Electronic Supplementary Material (ESI) for RSC Advances.

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Supplementary Information

Asymmetric Transfer Hydrogenation of γ-aryl α, γ-dioxo-butyric acid esters†

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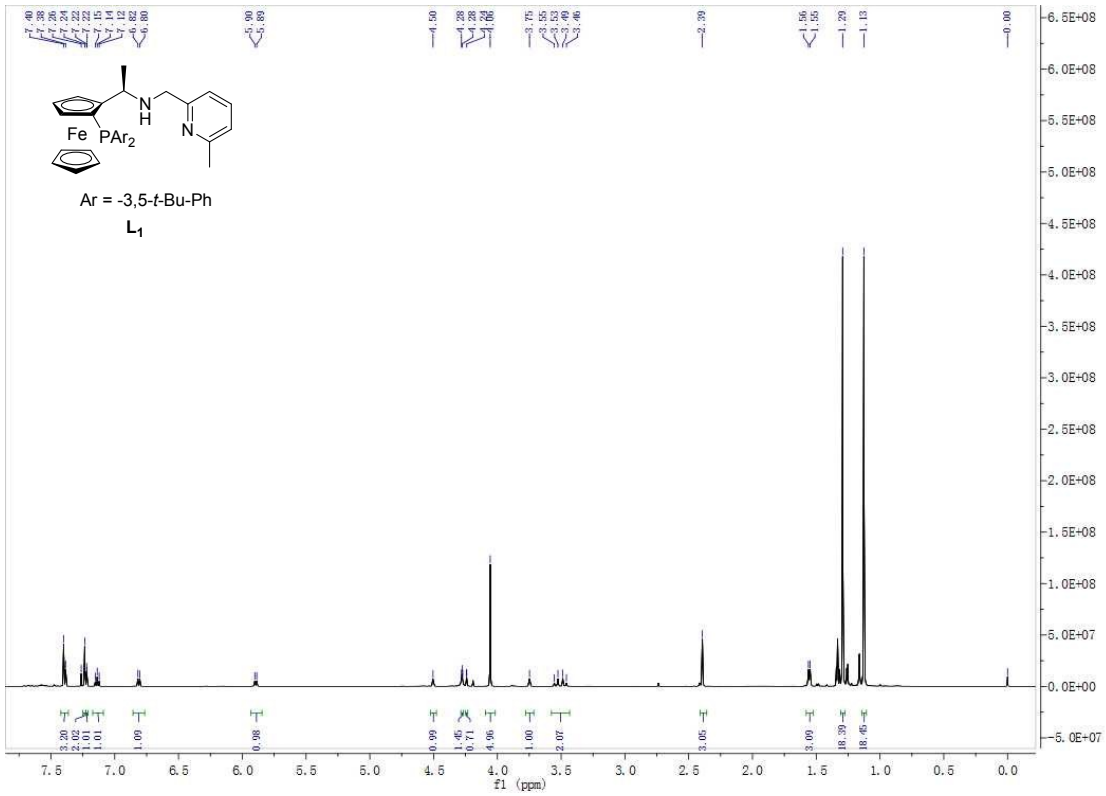
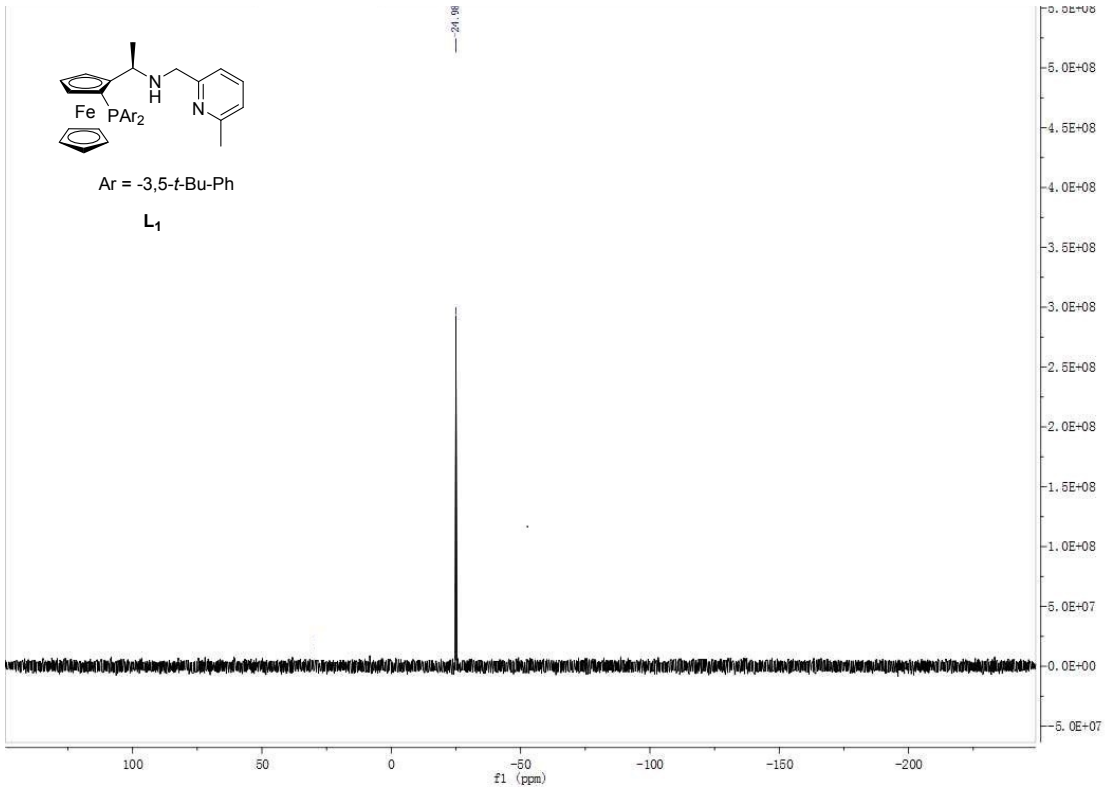
1.^{1}HNMR, ^{13}CNMR and ^{31}P NMR Spectra of L_1-L_6

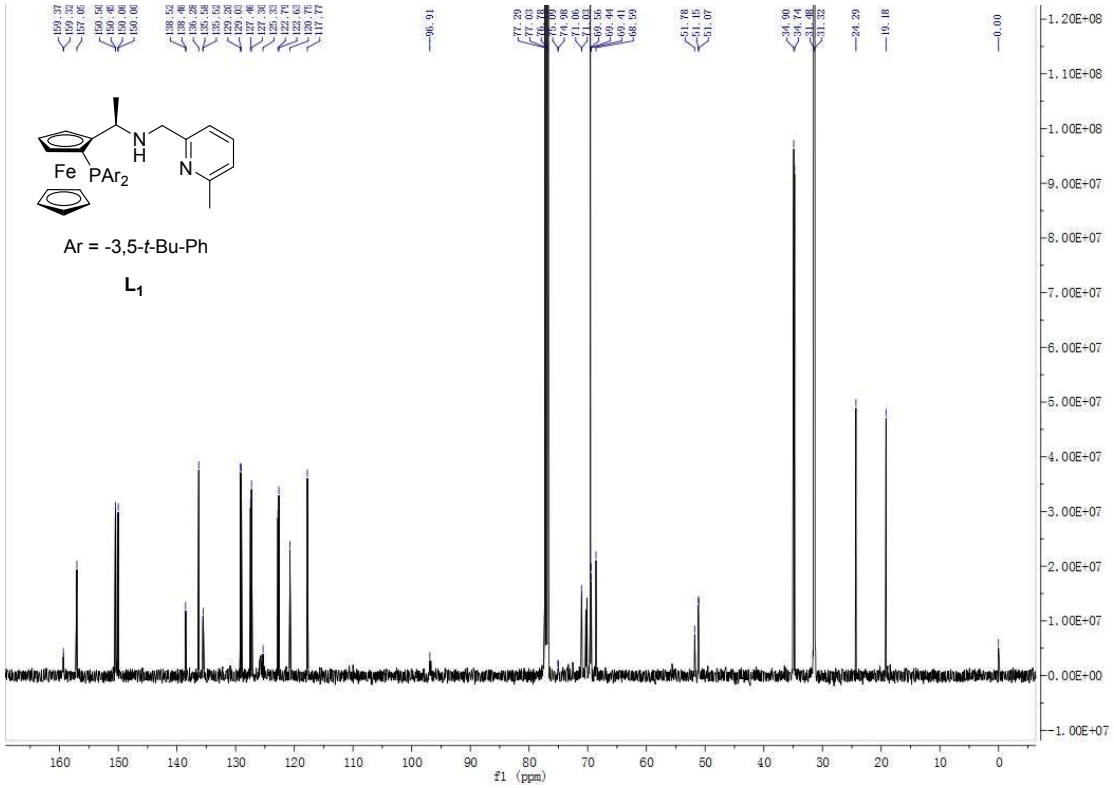
^{1}H NMR Spectra of L_1



^{31}P NMR Spectra of L_1

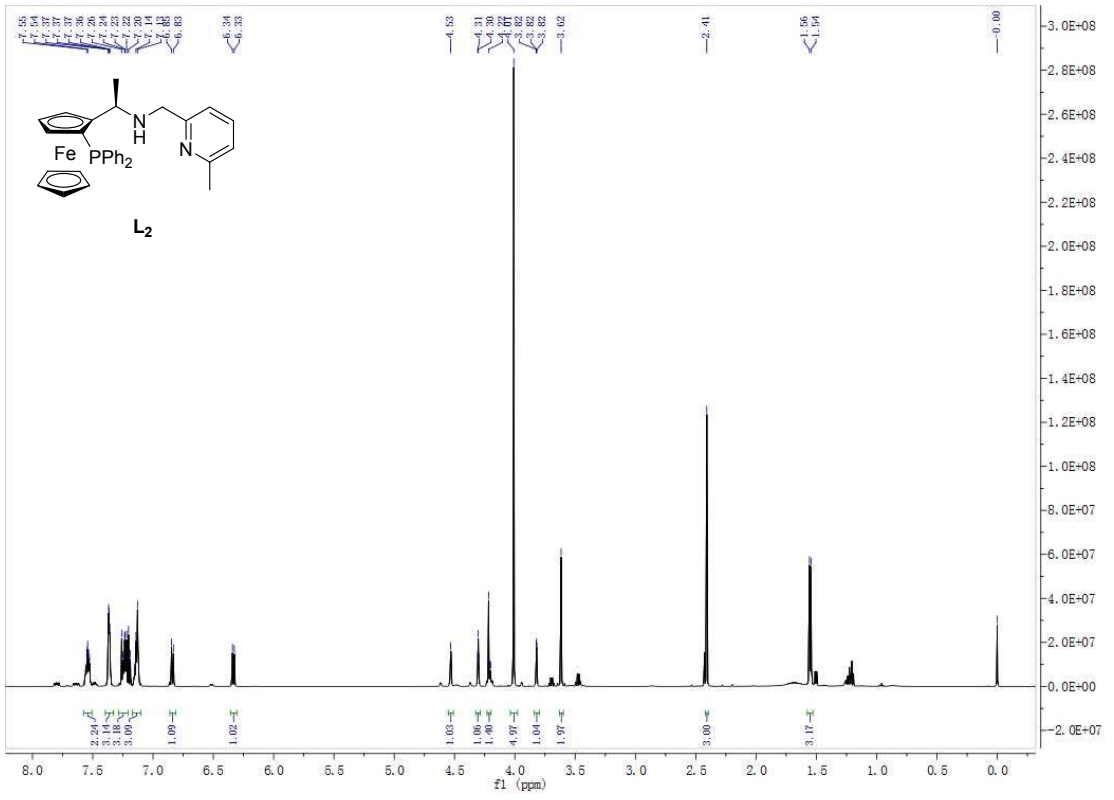


^{13}C NMR Spectra of L_1



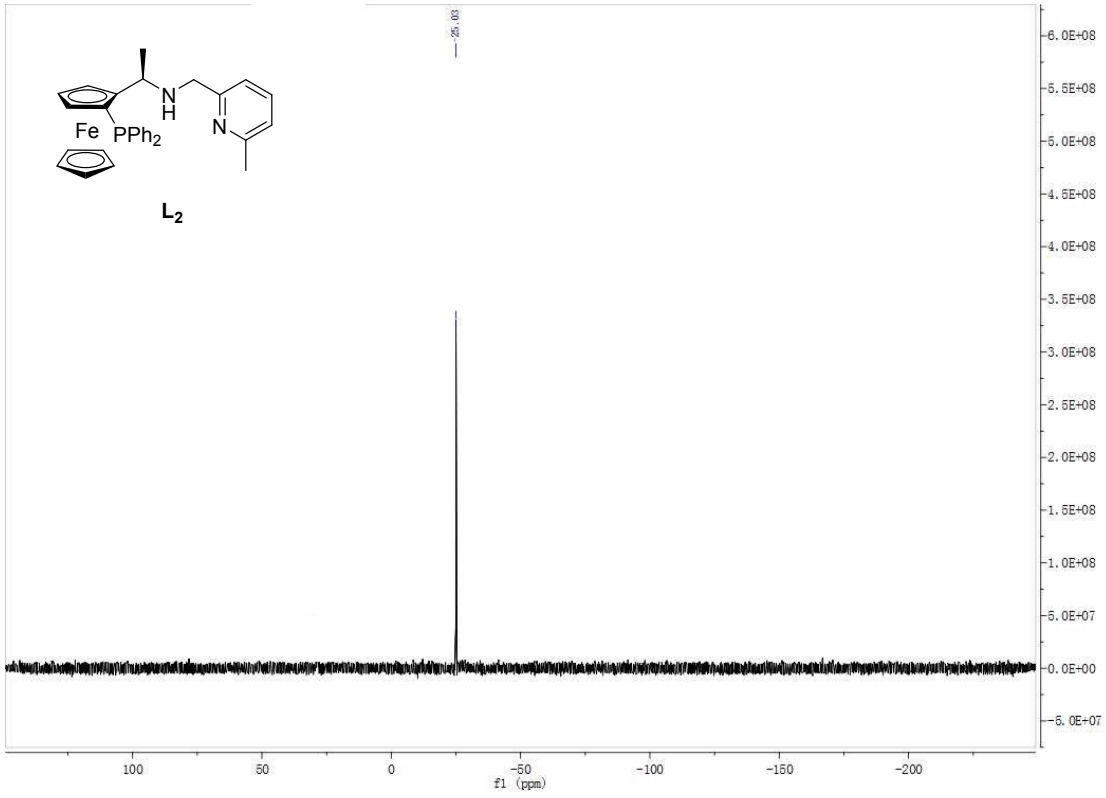
L_1

^{1}H NMR Spectra of L_2

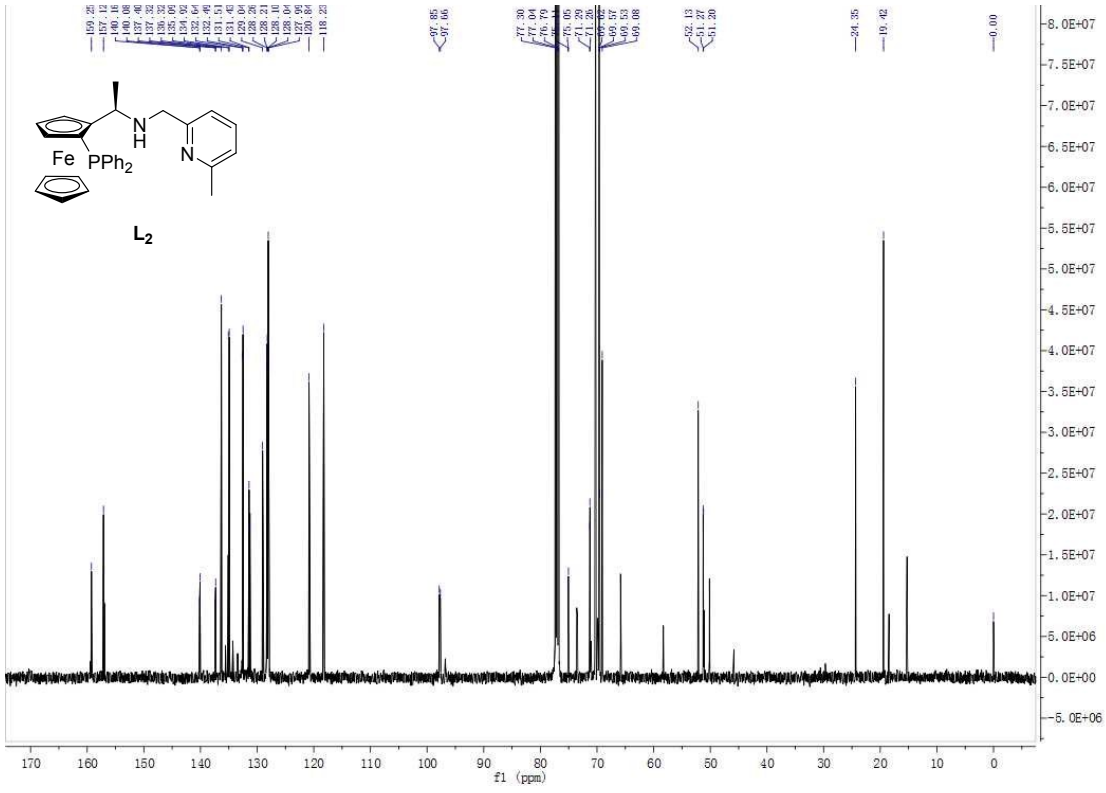


L_2

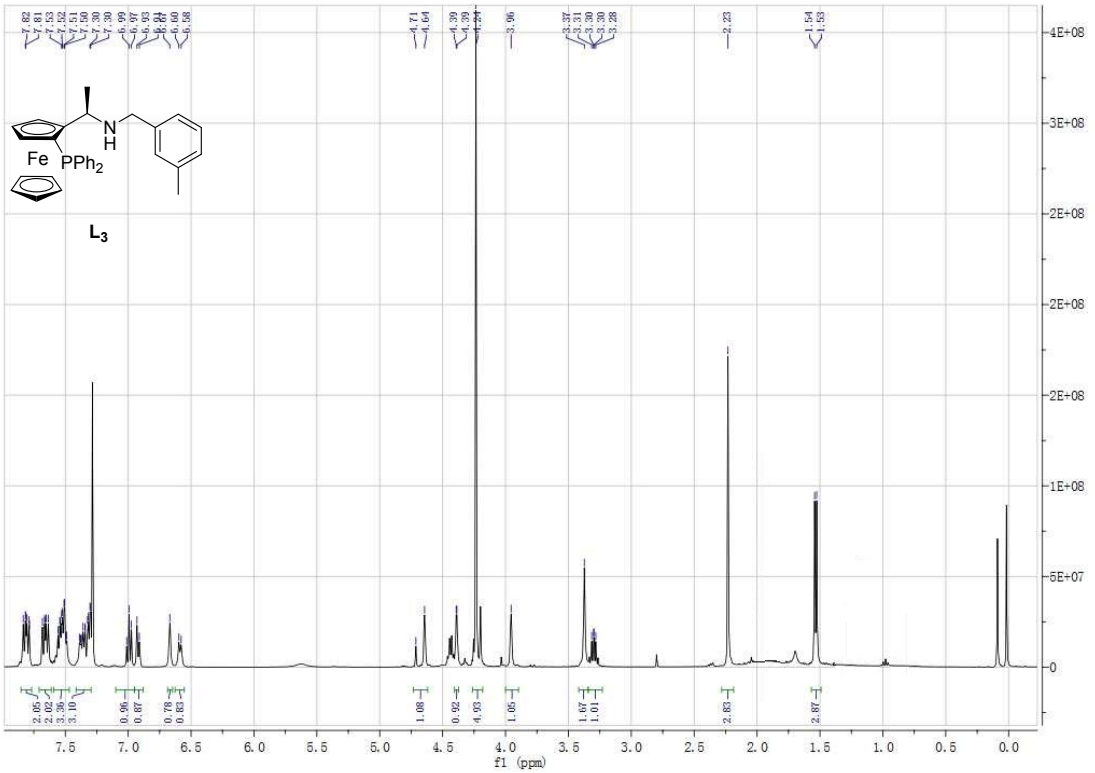
^{31}P NMR Spectra of L_2



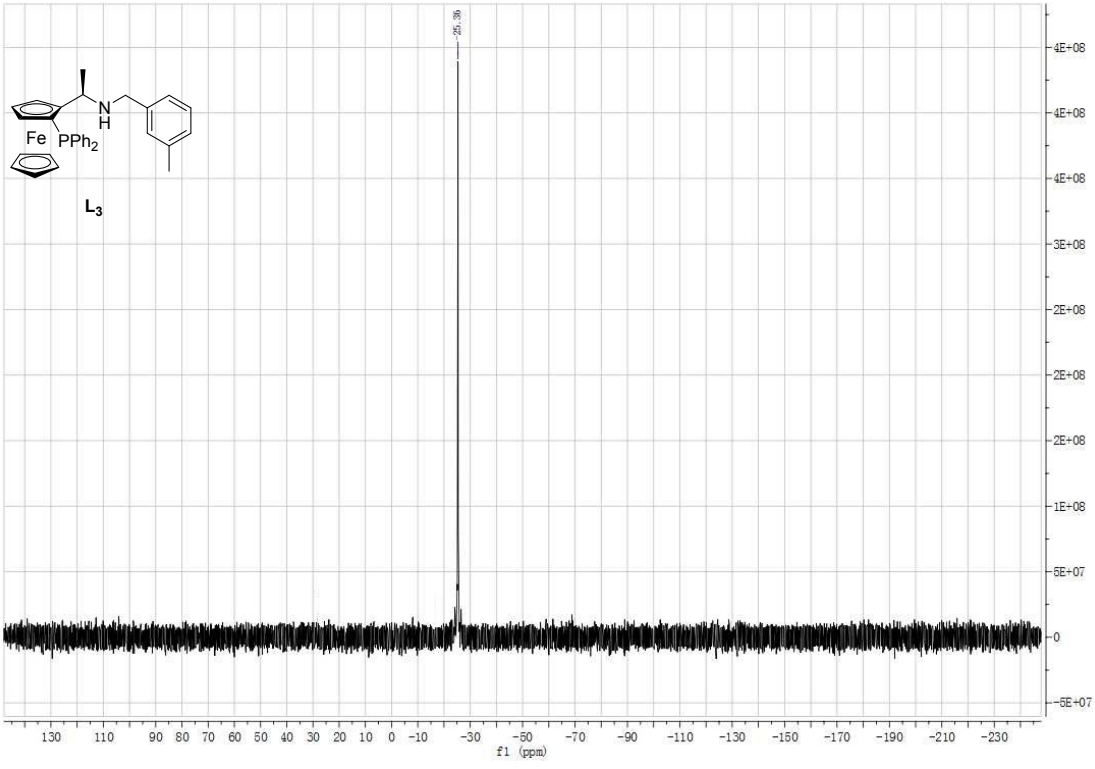
^{13}C NMR Spectra of L_2



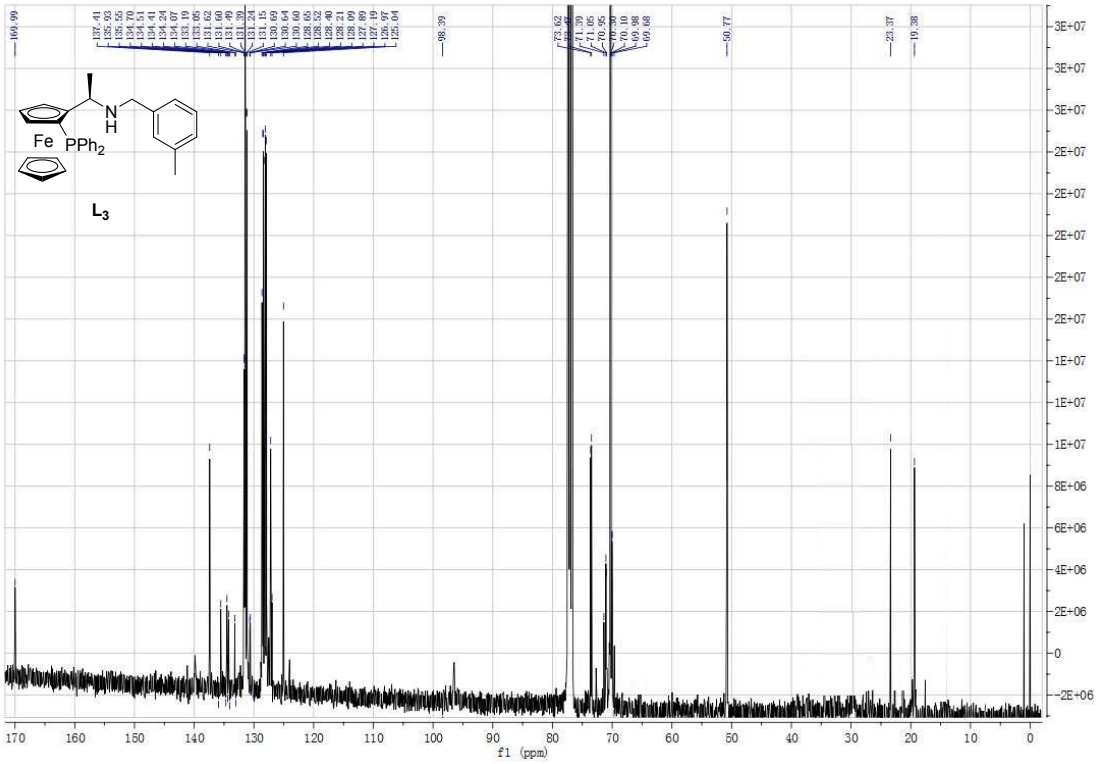
^{1}H NMR Spectra of L_3



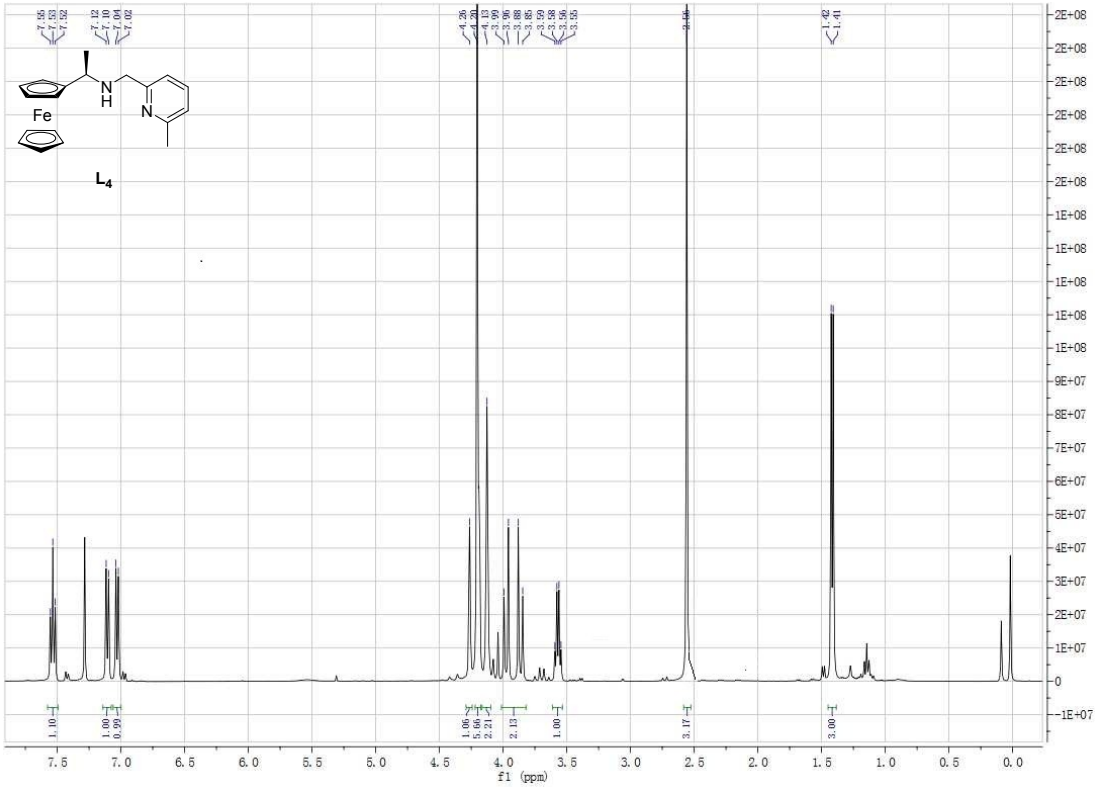
^{31}P NMR Spectra of L_3



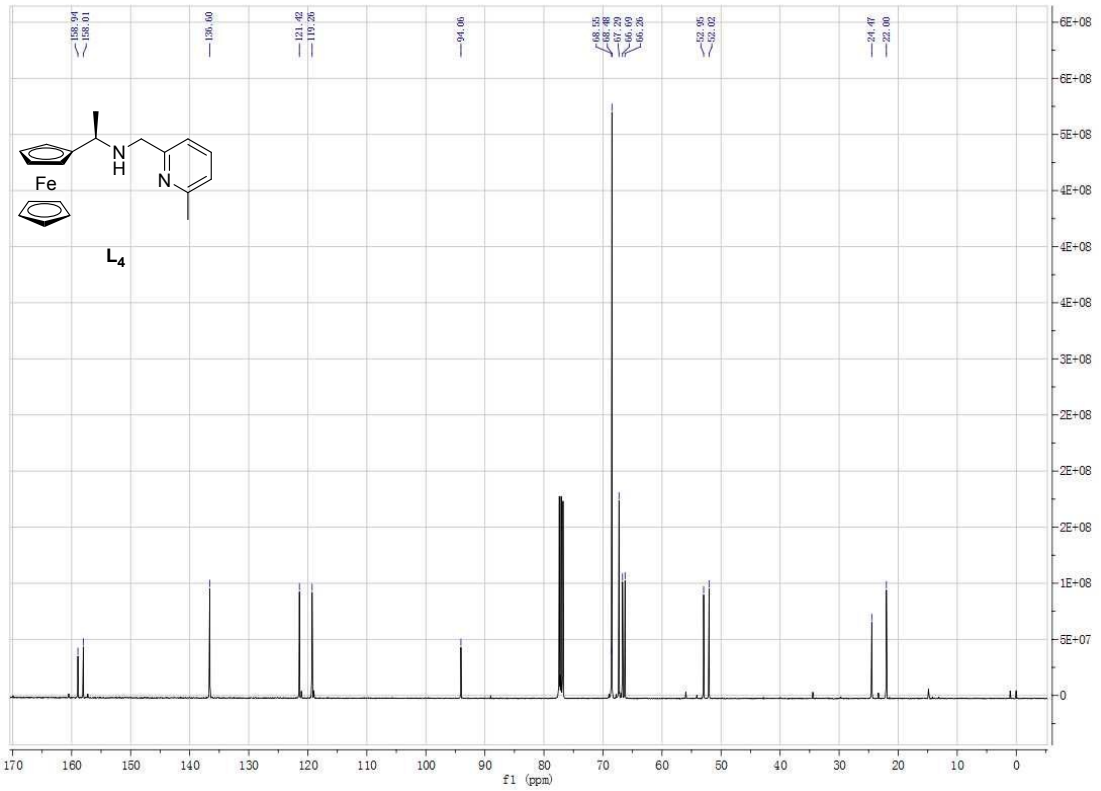
^{13}C NMR Spectra of L_3



^{1}H NMR Spectra of L_4

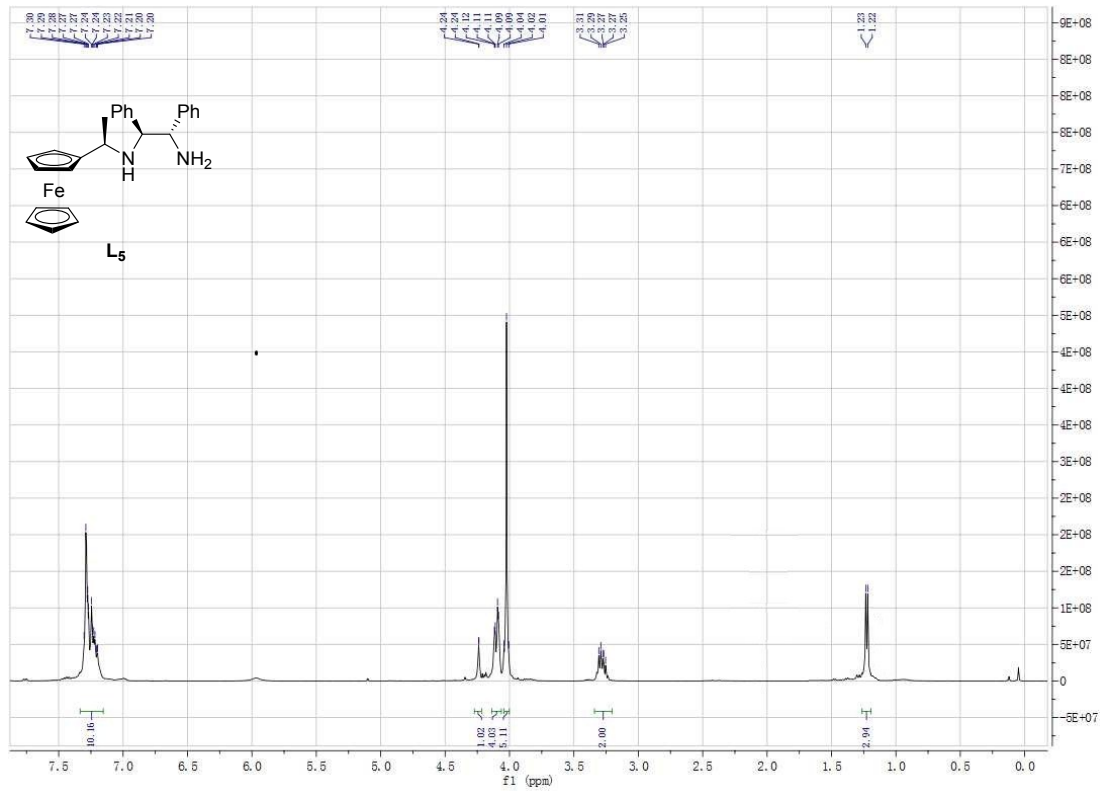


^{13}C NMR Spectra of L_4



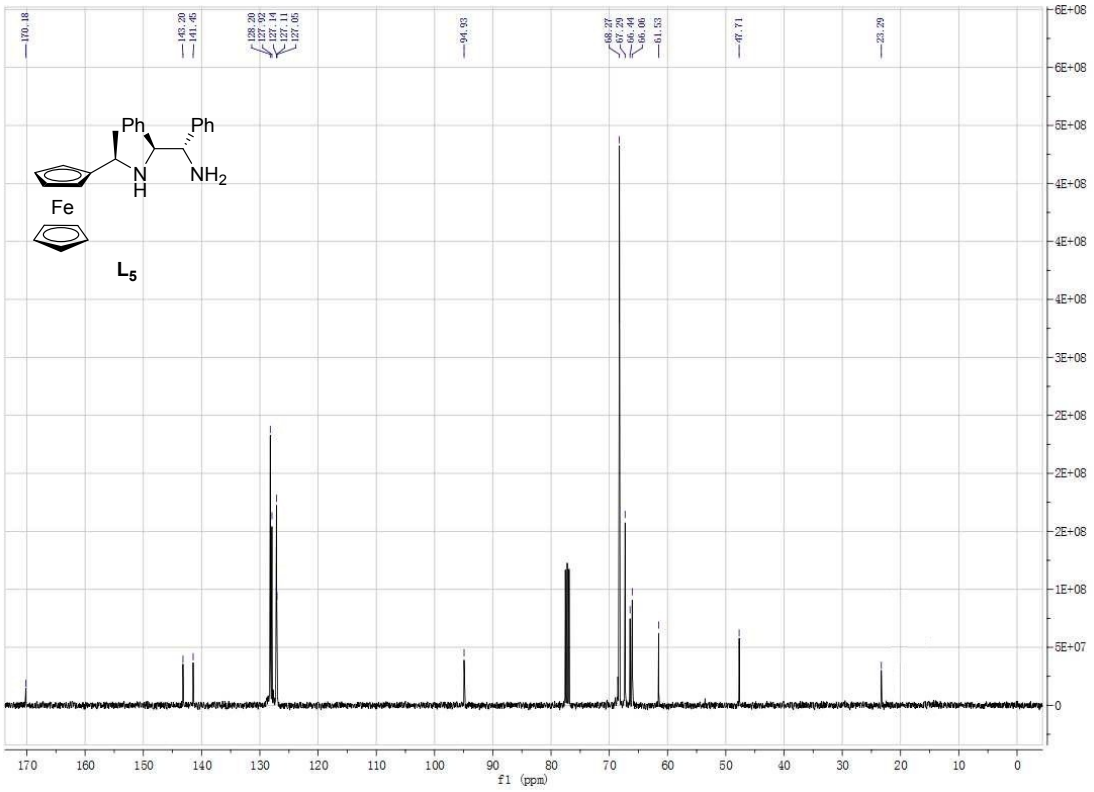
L_4

^{1}H NMR Spectra of L_5



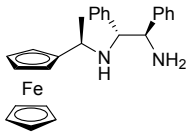
L_5

^{13}C NMR Spectra of L_5

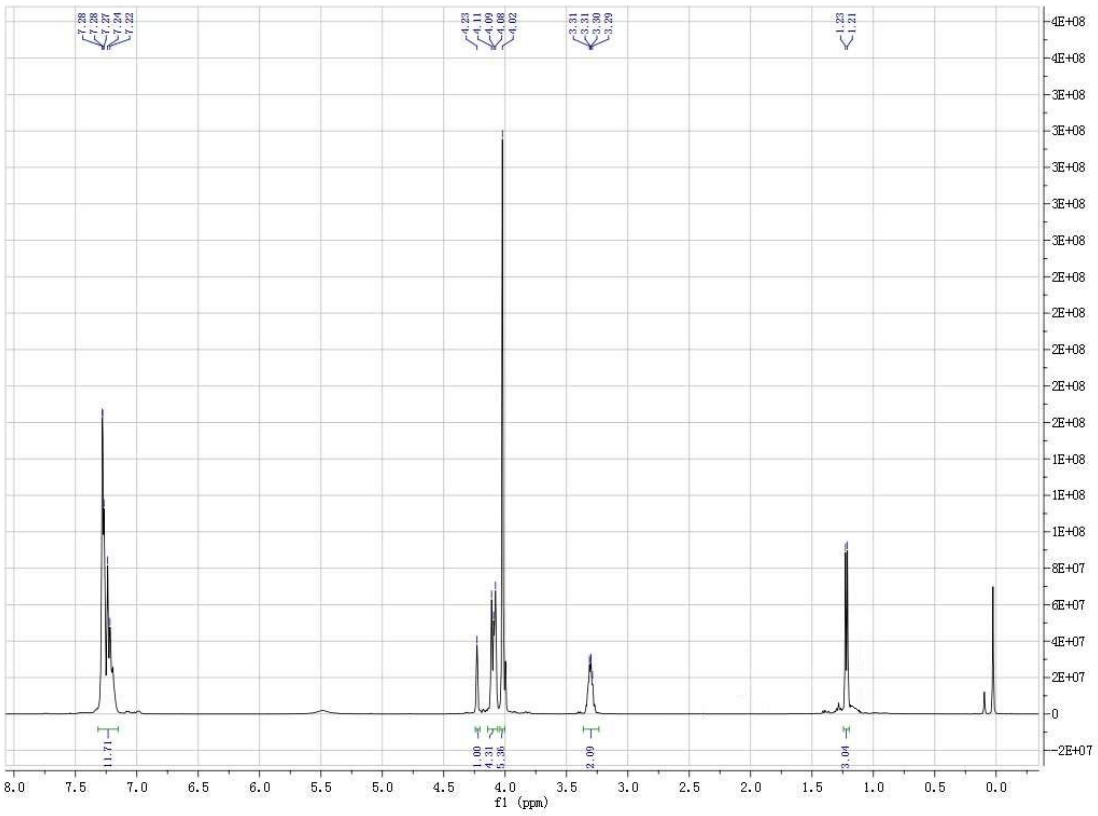


L_5

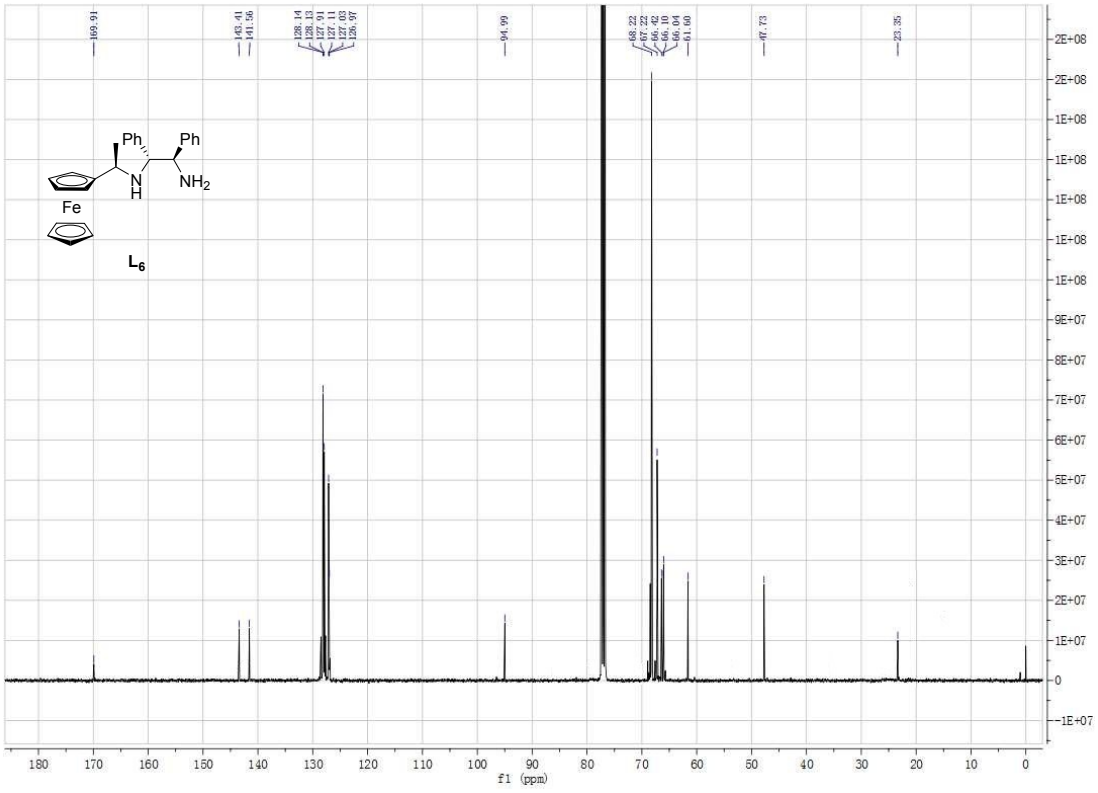
^{1}H NMR Spectra of L_6



L_6



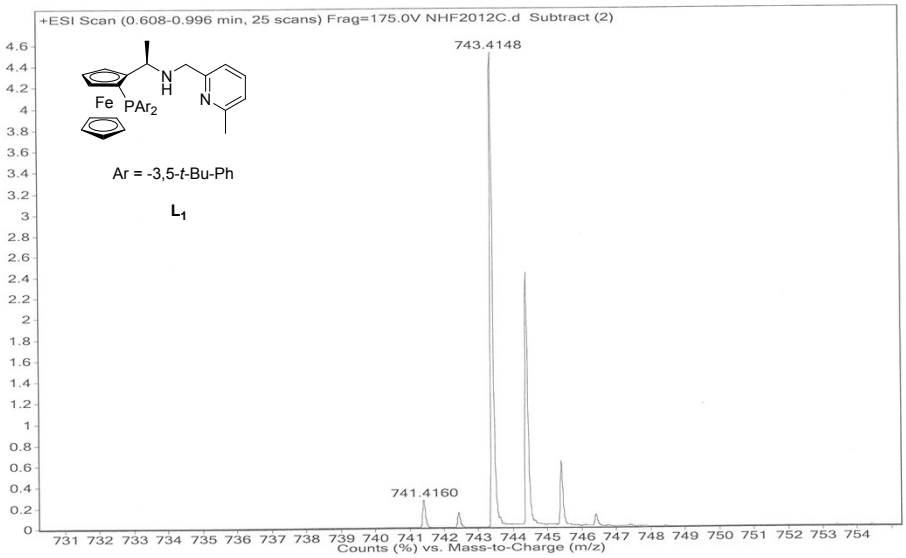
^{13}C NMR Spectra of L_6



2.HRMS of L_1-L_6

HRMS of L_1

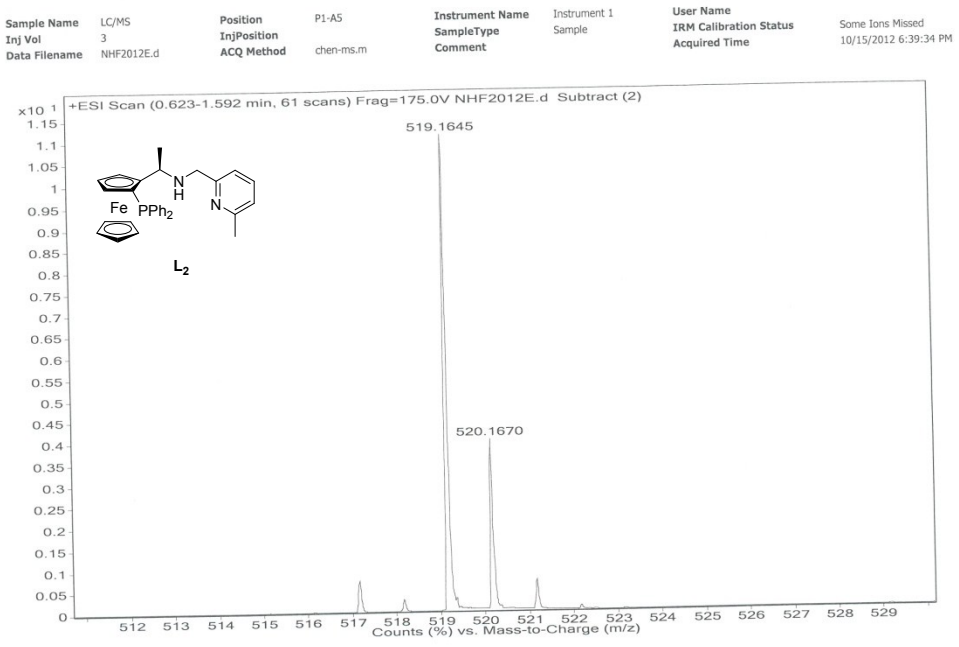
Sample Name	LC/MS	**Position**	P1-A3	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	3	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NHF2012C.d	**ACQ Method**	chen-ms.m	**Comment**		**Acquired Time**	10/15/2012 6:31:57 PM



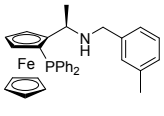
Ar = -3,5-*t*-Bu-Ph

L_1

HRMS of L_2



HRMS of L_3



L_3

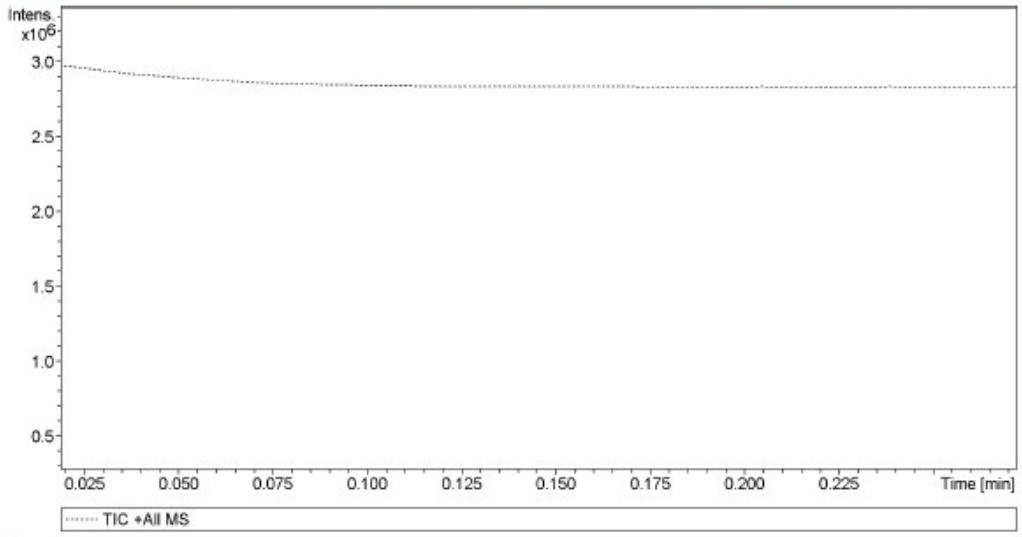
Display Report

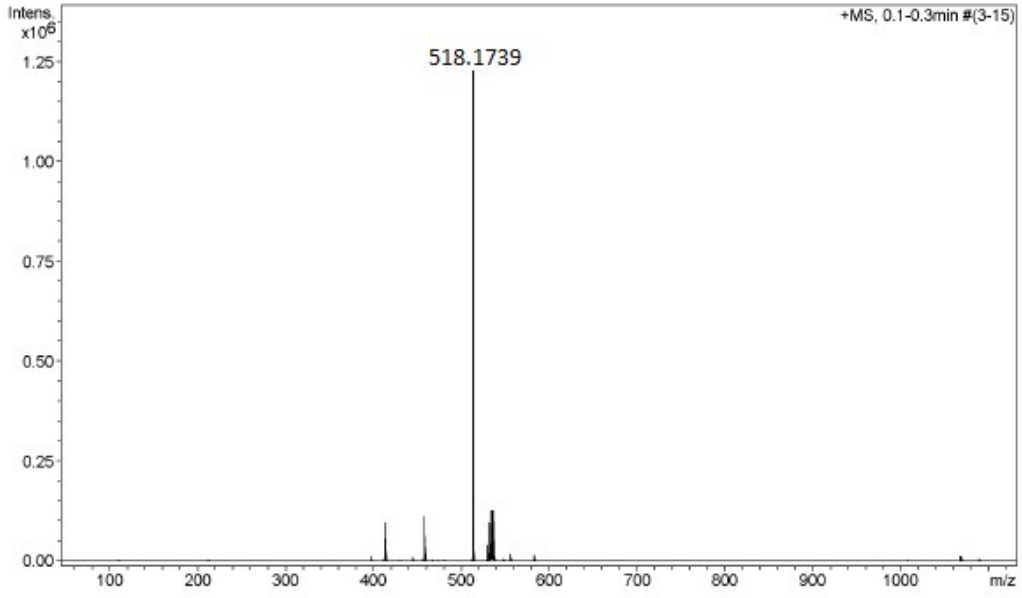
Analysis Info

Analysis Name	H:\siyida_MYZ-P20.d	Acquisition Date	2015-11-25 16:00:19
Method	tune_low 50-500.m	Operator	NWU
Sample Name		Instrument	micrOTOF-Q II 10280
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	110.0 Vpp	Set Divert Valve	Source





HRMS of L_4

Display Report

Analysis Info

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Method	tune_low 50-500.m	Operator	NWU
Sample Name		Instrument	micrOTOF-Q II 10280
Comment			

Acquisition Parameter

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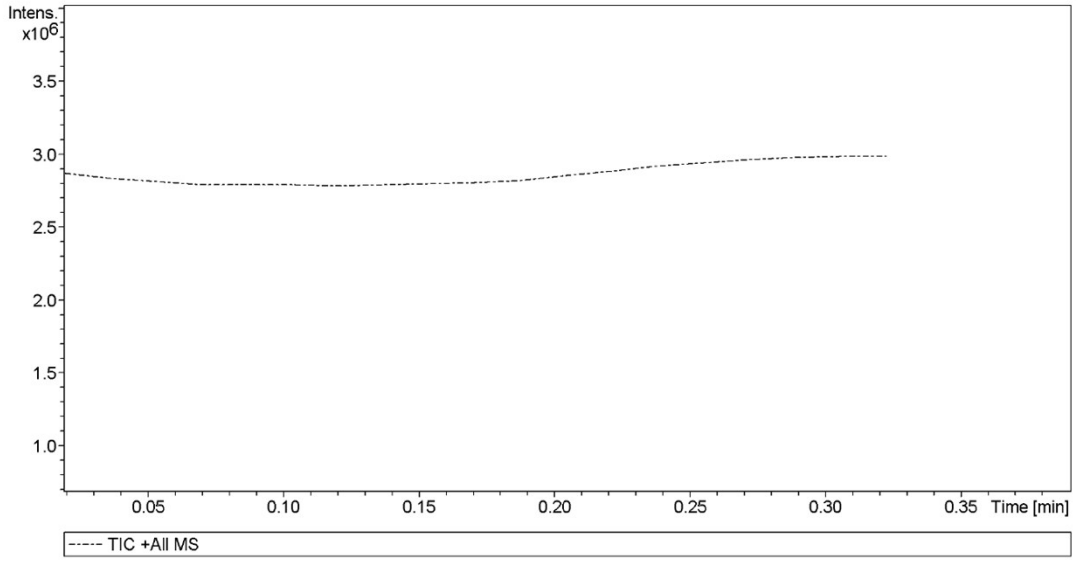
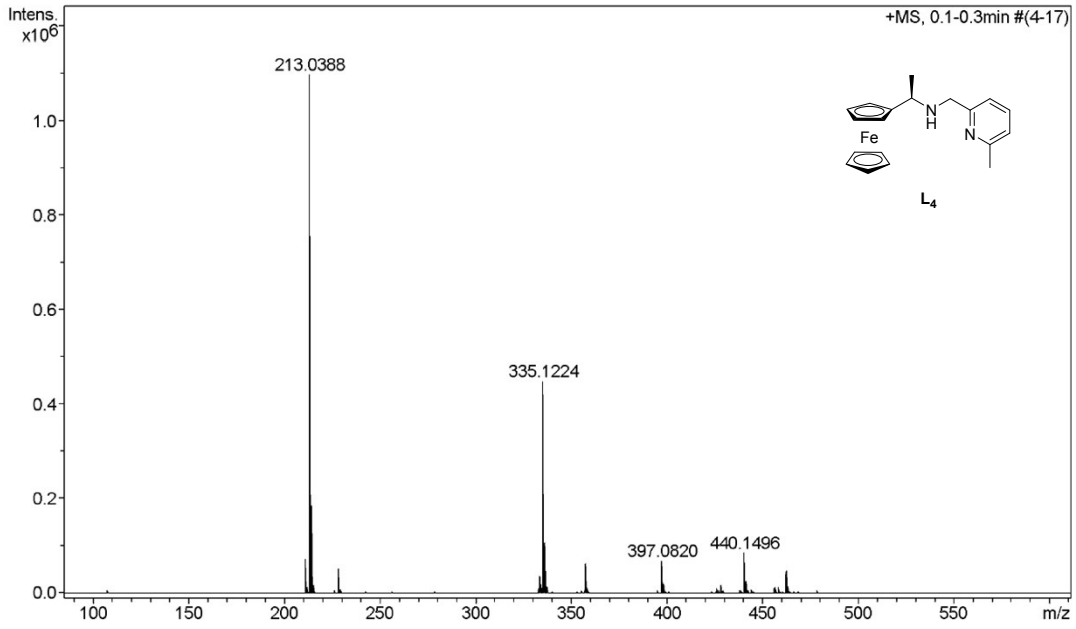
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	110.0 Vpp	Set Divert Valve	Source



----- TIC +All MS



L_4

HRMS of L_5

Display Report

Analysis Info

Analysis Name	D:\data1\siyida_he_10.d	Acquisition Date	2015-10-29 16:09:26
Method	tune_low 50-500.m	Operator	NWU
Sample Name		Instrument	micrOTOF-Q II 10280
Comment			

Acquisition Parameter

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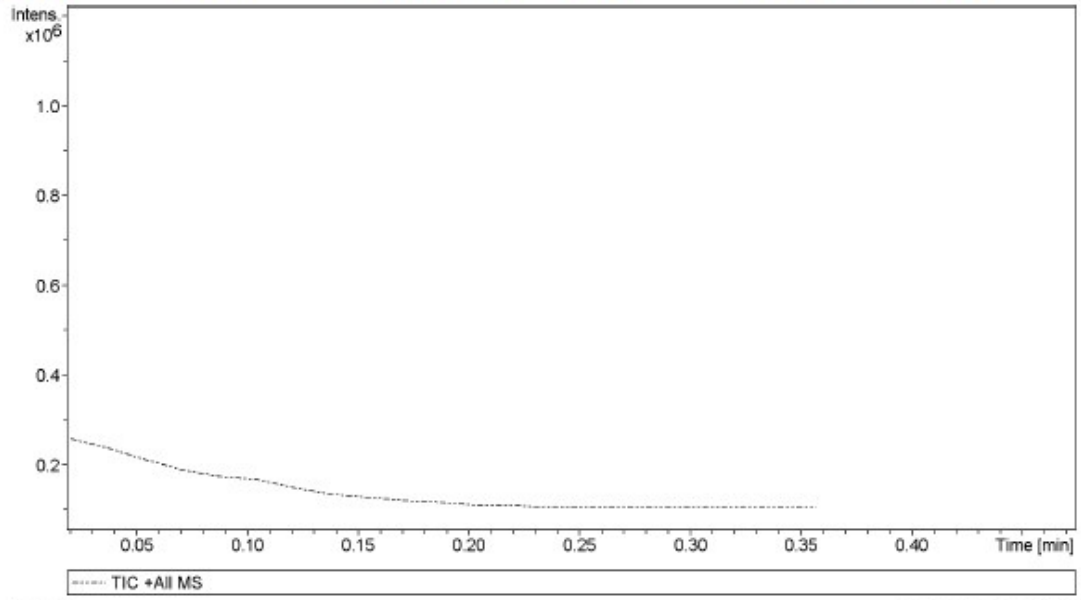
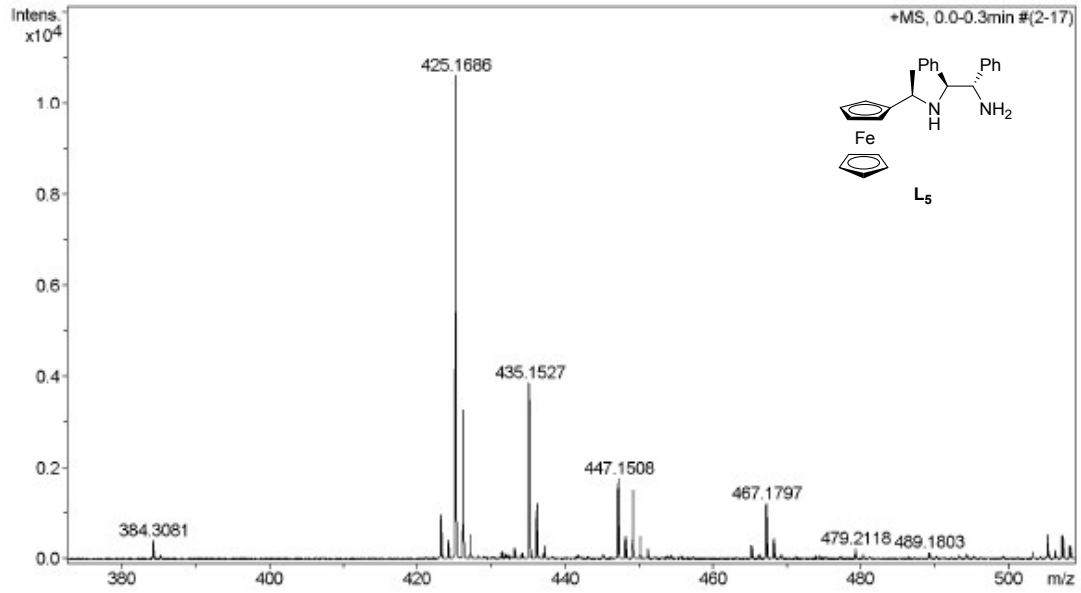
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	110.0 Vpp	Set Divert Valve	Source





L_5

Bruker Compass DataAnalysis 4.0 printed: 2015-10-30 9:27:19 Page 1 of 1

HRMS of L_6

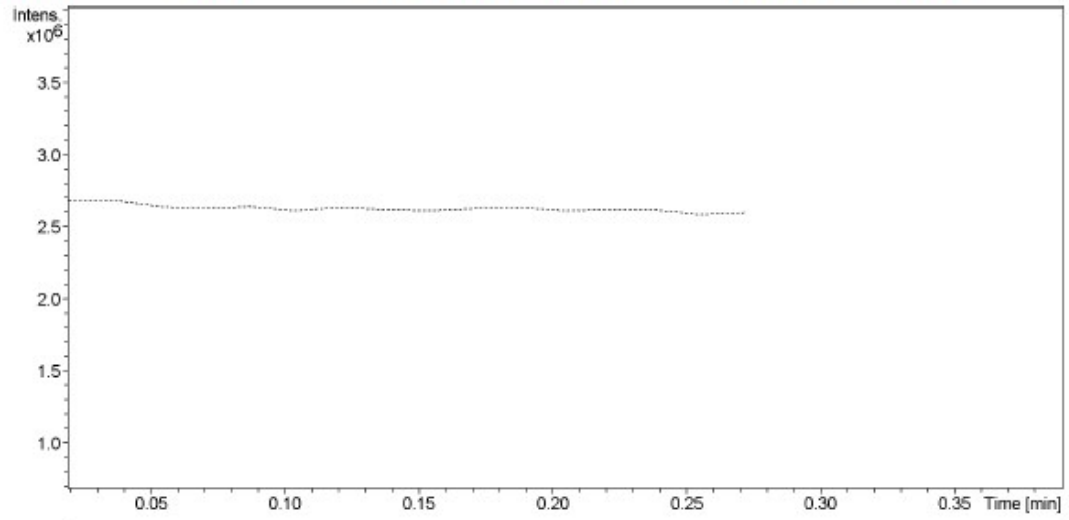
Display Report

Analysis Info

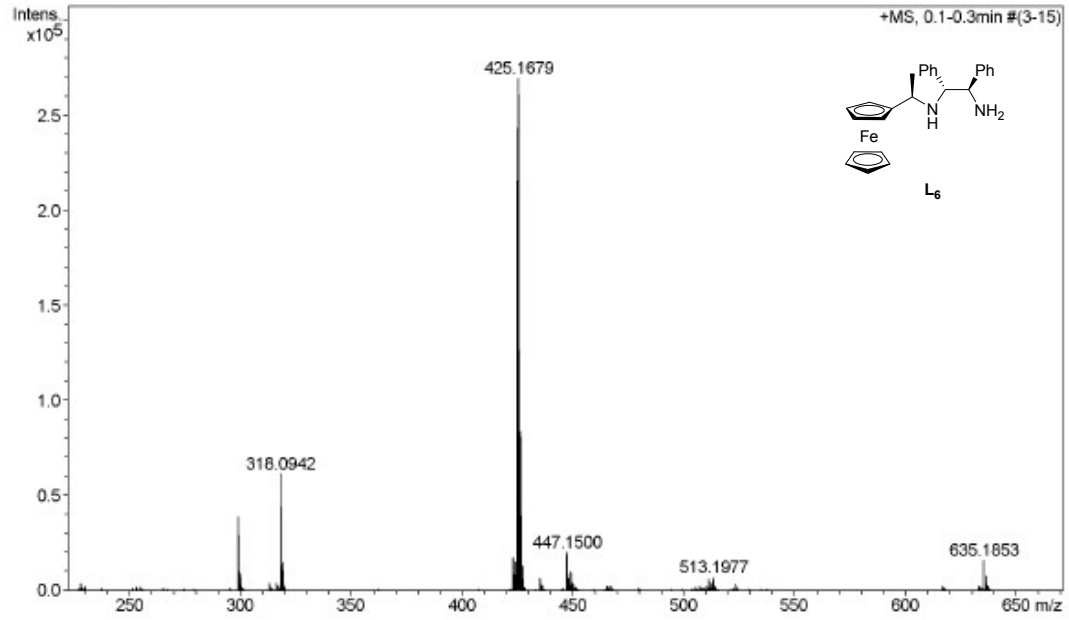
Analysis Name	H:\siyida_he_68.d	Acquisition Date	2015-9-25 15:28:31
Method	tune_low 50-500.m	Operator	NWU
Sample Name		Instrument	micrOTOF-Q II 10280
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	110.0 Vpp	Set Divert Valve	Source



TIC +All MS

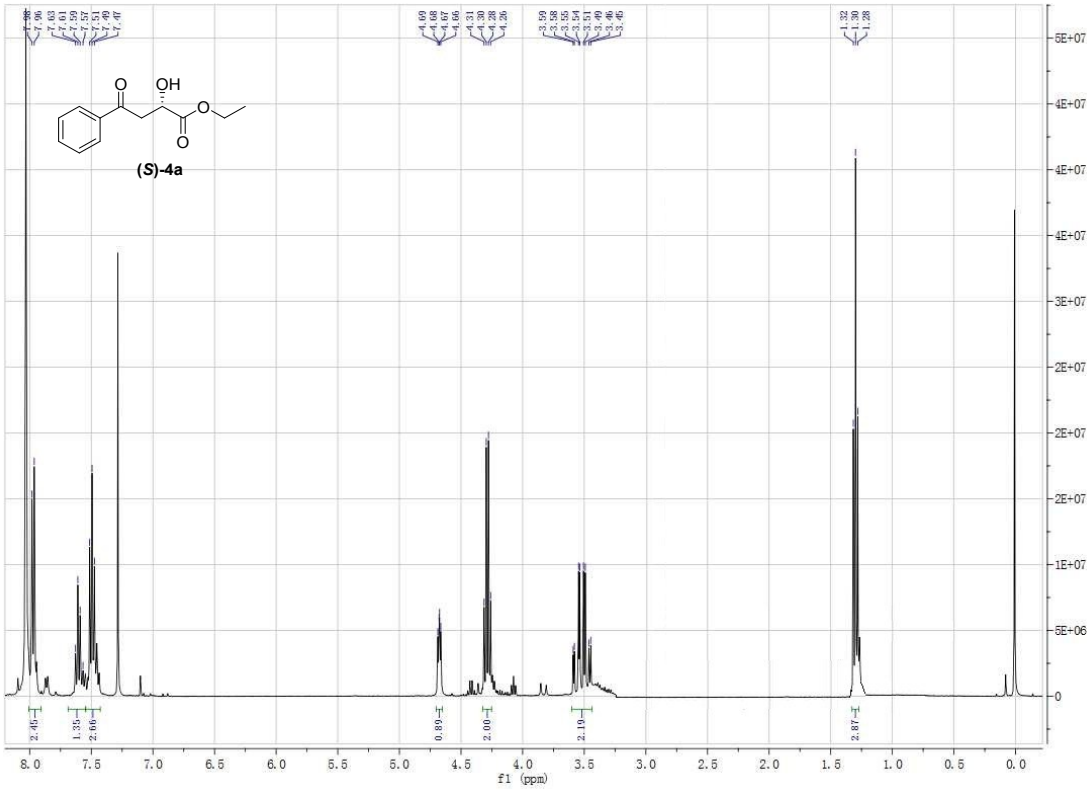


L_6

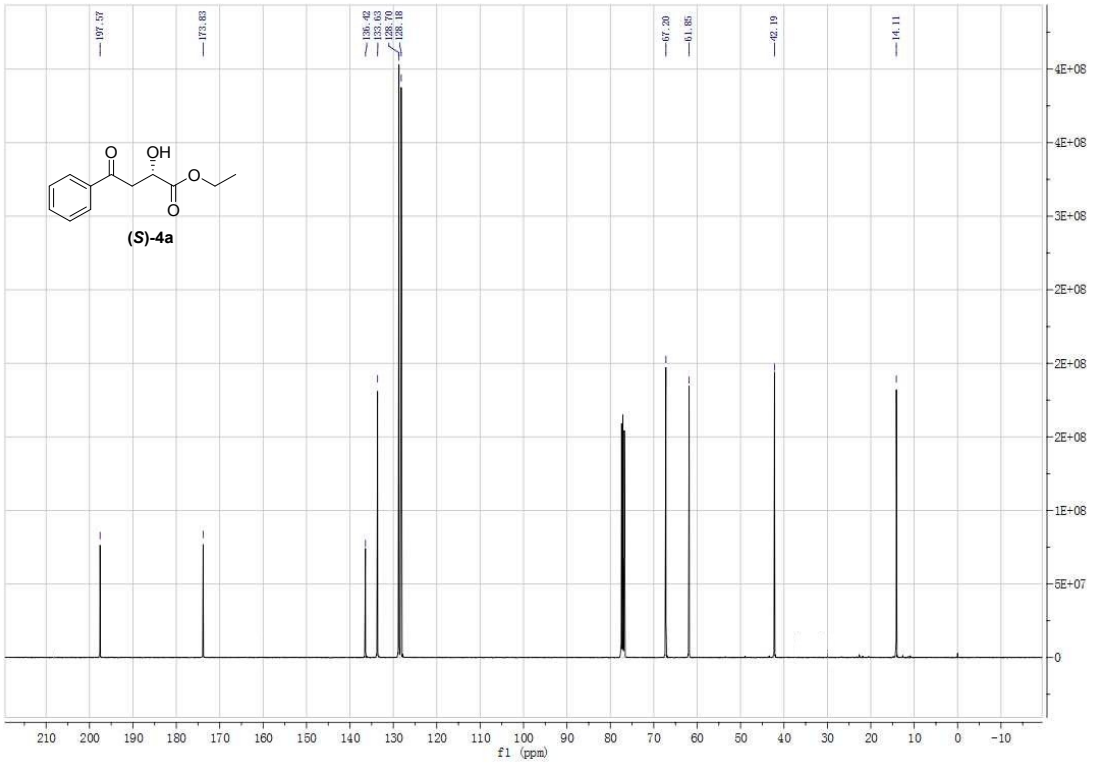
Bruker Compass DataAnalysis 4.0 printed: 2015-9-29 9:50:05 Page 1 of 1

3.^{1}H and ^{13}C NMR Spectra of 4a-g

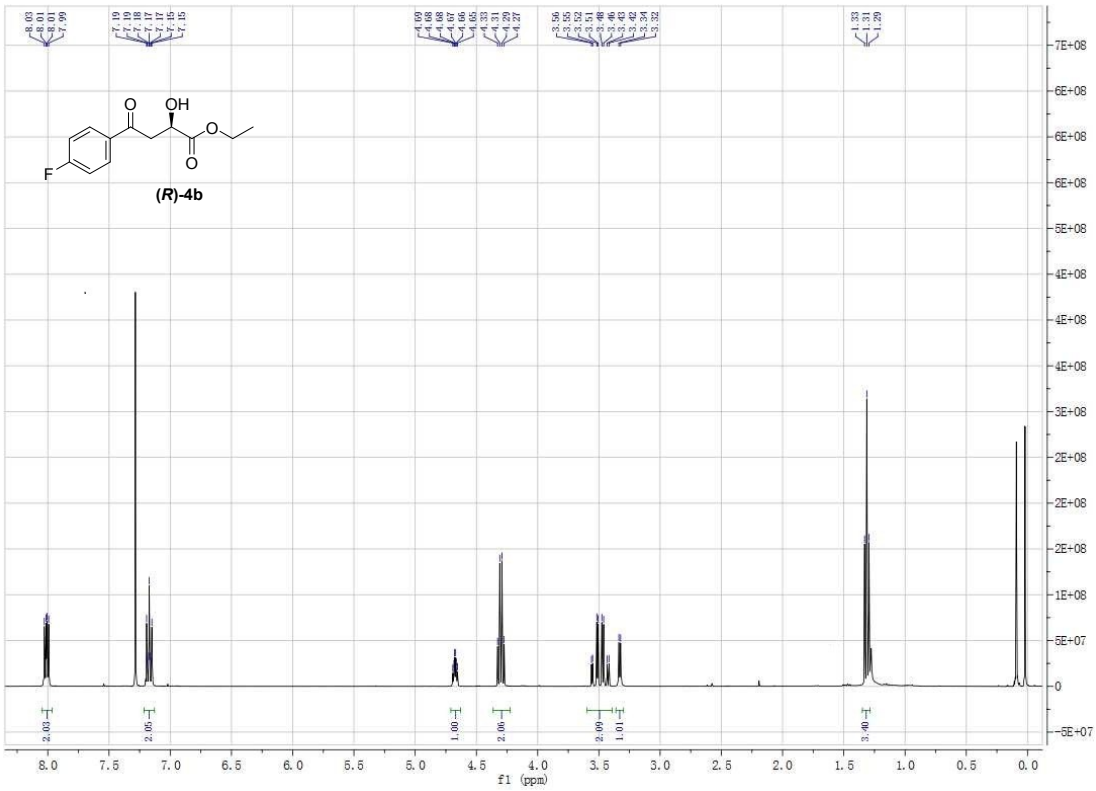
^{1}H NMR Spectra of (*S*)-4a



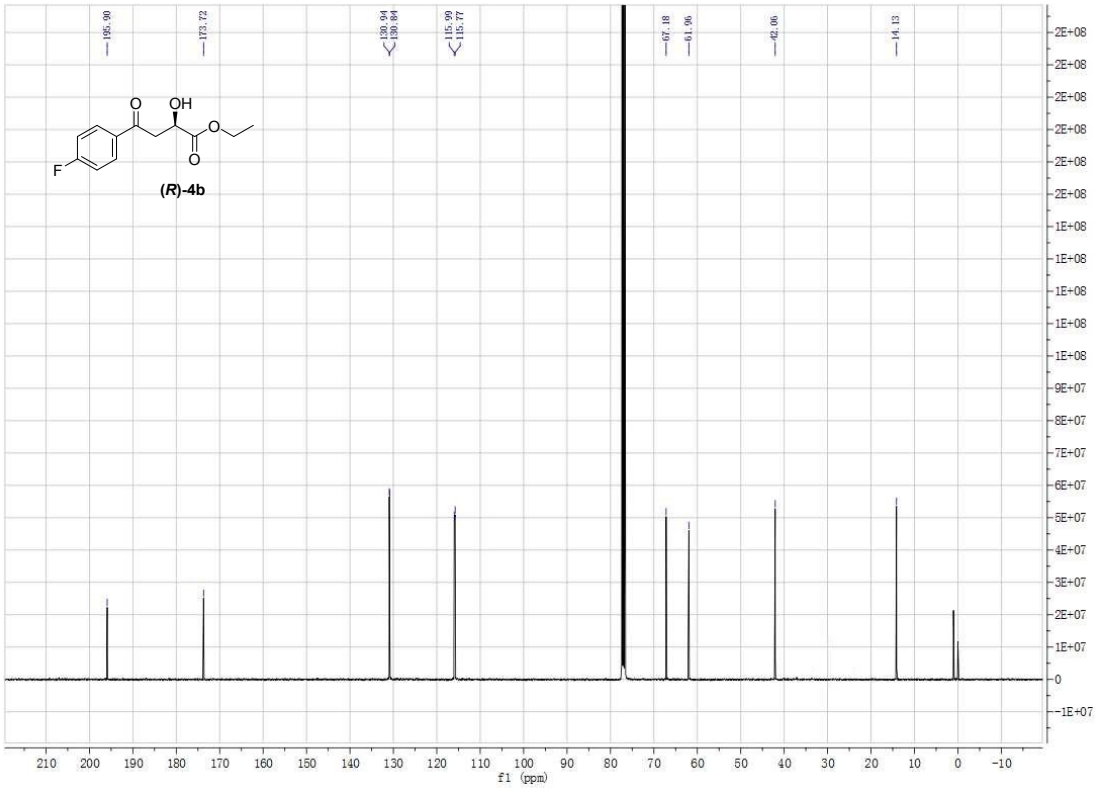
^{13}C NMR Spectra of (*S*)-4a



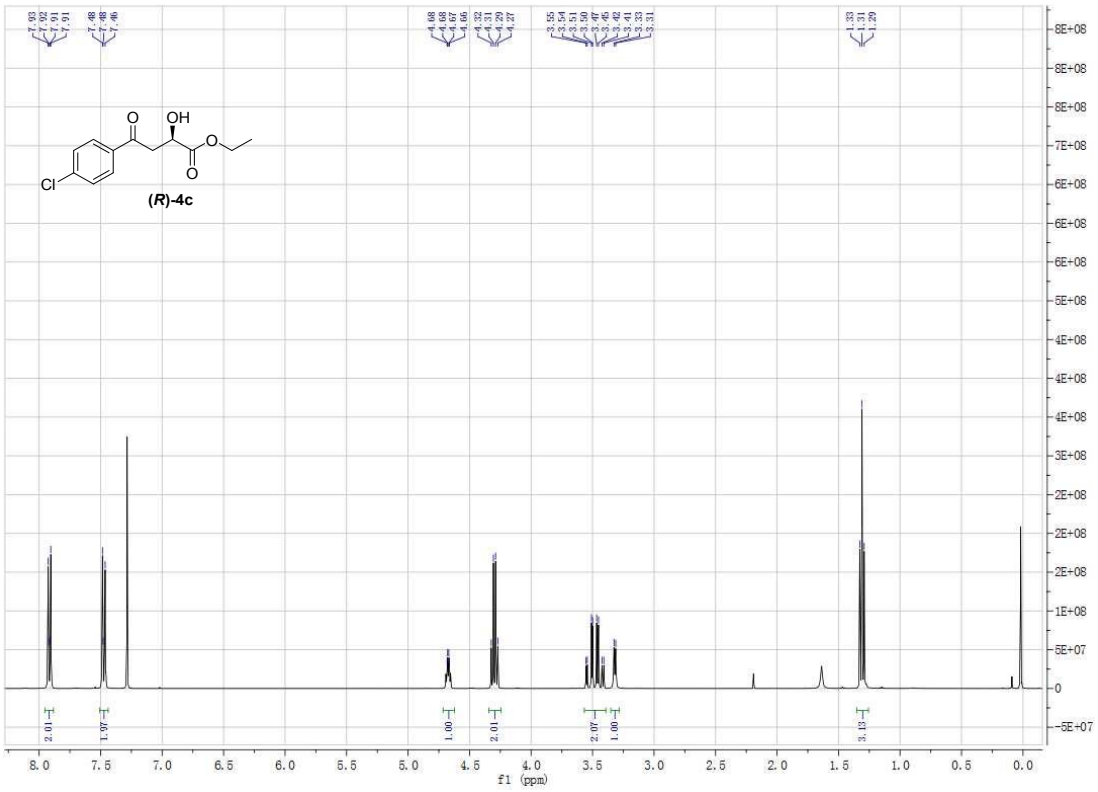
^{1}H NMR Spectra of (*R*)-4b



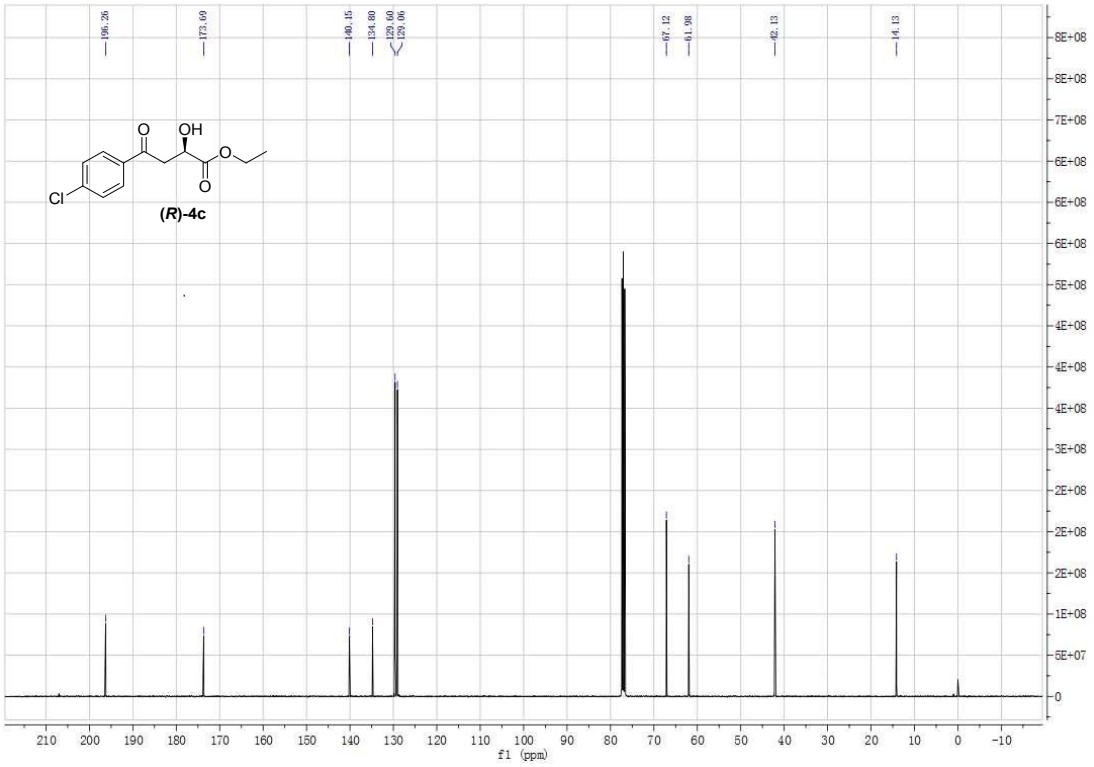
^{13}C NMR Spectra of (*R*)-4b



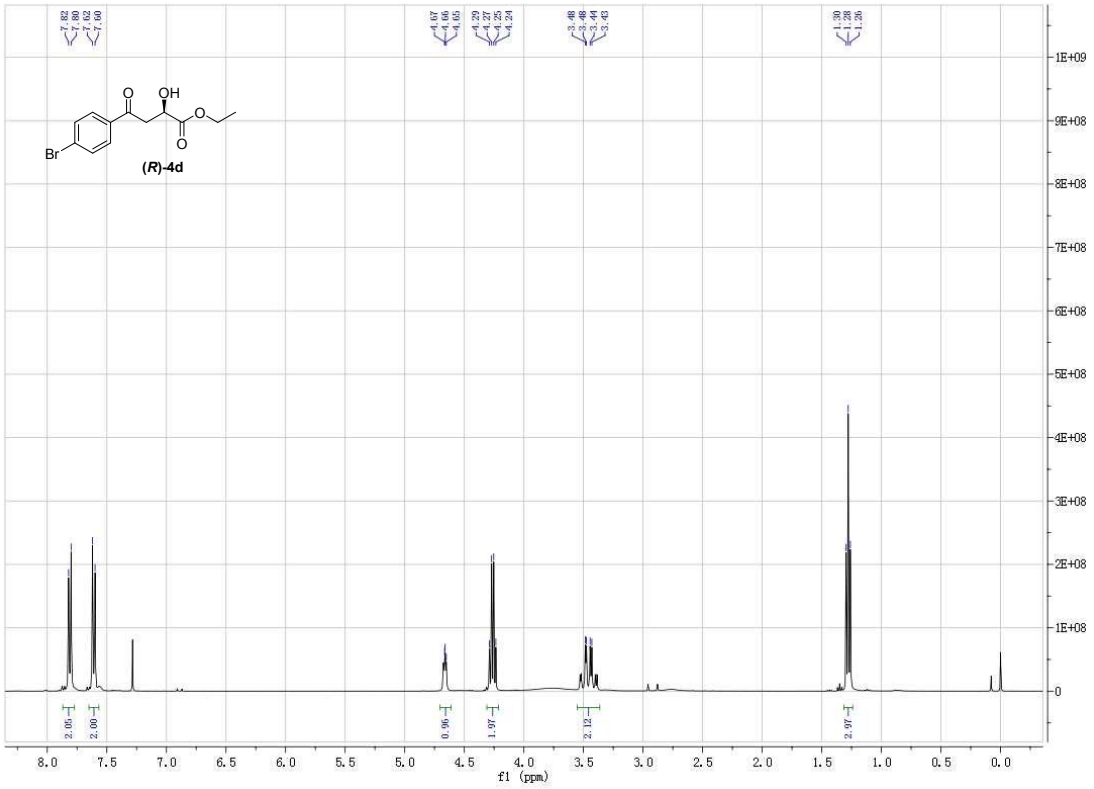
^{1}H NMR Spectra of (*R*)-4c



^{13}C NMR Spectra of (*R*)-4c



^{1}H NMR Spectra of (*R*)-4d



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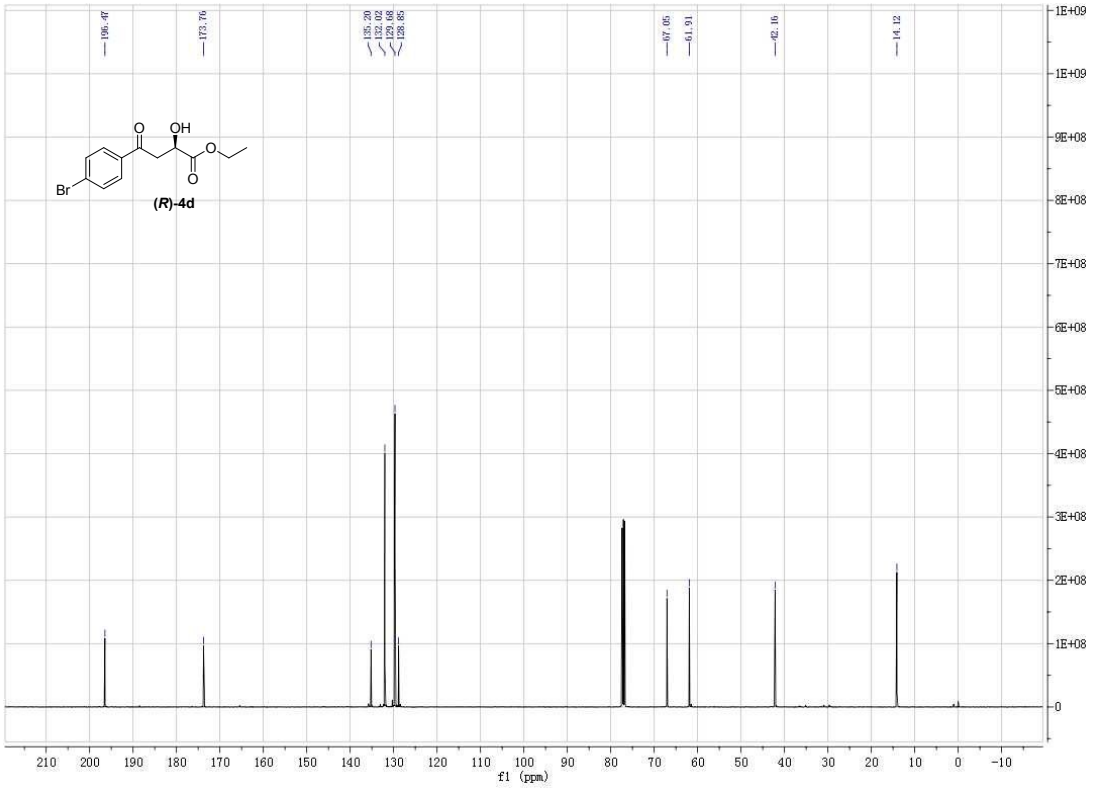
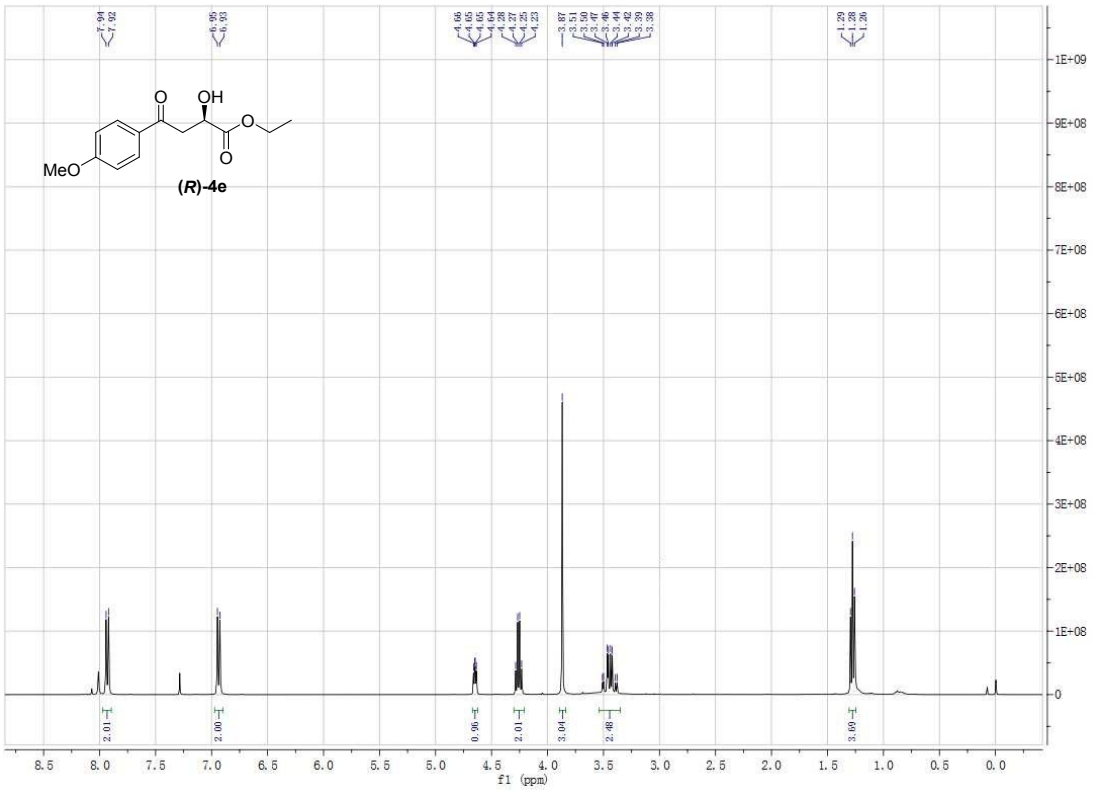


^{1}H NMR Spectra of (*R*)-4e



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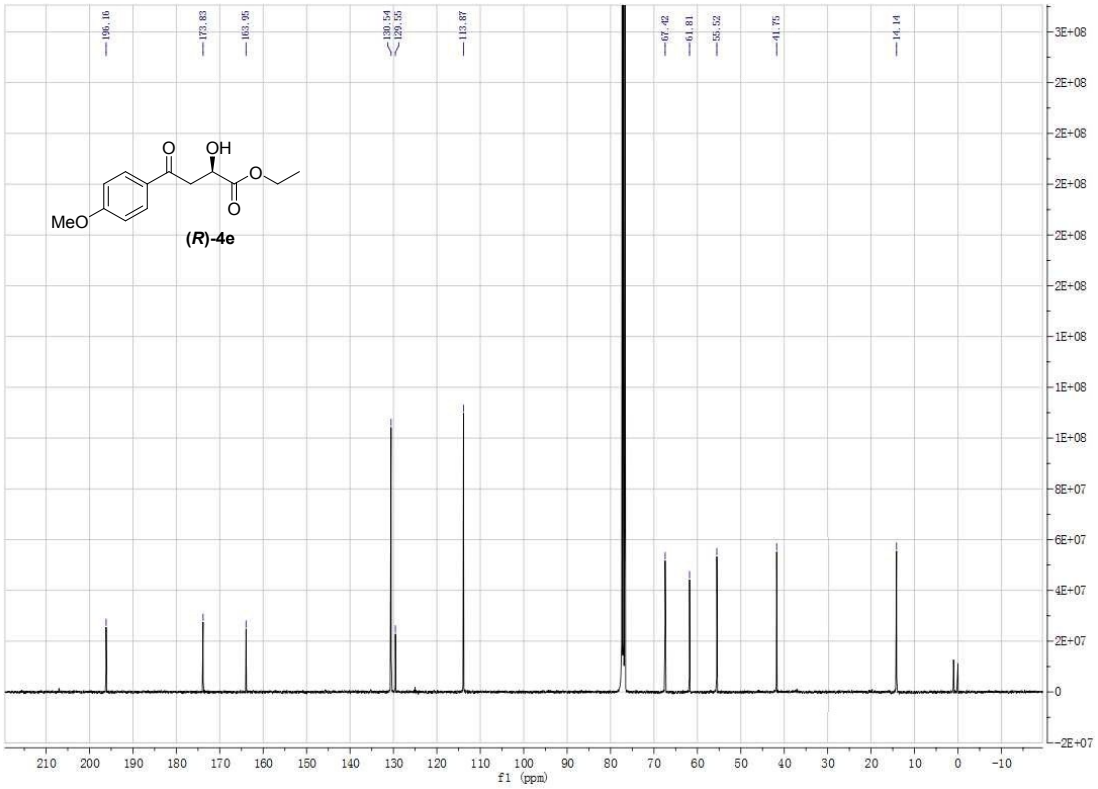
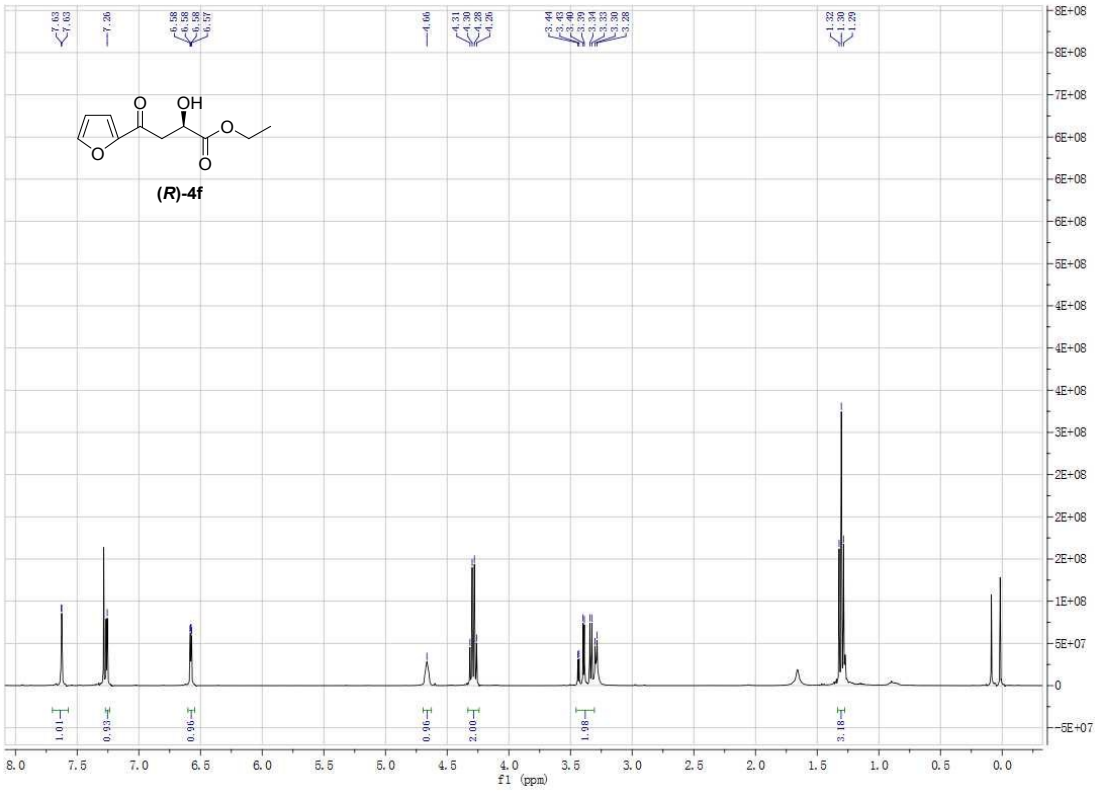
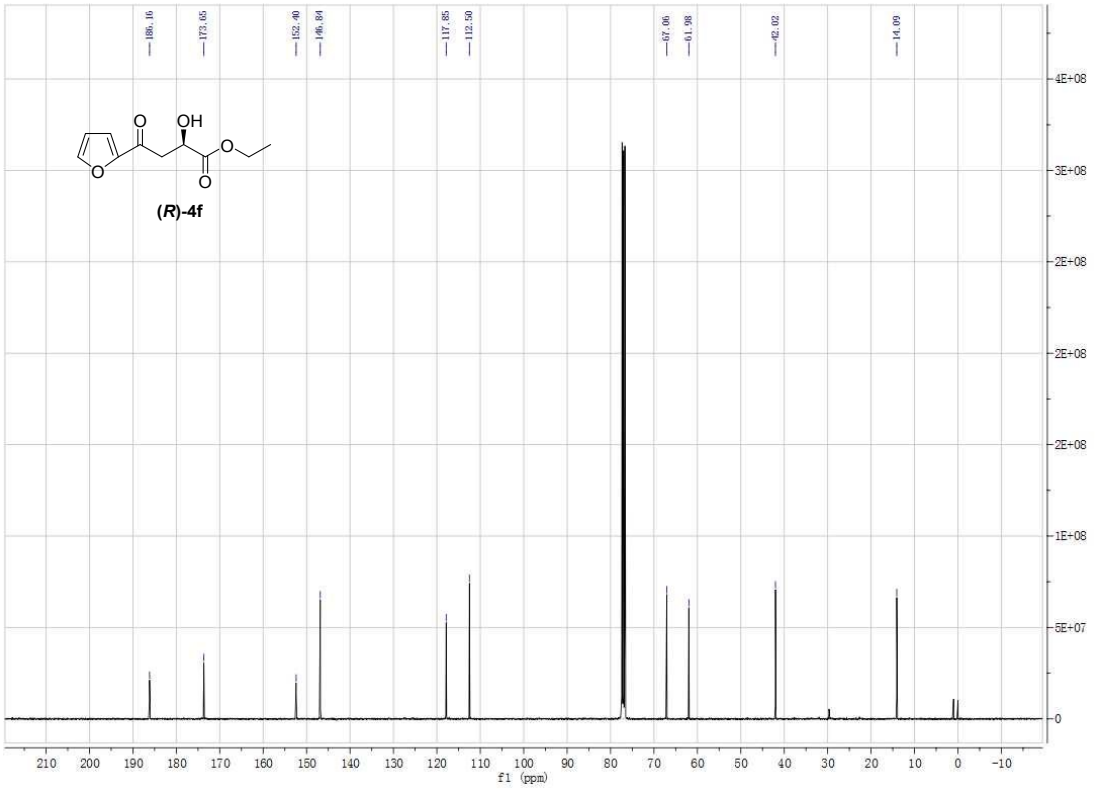


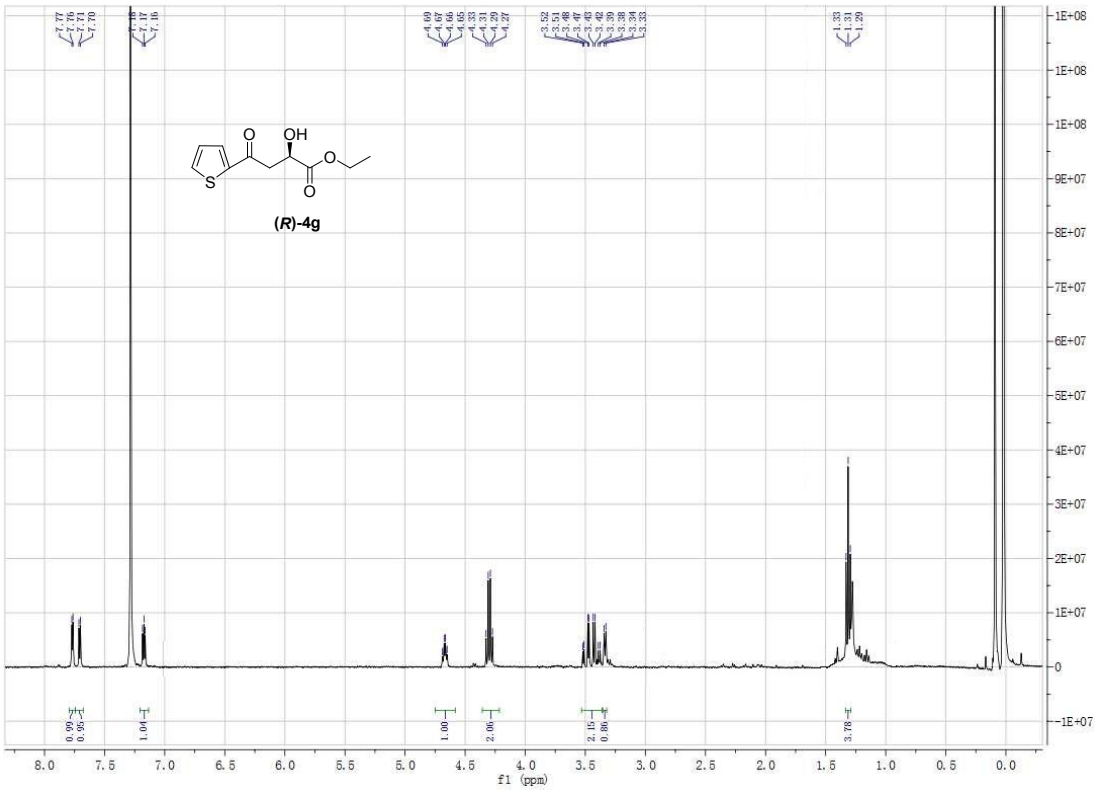
^{1}H NMR Spectra of (*R*)-4f



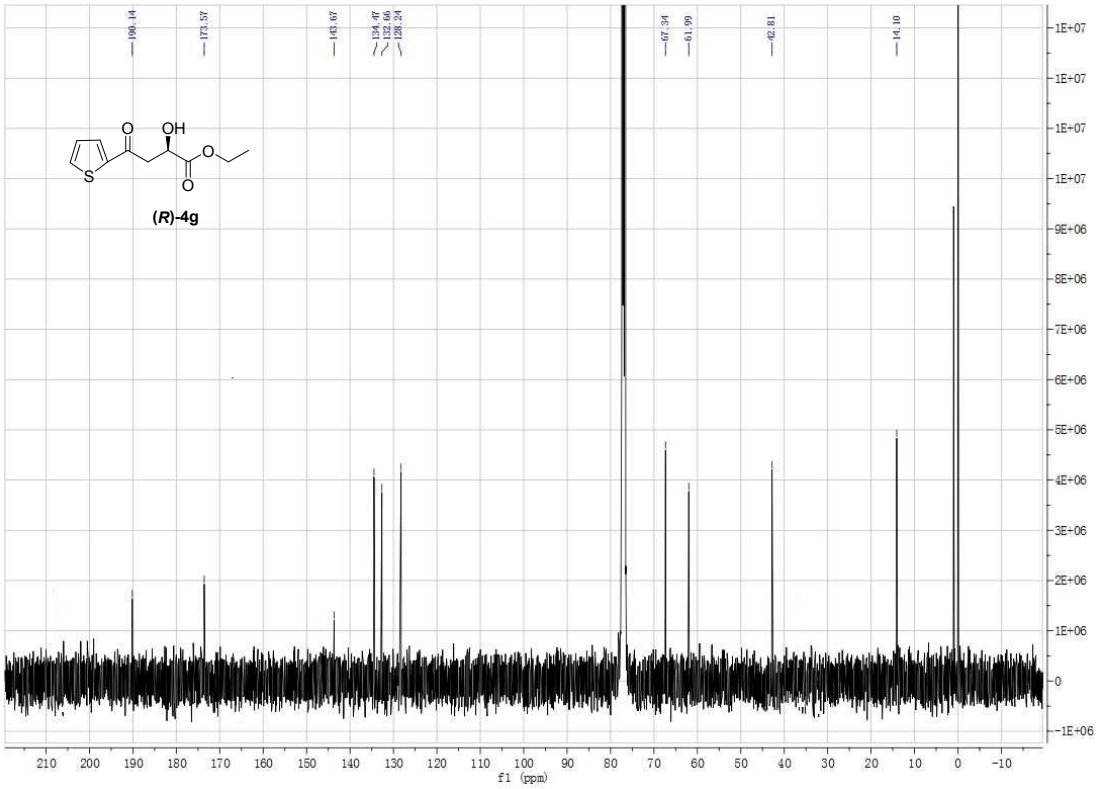
^{13}C NMR Spectra of (*R*)-4f



^{1}H NMR Spectra of (*R*)-4g



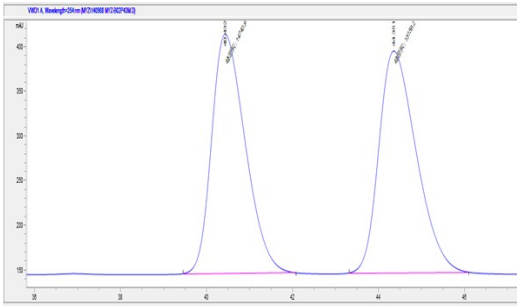
^{13}C NMR Spectra of (*R*)-4g

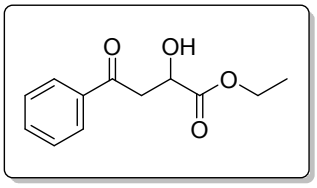


4.HPLC charts of [catalyst was RuCl [TsDPEN](cymene)]

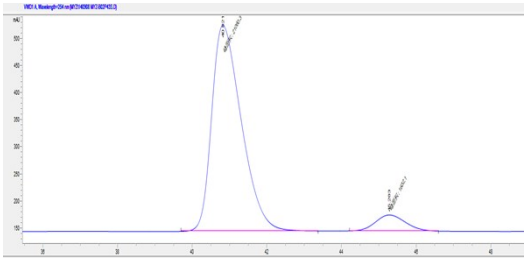
4-phenyl-2-hydroxy-4-oxo-butyric acid ethyl ester (4a)

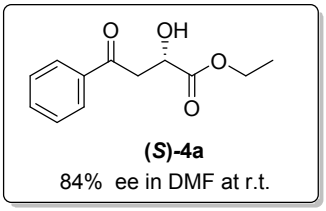
85% yield, 84% ee in DMF, 30% yield, 73% ee in MeOH, 73% yield, 50% ee in THF, 80% yield, 22% ee in EtOAc, 77% yield, 60% ee in dixane., 82% yield, 81% ee in *t*-BuOMe , 75% yield, 79% ee without solvent at r.t., 80% yield, 84% ee in DMF at 0°C, 68% yield, 94% ee in DMF at -20°C, 68% yield, 94% ee in DMF at -20°C (*R*-configuration) for 4days, determined by HPLC analysis (Chiralcel OD-H column, Hexane/i-PrOH=95/5, Flow rate: 0.5 mL/min, UV detection at 254 nm).





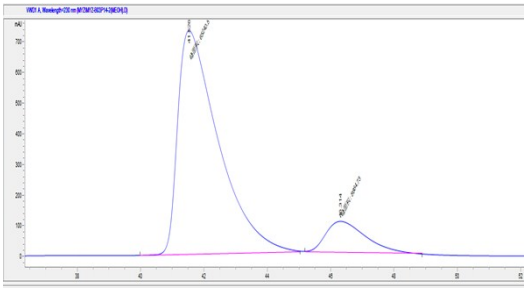
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	40.432	14740.9	269.7	0.911	0.668	49.301
2	44.351	15159.2	249.9	1.011	0.656	50.699

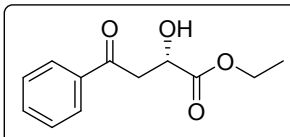




(*S*)-4a
84% ee in DMF at r.t.

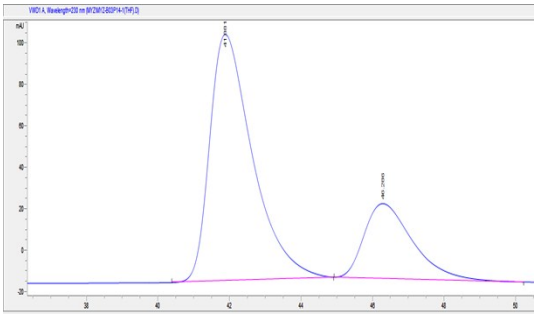
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	40.821	21060.3	381.8	0.9193	0.596	92.808
2	45.283	1632.1	29.6	0.9186	0.859	7.192

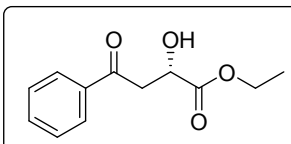




(*S*)-4a
73% ee in MeOH

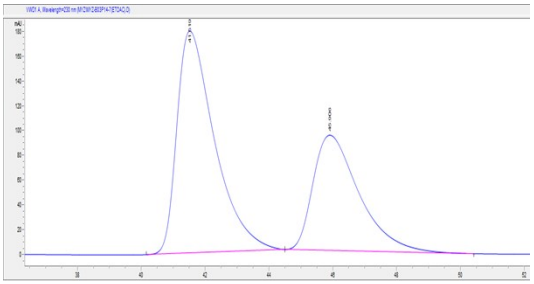
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	41.526	65240.5	728.7	1.4923	0.377	86.276
2	46.314	8664.7	100.3	1.4404	0.583	13.724

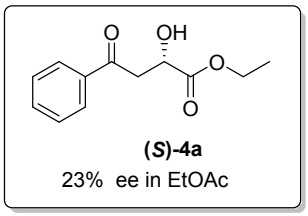




(*S*)-4a
50% ee in THF

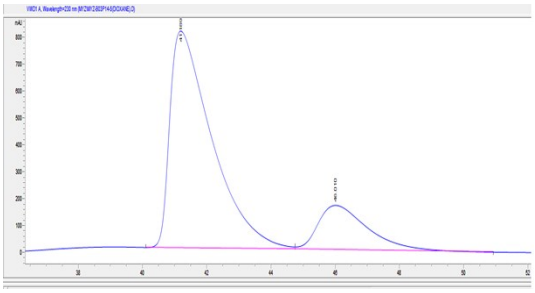
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	41.881	9898.4	119.1	1.2439	0.54	74.470
2	46.286	3393.5	36.2	1.4092	0.589	25.530

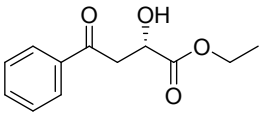




(*S*)-4a
23% ee in EtOAc

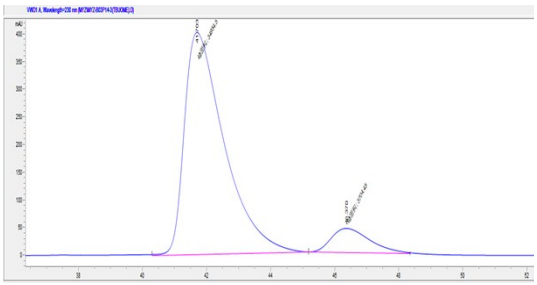
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	41.519	14674.8	179.2	1.2296	0.492	61.156
2	45.906	9320.8	93	1.5052	0.54	38.844

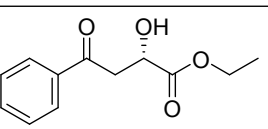




(*S*)-4a
60% ee in dixane

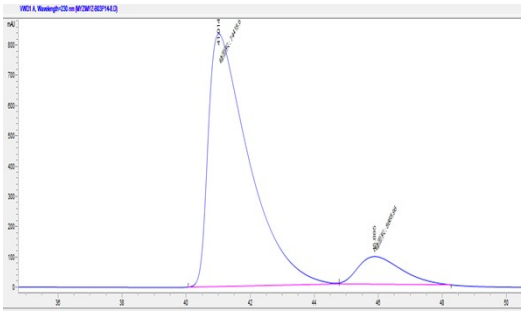
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	41.189	73990.8	810.6	1.3239	0.324	80.313
2	46.01	18136.9	166.9	1.6179	0.512	19.687

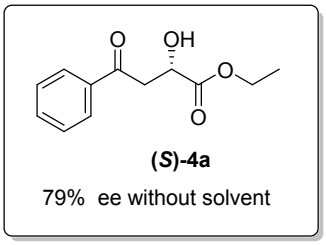




(*S*)-4a
81% ee in t-BuOMe

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	41.703	34954.5	403.9	1.4423	0.457	90.418
2	46.37	3704.5	44	1.4038	0.625	9.582

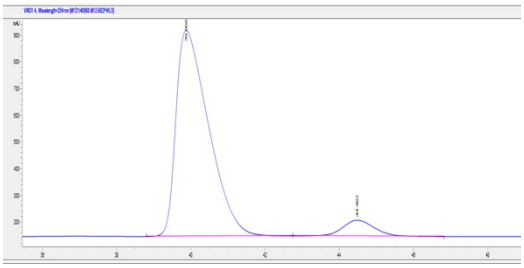


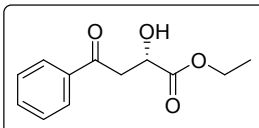


(*S*)-4a

79% ee without solvent

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	41.014	74418.6	841.1	1.4747	0.336	89.566
2	45.885	8669	91.9	1.5723	0.613	10.434

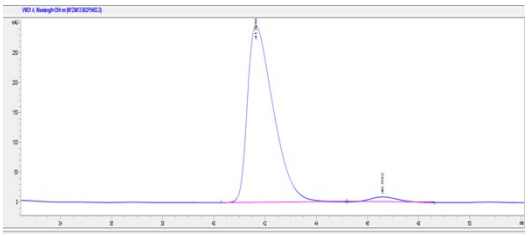


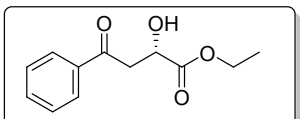


(*S*)-4a

84% ee in DMF at 0℃

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	39.866	2708.3	772.1	0.9325	0.429	92.953
2	44.482	2569.7	59.8	0.9117	0.851	7.047

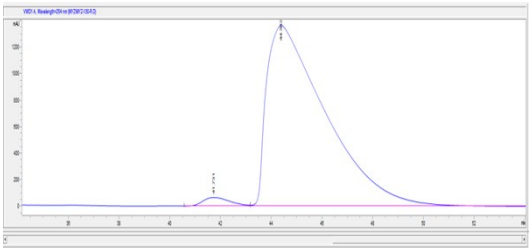


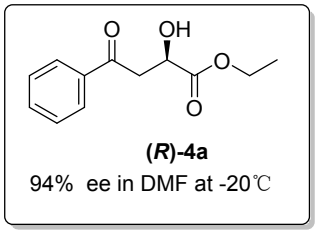


(*S*)-4a

94% ee in DMF at -20℃

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	41.638	20774.9	295.9	1.0682	0.467	96.944
2	46.592	655	8.7	1.1186	0.825	3.056





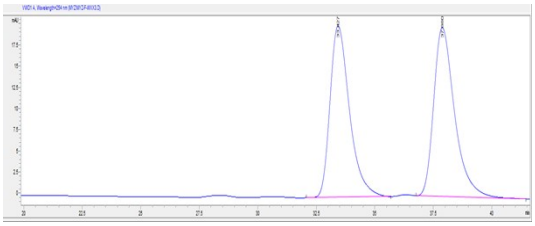
(*R*)-4a

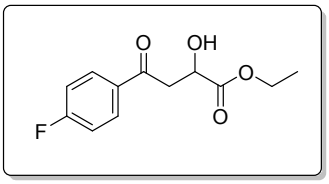
94% ee in DMF at -20℃

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	42.301	8928.5	112.6	1.2002	0.682	2.997
2	44.533	319693.1	1908.1	2.3337	0.115	97.003

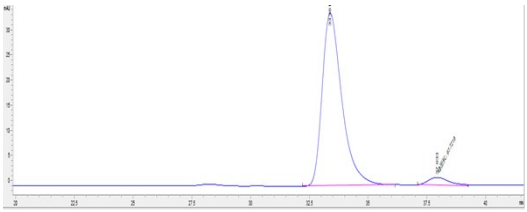
4-(4-F-phenyl)-2-hydroxy-4-oxo-butyric acid ethyl ester (4b) ——RuCl(*p*-cymene)[(*R*,*R*)-Ts-DPEN]

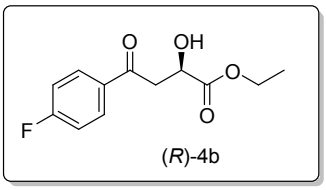
61% yield, 91% ee determined by HPLC analysis (Chiralcel OJ-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	33.427	1175	20.3	0.882	0.665	48.600
2	37.88	1242.7	20	0.9325	0.648	51.400





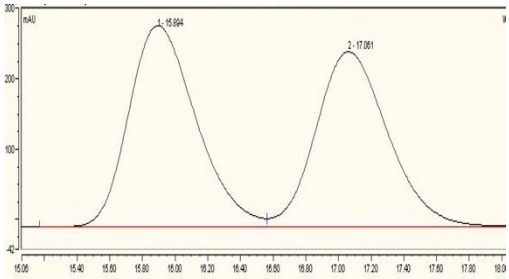
(*R*)-4b

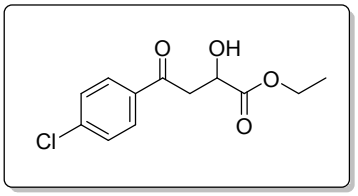
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	33.381	2006.7	34.3	0.8881	0.643	95.356
2	37.933	97.7	1.6	1.0393	0.719	4.644

4-(4-Cl-phenyl)-2-hydroxy-4-oxo-butyric acid ethyl ester (4c) ——RuCl(*p*-cymene)[(*R*,*R*)-Ts-DPEN]

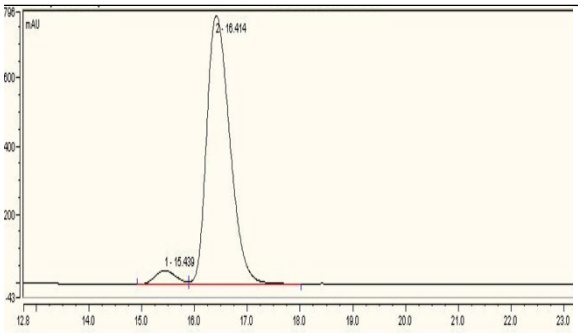
58% yield, 91% ee determined by HPLC analysis (Chiralcel OD-H column, Hexane/i-PrOH=93/7, Flow rate: 1

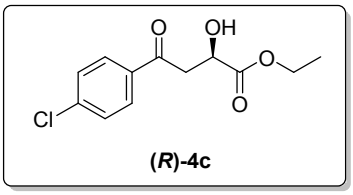
mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak area%
1	15.894	137.1400	286.632	51.78
2	17.061	127.7091	249.467	48.22



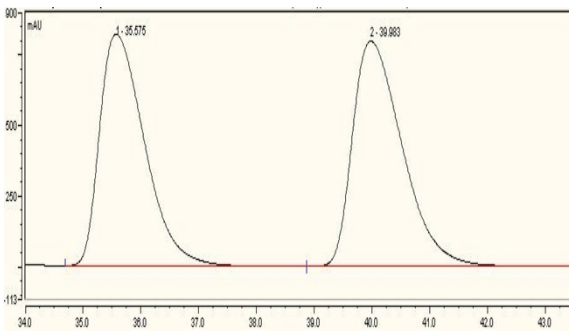


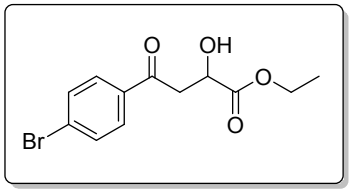
(*R*)-4c

#	Time	Peak area	Peak height	Peak area%
1	15.439	19.2280	41.408	4.59
2	16.414	399.4082	787.955	95.41

4-(4-Br-phenyl)-2-hydroxy-4-oxo-butyric acid ethyl ester (4d) ——RuCl(*p*-cymene)[(*R*,*R*)-Ts-DPEN]

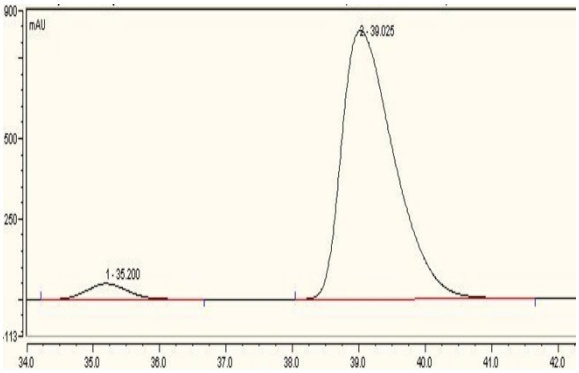
60% yield, 91% ee determined by HPLC analysis (Chiralcel OD-H column, Hexane/i-PrOH=90/10, Flow rate: 0.5 mL/min, UV detection at 254 nm).

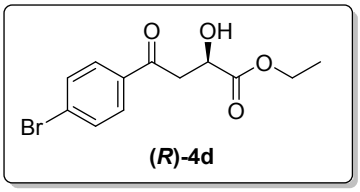




#	Time	Peak area	Peak height	Peak

				area%
1	35.575	735.0525	818.551	48.15
2	39.983	791.6492	795.476	51.85

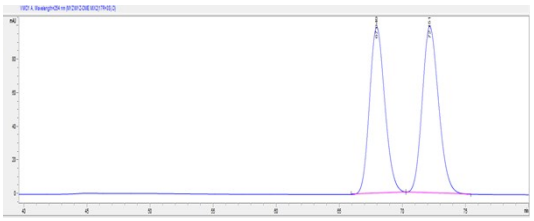


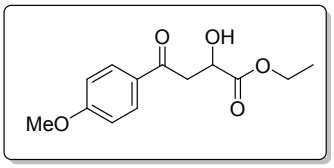


#	Time	Peak area	Peak height	Peak area%
1	35.200	35.4044	49.193	4.44
2	39.025	762.2163	835.512	95.56

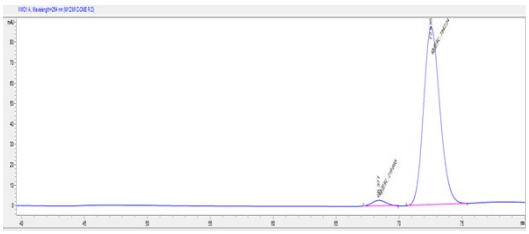
4-(4-OMe-phenyl)-2-hydroxy-4-oxo-butyric acid ethyl ester (4e) ——RuCl(*p*-cymene)[(*R*,*R*)-Ts-DPEN]

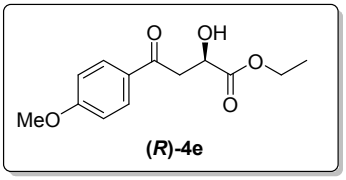
58% yield, 94.5% ee determined by HPLC analysis (Chiralcel AD-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	67.949	8372.3	99.3	1.2843	0.853	48.268
2	72.151	8973.2	99.5	1.4086	0.812	51.732

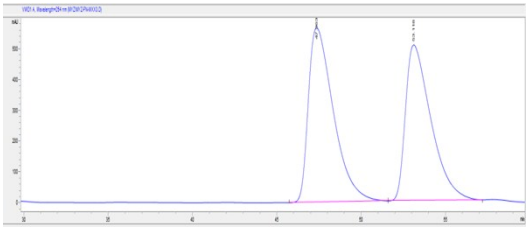


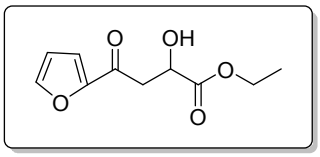


#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	68.377	219.7	2.8	1.3139	0.909	2.725
2	72.535	7842	86.7	1.5071	0.842	97.275

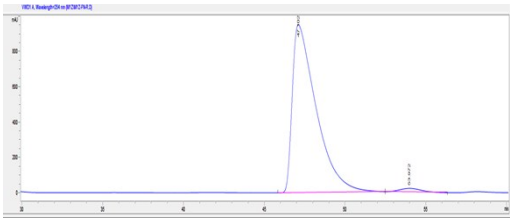
4-furyl-2-hydroxy-4-oxo-butyric acid ethyl ester (4f) ——RuCl(*p*-cymene)[(*R*,*R*)-Ts-DPEN]

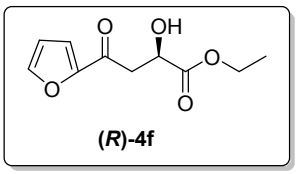
55% yield, 96% ee determined by HPLC analysis (Chiralcel OJ-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	47.363	58545.2	567.3	1.5931	0.467	52.401
2	53.118	53179.9	506.5	1.578	0.451	47.599



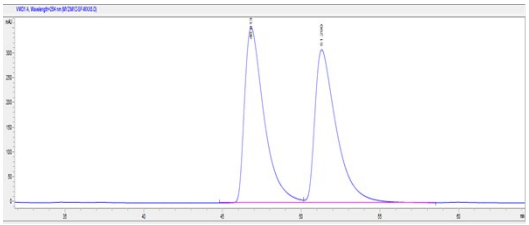


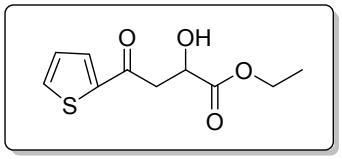
(*R*)-4f

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	47.102	97470	951.2	1.4575	0.321	98.127
2	53.972	1860.2	20.3	1.3956	0.813	1.873

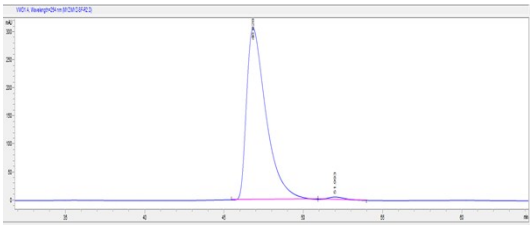
4- thienyl -2-hydroxy-4-oxo-butyric acid ethyl ester (4g) —— RuCl(*p*-cymene)[(*R*,*R*)-Ts-DPEN]

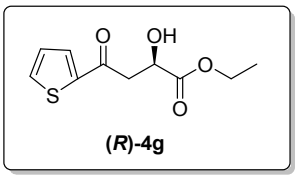
71% yield, 95% ee. determined by HPLC analysis (Chiralcel OJ-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	46.813	30557.2	354.4	1.3079	0.468	51.012
2	51.29	29344.4	309.6	1.4038	0.429	48.988





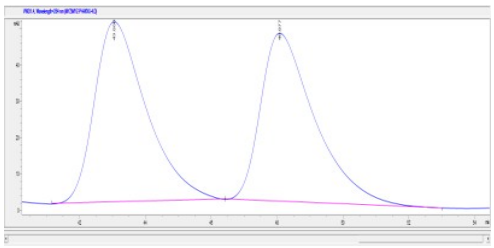
(*R*)-4g

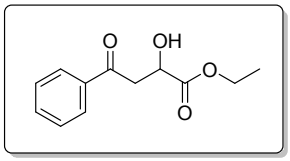
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	46.828	26226.1	306.8	1.2814	0.471	98.708
2	51.993	343.2	4.9	0.9975	0.726	1.292

5.HPLC charts 4a-4g [catalyst was ferrocene-based chiral ligand]

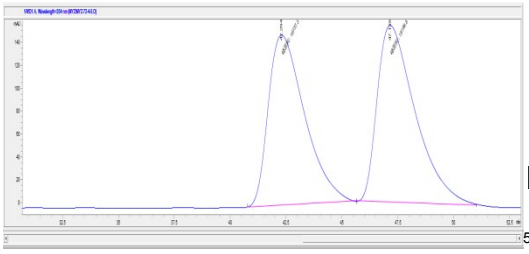
4-phenyl-2-hydroxy-4-oxo-butyric acid ethyl ester (4a)

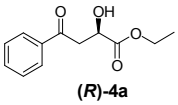
72% yield, 5% ee catalyzed by $\mathbf{L_2}$ in DMF at r.t., 57% yield, 90% ee catalyzed by $\mathbf{L_1}$, 60% yield, 65% ee catalyzed by $\mathbf{L_2}$, 55% yield, 50% ee catalyzed by $\mathbf{L_3}$, 47% yield, racemic catalyzed by $\mathbf{L_4}$, 50% yield, 40% ee catalyzed by $\mathbf{L_5}$, 52% yield, 37% ee catalyzed by $\mathbf{L_6}$ in DMF at -20℃ for 4days, determined by HPLC analysis (Chiralcel OD-H column, Hexane/i-PrOH=95/5, Flow rate: 0.5 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	43.05	5475.5	49.9	1.614	0.588	50.736
2	48.077	5317	46.5	1.6733	0.517	49.265



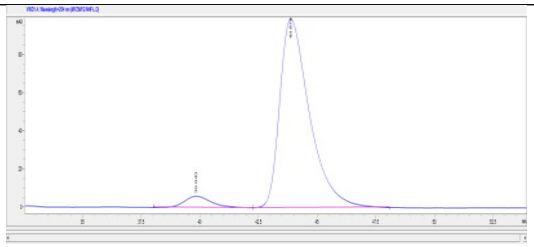


(*R*)-4a

5% ee catalyzed by L2 at r.t.

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	42.284	16737.3	148.2	1.8828	0.562	47.507
2	47.139	18199.8	153.8	1.9722	0.501	52.493

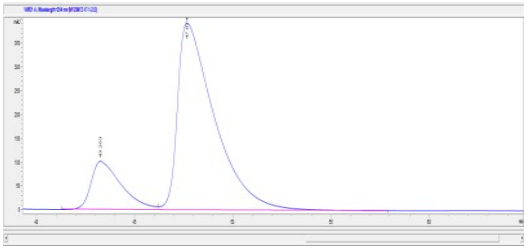
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	40.432	14740.9	269.7	0.911	0.668	49.301
2	44.351	15159.2	249.9	1.011	0.656	50.699



(*R*)-4a

90% ee (L_1)

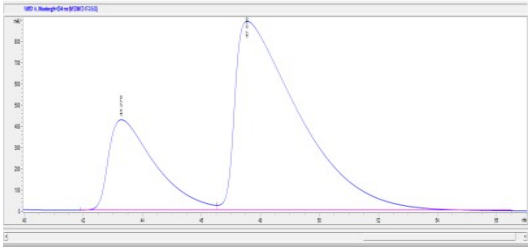
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	39.483	469.3	5.9	1.1422	0.765	5.091
2	43.878	8748.5	99.2	1.33	0.554	94.909



(*R*)-4a

65% ee (L_2)

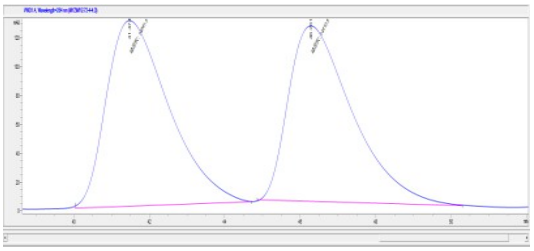
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	43.253	10867.7	102	1.4165	0.435	17.423
2	47.674	51506.5	392.8	1.8838	0.33	82.577

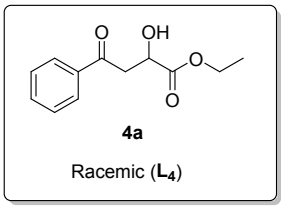


(*R*)-4a

50% ee (L_3)

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	43.279	46839.3	426.6	1.6099	0.349	25.757
2	47.545	135011.3	891.2	2.1804	0.215	74.243

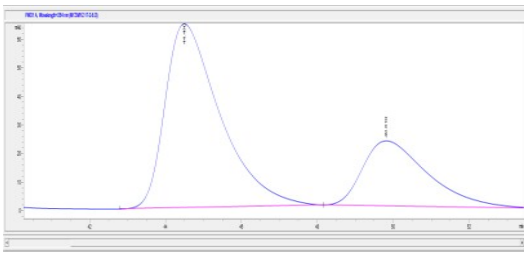


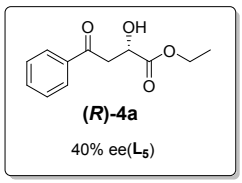


4a

Racemic (**L$_4$**)

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	41.474	14941.5	129..3	1.9256	0.576	50.904
2	46.81	14410.8	122.2	1.966	0.527	49.096

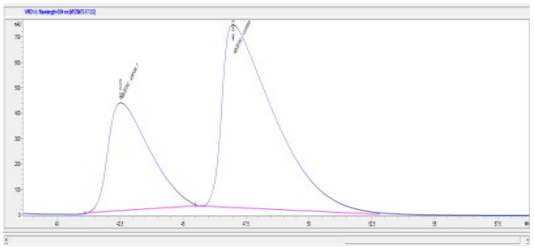


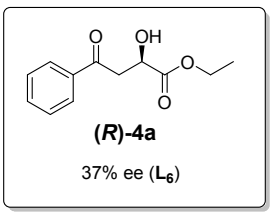


(***R***)**-4a**

40% ee(**L$_5$**)

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	44.49	6473.9	64.3	1.4882	0.51	70.528
2	49.81	2705.3	22.8	1.6719	0.542	29.472





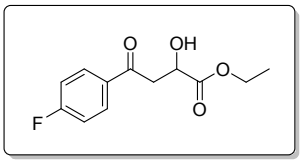
(***R***)**-4a**

37% ee (**L$_6$**)

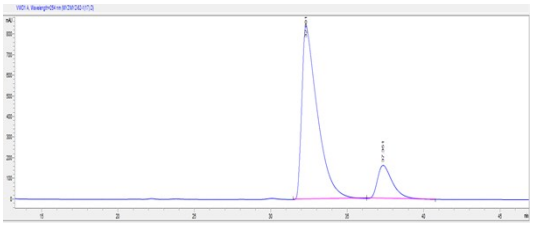
#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	42.525	48606.7	423.6	1.9124	0.43	31.943
2	46.993	716.7	22.8	2.4084	0.261	68.057

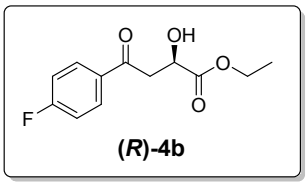
4-(4-F-phenyl)-2-hydroxy-4-oxo-butyric acid ethyl ester (4b) ——L_1

55% yield, 69% ee determined by HPLC analysis (Chiralcel OJ-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).



#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	33.427	1175	20.3	0.882	0.665	48.600
2	37.88	1242.7	20	0.9325	0.648	51.400



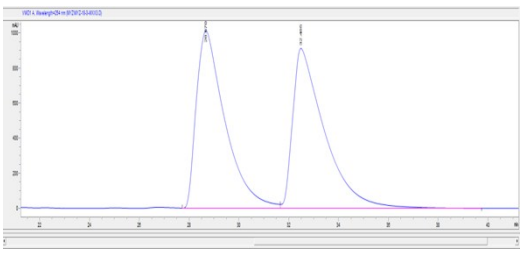


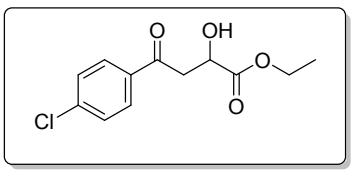
(*R*)-4b

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	32.301	59937.7	841.9	1.0216	0.335	84.408
2	37.351	11072.1	162.1	1.0298	0.543	15.592

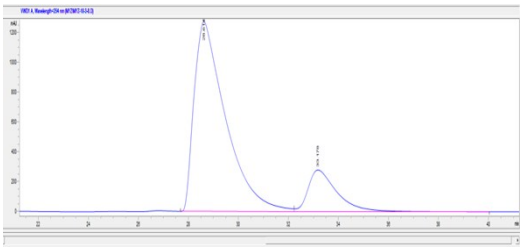
4-(4-Cl-phenyl)-2-hydroxy-4-oxo-butyric acid ethyl ester (4c) ——L_1

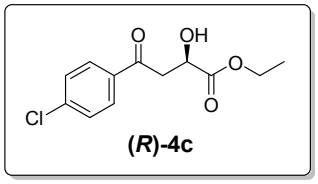
58% yield, 67% ee determined by HPLC analysis (Chiralcel OJ-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	31.338	5549	86.3	0.9581	0.595	53.769
2	34.585	4771	70.1	1.0145	0.569	46.231



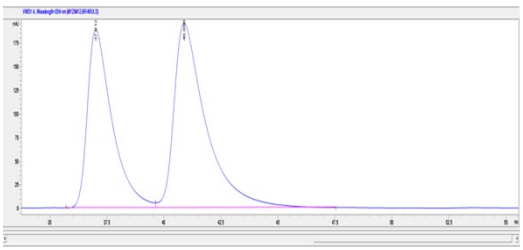


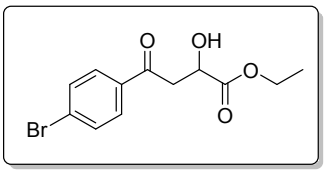
(*R*)-4c

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	31.41	6620.2	102.1	0.9703	0.574	83.369
2	34.772	1320.7	20.2	0.9871	0.648	16.631

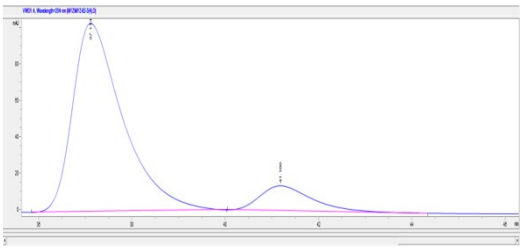
4-(4-Br-phenyl)-2-hydroxy-4-oxo-butyric acid ethyl ester (4d) ——L_1

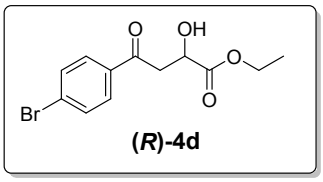
53% yield, 75% ee determined by HPLC analysis (Chiralcel OD-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	37.012	16215.8	18808	1.1046	0.474	47.798
2	40.888	17000.3	190.5	1.4116	0.431	52.202



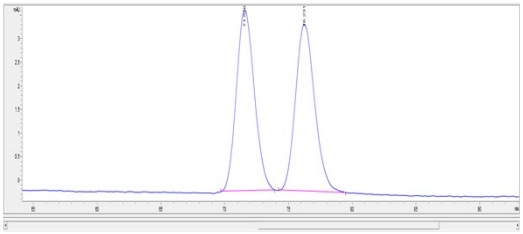


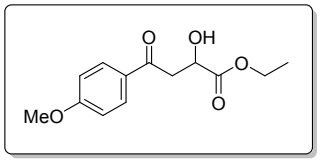
(*R*)-4d

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	37.114	7698.2	102.9	1.1047	0.531	87.897
2	41.186	1060	13.6	1.1441	0.619	12.103

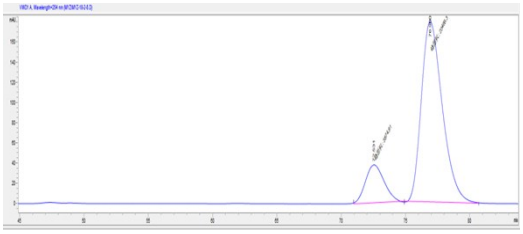
4-(4-OMe-phenyl)-2-hydroxy-4-oxo-butyric acid ethyl ester (4e) ——L_1

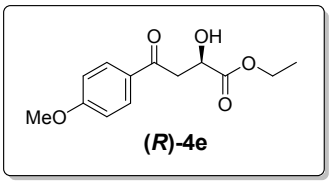
50% yield, 69% ee determined by HPLC analysis (Chiralcel AD-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	71.556	375.8	3.8	1.1572	0.849	50.267
2	76.231	371.8	3.6	1.236	0.846	49.733



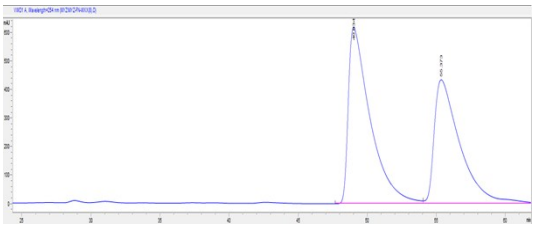


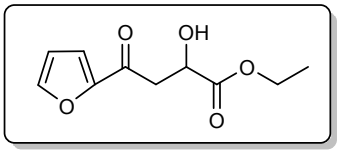
(*R*)-4e

#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	72.531	3874.9	38	1.7005	0.834	15.917
2	76.9	20469.3	179.6	1.8993	0.702	84.083

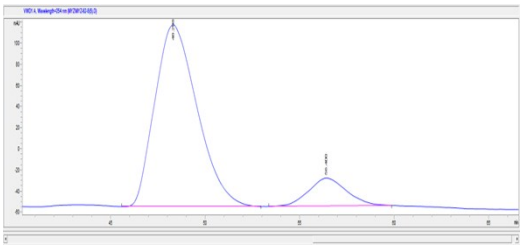
4-furyl-2-hydroxy-4-oxo-butyric acid ethyl ester (4f) ——L_1

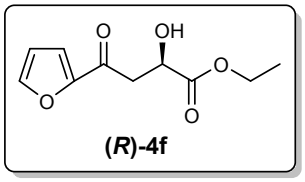
48% yield, 75% ee determined by HPLC analysis (Chiralcel OJ-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	49.034	68005.2	615.1	1.5849	0.297	54.691
2	55.373	56339	433.3	1.8509	0.345	45.309

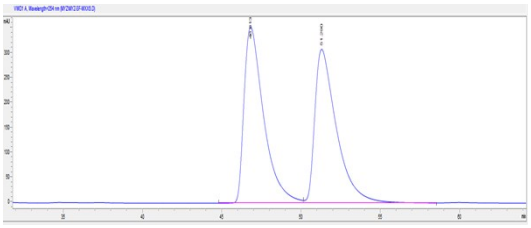


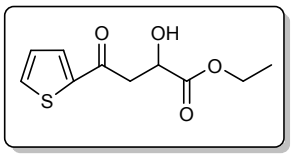


#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	48.298	26841	172.8	2.2983	0.725	87.815
2	56.4	3724.4	26.5	1.9414	0.863	12.185

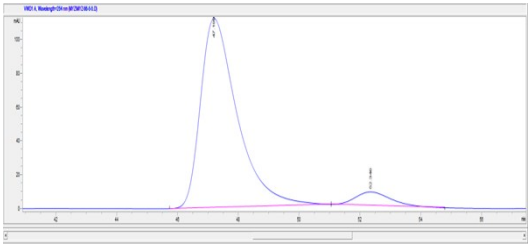
4- thienyl -2-hydroxy-4-oxo-butyric acid ethyl ester (4g) ——L_1

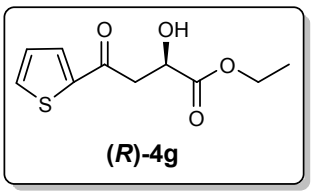
45% yield, 87% ee determined by HPLC analysis (Chiralcel OJ-H column, Hexane/i-PrOH=95/5, Flow rate: 1 mL/min, UV detection at 254 nm).





#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	46.813	30557.2	354.4	1.3079	0.468	51.012
2	51.29	29344.4	309.6	1.4038	0.429	48.988

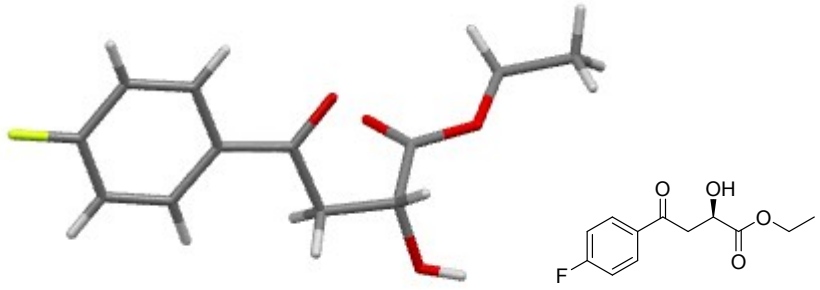




#	Time	Peak area	Peak height	Peak width	Symmetry factor	Peak area%
1	47.199	9358.3	111.7	1.2536	0.548	93.505
2	52.346	650	8	1.1669	0.717	6.495

6.Single crystal diffraction of (*R*)-4b and (*R*)-4e

（1）Single crystal diffraction of 4b



(*R*)-4b

Table 1. Crystal data and structure refinement for xb8331_0m.

Identification code	xb8331_0m
Empirical formula	C12 H13 F O4
Formula weight	240.22
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 5.059(5) A alpha = 90 deg.
	b = 10.704(12) A beta = 101.62(2) deg.
	c = 11.363(12) A gamma = 90 deg.
Volume	602.6(11) A^3
Z, Calculated density	2, 1.324 Mg/m^3
Absorption coefficient	0.109 mm^-1
F(000)	252
Crystal size	0.31 x 0.23 x 0.12 mm
Theta range for data collection	1.83 to 25.08 deg.
Limiting indices	-6<=h<=5, -12<=k<=12, -13<=l<=8
Reflections collected / unique	2945 / 2093 [R(int) = 0.0512]
Completeness to theta = 25.08	99.2 %
Absorption correction	None
Max. and min. transmission	0.9865 and 0.9674
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2093 / 1 / 157
Goodness-of-fit on F^2	1.064
Final R indices [I>2sigma(I)]	R1 = 0.0777, wR2 = 0.1627

R indices (all data) R1 = 0.1518, wR2 = 0.1941

Absolute structure parameter 0(3)

Extinction coefficient 0.036(9)

Largest diff. peak and hole 0.205 and -0.224 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for xb8331_0m. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
F(1)	3210(9)	7545(5)	13469(4)	94(2)
O(1)	8529(10)	9672(5)	9354(5)	74(2)
O(2)	13191(9)	7139(4)	7944(4)	59(1)
O(3)	7179(10)	8086(6)	6865(4)	78(2)
O(4)	10569(8)	9223(4)	6455(4)	61(1)
C(1)	7262(14)	7087(6)	11221(6)	57(2)
C(2)	5922(14)	6851(7)	12142(6)	61(2)
C(3)	4501(16)	7770(8)	12558(6)	68(2)
C(4)	4479(16)	8989(7)	12129(7)	70(2)
C(5)	5812(13)	9240(7)	11218(6)	52(2)
C(6)	7258(12)	8294(6)	10755(5)	46(2)
C(7)	8602(13)	8612(6)	9757(6)	48(2)
C(8)	10246(12)	7610(6)	9244(6)	47(2)
C(9)	11518(12)	8114(6)	8254(5)	45(2)
C(10)	9492(15)	8481(6)	7132(6)	52(2)
C(11)	8891(17)	9574(10)	5287(7)	92(3)
C(12)	10490(20)	10447(11)	4691(7)	114(4)

Table 3. Bond lengths [A] and angles [deg] for xb8331_0m.

F(1)-C(3)	1.352(8)
O(1)-C(7)	1.221(7)
O(2)-C(9)	1.431(7)
O(2)-H(2)	0.8200
O(3)-C(10)	1.223(8)
O(4)-C(10)	1.300(7)
O(4)-C(11)	1.473(8)

C(1)-C(2)	1.379(9)
C(1)-C(6)	1.396(9)
C(1)-H(1)	0.9300
C(2)-C(3)	1.358(10)
C(2)-H(2A)	0.9300
C(3)-C(4)	1.392(10)
C(4)-C(5)	1.371(9)
C(4)-H(4)	0.9300
C(5)-C(6)	1.411(9)
C(5)-H(5)	0.9300
C(6)-C(7)	1.475(9)
C(7)-C(8)	1.542(8)
C(8)-C(9)	1.505(8)
C(8)-H(8A)	0.9700
C(8)-H(8B)	0.9700
C(9)-C(10)	1.517(8)
C(9)-H(9)	0.9800
C(11)-C(12)	1.485(11)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600

C(9)-O(2)-H(2)	109.5
C(10)-O(4)-C(11)	117.1(6)
C(2)-C(1)-C(6)	119.6(7)
C(2)-C(1)-H(1)	120.2
C(6)-C(1)-H(1)	120.2
C(3)-C(2)-C(1)	120.5(7)
C(3)-C(2)-H(2A)	119.8
C(1)-C(2)-H(2A)	119.8
F(1)-C(3)-C(2)	120.6(7)
F(1)-C(3)-C(4)	117.6(7)
C(2)-C(3)-C(4)	121.6(7)
C(5)-C(4)-C(3)	118.5(7)
C(5)-C(4)-H(4)	120.8
C(3)-C(4)-H(4)	120.8
C(4)-C(5)-C(6)	120.8(7)
C(4)-C(5)-H(5)	119.6
C(6)-C(5)-H(5)	119.6
C(1)-C(6)-C(5)	118.9(6)

C(1)-C(6)-C(7)	122.7(6)
C(5)-C(6)-C(7)	118.4(6)
O(1)-C(7)-C(6)	121.3(6)
O(1)-C(7)-C(8)	118.9(6)
C(6)-C(7)-C(8)	119.8(6)
C(9)-C(8)-C(7)	112.2(5)
C(9)-C(8)-H(8A)	109.2
C(7)-C(8)-H(8A)	109.2
C(9)-C(8)-H(8B)	109.2
C(7)-C(8)-H(8B)	109.2
H(8A)-C(8)-H(8B)	107.9
O(2)-C(9)-C(8)	106.6(5)
O(2)-C(9)-C(10)	108.3(5)
C(8)-C(9)-C(10)	113.8(5)
O(2)-C(9)-H(9)	109.3
C(8)-C(9)-H(9)	109.3
C(10)-C(9)-H(9)	109.3
O(3)-C(10)-O(4)	124.0(6)
O(3)-C(10)-C(9)	124.7(6)
O(4)-C(10)-C(9)	111.3(6)
O(4)-C(11)-C(12)	107.7(6)
O(4)-C(11)-H(11A)	110.2
C(12)-C(11)-H(11A)	110.2
O(4)-C(11)-H(11B)	110.2
C(12)-C(11)-H(11B)	110.2
H(11A)-C(11)-H(11B)	108.5
C(11)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for xb8331_0m.

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
F(1)	109(4)	115(4)	73(3)	4(3)	53(3)	-5(3)
O(1)	80(4)	53(3)	93(4)	19(3)	25(3)	9(3)
O(2)	47(3)	50(3)	91(4)	13(3)	39(3)	14(2)
O(3)	42(3)	132(5)	60(3)	13(3)	8(3)	-23(3)
O(4)	50(3)	73(3)	59(3)	22(3)	10(2)	-8(3)
C(1)	68(5)	48(5)	52(4)	5(4)	9(4)	1(4)
C(2)	66(5)	65(5)	53(5)	15(4)	15(4)	1(4)
C(3)	81(5)	78(6)	46(4)	1(4)	15(4)	-3(5)
C(4)	88(6)	51(5)	69(6)	-9(4)	7(5)	3(5)
C(5)	58(4)	48(4)	56(5)	-1(4)	25(4)	1(4)
C(6)	40(4)	46(4)	49(4)	0(3)	5(3)	3(3)
C(7)	42(4)	41(4)	57(4)	1(4)	2(3)	2(3)
C(8)	38(4)	50(4)	56(4)	2(3)	14(3)	2(3)
C(9)	40(4)	36(4)	55(4)	8(3)	2(3)	-1(3)
C(10)	50(4)	45(4)	60(5)	14(4)	8(4)	2(4)
C(11)	81(6)	125(8)	61(5)	32(5)	-6(5)	-20(6)
C(12)	121(8)	143(9)	66(7)	39(6)	-6(6)	-16(7)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for xb8331_0m.

	x	y	z	U(eq)
H(2)	14510	7447	7737	88
H(1)	8162	6446	10913	68
H(2A)	5992	6058	12480	73
H(4)	3580	9619	12453	85
H(5)	5763	10043	10902	62
H(8A)	11649	7293	9885	57
H(8B)	9068	6918	8937	57
H(9)	12640	8838	8551	53
H(11A)	7244	9976	5404	110
H(11B)	8410	8837	4794	110
H(12A)	9617	11248	4595	170
H(12B)	10617	10123	3917	170
H(12C)	12268	10535	5176	170

Table 6. Torsion angles [deg] for xb8331_0m.

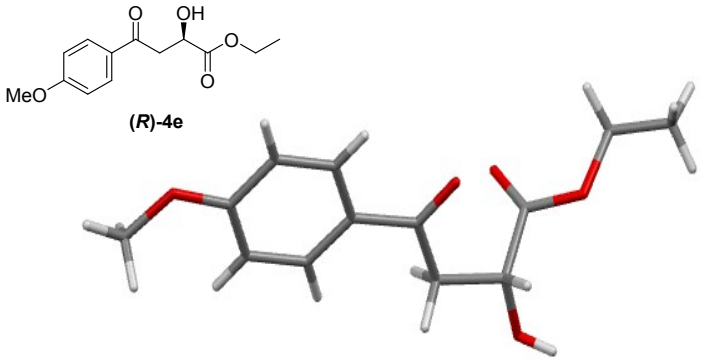
C(6)-C(1)-C(2)-C(3)	3.0(10)
C(1)-C(2)-C(3)-F(1)	-179.3(6)
C(1)-C(2)-C(3)-C(4)	-4.0(11)
F(1)-C(3)-C(4)-C(5)	179.0(7)
C(2)-C(3)-C(4)-C(5)	3.6(11)
C(3)-C(4)-C(5)-C(6)	-2.3(11)
C(2)-C(1)-C(6)-C(5)	-1.7(9)
C(2)-C(1)-C(6)-C(7)	-178.8(6)
C(4)-C(5)-C(6)-C(1)	1.4(10)
C(4)-C(5)-C(6)-C(7)	178.6(6)
C(1)-C(6)-C(7)-O(1)	178.8(7)
C(5)-C(6)-C(7)-O(1)	1.7(10)
C(1)-C(6)-C(7)-C(8)	-4.1(9)
C(5)-C(6)-C(7)-C(8)	178.8(6)
O(1)-C(7)-C(8)-C(9)	-1.4(8)
C(6)-C(7)-C(8)-C(9)	-178.6(5)
C(7)-C(8)-C(9)-O(2)	174.2(5)
C(7)-C(8)-C(9)-C(10)	-66.4(7)
C(11)-O(4)-C(10)-O(3)	-1.6(10)
C(11)-O(4)-C(10)-C(9)	175.2(6)
O(2)-C(9)-C(10)-O(3)	96.7(8)
C(8)-C(9)-C(10)-O(3)	-21.6(9)
O(2)-C(9)-C(10)-O(4)	-80.0(6)
C(8)-C(9)-C(10)-O(4)	161.6(6)
C(10)-O(4)-C(11)-C(12)	178.4(7)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for xb8331_0m [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

(2) Single crystal diffraction of (*R*)-4e



(*R*)-4e

Table 1. Crystal data and structure refinement for xb8523_0m.

Identification code	xb8523_0m
Empirical formula	C13 H16 O5
Formula weight	252.26
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 5.0181(11) A alpha = 90 deg. b = 11.253(2) A beta = 90.618(4) deg. c = 22.901(5) A gamma = 90 deg.
Volume	1293.1(5) A^3
Z, Calculated density	4, 1.296 Mg/m^3

Absorption coefficient	0.100 mm^-1
F(000)	536
Crystal size	0.37 x 0.25 x 0.14 mm
Theta range for data collection	0.89 to 25.10 deg.
Limiting indices	-5<=h<=5, -13<=k<=12, -25<=l<=27
Reflections collected / unique	6419 / 4112 [R(int) = 0.0225]
Completeness to theta = 25.10	99.8 %
Absorption correction	None
Max. and min. transmission	0.9860 and 0.9643
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4112 / 1 / 331
Goodness-of-fit on F^2	1.035
Final R indices [I>2sigma(I)]	R1 = 0.0538, wR2 = 0.1144
R indices (all data)	R1 = 0.0868, wR2 = 0.1303
Absolute structure parameter	0.9(14)
Extinction coefficient	0.0127(16)
Largest diff. peak and hole	0.175 and -0.183 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for xb8523_0m. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)

O(1)	6972(6)	6430(3)	11332(1)	84(1)
O(2)	-194(5)	4730(2)	9226(1)	80(1)
O(3)	-5432(5)	7218(3)	8586(1)	82(1)
O(4)	-3452(5)	5140(3)	7878(1)	80(1)
O(5)	-55(5)	6326(3)	8073(1)	87(1)
O(6)	12397(6)	6254(2)	6411(1)	79(1)
O(7)	5117(6)	4451(2)	4341(1)	79(1)
O(8)	-218(5)	6898(3)	3712(1)	80(1)
O(9)	1665(5)	4901(3)	2973(1)	109(1)
O(10)	5200(5)	6002(3)	3174(1)	84(1)
C(1)	7251(10)	7545(4)	11623(2)	102(2)
C(2)	5217(7)	6348(3)	10886(2)	62(1)
C(3)	3612(7)	7248(3)	10684(2)	62(1)
C(4)	1877(7)	7045(3)	10222(2)	58(1)
C(5)	1704(6)	5952(3)	9953(1)	52(1)
C(6)	3353(7)	5043(3)	10163(2)	65(1)
C(7)	5077(8)	5231(4)	10618(2)	68(1)
C(8)	-130(7)	5702(3)	9458(2)	57(1)
C(9)	-1954(7)	6684(3)	9233(2)	59(1)
C(10)	-3731(7)	6256(3)	8737(2)	59(1)
C(11)	-2156(7)	5912(4)	8208(2)	64(1)
C(12)	-2225(10)	4806(6)	7327(2)	109(2)
C(13)	-3813(13)	3976(5)	7032(2)	137(2)
C(14)	12651(10)	7386(4)	6686(2)	95(2)
C(15)	10603(7)	6141(4)	5966(2)	60(1)
C(16)	8978(7)	7041(3)	5765(2)	60(1)
C(17)	7206(7)	6812(3)	5311(2)	58(1)
C(18)	7023(7)	5697(3)	5054(1)	52(1)
C(19)	8693(7)	4808(3)	5267(2)	62(1)
C(20)	10459(8)	5025(3)	5717(2)	69(1)
C(21)	5166(7)	5436(3)	4564(2)	58(1)
C(22)	3354(7)	6388(3)	4338(2)	60(1)
C(23)	1556(7)	5964(3)	3851(2)	63(1)
C(24)	3059(7)	5633(4)	3310(2)	66(1)
C(25)	2743(10)	4611(8)	2400(2)	161(3)
C(26)	970(13)	4493(7)	1995(2)	158(3)

Table 3. Bond lengths [A] and angles [deg] for xb8523_0m.

O(1)-C(2)	1.345(4)
O(1)-C(1)	1.427(5)
O(2)-C(8)	1.217(4)
O(3)-C(10)	1.418(4)
O(3)-H(3)	0.8200
O(4)-C(11)	1.318(4)
O(4)-C(12)	1.458(4)
O(5)-C(11)	1.196(4)
O(6)-C(15)	1.358(4)
O(6)-C(14)	1.425(5)
O(7)-C(21)	1.221(4)
O(8)-C(23)	1.412(4)
O(8)-H(8)	0.8200
O(9)-C(24)	1.323(4)
O(9)-C(25)	1.463(5)
O(10)-C(24)	1.196(4)
C(1)-H(1A)	0.9600
C(1)-H(1B)	0.9600
C(1)-H(1C)	0.9600
C(2)-C(3)	1.370(5)
C(2)-C(7)	1.400(5)
C(3)-C(4)	1.382(5)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.377(5)
C(4)-H(4)	0.9300
C(5)-C(6)	1.398(5)
C(5)-C(8)	1.479(5)
C(6)-C(7)	1.364(5)
C(6)-H(6)	0.9300
C(7)-H(7)	0.9300
C(8)-C(9)	1.522(5)
C(9)-C(10)	1.515(4)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-C(11)	1.505(5)
C(10)-H(10)	0.9800
C(12)-C(13)	1.397(7)
C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600

C(14)-H(14A)	0.9600
C(14)-H(14B)	0.9600
C(14)-H(14C)	0.9600
C(15)-C(16)	1.377(5)
C(15)-C(20)	1.380(5)
C(16)-C(17)	1.385(5)
C(16)-H(16)	0.9300
C(17)-C(18)	1.388(4)
C(17)-H(17)	0.9300
C(18)-C(19)	1.389(5)
C(18)-C(21)	1.482(5)
C(19)-C(20)	1.375(5)
C(19)-H(19)	0.9300
C(20)-H(20)	0.9300
C(21)-C(22)	1.494(5)
C(22)-C(23)	1.504(4)
C(22)-H(22A)	0.9700
C(22)-H(22B)	0.9700
C(23)-C(24)	1.504(5)
C(23)-H(23)	0.9800
C(25)-C(26)	1.284(7)
C(25)-H(25A)	0.9700
C(25)-H(25B)	0.9700
C(26)-H(26A)	0.9600
C(26)-H(26B)	0.9600
C(26)-H(26C)	0.9600

C(2)-O(1)-C(1)	118.4(3)
C(10)-O(3)-H(3)	109.5
C(11)-O(4)-C(12)	117.1(3)
C(15)-O(6)-C(14)	118.1(3)
C(23)-O(8)-H(8)	109.5
C(24)-O(9)-C(25)	117.6(3)
O(1)-C(1)-H(1A)	109.5
O(1)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
O(1)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
O(1)-C(2)-C(3)	125.6(4)
O(1)-C(2)-C(7)	115.1(3)
C(3)-C(2)-C(7)	119.3(4)

C(2)-C(3)-C(4) 119.9(4)
C(2)-C(3)-H(3A) 120.1
C(4)-C(3)-H(3A) 120.1
C(5)-C(4)-C(3) 121.8(3)
C(5)-C(4)-H(4) 119.1
C(3)-C(4)-H(4) 119.1
C(4)-C(5)-C(6) 117.7(3)
C(4)-C(5)-C(8) 123.2(3)
C(6)-C(5)-C(8) 119.0(3)
C(7)-C(6)-C(5) 121.1(4)
C(7)-C(6)-H(6) 119.4
C(5)-C(6)-H(6) 119.4
C(6)-C(7)-C(2) 120.2(4)
C(6)-C(7)-H(7) 119.9
C(2)-C(7)-H(7) 119.9
O(2)-C(8)-C(5) 121.3(4)
O(2)-C(8)-C(9) 119.4(3)
C(5)-C(8)-C(9) 119.3(3)
C(10)-C(9)-C(8) 111.7(3)
C(10)-C(9)-H(9A) 109.3
C(8)-C(9)-H(9A) 109.3
C(10)-C(9)-H(9B) 109.3
C(8)-C(9)-H(9B) 109.3
H(9A)-C(9)-H(9B) 107.9
O(3)-C(10)-C(11) 108.7(3)
O(3)-C(10)-C(9) 106.7(3)
C(11)-C(10)-C(9) 112.1(3)
O(3)-C(10)-H(10) 109.8
C(11)-C(10)-H(10) 109.8
C(9)-C(10)-H(10) 109.8
O(5)-C(11)-O(4) 122.7(4)
O(5)-C(11)-C(10) 125.4(4)
O(4)-C(11)-C(10) 111.8(3)
C(13)-C(12)-O(4) 110.2(4)
C(13)-C(12)-H(12A) 109.6
O(4)-C(12)-H(12A) 109.6
C(13)-C(12)-H(12B) 109.6
O(4)-C(12)-H(12B) 109.6
H(12A)-C(12)-H(12B) 108.1
C(12)-C(13)-H(13A) 109.5
C(12)-C(13)-H(13B) 109.5
H(13A)-C(13)-H(13B) 109.5

C(12)-C(13)-H(13C) 109.5
H(13A)-C(13)-H(13C) 109.5
H(13B)-C(13)-H(13C) 109.5
O(6)-C(14)-H(14A) 109.5
O(6)-C(14)-H(14B) 109.5
H(14A)-C(14)-H(14B) 109.5
O(6)-C(14)-H(14C) 109.5
H(14A)-C(14)-H(14C) 109.5
H(14B)-C(14)-H(14C) 109.5
O(6)-C(15)-C(16) 124.5(4)
O(6)-C(15)-C(20) 115.2(4)
C(16)-C(15)-C(20) 120.2(4)
C(15)-C(16)-C(17) 119.1(3)
C(15)-C(16)-H(16) 120.4
C(17)-C(16)-H(16) 120.4
C(16)-C(17)-C(18) 121.7(3)
C(16)-C(17)-H(17) 119.2
C(18)-C(17)-H(17) 119.2
C(17)-C(18)-C(19) 117.8(3)
C(17)-C(18)-C(21) 122.6(3)
C(19)-C(18)-C(21) 119.6(3)
C(20)-C(19)-C(18) 121.1(3)
C(20)-C(19)-H(19) 119.5
C(18)-C(19)-H(19) 119.5
C(19)-C(20)-C(15) 120.1(4)
C(19)-C(20)-H(20) 120.0
C(15)-C(20)-H(20) 120.0
O(7)-C(21)-C(18) 120.4(4)
O(7)-C(21)-C(22) 119.8(4)
C(18)-C(21)-C(22) 119.8(3)
C(21)-C(22)-C(23) 112.8(3)
C(21)-C(22)-H(22A) 109.0
C(23)-C(22)-H(22A) 109.0
C(21)-C(22)-H(22B) 109.0
C(23)-C(22)-H(22B) 109.0
H(22A)-C(22)-H(22B) 107.8
O(8)-C(23)-C(22) 107.6(3)
O(8)-C(23)-C(24) 108.6(3)
C(22)-C(23)-C(24) 112.8(3)
O(8)-C(23)-H(23) 109.3
C(22)-C(23)-H(23) 109.3
C(24)-C(23)-H(23) 109.3

O(10)-C(24)-O(9)	122.4(4)
O(10)-C(24)-C(23)	126.0(4)
O(9)-C(24)-C(23)	111.6(3)
C(26)-C(25)-O(9)	114.2(5)
C(26)-C(25)-H(25A)	108.7
O(9)-C(25)-H(25A)	108.7
C(26)-C(25)-H(25B)	108.7
O(9)-C(25)-H(25B)	108.7
H(25A)-C(25)-H(25B)	107.6
C(25)-C(26)-H(26A)	109.5
C(25)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(25)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for xb8523_0m.

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	100(2)	78(2)	73(2)	2(2)	-22(2)	9(2)
O(2)	88(2)	56(2)	96(2)	-21(2)	-5(2)	-2(2)
O(3)	53(2)	83(2)	110(2)	-14(2)	-11(2)	4(1)
O(4)	66(2)	102(2)	73(2)	-25(2)	17(1)	-20(2)
O(5)	57(2)	125(3)	79(2)	-8(2)	11(1)	-32(2)
O(6)	95(2)	76(2)	66(2)	0(2)	-8(2)	-3(2)
O(7)	93(2)	53(2)	93(2)	-25(2)	-1(2)	-3(1)
O(8)	55(2)	79(2)	106(2)	-20(2)	-5(2)	-3(2)
O(9)	62(2)	163(3)	102(2)	-70(2)	20(2)	-33(2)
O(10)	56(2)	117(3)	79(2)	-10(2)	13(1)	-25(2)
C(1)	126(4)	104(4)	76(3)	-12(3)	-26(3)	-2(3)
C(2)	63(2)	65(3)	57(2)	3(2)	5(2)	1(2)
C(3)	68(2)	58(2)	61(2)	-7(2)	1(2)	1(2)
C(4)	61(2)	50(2)	61(2)	1(2)	2(2)	8(2)
C(5)	53(2)	44(2)	58(2)	3(2)	14(2)	-3(2)

C(6)	75(3)	45(2)	75(3)	2(2)	15(2)	3(2)
C(7)	76(3)	59(3)	69(3)	11(2)	-2(2)	8(2)
C(8)	54(2)	55(3)	63(2)	-7(2)	12(2)	-7(2)
C(9)	50(2)	62(3)	64(2)	-5(2)	8(2)	-3(2)
C(10)	43(2)	65(3)	69(2)	-9(2)	4(2)	-8(2)
C(11)	48(2)	74(3)	69(2)	-1(2)	1(2)	-10(2)
C(12)	101(4)	154(5)	73(3)	-36(3)	33(3)	-29(4)
C(13)	177(6)	133(5)	102(4)	-42(4)	42(4)	-33(5)
C(14)	127(4)	87(3)	71(3)	-13(3)	-16(3)	-15(3)
C(15)	63(2)	63(3)	53(2)	1(2)	8(2)	-6(2)
C(16)	70(2)	49(2)	61(2)	-4(2)	5(2)	-2(2)
C(17)	61(2)	45(2)	67(2)	-9(2)	8(2)	2(2)
C(18)	57(2)	47(2)	53(2)	-1(2)	13(2)	-2(2)
C(19)	75(3)	42(2)	70(3)	-4(2)	17(2)	-4(2)
C(20)	81(3)	51(3)	74(3)	3(2)	3(2)	6(2)
C(21)	59(2)	49(2)	68(2)	-8(2)	22(2)	-9(2)
C(22)	56(2)	52(2)	72(2)	-13(2)	7(2)	-8(2)
C(23)	51(2)	59(2)	78(2)	-15(2)	11(2)	-12(2)
C(24)	47(2)	78(3)	72(2)	-12(2)	2(2)	-7(2)
C(25)	72(4)	303(9)	107(4)	-109(6)	14(3)	-15(5)
C(26)	149(6)	240(9)	84(4)	25(5)	4(4)	59(6)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for xb8523_0m.

	x	y	z	U(eq)
H(3)	-6863	6959	8466	123
H(8)	-1453	6640	3512	120
H(1A)	7904	8128	11354	154
H(1B)	8486	7465	11943	154
H(1C)	5550	7793	11767	154
H(3A)	3690	7994	10858	75
H(4)	797	7662	10089	69
H(6)	3274	4297	9989	78
H(7)	6163	4616	10750	82
H(9A)	-3055	6968	9550	71
H(9B)	-881	7345	9099	71

H(10)	-4798	5578	8867	71
H(12A)	-2005	5506	7085	131
H(12B)	-476	4469	7403	131
H(13A)	-3723	3226	7232	205
H(13B)	-3183	3881	6641	205
H(13C)	-5626	4249	7022	205
H(14A)	13228	7962	6405	143
H(14B)	13936	7336	6998	143
H(14C)	10958	7621	6839	143
H(16)	9068	7794	5932	72
H(17)	6111	7421	5175	69
H(19)	8614	4054	5101	75
H(20)	11559	4419	5854	82
H(22A)	4420	7046	4198	72
H(22B)	2270	6679	4655	72
H(23)	537	5275	3985	75
H(25A)	3974	5232	2287	193
H(25B)	3746	3876	2431	193
H(26A)	1824	4314	1633	237
H(26B)	-20	5219	1956	237
H(26C)	-220	3858	2095	237

Table 6. Torsion angles [deg] for xb8523_0m.

C(1)-O(1)-C(2)-C(3)	-0.9(6)
C(1)-O(1)-C(2)-C(7)	179.3(4)
O(1)-C(2)-C(3)-C(4)	-179.7(3)
C(7)-C(2)-C(3)-C(4)	0.1(5)
C(2)-C(3)-C(4)-C(5)	0.0(5)
C(3)-C(4)-C(5)-C(6)	0.0(5)
C(3)-C(4)-C(5)-C(8)	179.9(3)
C(4)-C(5)-C(6)-C(7)	-0.1(5)
C(8)-C(5)-C(6)-C(7)	180.0(3)
C(5)-C(6)-C(7)-C(2)	0.2(5)
O(1)-C(2)-C(7)-C(6)	179.6(3)
C(3)-C(2)-C(7)-C(6)	-0.2(6)
C(4)-C(5)-C(8)-O(2)	178.7(3)
C(6)-C(5)-C(8)-O(2)	-1.3(5)
C(4)-C(5)-C(8)-C(9)	-0.7(5)
C(6)-C(5)-C(8)-C(9)	179.3(3)

O(2)-C(8)-C(9)-C(10)	2.0(4)
C(5)-C(8)-C(9)-C(10)	-178.6(3)
C(8)-C(9)-C(10)-O(3)	176.8(3)
C(8)-C(9)-C(10)-C(11)	-64.4(4)
C(12)-O(4)-C(11)-O(5)	-0.6(6)
C(12)-O(4)-C(11)-C(10)	175.4(4)
O(3)-C(10)-C(11)-O(5)	88.8(4)
C(9)-C(10)-C(11)-O(5)	-28.8(5)
O(3)-C(10)-C(11)-O(4)	-86.9(4)
C(9)-C(10)-C(11)-O(4)	155.4(3)
C(11)-O(4)-C(12)-C(13)	179.3(5)
C(14)-O(6)-C(15)-C(16)	-0.9(5)
C(14)-O(6)-C(15)-C(20)	179.8(4)
O(6)-C(15)-C(16)-C(17)	-179.4(3)
C(20)-C(15)-C(16)-C(17)	-0.1(5)
C(15)-C(16)-C(17)-C(18)	0.1(5)
C(16)-C(17)-C(18)-C(19)	-0.2(5)
C(16)-C(17)-C(18)-C(21)	-179.2(3)
C(17)-C(18)-C(19)-C(20)	0.2(5)
C(21)-C(18)-C(19)-C(20)	179.3(3)
C(18)-C(19)-C(20)-C(15)	-0.2(6)
O(6)-C(15)-C(20)-C(19)	179.4(3)
C(16)-C(15)-C(20)-C(19)	0.1(6)
C(17)-C(18)-C(21)-O(7)	178.1(3)
C(19)-C(18)-C(21)-O(7)	-0.9(5)
C(17)-C(18)-C(21)-C(22)	-1.4(5)
C(19)-C(18)-C(21)-C(22)	179.6(3)
O(7)-C(21)-C(22)-C(23)	1.9(5)
C(18)-C(21)-C(22)-C(23)	-178.5(3)
C(21)-C(22)-C(23)-O(8)	174.6(3)
C(21)-C(22)-C(23)-C(24)	-65.6(4)
C(25)-O(9)-C(24)-O(10)	-4.2(7)
C(25)-O(9)-C(24)-C(23)	173.1(5)
O(8)-C(23)-C(24)-O(10)	94.6(5)
C(22)-C(23)-C(24)-O(10)	-24.6(6)
O(8)-C(23)-C(24)-O(9)	-82.6(4)
C(22)-C(23)-C(24)-O(9)	158.2(3)
C(24)-O(9)-C(25)-C(26)	-143.8(7)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for xb8523_0m [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)