

Supporting Information for

**Palladium Complexes Bearing Chiral bis(NHC) Chelating Ligands
on a Spiro Scaffold: Synthesis and Characterization, and Their
Application in Oxidative Kinetic Resolution of Secondary
Alcohols**

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CONTENTS

- 1. Full NMR Spectra for Bis-NHC-Pd Complexes together with all relevant Intermediates (Figures S1-22).....PS2-12**
- 2. Typical GC Characterization and analysis data of products.....PS13-20**

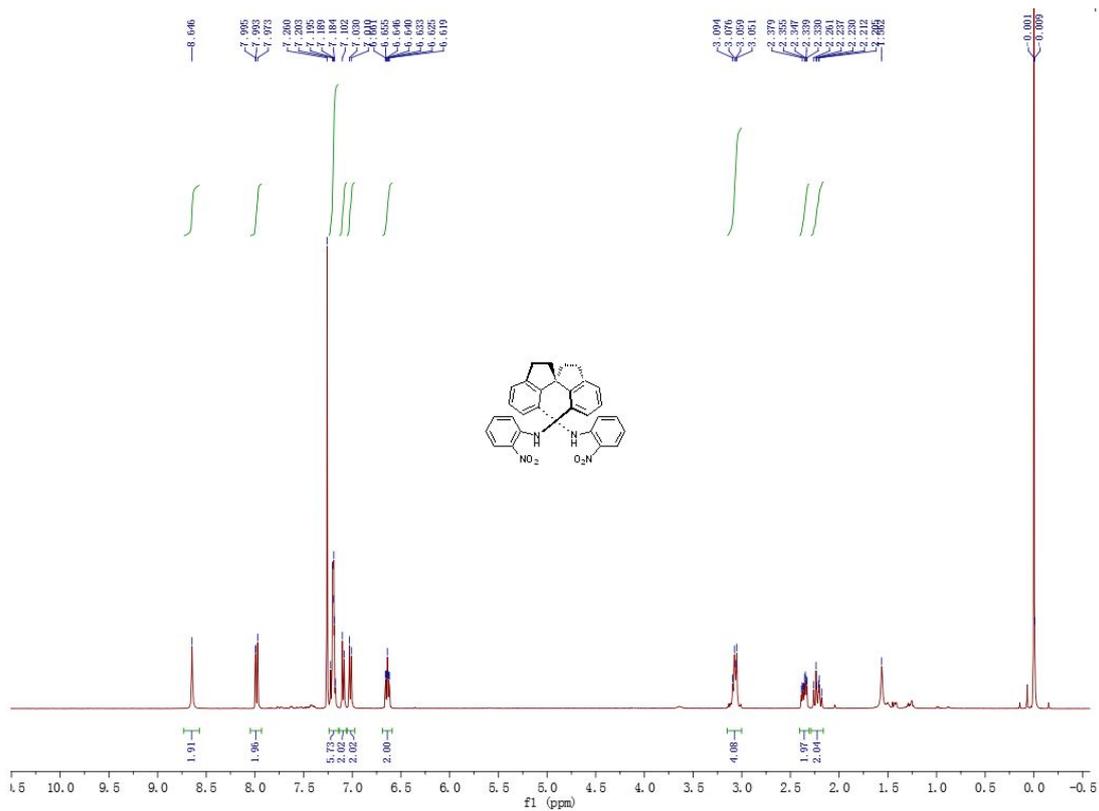


Figure S1. The ^1H NMR spectrum of (S)-5 in CDCl_3 at 20 °C

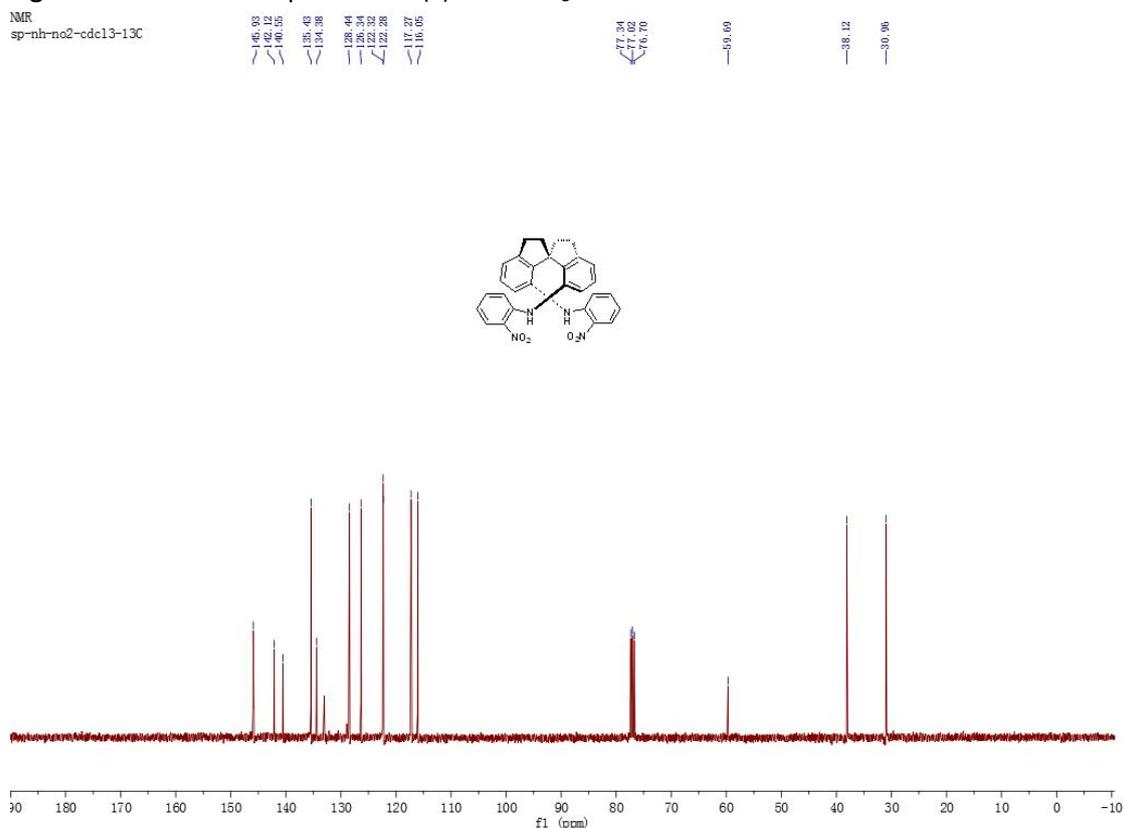


Figure S2. The ^{13}C NMR spectrum of (S)-5 in CDCl_3 at 20 °C

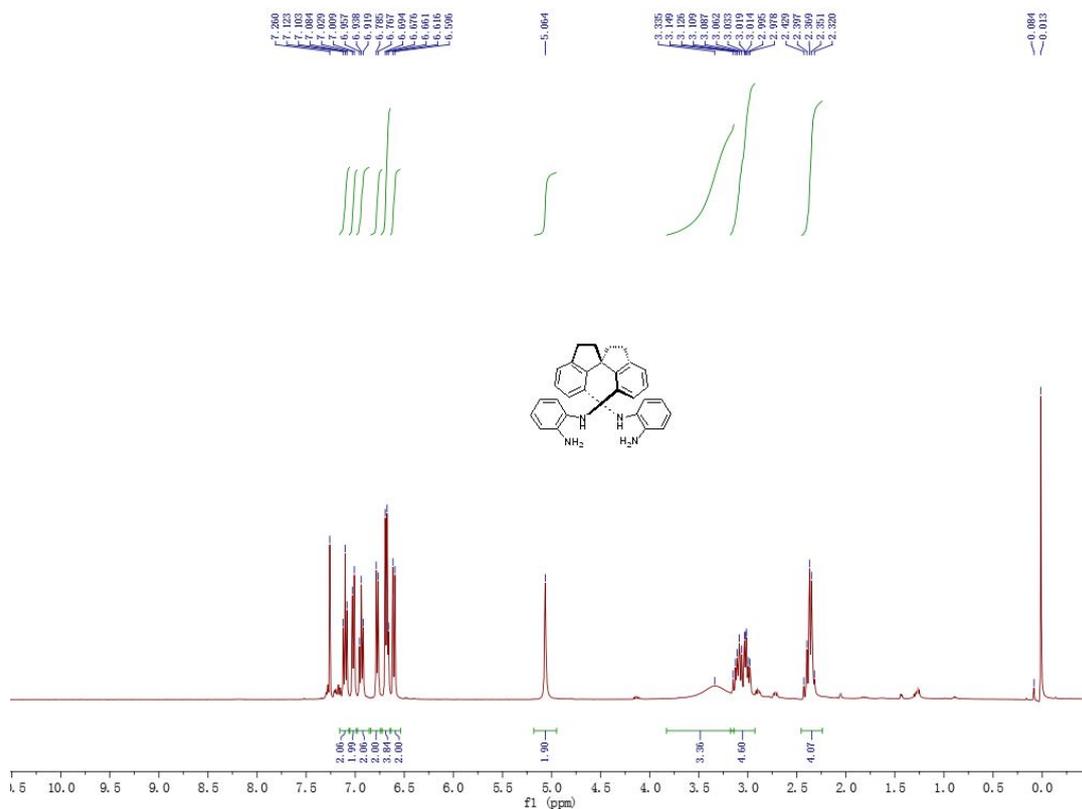


Figure S3. The ^1H NMR spectrum of (S)-6 in CDCl_3 at 20 °C

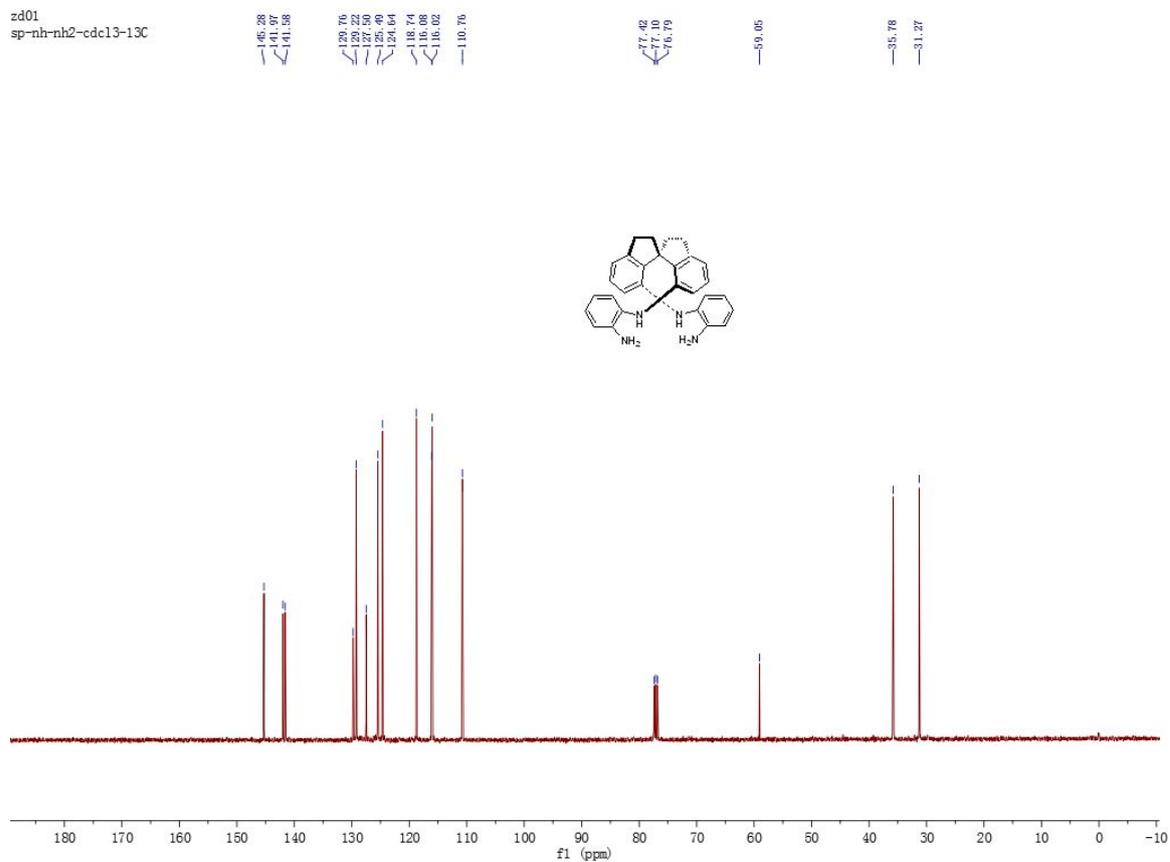


Figure S4. The ^{13}C NMR spectrum of (S)-6 in CDCl_3 at 20 °C

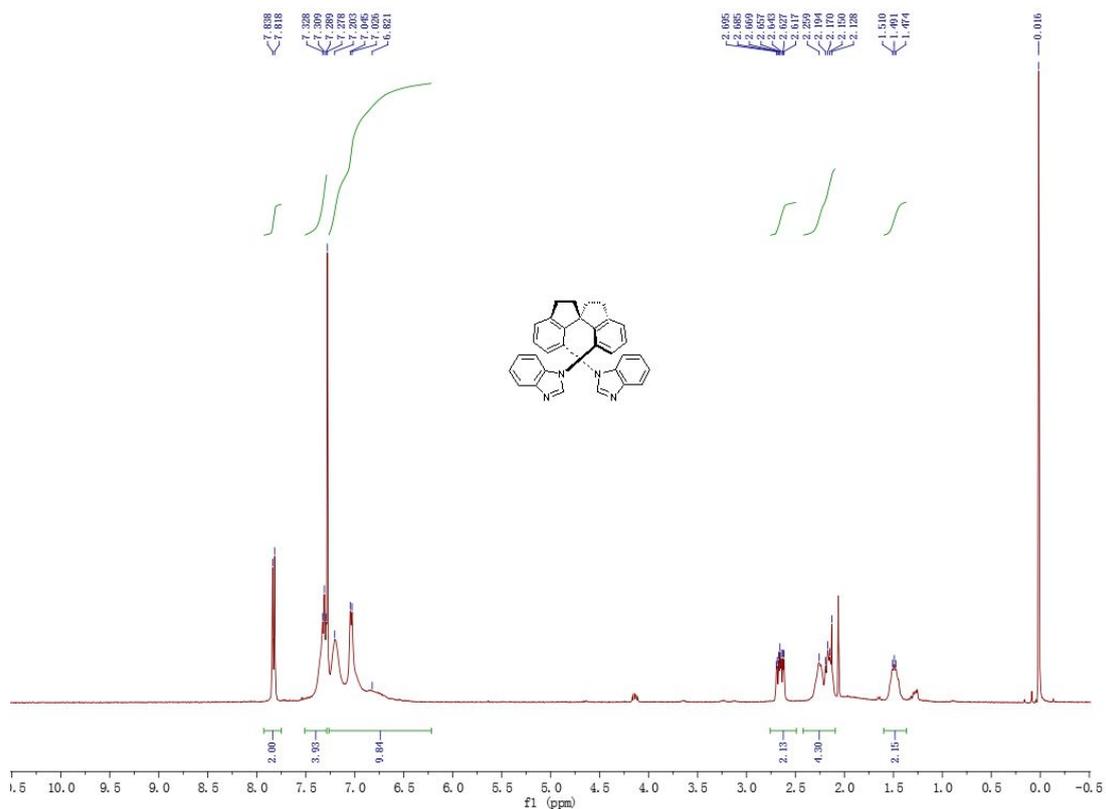


Figure S5. The ¹H NMR spectrum of (S)-7 in CDCl₃ at 20 °C

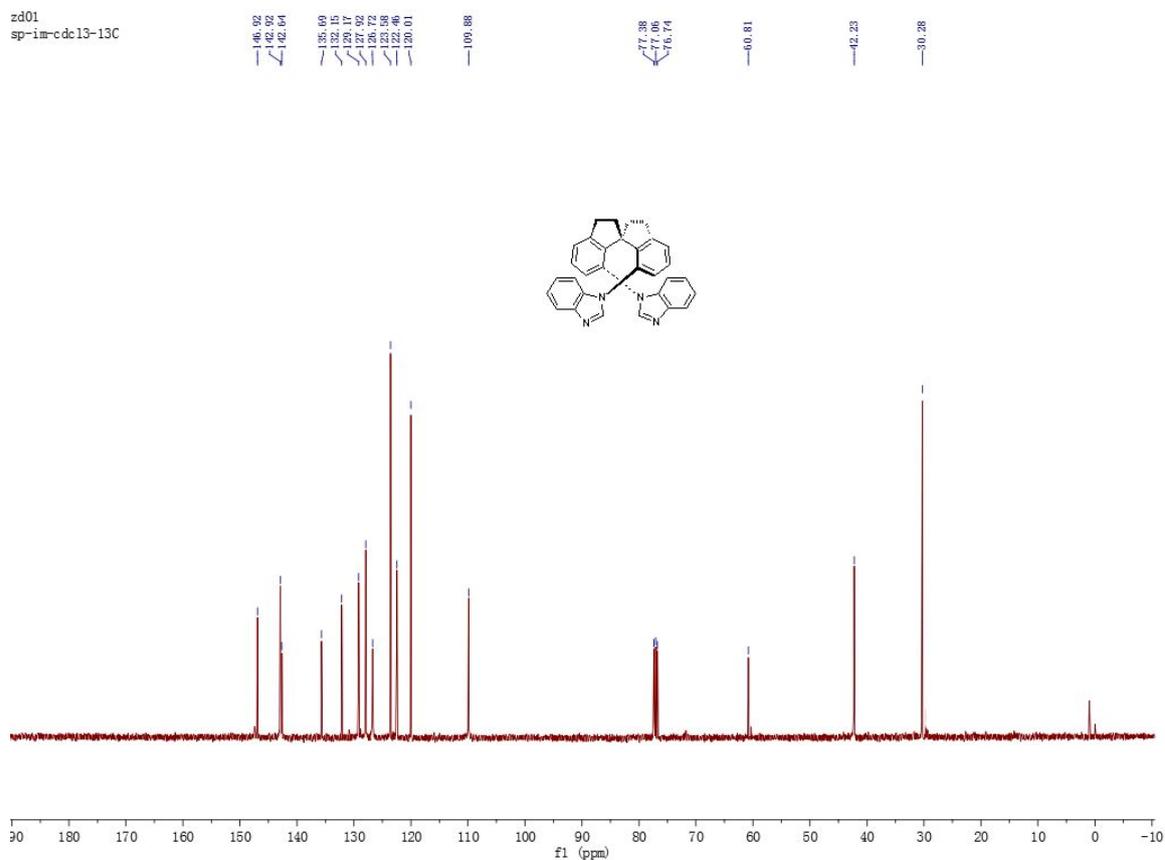


Figure S6. The ¹³C NMR spectrum of (S)-7 in CDCl₃ at 20 °C

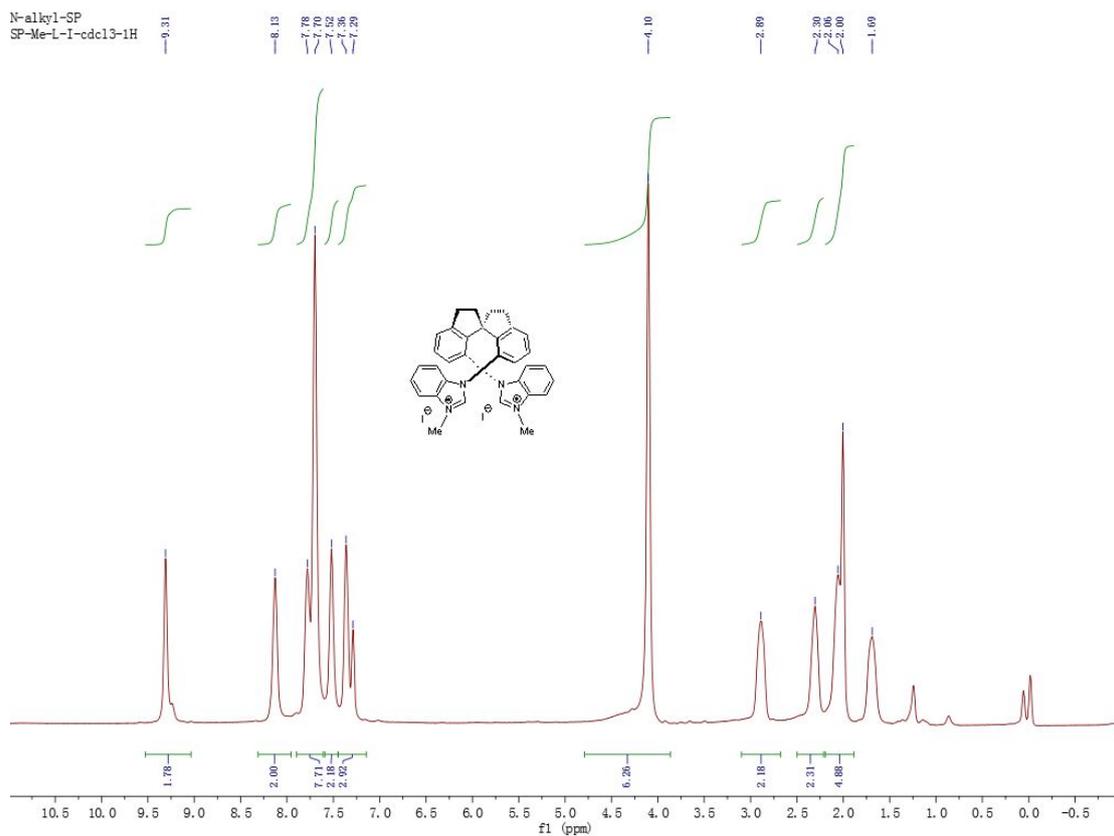


Figure S7. The ^1H NMR spectrum of $\text{H}_2[(\text{S})\text{-1a}]_2$ in CDCl_3 at $20\text{ }^\circ\text{C}$

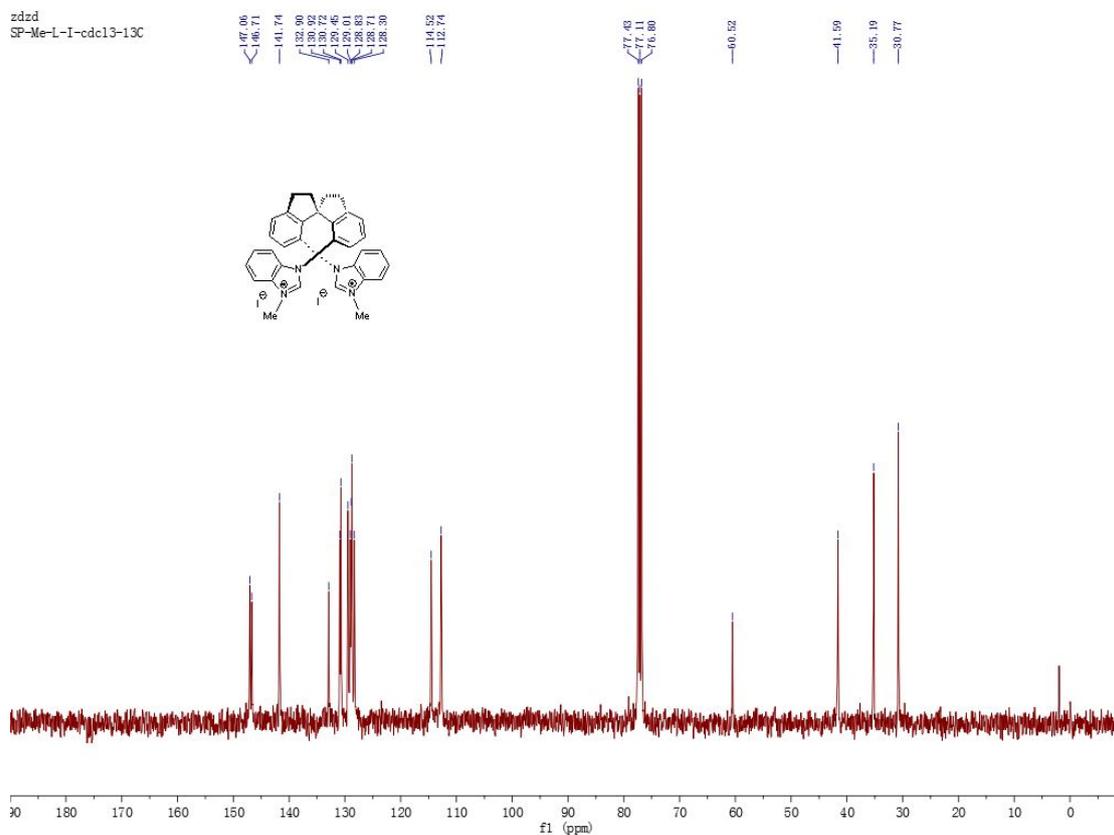


Figure S8. The ^{13}C NMR spectrum of $\text{H}_2[(\text{S})\text{-1a}]_2$ in CDCl_3 at $20\text{ }^\circ\text{C}$

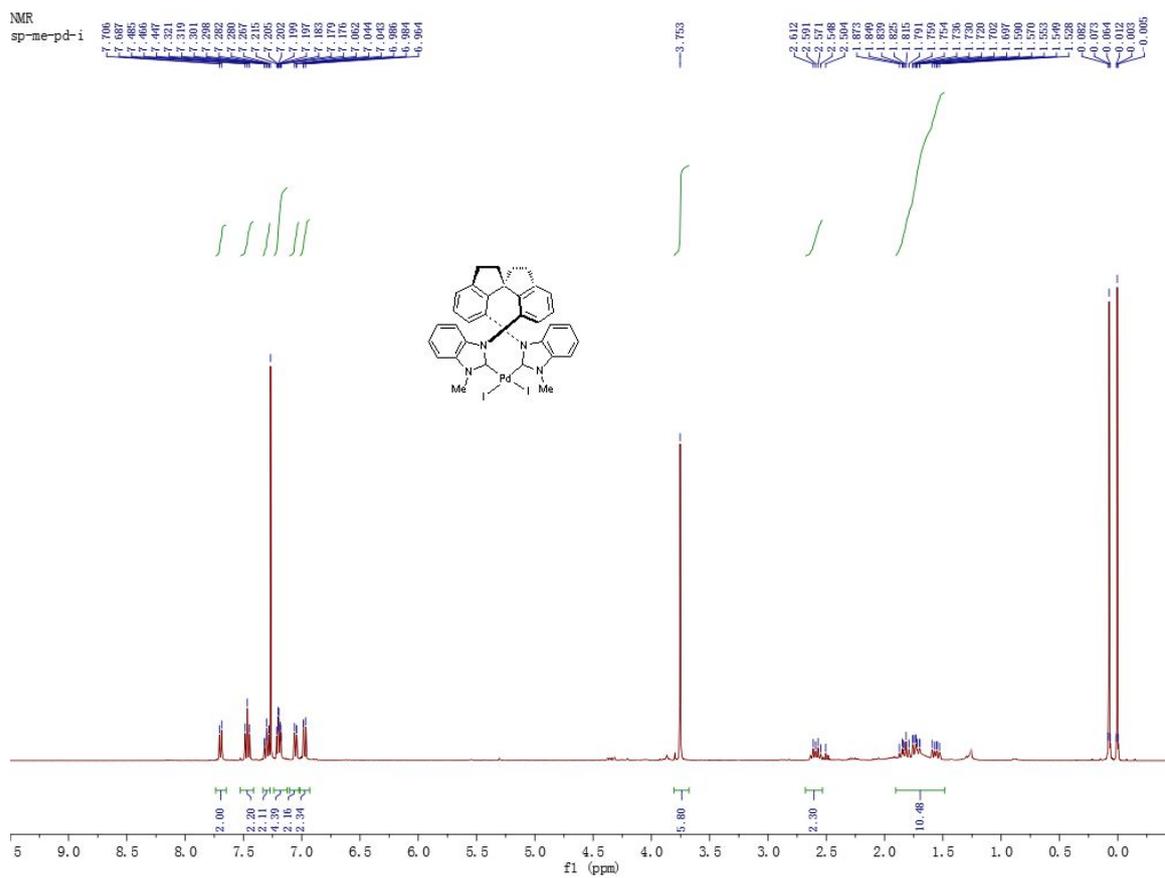


Figure S9. The ^1H NMR spectrum of (*S*)-2a in CDCl_3 at 20 °C

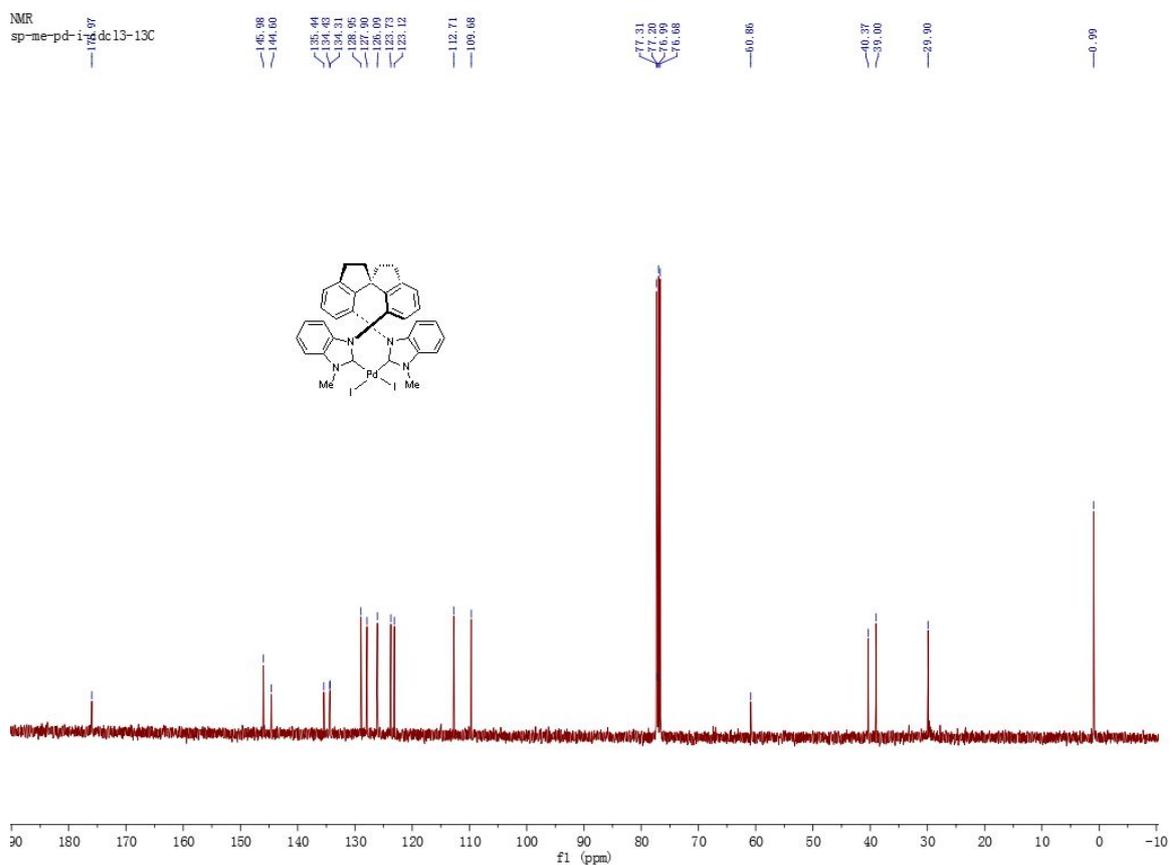


Figure S10. The ^{13}C NMR spectrum of (*S*)-2a in CDCl_3 at 20 °C

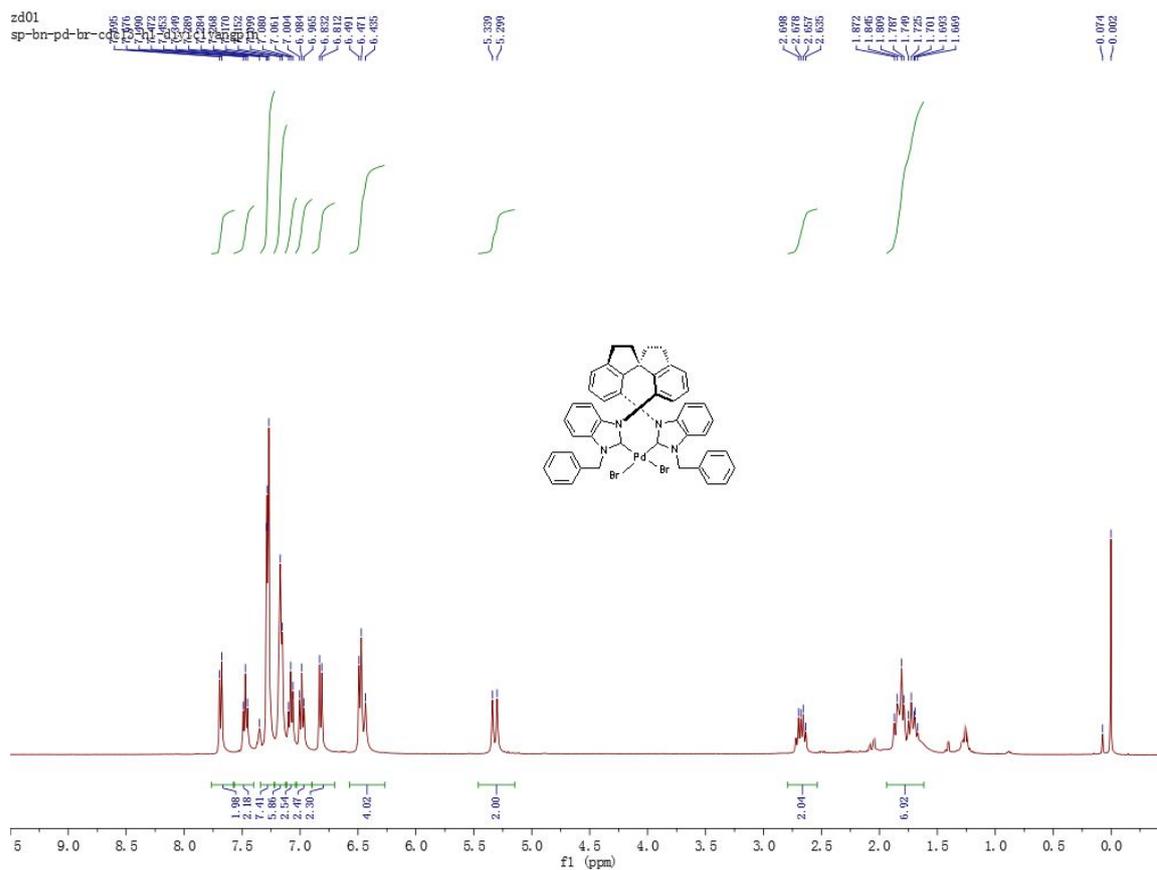


Figure S11. The ^1H NMR spectrum of (S)-2b in CDCl_3 at 20 °C

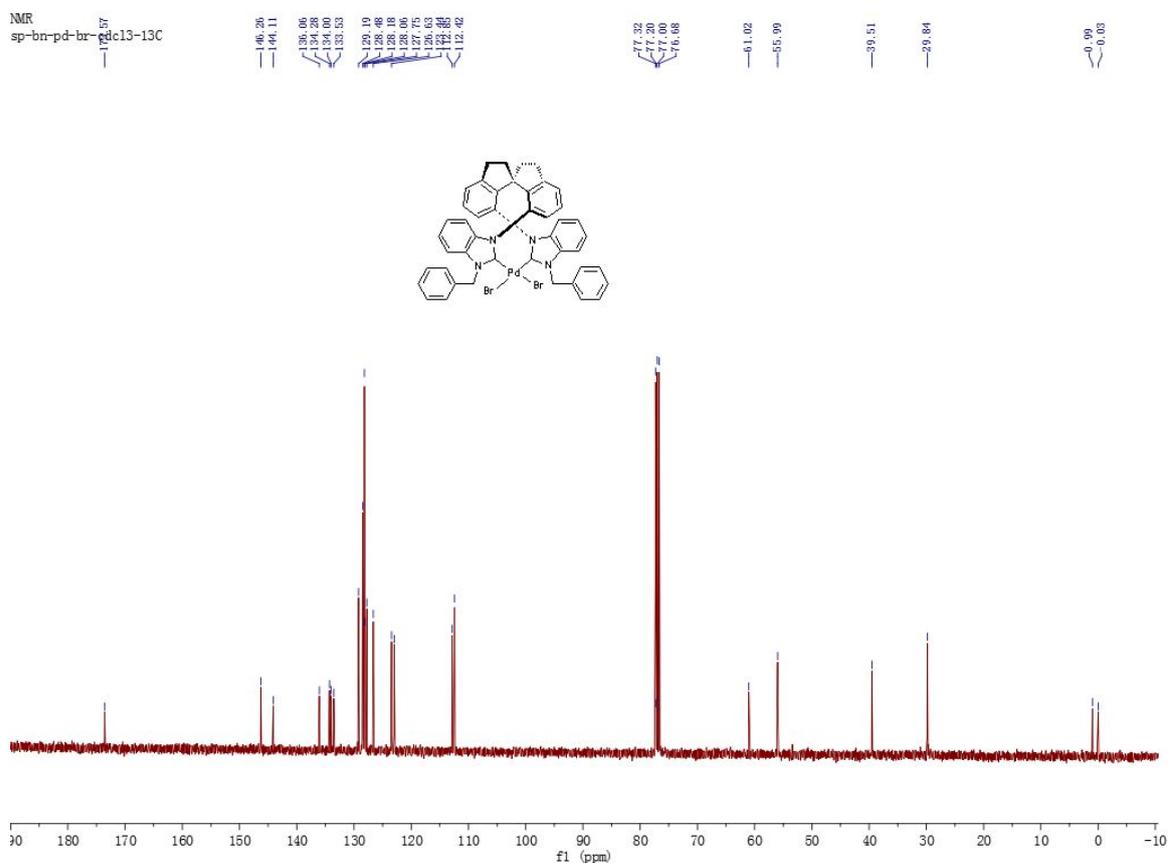


Figure S12. The ^{13}C NMR spectrum of (S)-2b in CDCl_3 at 20 °C

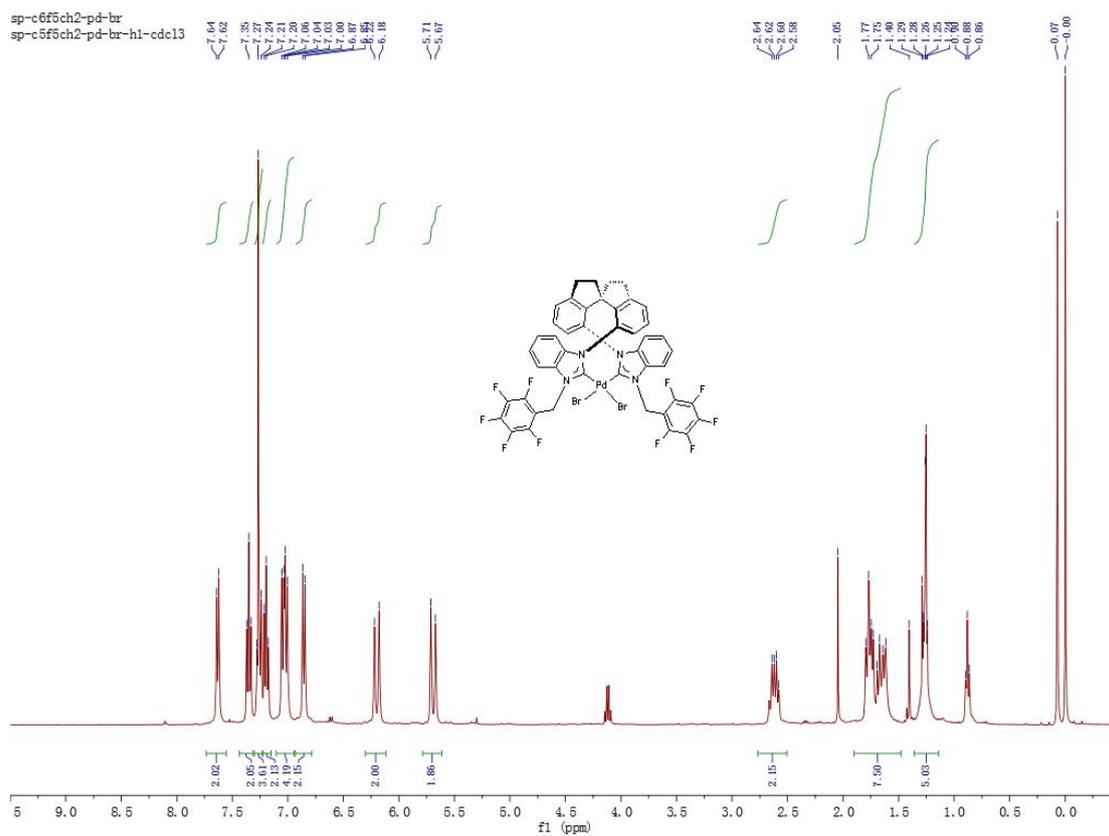


Figure S13. The ^1H NMR spectrum of (*S*)-2c in CDCl_3 at 20 °C

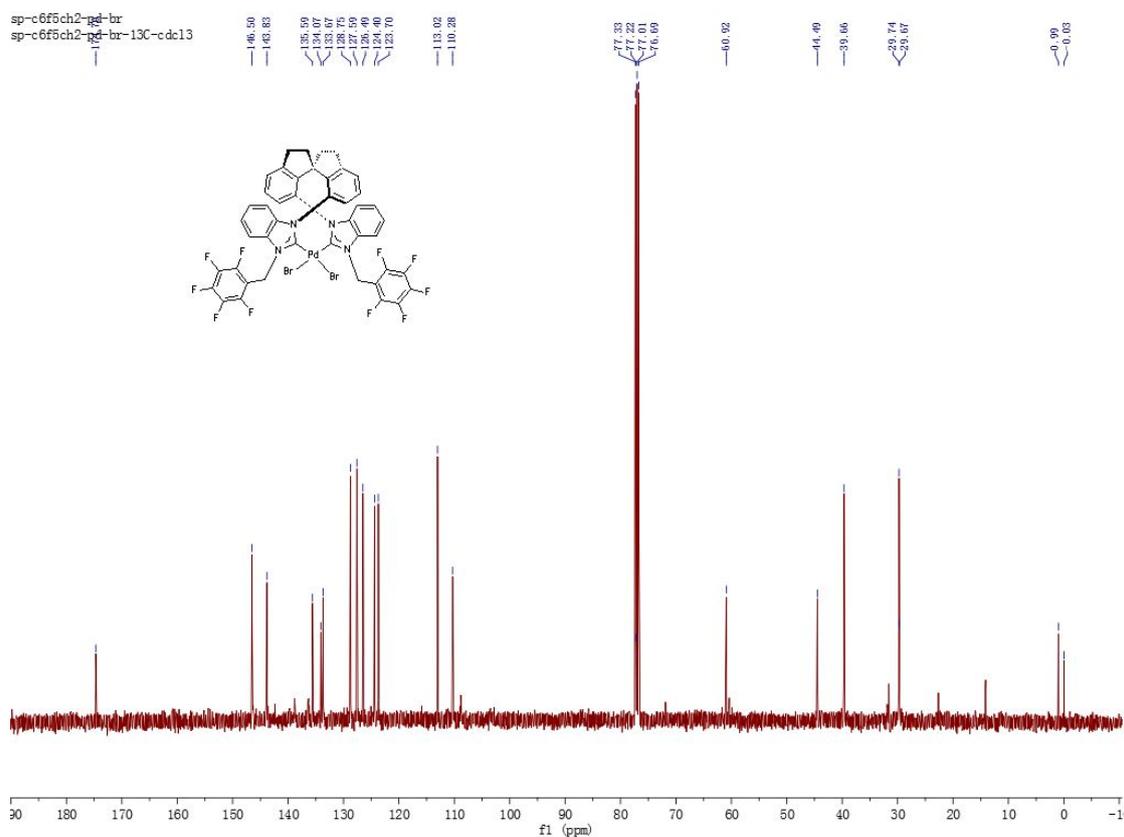


Figure S14. The ^{13}C NMR spectrum of (*S*)-2c in CDCl_3 at 20 °C

sp-c6f5ch2-pd-br
sp-c6f5ch2-pd-br-19F-cdcl3

137.71
137.71
137.77
137.79
153.83
153.83
153.94
161.25
161.27
161.32
161.36

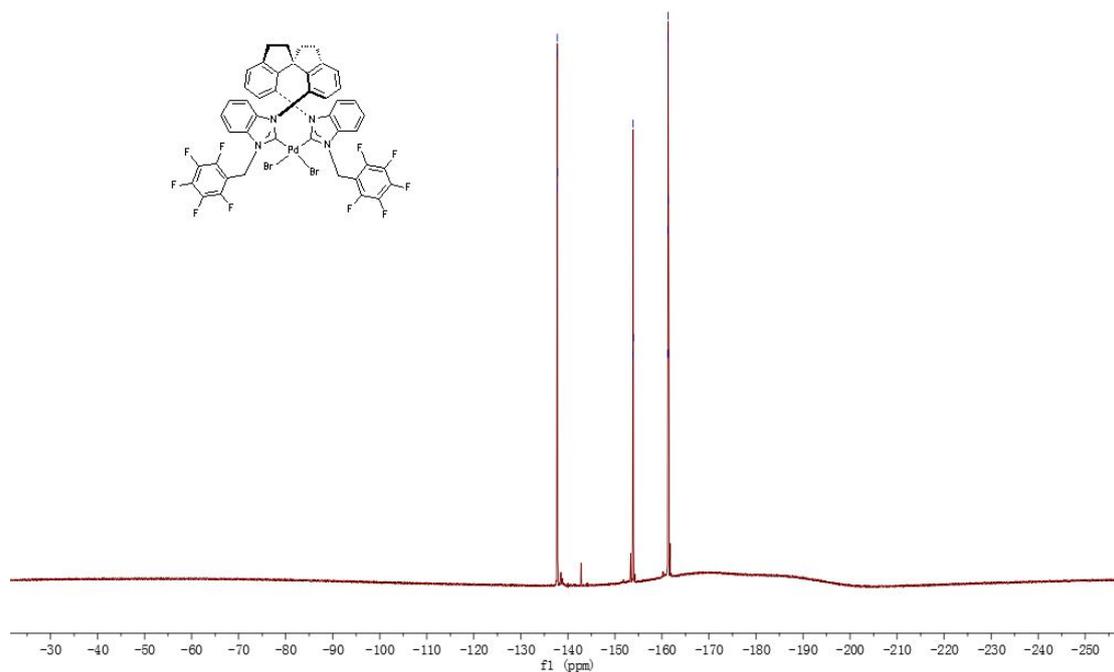


Figure S15. The ^{19}F NMR spectrum of (*S*)-**2c** in CDCl_3 at 20 °C

sp-2-cf3so3c6h4ch2-pd-br
sp-2-CF3SO3C6H4CH2-pd-br-1H-cdcl3

7.74
7.72
7.70
7.68
7.18
7.16
7.15
7.12
6.99
6.91
6.57

4.94
4.89

2.65
2.63
2.59
2.57
1.79
1.75
1.74
1.72
1.69
1.62
1.60
1.56
1.40
1.29
1.26
1.24

0.07
0.00

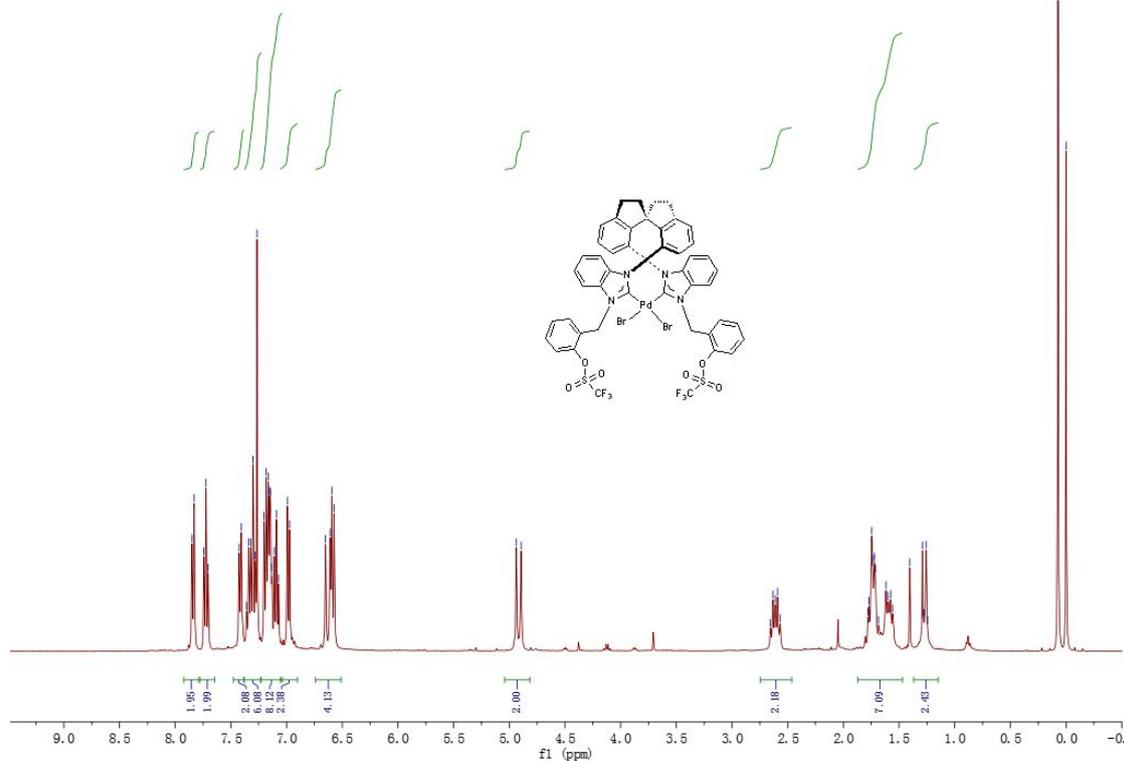
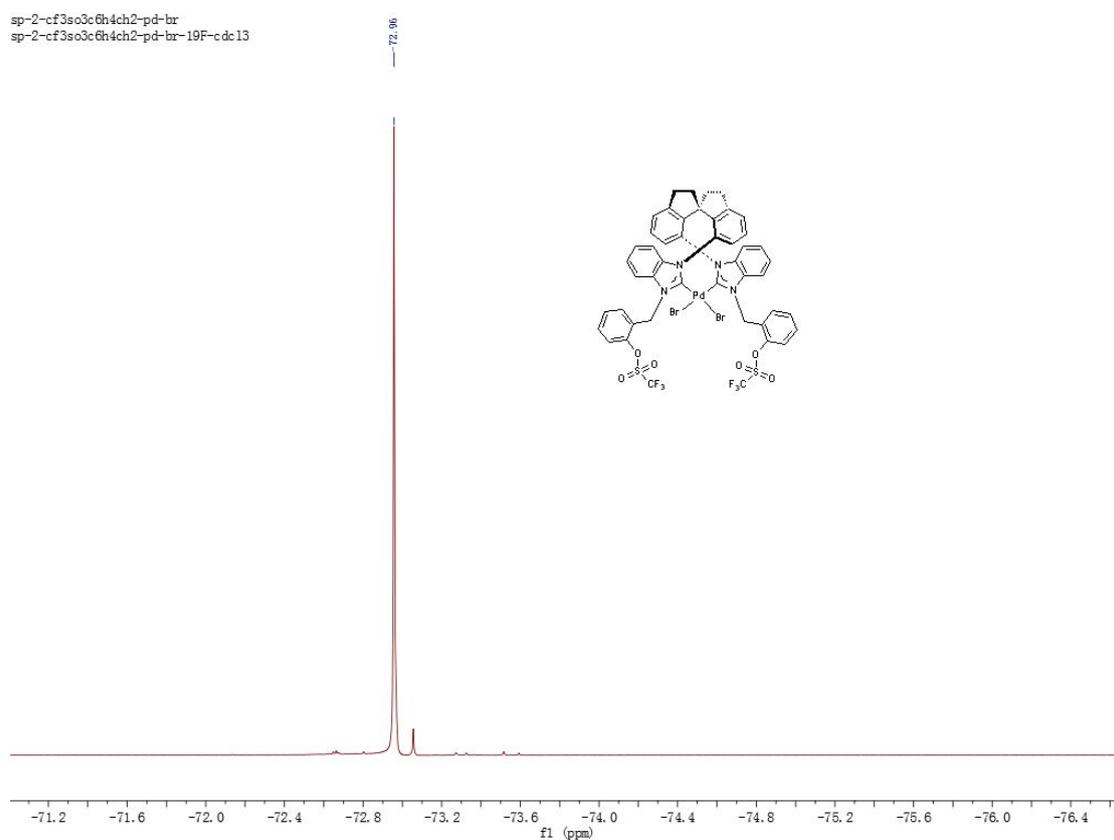
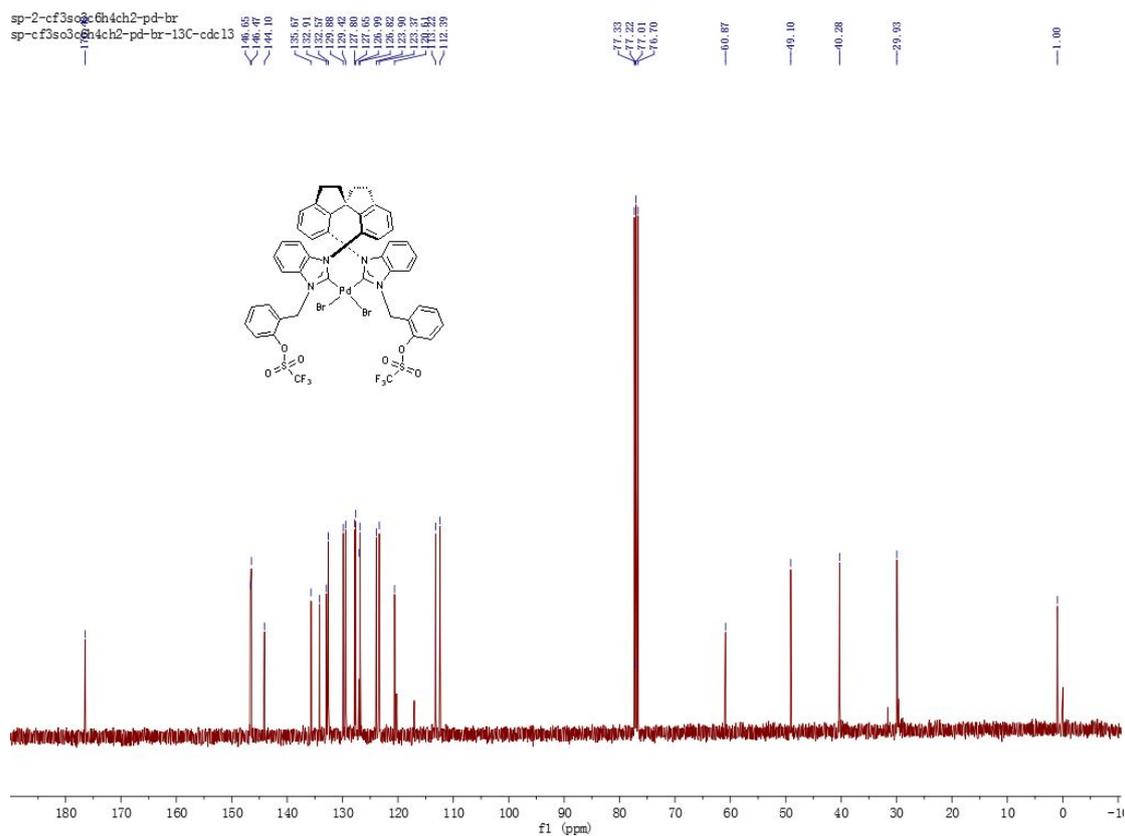


Figure S16. The ^1H NMR spectrum of (*S*)-**2d** in CDCl_3 at 20 °C



zhangdao

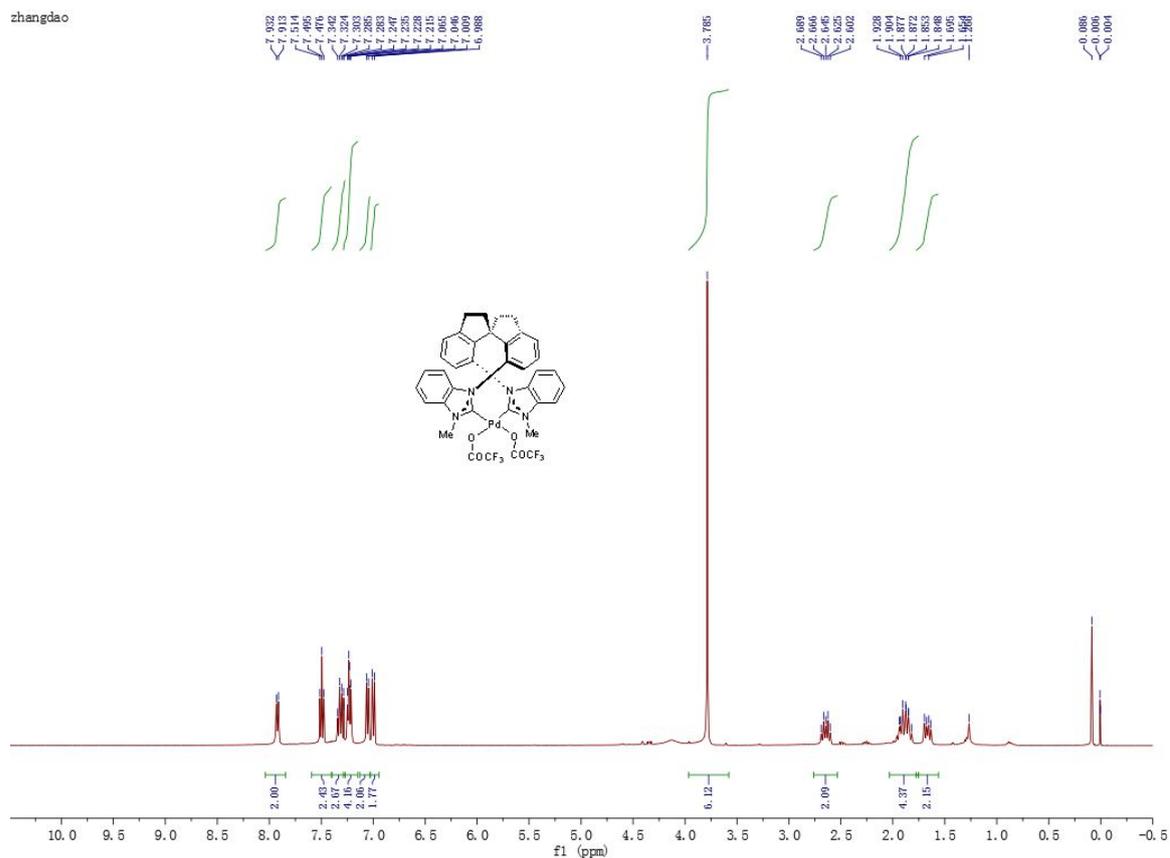


Figure S19. The ¹H NMR spectrum of (S)-3a in CDCl₃ at 20 °C

zhangdao

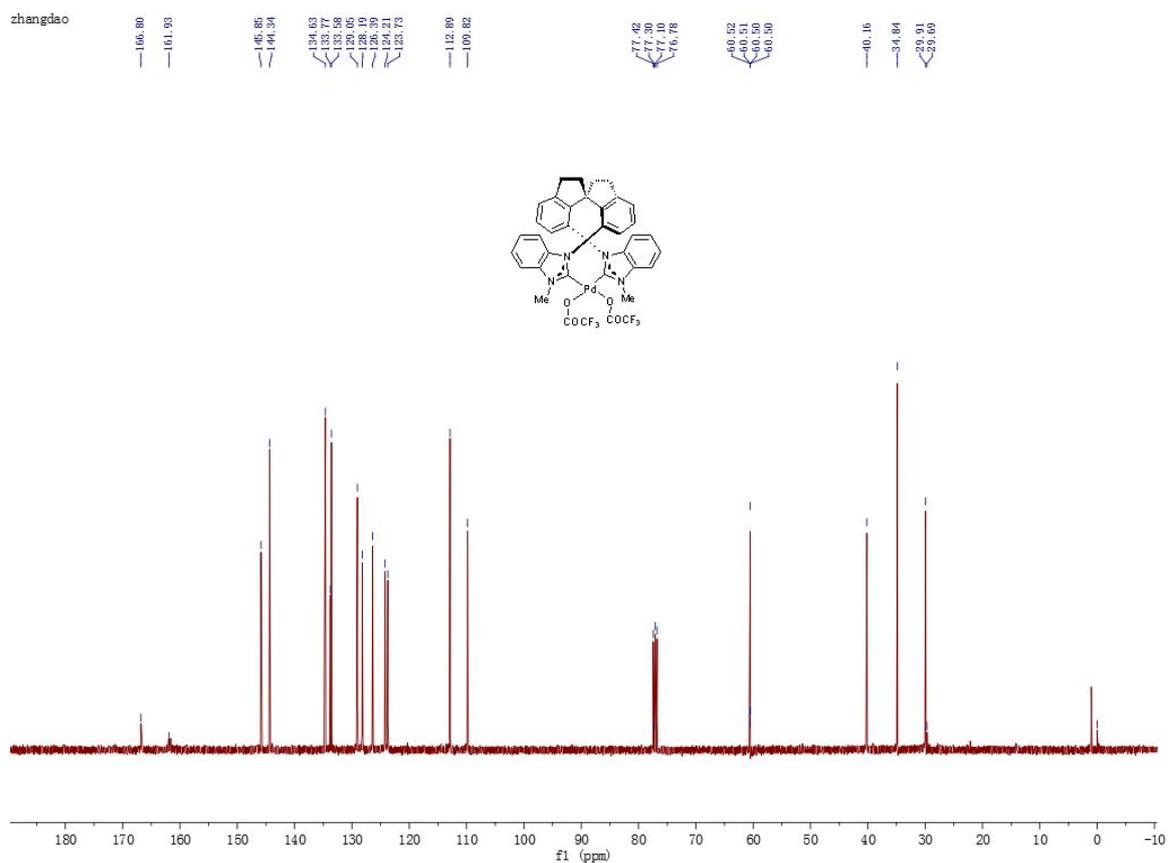


Figure S20. The ¹³C NMR spectrum of (S)-3a in CDCl₃ at 20 °C

zhangdao
sp-bn-pd-cdc13-h1

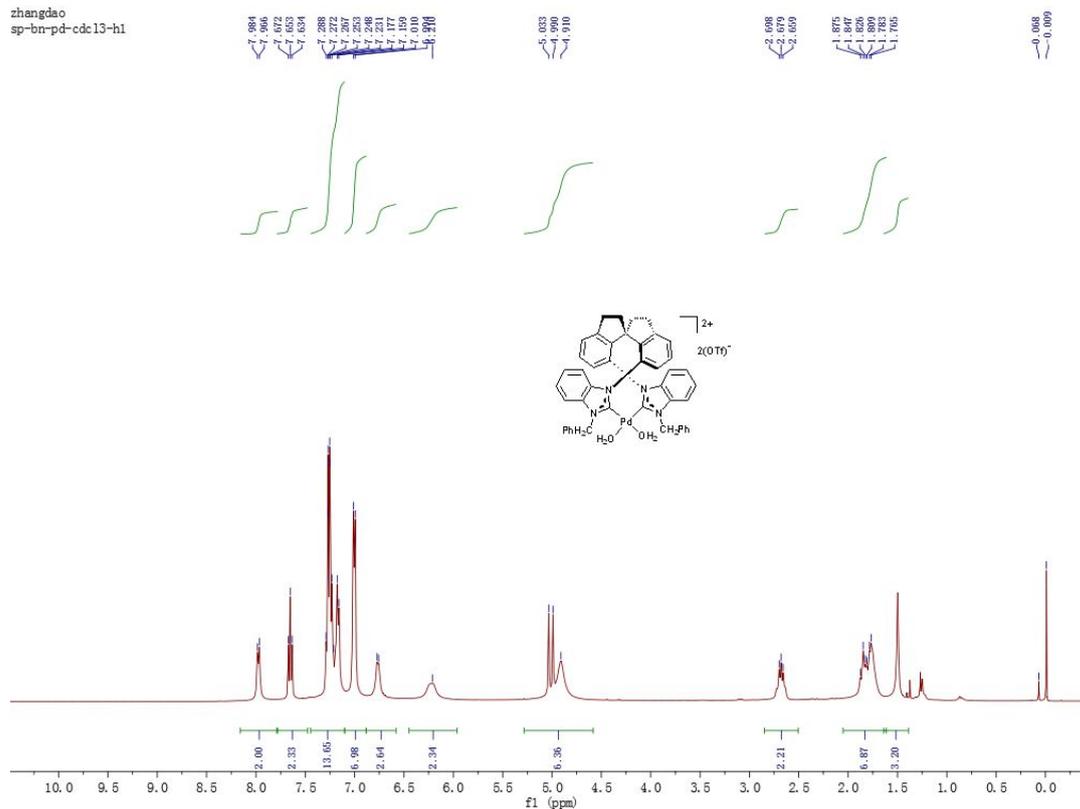


Figure S21. The ^1H NMR spectrum of (*S*)-**3b** in CDCl_3 at $20\text{ }^\circ\text{C}$

zhangdao
sp-bn-pd-13c-cdc13

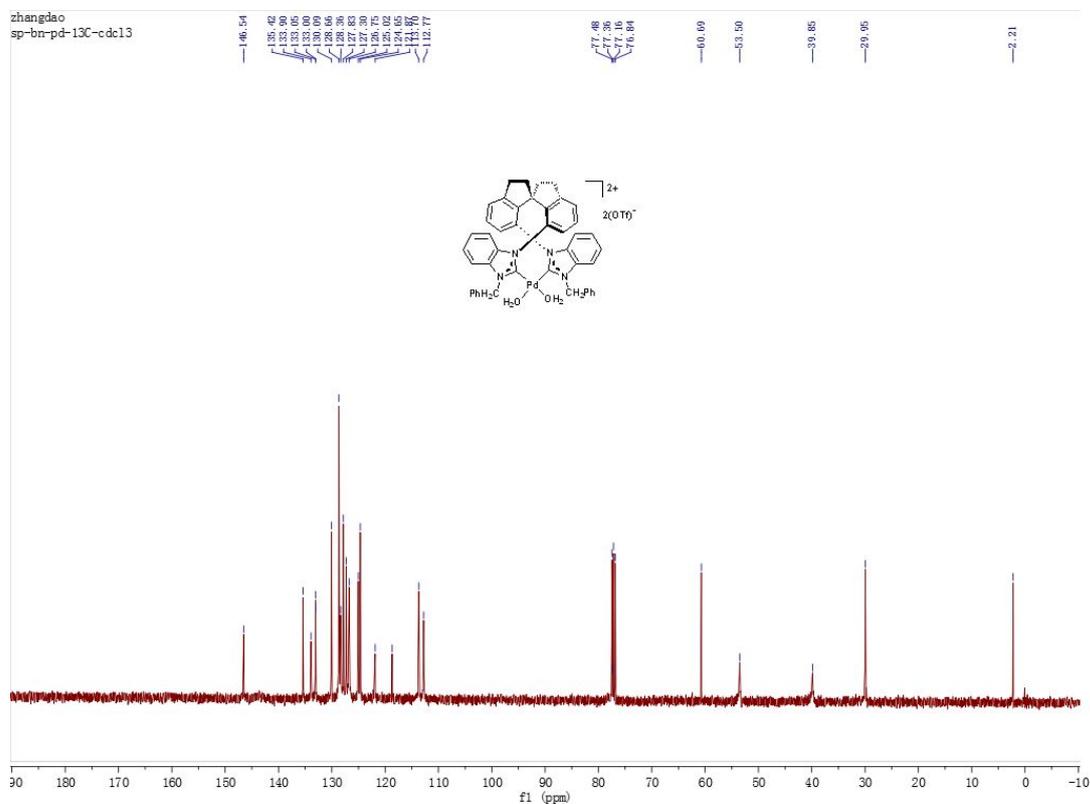


Figure S22. The ^{13}C NMR spectrum of (*S*)-**3b** in CDCl_3 at $20\text{ }^\circ\text{C}$

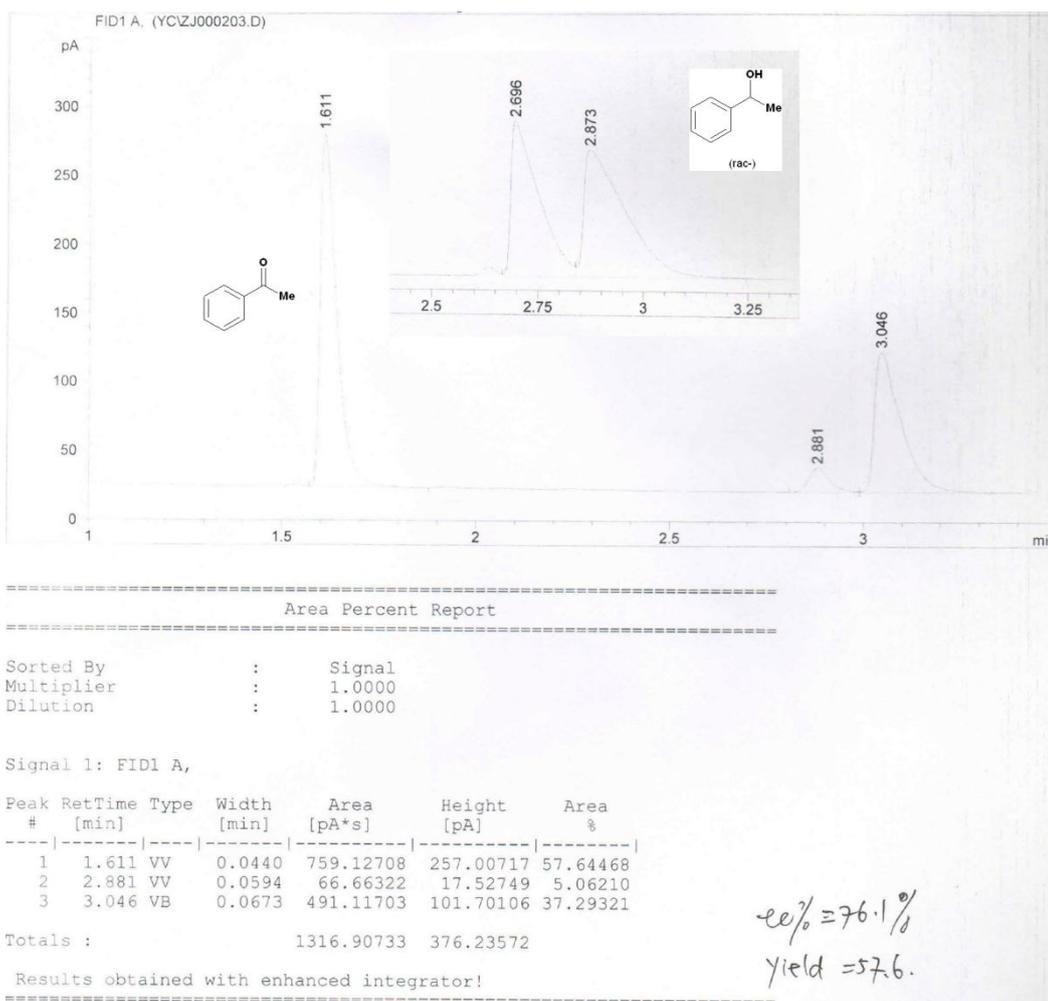
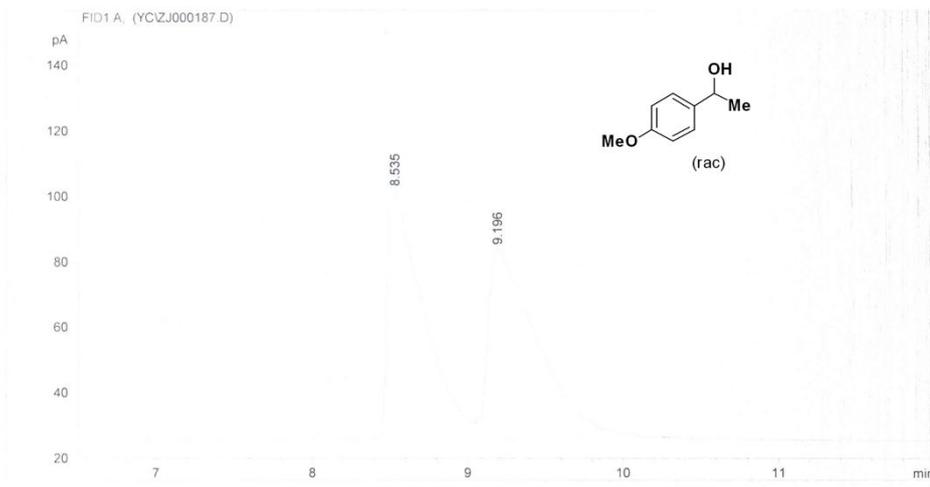


Figure S23. The GC curve and analysis data of **10a/11a**



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Area Percent Report
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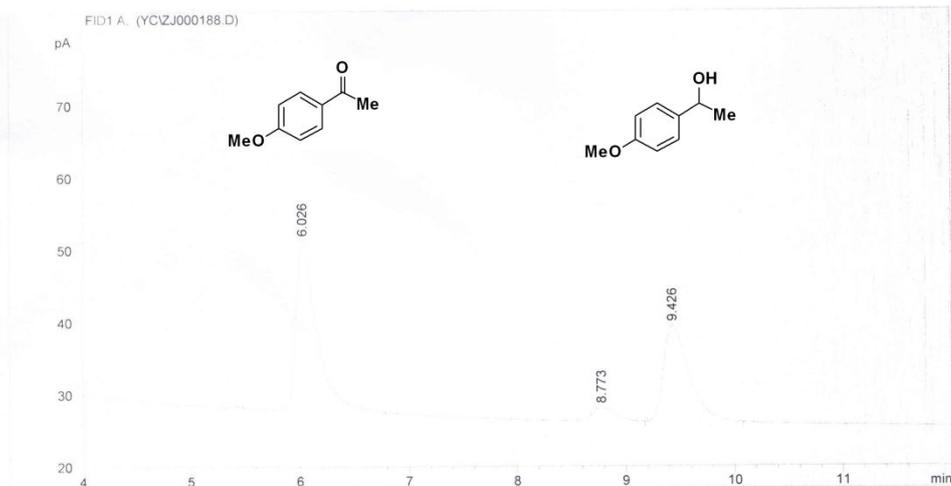
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	8.535	BV	0.2017	1206.12097	76.20535	48.37239
2	9.196	VB	0.2789	1287.28699	58.12667	51.62761

Totals : 2493.40796 134.33202

Results obtained with enhanced integrator!
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	6.026	VB	0.1660	318.71133	23.68497	56.79882
2	8.773	PP	0.1694	27.11762	1.93049	4.83274
3	9.426	BB	0.1917	215.29419	13.34763	38.36844

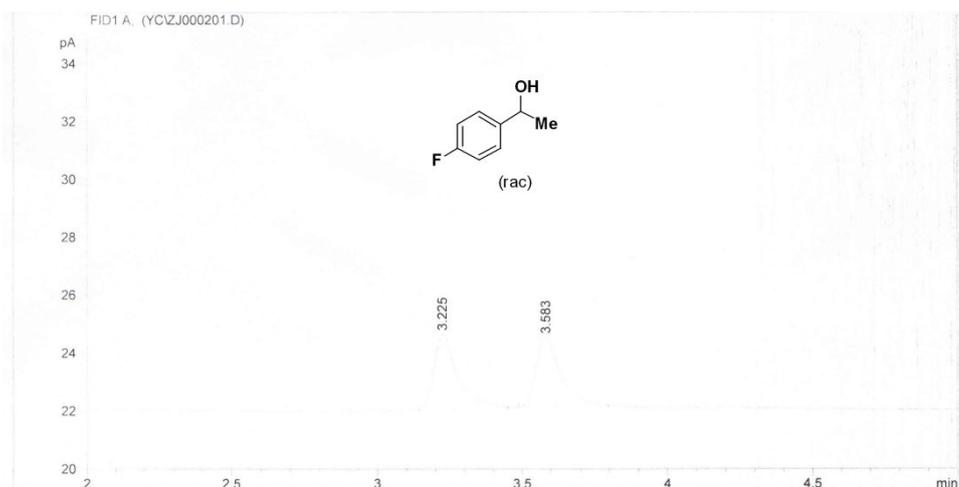
Totals : 561.12314 38.96309

Results obtained with enhanced integrator!
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$$ee\% = \frac{33.53}{43.19} \times 100\% = 77.6\%$$

$$Yield = 56.8\%$$

Figure S24. The GC curve and analysis data of 10b/11b



Area Percent Report

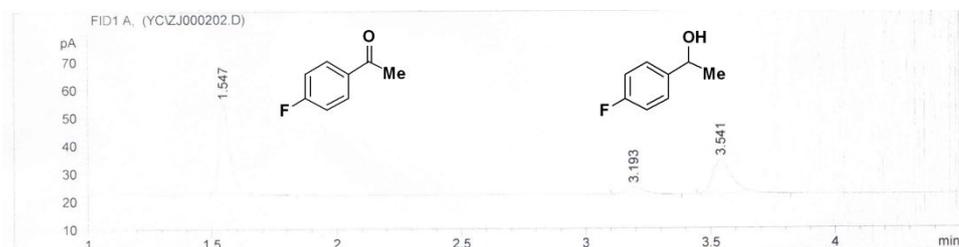
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	3.225	PB	0.0779	14.47087	2.60088	50.50811
2	3.583	PB	0.0853	14.17971	2.38573	49.49189

Totals : 28.65058 4.98661

Results obtained with enhanced integrator!



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	1.547	PB	0.0474	102.51325	32.81509	55.87810
2	3.193	BB	0.0737	13.25990	2.59030	7.22773
3	3.541	BB	0.0816	67.68559	12.12094	36.89418

Totals : 183.45874 47.52633

Results obtained with enhanced integrator!

ee% = 67.2%

Yield = 55.9%

Figure S25. The GC curve and analysis data of 10c/11c

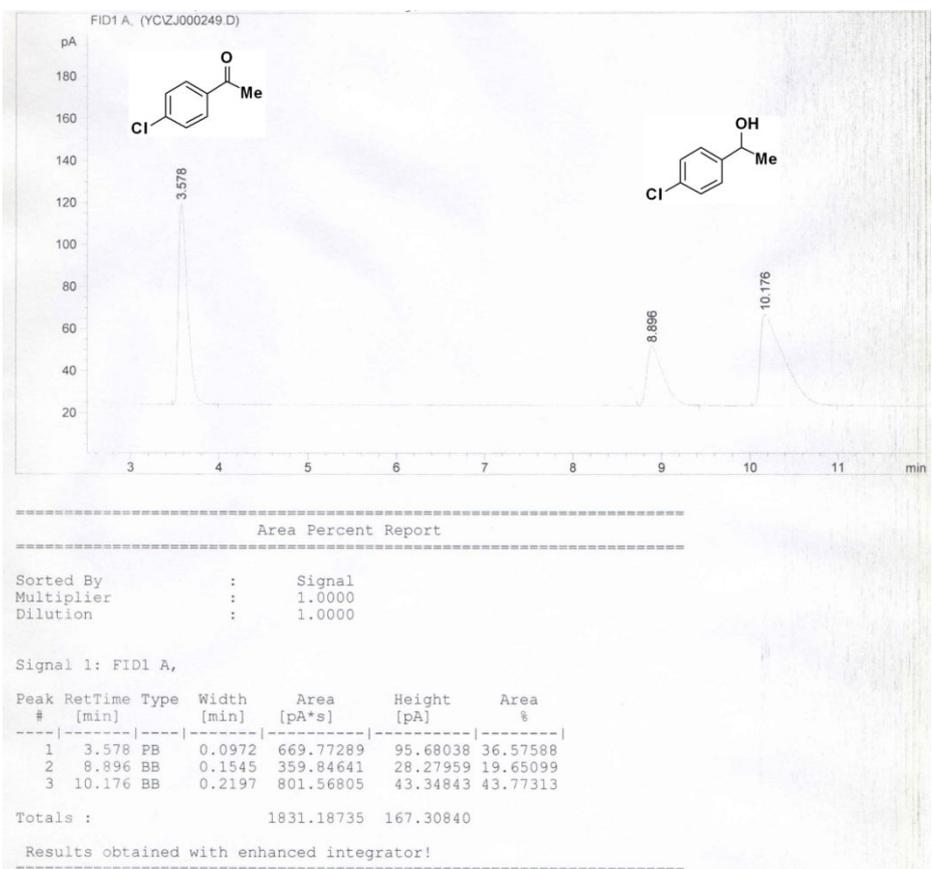
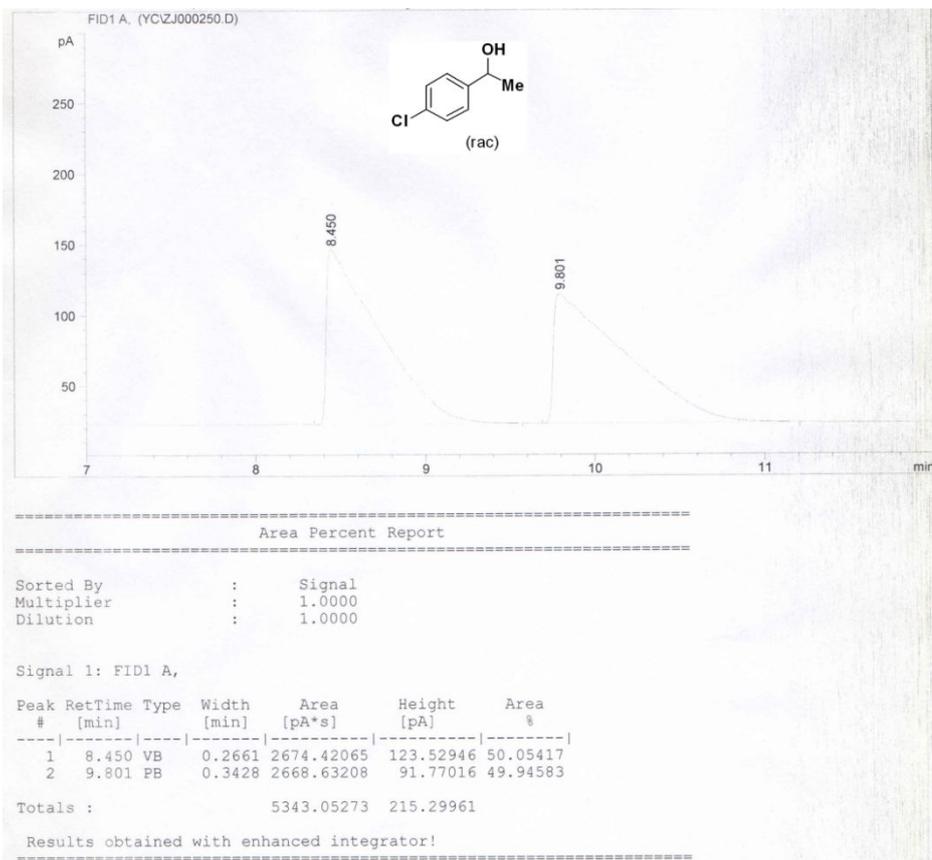


Figure S26. The GC curve and analysis data of 10d/11d

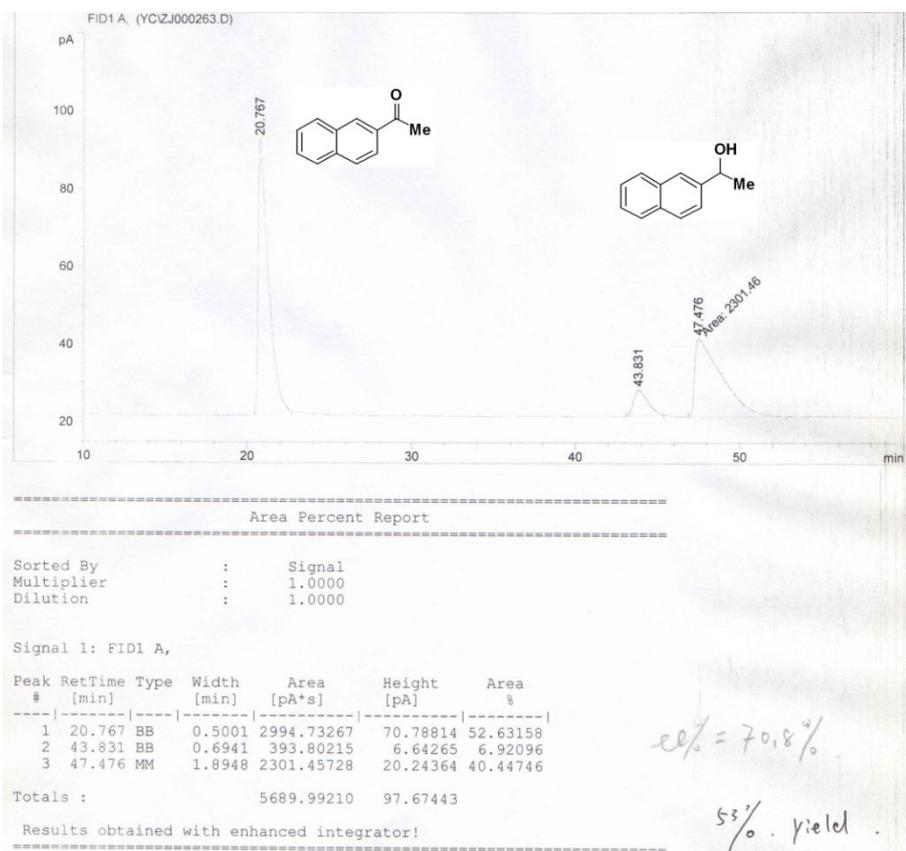
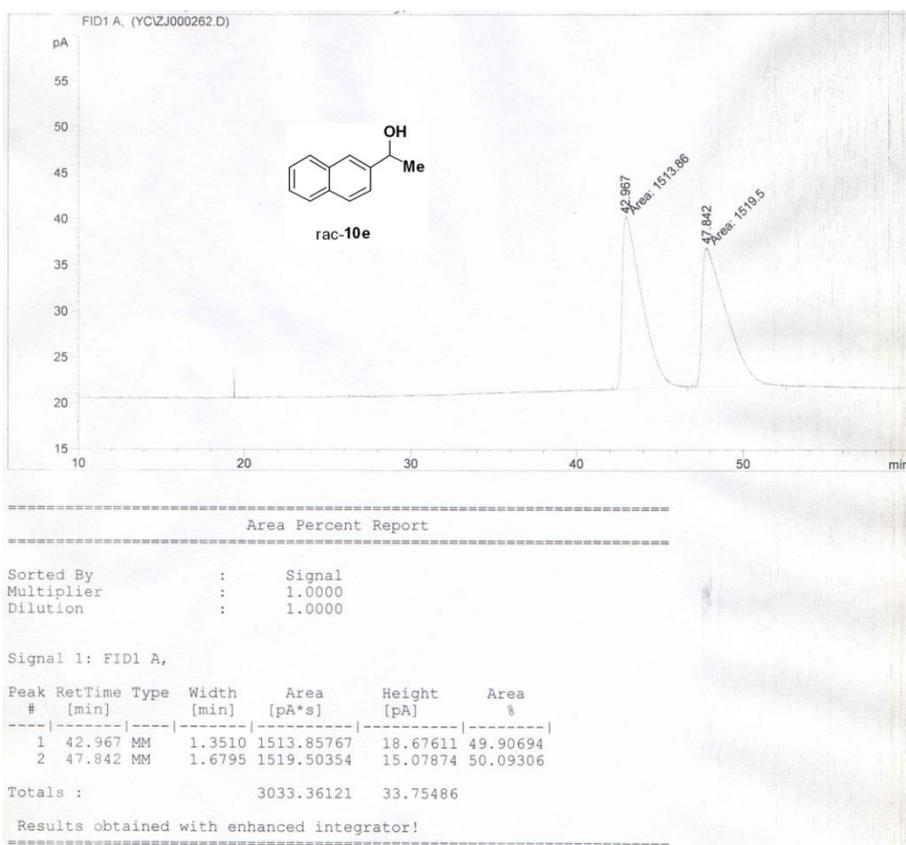
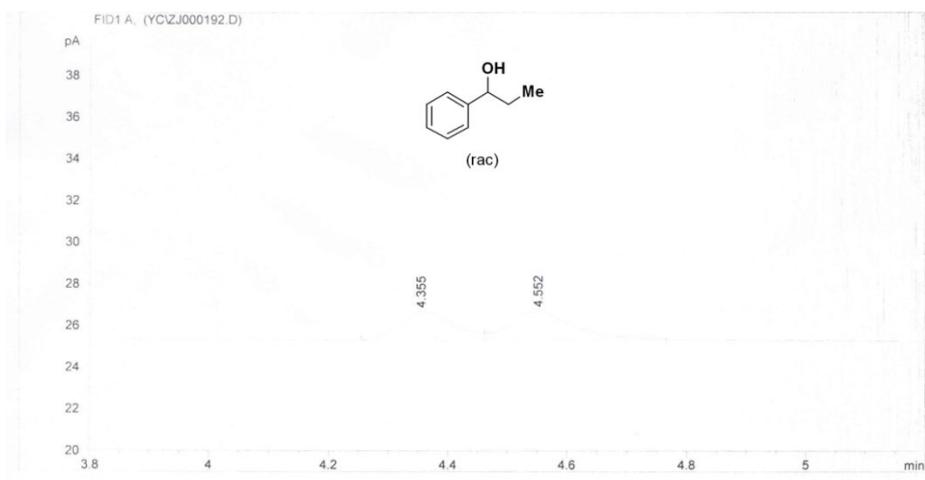


Figure S27. The GC curve and analysis data of 10e/11e



Area Percent Report

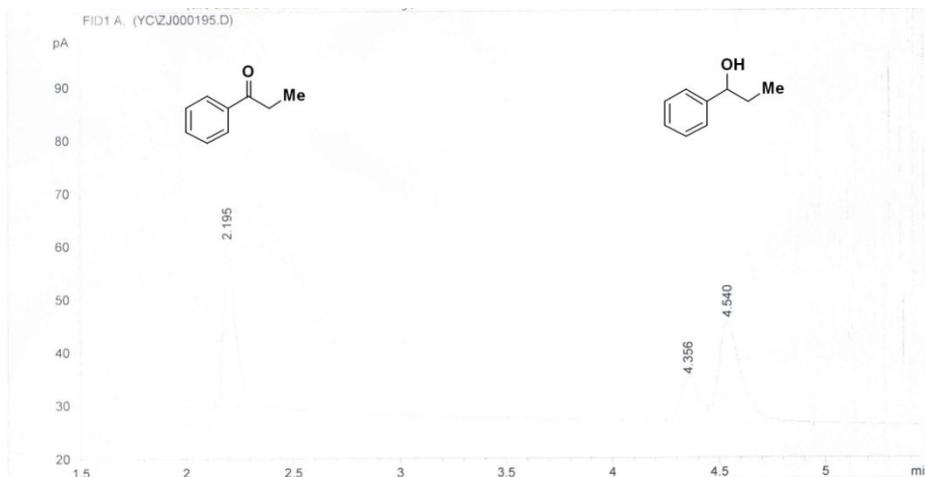
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.355	PV	0.0829	8.40319	1.36278	45.91667
2	4.552	VB	0.0911	9.89777	1.38916	54.08333

Totals : 18.30096 2.75194

Results obtained with enhanced integrator!



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	2.195	PP	0.0633	130.49976	30.89897	41.15000
2	4.356	BV	0.0896	48.27827	8.14625	15.22341
3	4.540	VB	0.0991	138.35381	18.86649	43.62659

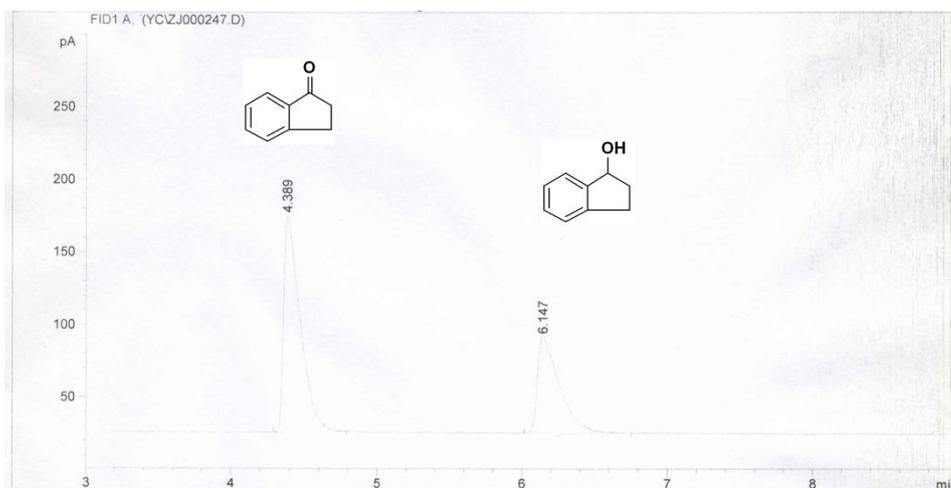
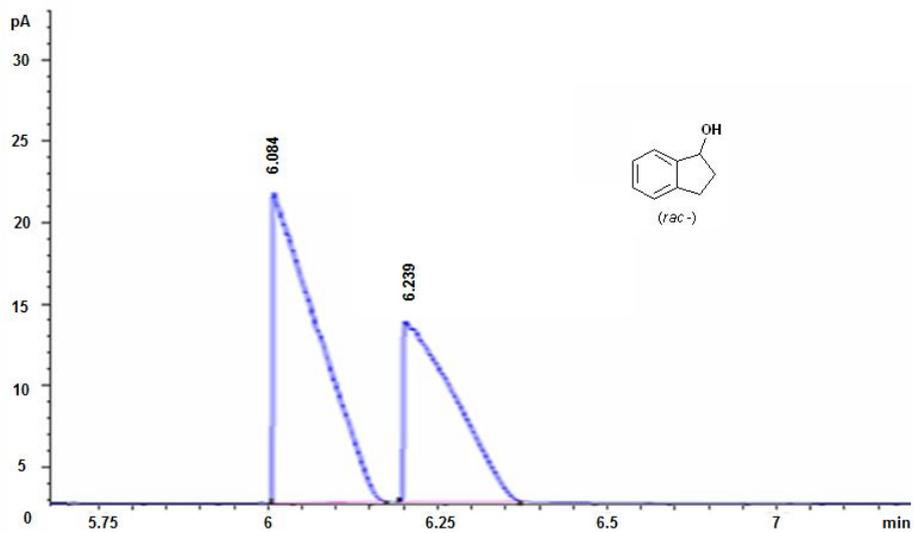
Totals : 317.13184 57.91171

Results obtained with enhanced integrator!

$$ee\% = \frac{28.40}{58.84} \times 100\% = 48.1\%$$

$$yield = 41.2\%$$

Figure S28. The GC curve and analysis data of 10f/11f



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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

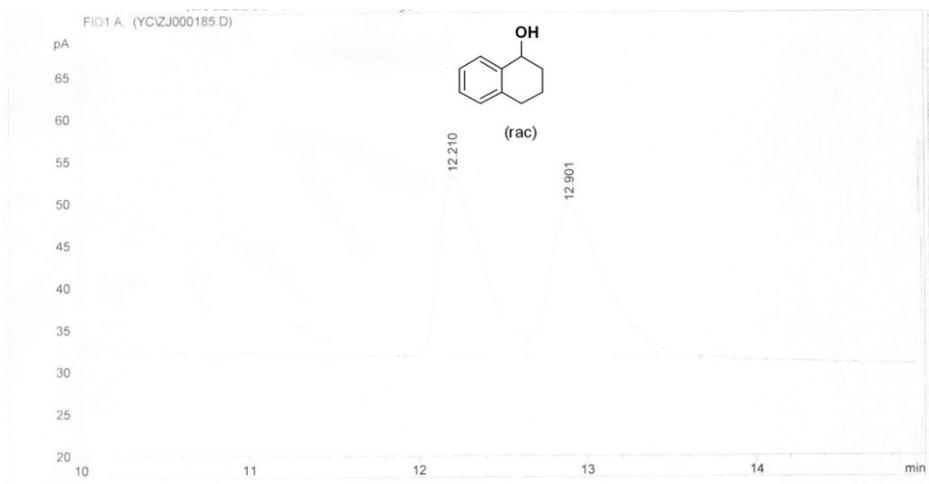
Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	4.389	PB	0.1071	1159.48413	148.54245	62.48375
2	6.147	BB	0.1383	696.17303	65.88248	37.51625

Totals : 1855.65717 214.42493

Results obtained with enhanced integrator!
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Figure S29. The GC curve and analysis data of **10g/11g**



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 Area Percent Report
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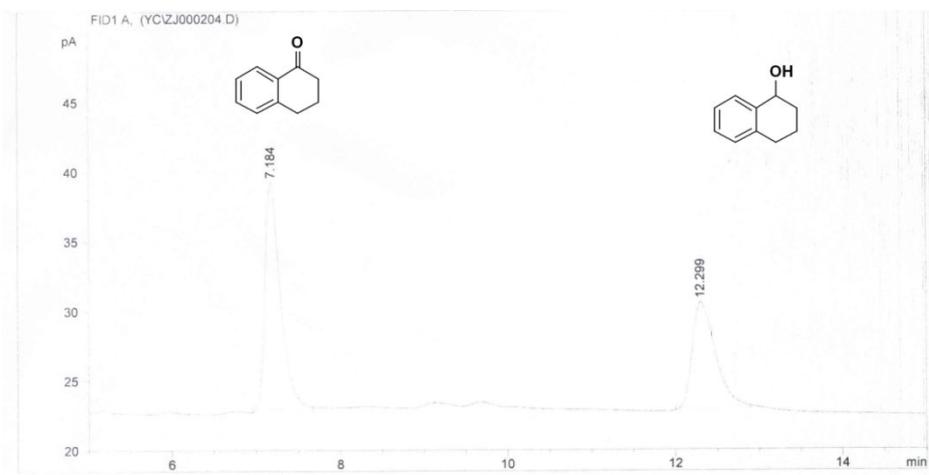
Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	12.210	BV	0.2163	388.26498	21.43347	48.89441
2	12.901	VB	0.2681	405.82370	18.03023	51.10559

Totals : 794.08868 39.46370

Results obtained with enhanced integrator!
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 Area Percent Report
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Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	7.184	BB	0.1498	206.55675	16.41781	58.41214
2	12.299	BB	0.2317	147.06277	7.67030	41.58786

Totals : 353.61952 24.08811

Results obtained with enhanced integrator!
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Figure S30. The GC curve and analysis data of 10h/11h