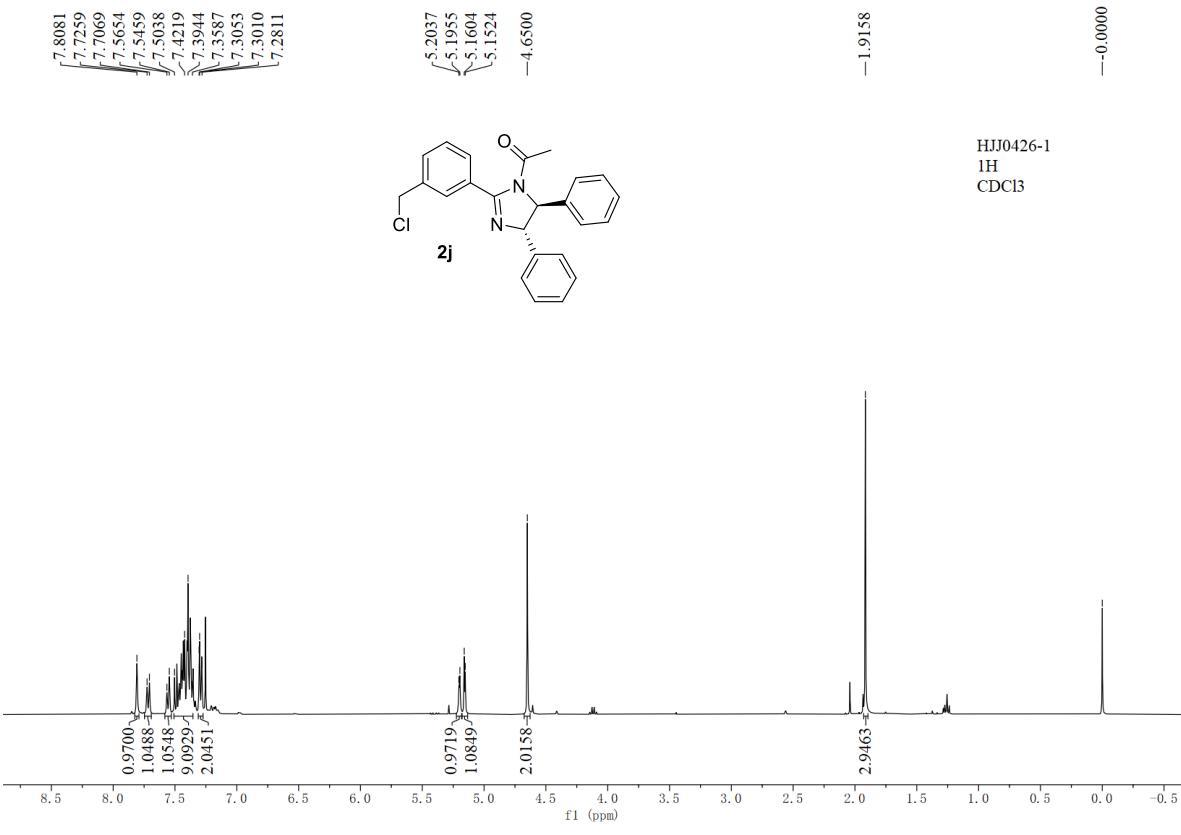
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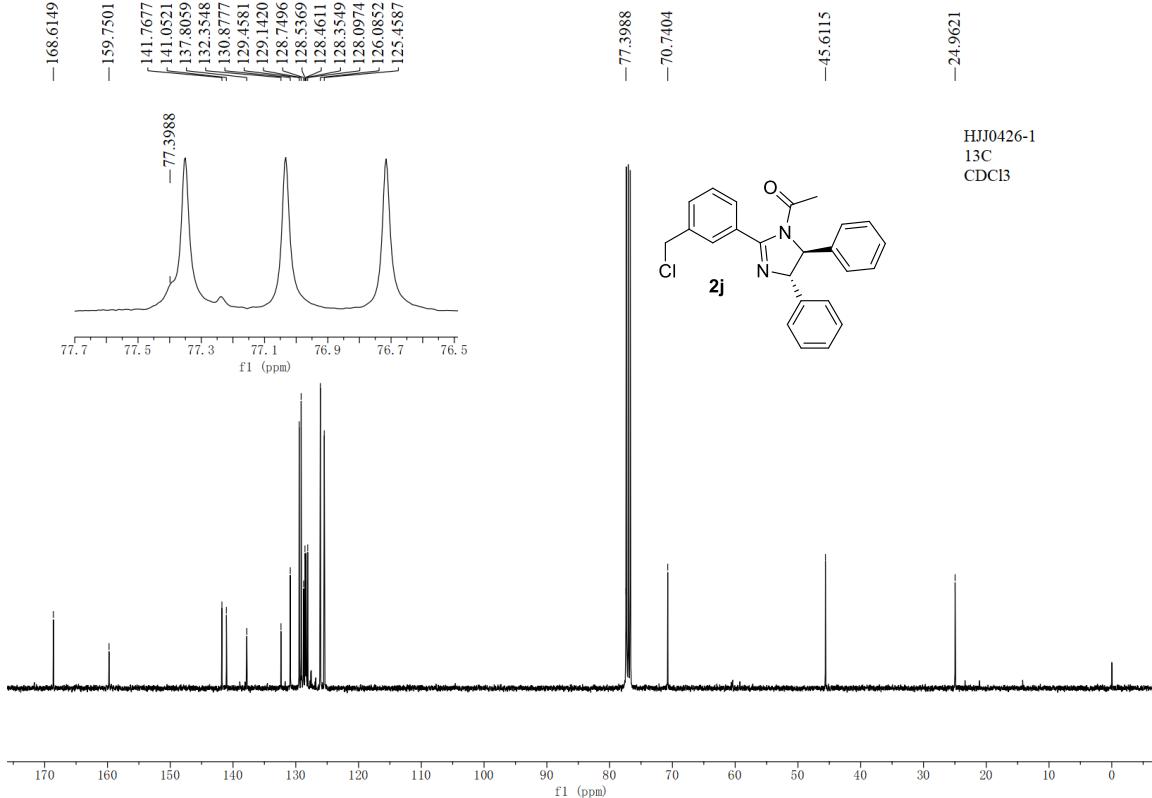
¹H NMR spectrum of **2i** (600 MHz, CDCl₃)



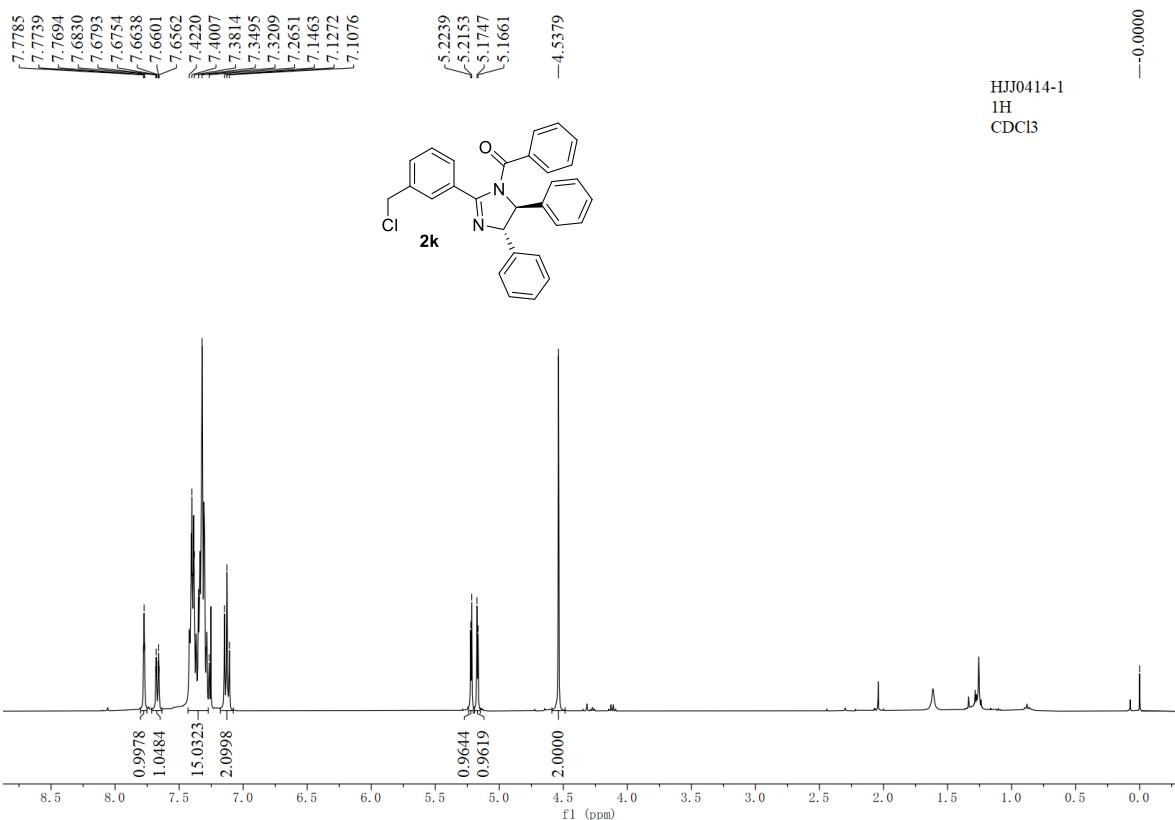
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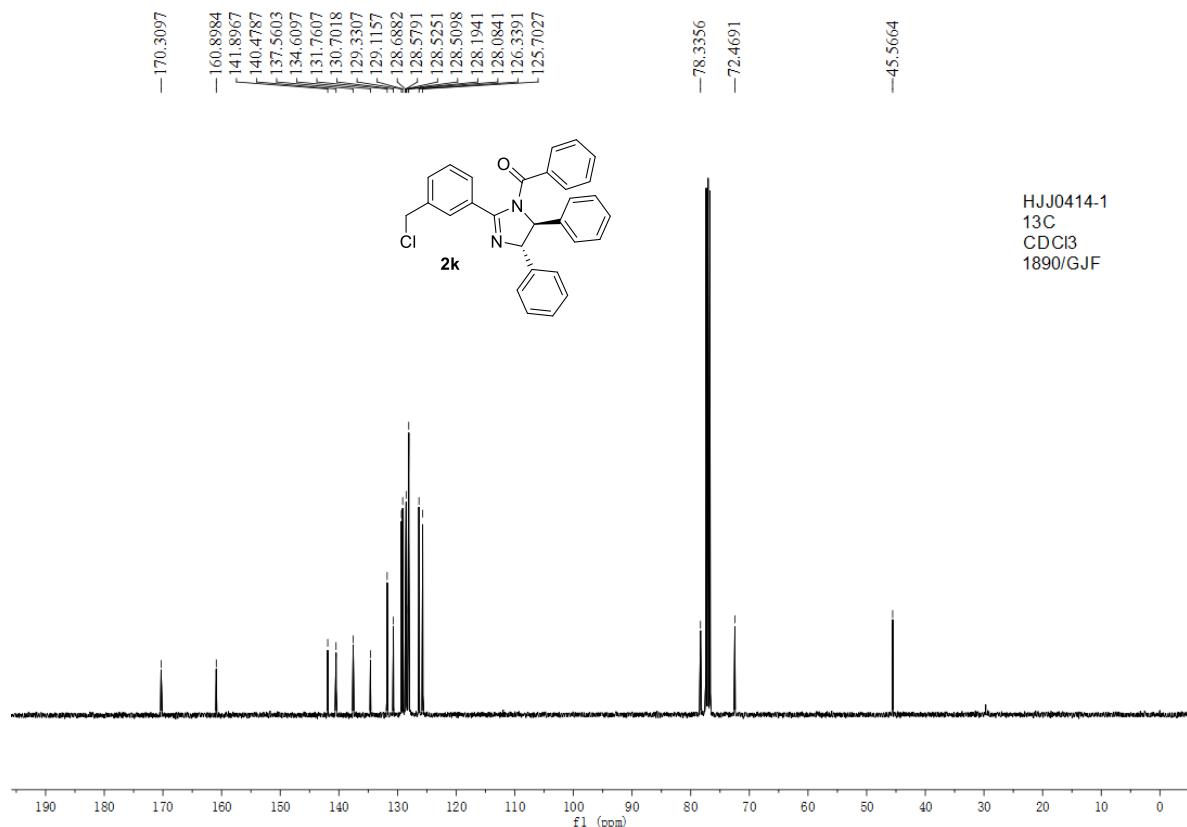
^1H NMR spectrum of **2j** (400 MHz, CDCl_3)



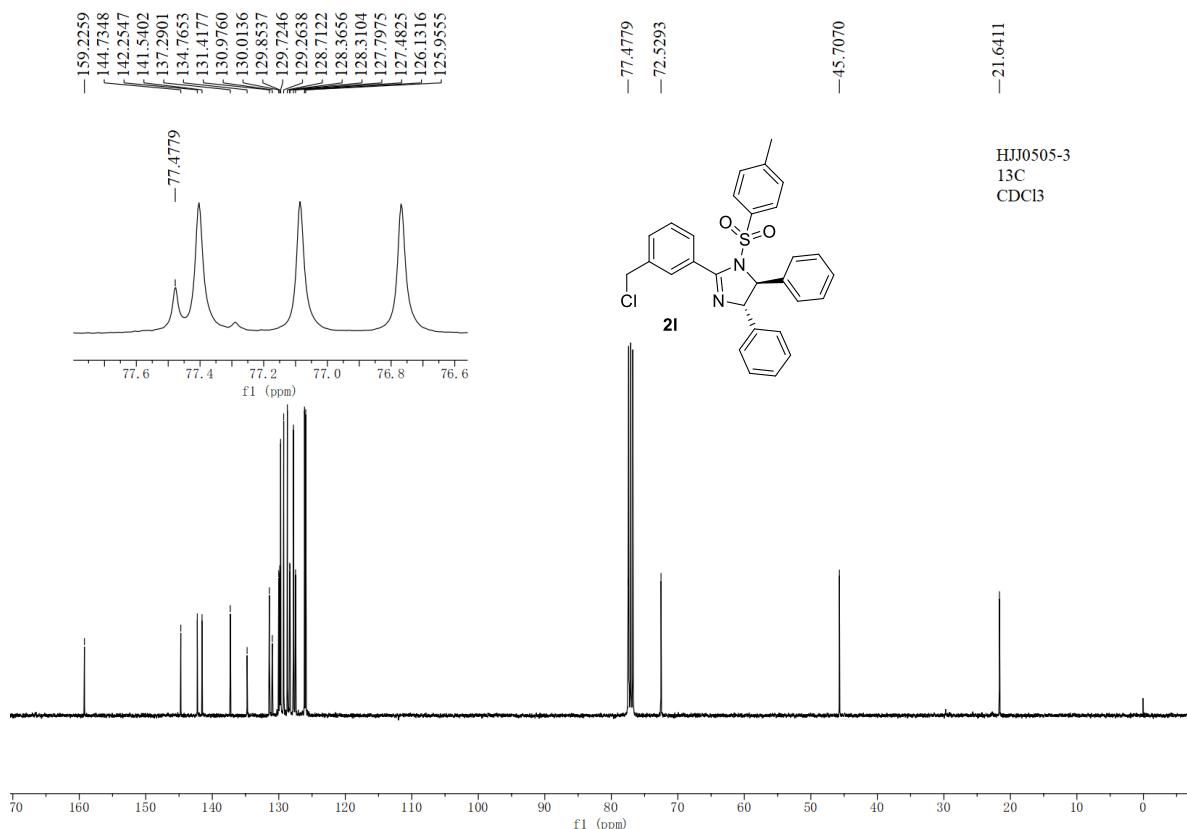
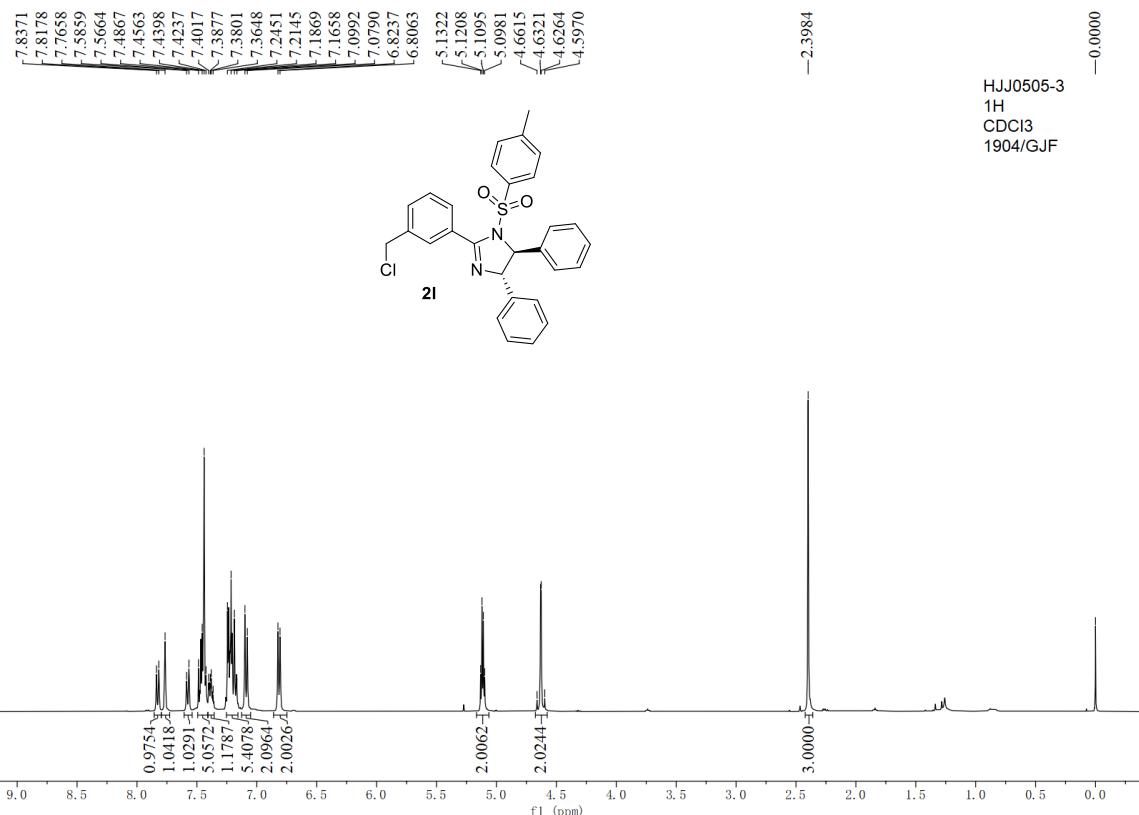
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2j** (100 MHz, CDCl_3)

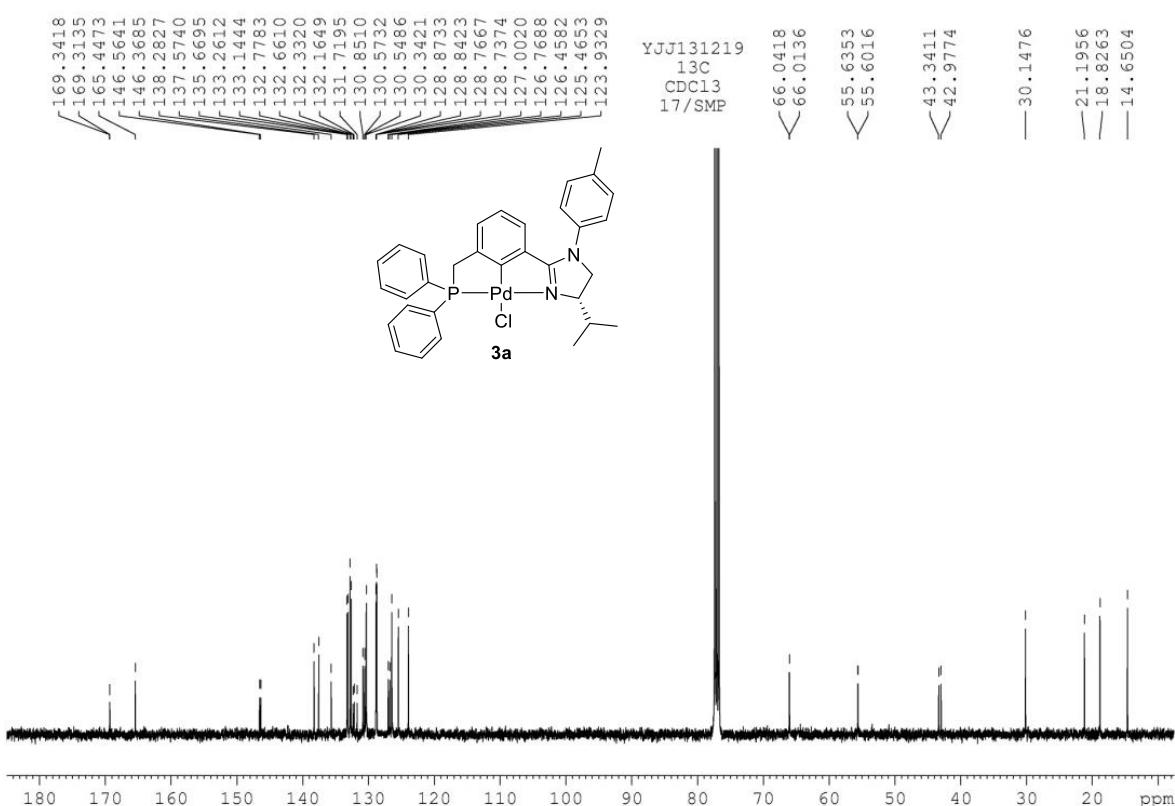
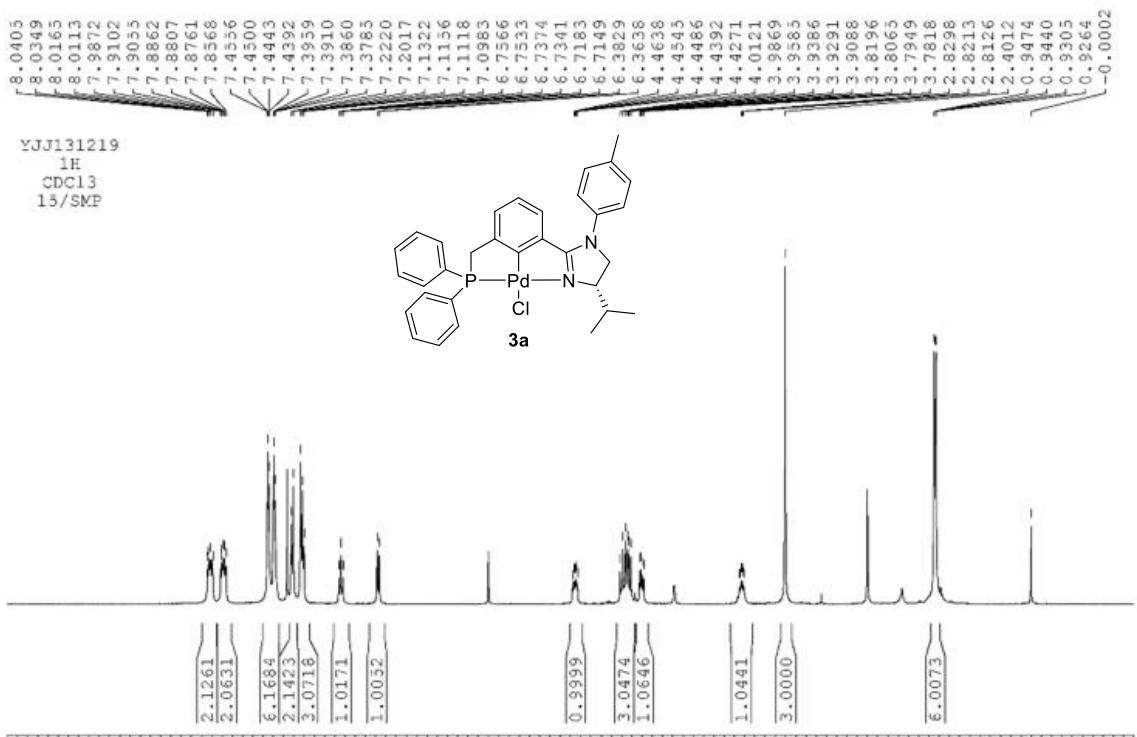


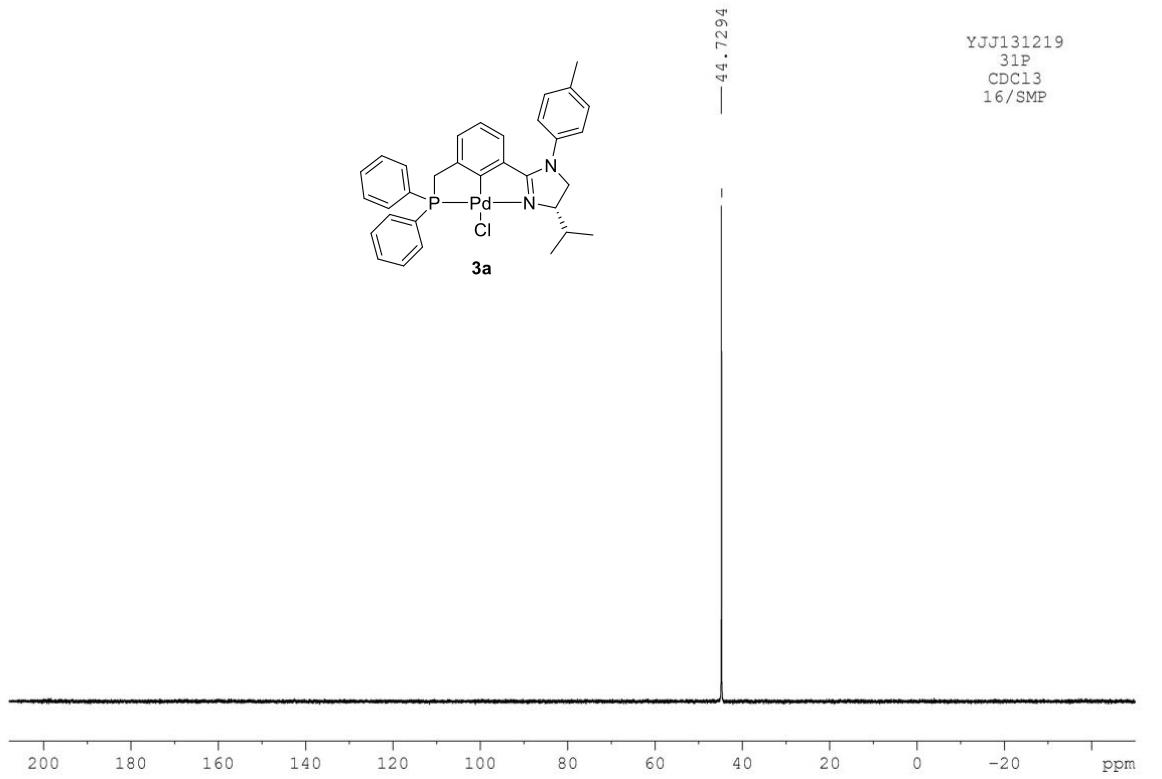
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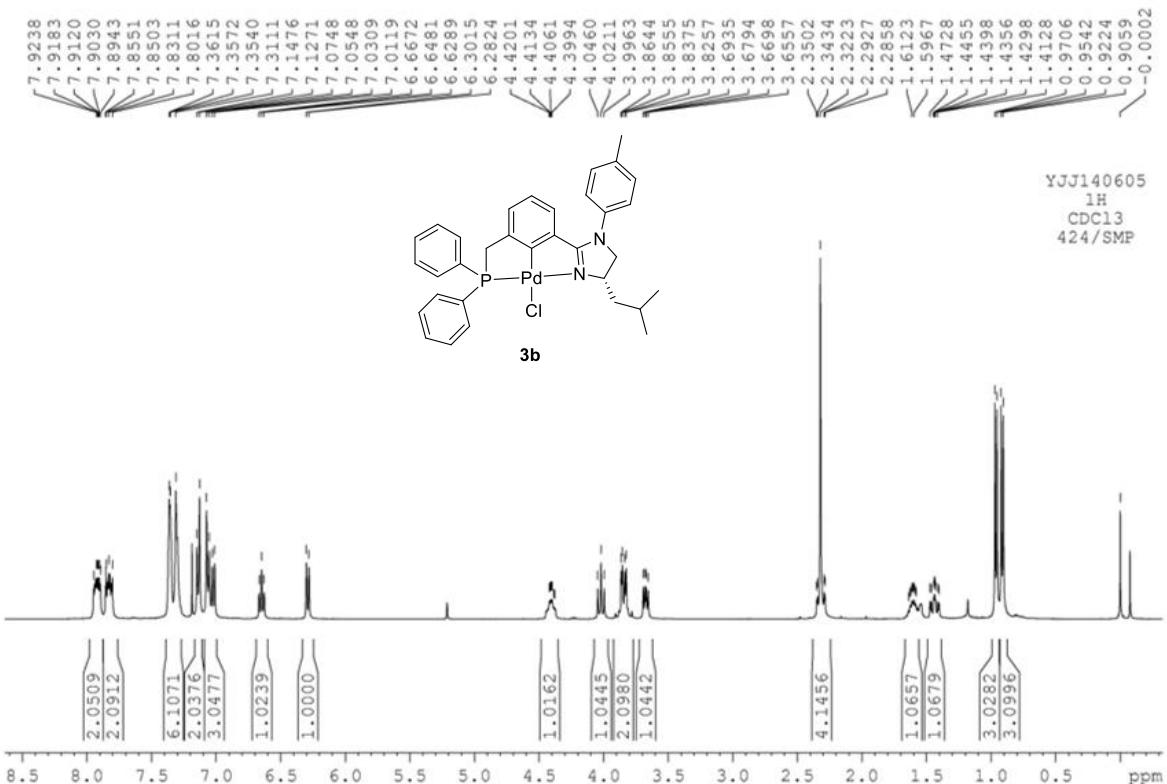
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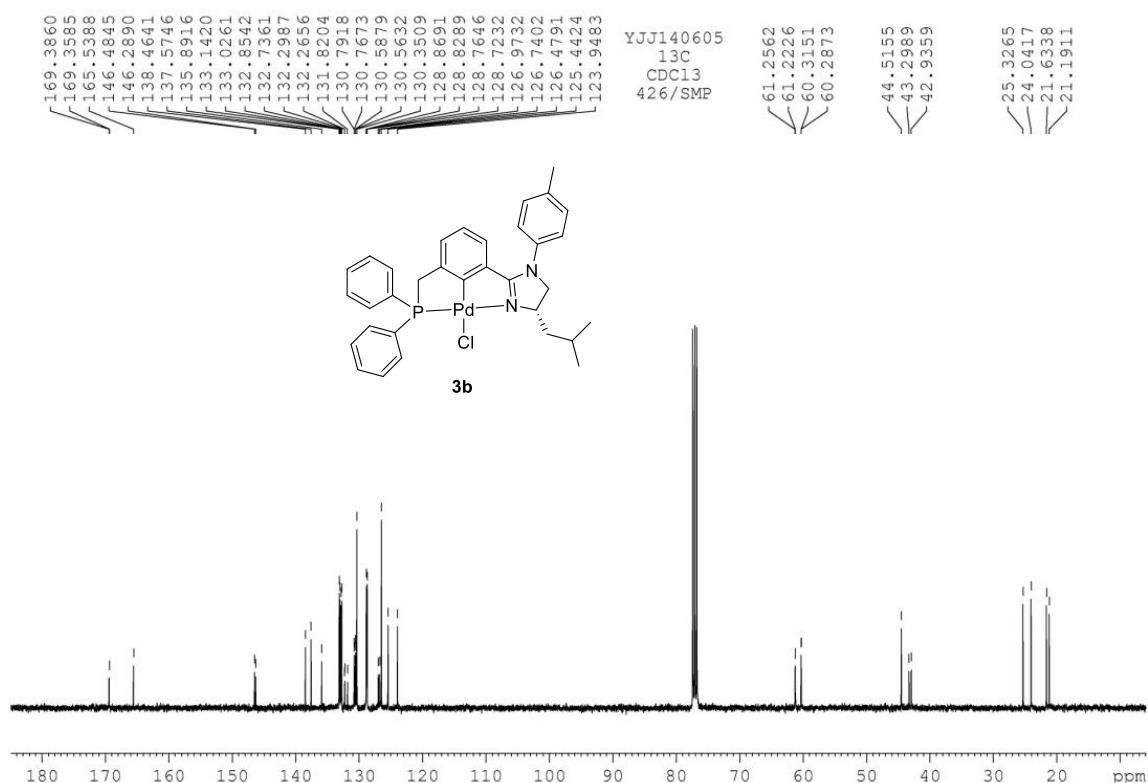




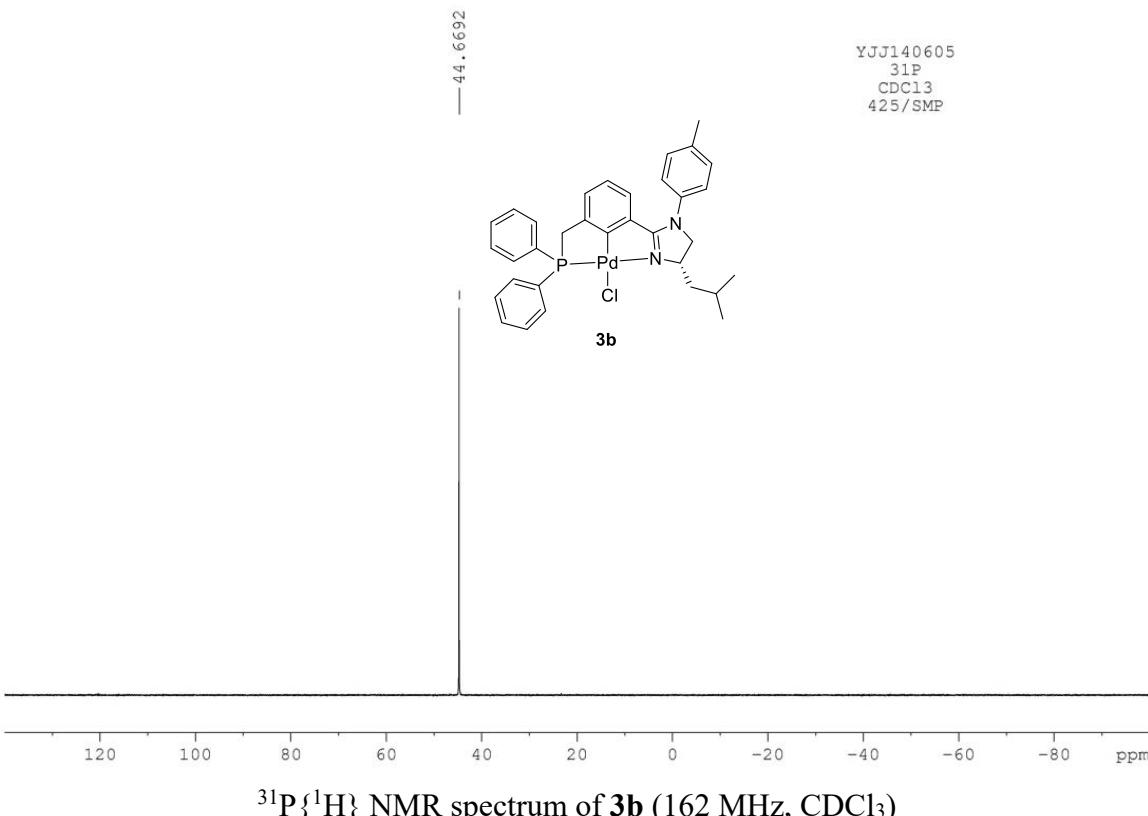
³¹P{¹H} NMR spectrum of **3a** (162 MHz, CDCl₃)



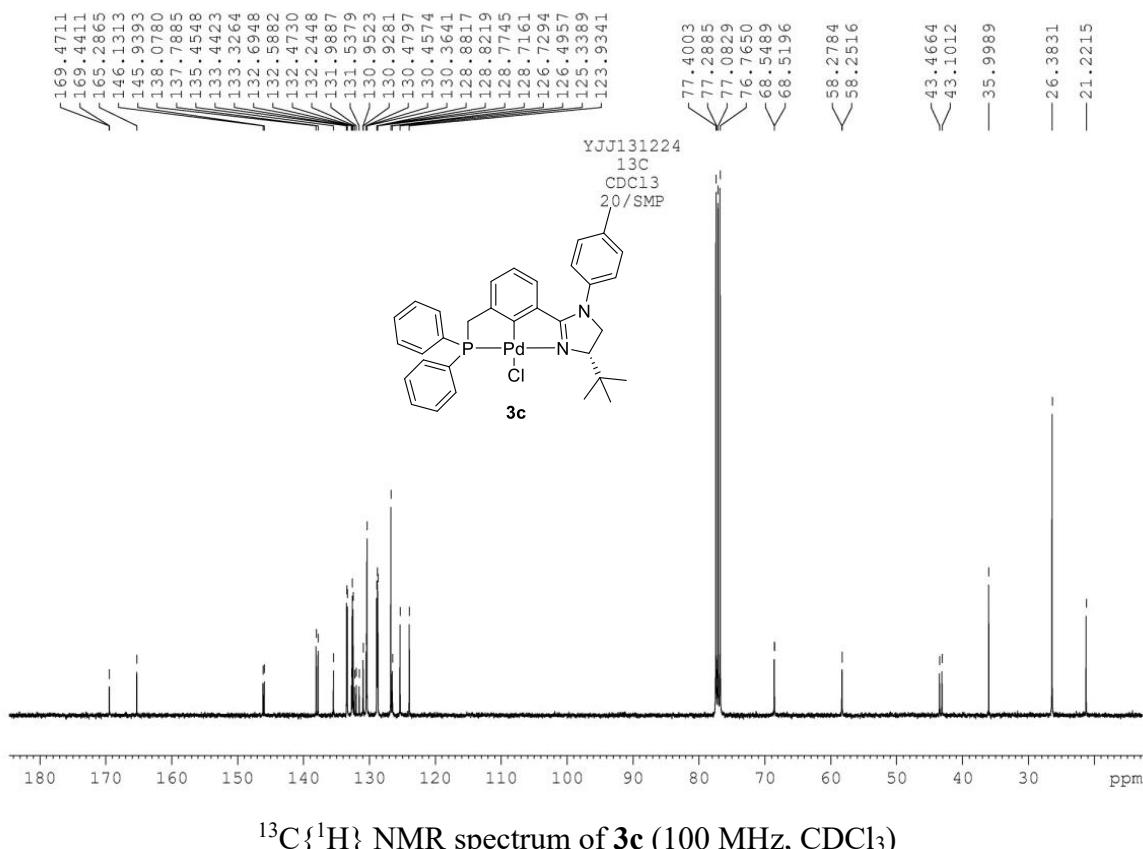
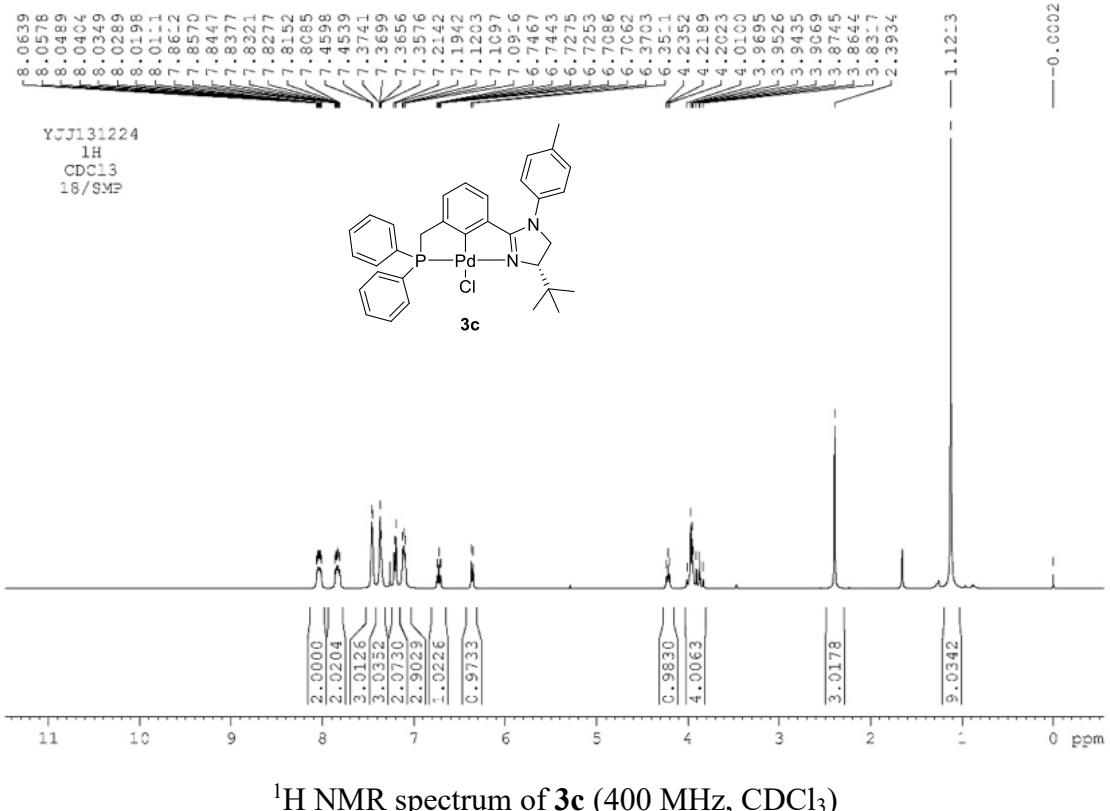
¹H NMR spectrum of **3b** (400 MHz, CDCl₃)

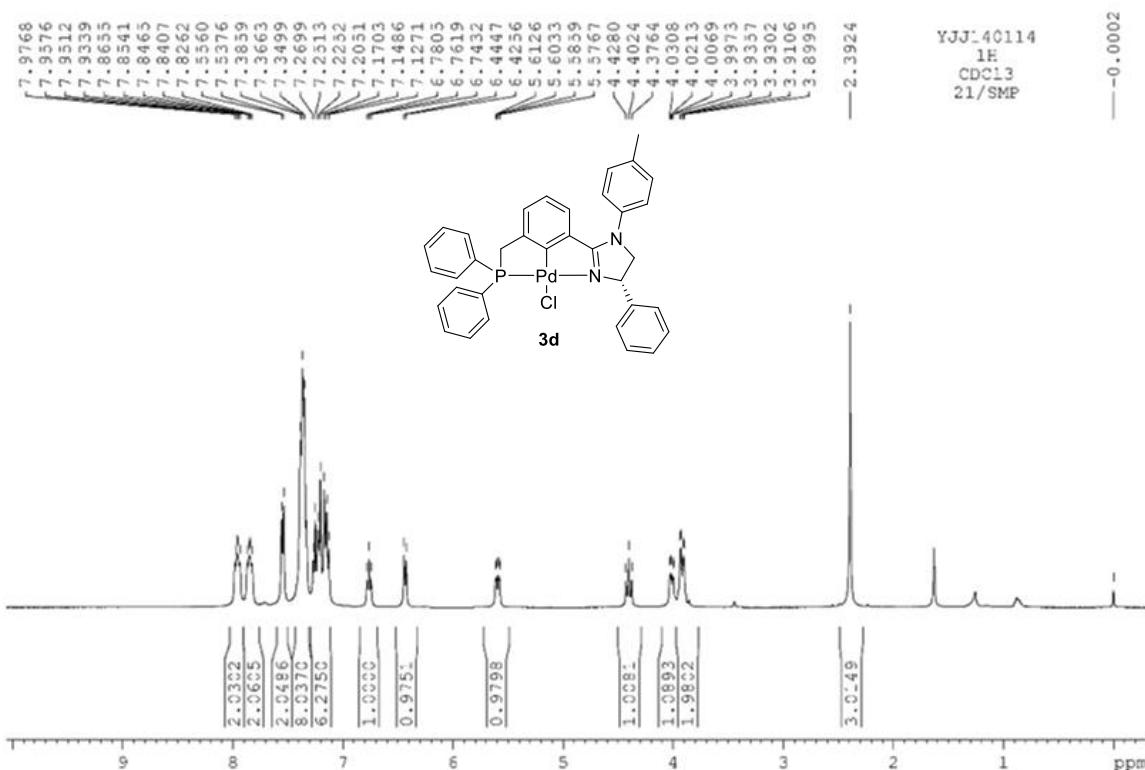
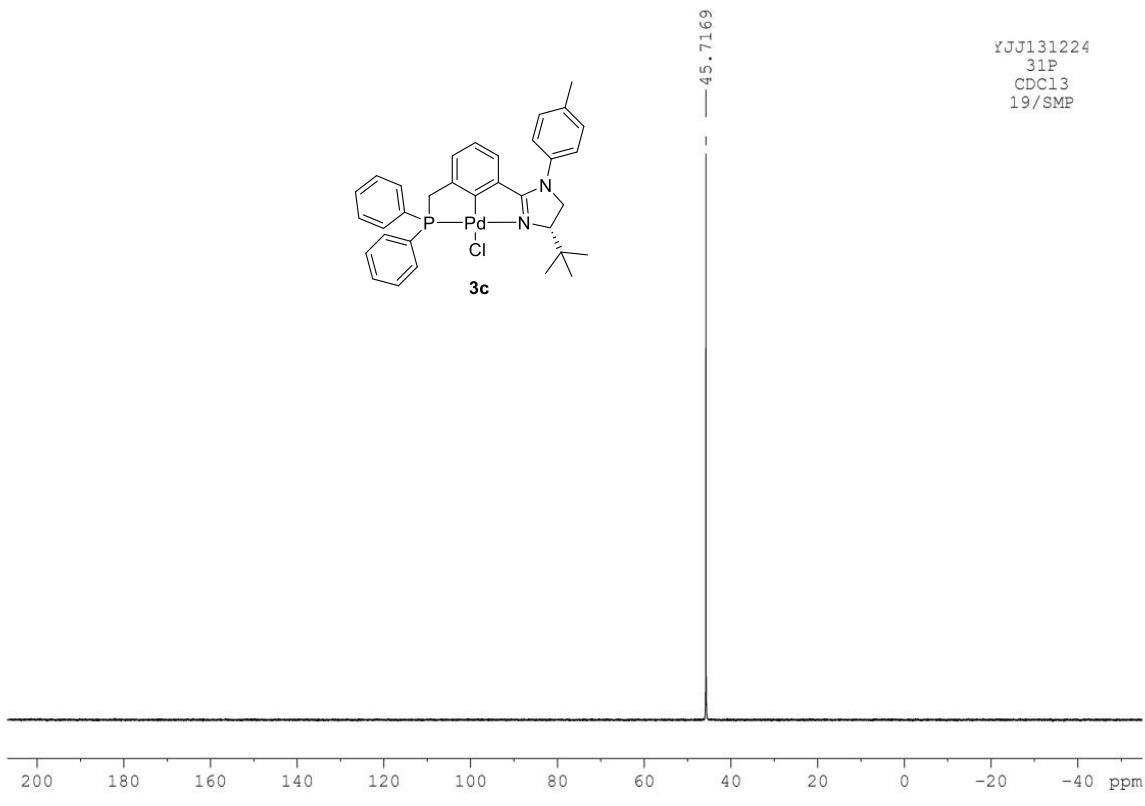


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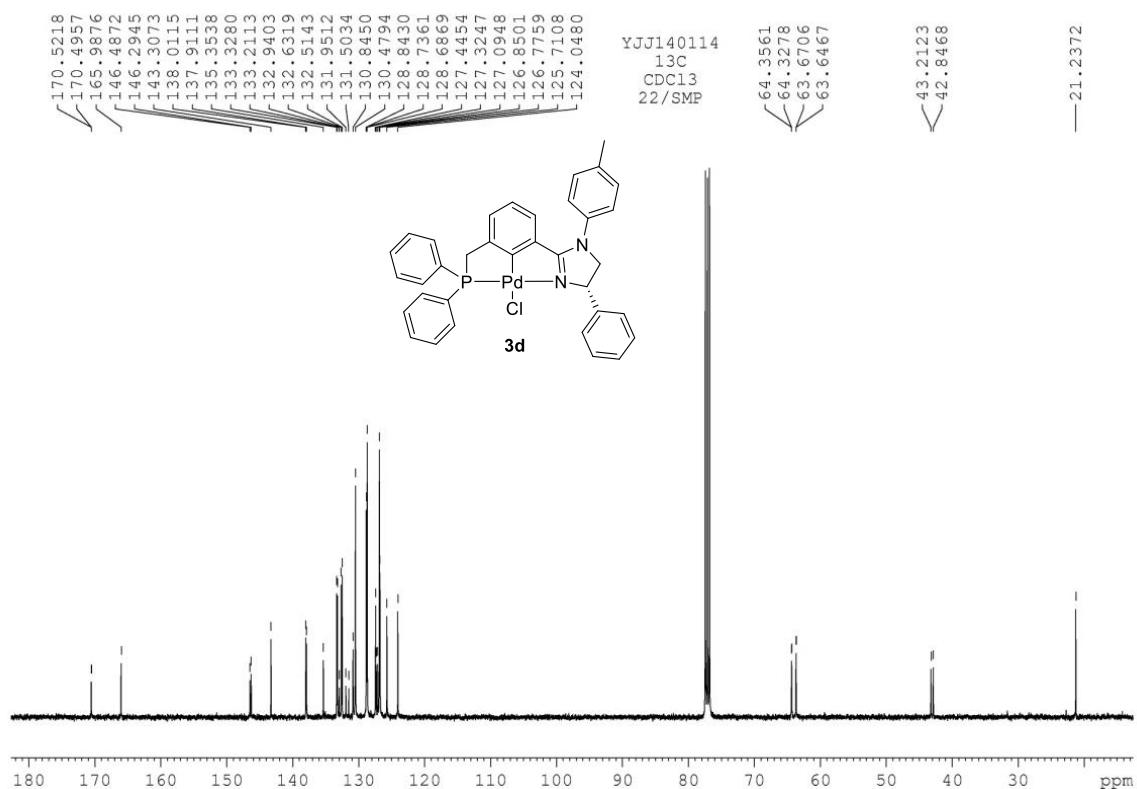


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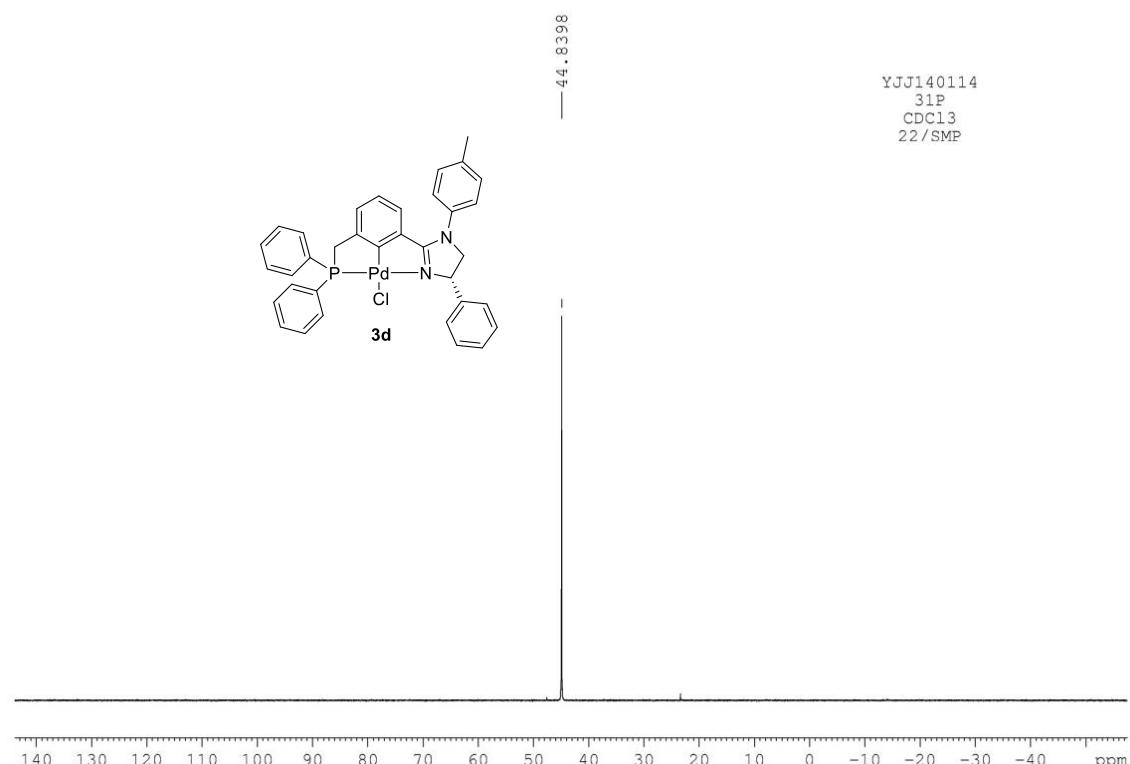




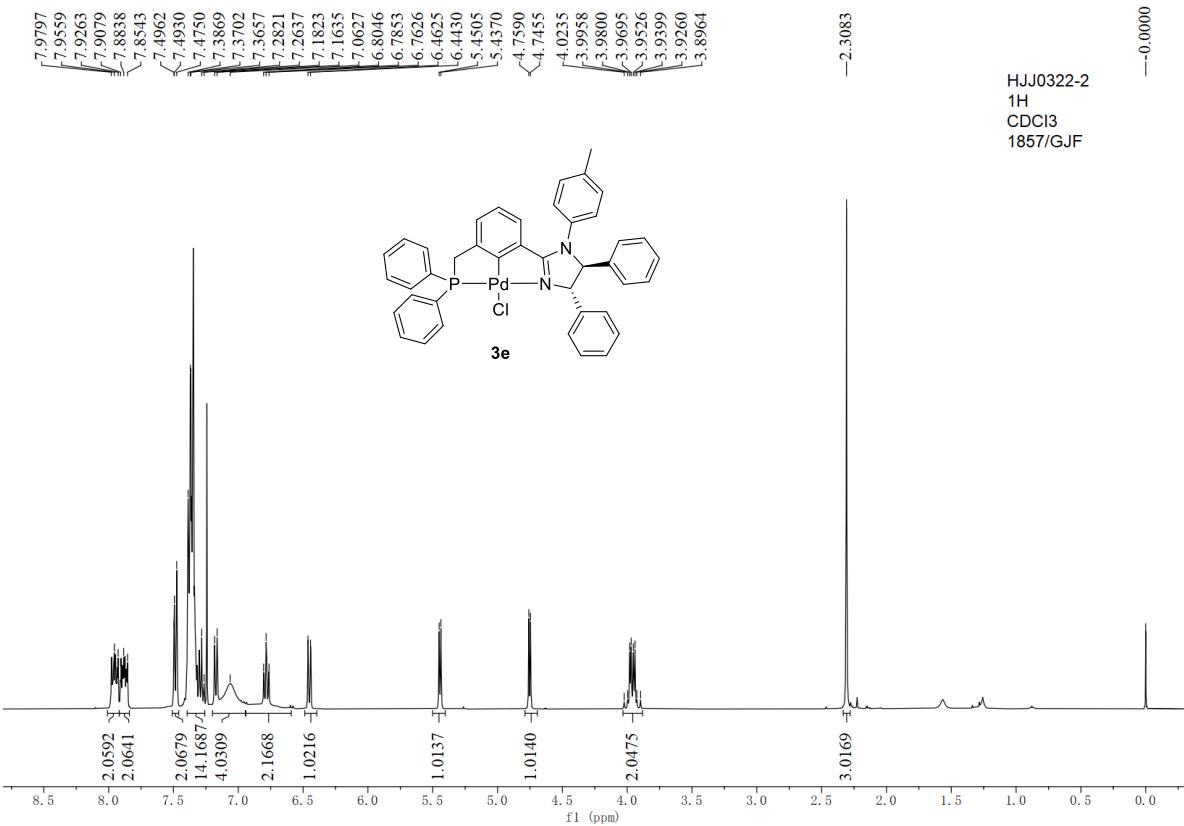
¹H NMR spectrum of **3d** (400 MHz, CDCl₃)



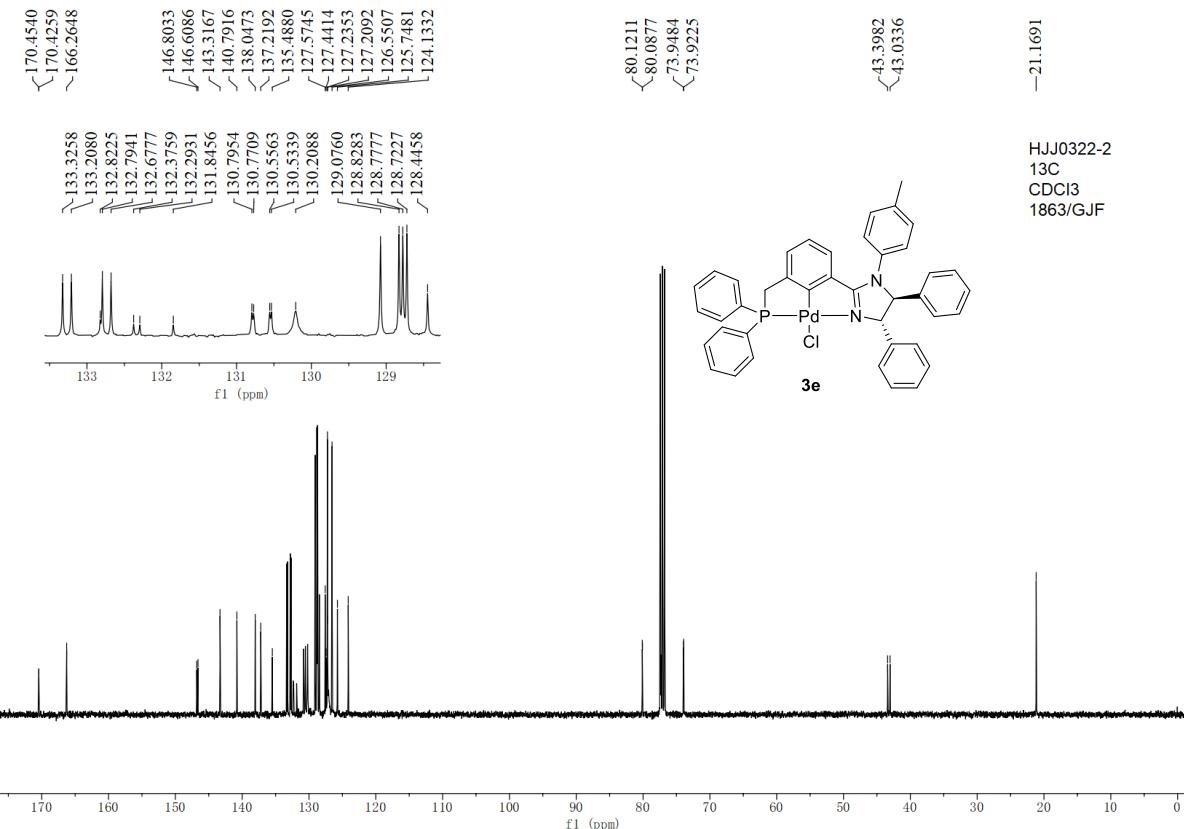
¹³C{¹H} NMR spectrum of **3d** (100 MHz, CDCl₃)



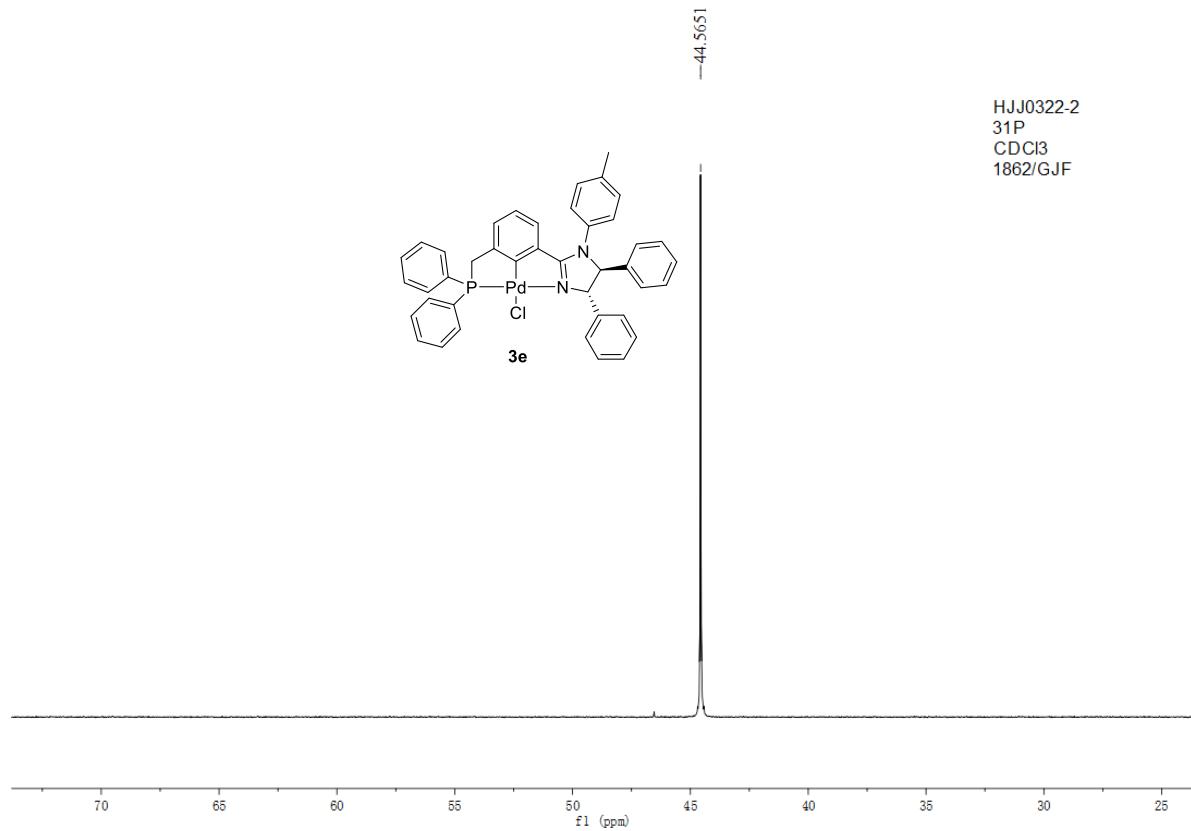
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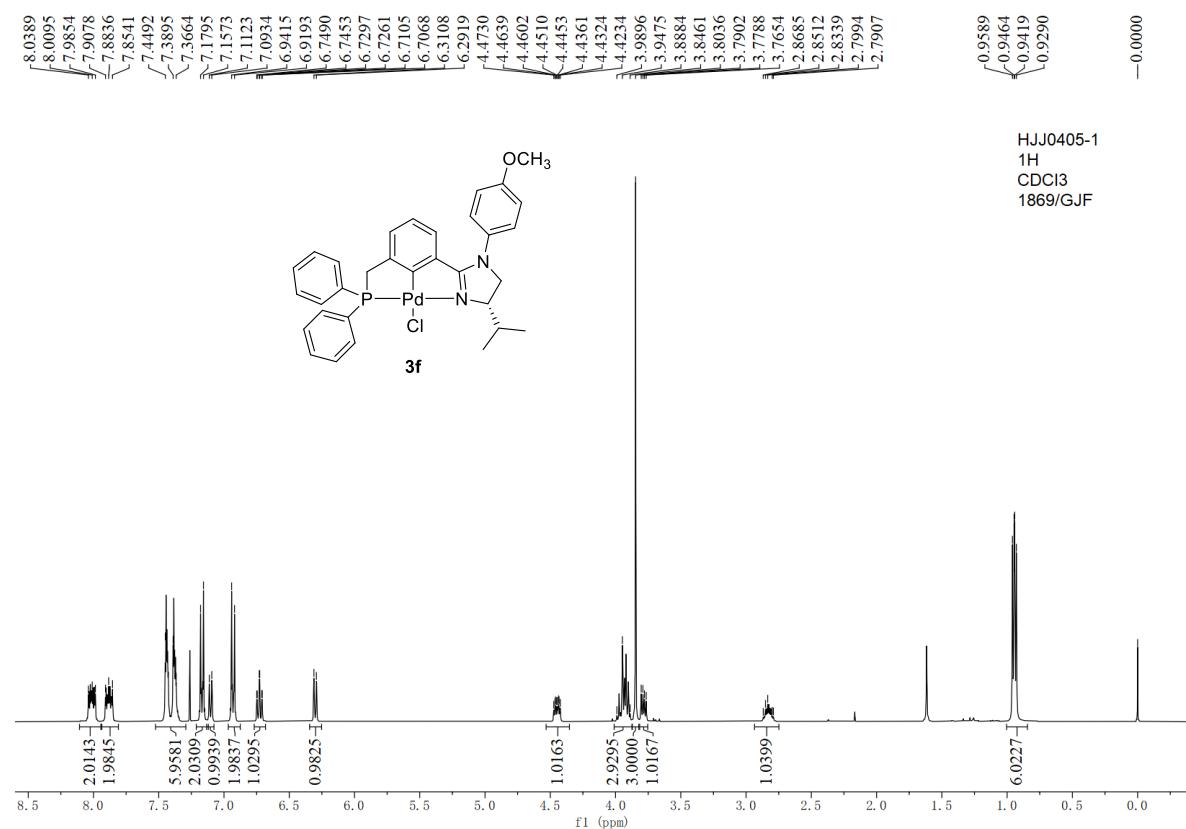
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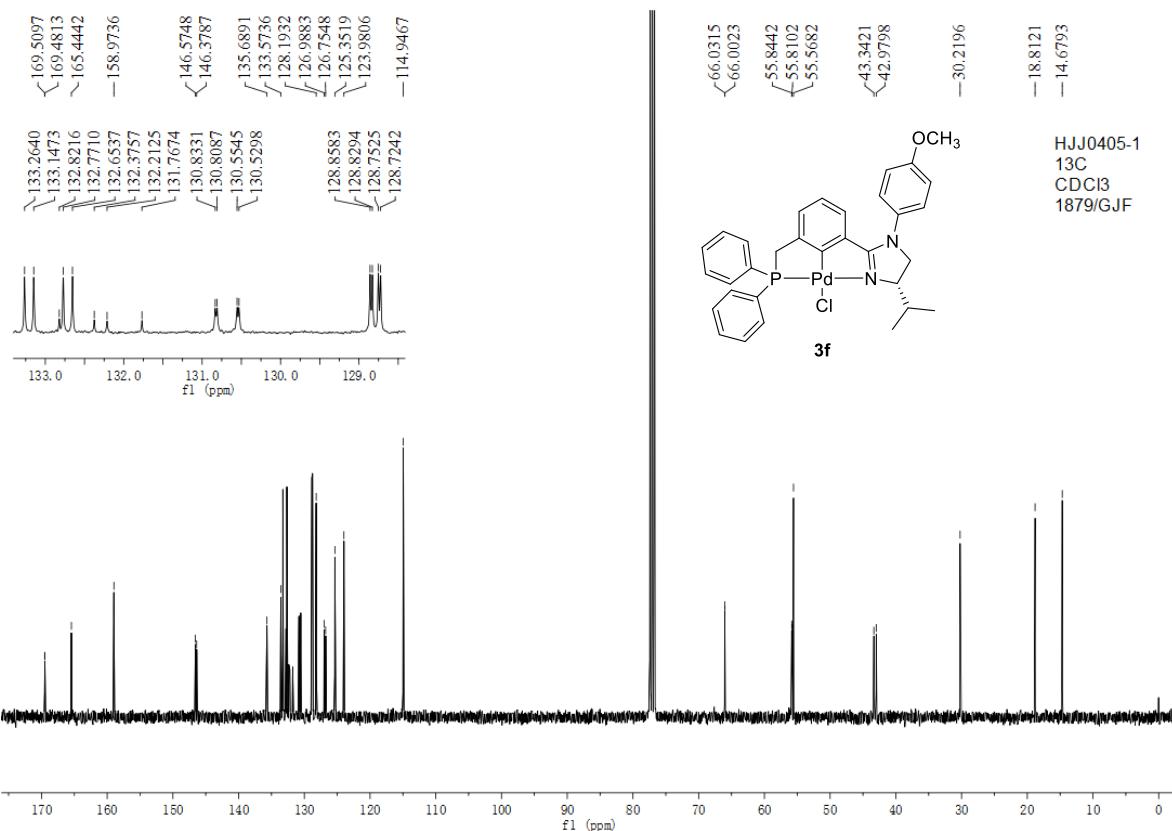
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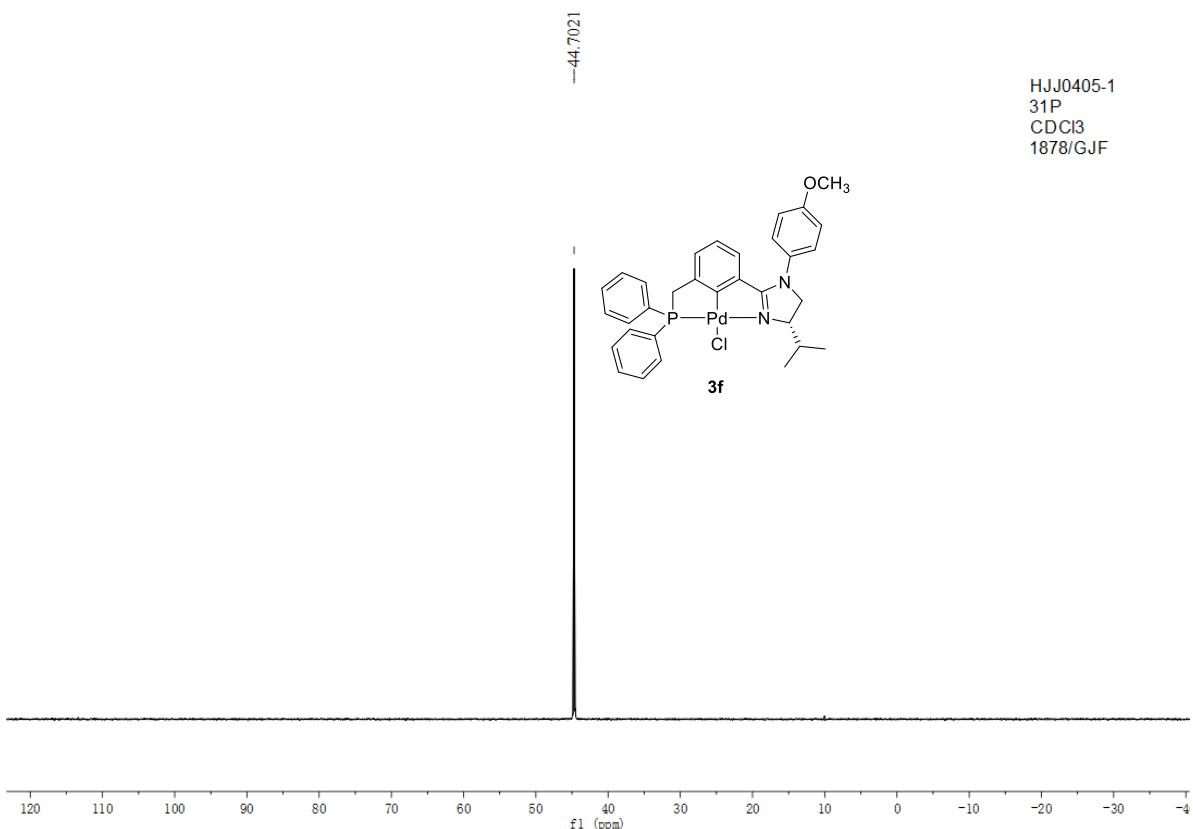
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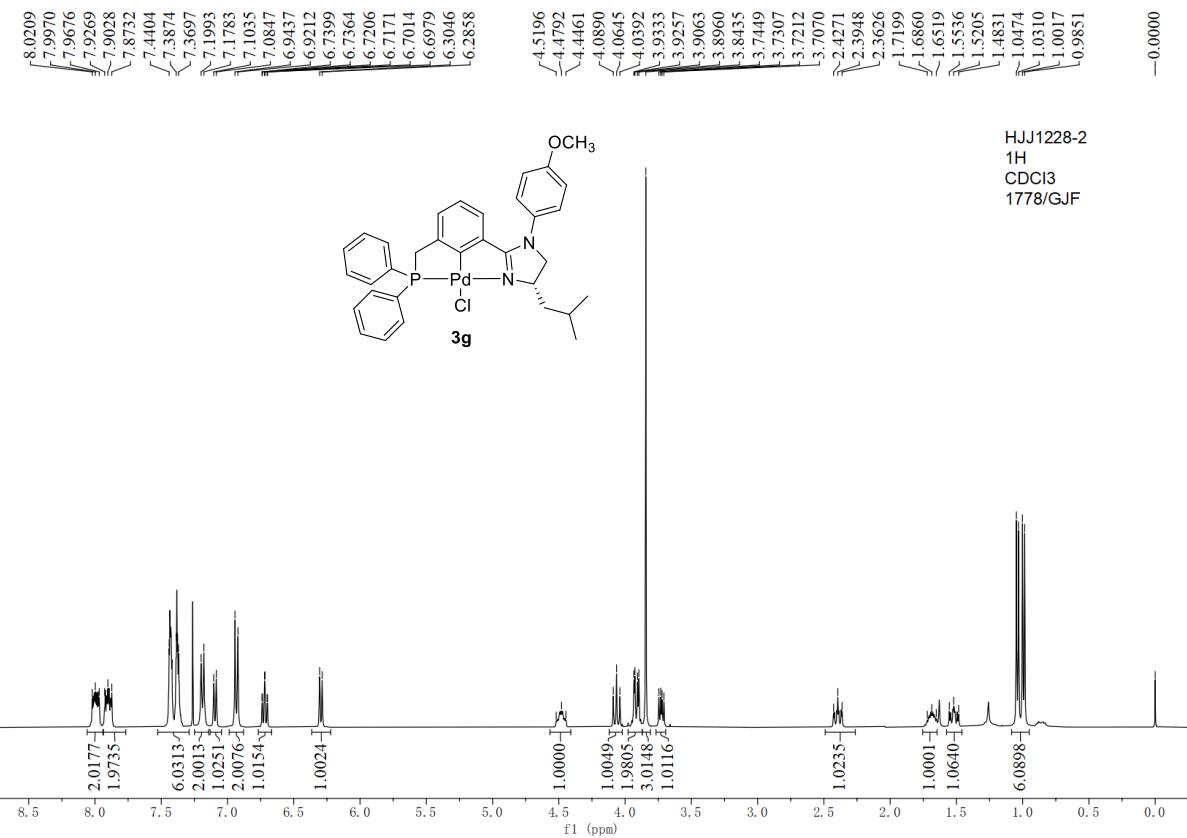
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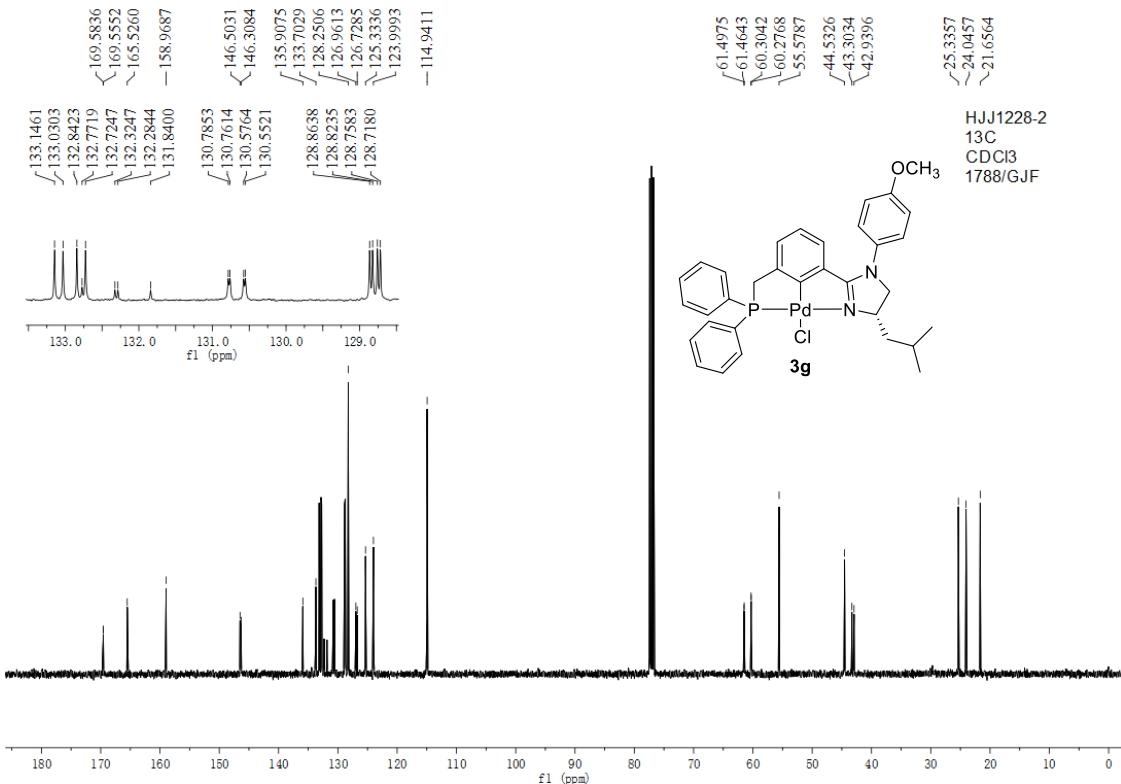
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3f** (100 MHz, CDCl_3)



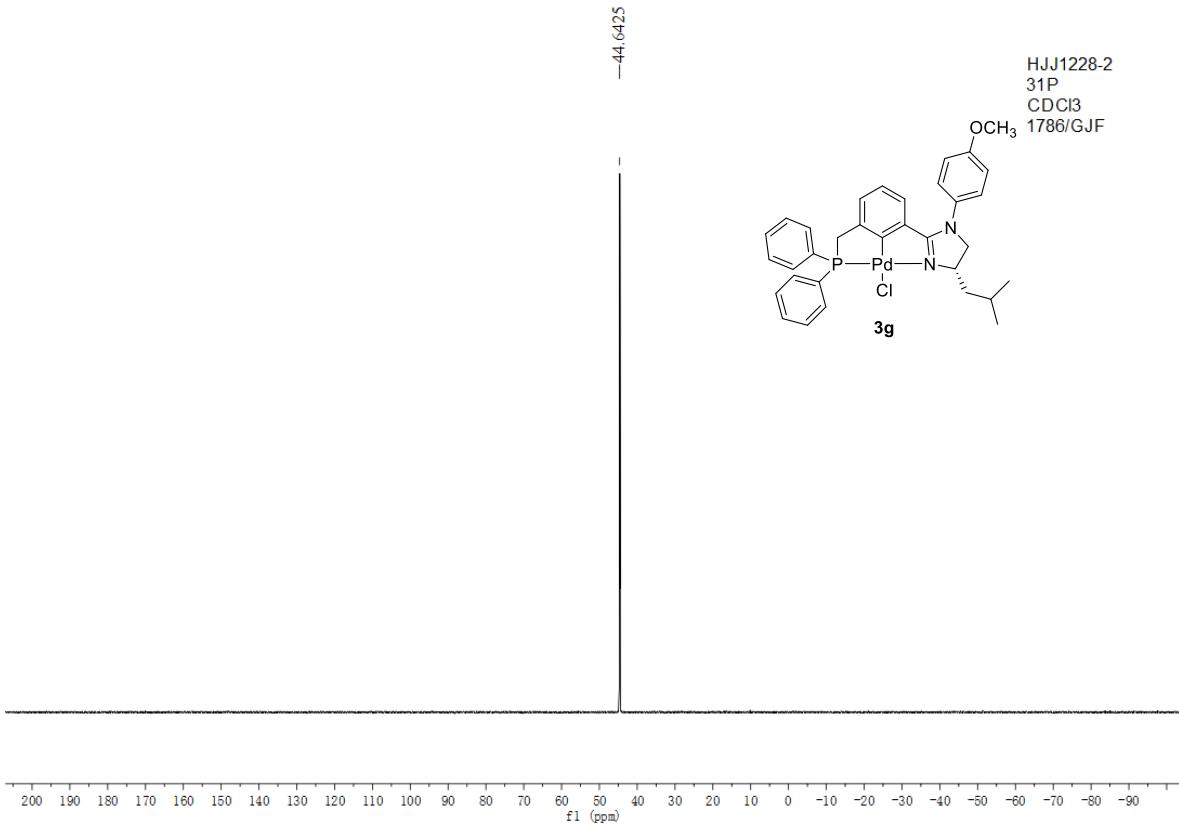
$^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of **3f** (162 MHz, CDCl_3)



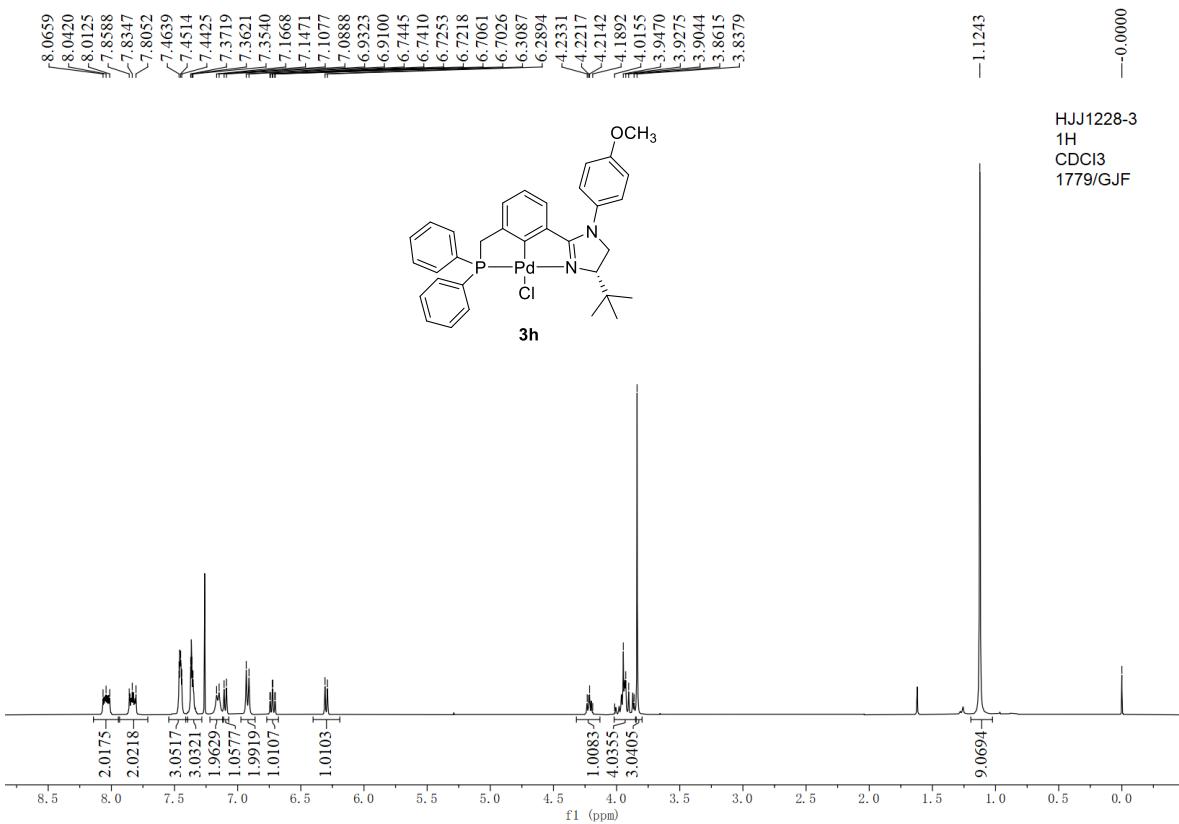
¹H NMR spectrum of **3g** (400 MHz, CDCl₃)



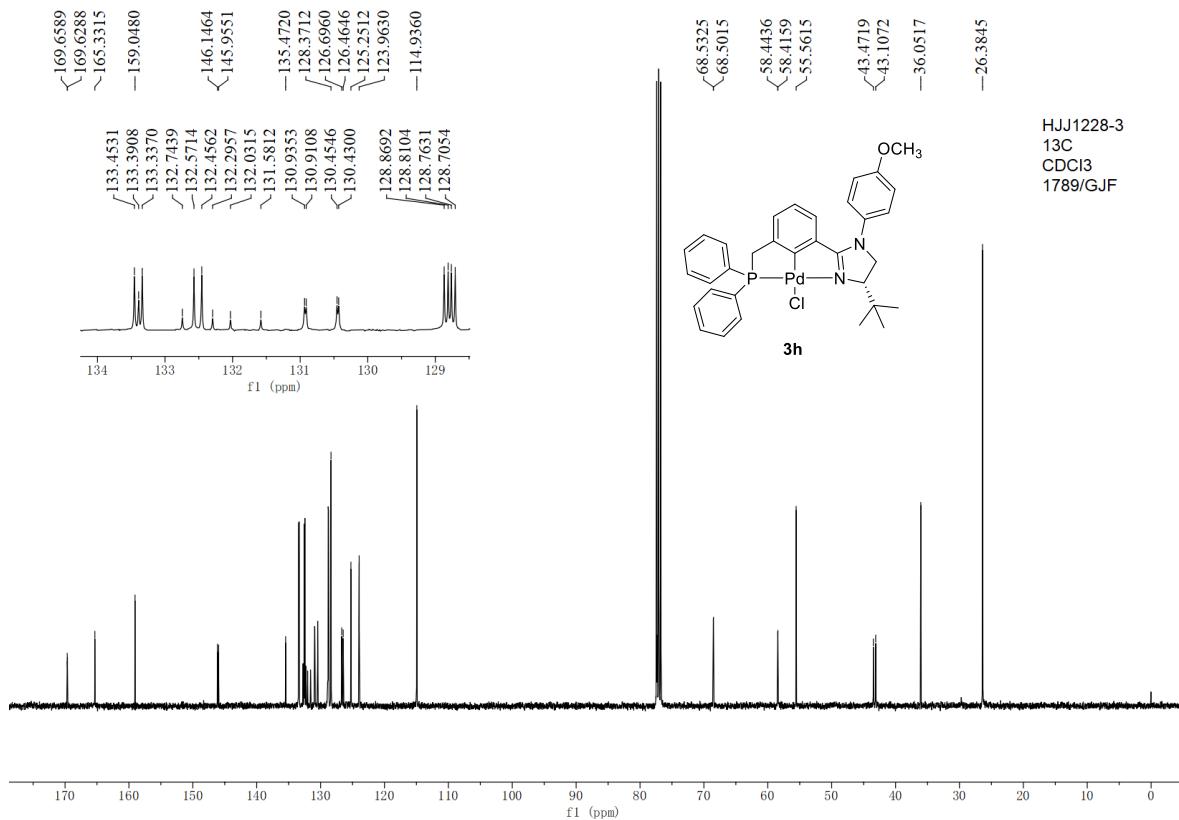
¹³C{¹H} NMR spectrum of **3g** (100 MHz, CDCl₃)



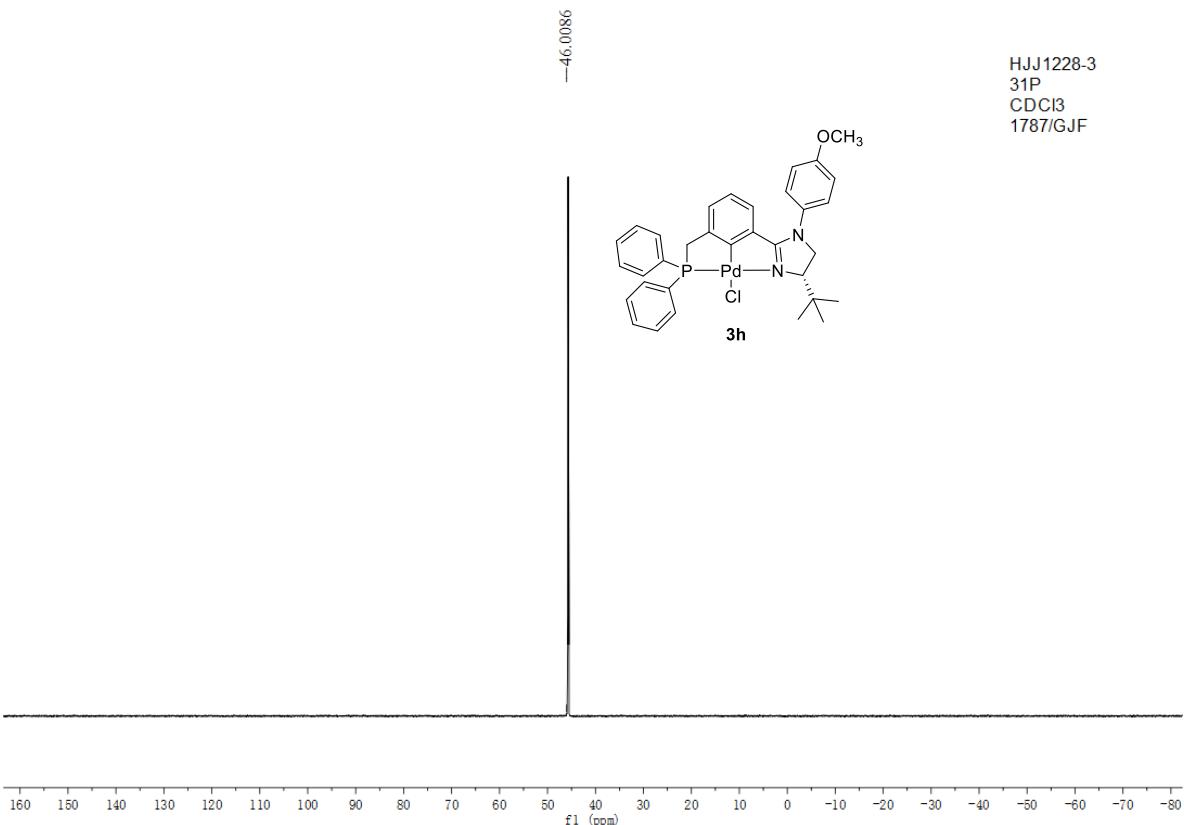
³¹P{¹H} NMR spectrum of **3g** (162 MHz, CDCl₃)



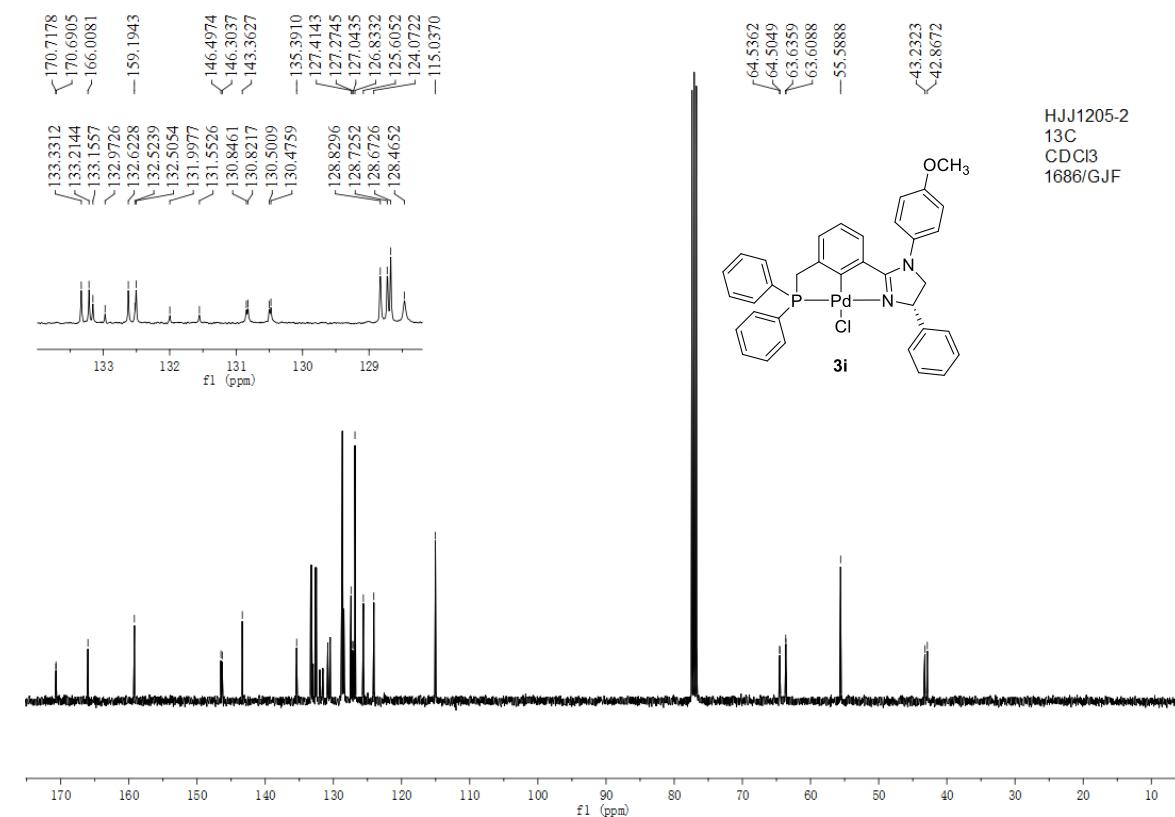
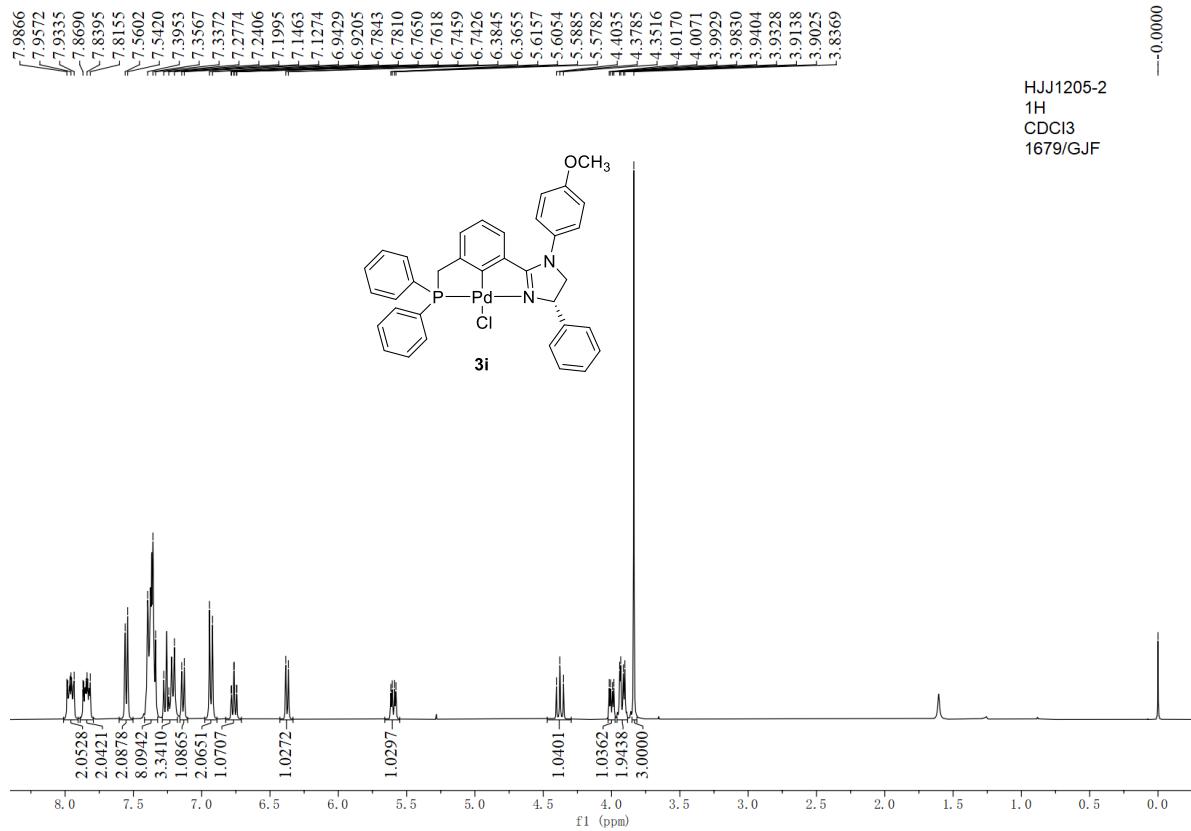
¹H NMR spectrum of **3h** (400 MHz, CDCl₃)

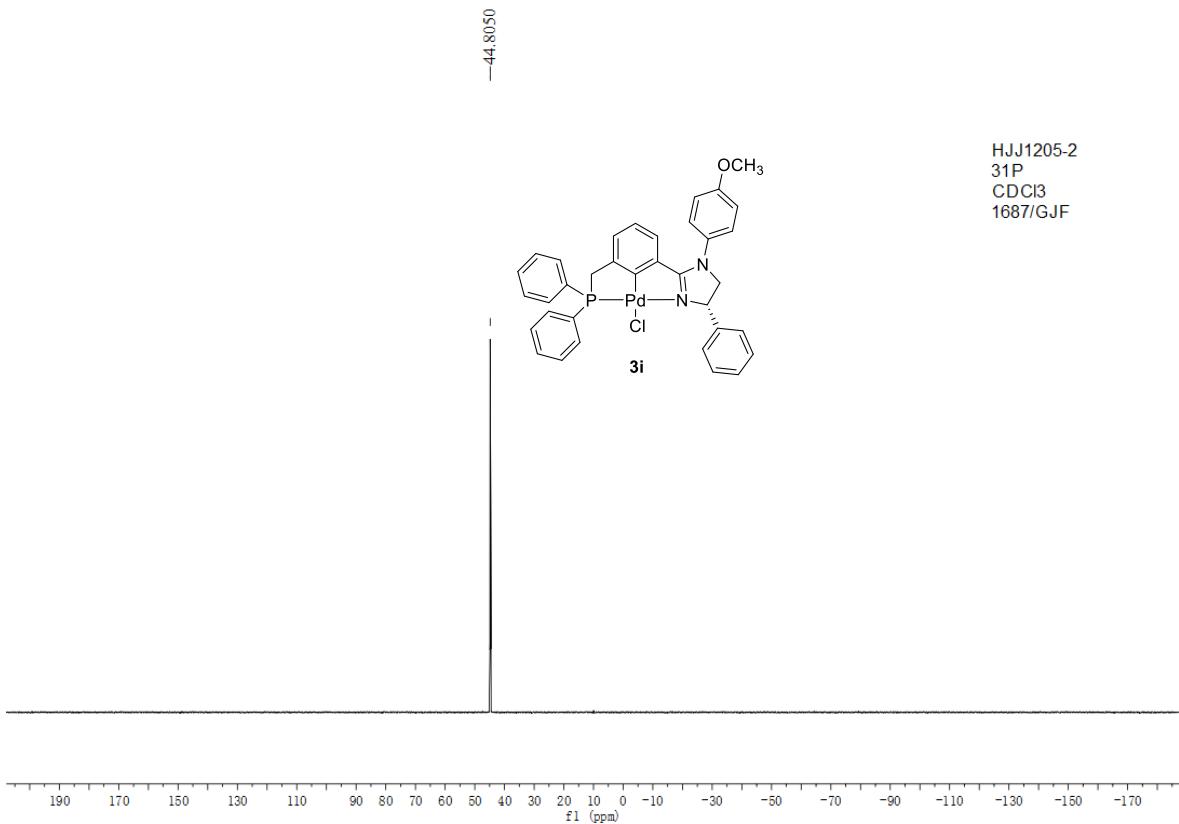


¹³C{¹H} NMR spectrum of **3h** (100 MHz, CDCl₃)

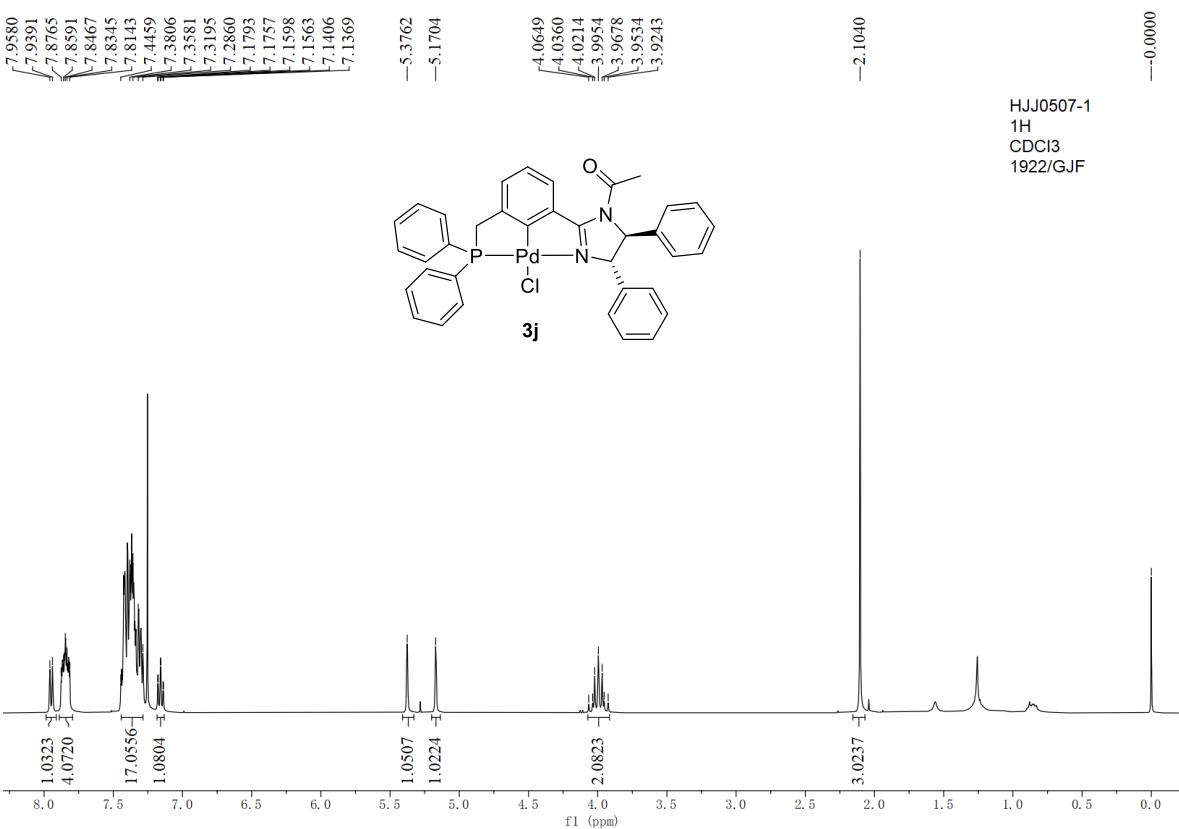


³¹P{¹H} NMR spectrum of **3h** (162 MHz, CDCl₃)

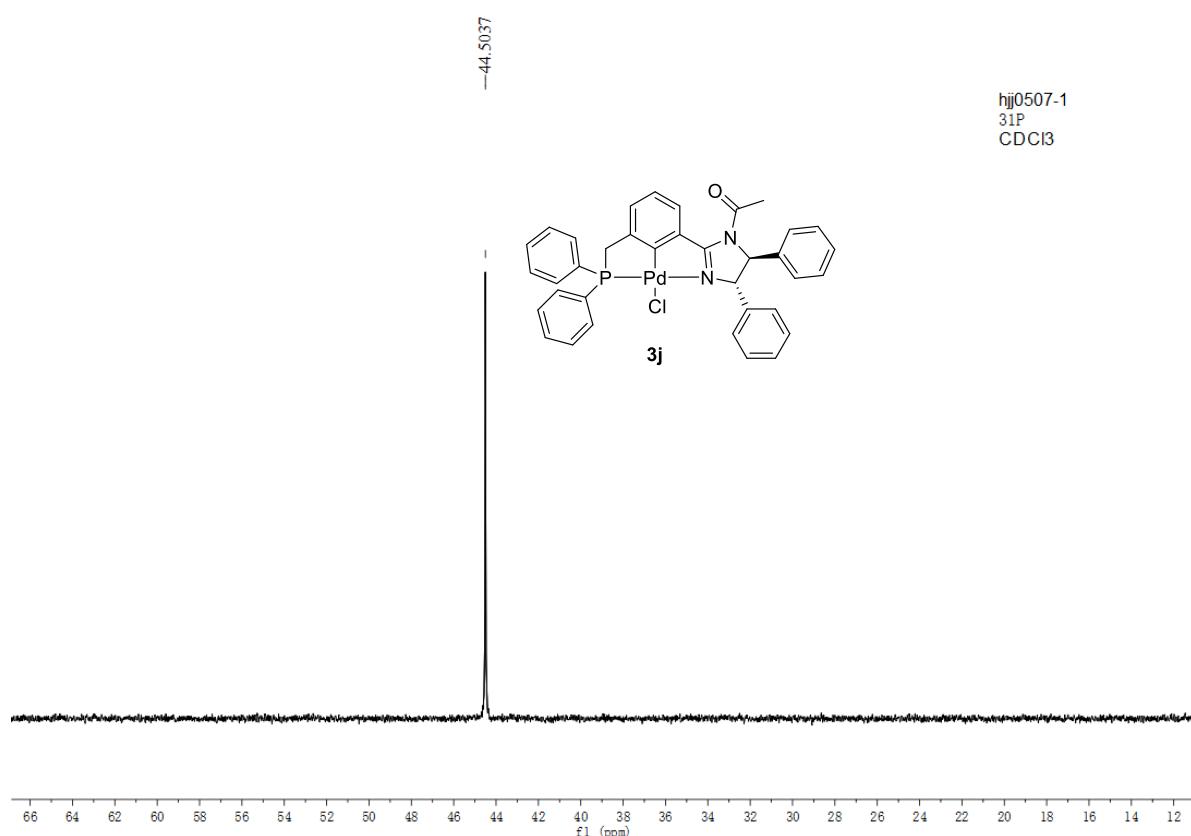
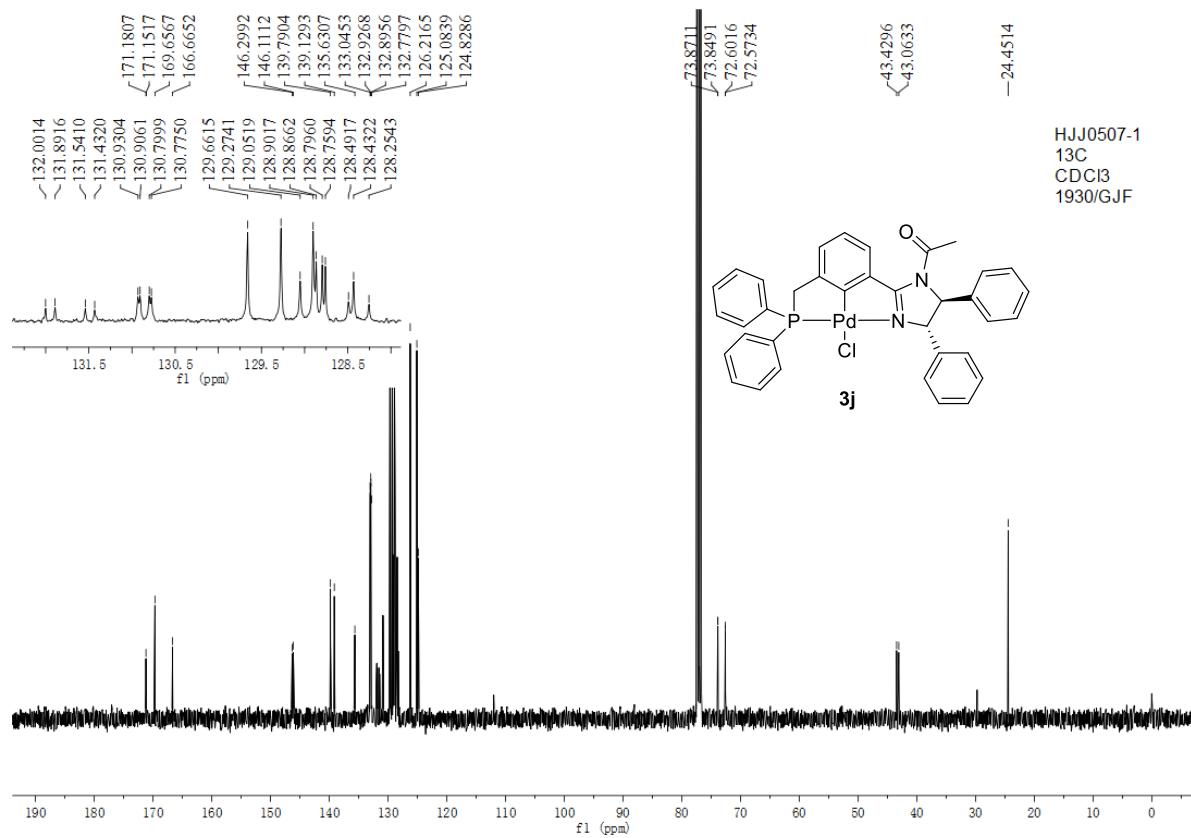


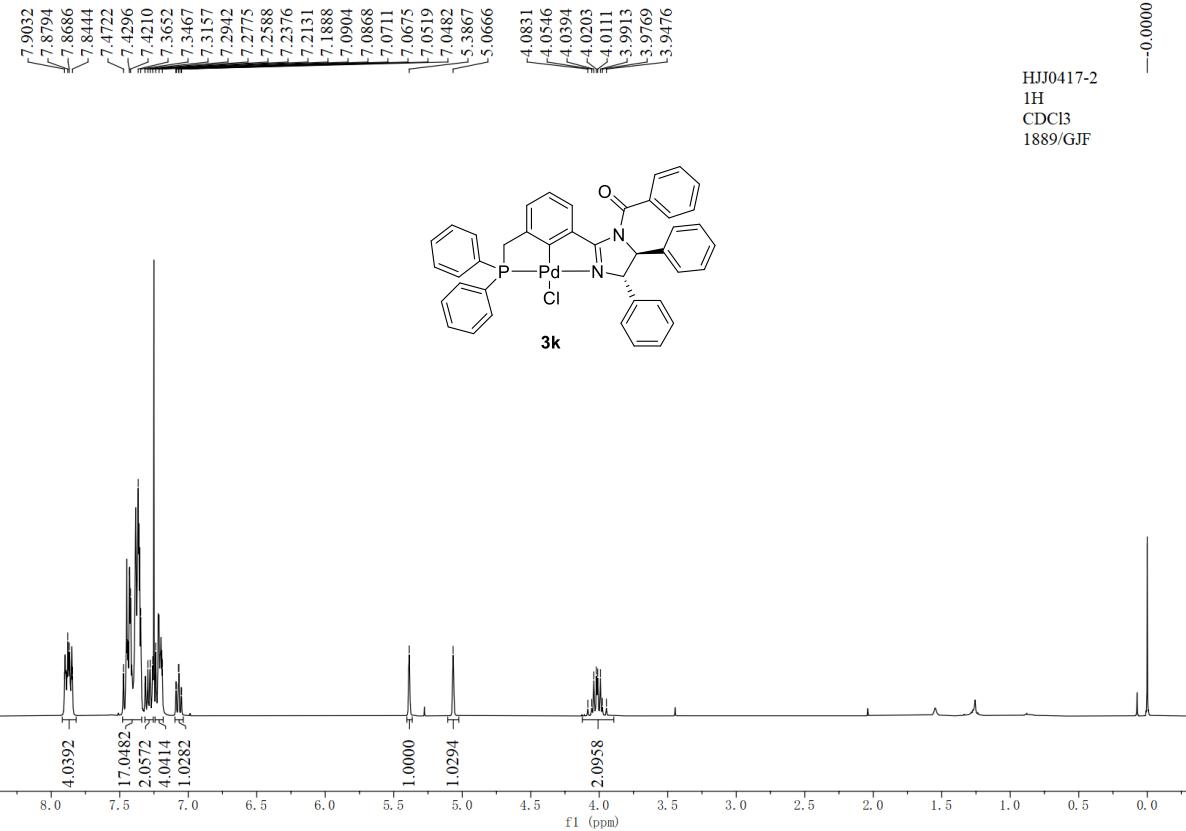


³¹P{¹H} NMR spectrum of **3i** (162 MHz, CDCl₃)

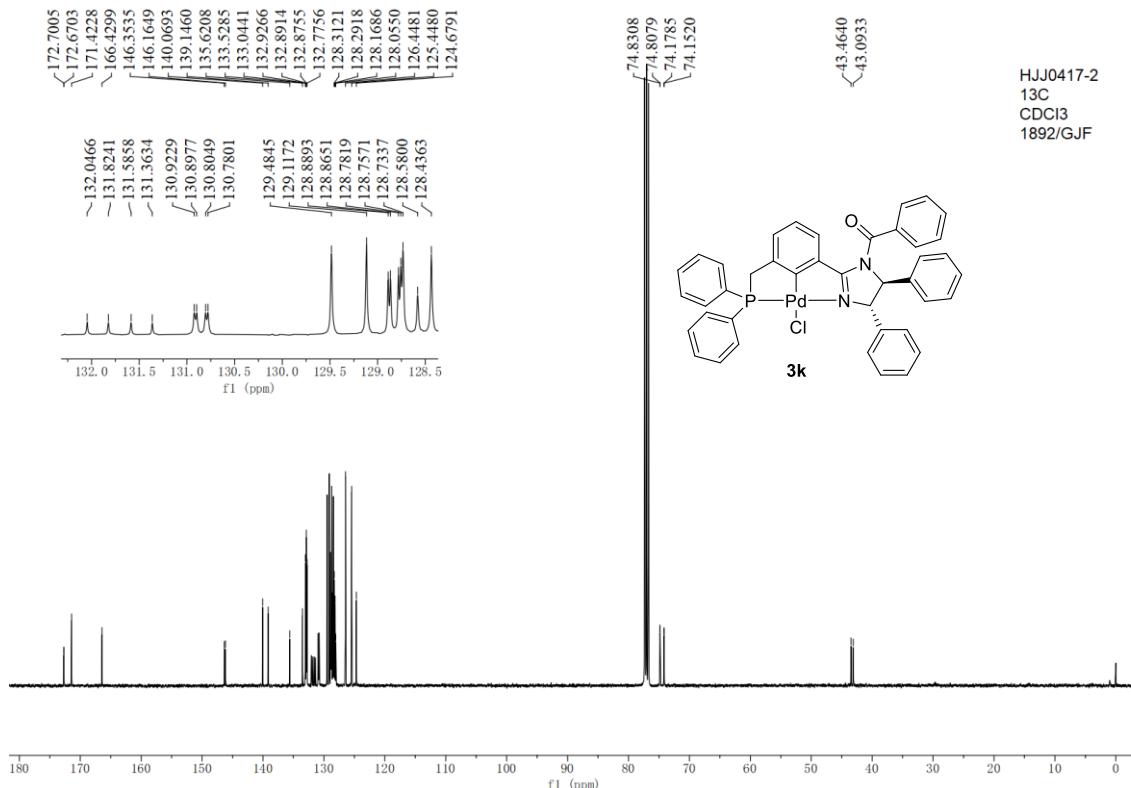


¹H NMR spectrum of **3j** (400 MHz, CDCl₃)

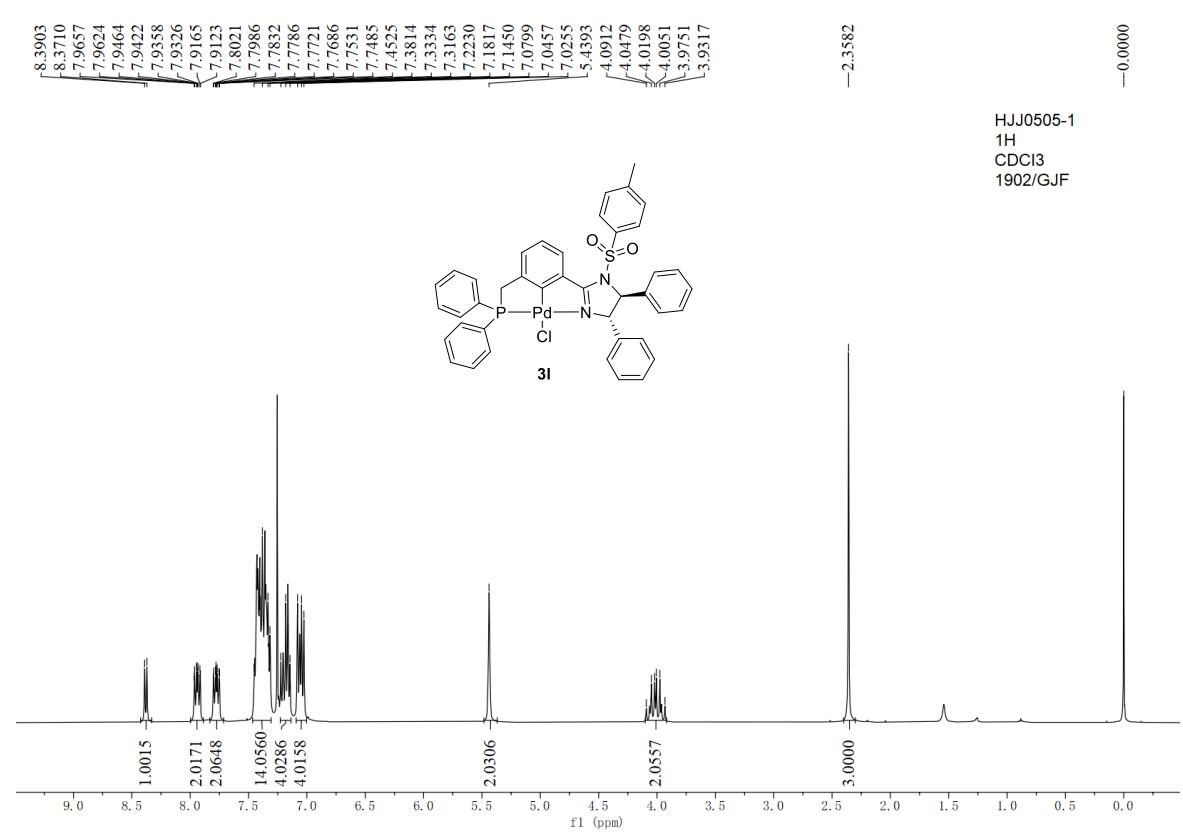
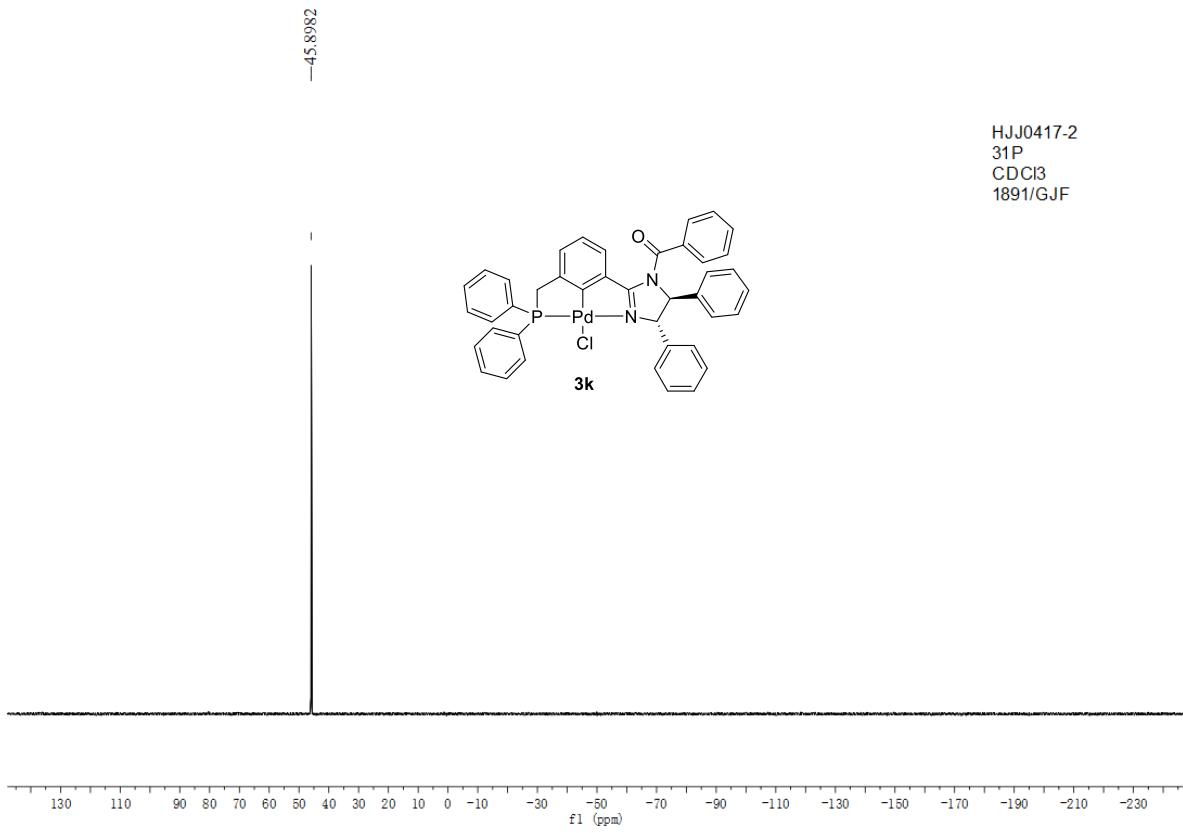


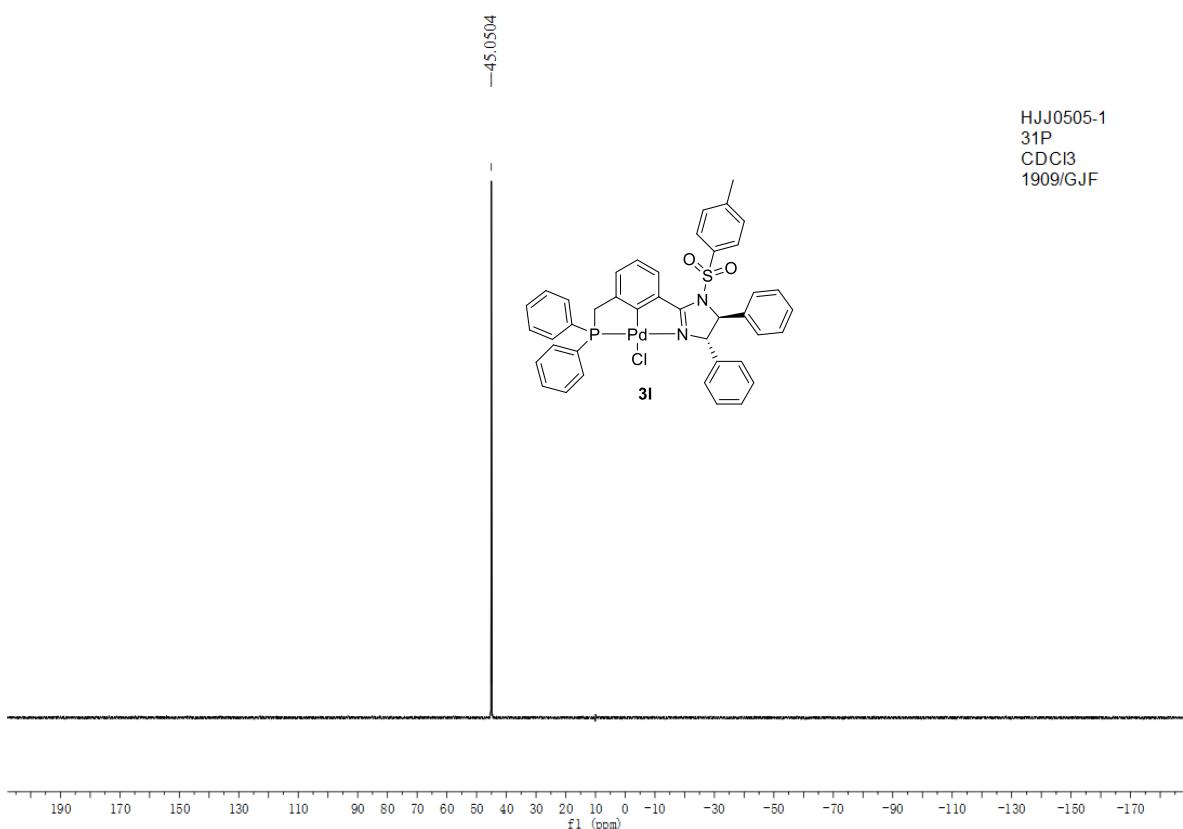
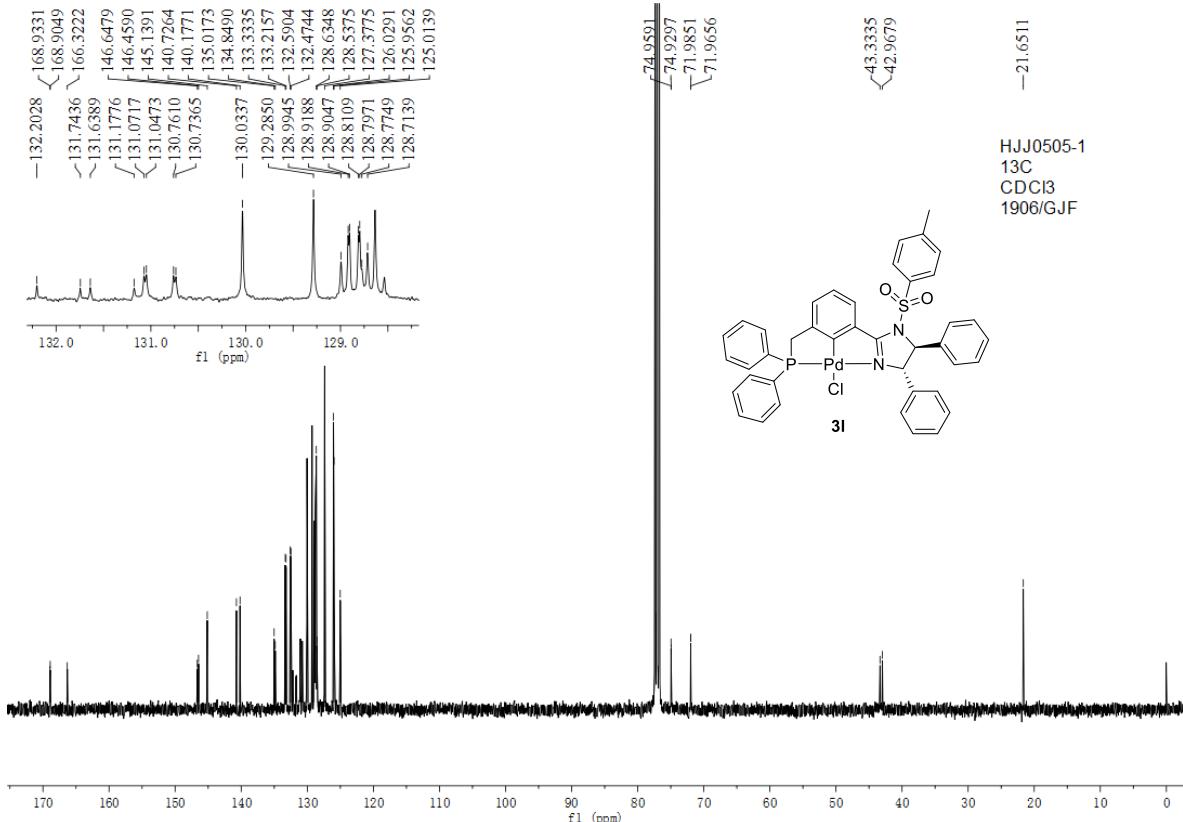


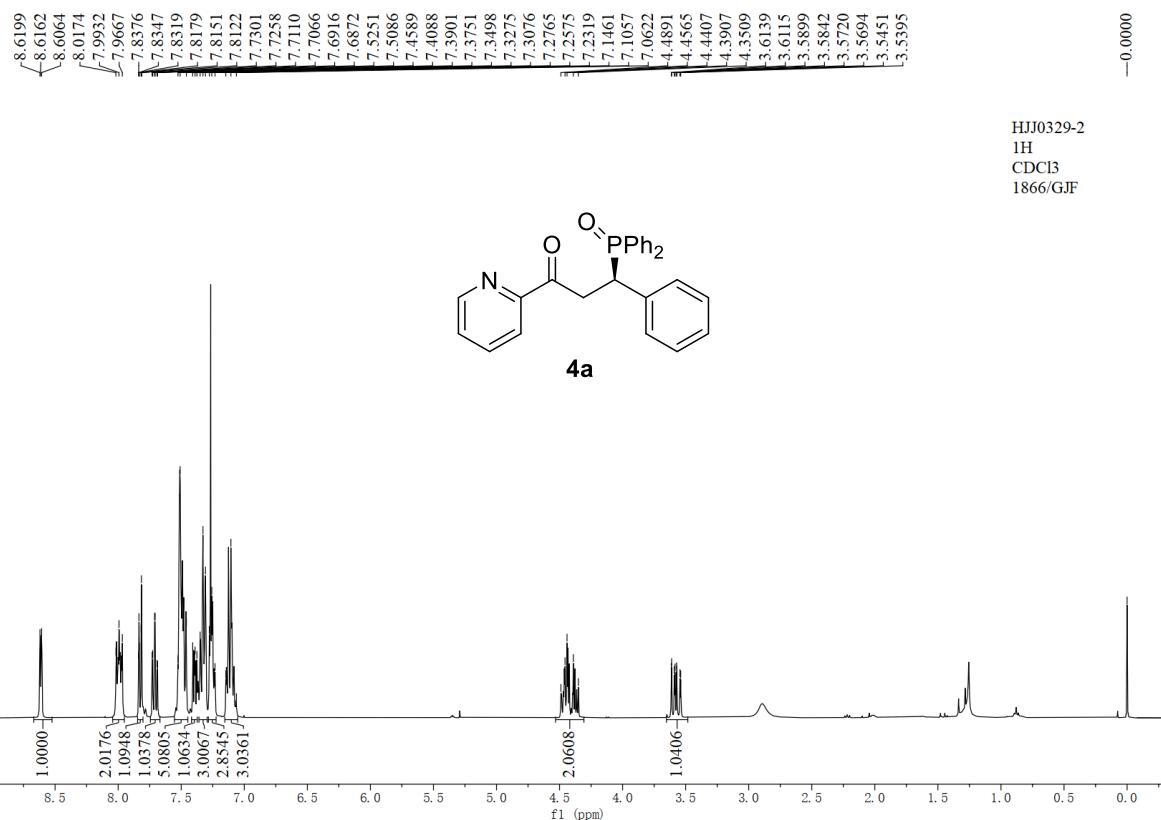
¹H NMR spectrum of **3k** (400 MHz, CDCl₃)



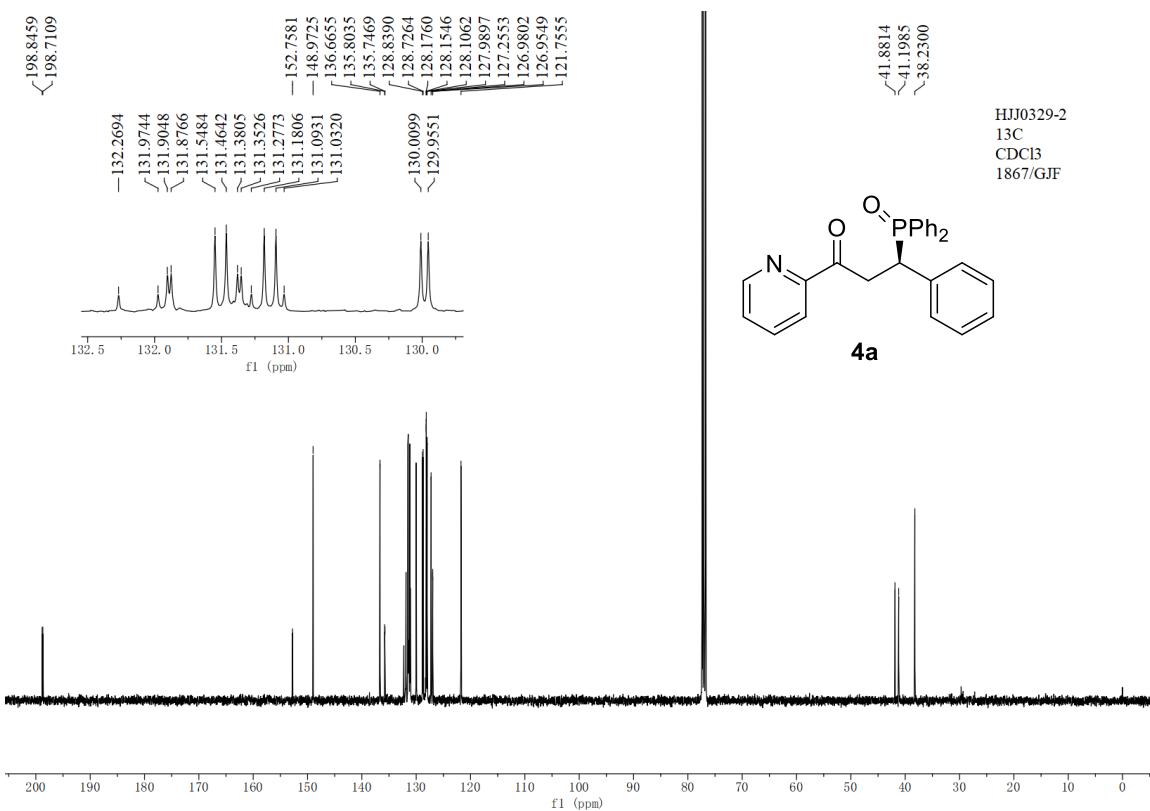
¹³C{¹H} NMR spectrum of **3k** (100 MHz, CDCl₃)



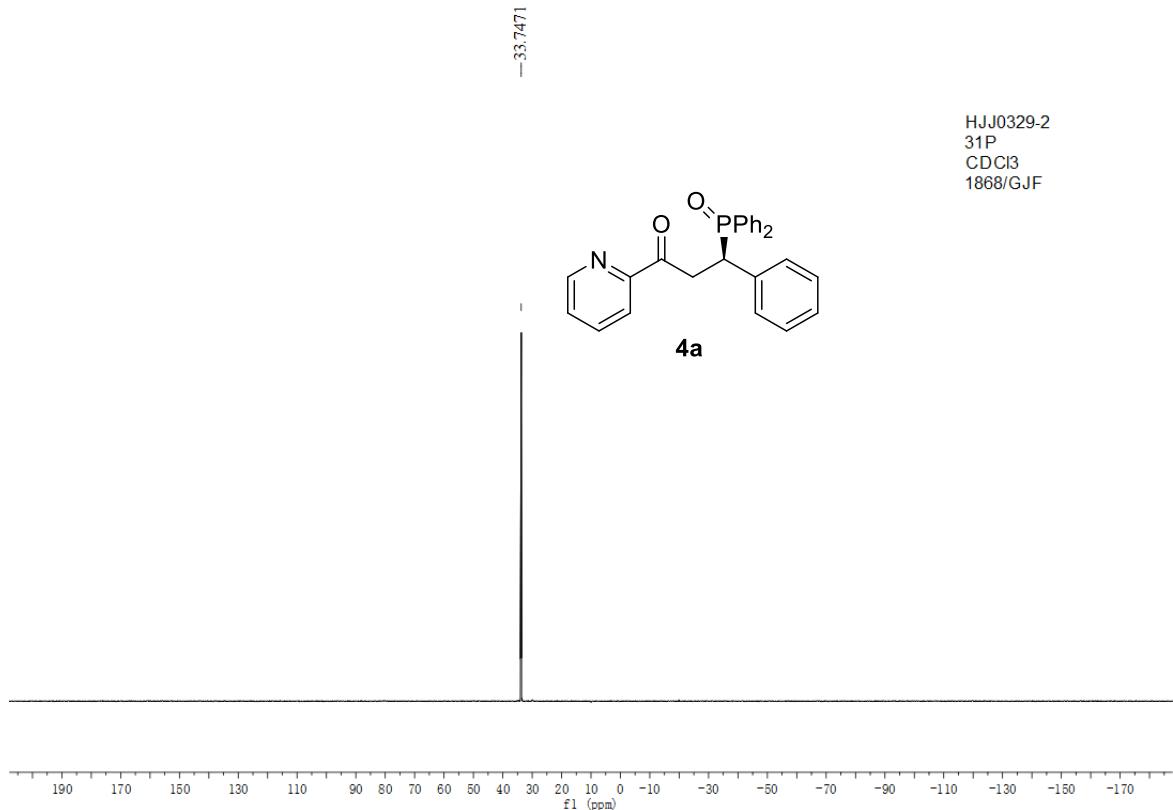




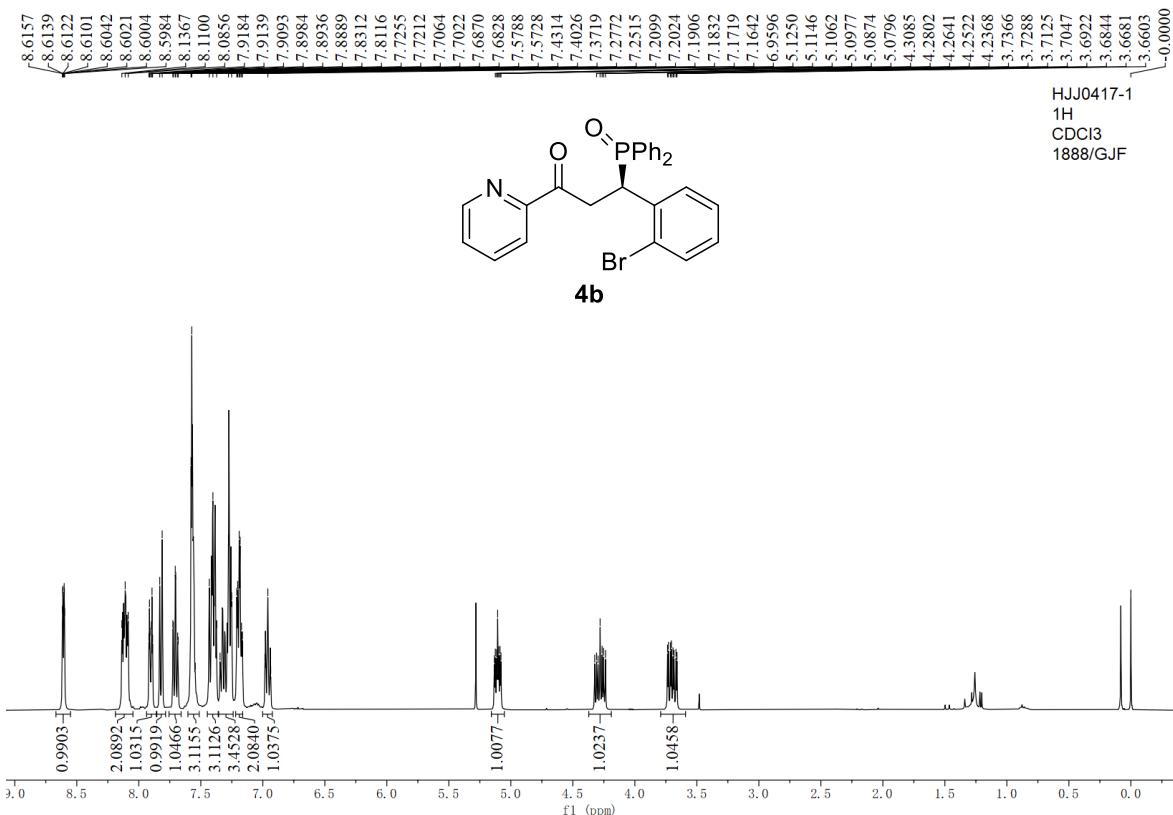
¹H NMR spectrum of **4a** (400 MHz, CDCl₃)



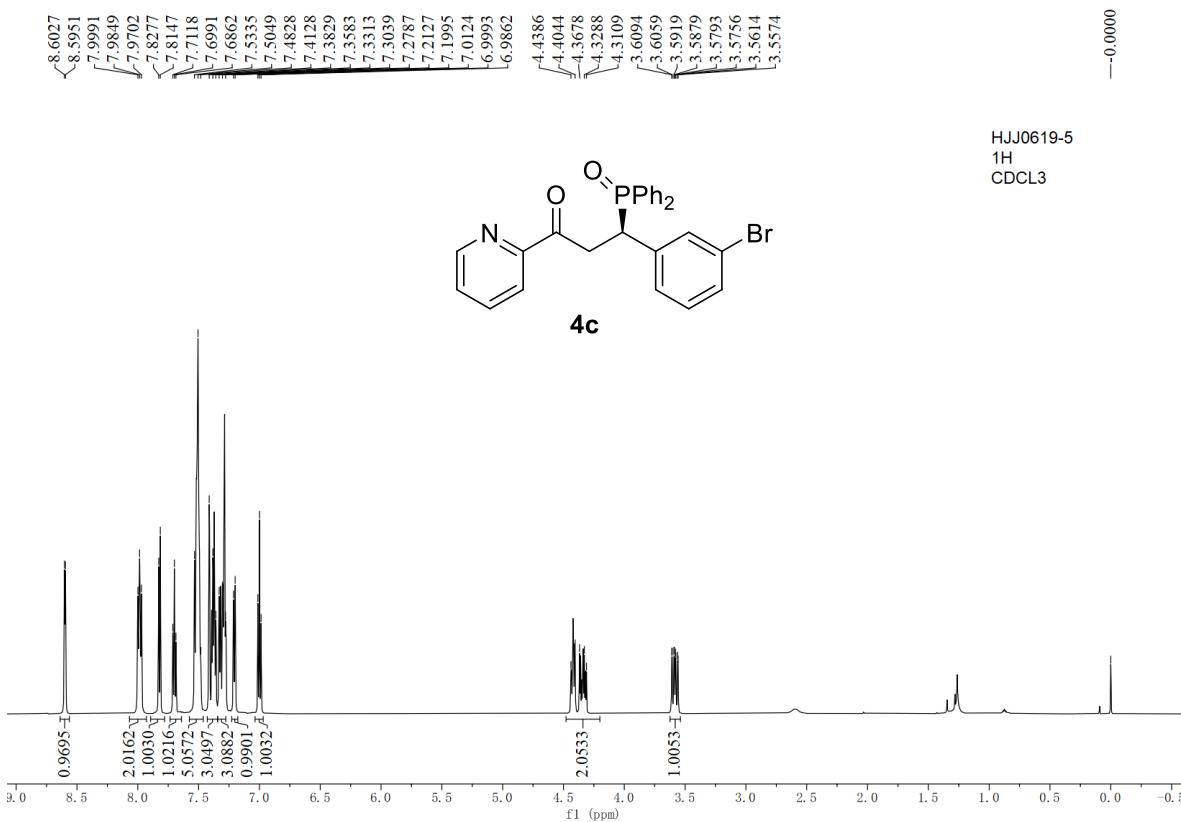
¹³C{¹H} NMR spectrum of **4a** (100 MHz, CDCl₃)



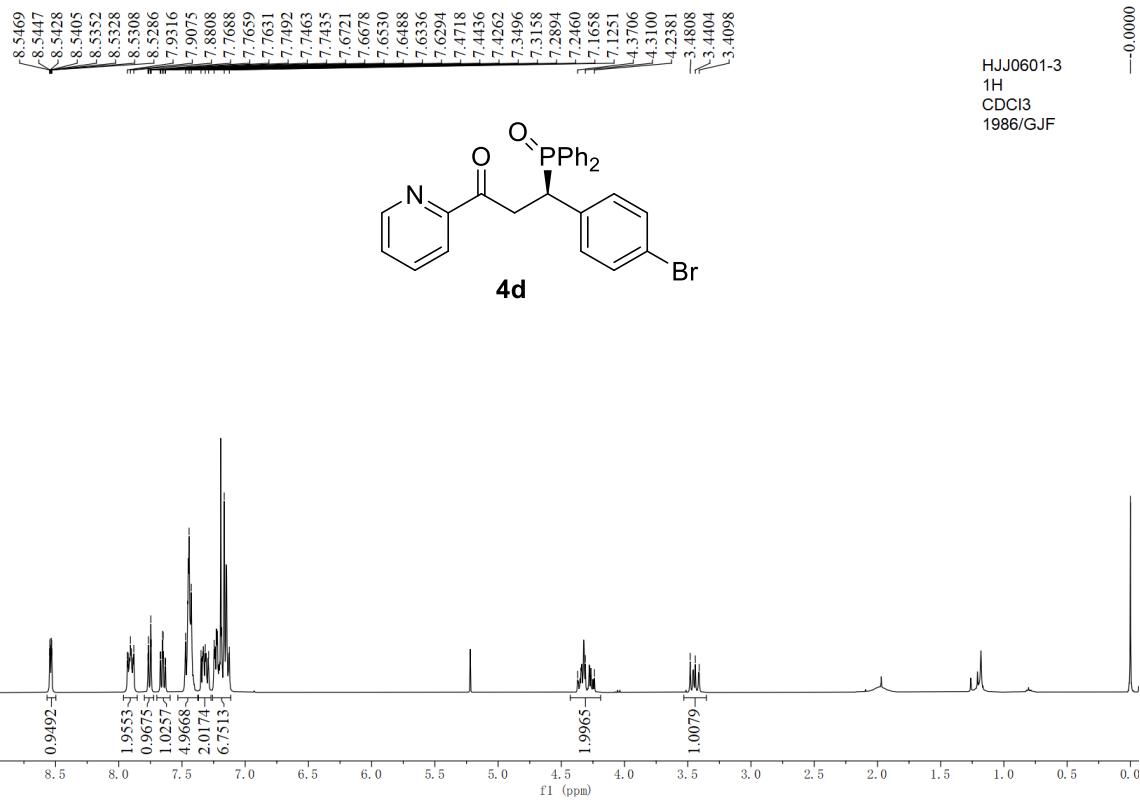
³¹P{¹H} NMR spectrum of **4a** (162 MHz, CDCl₃)



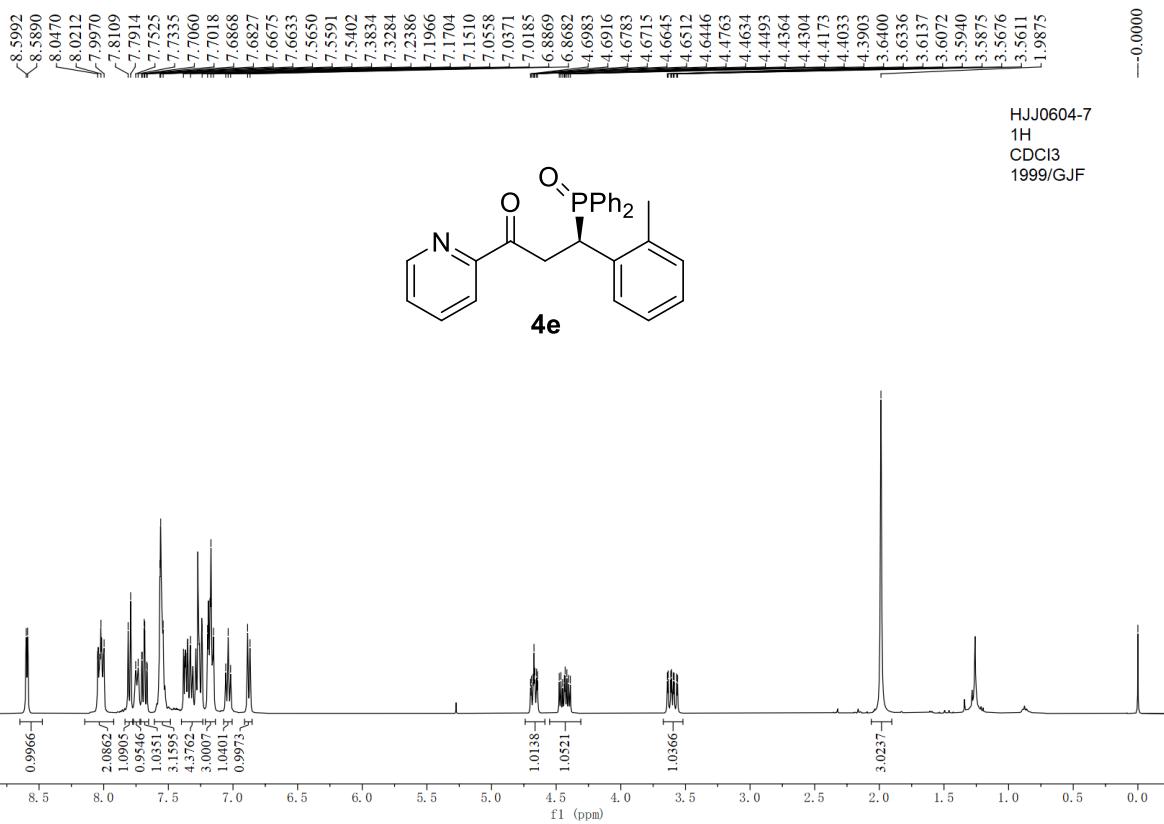
¹H NMR spectrum of **4b** (400 MHz, CDCl₃)



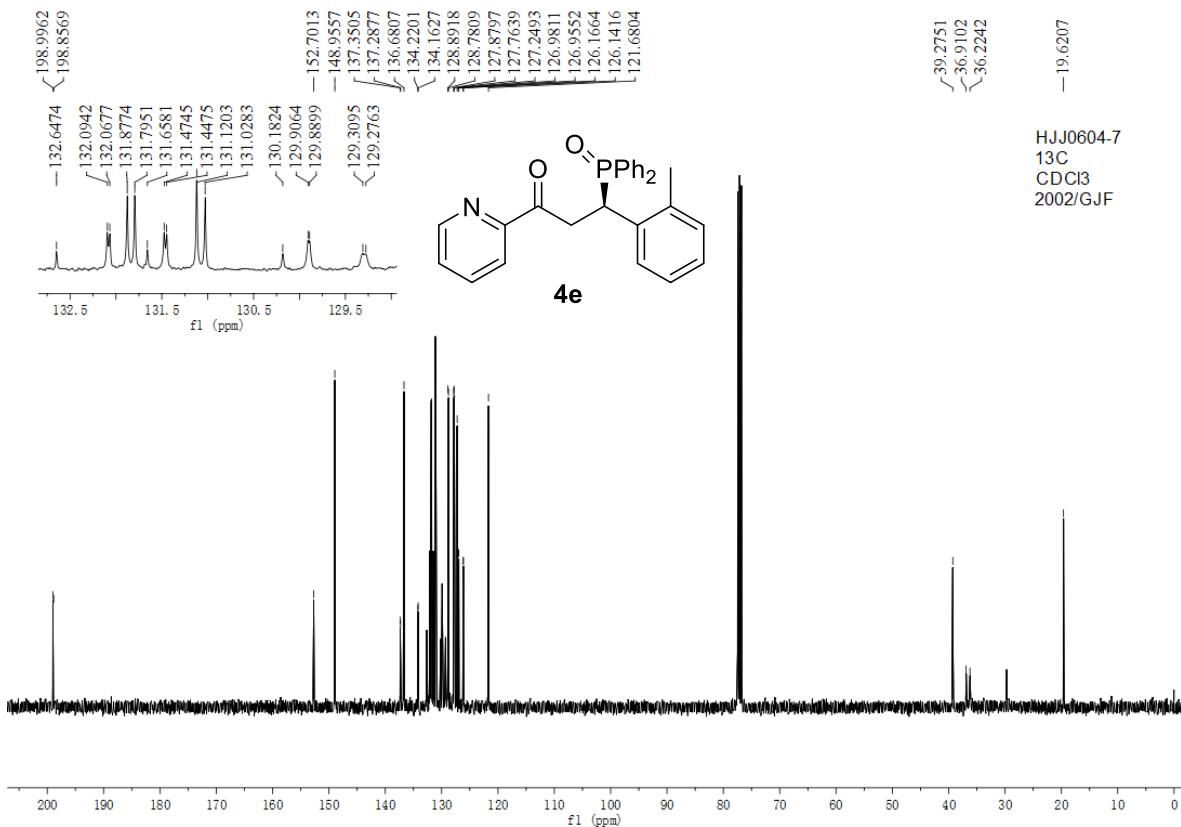
¹H NMR spectrum of **4c** (600 MHz, CDCl₃)



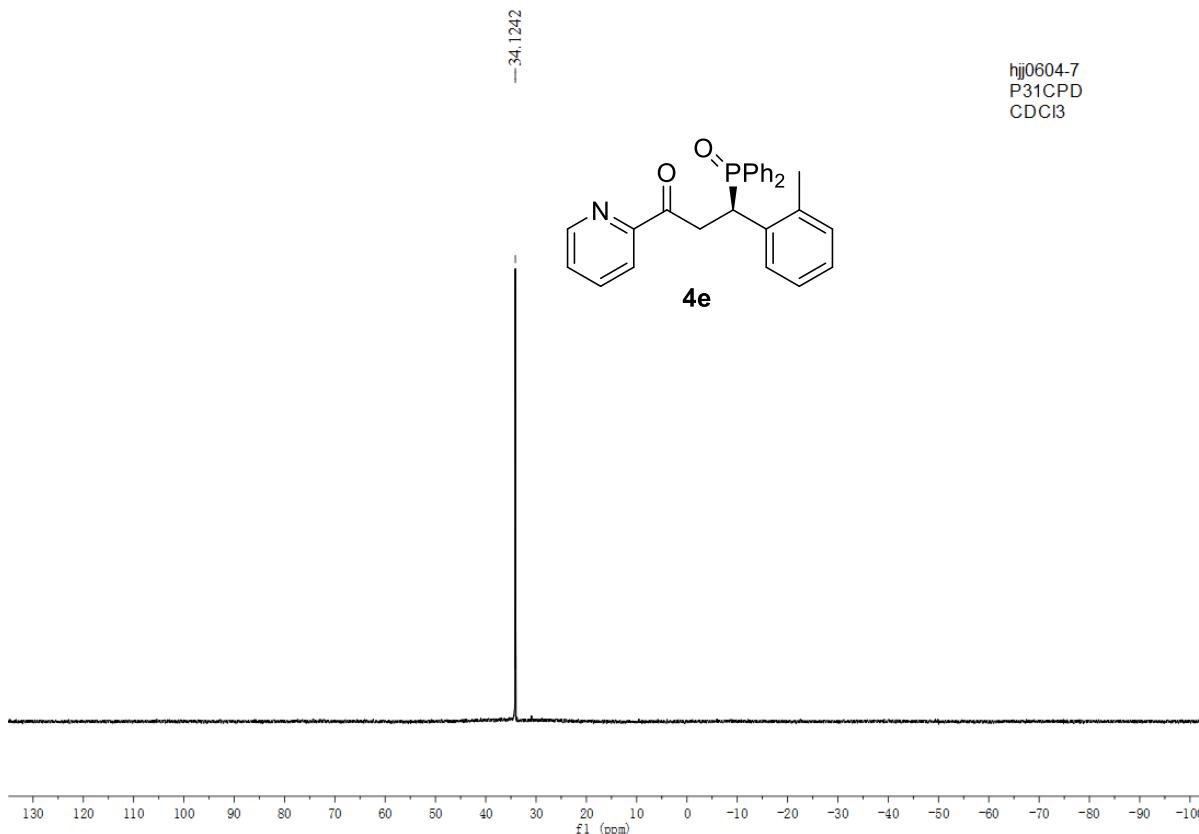
¹H NMR spectrum of **4d** (400 MHz, CDCl₃)



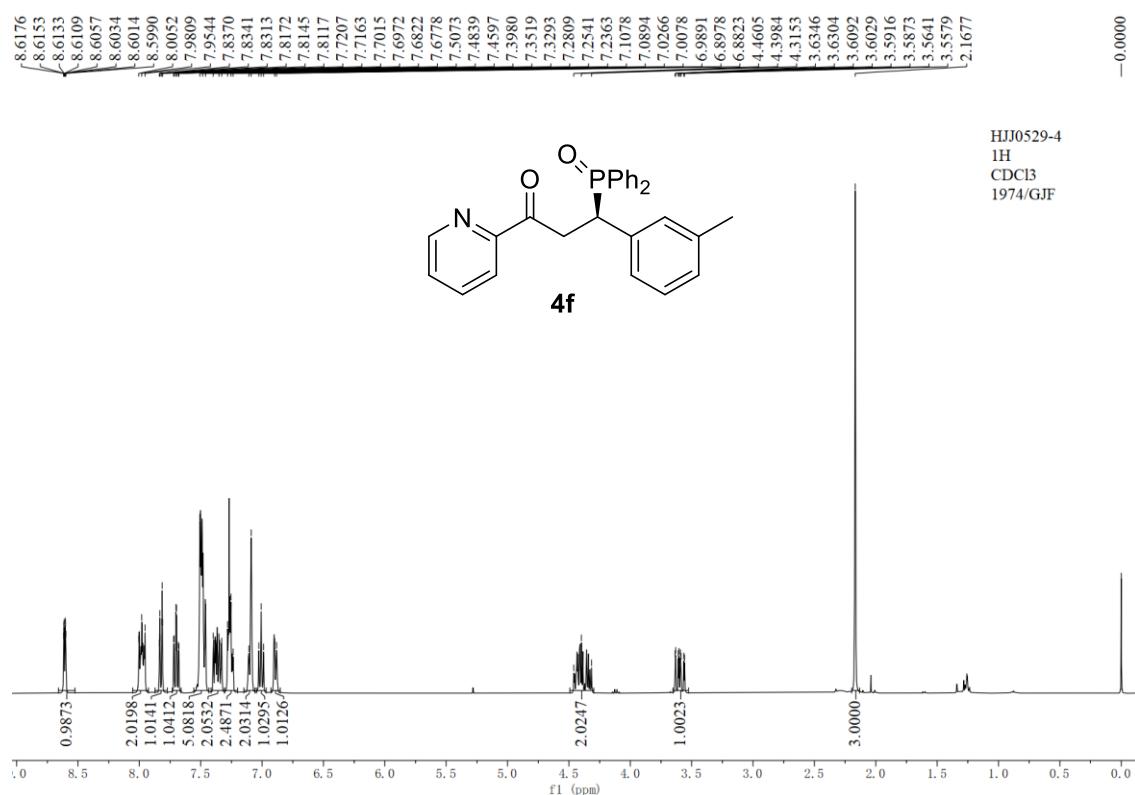
¹H NMR spectrum of **4e** (400 MHz, CDCl₃)



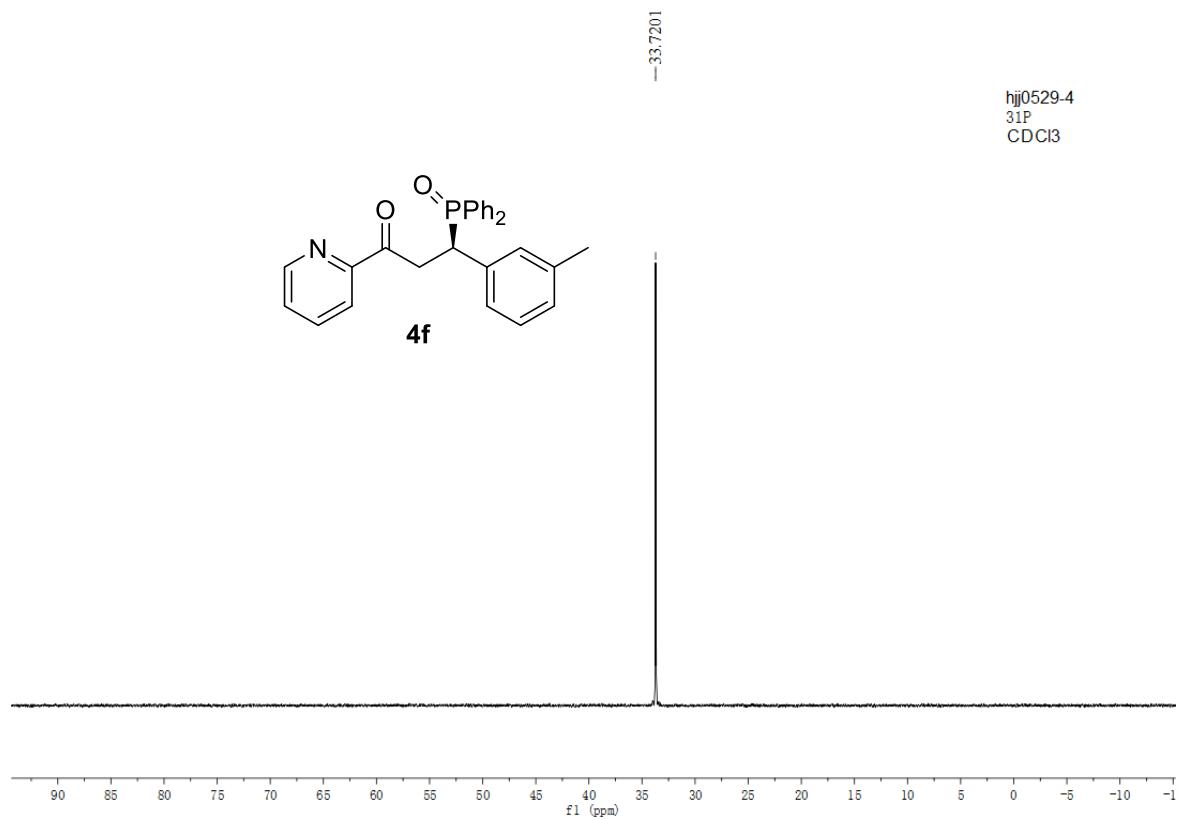
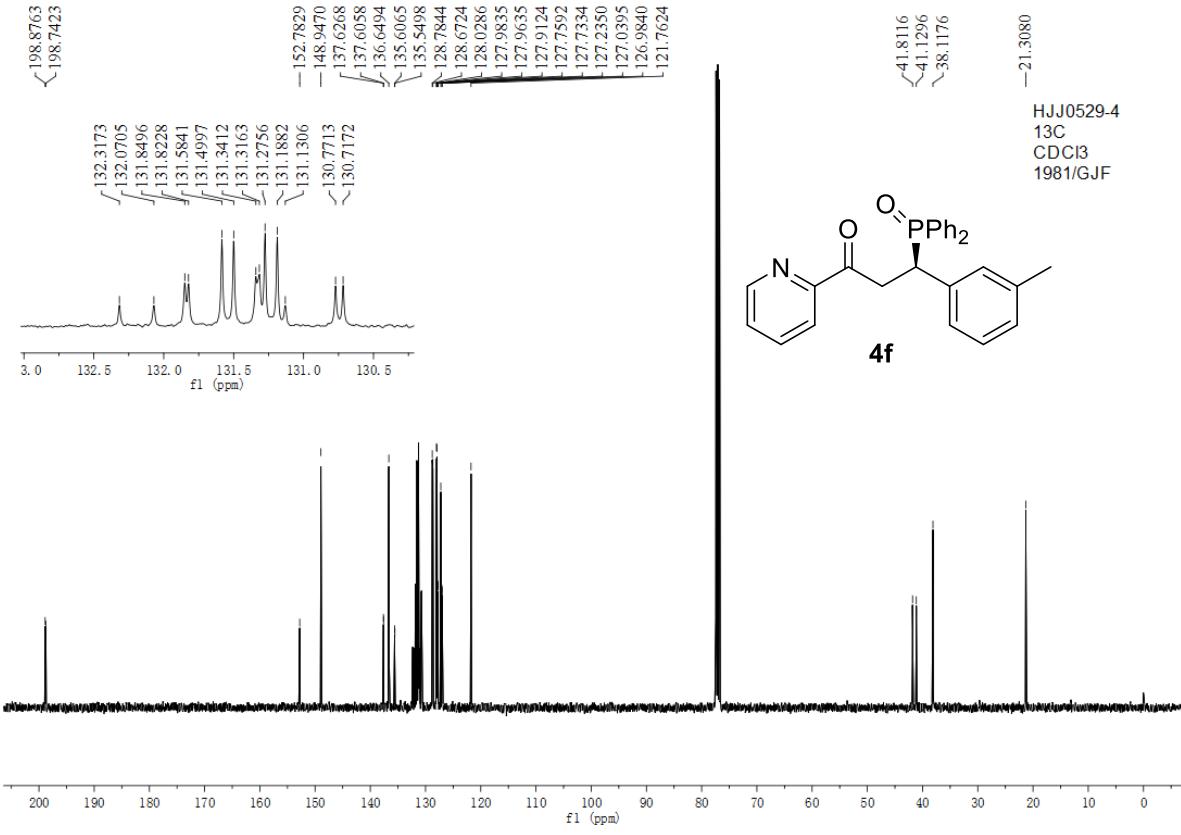
¹³C{¹H} NMR spectrum of **4e** (100 MHz, CDCl₃)

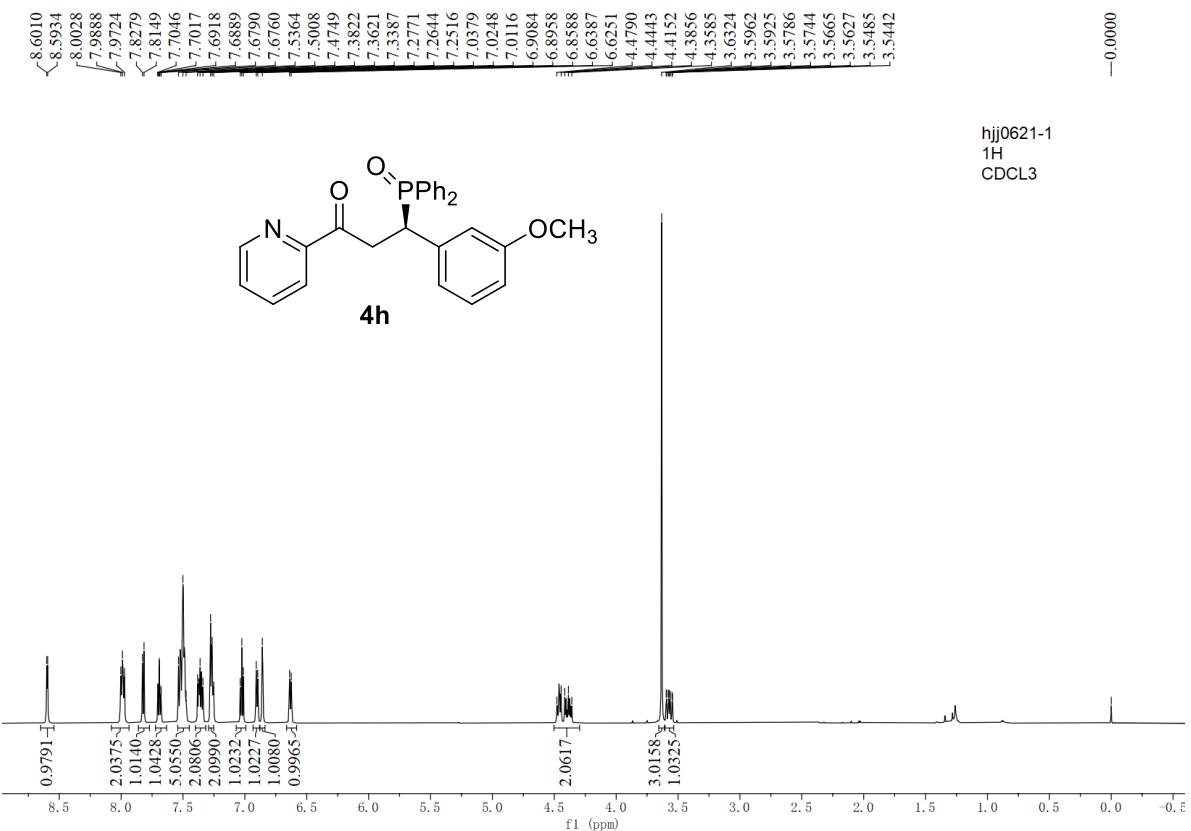
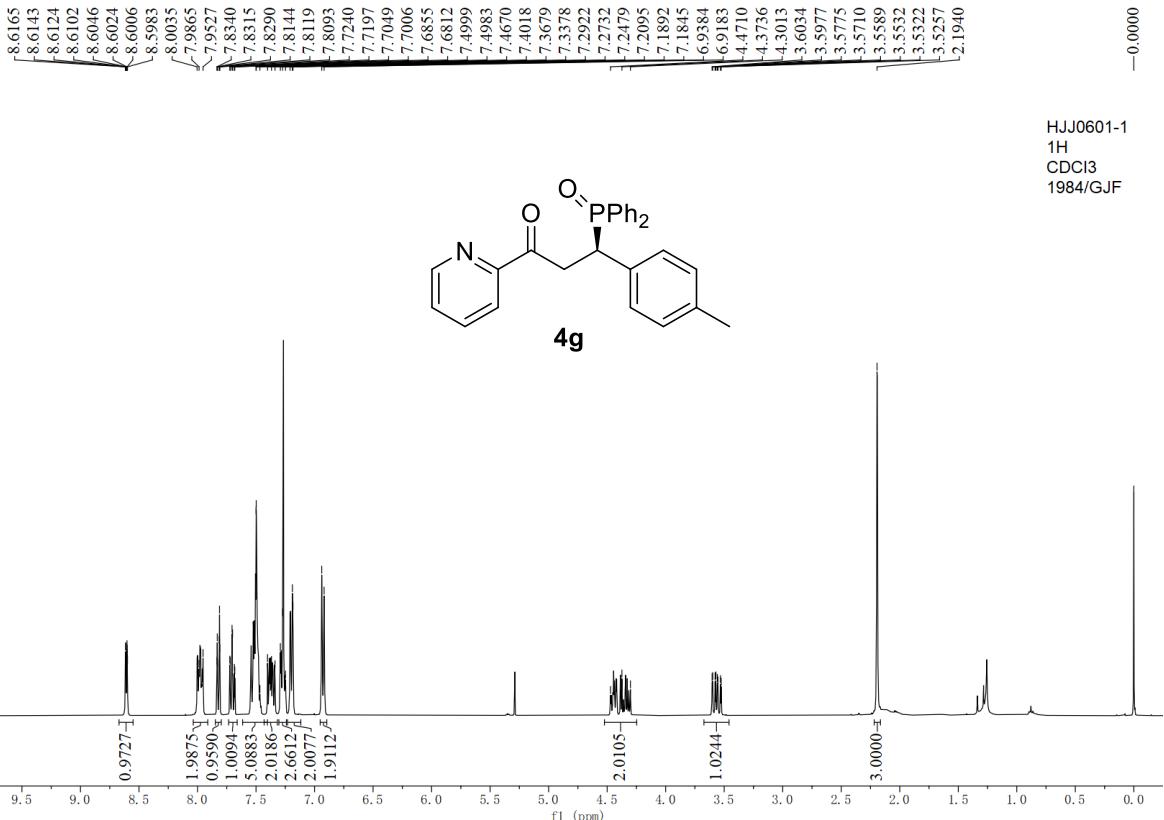


³¹P{¹H} NMR spectrum of **4e** (162 MHz, CDCl₃)

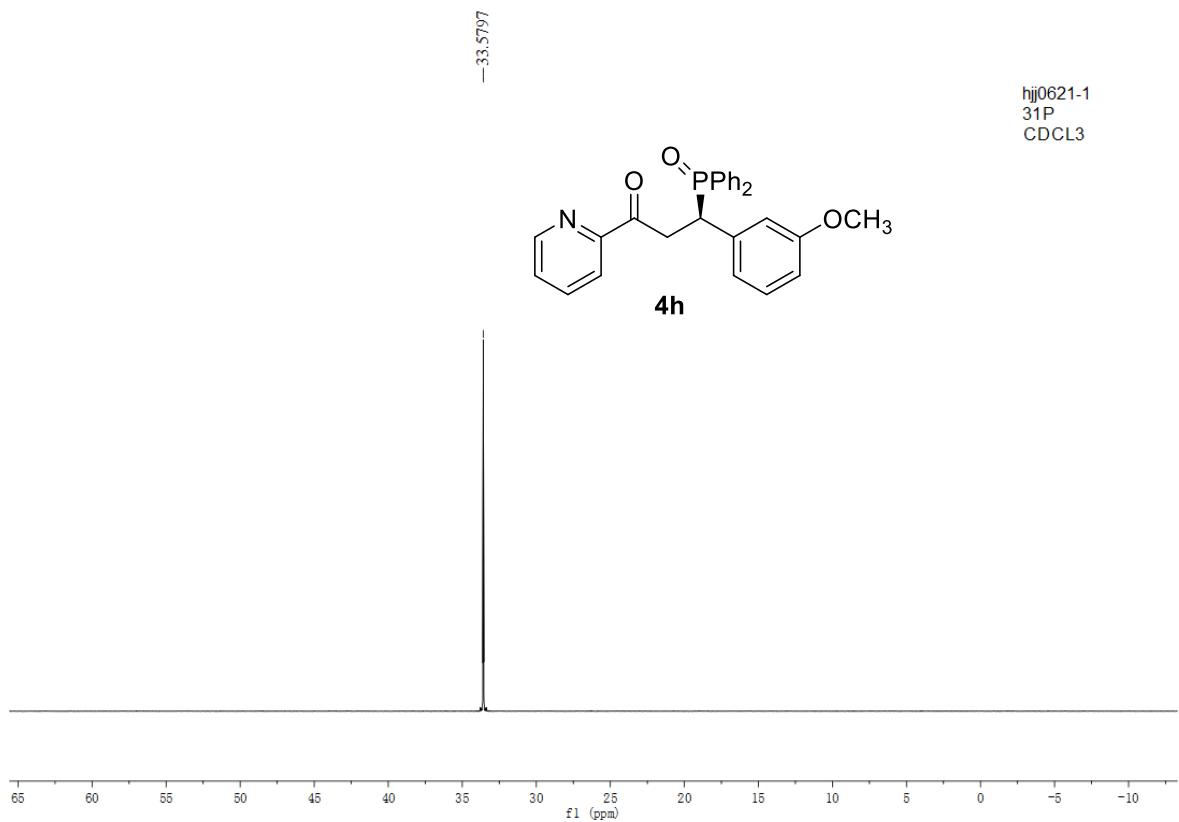
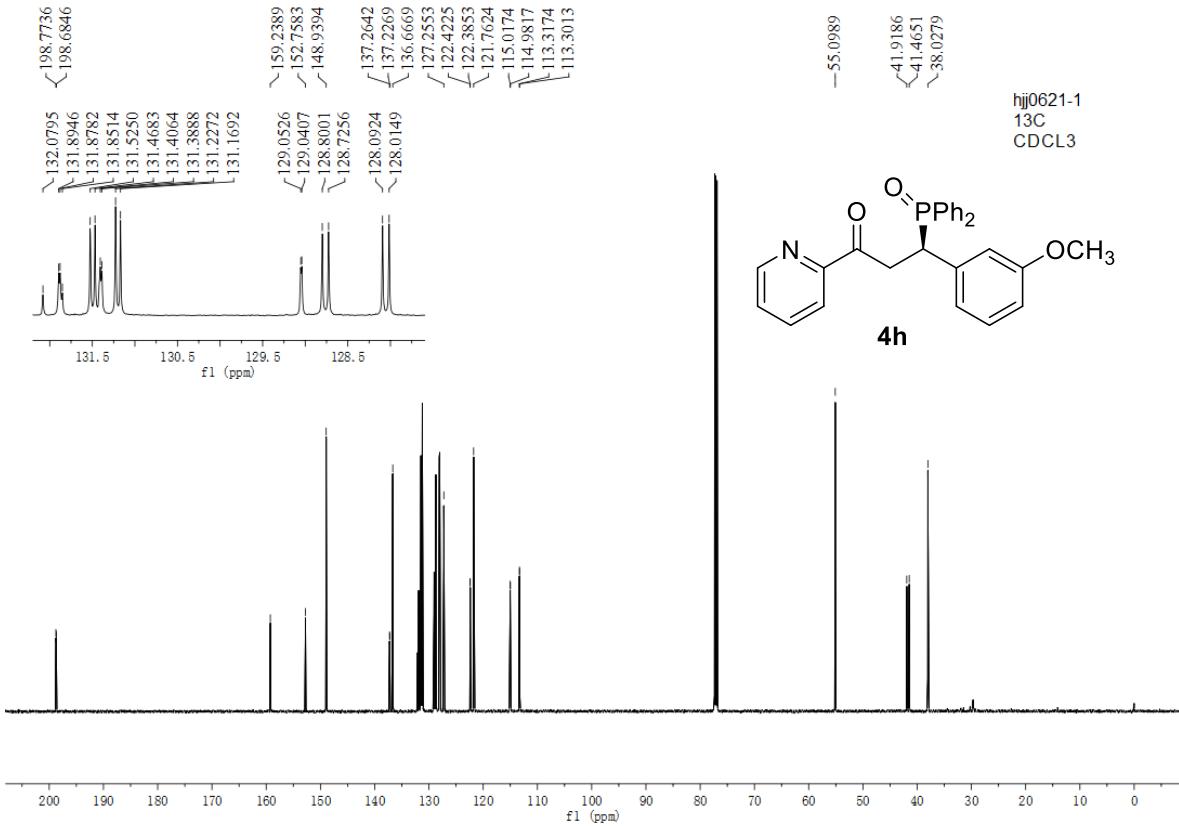


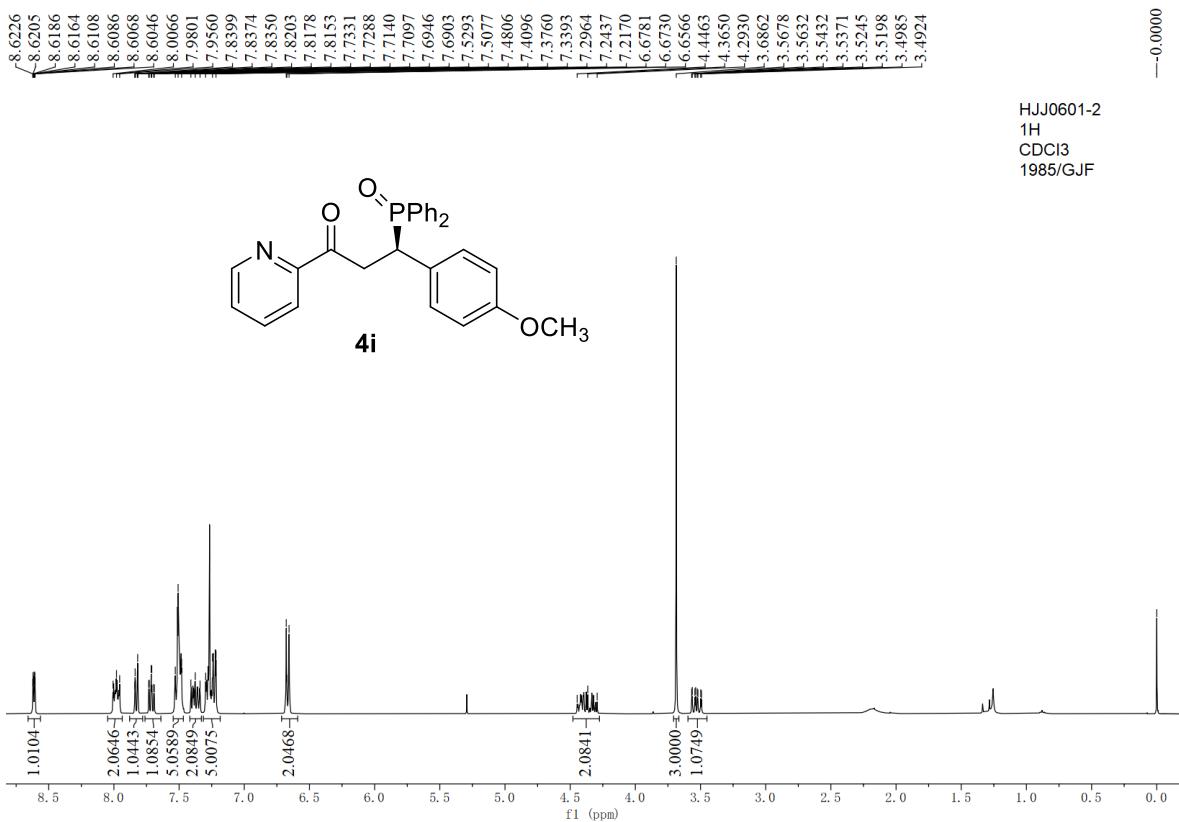
¹H NMR spectrum of **4f** (400 MHz, CDCl₃)



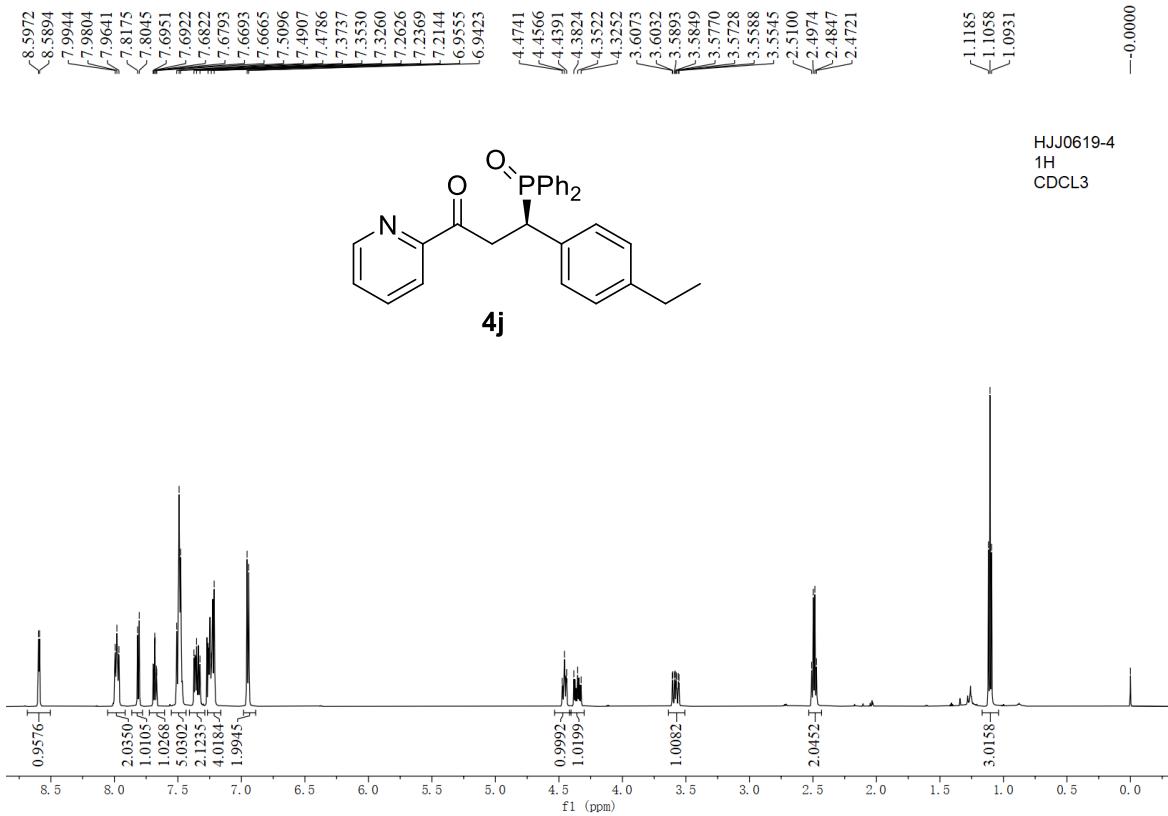


¹H NMR spectrum of **4h** (600 MHz, CDCl₃)

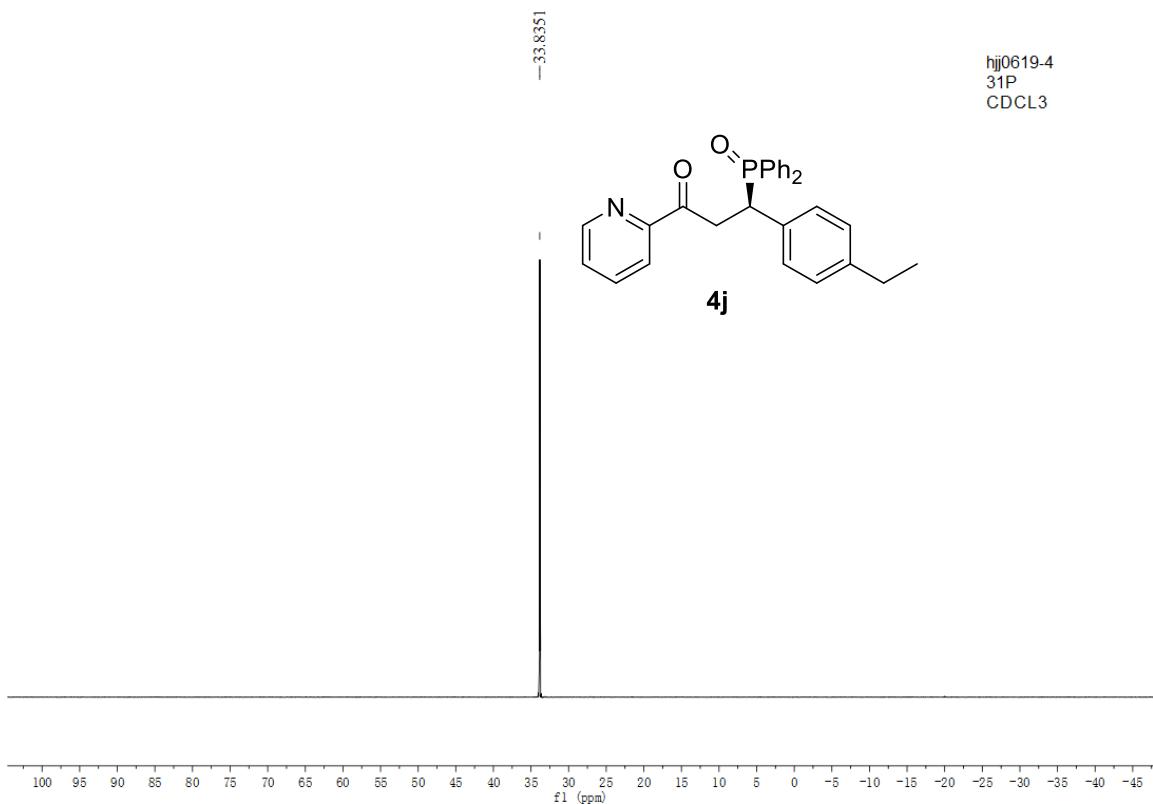
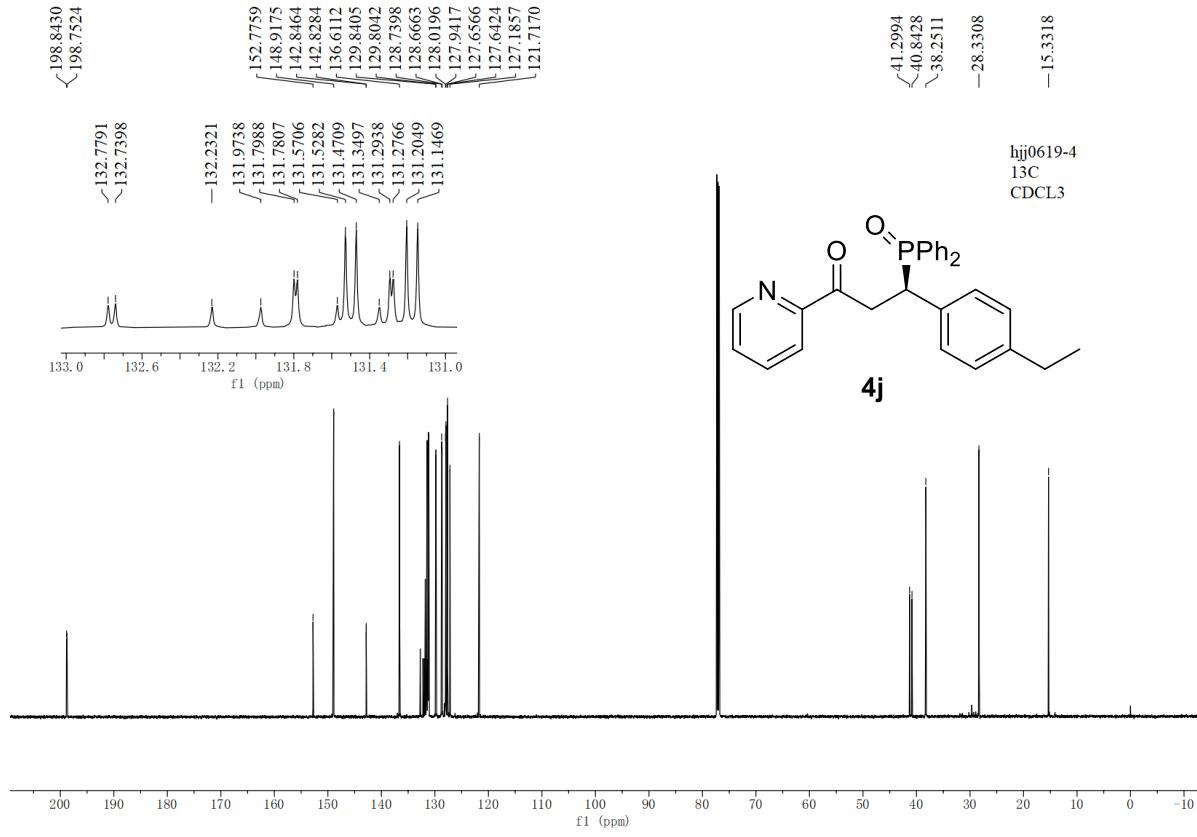


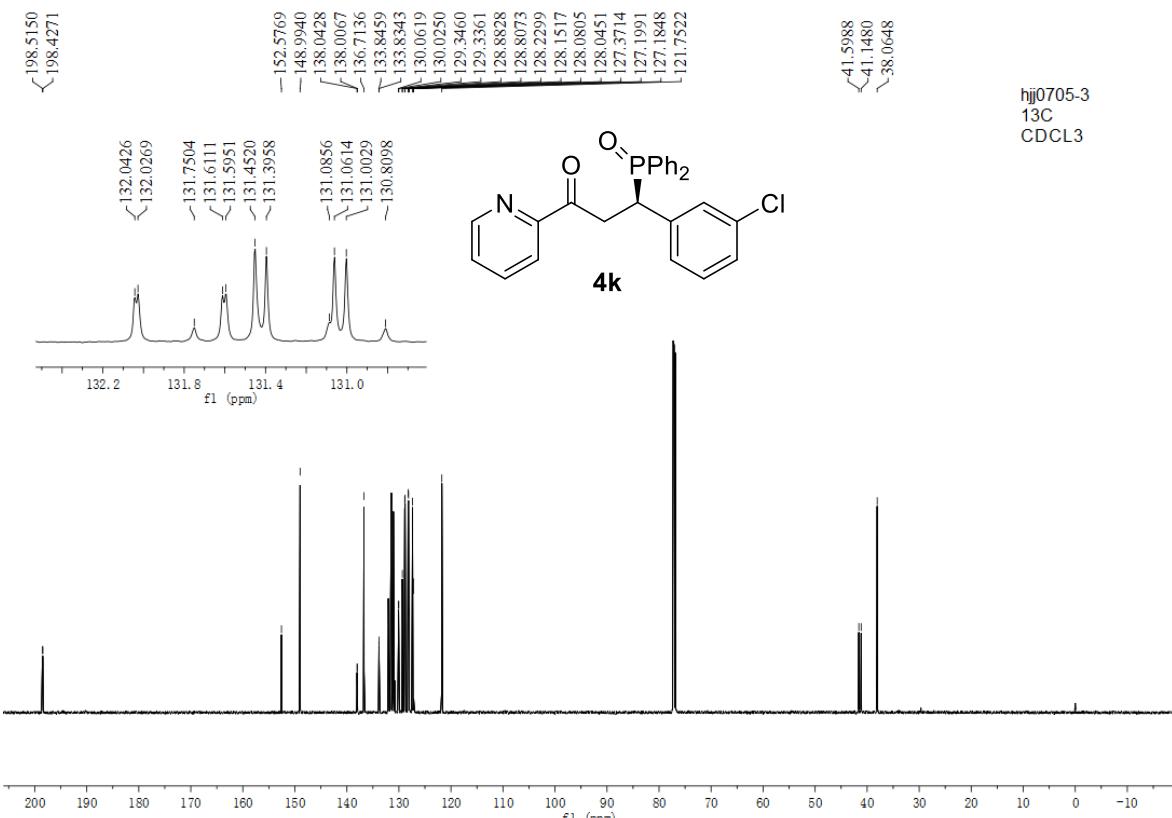
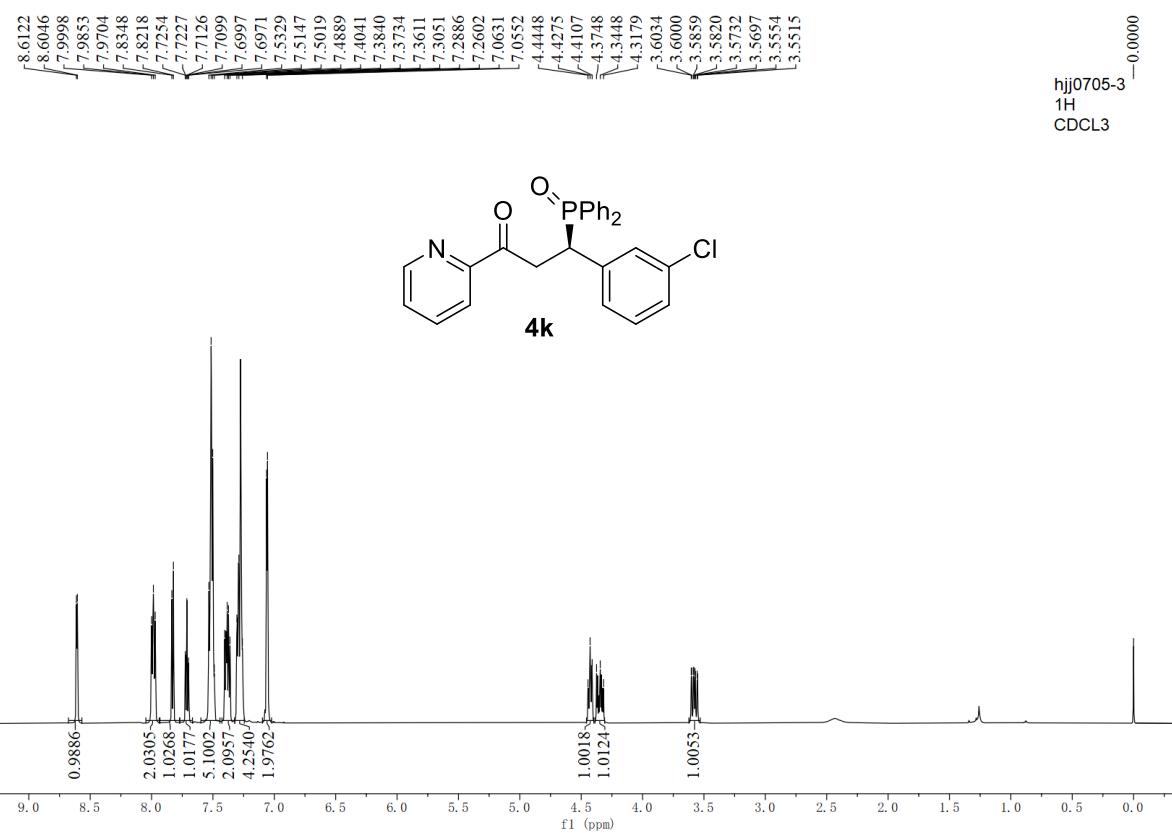


¹H NMR spectrum of **4i** (600 MHz, CDCl₃)



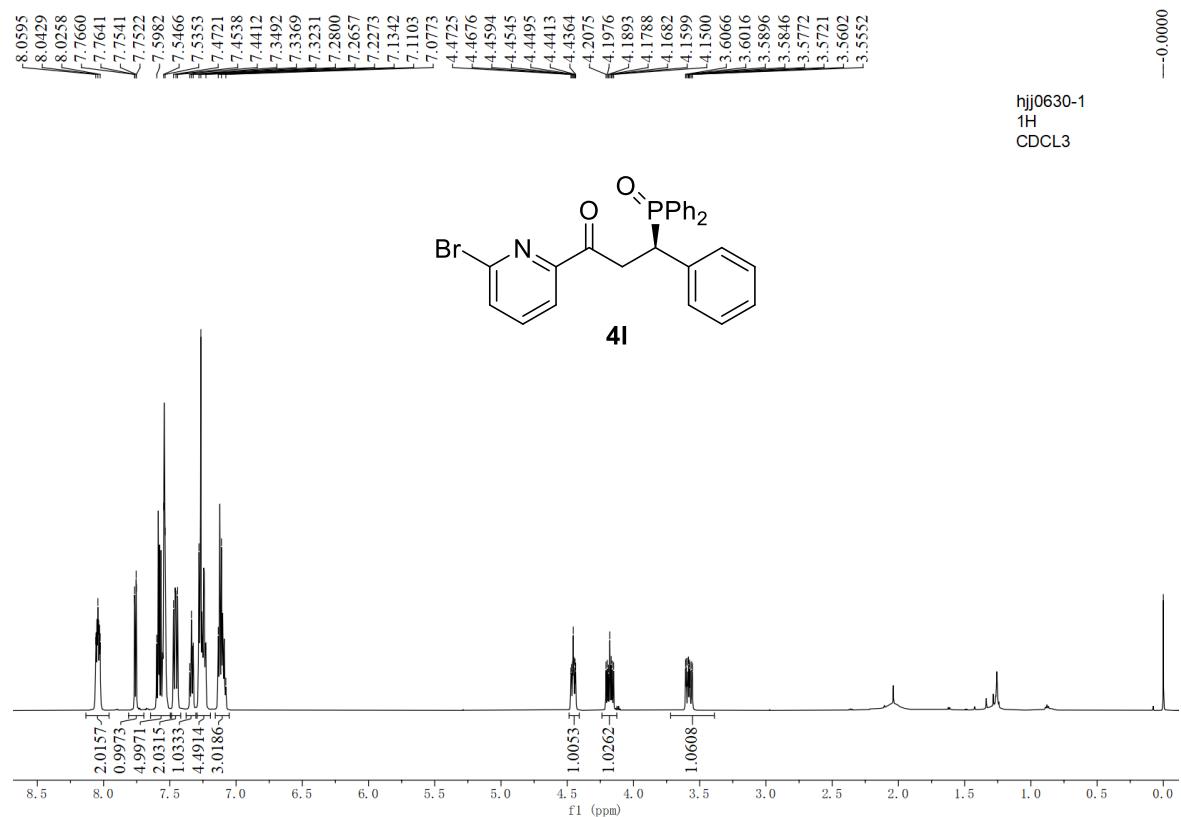
¹H NMR spectrum of **4j** (600 MHz, CDCl₃)



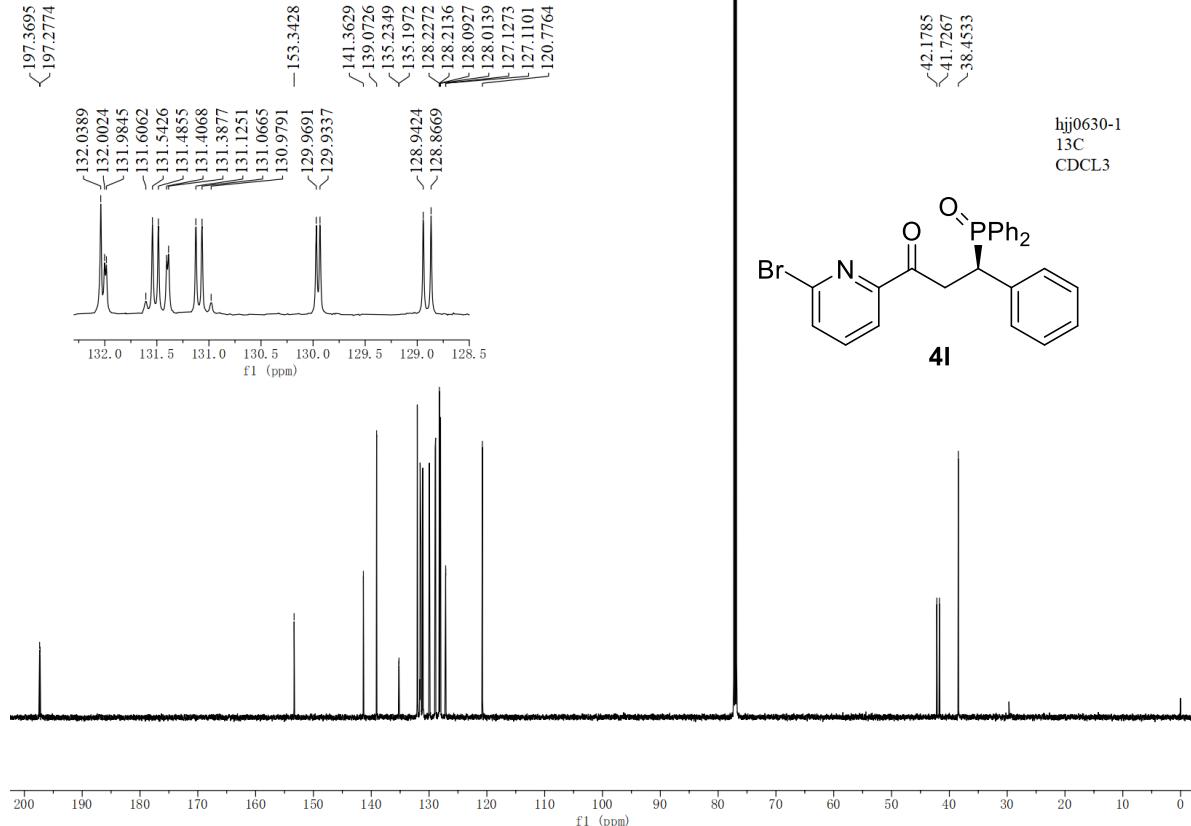


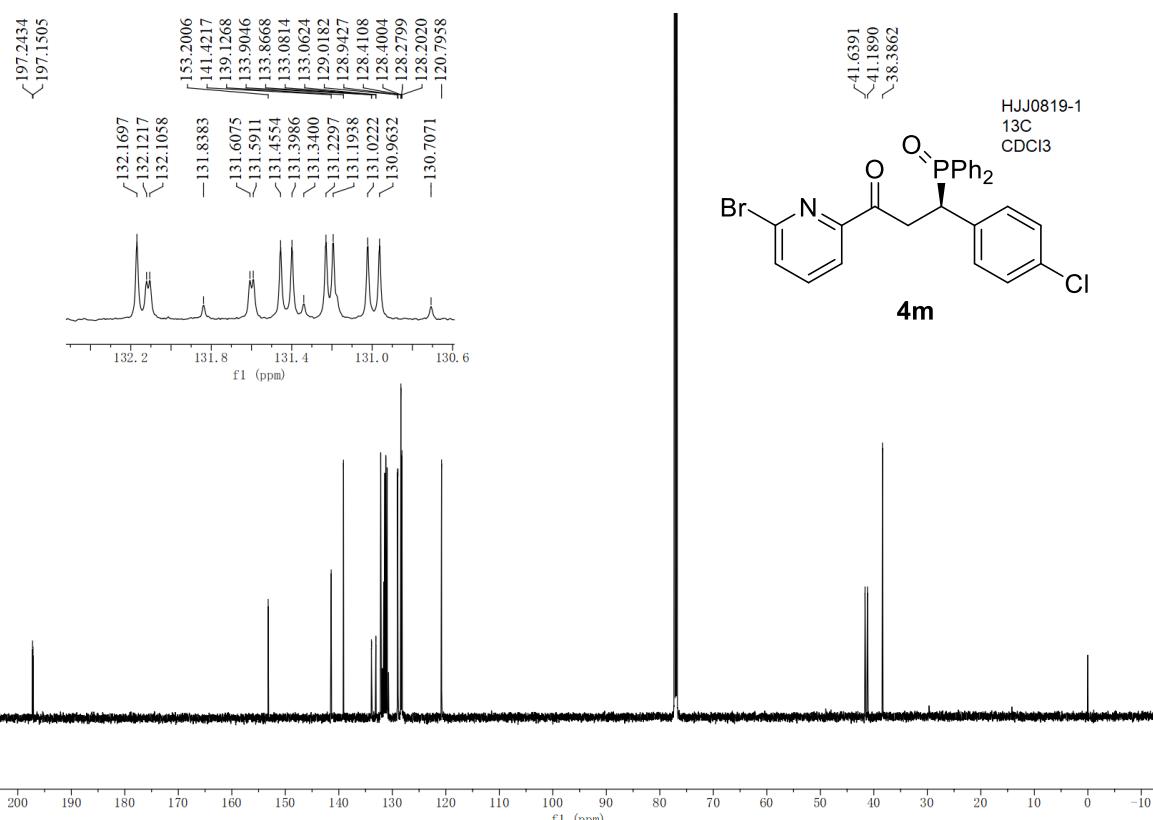
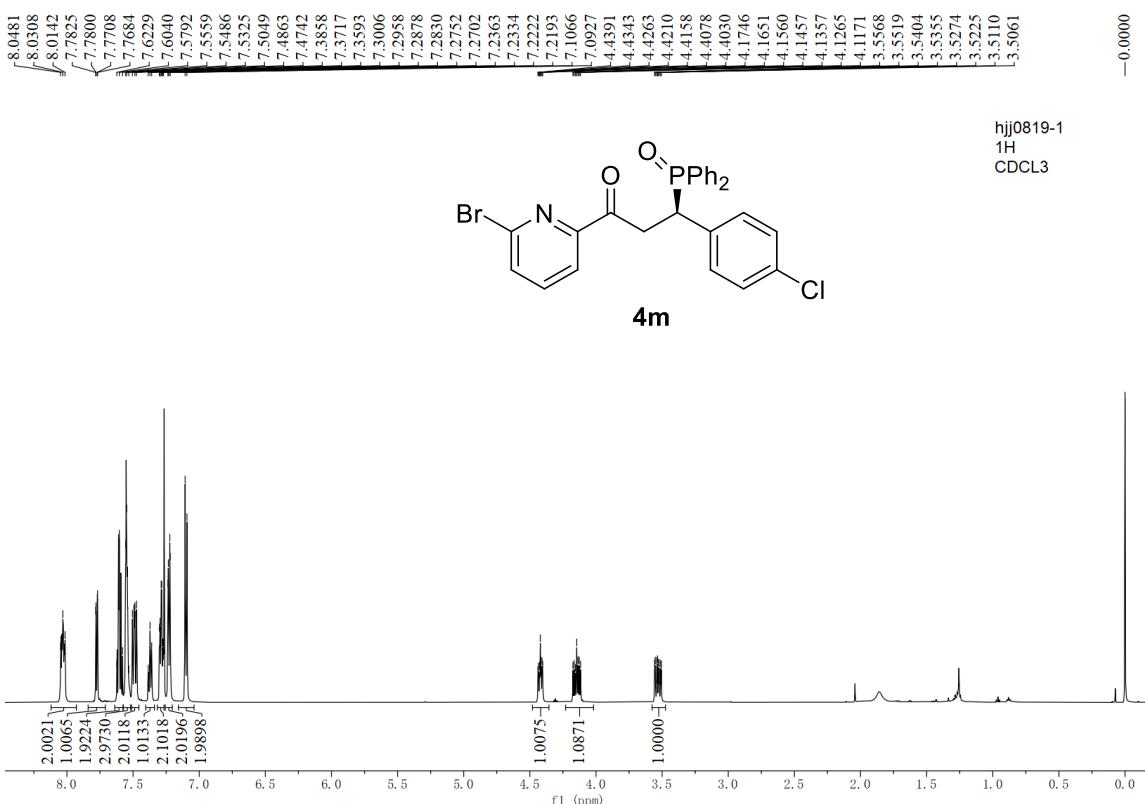


³¹P{¹H} NMR spectrum of **4k** (243 MHz, CDCl₃)

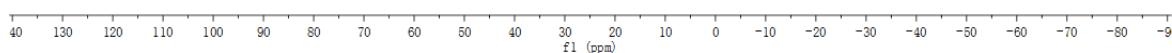
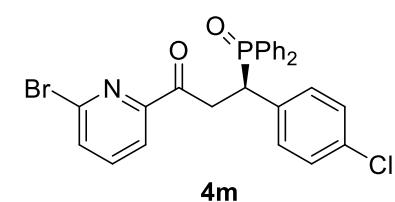


¹H NMR spectrum of **4l** (600 MHz, CDCl₃)



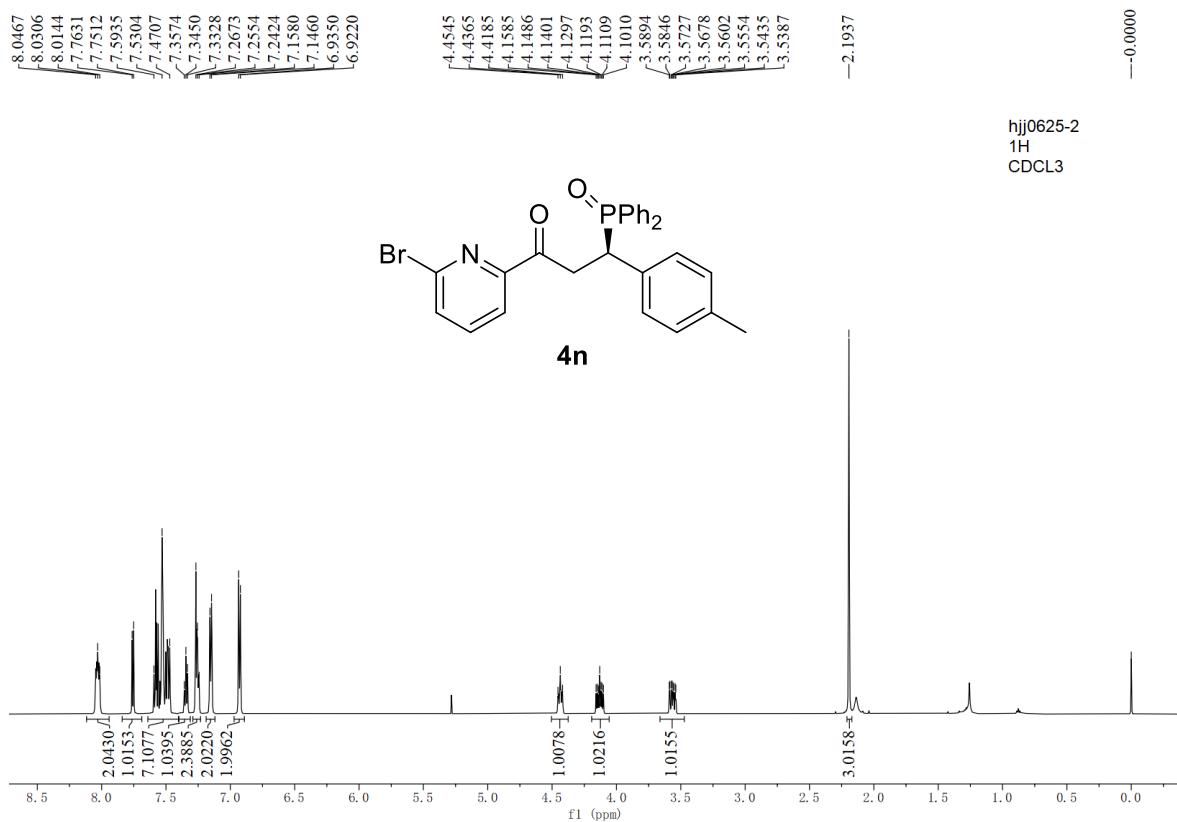
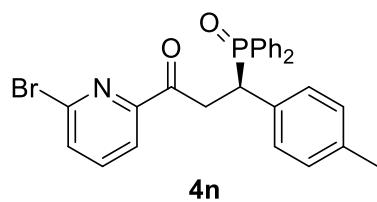


HJJ0819-1
31P
CDCl₃

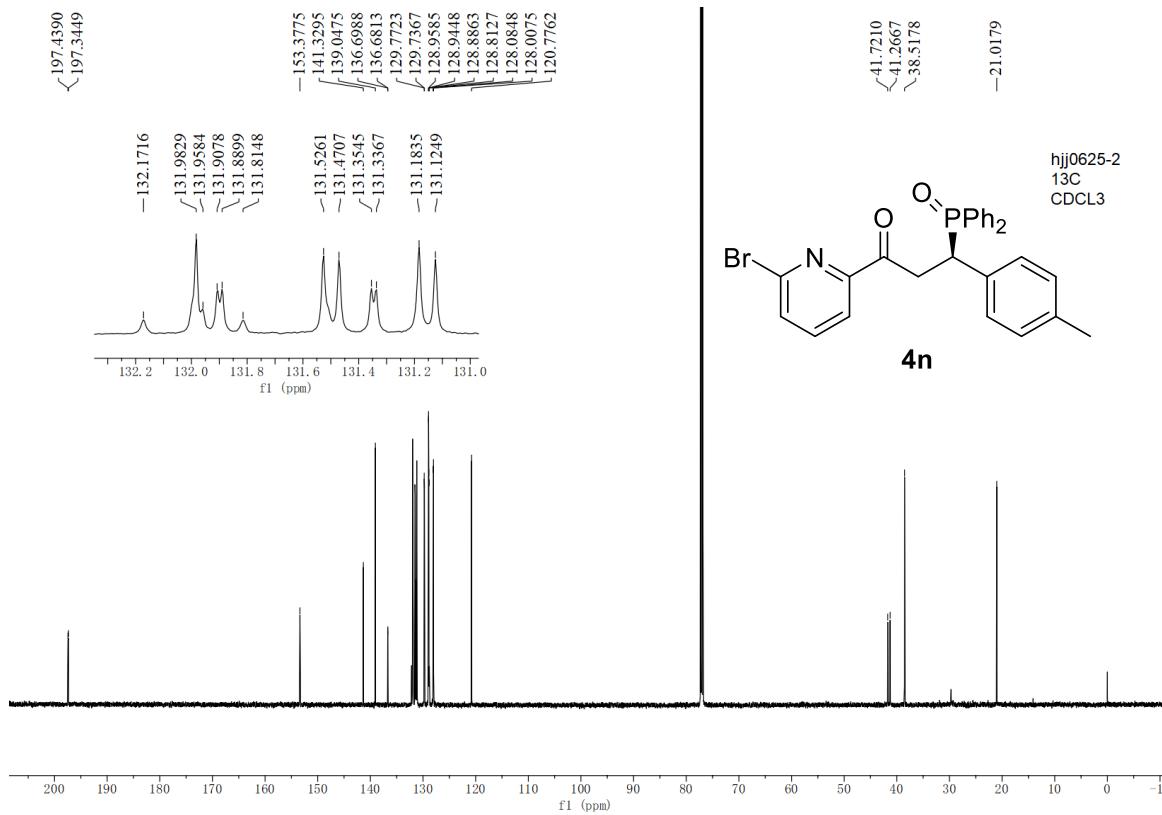


³¹P{¹H} NMR spectrum of **4m** (243 MHz, CDCl₃)

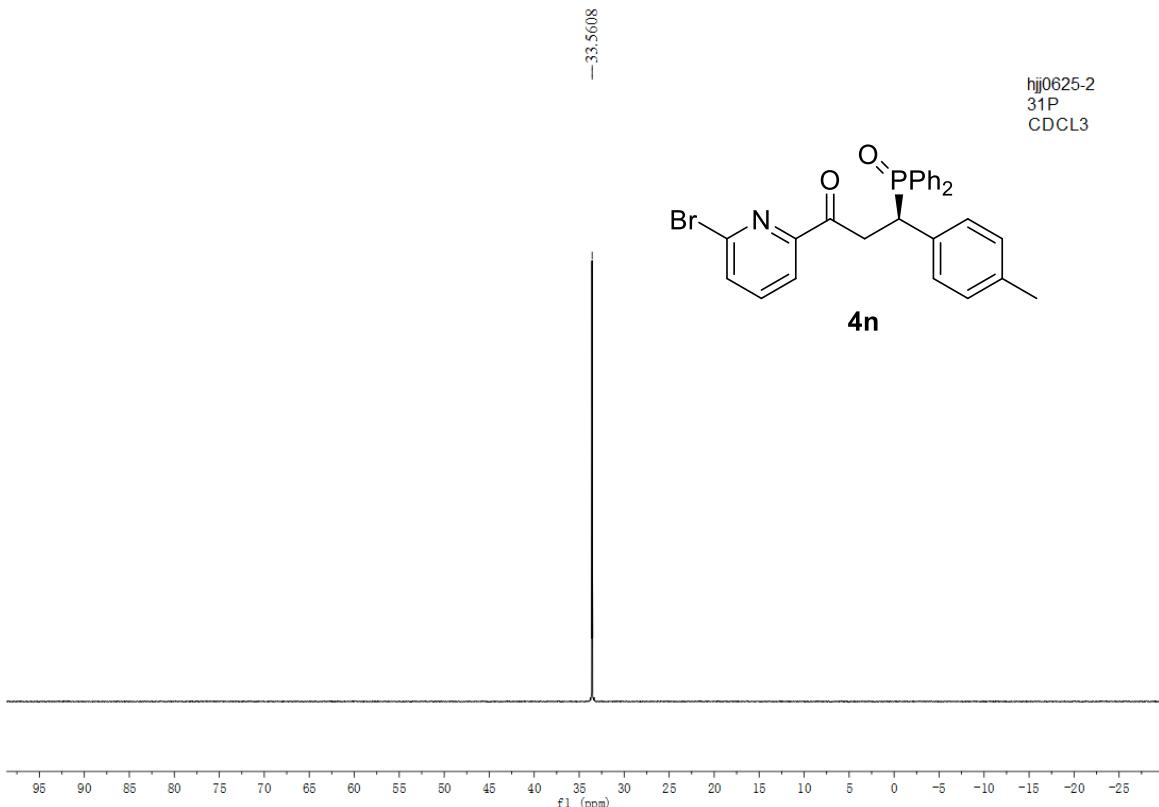
hjj0625-2
1H
CDCl₃



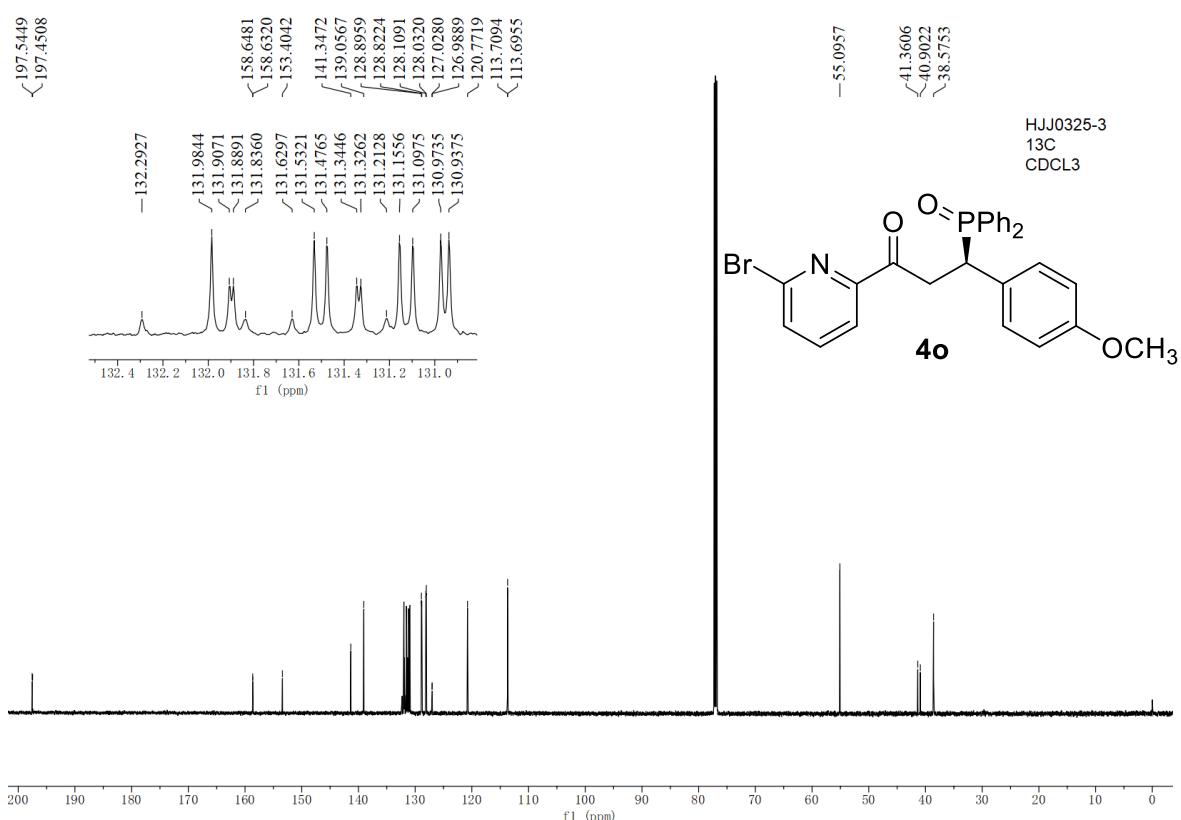
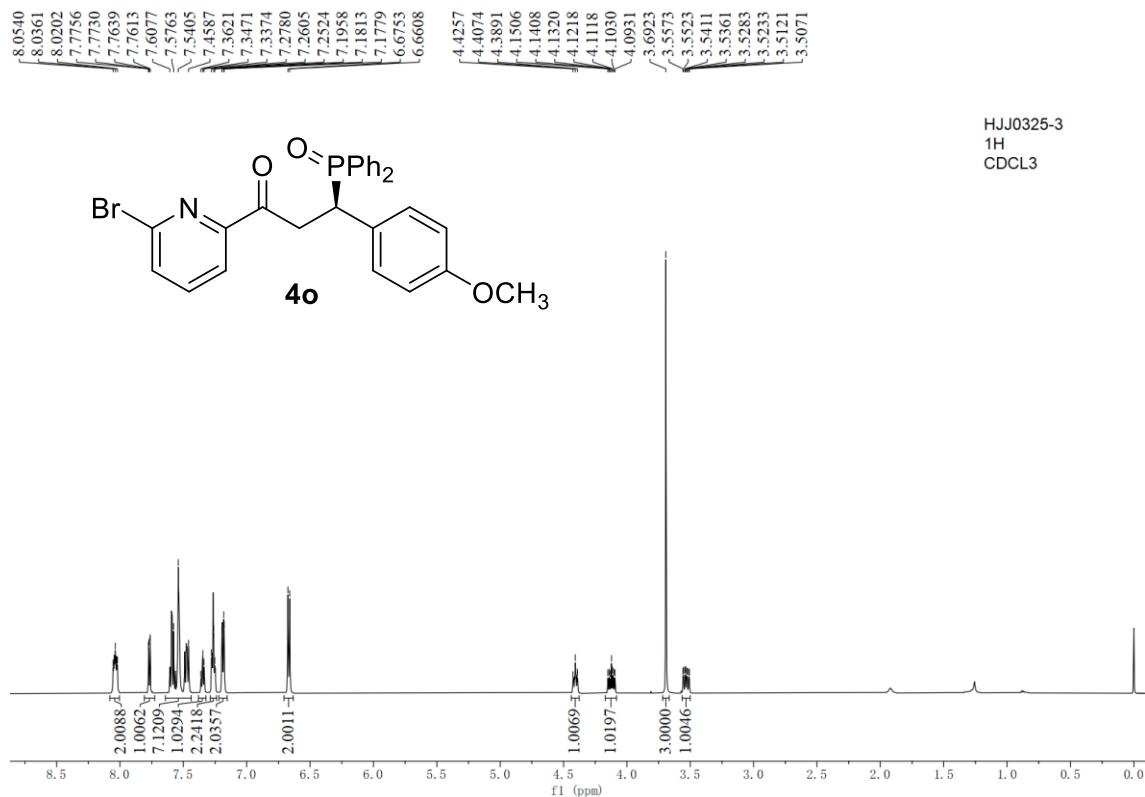
¹H NMR spectrum of **4n** (600 MHz, CDCl₃)



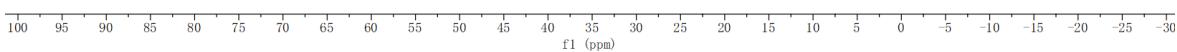
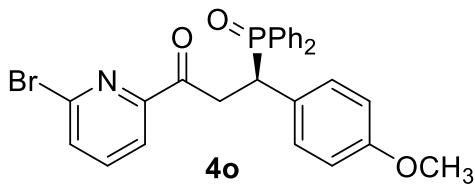
¹³C{¹H} NMR spectrum of **4n** (150 MHz, CDCl₃)



³¹P{¹H} NMR spectrum of **4n** (243 MHz, CDCl₃)

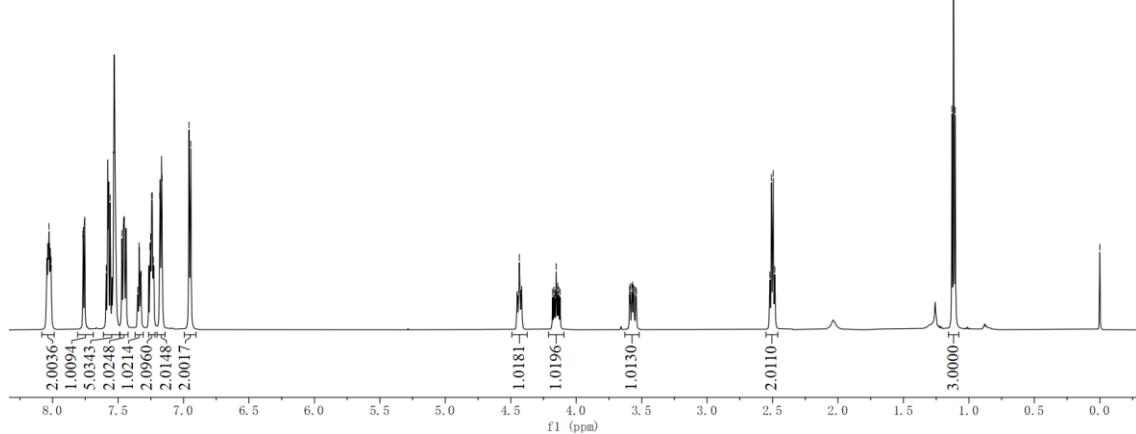
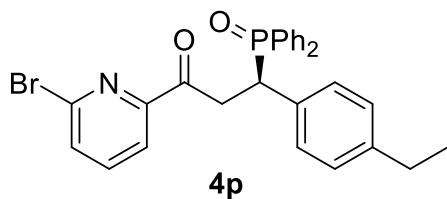


HJJ0325-3
31P
CDCl₃

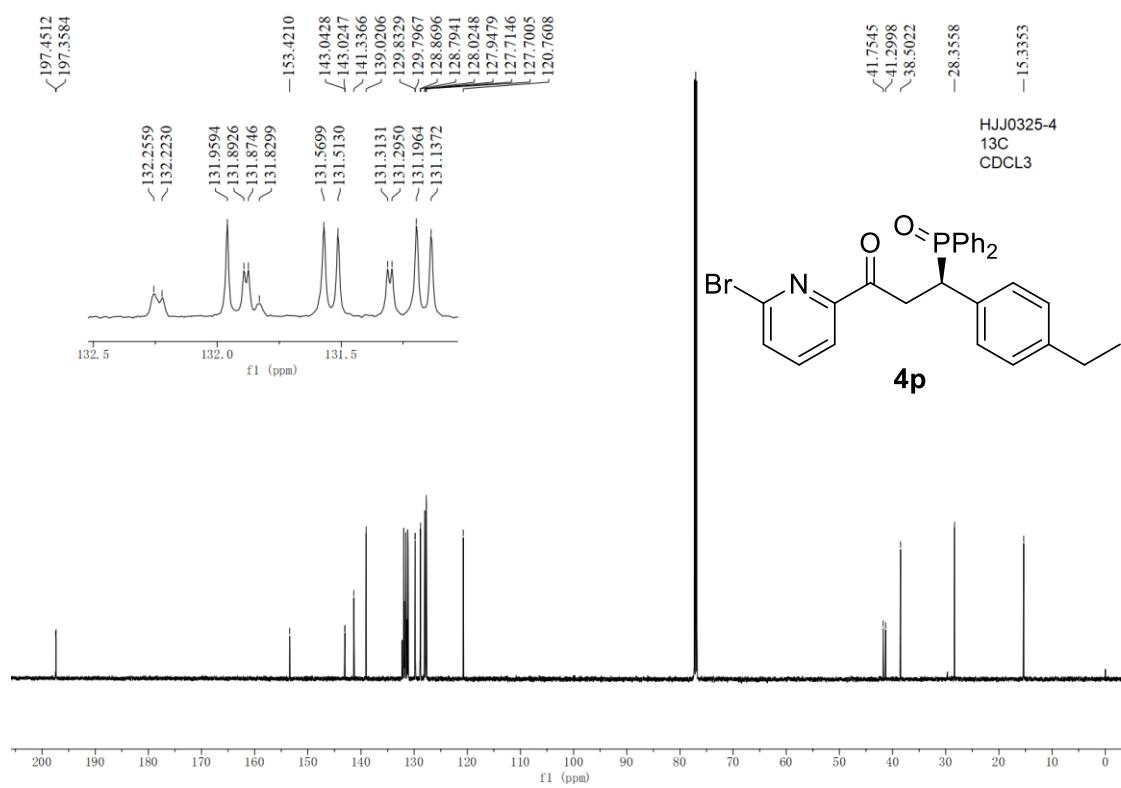


³¹P{¹H} NMR spectrum of **4o** (243 MHz, CDCl₃)

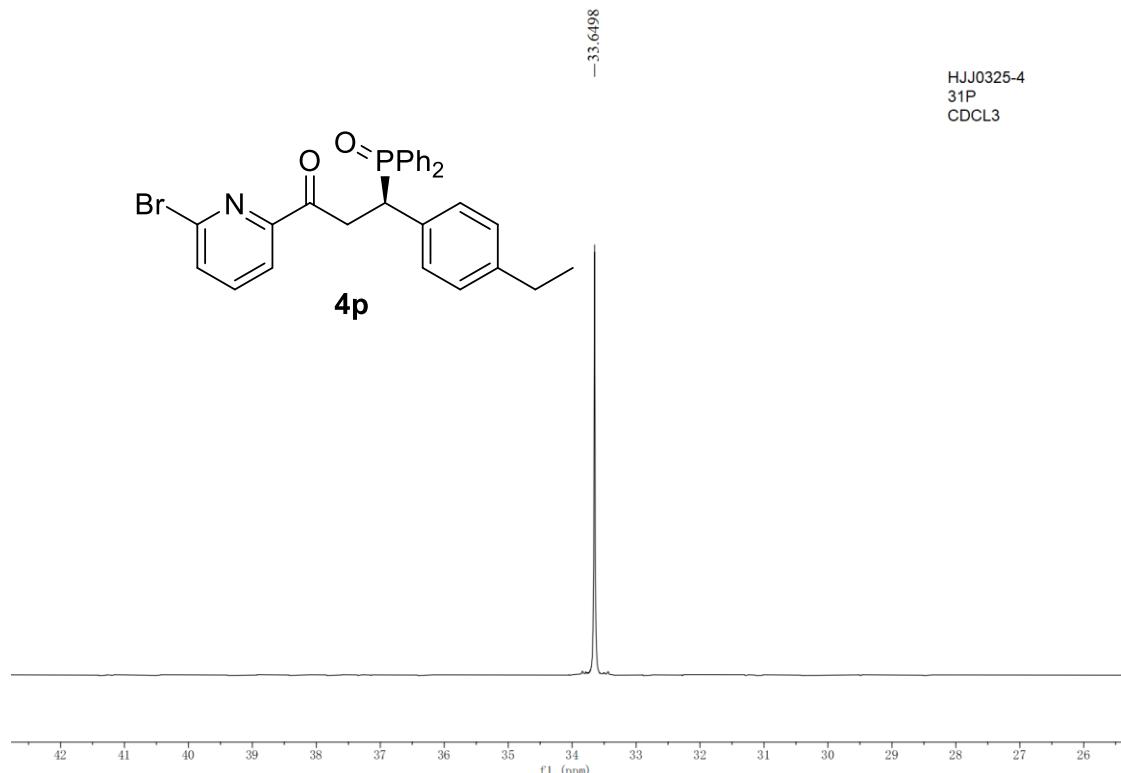
HJJ0325-4
1H
CDCl₃



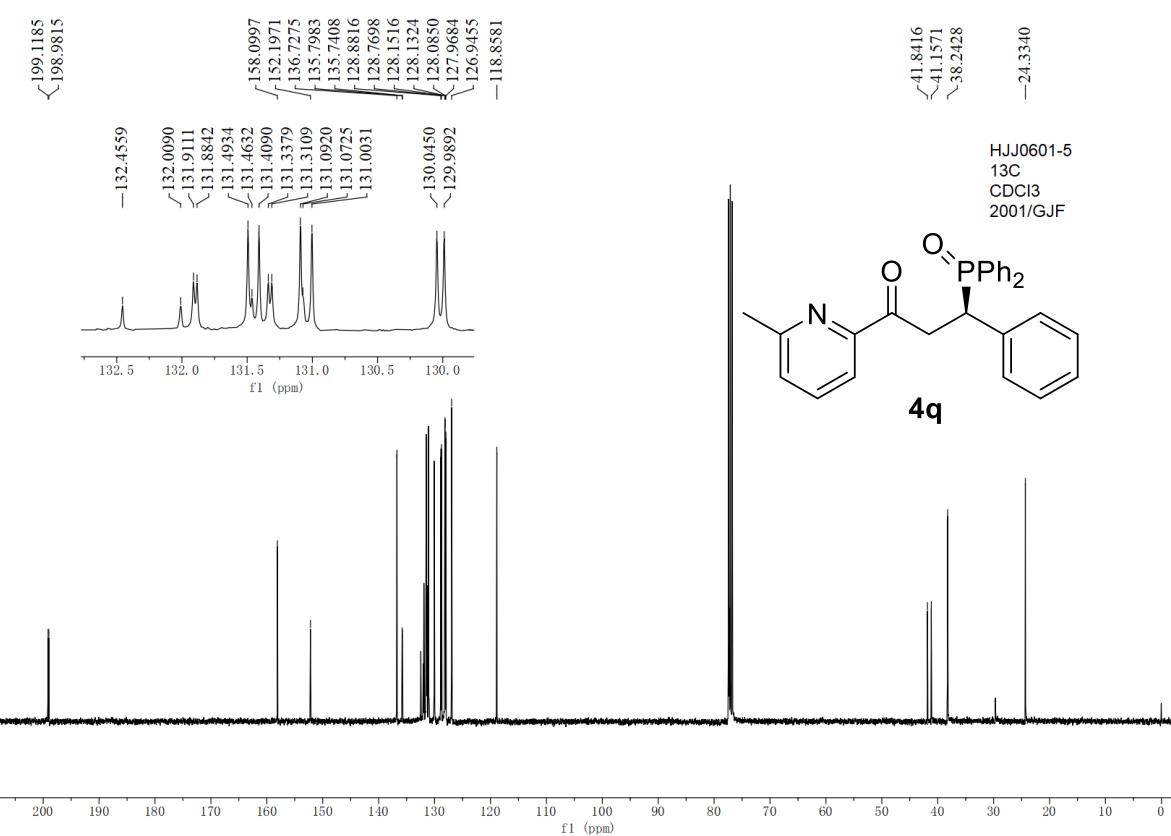
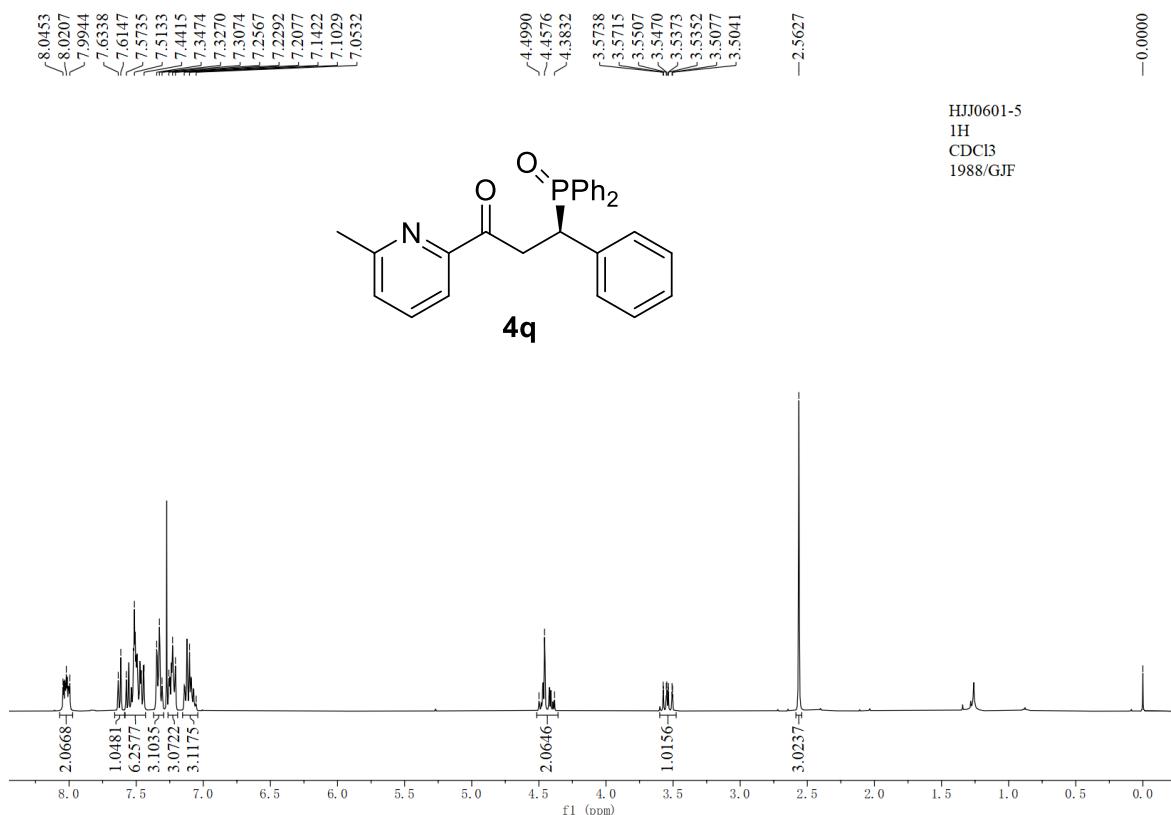
¹H NMR spectrum of **4p** (600 MHz, CDCl₃)

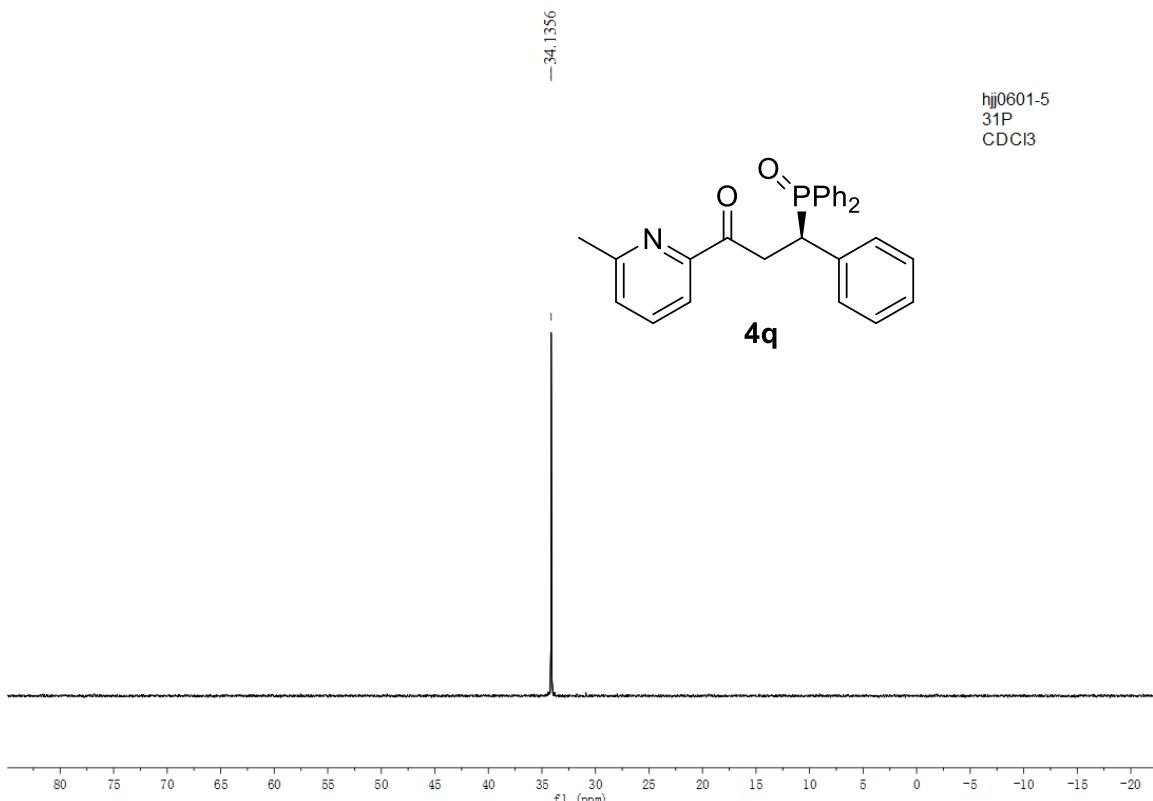


¹³C{¹H} NMR spectrum of **4p** (150 MHz, CDCl₃)

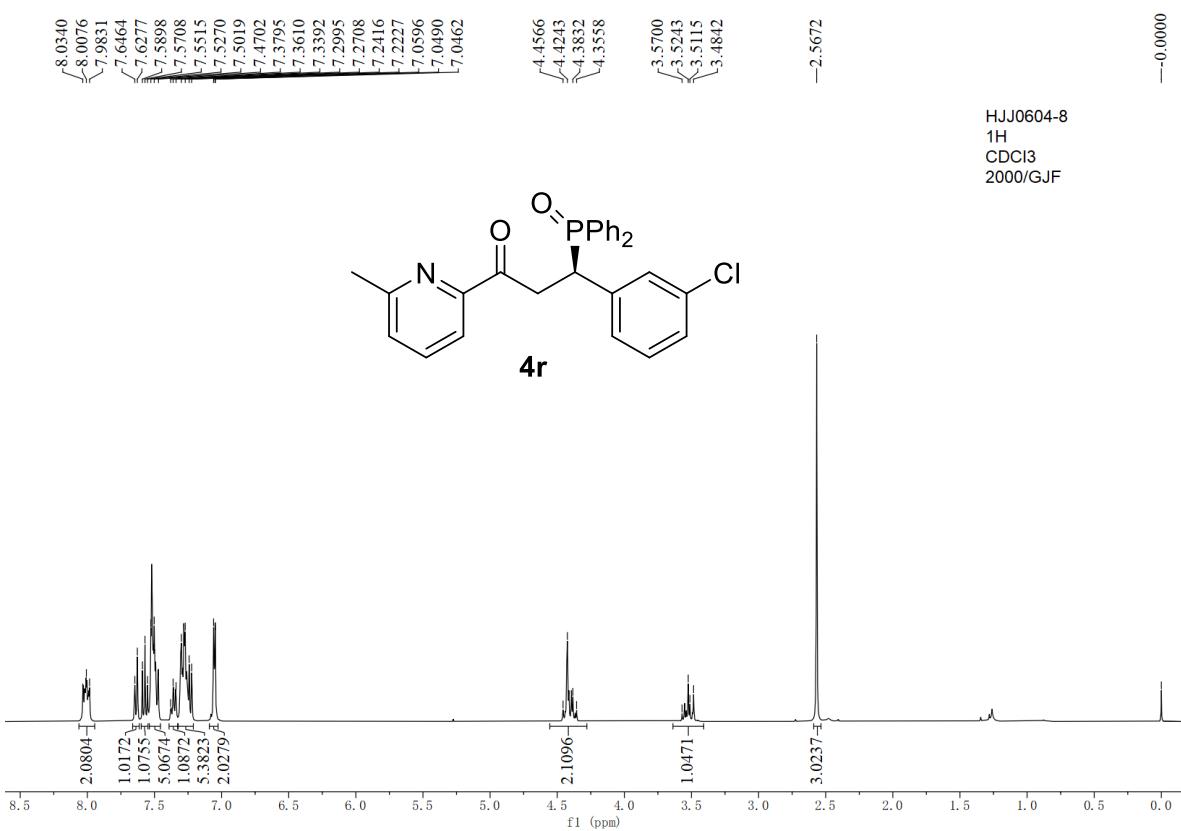


³¹P{¹H} NMR spectrum of **4p** (243 MHz, CDCl₃)

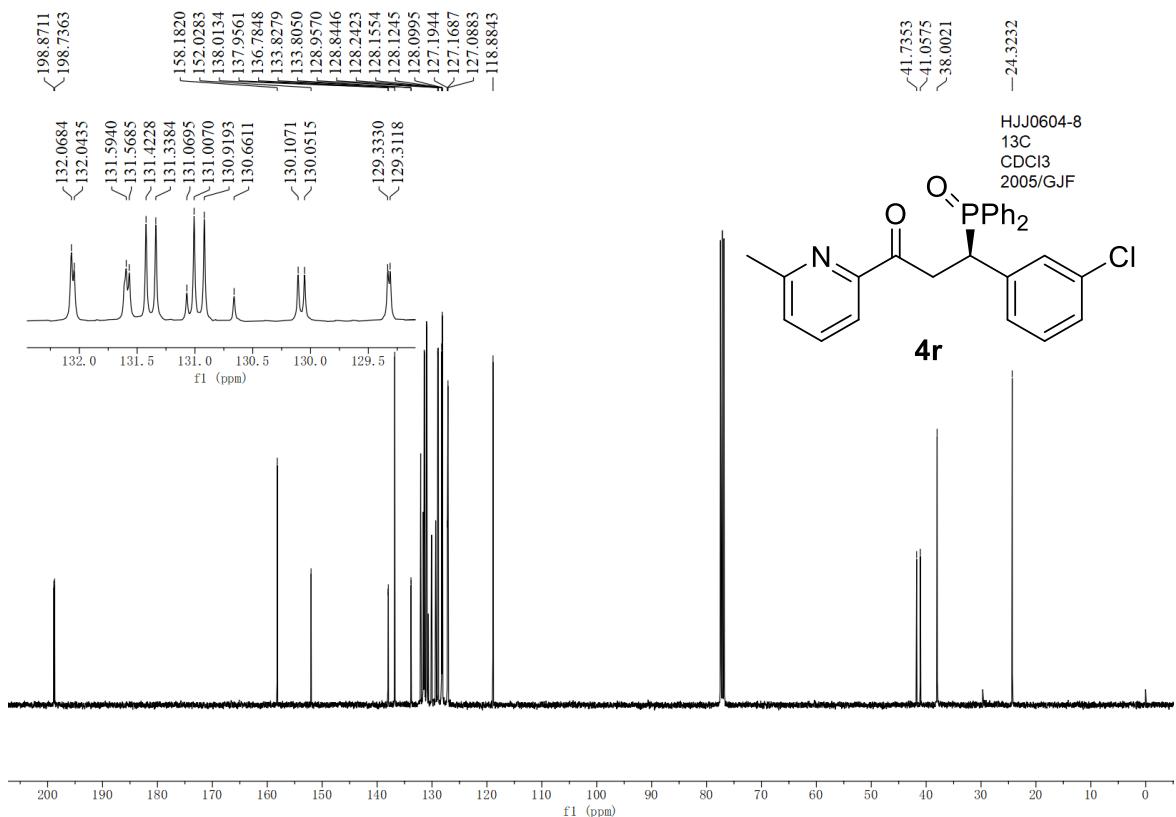


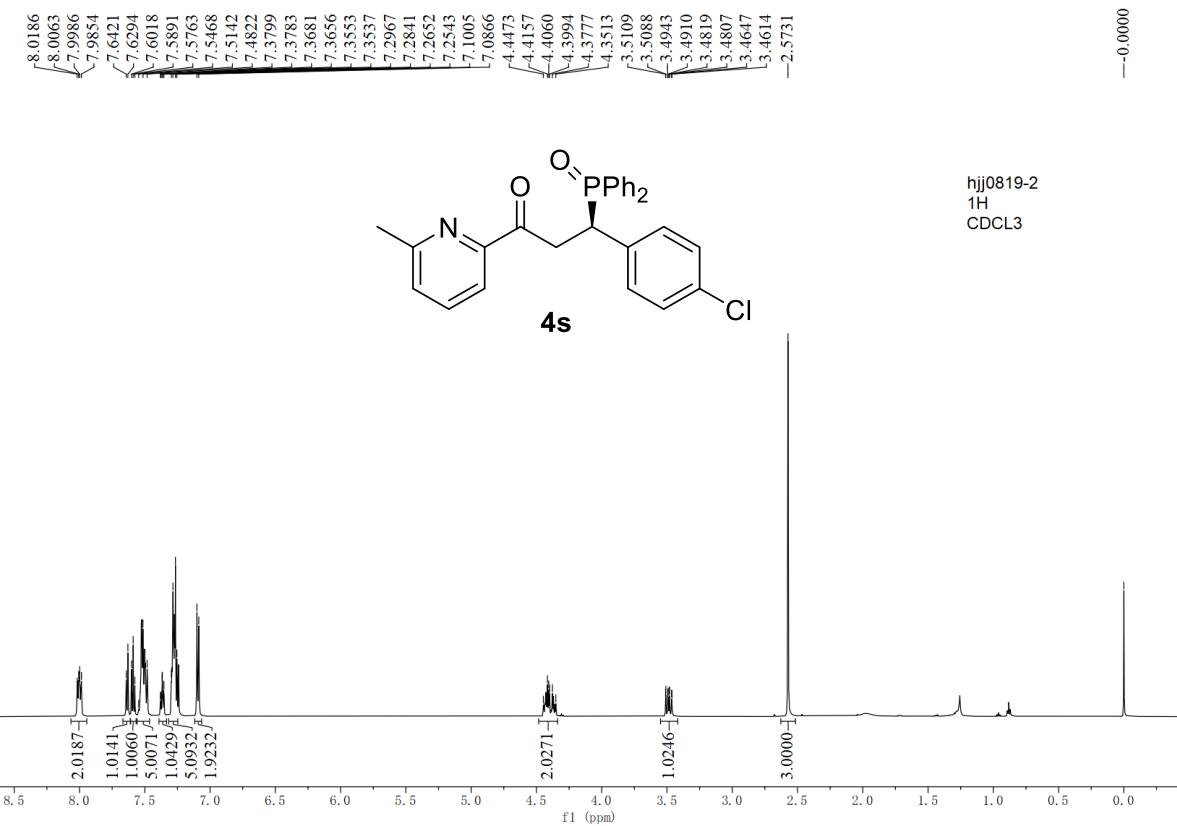


³¹P{¹H} NMR spectrum of **4q** (163 MHz, CDCl₃)

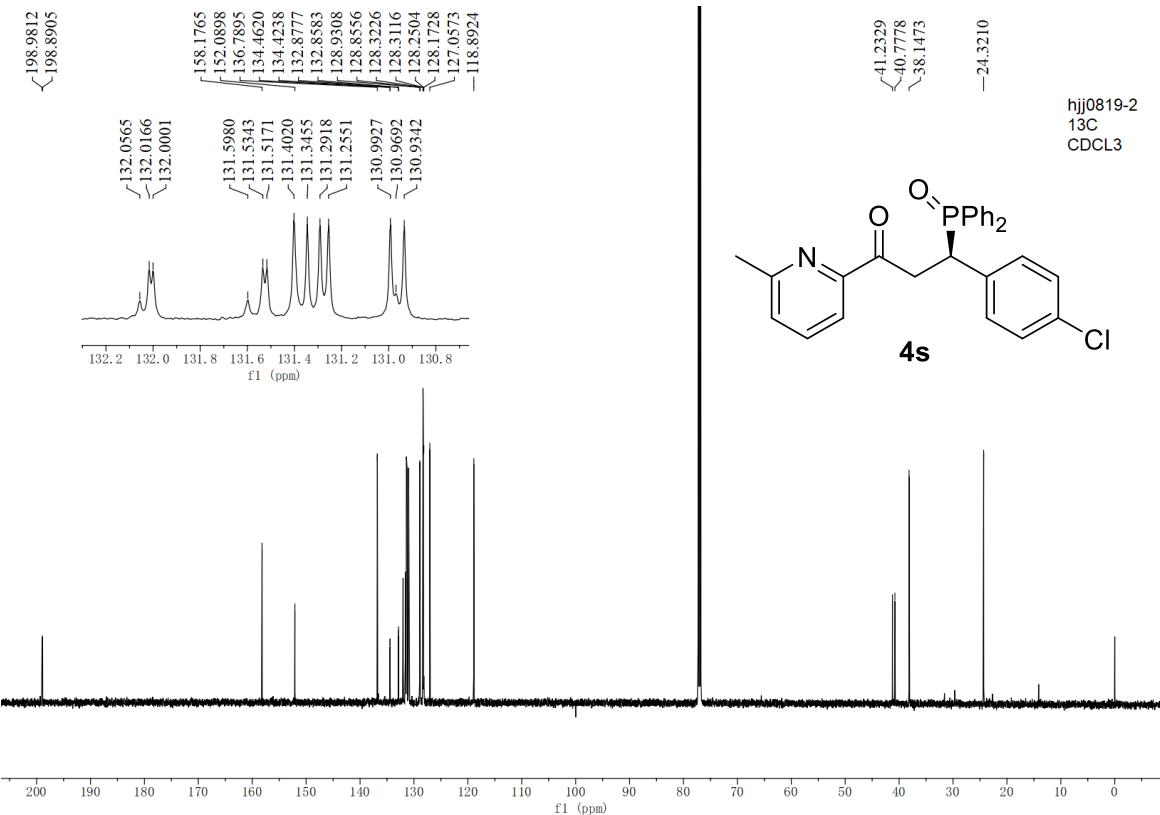


¹H NMR spectrum of **4r** (400 MHz, CDCl₃)

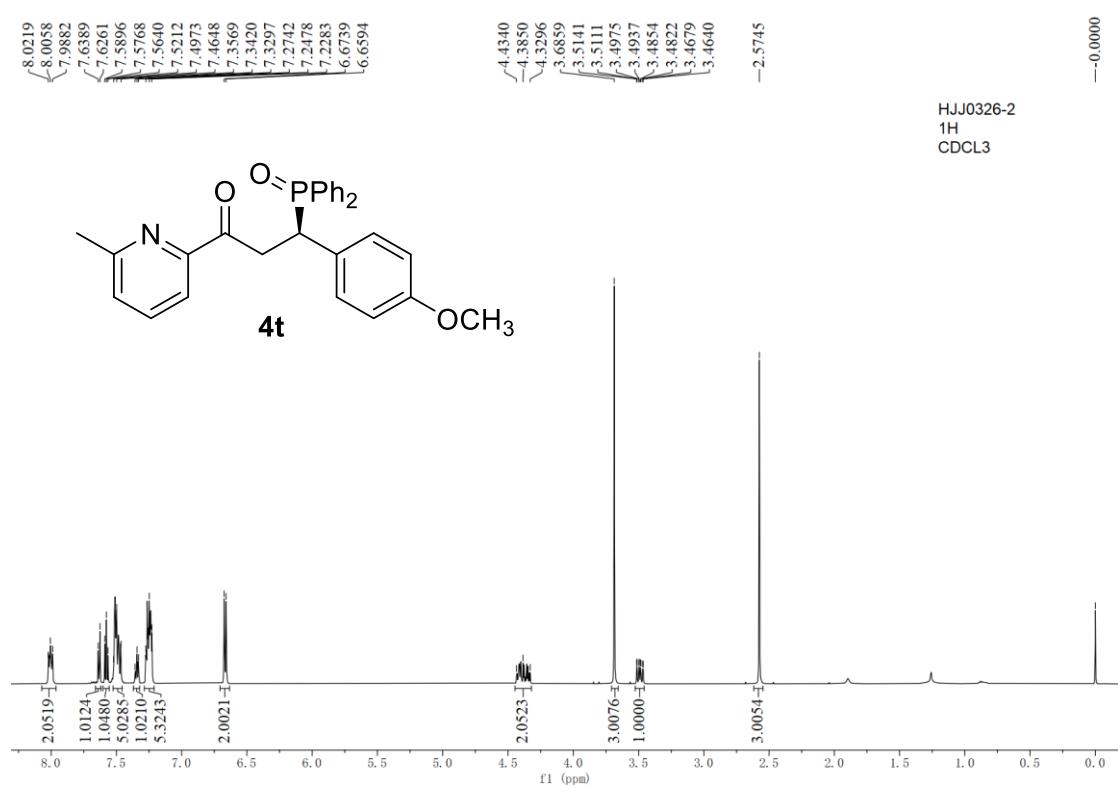
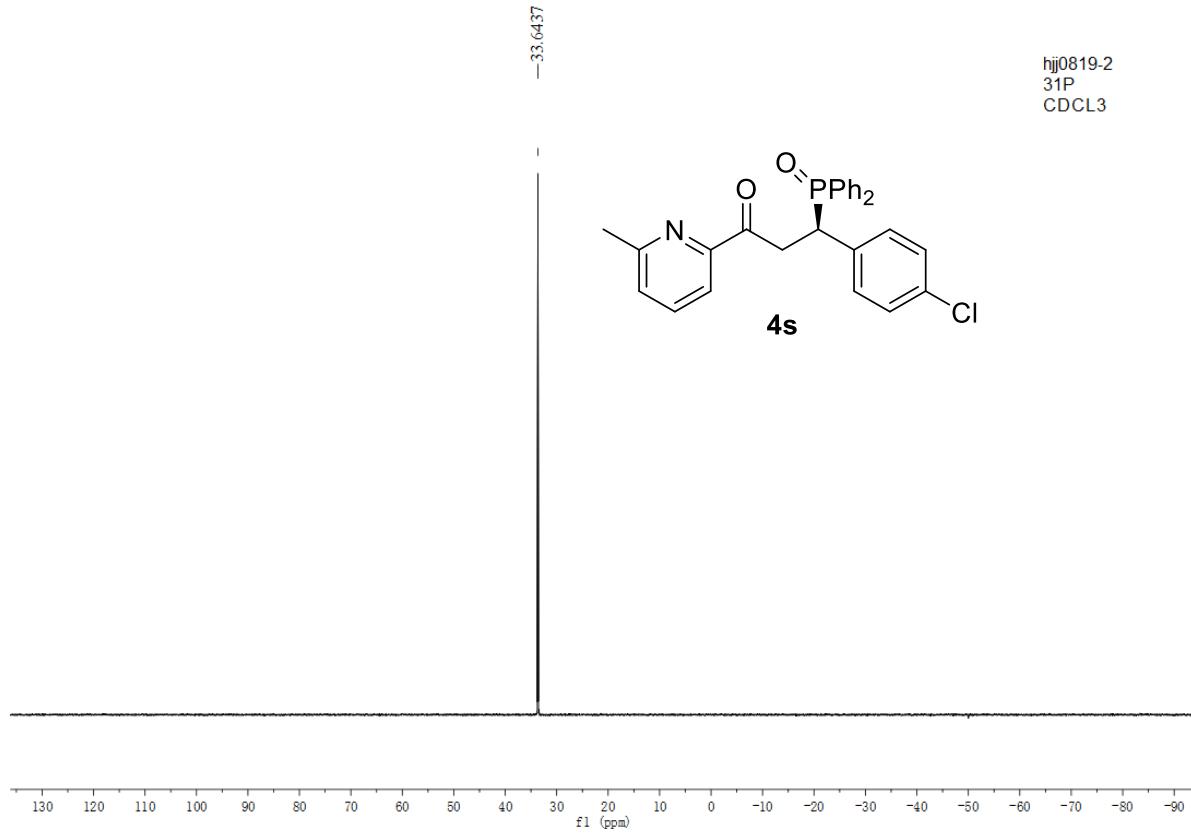


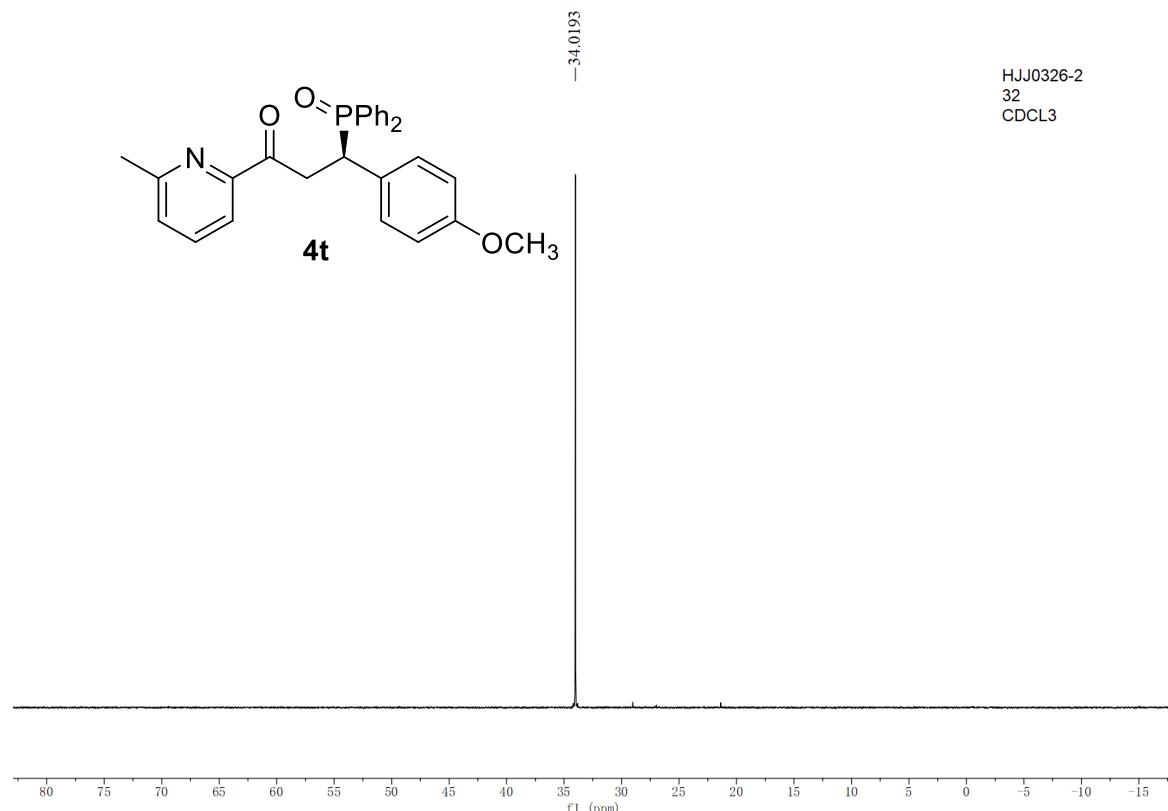
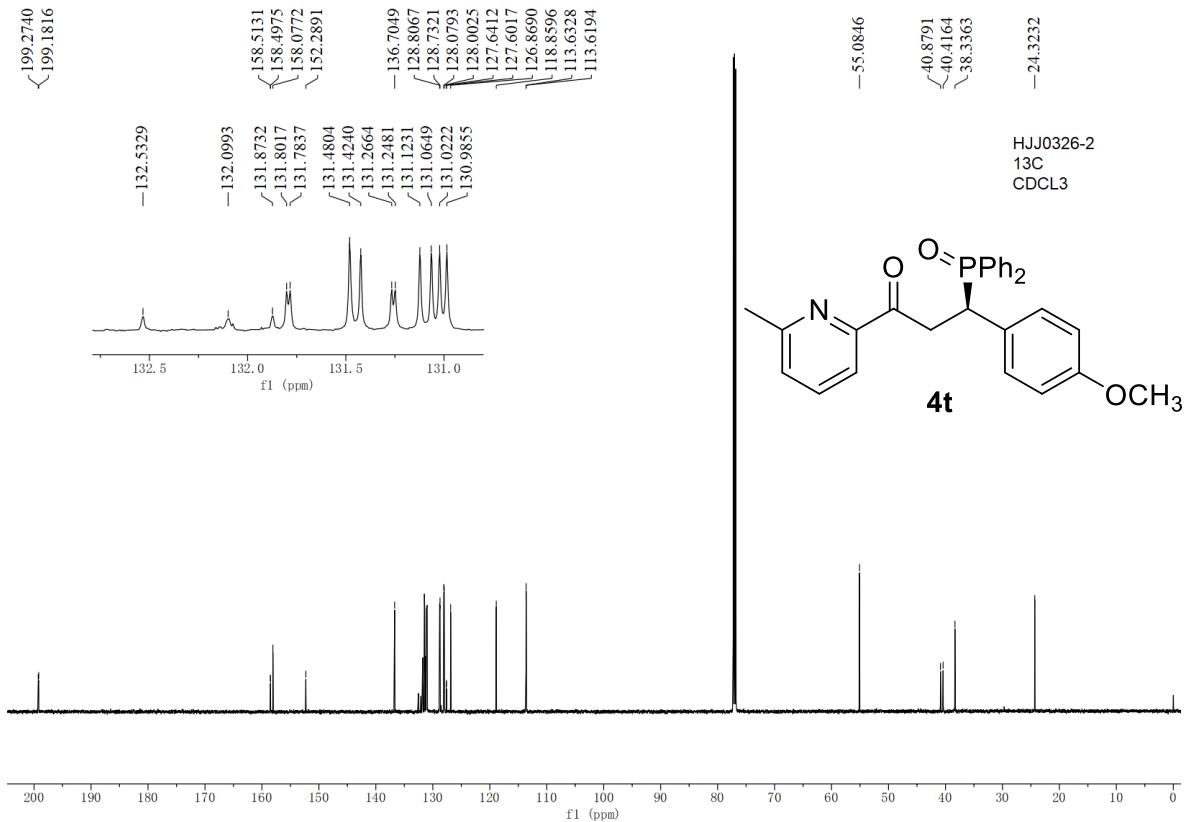


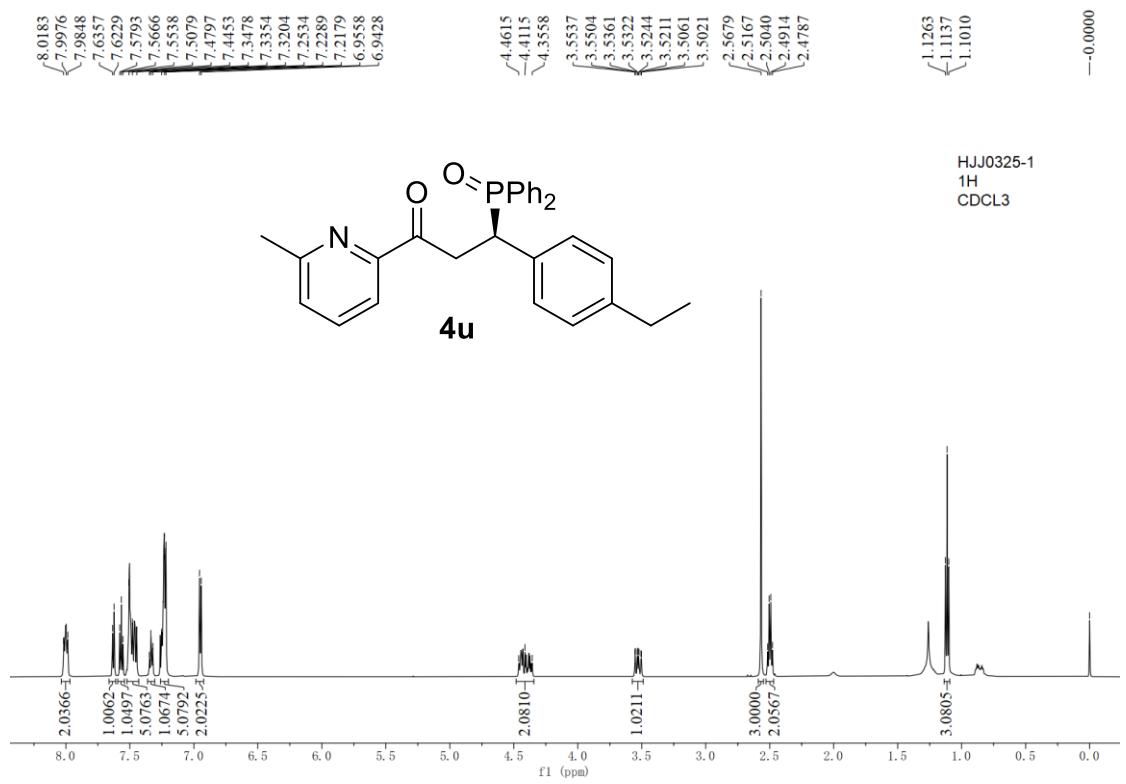
¹H NMR spectrum of **4s** (600 MHz, CDCl₃)



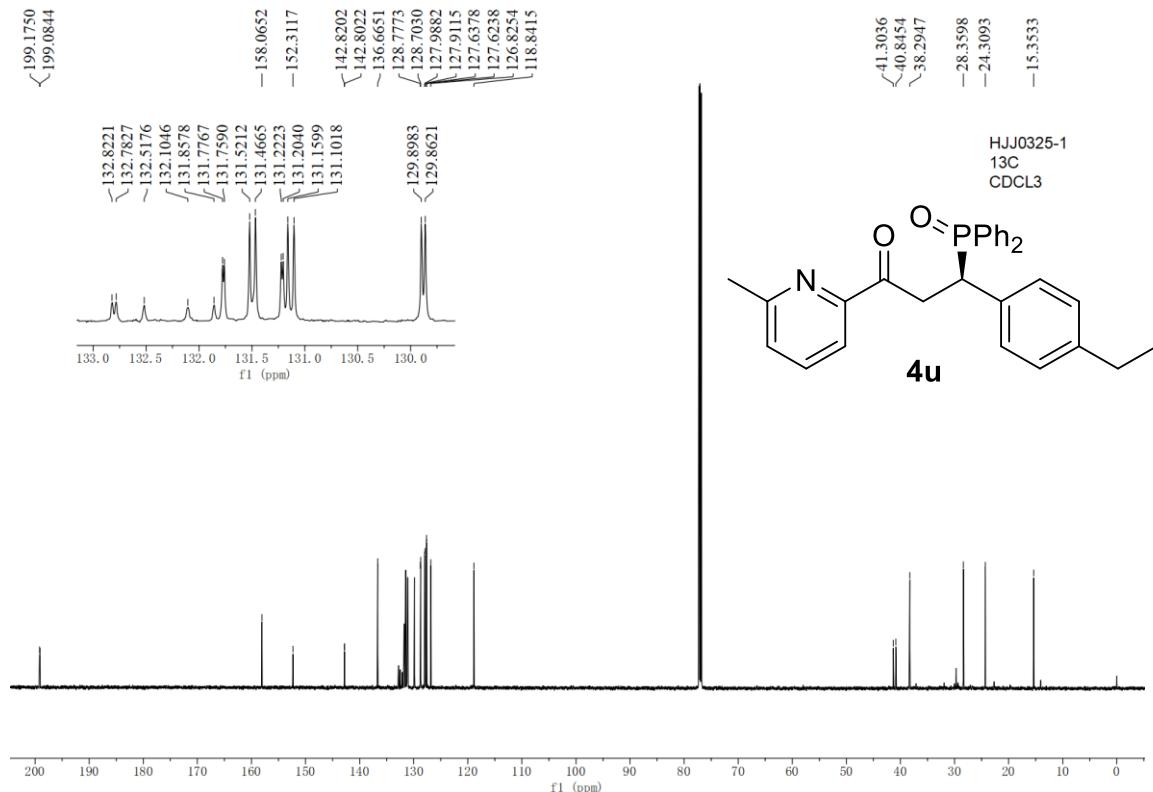
¹³C{¹H} NMR spectrum of **4s** (150 MHz, CDCl₃)



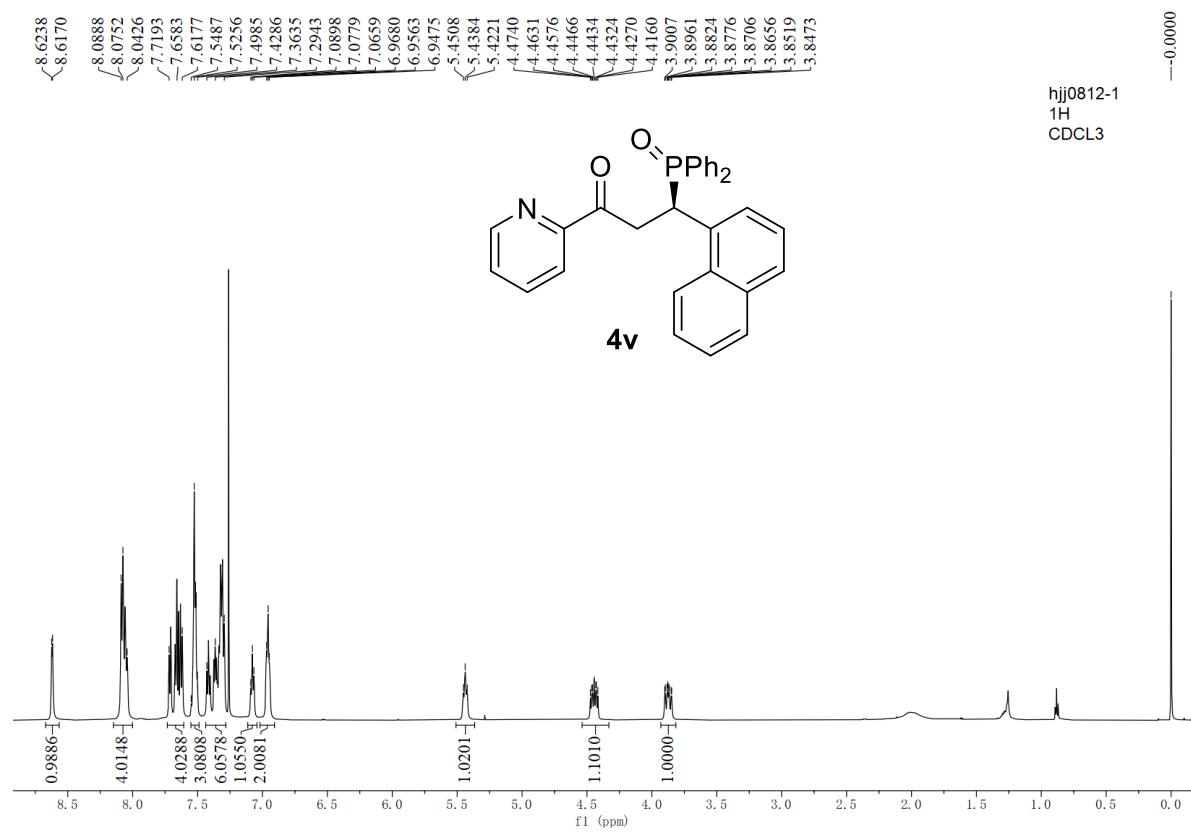
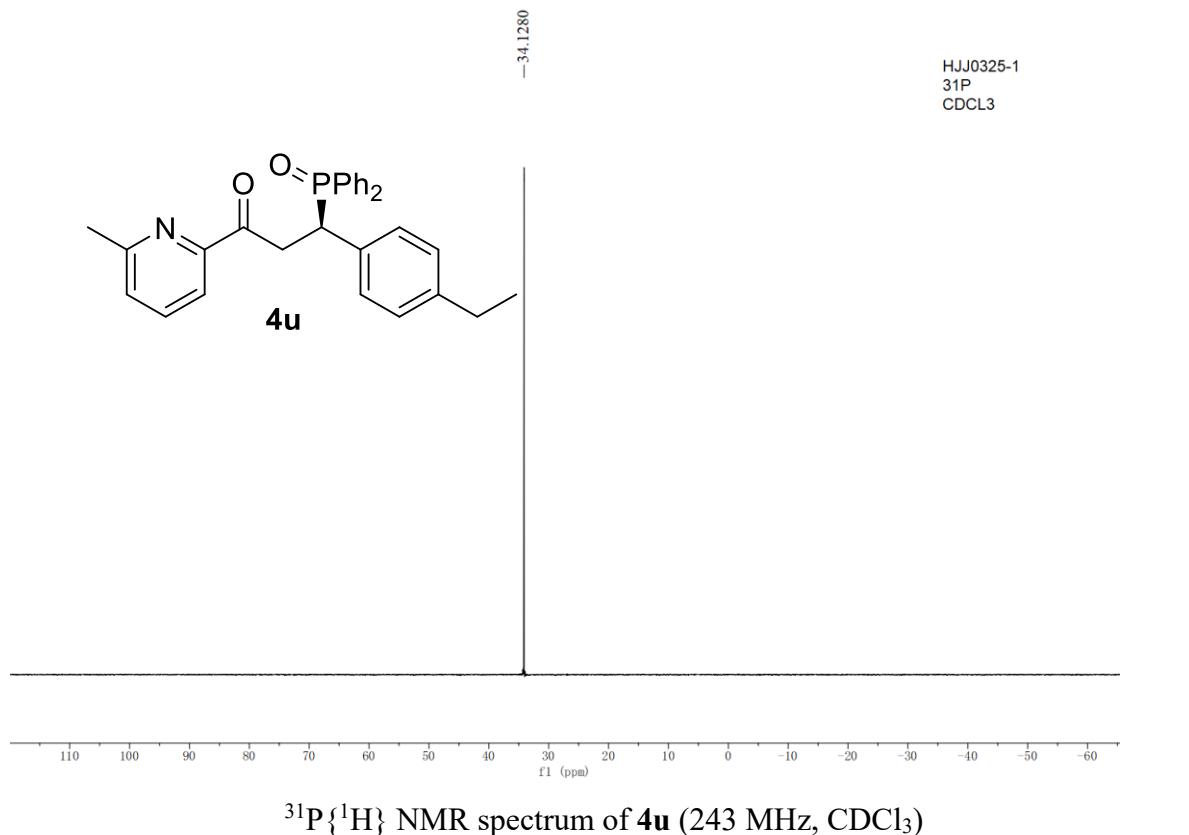


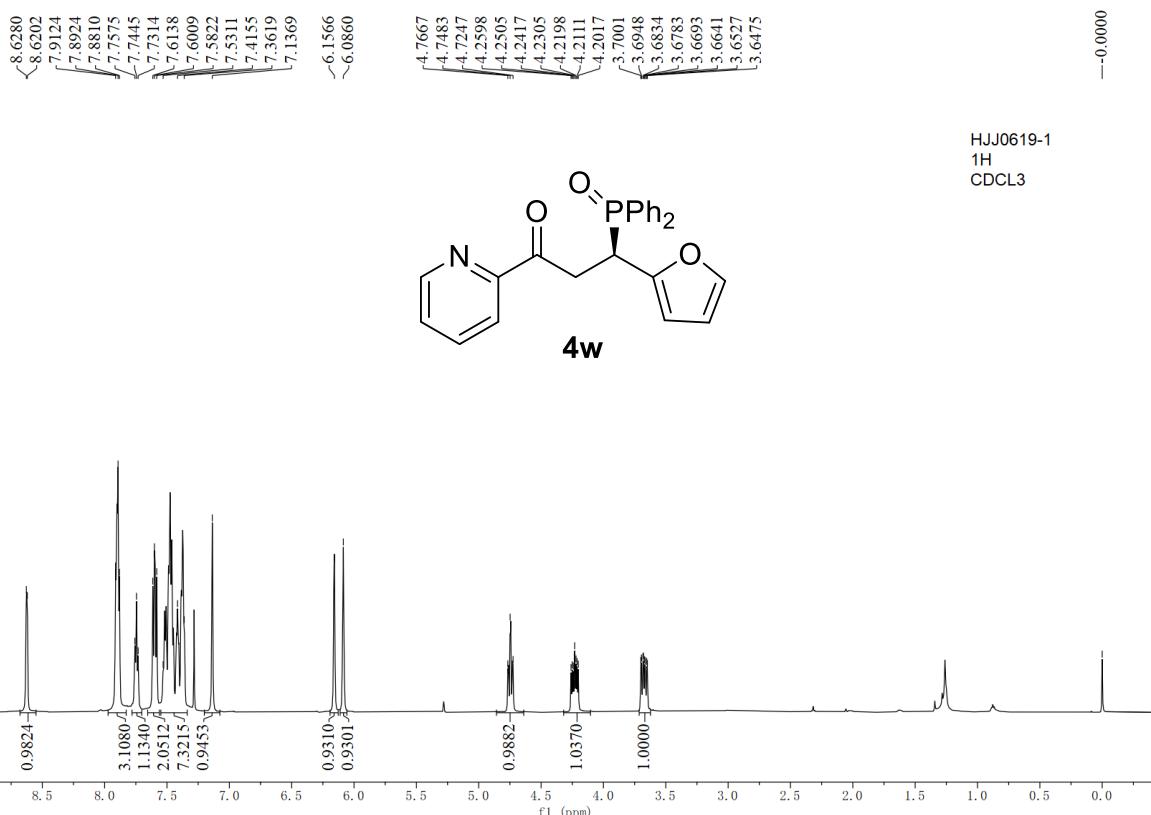


¹H NMR spectrum of **4u** (600 MHz, CDCl₃)

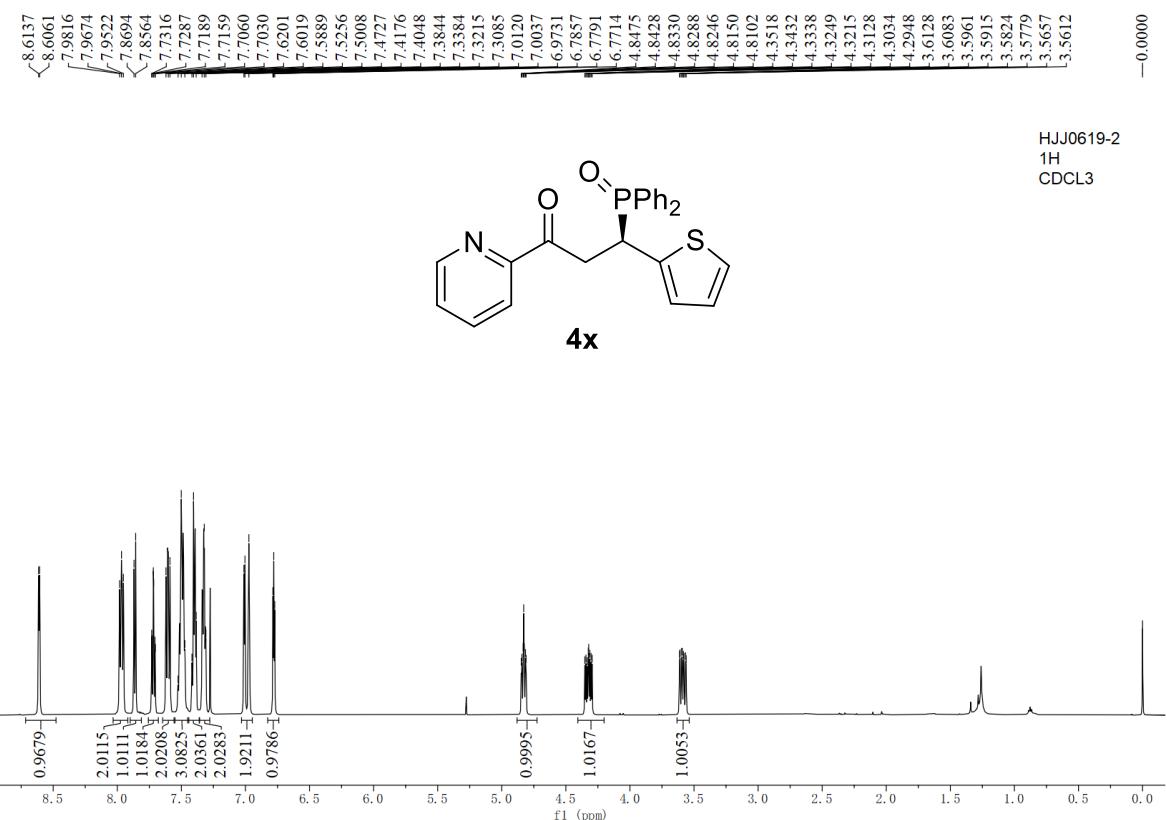


¹³C{¹H} NMR spectrum of **4u** (150 MHz, CDCl₃)

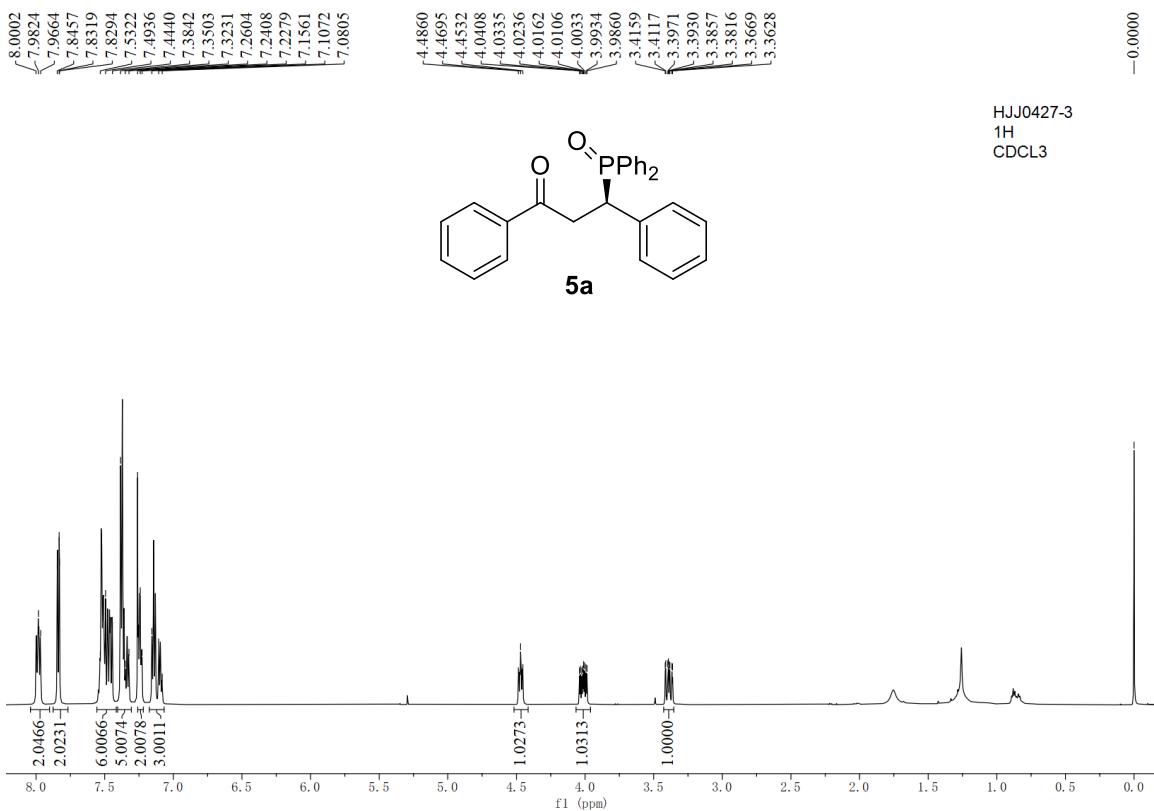




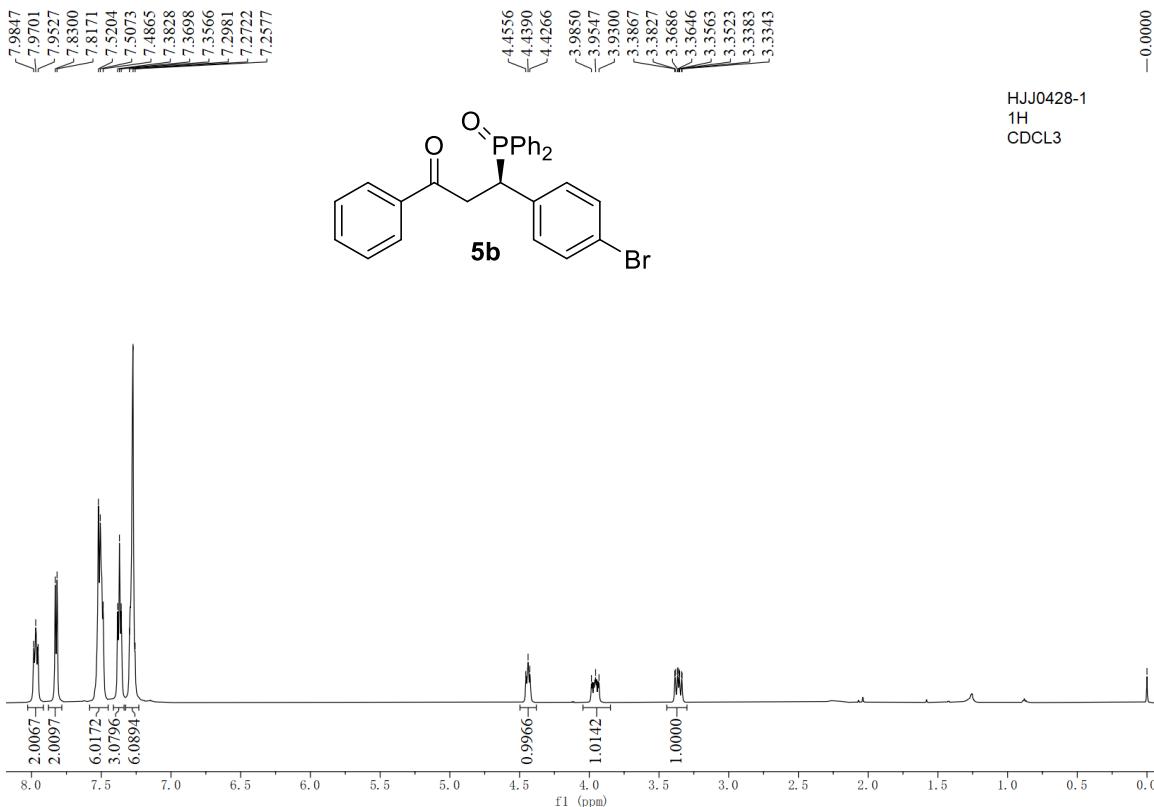
¹H NMR spectrum of **4w** (600 MHz, CDCl₃)



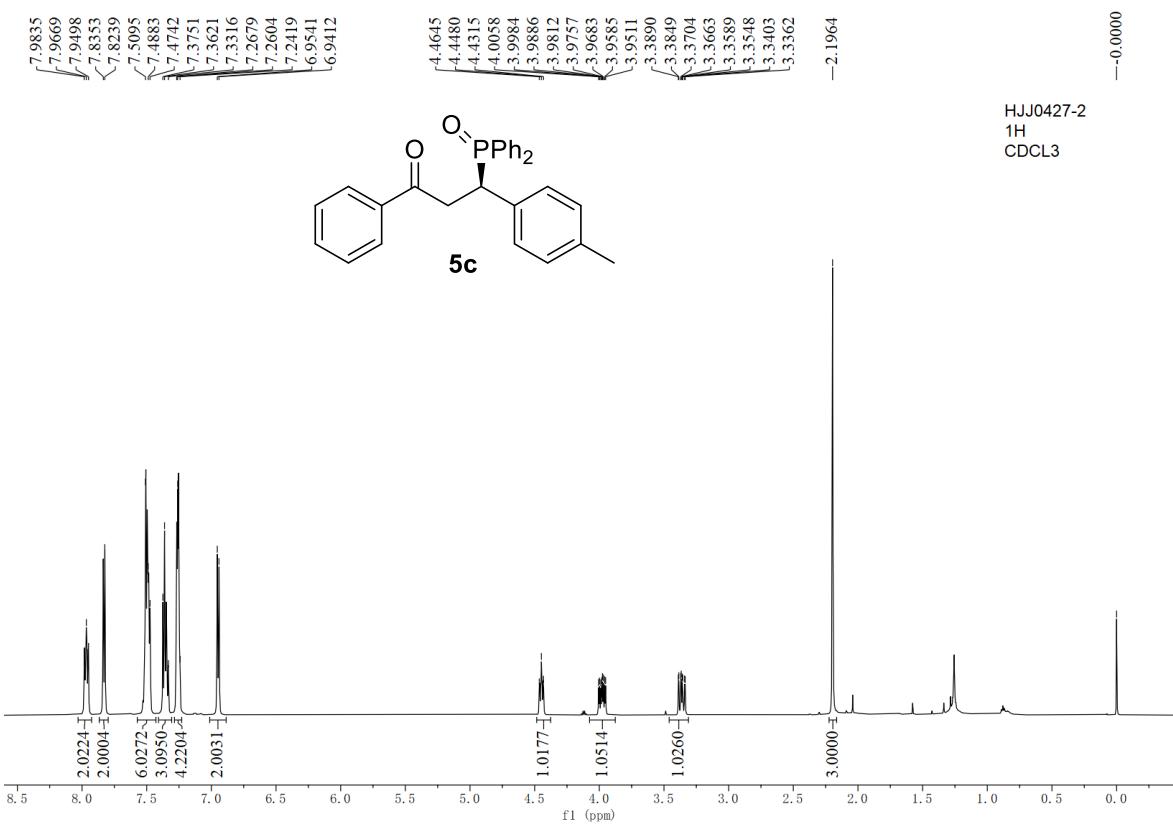
¹H NMR spectrum of **4x** (600 MHz, CDCl₃)



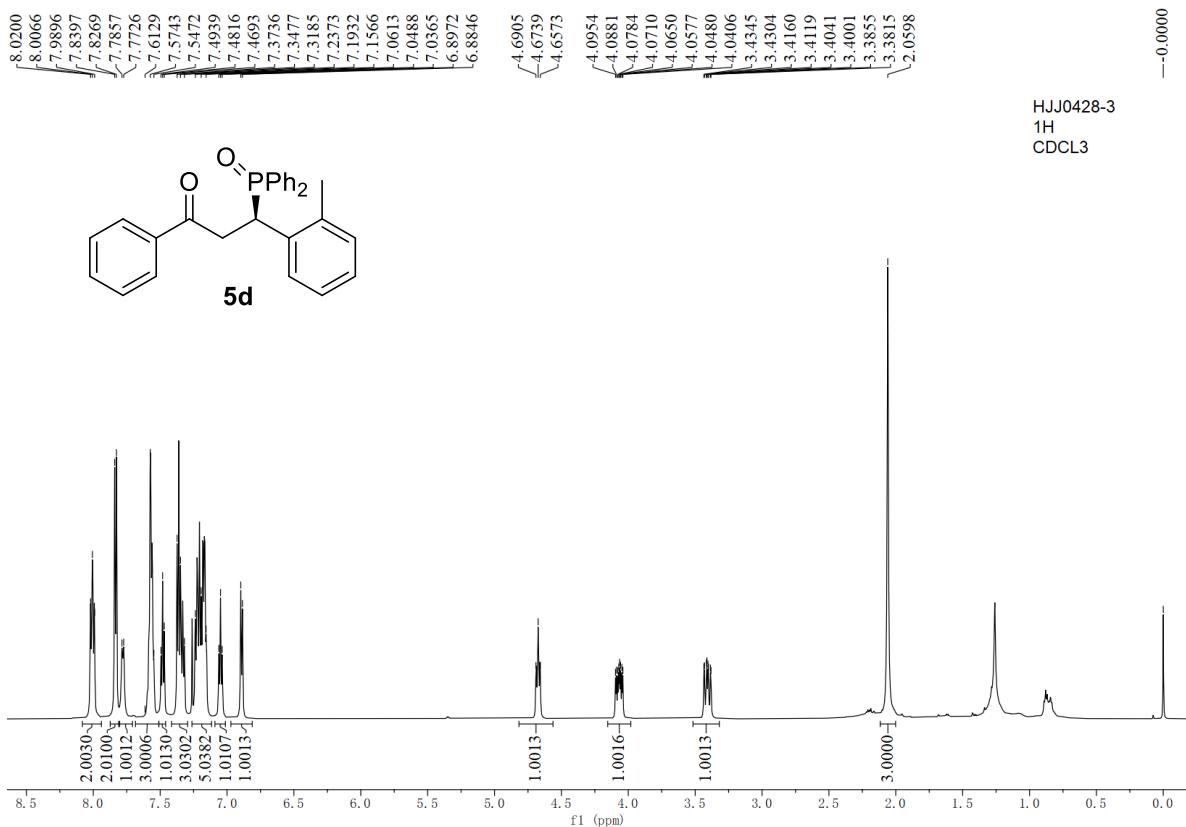
¹H NMR spectrum of **5a** (600 MHz, CDCl₃)



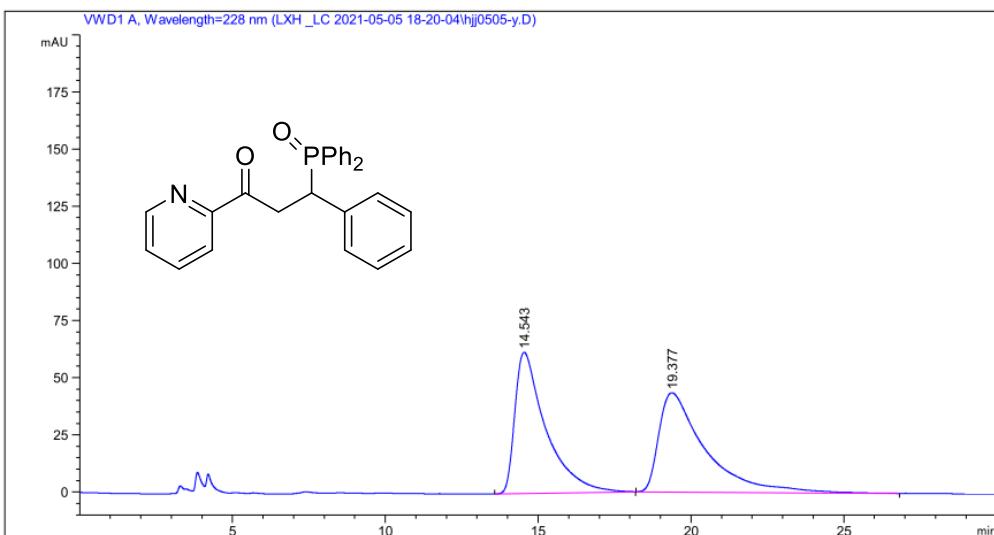
¹H NMR spectrum of **5b** (600 MHz, CDCl₃)



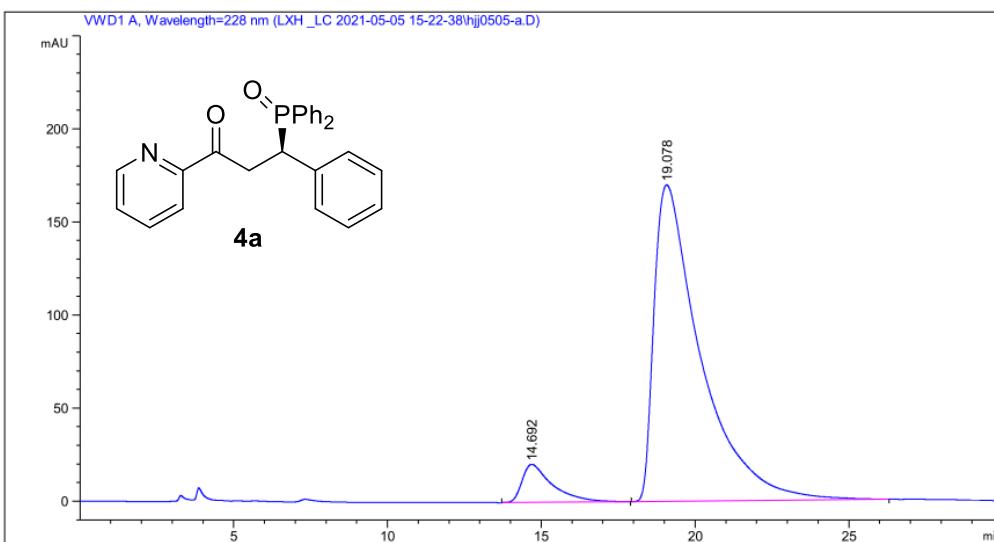
¹H NMR spectrum of **5c** (600 MHz, CDCl₃)



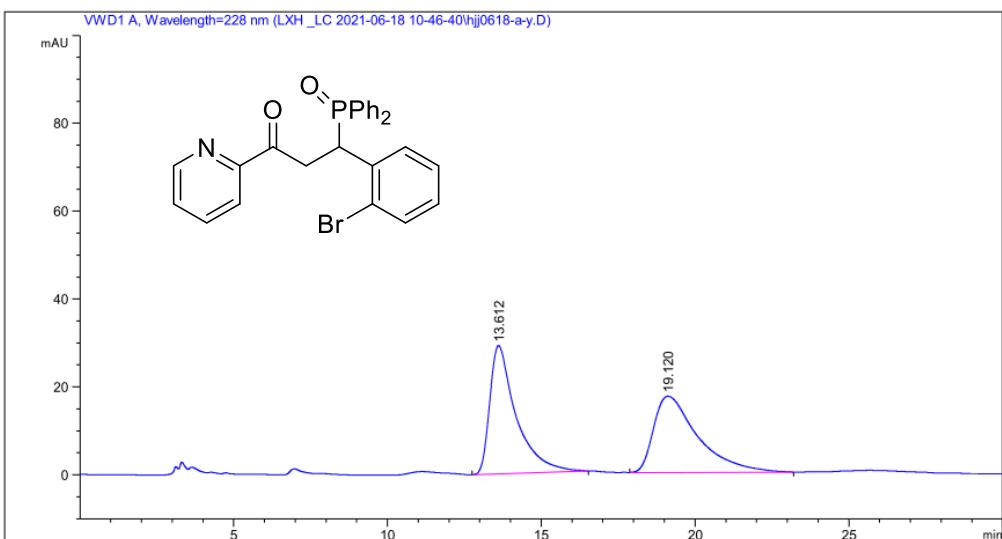
¹H NMR spectrum of **5d** (600 MHz, CDCl₃)



Chiral HPLC chromatogram for racemic **4a**

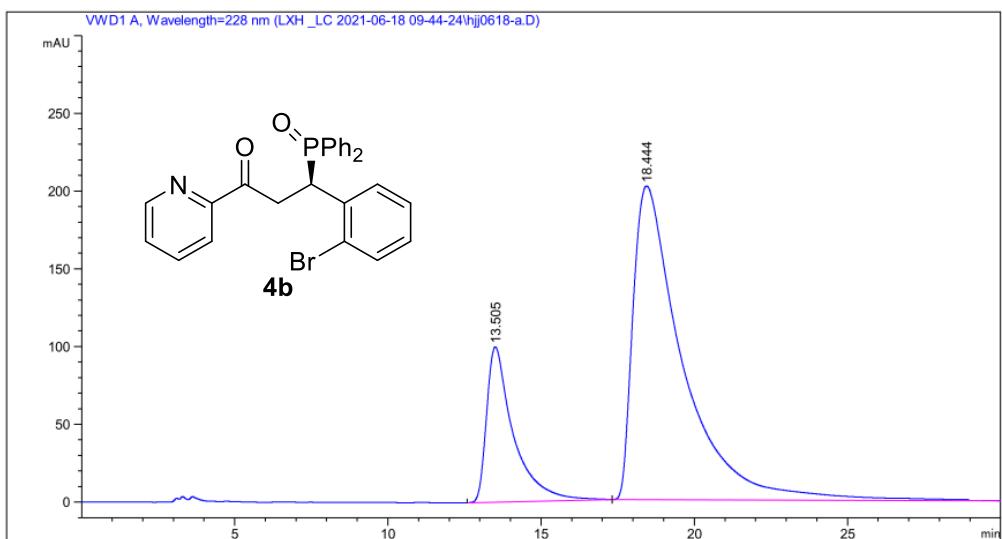


Chiral HPLC chromatogram for enantioenriched **4a**



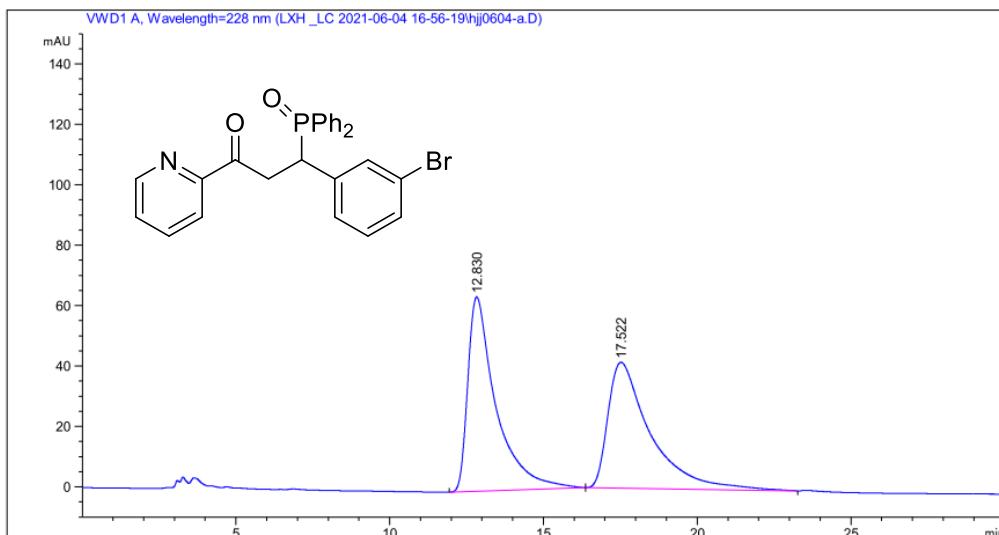
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	13.612	1737.38477	29.20243	50.2192
2	19.120	1722.21887	17.33938	49.7808

Chiral HPLC chromatogram for racemic **4b**

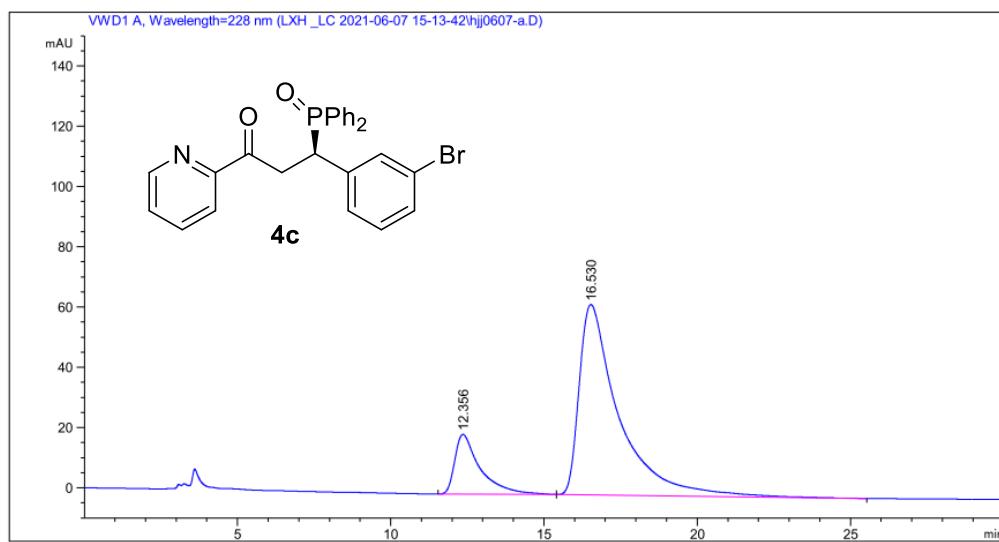


PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	13.505	6059.80762	99.86134	21.0260
2	18.444	2.27607e4	201.68936	78.9740

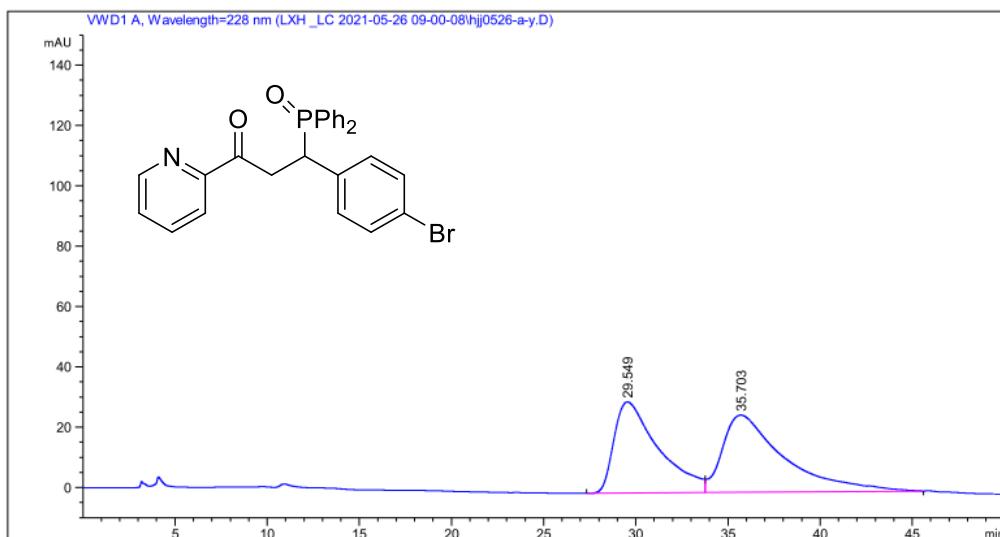
Chiral HPLC chromatogram for enantioenriched **4b**



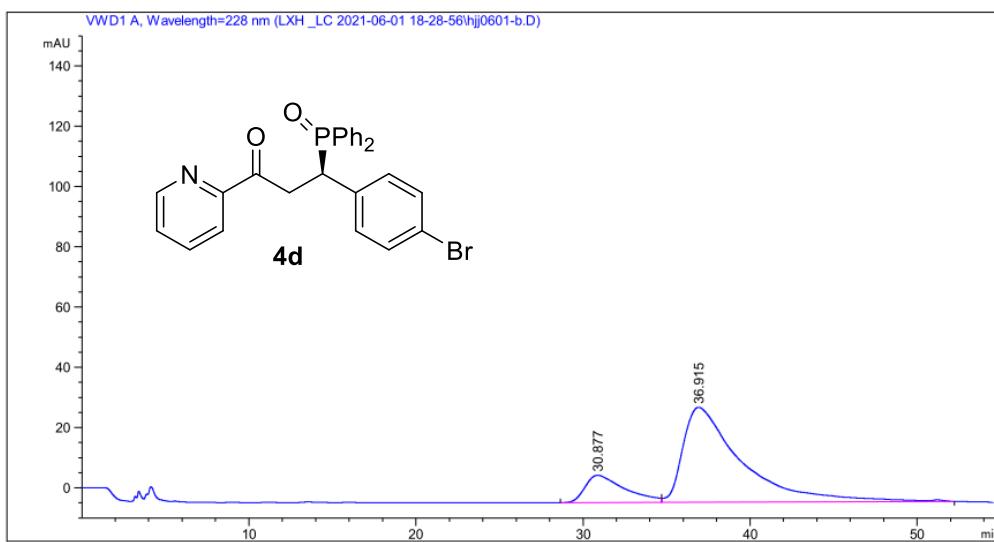
Chiral HPLC chromatogram for racemic **4c**



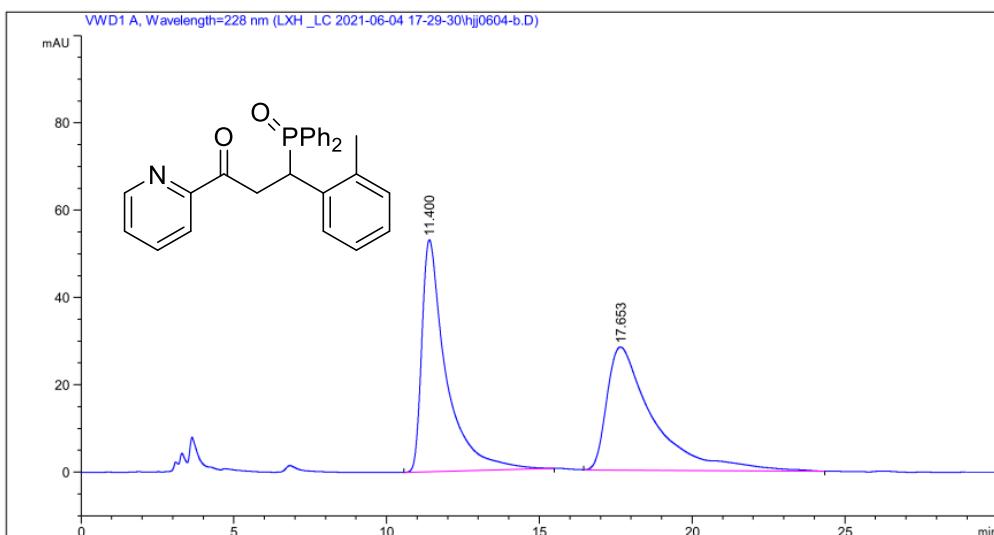
Chiral HPLC chromatogram for enantioenriched **4c**



Chiral HPLC chromatogram for racemic **4d**

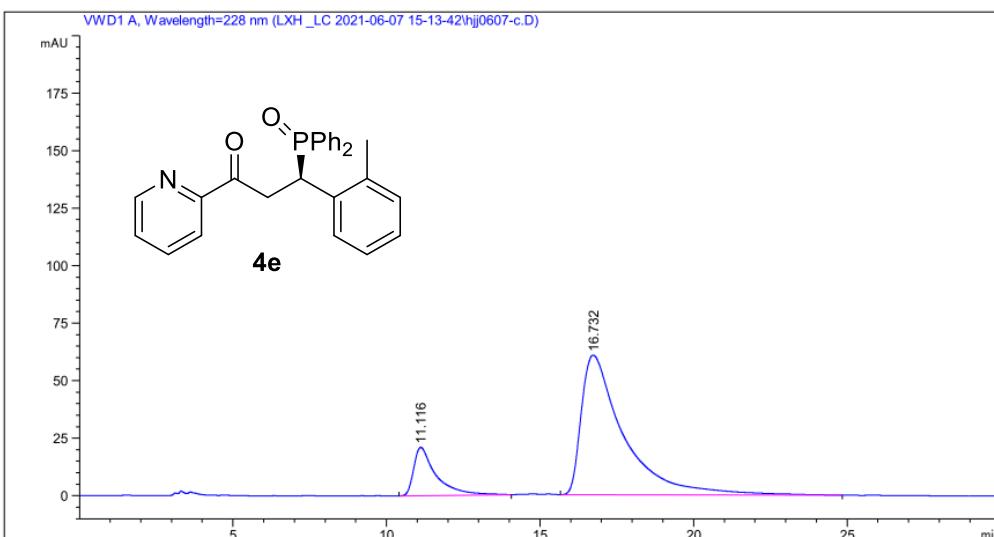


Chiral HPLC chromatogram for enantioenriched **4d**



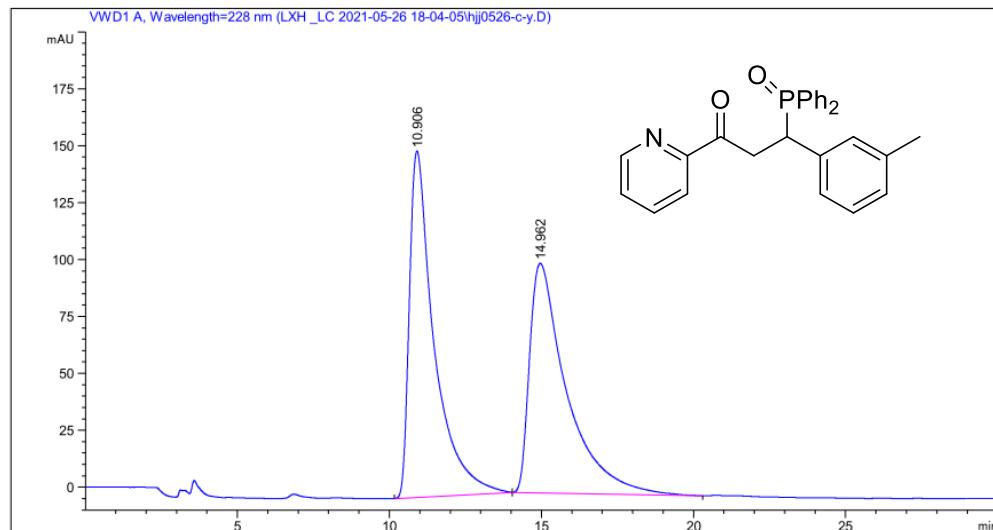
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	11.400	2936.97095	53.12720	49.7166
2	17.653	2970.45483	28.16958	50.2834

Chiral HPLC chromatogram for racemic **4e**



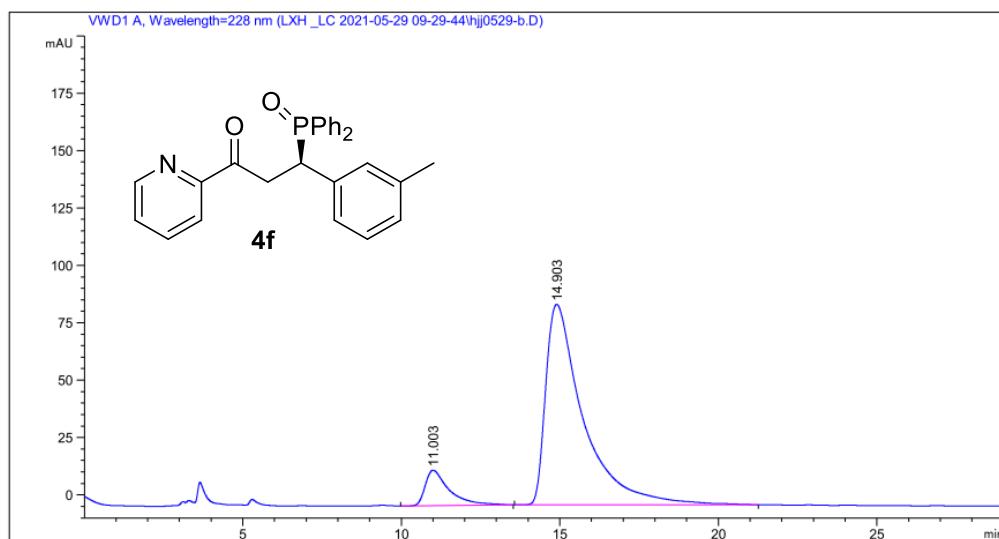
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	11.116	1058.98669	20.99481	15.3497
2	16.732	5840.08154	60.67751	84.6503

Chiral HPLC chromatogram for enantioenriched **4e**



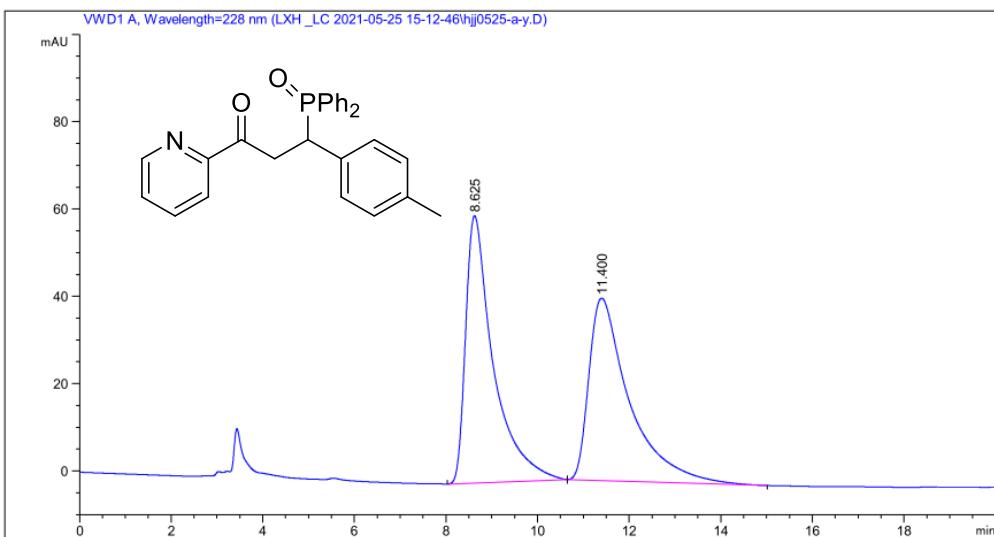
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	10.906	8583.00391	152.13066	50.2152
2	14.962	8509.45215	100.78463	49.7848

Chiral HPLC chromatogram for racemic **4f**

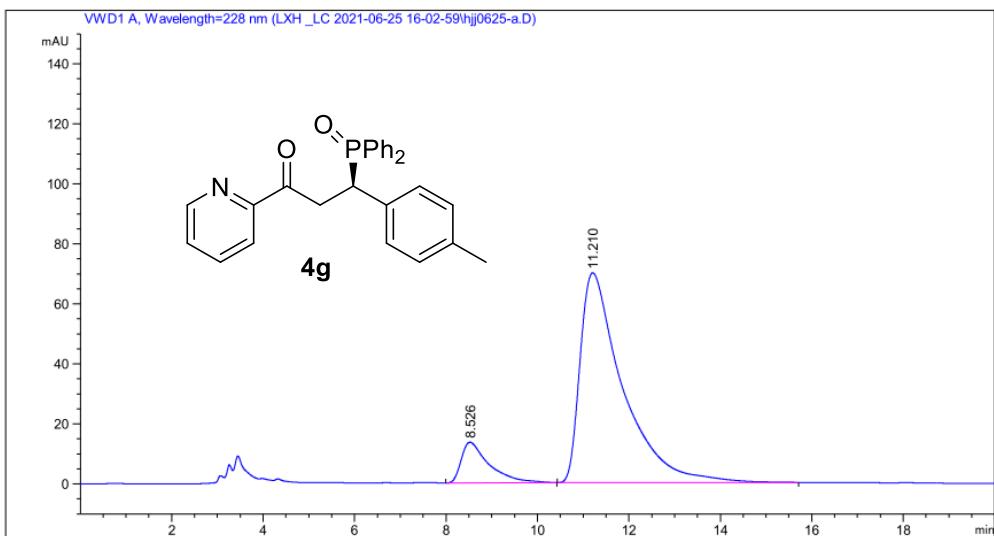


PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	11.003	807.63409	15.4145	10.0869
2	14.903	7199.14160	87.30708	89.9131

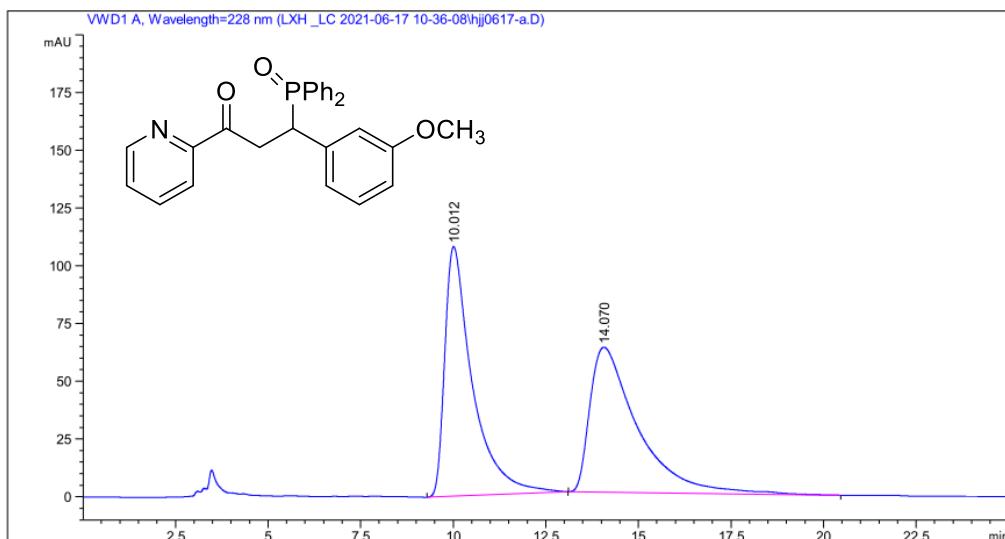
Chiral HPLC chromatogram for enantioenriched **4f**



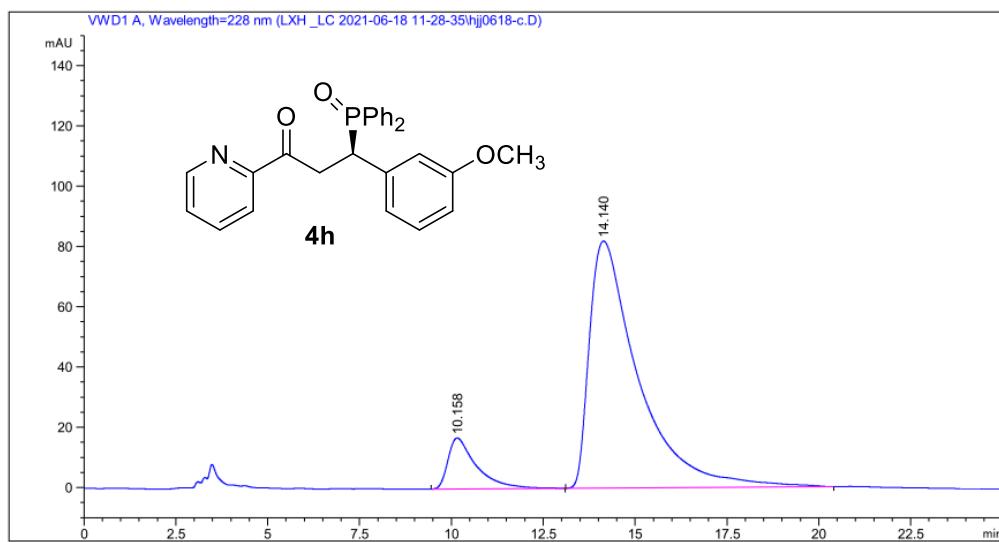
Chiral HPLC chromatogram for racemic **4g**



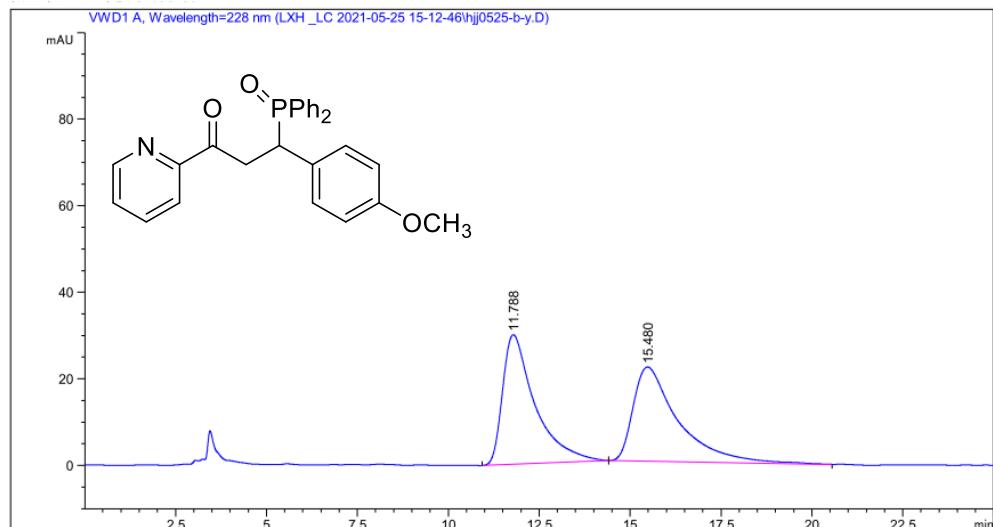
Chiral HPLC chromatogram for enantioenriched **4g**



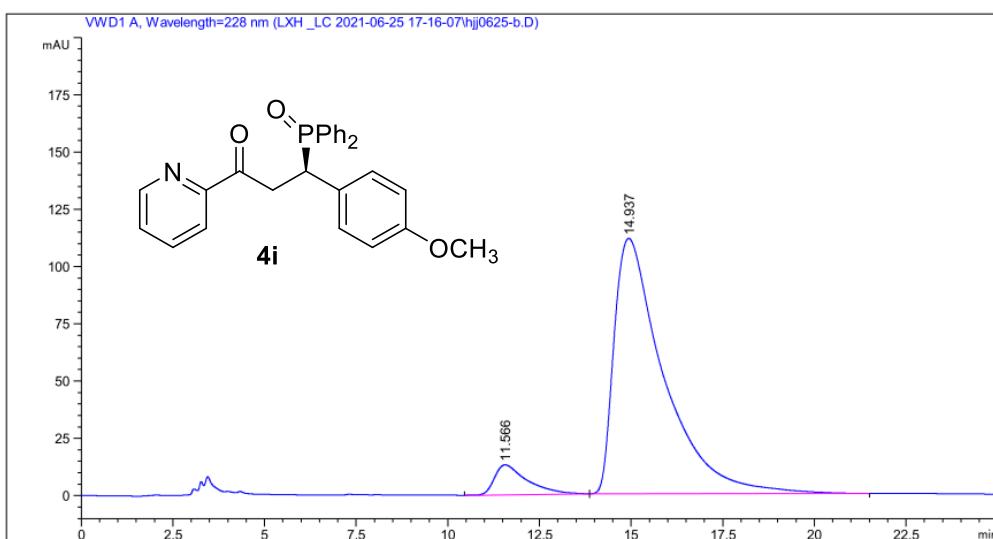
Chiral HPLC chromatogram for racemic **4h**



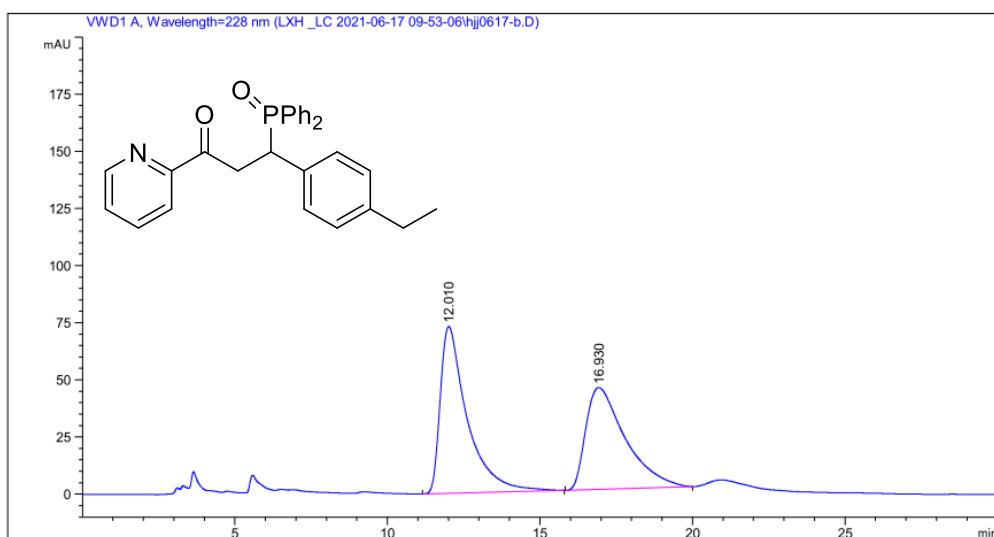
Chiral HPLC chromatogram for enantioenriched **4h**



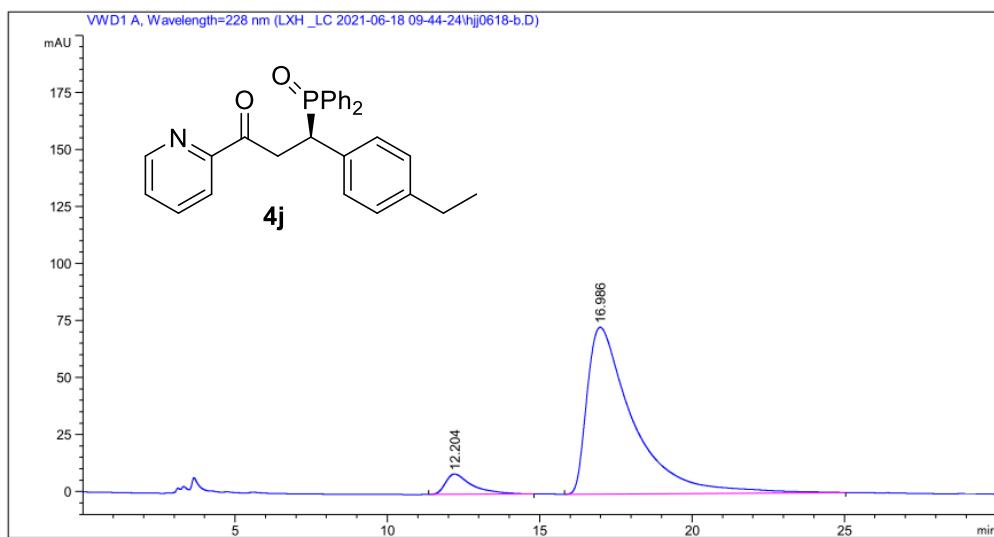
Chiral HPLC chromatogram for racemic **4i**



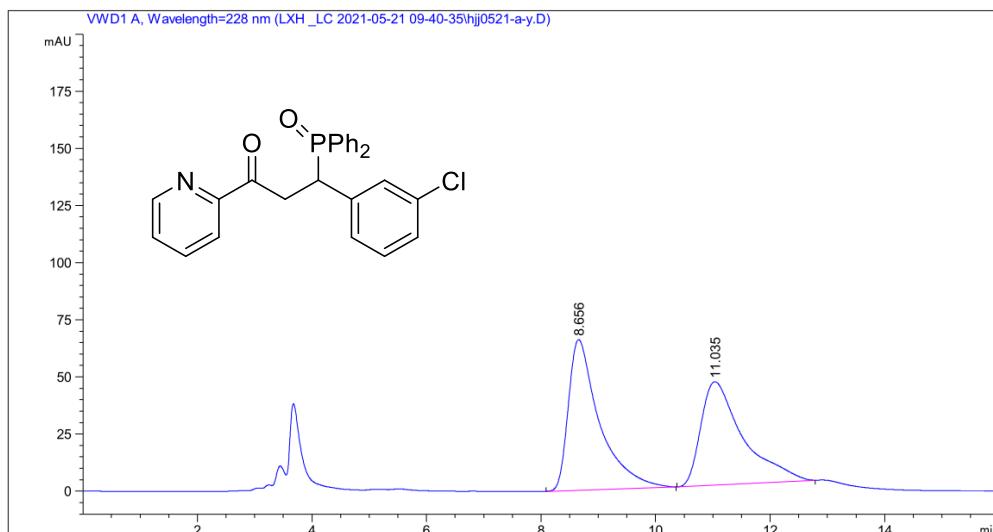
Chiral HPLC chromatogram for enantioenriched **4i**



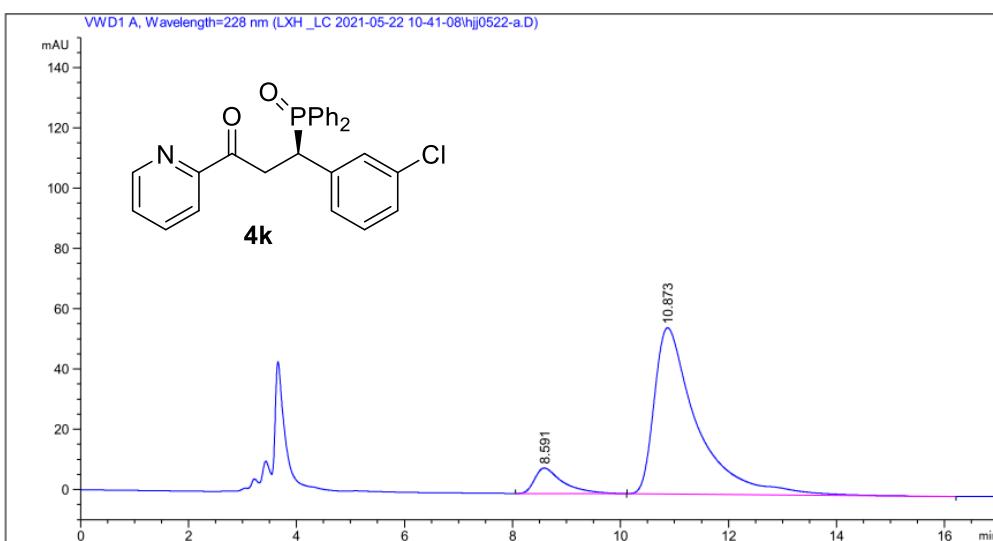
Chiral HPLC chromatogram for racemic **4j**



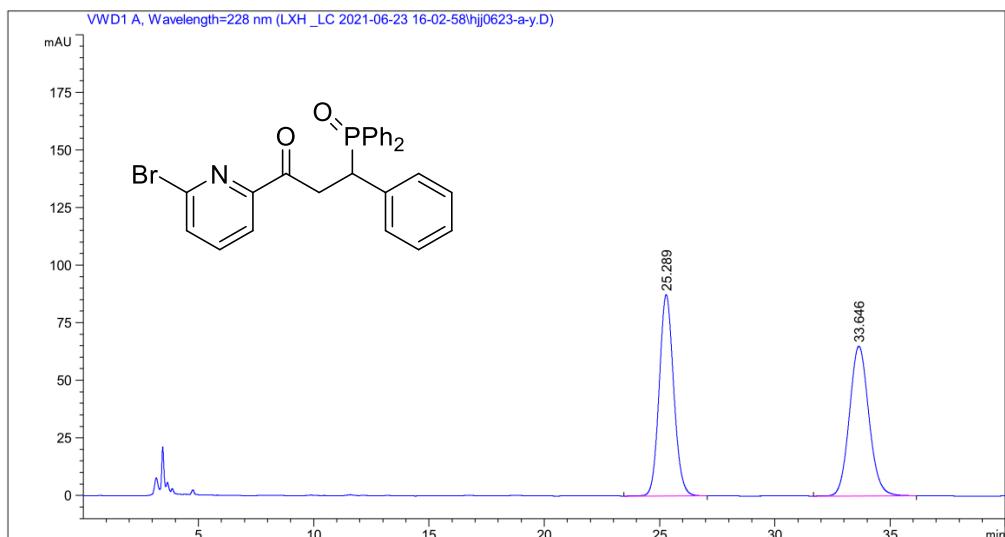
Chiral HPLC chromatogram for enantioenriched **4j**



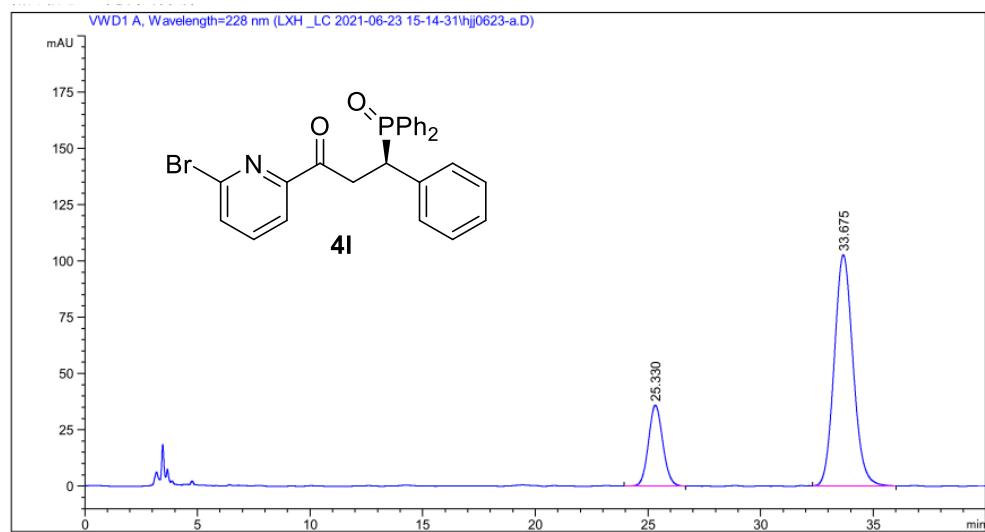
Chiral HPLC chromatogram for racemic **4k**



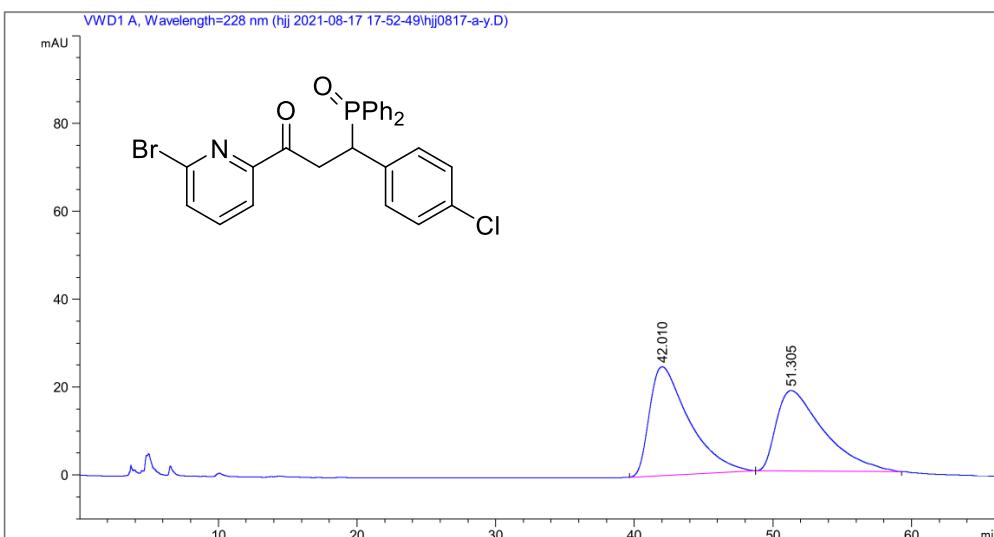
Chiral HPLC chromatogram for enantioenriched **4k**



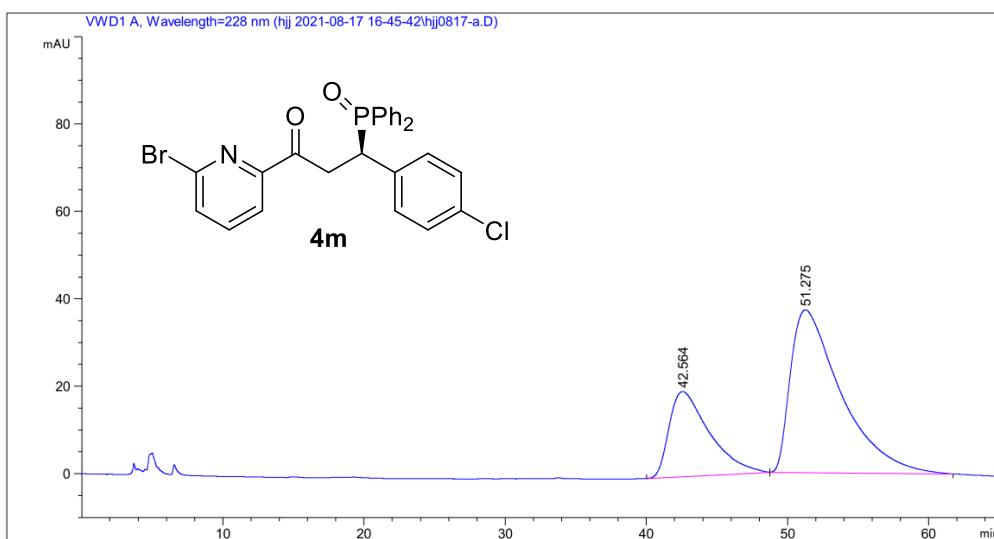
Chiral HPLC chromatogram for racemic **4l**



Chiral HPLC chromatogram for enantioenriched **4l**

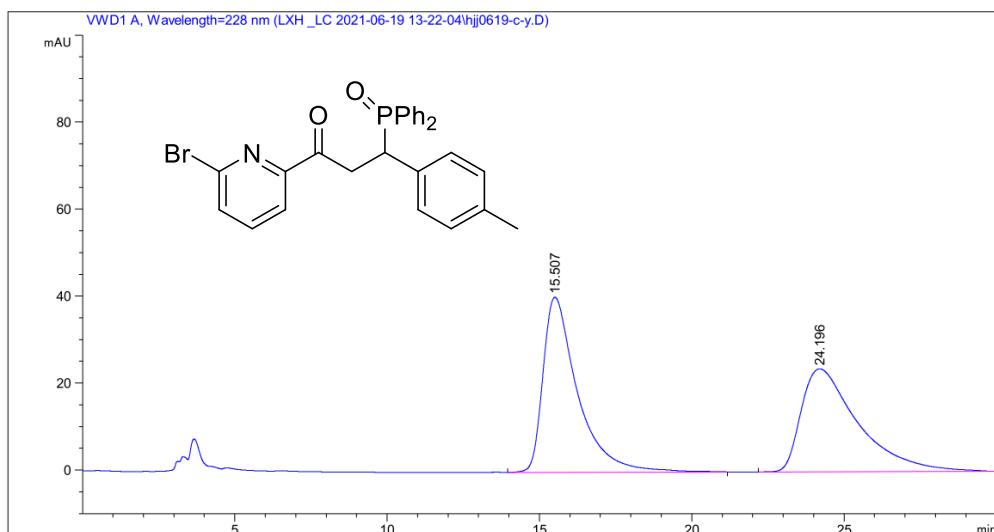


Chiral HPLC chromatogram for racemic **4m**



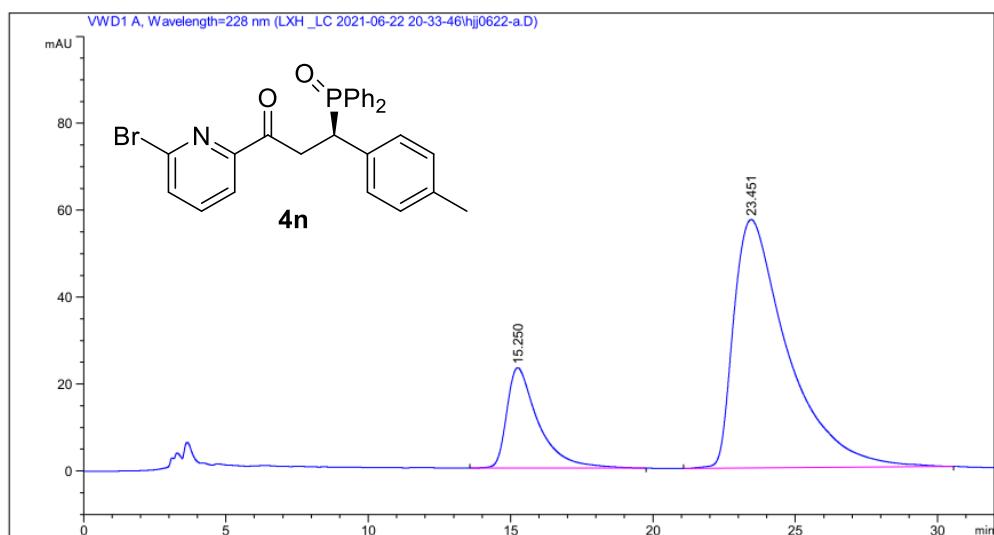
Chiral HPLC chromatogram for enantioenriched **4m**

PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	42.564	3842.41357	19.47927	29.2509
2	51.275	9293.63477	37.23388	70.7491



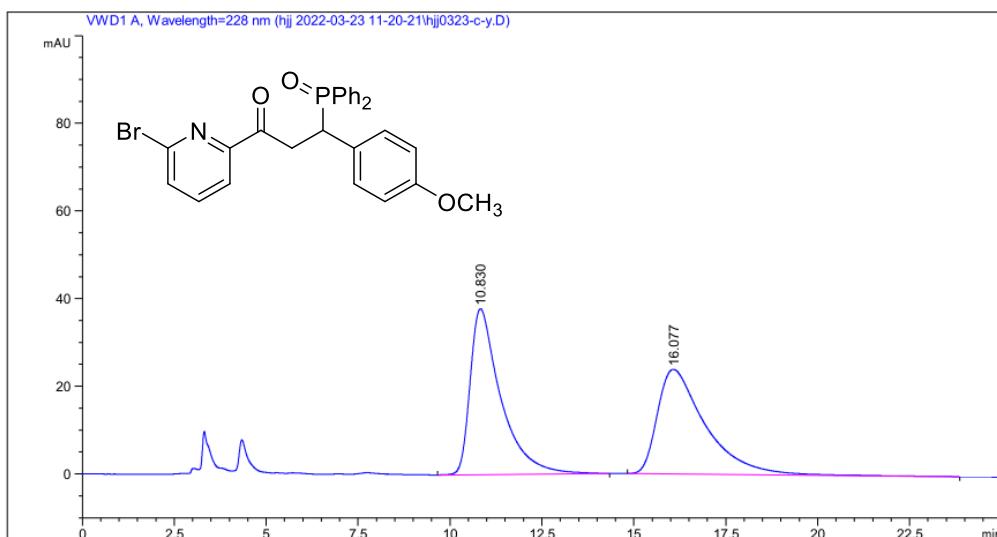
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	15.507	3184.20508	40.29589	50.9556
2	24.196	3064.77148	23.65708	49.0444

Chiral HPLC chromatogram for racemic **4n**

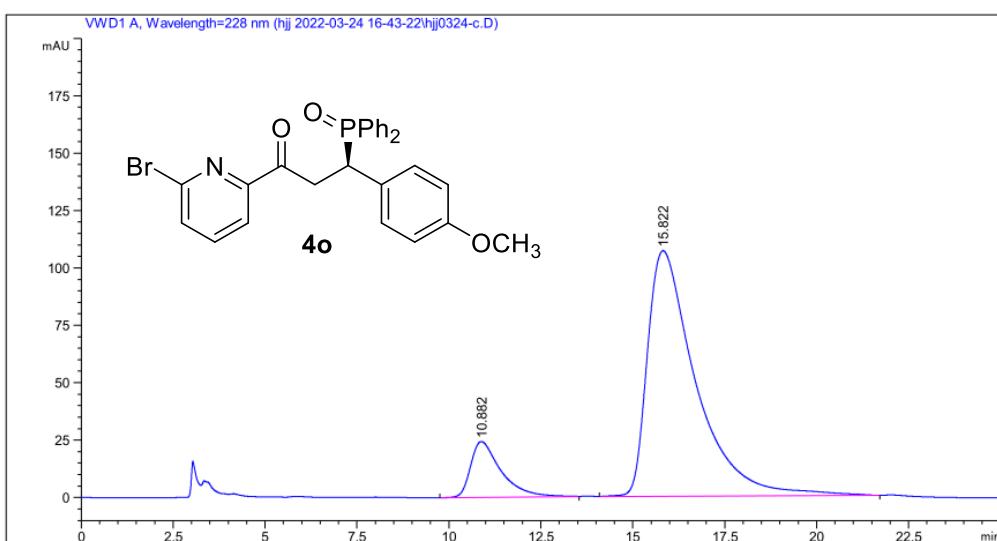


PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	15.250	1780.59644	23.09826	19.0620
2	23.451	7560.46826	57.18015	80.9380

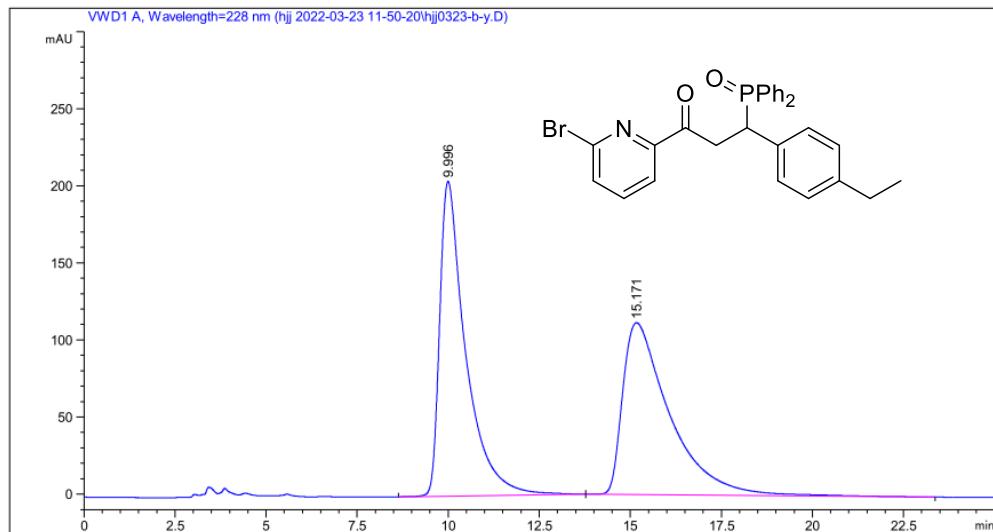
Chiral HPLC chromatogram for enantioenriched **4n**



Chiral HPLC chromatogram for racemic **4o**

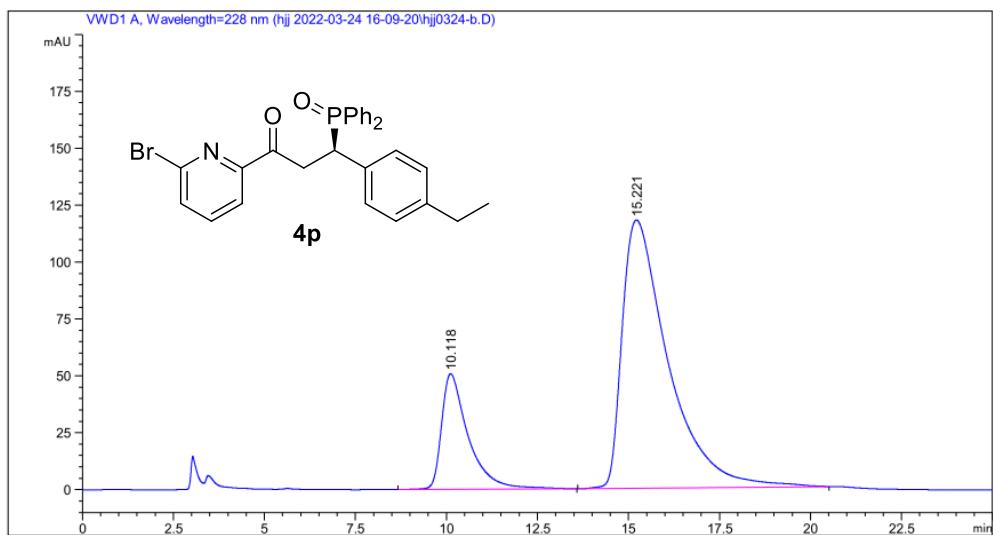


Chiral HPLC chromatogram for enantioenriched **4o**



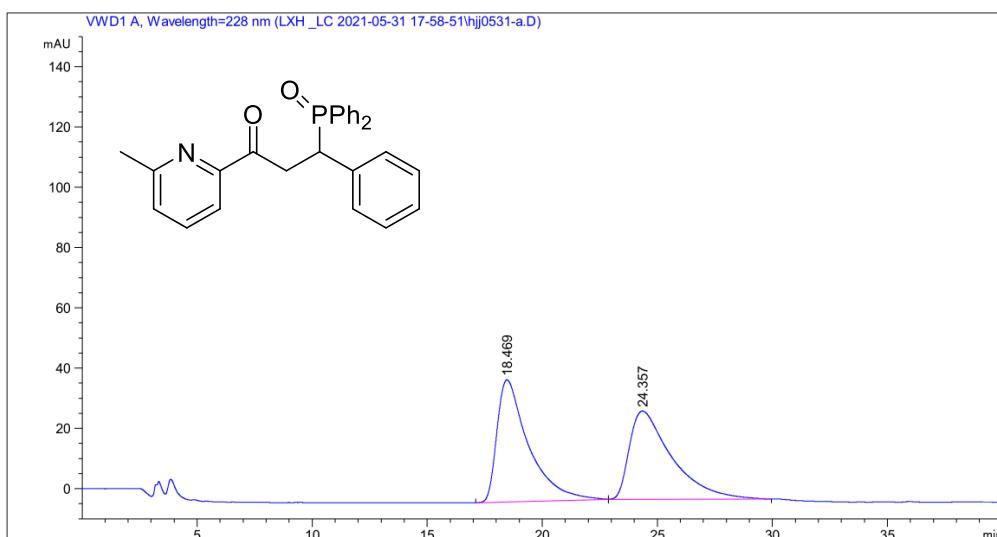
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	9.996	1.01281e4	204.05437	50.2544
2	15.171	1.00255e4	111.44259	49.7456

Chiral HPLC chromatogram for racemic **4p**

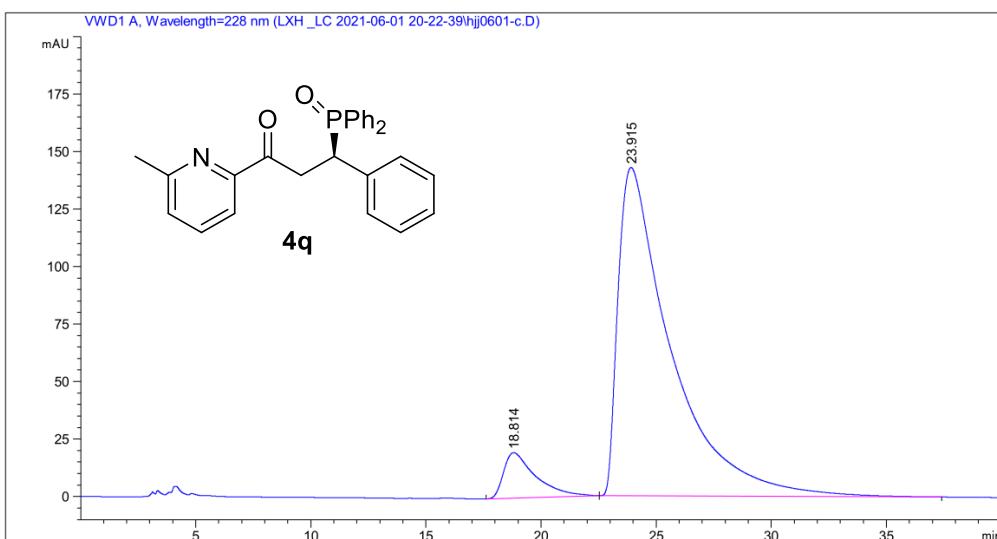


PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	10.118	2590.84888	50.73385	19.9893
2	15.221	1.03703e4	117.92656	80.0107

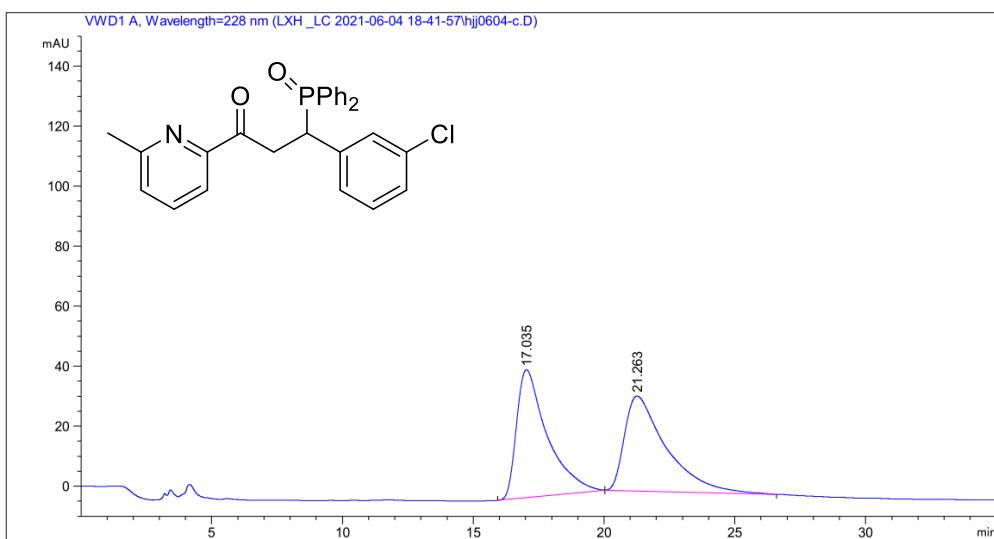
Chiral HPLC chromatogram for enantioenriched **4p**



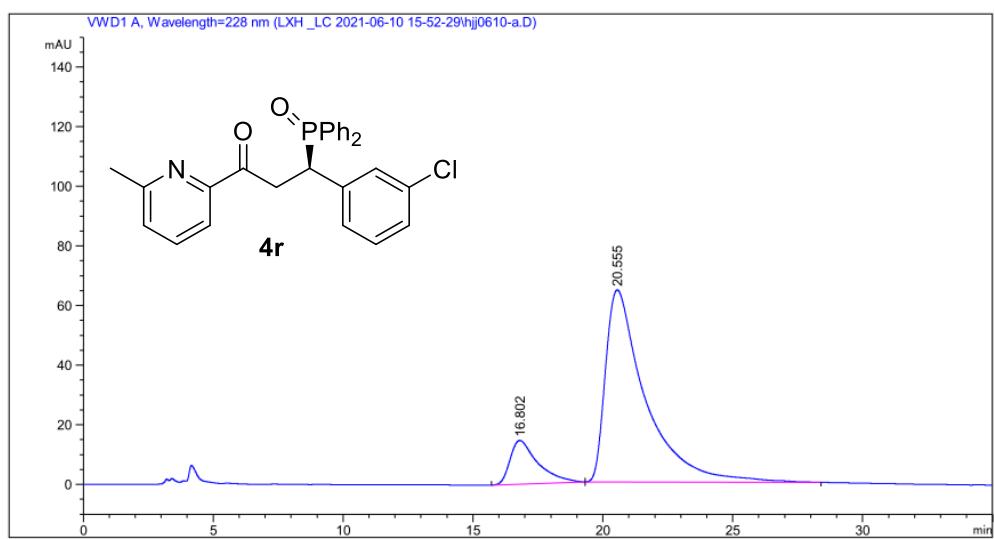
Chiral HPLC chromatogram for racemic **4q**



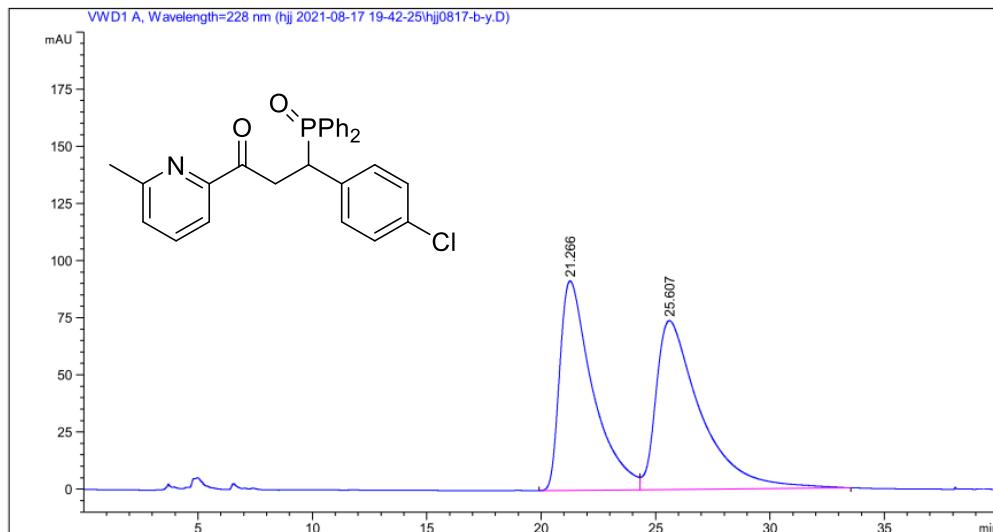
Chiral HPLC chromatogram for enantioenriched **4q**



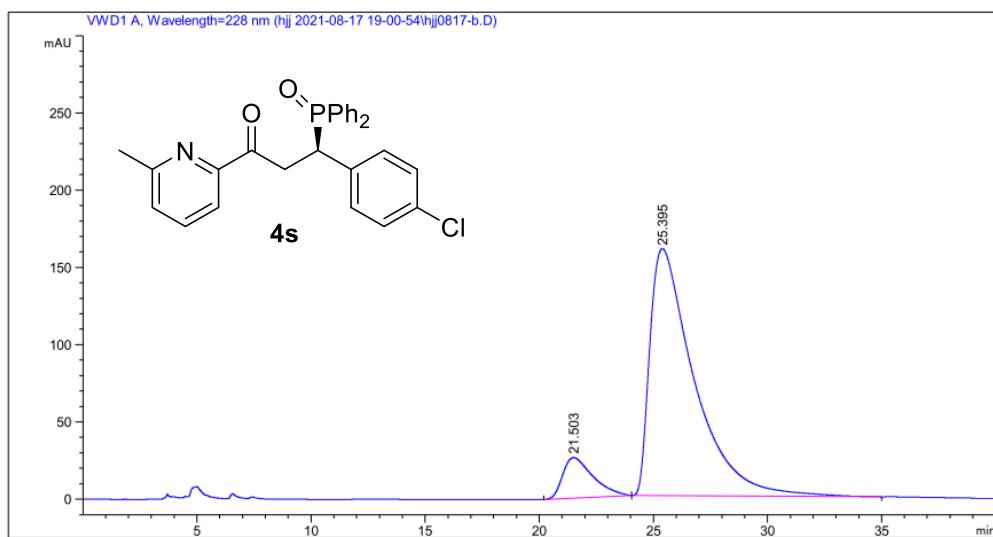
Chiral HPLC chromatogram for racemic **4r**



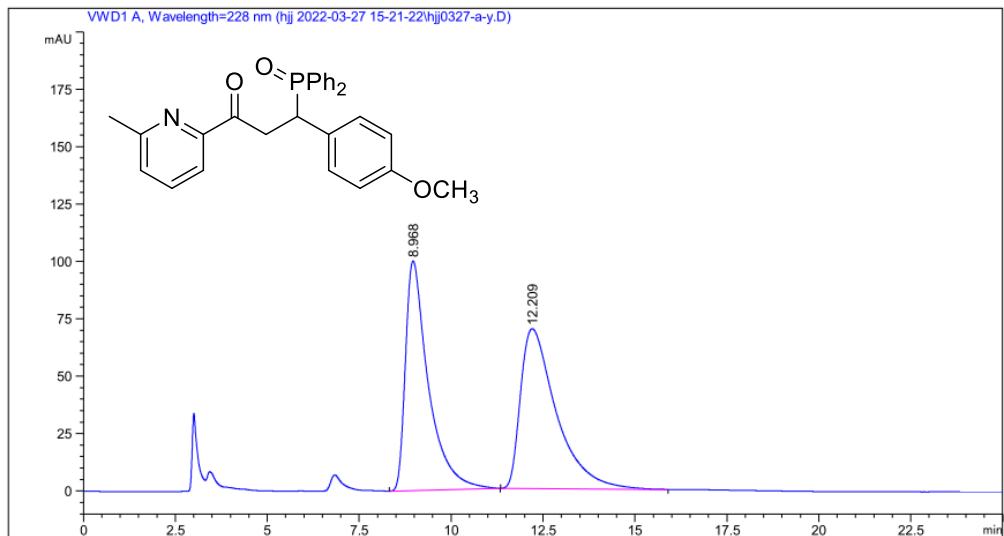
Chiral HPLC chromatogram for enantioenriched **4r**



Chiral HPLC chromatogram for racemic **4s**

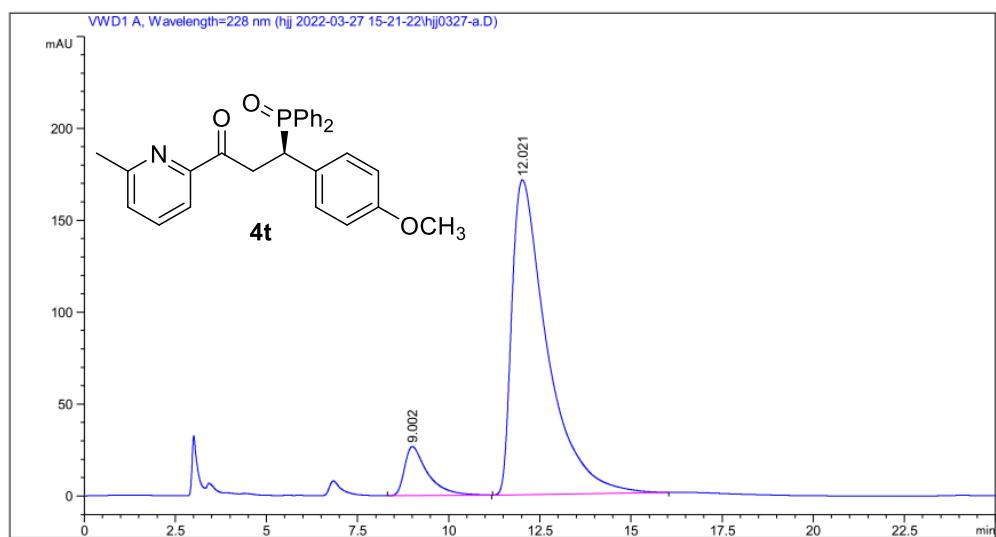


Chiral HPLC chromatogram for enantioenriched **4s**



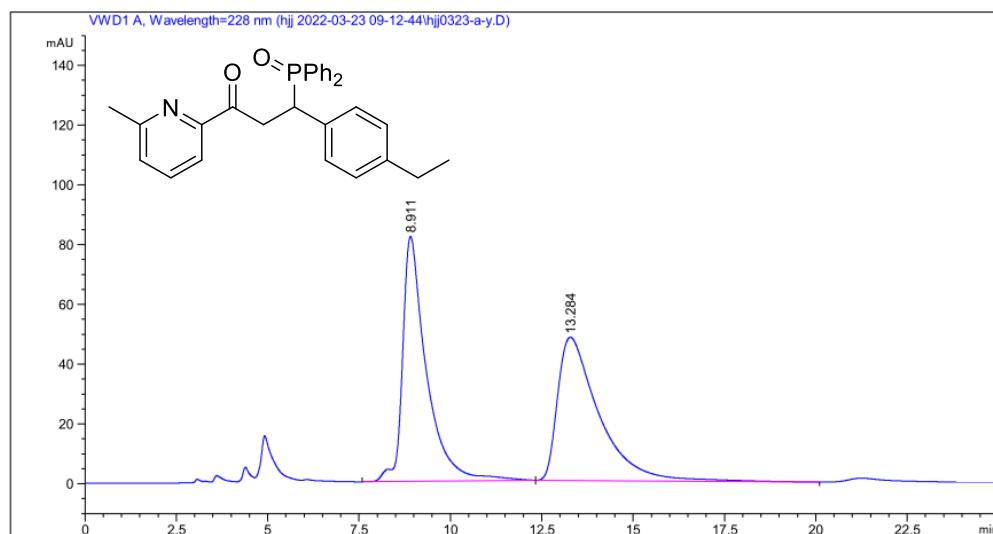
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	8.968	4341.99561	100.05734	47.8886
2	12.209	4724.86963	69.62543	52.1114

Chiral HPLC chromatogram for racemic **4t**

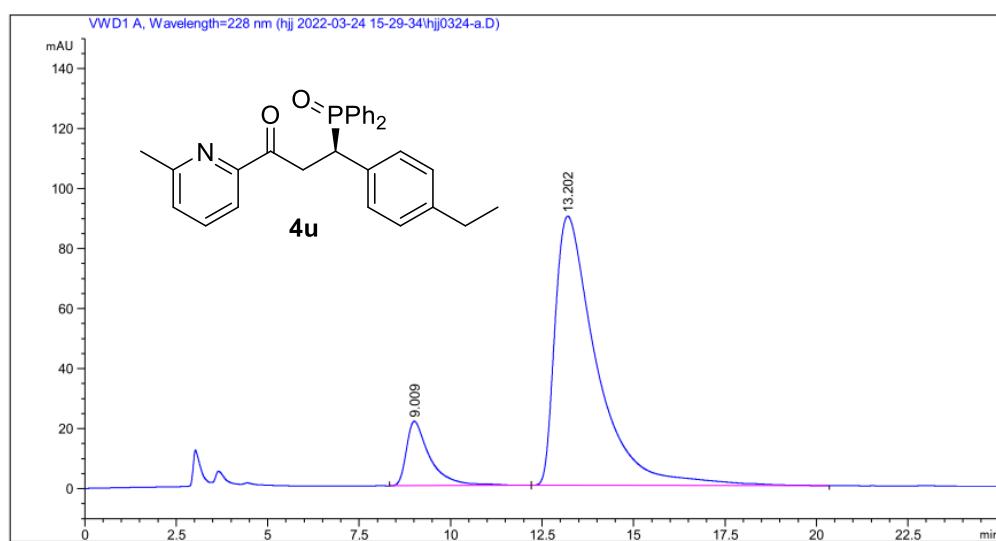


PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	9.002	1168.56824	26.66911	9.2120
2	12.021	1.15168e4	171.21254	90.7880

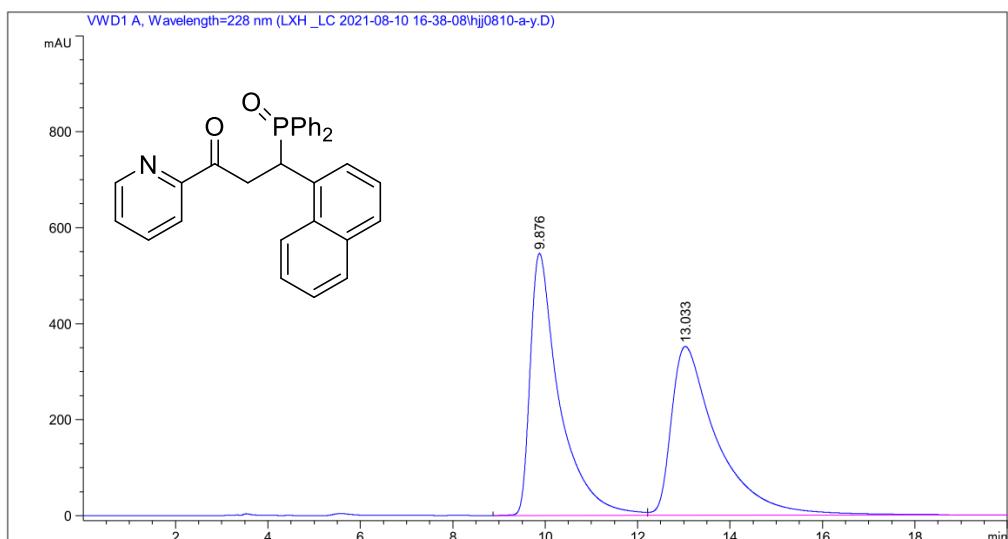
Chiral HPLC chromatogram for enantioenriched **4t**



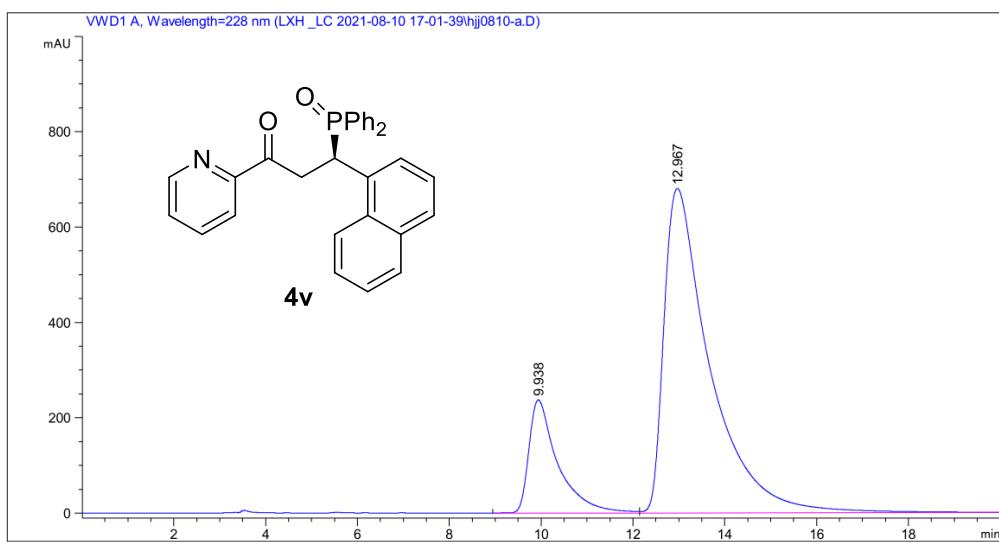
Chiral HPLC chromatogram for racemic **4u**



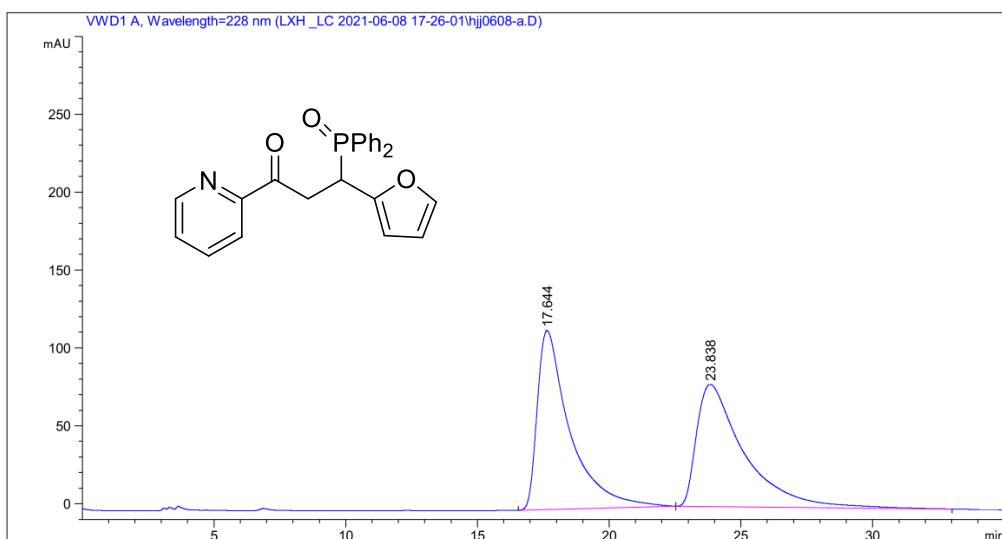
Chiral HPLC chromatogram for enantioenriched **4u**



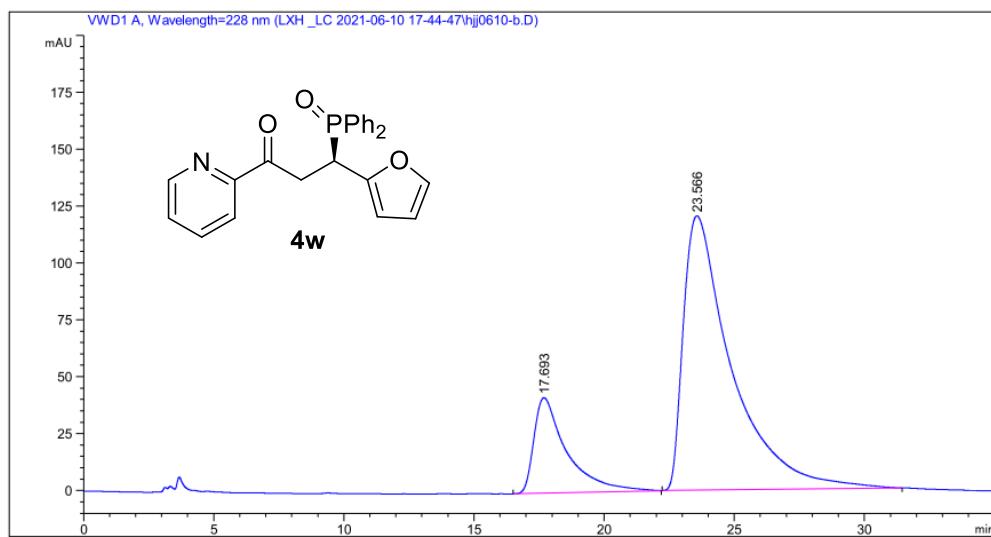
Chiral HPLC chromatogram for racemic **4v**



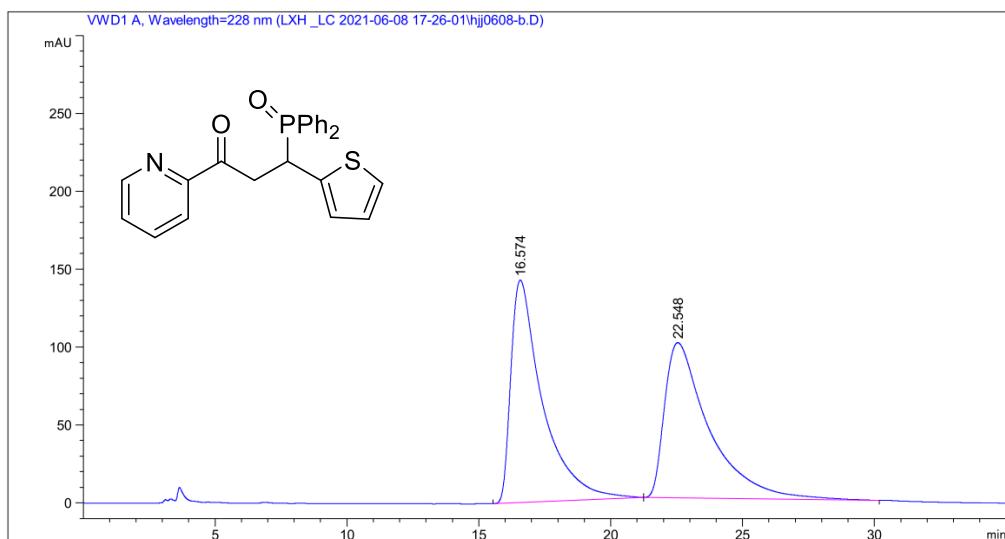
Chiral HPLC chromatogram for enantioenriched **4v**



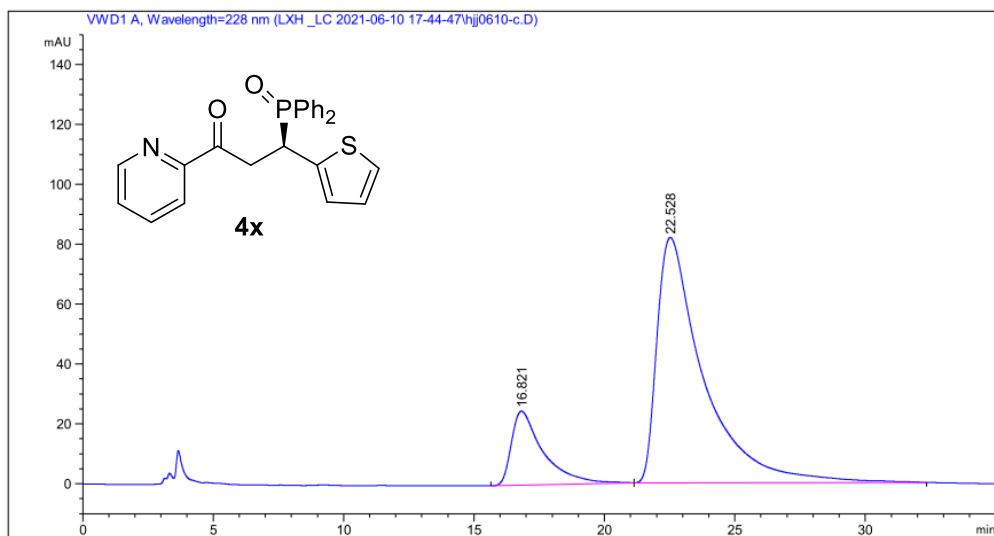
Chiral HPLC chromatogram for racemic **4w**



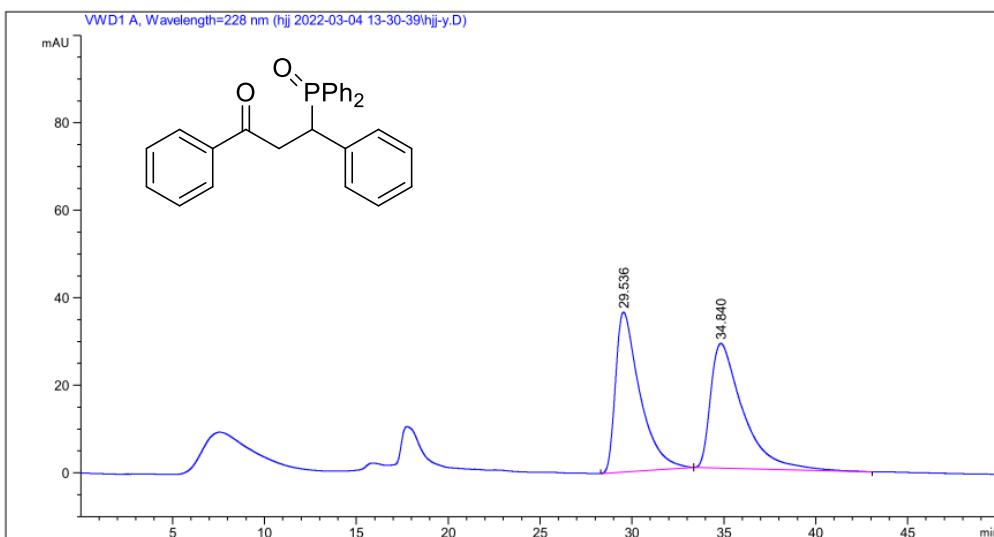
Chiral HPLC chromatogram for enantioenriched **4w**



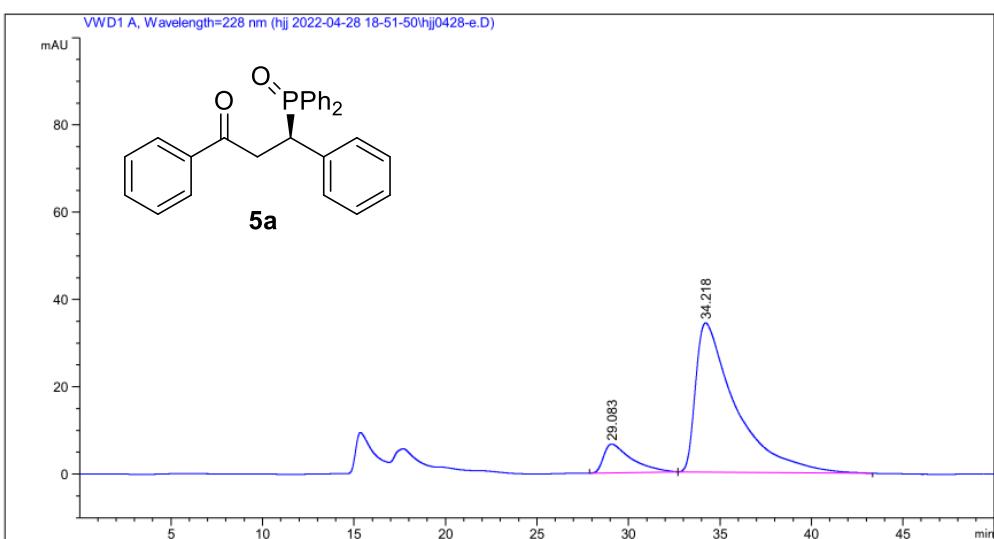
Chiral HPLC chromatogram for racemic **4x**



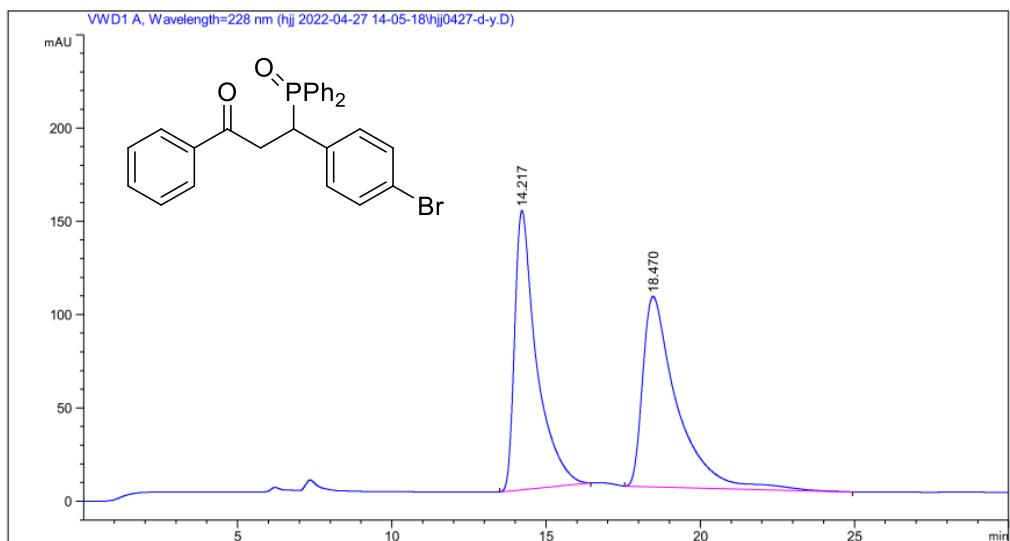
Chiral HPLC chromatogram for enantioenriched **4x**



Chiral HPLC chromatogram for racemic **5a**

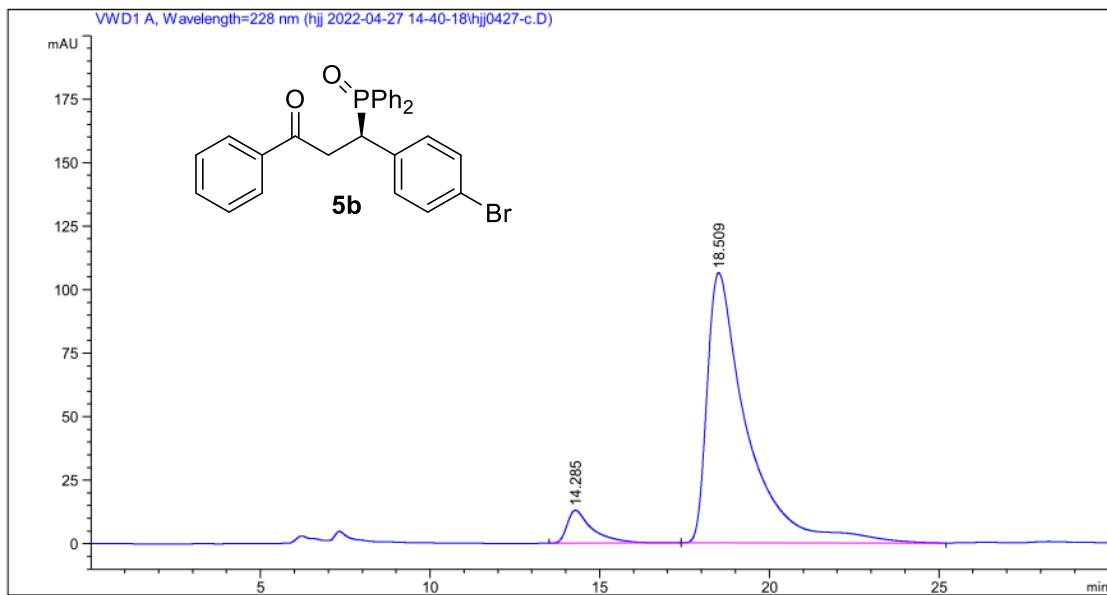


Chiral HPLC chromatogram for enantioenriched **5a**



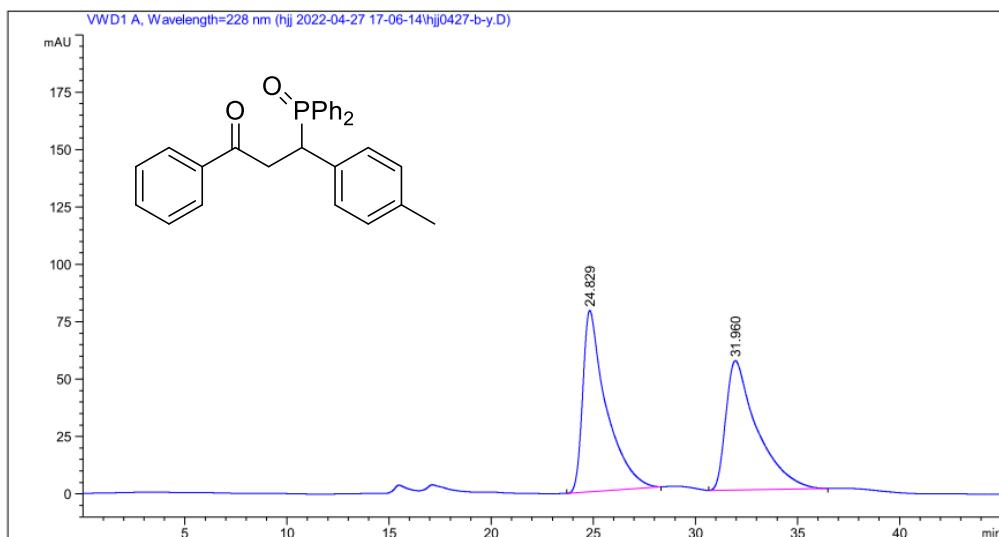
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	14.217	7367.39697	149.61864	48.3184
2	18.470	7880.20166	102.04332	51.6816

Chiral HPLC chromatogram for racemic **5b**



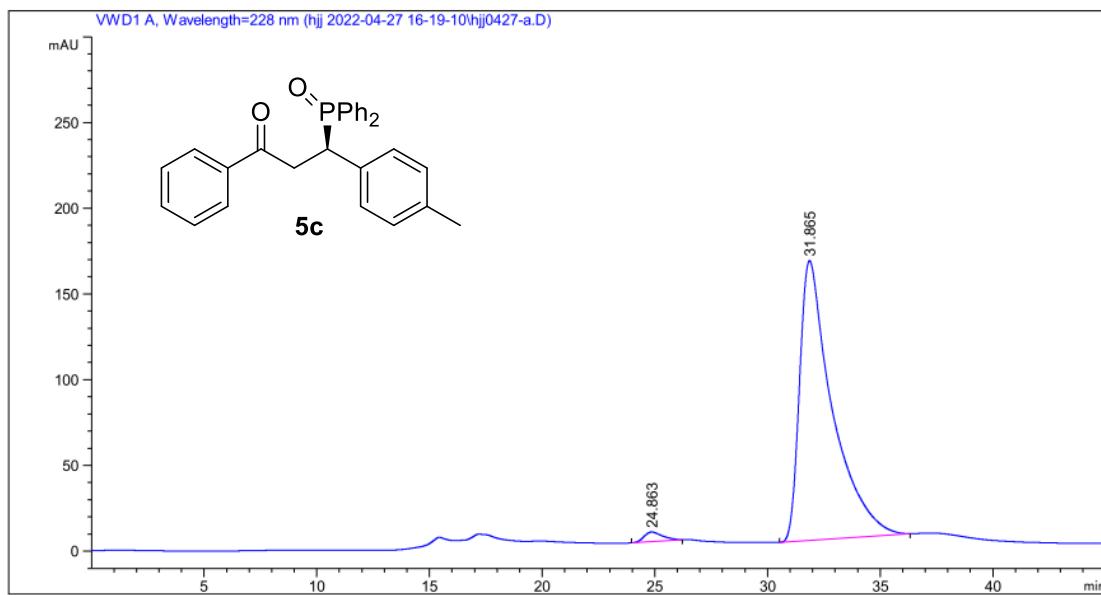
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	14.285	669.51398	12.96331	7.1524
2	18.509	8691.23926	106.29477	92.8476

Chiral HPLC chromatogram for enantioenriched **5b**



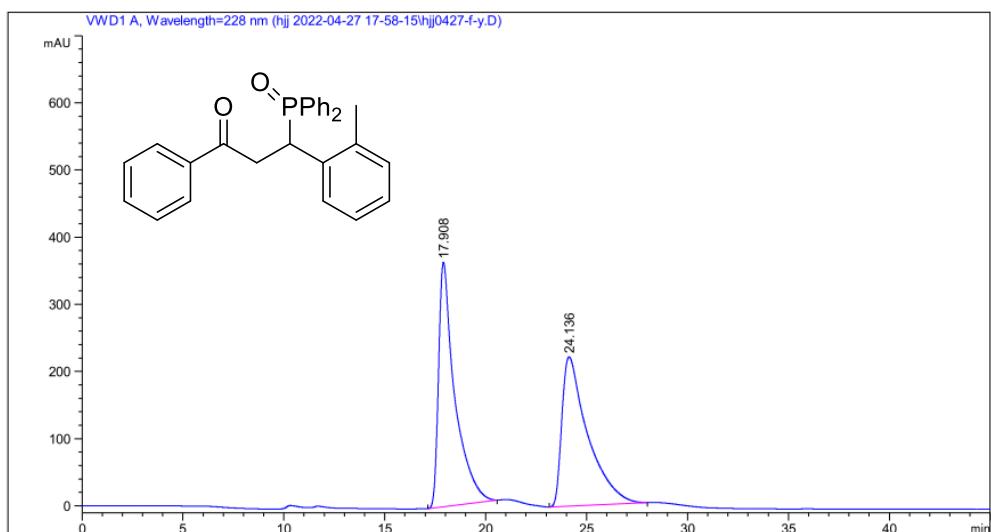
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	24.829	6301.59131	78.94345	51.3500
2	31.960	5970.25342	56.35709	48.6500

Chiral HPLC chromatogram for racemic **5c**



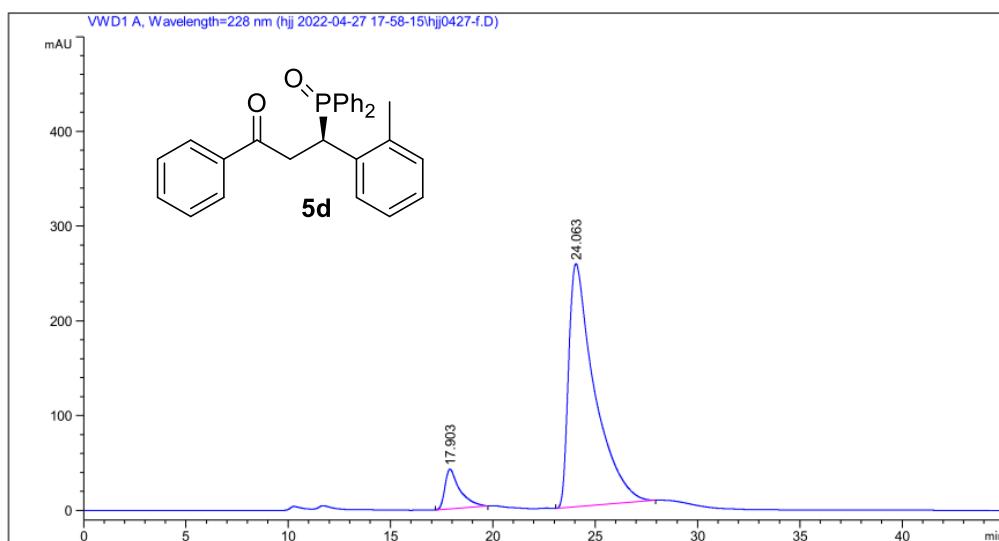
PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	24.863	316.61862	5.62219	1.8914
2	31.865	1.64237e4	163.20700	98.1086

Chiral HPLC chromatogram for enantioenriched **5c**



PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	17.908	2.02667e4	363.83209	50.7136
2	24.136	1.96963e4	222.19511	49.2864

Chiral HPLC chromatogram for racemic **5d**



PeakNO.	Ret.Time	PeakArea	PeakHeight	Area%
1	17.903	2114.00415	41.86325	8.5767
2	24.063	2.25343e4	256.58746	91.4233

Chiral HPLC chromatogram for enantioenriched **5d**