Supporting Information

Resin-Supported Peptide-Catalyzed Asymmetric Aldol Reaction of Aldehydes for the Synthesis of (*R*)-Pantolactone

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1. General Experimental Details

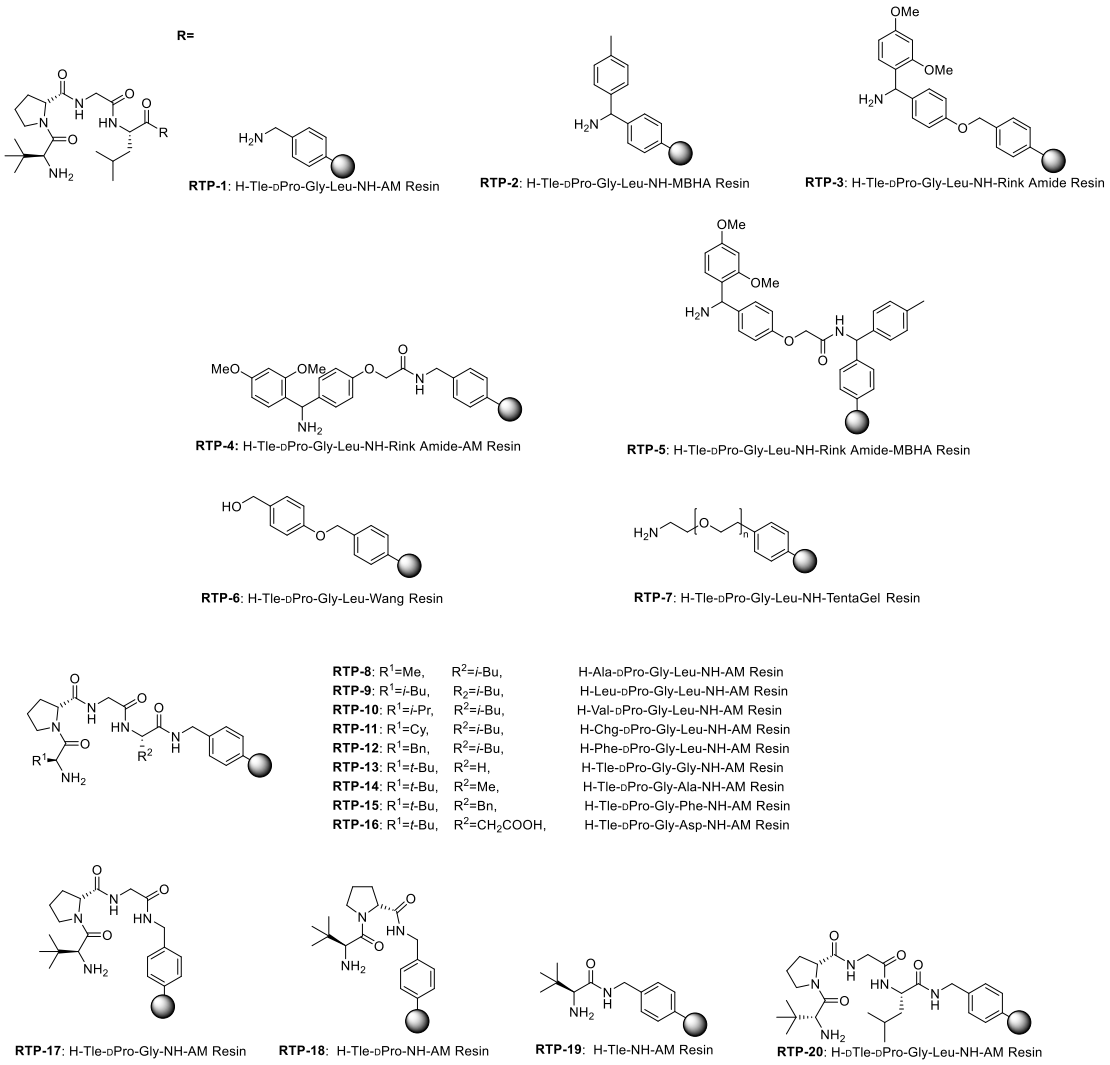
The reactions were carried out in vials and stirred with a magnetic bar without inert atmosphere unless specified. All commercial reagents were purchased with the analysis purity grade. They were used without further purification unless specified. All solvents used, mainly petroleum ether (PE) and ethyl acetate (EtOAc) were distilled. Anhydrous DCM and CH_3CN were freshly distilled from CaH_2, THF, Et_2O and toluene freshly distilled from sodium/benzophenone before use. Anhydrous methanol and ethanol were distilled from Mg.

The reactions were monitored by TLC (thin layer chromatography) method; column and preparative TLC purifications were carried out using silica gel. Melting points were uncorrected and recorded on XT-5 melting point apparatus.

NMR spectra were acquired on a Bruker 400/600 spectrometer, running at 400/600 MHz and 100/151 MHz for ^1H and ^{13}C, respectively. NMR in $CDCl_3$, D_2O, DMSO-d_6 with TMS as an internal standard, chemical shifts (δ) are reported in ppm relative to residual solvent signals ($CDCl_3$, 7.26 ppm for ^1H NMR and 77.00 ppm for ^{13}C NMR; D_2O, 4.80 ppm for ^1H NMR; DMSO-d_6, 2.50 ppm for ^1H NMR, 40.00 ppm for ^{13}C NMR). The following abbreviations are used to describe peak patterns when appropriate: s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sept (septuplet), m(multiplet), br (broad). High Resolution Mass spectra (HR-MS) were measured with ESI-Orbitrap mass spectrometer. Enantiomeric excess (ee) was decided with chiral HPLC, Waters 1525 Binary HPLC Pump/Waters 2998 Photodiode Array Detector of Lanzhou University State Key Laboratory of Applied Organic Chemistry.

All products in this text, if not stated otherwise, are oily liquids.

2. Preparation and Characterization of Peptide Catalysts



2.1 Preparation of Peptide Catalysts

Resin-supported peptides were synthesized by the standard method of the Fmoc solid-phase peptide synthesis.[1] As resins, Aminomethyl Polystyrene Resin, MBHA Resin, Fmoc-Rink Amide Resin, Fmoc-Rink Amide-AM Resin, Fmoc-Rink Amide-MBHA Resin, Wang Resin and TentaGel MB-NH_2 Resin were used. The coupling reaction of an amino acid was performed with 2.5 equiv each of an N-α-9-fluorenylmethoxycarbonyl (Fmoc) amino acid, O-(7-benzotriazol-1-yl)-N,N,N′,N′-tetramethyluronium hexafluorophosphate (HBTU), and 1-hydroxy-7-benzotriazole (HOBt) along with 5.0 equiv of diisopropylethylamine (DIPEA) in N, N-dimethylformamide (DMF) for 60 min. After washing the resin with DMF, completion of the peptide bond formation was confirmed by the chloranil test. To remove the Fmoc group,

the resin was soaked in 20% piperidine/DMF solution for 15 min and washed with DMF. This cycle, the coupling of an Fmoc-protected amino acid and removal of the Fmoc group, was repeated until an intended sequence was introduced on the resin. After the Fmoc group on the terminal amino acid residue was removed, the resin was washed with DMF and DCM, and dried under reduced pressure.

2.2 Resin loadings determination of resin-supported peptides

a) Establish a standard curve

Prepare 25 mL of a 2% DBU/DMF solution, referred to as solution D. Take 2 mL from solution D and transfer it to a 10 mL volumetric flask. Then, use acetonitrile to adjust the volume to 10 mL, forming solution E. Take 2 mL from solution E and transfer it to a 25 mL volumetric flask, then adjust the volume to 25 mL with acetonitrile to create solution F. Solution F serves as the blank solution for testing. Weigh 35.34 mg (0.1 mmol) of Fmoc-Leu-OH into a 10 mL round-bottom flask, add 5 mL of solution D, and stir for 30 minutes. Transfer the reaction solution to a 25 mL volumetric flask, wash it three times with DMF, and combine the washing solution in the same 25 mL volumetric flask, adjusting the volume to 25 mL using DMF to create the standard solution G. The standard solution was then diluted with acetonitrile to produce solutions of varying concentrations, and their absorbance was measured individually; the test results are presented in Table S1. Using concentration as the x-axis and absorbance as the y-axis, a standard curve was plotted, and the results are illustrated in Figure S1.

Table S1. Determining the absorbance of the standard curve

concentration (mmol/L)	0.025	0.05	0.08	0.1	0.2
absorbance	0.096	0.191	0.301	0.385	0.762

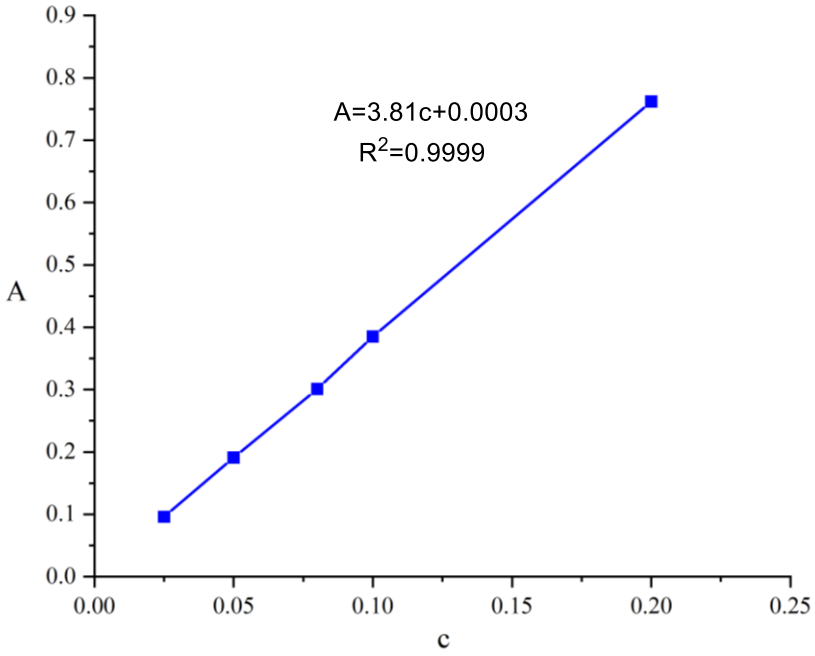


Figure S1. Fmoc-absorbance standard curve

According to the standard curve, the relationship between absorbance (A) and concentration (c) is expressed as $c = 0.262A$. The substitution value can be calculated as follows:

$$\frac{n}{m} = \frac{c \times V}{m} = \frac{0.262A \times 62.5}{1000m} = \frac{16.4A}{1000m}$$

c (mmol/L) refers to the concentration of the tested solution, V (L) indicates the volume of the solution, m (g) represents the weight of the resin to be measured, and 62.5 is a multiplier for solution D diluted to solution F.

b) The quantitative Fmoc test

Using **RTP-1** as an example, Fmoc-Leu-NH-AM resin was synthesized following method **2.1**. A portion of the synthesized Fmoc-Leu-NH-AM resin was extracted, washed with solvent, and placed in a vacuum drying oven for one hour. It was then divided into three parts, each transferred into 2 mL syringes. Next, 1 mL of solution D was added to each syringe, and they were placed on a shaker for 30 minutes at room temperature. The reaction solution was collected into a 10 mL volumetric flask, and the resin was washed with 1 mL of acetonitrile, with the wash solution also collected in the 10 mL volumetric flask. Acetonitrile was then used to bring the solution in the volumetric flask to a total volume of 10 mL. From this, 2 mL was taken and transferred to a 25 mL volumetric flask, and acetonitrile was used again to adjust the volume to 25 mL. The same procedure was followed for the three samples. For the control group, 1 mL of solution D was added to a 10 mL volumetric flask, and acetonitrile was used to bring the volume to 10 mL. Then, 2 mL was taken from this flask and transferred to a 25 mL volumetric flask, with acetonitrile again used to adjust the volume to 25 mL. The ultraviolet absorption of the final diluted solution at a wavelength of 304 nm was measured

against the control group, and the substitution value of the first amino acid was calculated using the formula. The values from the three groups were averaged, yielding a resin loading 0.97 for **RTP-1**. Similarly, resin loadings for other supported peptides were measured.

Table S2. The data for the quantitative Fmoc test of Fmoc-aa-NH-resin.

peptide	m(mg)	A	peptide loadings	averaged peptide loadings
	17.7	1.046	0.97	
RTP-1	18.1	1.081	0.98	0.97
	18.4	1.081	0.96	
	17.2	0.558	0.53	
RTP-1	16.7	0.558	0.55	0.54
	15.3	0.498	0.53	
	23.2	1.125	0.80	
RTP-1	22.7	1.084	0.78	0.78
	23.1	1.083	0.77	
	19.3	1.423	1.21	
RTP-1	17.6	1.423	1.33	1.23
	19.5	1.367	1.15	
	15.4	0.885	0.94	
RTP-2	15.0	0.885	0.97	0.95
	14.5	0.844	0.95	
	15.7	0.830	0.87	
RTP-3	16.3	0.853	0.86	0.86
	16.4	0.854	0.85	
	19.6	1.054	0.88	
RTP-4	19.7	1.054	0.88	0.89
	21.4	1.200	0.92	
	20.2	1.044	0.85	
RTP-5	20.2	1.044	0.85	0.85
	19.6	1.023	0.86	
	18.8	0.956	0.83	
RTP-6	20.7	1.097	0.87	0.85
	21.0	1.097	0.86	
	15.4	0.291	0.31	
RTP-7	14.7	0.291	0.32	0.31
	14.5	0.267	0.30	
	15.3	0.853	0.91	
RTP-8	15.7	0.892	0.93	0.93
	15.6	0.904	0.95	
	15.2	0.878	0.95	

RTP-9	14.3	0.844	0.97	0.97
	14.2	0.846	0.98	
	13.9	0.793	0.94	
RTP-10	15.5	0.873	0.92	0.94
	15.3	0.889	0.95	
	15.4	0.963	1.03	
RTP-11	16.3	0.949	0.95	0.98
	14.3	0.829	0.95	
	14.1	0.752	0.87	
RTP-12	14.3	0.835	0.96	0.95
	14.6	0.914	1.03	
	20.5	1.166	0.93	
RTP-13	19.9	1.094	0.90	0.92
	19.2	1.094	0.93	
	18.3	1.036	0.93	
RTP-14	18.2	1.036	0.93	0.94
	19.7	1.146	0.95	
	17.8	1.041	0.96	
RTP-15	18.0	1.057	0.96	0.96
	18.3	1.057	0.95	
	19.5	1.084	0.91	
RTP-16	19.2	1.084	0.93	0.92
	19.5	1.106	0.93	
	15.7	0.872	0.91	
RTP-17	14.8	0.838	0.93	0.92
	14.3	0.812	0.93	
	14.7	0.719	0.80	
RTP-18	15.0	0.772	0.84	0.82
	15.1	0.764	0.83	
	17.0	0.818	0.79	
RTP-19	15.4	0.755	0.80	0.79
	15.0	0.726	0.79	
	14.8	0.797	0.88	
RTP-20	15.4	0.814	0.87	0.88
	15.1	0.809	0.88	

2.3 Characterization of Peptide Catalysts

RTP-1: **H-Tle-ᴅPro-Gly-Leu-NH-AM Resin:** Loading = 0.97 mmol/g or 0.54 mmol/g or 0.78

mmol/g or 1.23 mmol/g, FT-IR (solid): 3386, 1655, 1543, 1282 cm^{-1}.

TP-1: **H-Tle-ᴅPro-Gly-Leu-OH:** a white solid, mp 223 – 225°C, $[\alpha]_D^{25}$ = + 4.0 (*c* 0.5, MeOH). ^1H NMR (600 MHz, D_2O) δ 4.40 – 4.29 (m, 2H), 4.06 (s, 1H), 3.84 (s, 2H), 3.72 – 3.63 (m, 2H), 2.24 – 2.19 (m, 1H), 1.93 – 1.89 (m, 3H), 1.64 – 1.52 (m, 3H), 0.98 (s, 9H), 0.78 (dd, *J* = 27.0, 6.6 Hz, 6H); ^{13}C NMR (101 MHz, D_2O) δ 179.9, 174.4, 170.5, 168.2, 61.1, 59.1, 53.8, 49.1, 42.5, 40.6, 34.0, 29.4, 25.4, 24.6, 24.3, 22.5, 20.6. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{19}H_{35}N_4O_5$ 399.2602, Found 399.2603.

RTP-2: **H-Tle-ᴅPro-Gly-Leu-NH-MBHA Resin:** Loading = 0.95 mmol/g, FT-IR (solid): 3386, 1624, 1541, 1258 cm^{-1}.

RTP-3: **H-Tle-ᴅPro-Gly-Leu-NH-Rink Amide Resin:** Loading = 0.86 mmol/g, FT-IR (solid): 3307, 1666, 1507, 1207 cm^{-1}.

RTP-4: **H-Tle-ᴅPro-Gly-Leu-NH-Rink Amide-AM Resin:** Loading = 0.89 mmol/g, FT-IR (solid): 3302, 1657, 1508, 1294 cm^{-1}.

RTP-5: **H-Tle-ᴅPro-Gly-Leu-NH-Rink Amide-MBHA Resin:** Loading = 0.85 mmol/g, FT-IR (solid): 3427, 1659, 1506, 1296 cm^{-1}.

RTP-6: **H-Tle-ᴅPro-Gly-Leu-O-Wang Resin:** Loading = 0.85 mmol/g, FT-IR (solid): 3304, 1671, 1512, 1242 cm^{-1}.

RTP-7: **H-Tle-ᴅPro-Gly-Leu-NH-TentaGel Resin:** Loading = 0.31 mmol/g, FT-IR (solid): 3398, 1673, 1538, 1251 cm^{-1}.

RTP-8: **H-Ala-ᴅPro-Gly-Leu-NH-AM Resin:** Loading = 0.93 mmol/g, FT-IR (solid): 3406, 1657,1539, 1255 cm^{-1}.

RTP-9: **H-Leu-ᴅPro-Gly-Leu-NH-AM Resin:** Loading = 0.97 mmol/g, FT-IR (solid): 3402, 1632, 1535, 1254 cm^{-1}.

RTP-10: **H-Val-ᴅPro-Gly-Leu-NH-AM Resin:** Loading = 0.94 mmol/g, FT-IR (solid): 3404, 1657,1543, 1255 cm^{-1}.

RTP-11: **H-Chg-ᴅPro-Gly-Leu-NH-AM Resin:** Loading = 0.98 mmol/g, FT-IR (solid): 3417, 1637, 1547, 1257 cm^{-1}.

RTP-12: **H-Phe-ᴅPro-Gly-Leu-NH-AM Resin:** Loading = 0.95 mmol/g, FT-IR (solid): 3421, 1659, 1545, 1255 cm^{-1}.

RTP-13: **H-Tle-ᴅPro-Gly-Gly-NH-AM Resin:** Loading = 0.92 mmol/g, FT-IR (solid): 3381, 1655,1526, 1255 cm^{-1}.

RTP-14: **H-Tle-ᴅPro-Gly-Ala-NH-AM Resin:** Loading = 0.94 mmol/g, FT-IR (solid): 3336, 1657, 1542, 1254 cm^{-1}.

RTP-15: **H-Tle-ᴅPro-Gly-Phe-NH-AM Resin:** Loading = 0.96 mmol/g, FT-IR (solid): 3412,

1657, 1541, 1254 cm^{-1}.

RTP-16: **H-Tle-ᴅPro-Gly-Asp-NH-AM Resin:** Loading = 0.92 mmol/g, FT-IR (solid): 3311, 1728, 1663, 1543, 1252 cm^{-1}.

RTP-17: **H-Tle-ᴅPro-Gly-NH-AM Resin:** Loading = 0.92 mmol/g, FT-IR (solid): 3385, 1655, 1541, 1254 cm^{-1}.

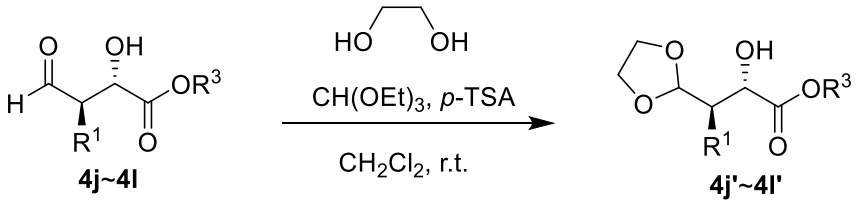
RTP-18: **H-Tle-ᴅPro-NH-AM Resin:** Loading = 0.82 mmol/g, FT-IR (solid): 3387, 1634, 1540, 1231 cm^{-1}.

RTP-19: **H-Tle-NH-AM Resin:** Loading = 0.79 mmol/g, FT-IR (solid): 3382, 1653, 1600, 1256 cm^{-1}.

RTP-20: **H-ᴅTle-ᴅPro-Gly-Leu-NH-AM Resin:** Loading = 0.88 mmol/g, FT-IR (solid): 3334, 1657, 1543, 1254cm^{-1}.

3. General Procedure for synthesizing the aldol adduct derivative for ee determination

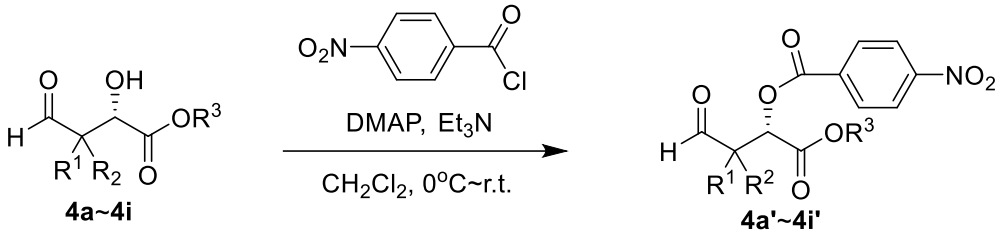
3.1 Synthesis of the Acetal Derivatives of 4j ~ 4l[2]



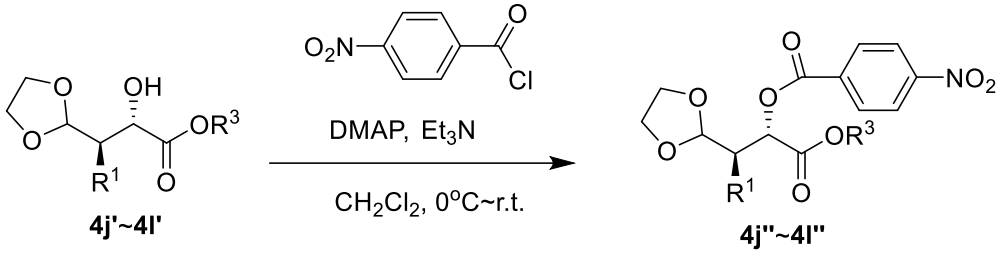
In a 5 mL round bottom flask equipped with a magnetic stirrer, **4j** ~ **4l** (0.5 mmol), CH_2Cl_2 (1.0 mL), $(CH_2OH)_2$ (0.11 mL, 1.0 mmol), triethylorthoformate (165 μL, 1.0 mmol) and *p*-toluenesulfonic acid (19 mg, 0.1 mmol) were added at room temperature. After 2 h of stirring, the reaction was quenched with water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel (petroleum ether: EtOAc = 8 : 1) to afford **4j′** ~ **4l′**.

3.2 Synthesis of 4a′ ~ 4i′ and 4j′′ ~ 4l′′ for Determining ee values of 4a ~ 4i and 4j′ ~ 4l′[3]

a)



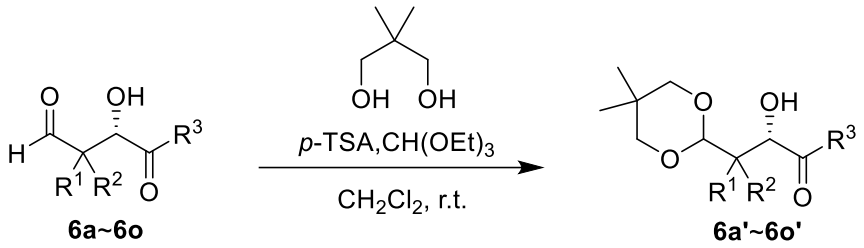
b)



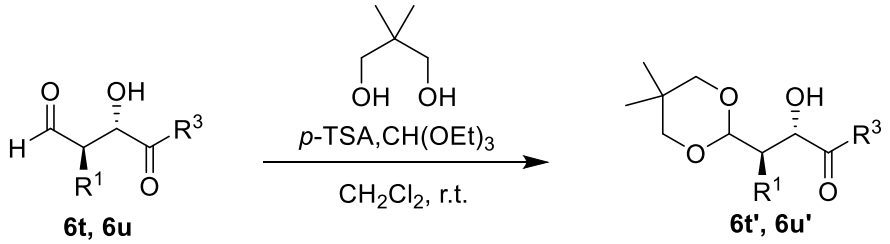
The product **4a** ~ **4i** and **4j′** ~ **4l′** (0.3 mmol) was added into the mixture of dimethylamino-pyridine (DMAP) (3.7 mg, 0.03 mmol), *p*-nitro benzoyl chloride (67 mg, 0.36 mmol) and triethylamine (50 mL, 0.36 mmol) in 1.0 mL of dichloromethane at 0 °C in a 5.0 mL round bottom flask. After the reaction was completed with the detection of TLC, a drop of water was added and the mixture was purified by column chromatography on silica gel (petroleum ether: EtOAc = 8 : 1) to give the product **4a′** ~ **4i′** and **4j′′** ~ **4l′′**.

3.3 Synthesis of 6a′ ~ 6o′, 6t′ and 6u′ for determining ee values of 6a ~ 6o, 6t and 6u[3]

a)



b)

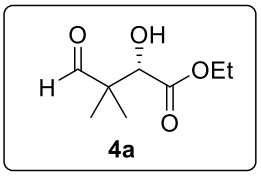


To a solution of **6** (0.2 mmol) in CH_2Cl_2 (1.0 mL) were sequentially added 2,2-dimethyl-1,3-propanediol (31.2 mg, 0.3 mmol), triethyl orthoformate (50 μL, 0.3 mmol) and *p*-toluenesulfonic acid (7.6 mg, 0.04 mmol) at room temperature. After 2 h of stirring, the reaction was quenched with water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried over Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel (petroleum ether: EtOAc = 8 : 1) to afford **6a′** ~ **6o′**, **6t′** and **6u′**.

4. Characterization of Cross-Aldol Reaction Product

4.1 Products of Substrate Scope

(*S*)-Ethyl-2-hydroxy-3, 3-dimethyl-4-oxobutanoate (4a)[3]



4a

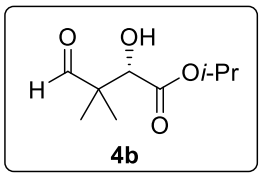
4a from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 83 mg, 95% yield, 98% ee, $[\alpha]_D^{23}$ = + 16.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.58 (s, 1H), 4.32 (s, 1H), 4.30 – 4.22 (m, 2H), 1.28 (t, *J* = 7.2 Hz, 3H), 1.15 (s, 3H), 1.06 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.5, 172.9, 73.6, 62.3, 50.4, 18.3, 16.9, 14.19.9. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_8H_{15}O_4$ 175.0965, Found 175.0966. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.7 min (minor) and 9.6 min (major).

4a from condition B, colorless oil, 85 mg, 98% yield, 95% ee, $[\alpha]_D^{23}$ = + 21.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.6 min (minor) and 9.4 min (major).

ent-**4a from condition A**, the title compound was purified by column chromatography on silica gel (PE : EA = 10 : 1), colorless oil, 82 mg, 94% yield, – 98% ee, $[\alpha]_D^{24}$ = – 20.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 7.1 min (major) and 9.8 min(minor).

ent-**4a from condition B**, the title compound was purified by column chromatography on silica gel (PE : EA = 10 : 1), colorless oil, 85 mg, 98% yield, – 95% ee, $[\alpha]_D^{24}$ = – 21.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 7.0 min (major) and 9.6 min (minor).

(*S*)-Isopropyl-2-hydroxy-3,3-dimethyl-4-oxobutanoate(4b)[2]

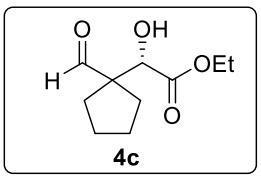


4b

4b from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 81 mg, 86% yield, 98% ee, $[\alpha]_D^{24}$ = + 22.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.57 (s, 1H), 5.12 – 5.07 (m, 1H), 4.28 (d, *J* = 6.0 Hz, 1H), 3.03 (d, *J* = 5.4 Hz, 1H), 1.25 (dd, *J* = 12.0, 6.0 Hz, 6H), 1.13 (s, 3H), 1.05 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.5, 172.4, 73.4, 70.8, 50.5, 21.8, 21.7, 18.4, 16.7. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_9H_{17}O_4$ 189.1121, Found 189.1119. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 80/20, flow rate 1.0 mL/min, 254 nm, retention time : 7.6 min (minor) and 11.1 min (major).

4b from condition B, colorless oil, 85 mg, 90% yield, 95% ee, $[\alpha]_D^{24}$ = + 19.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 80/20, flow rate 1.0 mL/min, 254 nm, retention time : 7.6 min (minor) and 11.1 min (major).

(*S*)-Ethyl-2-(1-formylcyclopentyl)-2-hydroxyacetate(4c)[3]

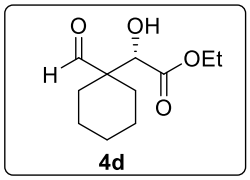


4c

4c from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 91 mg, 91% yield, 98% ee, $[\alpha]_D^{25}$ = + 9.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.56 (s, 1H), 4.35 (s, 1H), 4.27 – 4.19 (m, 2H), 3.09 (s, 1H), 1.97 – 1.84 (m, 3H), 1.73 – 1.52 (m, 5H), 1.26 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.7, 173.3, 72.4, 62.3, 61.7, 30.3, 28.1, 25.9, 25.7, 14.1. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{10}H_{17}O_4$ 201.1121, Found 201.1121. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 80/20, flow rate 1.0 mL/min, 254 nm, retention time : 8.1 min (minor) and 11.9 min (major).

4c from condition B, colorless oil, 93 mg, 93% yield, 95% ee, $[\alpha]_D^{25}$= + 4.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 80/20, flow rate 1.0 mL/min, 254 nm, retention time : 8.1 min (minor) and 11.9 min (major).

(*S*)-Ethyl-2-(1-formylcyclohexyl)-2-hydroxyacetate(4d)[3]



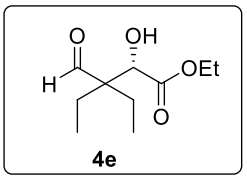
4d

4d from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 94 mg, 88% yield, 98% ee, $[\alpha]_D^{26}$ = + 22.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.59 (s, 1H), 4.31 – 4.21 (m, 2H), 4.15 (d, *J* = 6.6 Hz, 1H), 2.95 (d, *J* = 6.6 Hz, 1H), 1.97 (d, *J* = 15.0 Hz, 1H), 1.80 (d, *J* = 13.2 Hz, 1H), 1.67 – 1.59 (m, 4H), 1.53 (d, *J* =13.2, 3.6 Hz, 1H), 1.45 – 1.39 (m, 1H), 1.30 (t, *J* = 6.6 Hz, 3H), 1.26 – 1.14 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.5, 172.8, 73.9, 62.3, 53.5,

27.9, 25.9, 25.2, 22.3, 22.1, 14.1. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{11}H_{19}O_4$ 215.1278, Found 215.1278. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 80/20, flow rate 1.0 mL/min, 254 nm, retention time : 8.1 min (minor) and 12.3 min (major).

4d from condition B, colorless oil, 96 mg, 90% yield, 95% ee, $[\alpha]_D^{26}$= + 19.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 80/20, flow rate 1.0 mL/min, 254 nm, retention time : 8.0 min (minor) and 12.3 min (major).

(*S*)-Ethyl-3-ethyl-3-formyl-2-hydroxypentanoate(4e)[3]

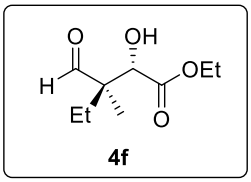


4e

4e from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 81 mg, 80% yield, 98% ee, $[\alpha]_D^{26}$ = + 14.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.61 (s, 1H), 4.42 (s, 1H), 4.30 – 4.24 (m, 2H), 2.96 (s, 1H), 1.77 – 1.66 (m, 4H), 1.30 (t, *J* = 7.2 Hz, 3H), 0.89 (td, *J* = 7.8, 3.0 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.1, 173.6, 72.7, 62.3, 55.9, 22.6, 21.7, 14.1, 8.1, 7.9. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{10}H_{19}O_4$ 203.1278, Found 203.1279. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 80/20, flow rate 1.0 mL/min, 254 nm, retention time : 7.8 min (minor) and 11.0 min (major).

4e from condition B, colorless oil, 85 mg, 84% yield, 96% ee, $[\alpha]_D^{26}$= + 16.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 80/20, flow rate 1.0 mL/min, 254 nm, retention time : 7.7 min (minor) and 11.2 min (major).

(2*S*,3*R*)-Ethyl-3-formyl-2-hydroxy-3-methylpentanoate(4f)[2]



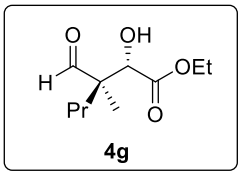
4f

4f from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 87 mg, 92% yield, 98/97% ee, *anti*/*syn* = 53:47, $[\alpha]_D^{19}$ = + 17.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.59 (s, 1H), 4.41 (s, 1H), 4.29 – 4.19 (m, 2H), 2.93 (s, 1H), 1.83 – 1.77 (m, 1H), 1.74 – 1.68 (m, 1H), 1.26 (t, *J* = 7.2 Hz, 3H), 0.97 (s, 3H), 0.86 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.4, 203.1, 173.2, 172.9, 73.4, 72.9, 62.4, 62.2, 53.8, 53.5, 26.0, 25.0, 14.2, 14.1, 12.8, 8.2, 8.1. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_9H_{17}O_4$ 189.1121, Found 189.1122. The *ee* was determined by HPLC analysis. CHIRALPAK OJ-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 24.0 min (minor) and 47.7 min (major).

4f from condition B, colorless oil, 89 mg, 95% yield, 97/92% ee, *anti*/*syn* = 63:37, $[\alpha]_D^{19}$ = + 24.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK OJ-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 25.6 min (minor)

and 51.4 min (major).

(2*S*,3*R*)-Ethyl-3-formyl-2-hydroxy-3-methylhexanoate(4g)[2]

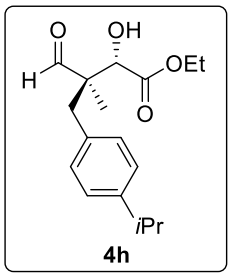


4g

4g from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 97 mg, 96% yield, 98/94% ee, *anti*/*syn* = 58:42, $[\alpha]_D^{20}$ = + 17.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.61 (s, 1H), 4.41 (d, *J* = 6.0 Hz, 1H), 4.29 – 4.20 (m, 2H), 2.98 (d, *J* = 5.4 Hz, 1H), 1.74 –1.61 (m, 2H), 1.33 – 1.30 (m, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.22 – 1.20 (m, 1H), 0.99 (s, 3H), 0.91 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 203.1, 173.1, 73.1, 62.4, 53.8, 35.6, 17.0, 14.7, 14.1, 13.3. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{10}H_{19}O_4$ 203.1278, Found 203.1278. The *ee* was determined by HPLC analysis. CHIRALPAK OJ-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 18.5 min (minor) and 38.2 min (major).

4g from condition B, colorless oil, 97 mg, 96% yield, 94/85% ee, *anti*/*syn* = 65:35, $[\alpha]_D^{20}$ = + 43.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK OJ-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 18.7 min (minor) and 38.0 min (major).

(2*S*,3*R*)-Ethyl-3-formyl-2-hydroxy-4-(4-isopropylphenyl)-3-methylbutanoate(4h)[2]

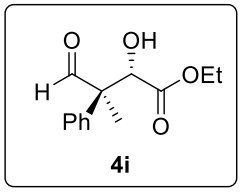


4h

4h from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 139mg, 95% yield, 95/85% ee, *anti*/*syn* = 52:48, $[\alpha]_D^{20}$ = + 18.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.59 (s, 1H), 7.11 (dd, *J* = 22.8, 8.4 Hz, 4H), 4.47 (s, 1H), 4.23 – 4.14 (m, 2H), 3.16 (d, *J* = 13.8 Hz, 1H), 2.89 – 2.84 (m, 1H), 2.80 (d, *J* = 13.8 Hz, 1H), 1.23 – 1.20 (m, 9H), 0.99 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.0, 173.4, 147.5, 132.6, 130.5, 126.4, 71.5, 62.5, 54.2, 40.3, 33.7, 24.0, 14.1, 14.0. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{17}H_{25}O_4$ 293.1747, Found 293.1748. The *ee* was determined by HPLC analysis. CHIRALPAK IH, Hexane/2-propanol = 95/5, flow rate 0.8 mL/min, 254 nm, retention time : major isomer 22.5 min (minor) and 29.8 min (major).

4h from condition B, colorless oil, 131 mg, 90% yield, 90/79% ee, *anti*/*syn* = 62:38, $[\alpha]_D^{21}$ = + 7.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK IH, Hexane/2-propanol = 95/5, flow rate 0.8 mL/min, 254 nm, retention time : major isomer 22.5 min (minor) and 29.4 min (major).

(2*S*,3*R*)-Ethyl -2-hydroxy-3-methyl-4-oxo-3-phenylbutanoate(4i)[3]

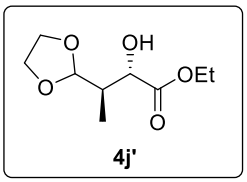


4i

4i from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 100mg, 85% yield, 86/26% ee, *anti*/*syn* = 65:35, $[\alpha]_D^{21}$ = + 19.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.65 (s, 1H), 7.39 – 7.37 (m, 2H), 7.33 – 7.29 (m, 3H), 4.91 (s, 1H), 3.96 (q, *J* = 7.2 Hz, 2H), 3.10 (s, 1H), 1.59 (s, 3H), 0.94 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.5, 199.0, 172.4, 172.3, 136.2, 135.8, 128.9, 128.8, 127.9, 127.8, 127.6, 73.9, 73.6, 62.4, 61.6, 58.0, 57.8, 15.3, 14.0, 13.9, 13.64. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{13}H_{17}O_4$ 237.1121, Found 237.1122. The *ee* was determined by HPLC analysis. CHIRALPAK IB, Hexane/2-propanol = 98/2, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 23.1min (major) and 27.4 min (minor).

4i from condition B, colorless oil, 83 mg, 70% yield, 66/10% ee, *anti*/*syn* = 79:21, $[\alpha]_D^{21}$ = + 14.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK IB, Hexane/2-propanol = 98/2, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 21.8 min (major) and 25.7 min (minor).

(2*S*,3*R*)-Ethyl -3-(1,3-dioxolan-2-yl)-2-hydroxybutanoate(4j')[2]

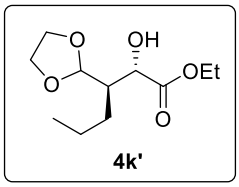


4j'

4j' from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 74mg, 73% yield, 81/80% ee, *anti*/*syn* = 62:38, $[\alpha]_D^{23}$ = + 1.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 4.89 – 4.87 (m, 1H), 4.31 – 4.22 (m, 2H), 4.01 – 3.83 (m, 4H), 3.22 – 3.00 (m, 1H), 2.32 – 2.18 (m, 1H), 1.30 (t, *J* = 7.2 Hz, 3H), 1.15 (d, *J* = 7.2 Hz, 1H), 0.96 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.1, 105.6, 105.5, 72.7, 70.8, 65.1, 65.0, 64.9, 64.8, 61.7, 61.3, 40.6, 40.2, 14.2, 14.1, 12.4, 8.7. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_9H_{17}O_5$ 205.1071, Found 205.1071. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 20.3 min (minor) and 47.6 min (major).

4j' from condition B, colorless oil, 82 mg, 80% yield, 43/48% ee, *anti*/*syn* = 57:43, $[\alpha]_D^{23}$ = + 3.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 20.2 min (minor) and 47.0 min (major).

(2*S*,3*R*)-Ethyl -3-(1,3-dioxolan-2-yl)-2-hydroxyhexanoate(4k')[2]

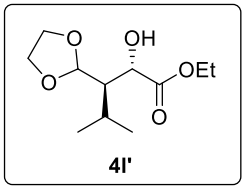


4k'

4k' from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 106mg, 91% yield, 81/81% ee, *anti*/*syn* = 53:47, $[\alpha]_D^{23}$ = + 6.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 4.92 – 4.88 (m, 1H), 4.27 – 4.20 (m, 2H), 4.04 – 3.78 (m, 4H), 3.28– 3.20 (m, 1H), 2.24 – 2.17 (m, 1H), 1.58 – 1.33 (m, 4H), 1.31– 1.29 (m, 3H), 0.97– 0.88 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.7, 174.2, 105.6, 105.5, 70.3, 70.2 , 65.0, 64.8, 64.7, 61.5, 61.2, 45.1, 44.2, 29.7, 29.2, 27.5, 20.7, 20.3, 14.3, 14.2, 14.1. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{11}H_{21}O_5$ 233.1384, Found 233.1384. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H; Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 15.5 min (minor) and 32.1 min (major).

4k' from condition B, colorless oil, 100 mg, 86% yield, 36/37% ee, *anti*/*syn* = 57:43, $[\alpha]_D^{23}$ = + 2.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 15.6 min (minor) and 31.9 min (major).

(2*S*,3*R*)-Ethyl-3-(1,3-dioxolan-2-yl)-2-hydroxy-4-methylpentanoate(4l')[2]

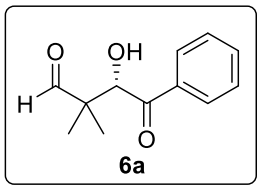


4l'

4l' from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 111mg, 96% yield, 85/86% ee, *anti*/*syn* = 68:32, $[\alpha]_D^{24}$ = + 5.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 5.05 (d, *J* = 3.6 Hz, 1H), 4.27 (s, 1H), 4.22 – 4.14 (m, 2H), 3.99 – 3.94 (m, 1H), 3.88 – 3.84 (m, 1H), 3.80 – 3.75 (m, 2H), 3.42 (s, 1H), 2.13 – 2.07 (m, 1H), 2.03 – 2.00 (m, 1H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.08 (dd, *J* = 13.8, 6.6 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.0, 104.8, 69.8, 64.8, 64.4, 61.0, 49.8, 27.0, 21.2, 20.8, 14.2. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{11}H_{21}O_5$ 233.1384, Found 233.1385. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H; Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 15.0 min (minor) and 21.1 min (major).

4l' from condition B, colorless oil, 104 mg, 90% yield, 49/50% ee, *anti*/*syn* = 62:38, $[\alpha]_D^{25}$ = + 5.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 15.2 min (minor) and 21.7 min (major).

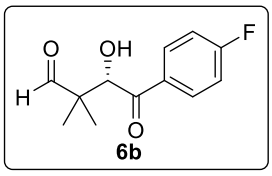
(*S*)-3-Hydroxy-2,2-dimethyl-4-oxo-4-phenylbutanal(6a)[3]



6a from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 98 mg, 95% yield, 95% ee, $[\alpha]_D^{27}$ = + 61.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.61 (s, 1H), 7.86 –7.85 (m, 2H), 7.63 – 7.60 (m, 1H), 7.49 (t, *J* = 7.8 Hz, 2H), 5.26 (s, 1H), 3.74 (s, 1H), 1.07 (s, 3H), 0.94 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.8, 201.1, 136.1, 134.2, 128.9, 128.7, 76.0, 50.1, 19.6, 17.6.HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{12}H_{15}O_3$ 207.1016, Found 207.1016. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 6.9 min (major) and 12.1 min (minor).

6a from condition B, colorless oil, 88 mg, 85% yield, 97% ee, $[\alpha]_D^{27}$ = + 44.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.0 min (major) and 12.1 min (minor).

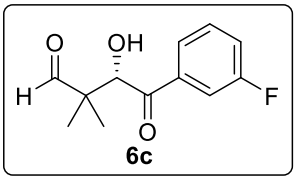
(*S*)-4-(4-Fluorophenyl)-3-hydroxy-2,2-dimethyl-4-oxobutanal (6b)[3]



6b from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 109 mg, 97% yield, 92% ee, $[\alpha]_D^{27}$ = + 42.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.62 (s, 1H), 7.93 (dd, *J* = 8.4, 5.4 Hz, 2H), 7.17 (t, *J* = 8.4 Hz, 2H), 5.19 (s, 1H), 3.67 (s, 1H), 1.09 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.2, 199.1, 167.6, 165.0, 132.4, 132.3, 131.6, 131.5, 116.3, 116.1, 76.3, 49.9, 19.7, 17.8. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{12}H_{14}FO_3$ 225.0921, Found 225.0921. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.3 min (major) and 12.0 min (minor).

6b from condition B, colorless oil, 81 mg, 72% yield, 95% ee, $[\alpha]_D^{27}$= + 52.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.2 min (major) and 11.9 min (minor).

(*S*)-4-(3-Fluorophenyl)-3-hydroxy-2,2-dimethyl-4-oxobutanal (6c)

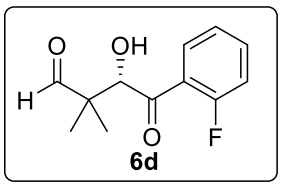


6c from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 101 mg, 90% yield, 95% ee, $[\alpha]_D^{30}$ = + 61.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.62 (s, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.59 – 7.56 (m, 1H), 7.50 – 7.46 (m, 1H), 7.33 – 7.30 (m, 1H), 5.18 (d, *J* = 4.8 Hz, 1H), 3.66 (d, *J* = 6.6 Hz, 1H), 1.10 (s, 3H), 0.99 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 204.0,

199.6, 163.8, 161.9, 138.0 , 130.6, 124.6, 121.2, 121.1, 115.6, 115.4, 76.5, 49.8, 19.7, 17.7. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{12}H_{14}FO_3$ 225.0921, Found 225.0919. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 6.3 min (major) and 11.5 min (minor).

6c from condition B, colorless oil, 76 mg, 68% yield, 92% ee, $[\alpha]_D^{30}$ = + 46.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 6.3 min (major) and 11.4 min (minor).

(*S*)-4-(2-Fluorophenyl)-3-hydroxy-2,2-dimethyl-4-oxobutanal (6d)

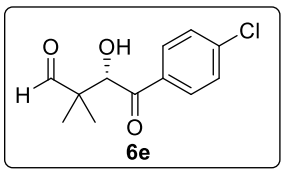


6d

6d from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 106 mg, 95% yield, 99% ee, $[\alpha]_D^{30}$ = + 51.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.59 (s, 1H), 7.71 – 7.68 (m, 1H), 7.59 – 7.55 (m, 1H), 7.28 –7.25 (m, 1H), 7.17 – 7.14 (m, 1H), 5.22 (s, 1H), 3.76 (d, *J* = 6.0 Hz, 1H), 1.07 (s, 3H), 0.96 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 203.0, 199.5, 161.7, 160.0, 135.5, 130.4, 125.1, 116.8, 79.1, 50.6, 18.2, 17.6. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{12}H_{14}FO_3$ 225.0921, Found 225.0920. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.1 min (major) and 15.6 min (minor).

6d from condition B, colorless oil, 80 mg, 71% yield, 97% ee, $[\alpha]_D^{30}$ = + 35.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.1 min (major) and 15.6 min (minor).

(*S*)-4-(4-Chlorophenyl)-3-hydroxy-2,2-dimethyl-4-oxobutanal (6e)[3]

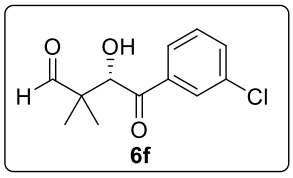


6e

6e from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 116 mg, 97% yield, 89% ee, $[\alpha]_D^{28}$ = + 34.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.62 (s, 1H), 7.83 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 9.0 Hz, 2H), 5.18 (s, 1H), 3.67 (s, 1H), 1.09 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.2, 199.6, 140.8, 134.3, 130.2, 129.3, 76.4, 49.9, 19.7, 17.8. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{12}H_{14}ClO_3$ 241.0626, Found 241.0627. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.0 min (major) and 10.0 min (minor).

6e from condition B, colorless oil, 72 mg, 60% yield, 84% ee, $[\alpha]_D^{28}$ = + 31.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 6.7 min (major) and 8.9 min (minor).

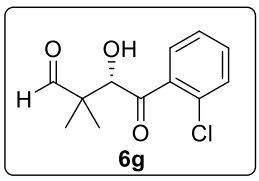
(*S*)-4-(3-Chlorophenyl)-3-hydroxy-2,2-dimethyl-4-oxobutanal (6f)[3]



6f from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 108 mg, 90% yield, 91% ee, $[\alpha]_D^{29}$ = + 61.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.64 (s, 1H), 7.88 (t, *J* = 1.8 Hz, 1H), 7.77 – 7.75 (m, 1H), 7.61 – 7.59 (m, 1H), 7.45 (t, *J* = 7.8 Hz, 1H), 5.18 (d, *J* = 7.2 Hz, 1H), 3.65 (d, *J* = 8.4 Hz, 1H), 1.11 (s, 3H), 1.01 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.2, 199.6, 137.6, 135.3, 134.0, 130.2, 128.8, 126.9, 76.6, 49.8, 19.8, 17.8. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{12}H_{14}ClO_3$ 241.0626, Found 241.0627. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 6.1 min (major) and 11.3 min (minor).

6f from condition B, colorless oil, 70 mg, 58% yield, 91% ee, $[\alpha]_D^{29}$ = + 36.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 6.2 min (major) and 11.9 min (minor).

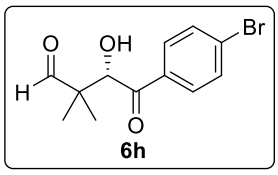
(*S*)-4-(2-Chlorophenyl)-3-hydroxy-2,2-dimethyl-4-oxobutanal (6g)[3]



6g from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 112 mg, 93% yield, 99% ee, $[\alpha]_D^{29}$ = +26.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.50 (s, 1H), 7.46 – 7.44 (m, 3H), 7.38 – 7.35 (m, 1H), 5.27 (s, 1H), 3.72 (s, 1H), 1.11 (s, 3H), 0.92 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.8, 202.7, 137.0, 133.0, 131.5, 131.1, 129.7, 127.2, 78.5, 50.9, 18.4, 17.8. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{12}H_{14}ClO_3$ 241.0626, Found 241.0627. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.2 min (major) and 14.1 min (minor).

6g from condition B, colorless oil, 34 mg, 28% yield, 98% ee, $[\alpha]_D^{26}$ = +15.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.4 min (major) and 14.4 min (minor).

(*S*)-4-(4-Bromophenyl)-3-hydroxy-2,2-dimethyl-4-oxobutanal (6h)[3]

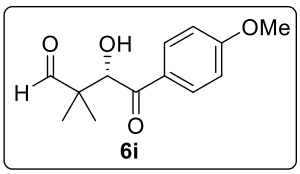


6h from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 131 mg, 92% yield, 86% ee, $[\alpha]_D^{30}$ = + 41.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.61 (s, 1H), 7.75 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 2H), 5.17 (s, 1H), 3.68 (s, 1H), 1.08 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.2, 199.8, 134.7, 132.3, 130.2, 129.6, 76.3, 49.8, 19.8, 17.8.

HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{12}H_{14}BrO_3$ 285.0121, Found 285.0120. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.5 min (major) and 10.0 min (minor).

6h from condition B, colorless oil, 72 mg, 51% yield, 85% ee, $[\alpha]_D^{30}$ = + 25.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.5 min (major) and 10.0 min (minor).

(*S*)-3-Hydroxy-4-(4-methoxyphenyl)-2,2-dimethyl-4-oxobutanal (6i)[3]

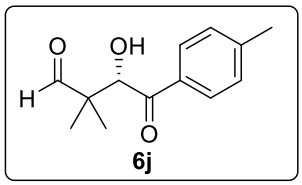


6i

6i from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 111 mg, 94% yield, 97% ee, $[\alpha]_D^{28}$ = + 101.0 (*c* 1.0, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.64 (s, 1H), 7.89 – 7.87 (m, 2H), 6.98 – 6.94 (m, 2H), 5.23 (s, 1H), 3.89 (s, 3H), 3.75 (s, 1H), 1.08 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.1, 199.0, 164.5, 131.3, 128.6, 114.2, 75.5, 55.6, 50.2, 19.6, 17.6. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{13}H_{17}O_4$ 237.1121, Found 237.1120. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : 11.0 min (major) and 13.2 min (minor).

6i from condition B, colorless oil, 96 mg, 81% yield, 94% ee, $[\alpha]_D^{28}$ = + 116.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : 11.5 min (major) and 13.3 min (minor).

(S)-3-Hydroxy-2,2-dimethyl-4-oxo-4-(*p*-tolyl)butanal (6j)[3]

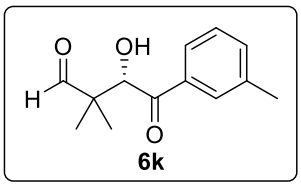


6j

6j from condition A, the title compound was purified by column chromatography on silica gel(PE : EA = 10 : 1), colorless oil, 106 mg, 96% yield, 91% ee, $[\alpha]_D^{29}$ = + 148.0 (*c* 1.0, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.61 (s, 1H), 7.76 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 5.24 (s, 1H), 3.73 (s, 1H), 2.42 (s, 3H), 1.06 (s, 3H), 0.94 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.9, 200.5, 145.4, 133.4, 129.6, 128.9, 75.7, 50.2, 21.8, 19.5, 17.5. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{13}H_{17}O_3$ 221.1172, Found 221.1171. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min; 254 nm; retention time : 9.0 min (major) and 12.6 min (minor).

6j from condition B, colorless oil, 92 mg, 84% yield, 92% ee, $[\alpha]_D^{29}$ = + 66.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 8.8 min (major) and 12.7 min (minor).

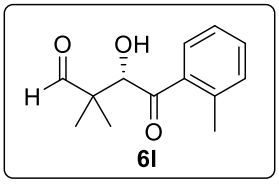
(*S*)-3-Hydroxy-2,2-dimethyl-4-oxo-4-(*m*-tolyl)butanal (6k)[3]



6k from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 105 mg, 95% yield, 93% ee, $[\alpha]_D^{29}$ = + 106.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.60 (s, 1H), 7.66 – 7.63 (m, 2H), 7.44 – 7.36 (m, 2H), 5.25 (s, 1H), 3.72 (s, 1H), 2.42 (s, 3H), 1.07 (s, 3H), 0.93 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.8, 201.3, 139.0, 136.1, 135.0, 129.1, 128.8, 126.0, 75.9, 50.1, 21.3, 19.5, 17.5. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{13}H_{17}O_3$ 221.1172, Found 221.1171. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.5 min (major) and 13.4 min (minor).

6k from condition B, colorless oil, 90 mg, 82% yield, 90% ee, $[\alpha]_D^{29}$ = + 68.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 7.2 min (major) and 13.1 min (minor).

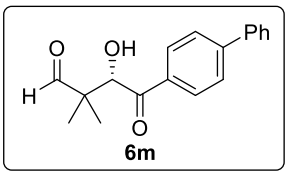
(*S*)-3-Hydroxy-2,2-dimethyl-4-oxo-4-(*o*-tolyl)butanal (6l)[3]



6l from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 103 mg, 94% yield, 98% ee, $[\alpha]_D^{29}$ = + 72.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.44 (s, 1H), 7.47 – 7.36 (m, 2H), 7.26 – 7.21 (m, 2H), 5.13 (s, 1H), 3.80 (s, 1H), 2.41 (s, 3H), 0.99 (s, 3H), 0.80 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.0, 202.9, 138.2, 136.0, 132.6, 132.4, 128.9, 125.8, 77.1, 50.9, 20.5, 18.9, 17.9. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{13}H_{17}O_3$ 221.1172, Found 221.1172. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 5.3 min (major) and 9.2 min (minor).

6l from condition B, colorless oil, 88 mg, 80% yield, 93% ee, $[\alpha]_D^{29}$ = + 120.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : 5.4 min (major) and 8.6 min (minor).

(*S*)-4-([1,1'-Biphenyl]-4-yl)-3-hydroxy-2,2-dimethyl-4-oxobutanal (6m)[3]

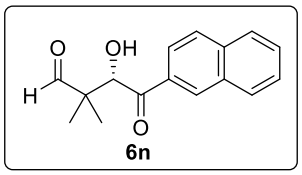


6m from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 133 mg, 94% yield, 98% ee, $[\alpha]_D^{29}$ = + 70.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.66 (s, 1H), 7.95 (d, *J* = 8.4 Hz, 2H), 7.71 (d, *J* = 8.4 Hz, 2H), 7.63 – 7.62 (m, 2H), 7.49 – 7.46 (m, 2H), 7.43 – 7.40 (m, 1H), 5.30 (s, 1H), 3.76 (s, 1H), 1.11 (s, 3H), 1.01 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.0, 200.4, 147.0, 139.4, 134.6, 129.4, 129.1, 128.6, 127.5, 127.3, 76.0, 50.1, 19.8, 17.6.

HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{18}H_{19}O_3$ 283.1329, Found 283.1328. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : 5.9 min (minor) and 7.0 min (major).

6m from condition B, colorless oil, 124 mg, 88% yield, 99% ee, $[\alpha]_D^{29}$ = + 41.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : 6.0 min (minor) and 7.0 min (major).

(*S*)-3-Hydroxy-2,2-dimethyl-4-(naphthalen-2-yl)-4-oxobutanal (6n)[3]

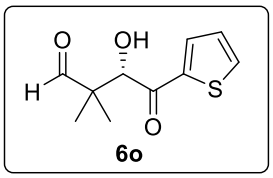


6n

6n from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 120 mg, 94% yield, 96% ee, $[\alpha]_D^{28}$ = + 54.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.66 (s, 1H), 8.40 (s, 1H), 7.98 – 7.88 (m, 4H), 7.65 – 7.57 (m, 2H), 5.43 (s, 1H), 3.80 (d, *J* = 5.4 Hz, 1H), 1.12 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.1, 200.9, 136.0, 133.3, 132.3, 130.9, 129.7, 129.2, 129.0, 127.9, 127.3, 123.9, 76.1, 50.2, 19.8, 17.6. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{16}H_{17}O_3$ 257.1172, Found 257.1169. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : 8.9 min (major) and 13.9 min (minor).

6n from condition B, colorless oil, 119 mg, 93% yield, 94% ee, $[\alpha]_D^{28}$ = + 41.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : 9.0 min (major) and 14.1 min (minor).

(*S*)-3-Hydroxy-2,2-dimethyl-4-oxo-4-(thiophen-2-yl)butanal (6o)[3]

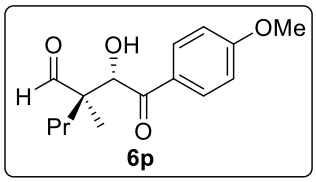


6o

6o from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 102 mg, 96% yield, 87% ee, $[\alpha]_D^{28}$ = + 93.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.65 (s, 1H), 7.82 – 7.81 (m, 1H), 7.75 – 7.74 (m, 1H), 7.17 – 7.16 (m, 1H), 5.00 (s, 1H), 3.49 (d, *J* = 5.4 Hz, 1H), 1.15 (s, 3H), 1.08 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.3, 192.4, 141.8, 135.8, 134.3, 128.5, 77.6, 50.0, 19.6, 17.7. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{10}H_{13}O_3S$ 213.0580, Found 213.0579. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : 10.0 min (major) and 13.7 min (minor).

6o from condition B, colorless oil, 83 mg, 78% yield, 86% ee, $[\alpha]_D^{28}$ = + 128.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : 9.3 min (major) and 12.5 min (minor).

(*R*)-2-((S)-1-hydroxy-2-(4-methoxyphenyl)-2-oxoethyl)-2-methylpentanal (6p)[3]

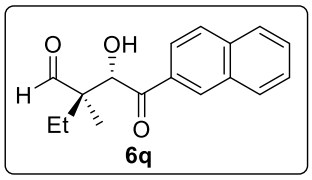


6p

6p from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 119 mg, 90% yield, 95/90% ee, *anti*/*syn* =51:49, $[\alpha]_D^{21}$ = + 29.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.61 (s, 1H), 7.84 – 7.82 (m, 2H), 6.97 – 6.94 (m, 2H), 5.17 (s, 1H), 3.88 (s, 3H), 3.70 (s, 1H), 1.83 – 1.78 (m, 1H), 1.53 – 1.48 (m, 1H), 1.29 – 1.21 (m, 2H), 0.96 (s, 3H), 0.89 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 205.1, 199.3, 164.4, 131.2, 128.4, 114.1, 75.8, 55.6, 53.4, 34.9, 17.3, 15.3, 14.7. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{15}H_{21}O_4$ 265.1434, Found 265.1434. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 20.5 min (major) and 44.8 min (minor).

6p from condition B, colorless oil, 94 mg, 71% yield, 95/88% ee, *anti*/*syn* =60:40, $[\alpha]_D^{21}$ = + 21.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 20.1 min (major) and 44.0 min (minor).

(2*R*,3*S*)-2-ethyl-3-hydroxy-2-methyl-4-(naphthalen-2-yl)-4-oxobutanal(6q)[3]

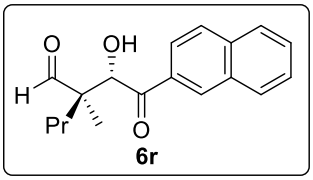


6q

6q from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 130 mg, 96% yield, 98/96% ee, *anti*/*syn* = 58:42, $[\alpha]_D^{22}$ = + 20.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.71 (s, 1H), 8.43 (s, 1H), 7.99 (d, *J* = 8.4 Hz, 1H), 7.94 (s, 2H), 7.90 (d, *J* = 8.4 Hz, 1H), 7.65 (t, *J* = 7.8 Hz, 1H), 7.60 (t, *J* = 7.2 Hz, 1H), 5.40 (d, *J* = 7.8 Hz, 1H), 3.77 (d, *J* = 8.4 Hz, 1H), 1.82 – 1.75 (m, 1H), 1.43 – 1.36 (m, 1H), 1.06 (s, 3H), 0.80 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 205.1, 200.6 136.0 133.5, 132.3, 130.8, 129.7, 129.1, 128.9, 127.9, 127.2, 124.0, 76.3, 53.5, 26.5, 14.0, 8.3. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{17}H_{19}O_3$ 271.1329, Found 271.1328. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 17.5 min (major) and 39.0 min (minor).

6q from condition B, colorless oil, 111 mg, 82% yield, 96/95% ee, *anti*/*syn* = 53:47, $[\alpha]_D^{22}$ = + 30.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 17.4 min (major) and 38.9 min (minor).

(*R*)-2-((*S*)-1-hydroxy-2-(naphthalen-2-yl)-2-oxoethyl)-2-methylpentanal(6r)[3]

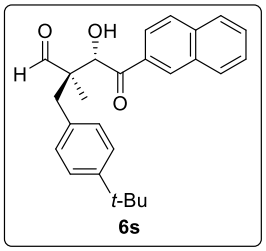


6r

6r from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 138 mg, 97% yield, 97/90% ee, *anti*/*syn* = 68:32, $[\alpha]_D^{22}$ = + 16.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.71 (s, 1H), 8.44 (s, 1H), 7.99 (d, *J* = 8.4 Hz, 1H), 7.94 (s, 2H), 7.89 (d, *J* = 7.8 Hz, 1H), 7.65 (t, *J* = 7.2 Hz, 1H), 7.59 (t, *J* = 6.6 Hz, 1H), 5.37 (d, *J* = 8.4 Hz, 1H), 3.77 (d, *J* = 8.4 Hz, 1H), 1.70 – 1.65 (m, 1H), 1.43 – 1.37 (m, 1H), 1.33 – 1.28 (m, 1H), 1.21 – 1.14 (m, 1H), 1.08 (s, 3H), 0.79 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 205.4, 200.5, 136.0, 133.4, 132.3, 130.9, 129.7, 129.1, 128.9, 127.9, 127.2, 124.0, 76.6, 53.2, 36.0, 17.3, 15.1, 14.6. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{18}H_{21}O_3$ 285.1485, Found 285.1486. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 14.0 min (major) and 32.8 min (minor).

6r from condition B, colorless oil, 104 mg, 73% yield, 96/90% ee, *anti*/*syn* = 69:31, $[\alpha]_D^{22}$ = + 20.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 14.2 min (major) and 33.1 min (minor).

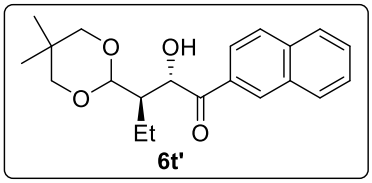
(2*R*,3*S*)-2-(4-(tert-butyl)benzyl)-3-hydroxy-2-methyl-4-(naphthalen-2-yl)-4 oxobutanal(6s)[3]



6s

6s from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 178 mg, 92% yield, 94/99% ee, *anti*/*syn* = 54:46, $[\alpha]_D^{22}$ = + 11.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 9.82 (s, 1H), 8.36 (s, 1H), 7.94 – 7.86 (m, 4H), 7.62 (t, *J* = 7.2 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.29 (d, *J* = 7.8 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 5.30 (d, *J* = 7.2 Hz, 1H), 3.86 (d, *J* = 8.4 Hz, 1H), 3.02 (d, *J* = 13.2 Hz, 1H), 2.88 (d, *J* = 13.2 Hz, 1H), 1.30 (s, 9H), 1.05 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 206.5, 200.2, 149.8, 136.0, 133.3, 132.4, 132.3, 131.1, 130.3, 129.8, 129.1, 128.9, 127.9, 127.1, 125.3, 124.1, 76.8, 53.0, 39.4, 34.5, 31.4, 16.3. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_{26}H_{29}O_3$ 389.2111, Found 389.2110. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 90/10, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 12.6 min (major) and 28.1 min (minor).

(2*S*,3*R*)-3-(5,5-dimethyl-1,3-dioxan-2-yl)-2-hydroxy-1-(naphthalen-2-yl)pentan-1-one(6t')

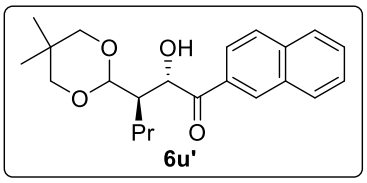


6t'

6t' from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 150 mg, 88% yield, 77/76% ee, *anti*/*syn* = 61:39, $[\alpha]_D^{24}$ = + 5.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 8.59 (s, 1H), 8.04 – 8.03 (m, 1H), 7.96 (d, *J* = 7.8 Hz, 1H), 7.91 (d, *J* = 9.0 Hz, 1H), 7.89 – 7.87 (m, 1H), 7.63 – 7.59 (m, 1H), 7.58 – 7.54 (m, 1H), 5.75 (s, 1H), 4.61 (d, *J* = 5.4 Hz, 1H), 3.78 (dd, *J* = 10.8, 2.4 Hz, 1H), 3.71 (dd, *J* = 10.8, 2.4 Hz, 1H), 3.51 (dd, *J* = 11.4, 6.0 Hz, 2H), 2.20 – 2.16 (m, 1H), 1.49 – 1.38 (m, 2H), 1.28 (s, 3H), 0.77 (s, 3H), 0.71 (t, *J* = 7.8 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 202.1, 135.8, 132.4, 130.9, 129.7, 128.7, 128.6, 127.8, 126.8, 124.3, 102.8, 77.5, 72.4, 48.8, 30.3, 23.2, 21.8, 17.0, 12.5. HRMS (ESI-Orbitrap) m/z: $[M+Na]^+$ Calcd for $C_{21}H_{26}O_4Na$ 365.1723, Found 365.1718. The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 14.1 min (minor) and 19.1 min (major).

6t' from condition B, colorless oil, 142 mg, 83% yield, 51/53% ee, *anti*/*syn* = 63:37, $[\alpha]_D^{24}$ = + 2.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK AS-H, Hexane/2-propanol = 95/5, flow rate 1.0 mL/min, 254 nm, retention time : major isomer 13.8 min (minor) and 18.3 min (major).

(2*S*,3*R*)-3-(5,5-dimethyl-1,3-dioxan-2-yl)-2-hydroxy-1-(naphthalen-2-yl)hexan-1-one(6u')



6u'

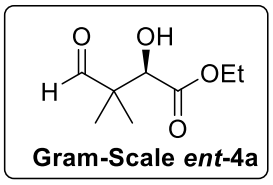
6u' from condition A, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 150 mg, 84% yield, 81/80%ee, *anti*/*syn* = 73:27, $[\alpha]_D^{24}$ = + 5.0 (*c* 1.0, $CHCl_3$). ^1H NMR (600 MHz, $CDCl_3$) δ 8.59 (s, 1H), 8.04 (dd, *J* = 8.4, 1.8Hz, 1H), 7.96 (d, *J* = 7.8 Hz, 1H), 7.93 – 7.90 (m, 1H), 7.89 – 7.87 (m, 1H), 7.62 – 7.54 (m, 2H), 5.75 (d, *J* = 1.2 Hz, 1H), 4.59 (d, *J* = 5.4 Hz, 1H), 3.78 (dd, *J* = 11.4, 3.0 Hz, 1H), 3.71 (dd, *J* = 10.8, 3.0 Hz, 1H), 3.51 (dd, *J* = 11.4, 7.2 Hz, 2H), 2.26 – 2.23 (m, 1H), 1.43 – 1.31 (m, 2H), 1.28 (s, 3H), 1.15 – 1.07 (m, 2H), 0.77 (s, 3H), 0.68 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 202.0, 135.8, 132.4, 130.9, 129.8, 128.7, 128.6, 127.8, 126.8, 124.3, 103.0, 77.7, 72.4, 47.0, 30.3, 26.2, 23.2, 21.8, 20.9, 14.3. HRMS (ESI-Orbitrap) m/z: $[M+Na]^+$ Calcd for $C_{22}H_{28}O_4Na$ 379.1880, Found 379.1876. The *ee* was determined by HPLC analysis. CHIRALPAK OD-H, Hexane/2-propanol = 96/4, flow rate 0.5 mL/min, 254 nm, retention time : major isomer 11.5 min (major) and 17.4 min (minor).

6u' from condition B, colorless oil, 142 mg, 80% yield, 53/56%ee, *anti*/*syn* = 71:29, $[\alpha]_D^{24}$ = + 9.0 (*c* 1.0, $CHCl_3$). The *ee* was determined by HPLC analysis. CHIRALPAK OD-H,

Hexane/2-propanol = 96/4, flow rate 0.5 mL/min, 254 nm, retention time : major isomer 11.5 min (major) and 17.1 min (minor).

4.2 Products of Gram-Scale Reaction

(*R*)-Ethyl-2-hydroxy-3, 3-dimethyl-4-oxobutanoate (4a)[3]



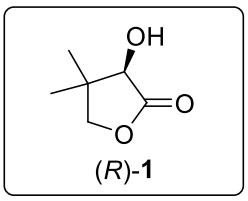
Gram-Scale *ent*-4a

Gram-Scale *ent*-4a from condition A, colorless oil, 1.61g, 93% yield, 98% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 7.2 min (major) and 10.0 min (minor).

Gram-Scale *ent*-4a from condition B, colorless oil, 1.42 g, 82% yield, 95% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 7.1 min (major) and 9.8 min (minor).

4.3 Products of Derivatization

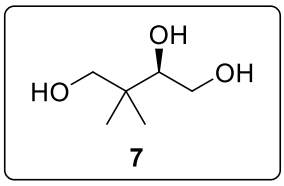
(*R*)-3-Hydroxy-4,4-dimethyldihydrofuran-2(3H)-one ((*R*)-1)[3]



(*R*)-**1**

(*R*)-**1**, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 4 : 1), white soild, mp: 90.3 – 94.0 °C, 120 mg, 92% yield, 98% ee, $[\alpha]_D^{24} = -15.0$ (c 1.0, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.15 (s, 1H), 4.03 (d, *J* = 9.0 Hz, 1H), 3.95 (d, *J* = 9.0 Hz, 1H), 3.21 (s, 1H), 1.24 (s, 3H), 1.09 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.7, 76.4, 75.8, 40.9, 22.9, 18.8. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_6H_{11}O_3$ 103.0703, Found 103.0706. The ee was determined with the *p*-nitrobenzoate of (*R*)-**1** by chiral HPLC. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 10.6 min (major) and 13.6 min (minor).

(*R*)-3,3-dimethylbutane-1,2,4-triol (7)[4]

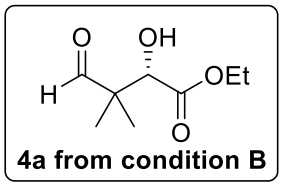


7

7, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 1 : 1), colorless oil, 129 mg, 96% yield, $[\alpha]_D^{24} = -9.0$ (c 1.0, $CHCl_3$). ^{1}H NMR (600 MHz, DMSO-d_6) δ 4.41 (t, *J* = 5.4 Hz, 1H), 4.34 – 4.32 (m, 1H), 4.27 (d, *J* = 4.2 Hz, 1H), 3.48 – 3.45 (m, 1H), 3.28 – 3.22 (m, 2H), 3.19 (dd, *J* = 10.8, 5.4 Hz, 1H), 3.13 (dd, *J* = 10.8, 5.4 Hz, 1H), 0.74 (d, *J* = 1.8 Hz, 6H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 76.4, 68.6, 62.9, 38.7, 21.6, 20.9. HRMS (ESI-Orbitrap) m/z: $[M+H]^+$ Calcd for $C_6H_{15}O_3$ 135.1016, Found 135.1018.

4.4 Products of Reusability of RTP-1

(*S*)-Ethyl-2-hydroxy-3, 3-dimethyl-4-oxobutanoate (4a)



Cycle 1, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 83.5 mg, 96% yield, 96% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.9 min (minor) and 9.4 min (major).

Cycle 2, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 84 mg, 97% yield, 96% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.8 min (minor) and 9.3 min (major).

Cycle 3, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 85 mg, 98% yield, 96% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.8 min (minor) and 9.2 min (major).

Cycle 4, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 85 mg, 98% yield, 96% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.8 min (minor) and 9.1 min (major).

Cycle 5, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 85 mg, 98% yield, 96% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.7 min (minor) and 9.0 min (major).

Cycle 6, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 83 mg, 96% yield, 95% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.7 min (minor) and 9.0 min (major).

Cycle 7, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 84.7 mg, 97% yield, 95% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.7 min (minor) and 9.0 min (major).

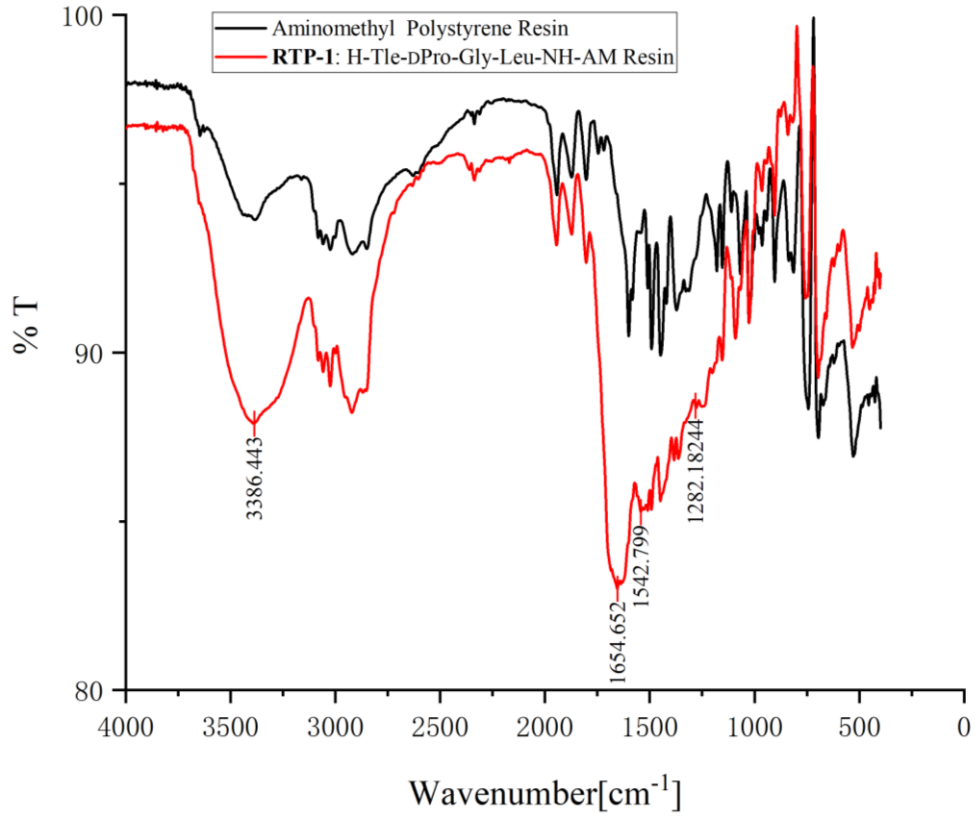
Cycle 8, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 81 mg, 93% yield, 95% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254

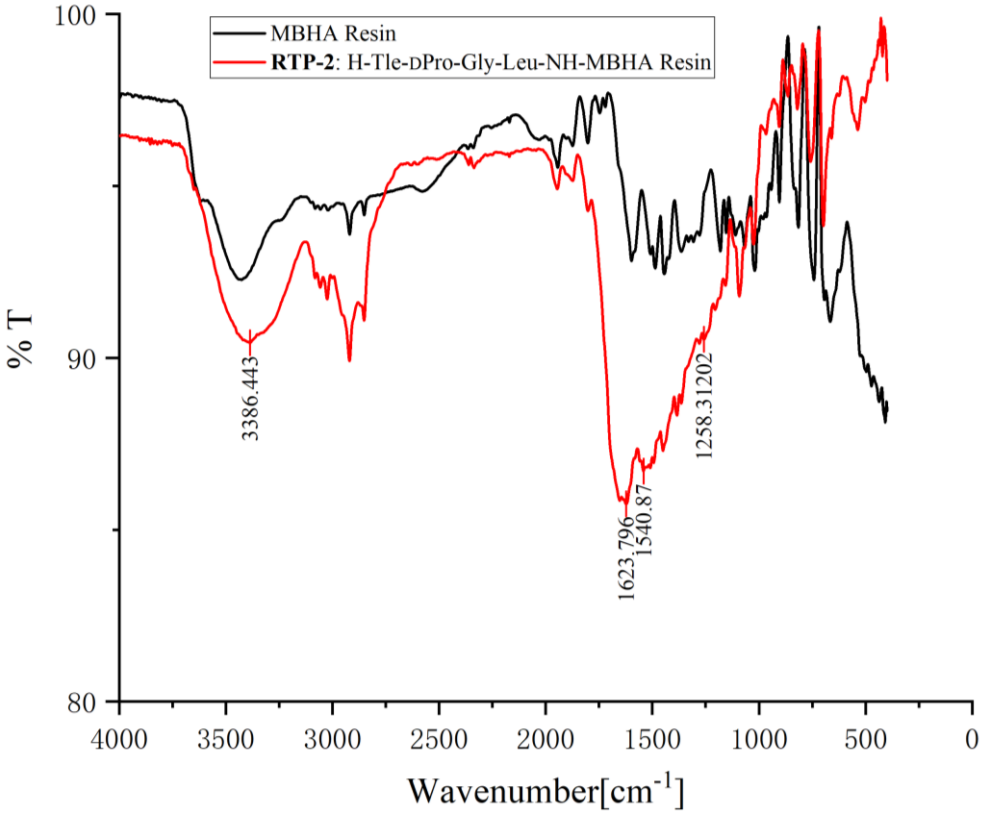
nm, retention time : 6.7 min (minor) and 8.9 min (major).

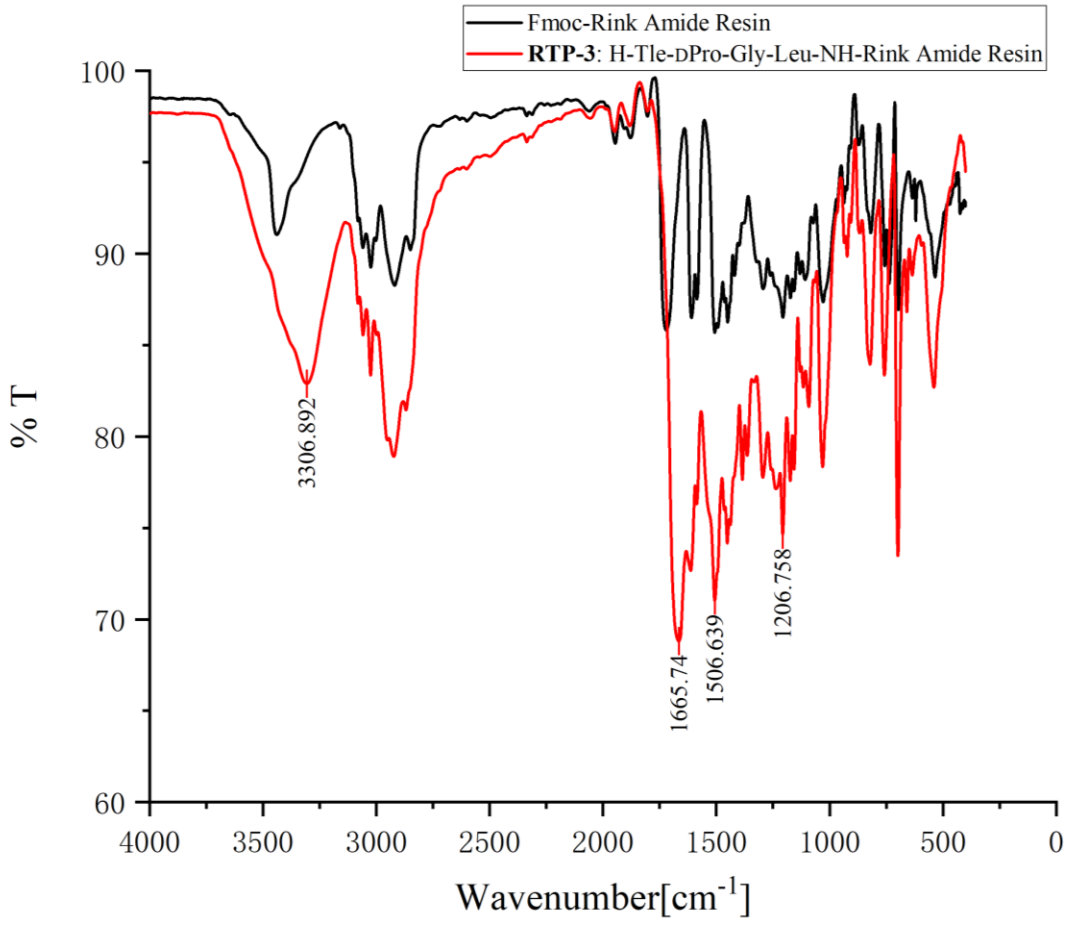
Cycle 9, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 67 mg, 77% yield, 95% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.7 min (minor) and 8.9 min (major).

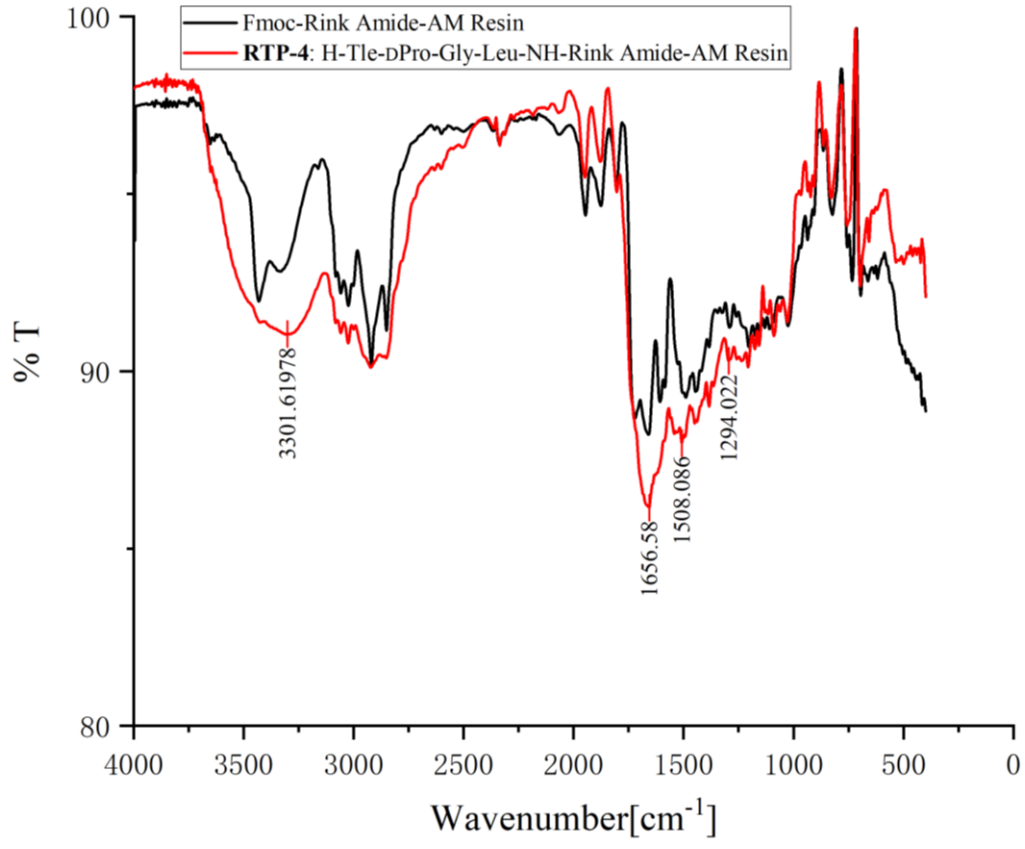
Cycle 10, the title compound was purified by column chromatography on silica gel (petroleum ether: EtOAc = 10 : 1), colorless oil, 52 mg, 60% yield, 94% ee. The *ee* was determined by HPLC analysis. CHIRALPAK AD-H, Hexane/2-propanol = 70/30, flow rate 1.0 mL/min, 254 nm, retention time : 6.7 min (minor) and 9.0 min (major).

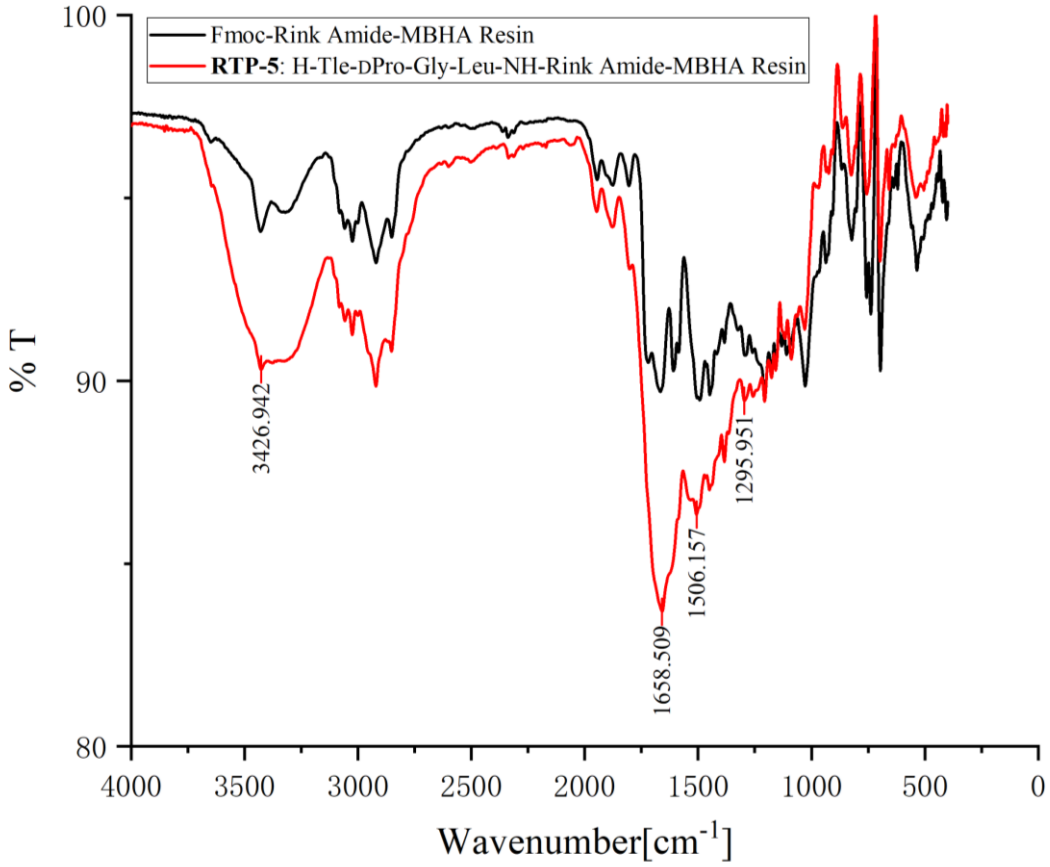
5. Copies of FT-IR spectra of resin-supported peptides

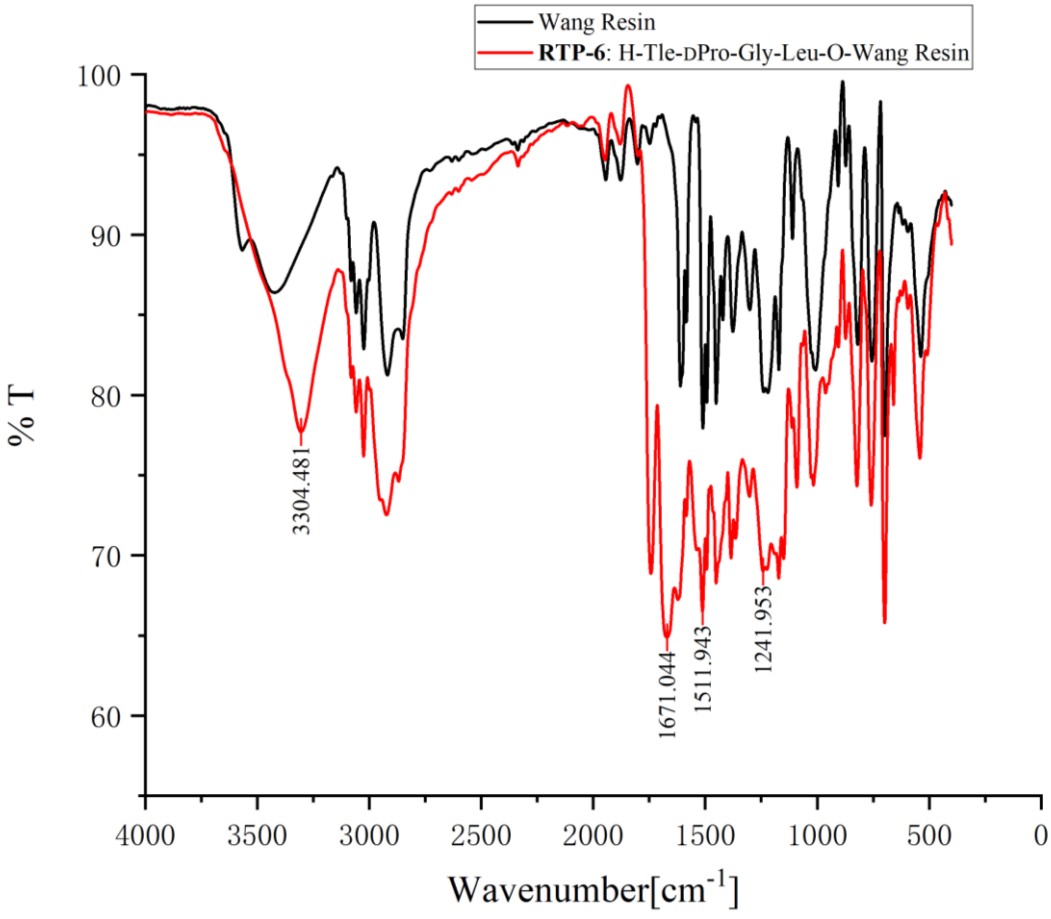


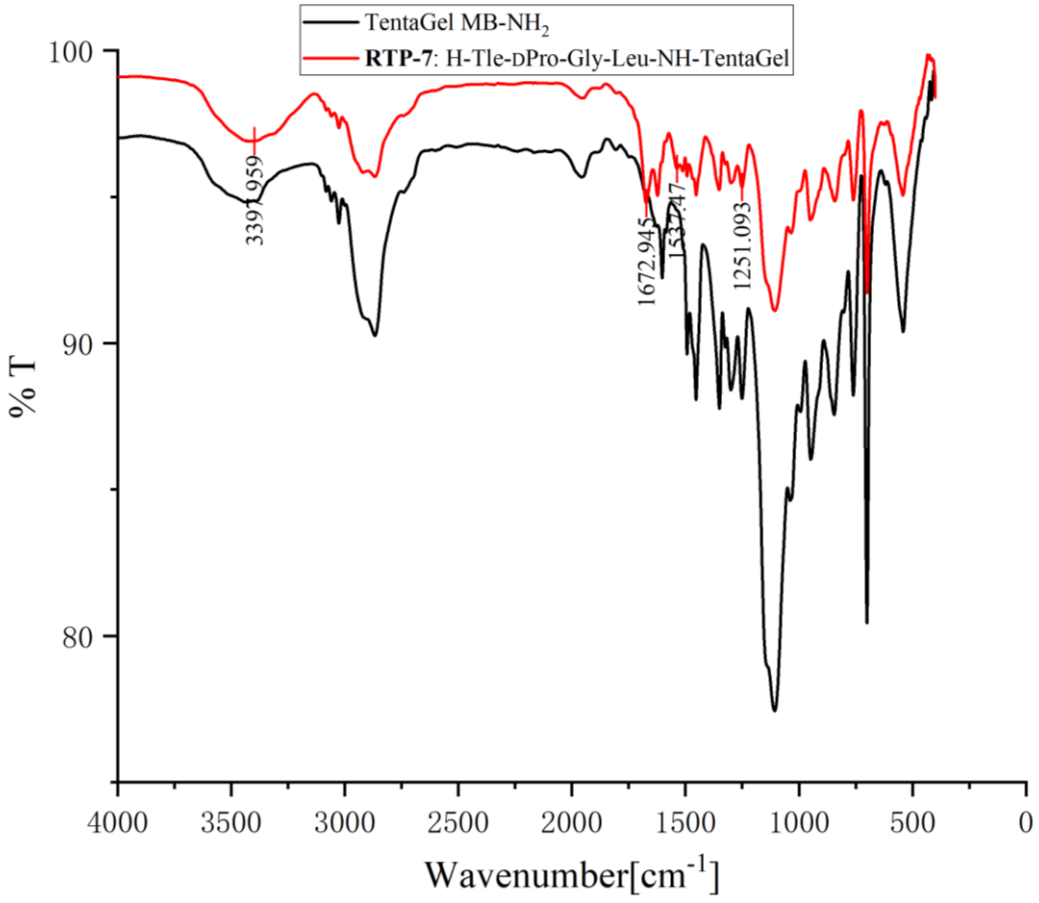


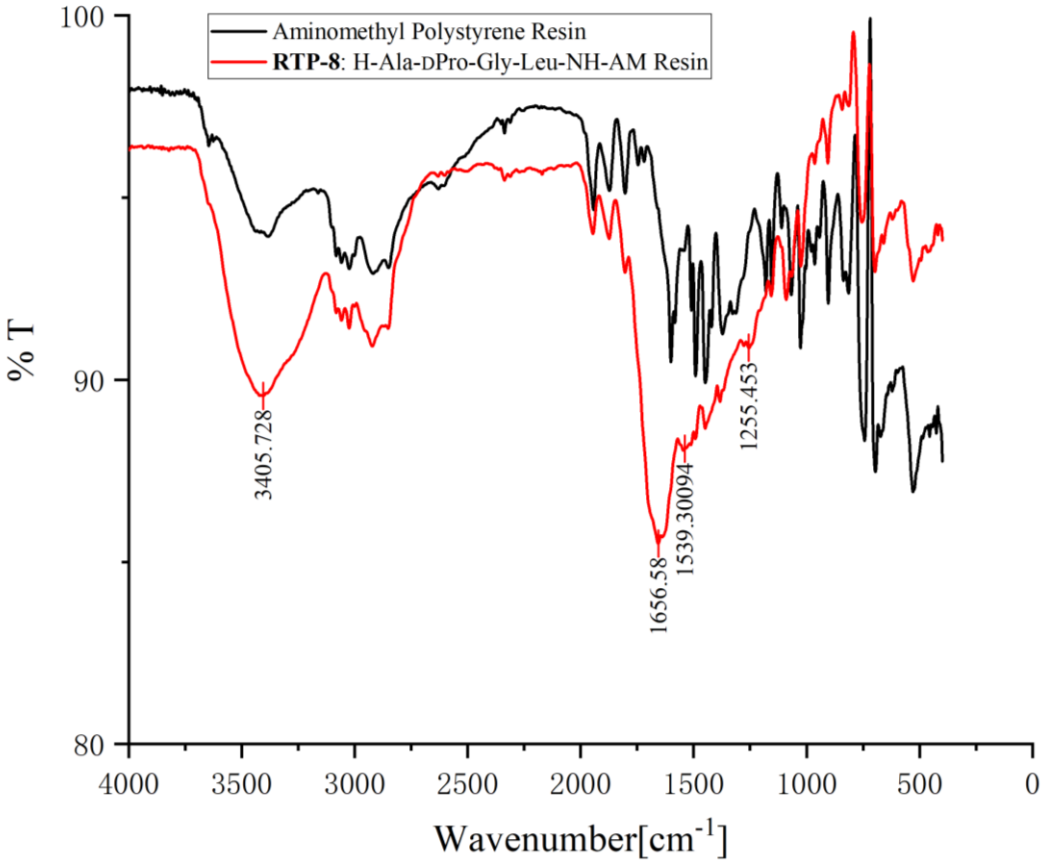

Fmoc-Rink Amide Resin
RTP-3: H-Tle-DPro-Gly-Leu-NH-Rink Amide Resin
100
90
80
70
60
% T
3306.892
1665.74
1506.639
1206.758
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

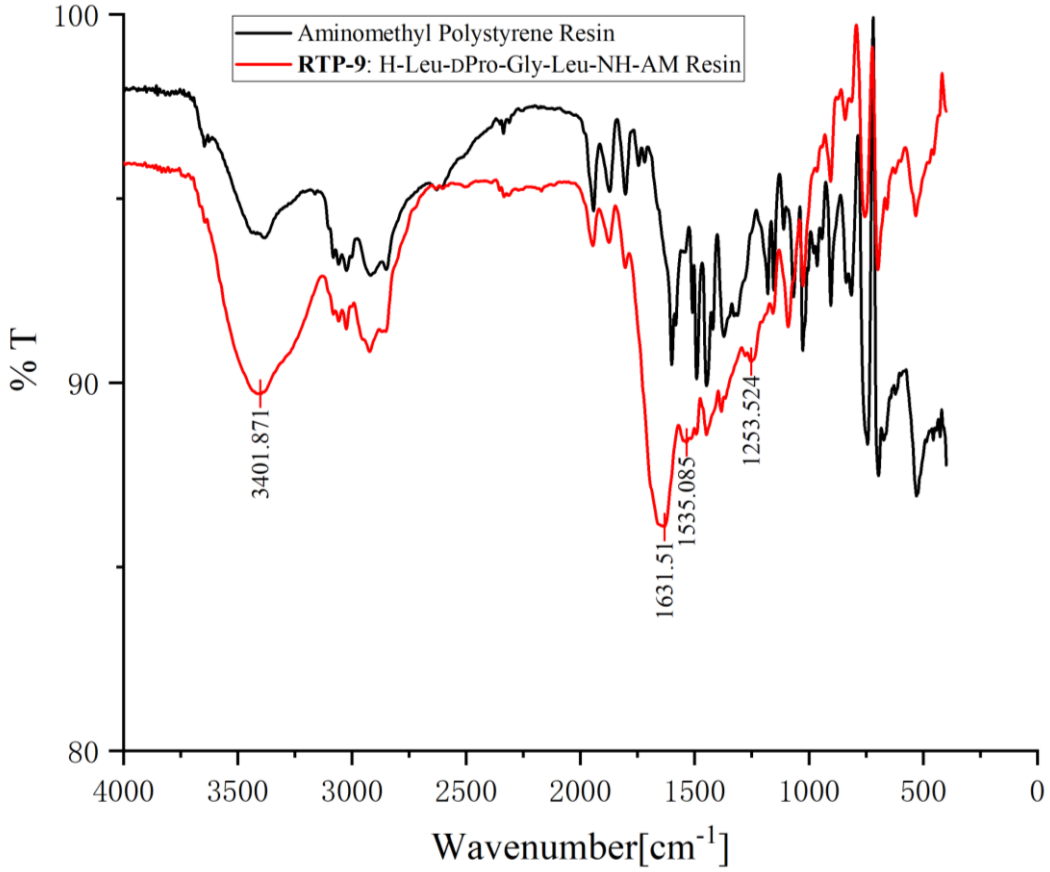

Fmoc-Rink Amide-AM Resin
RTP-4: H-Tle-DPro-Gly-Leu-NH-Rink Amide-AM Resin
100
90
80
% T
3301.61978
1656.58
1508.086
1294.022
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

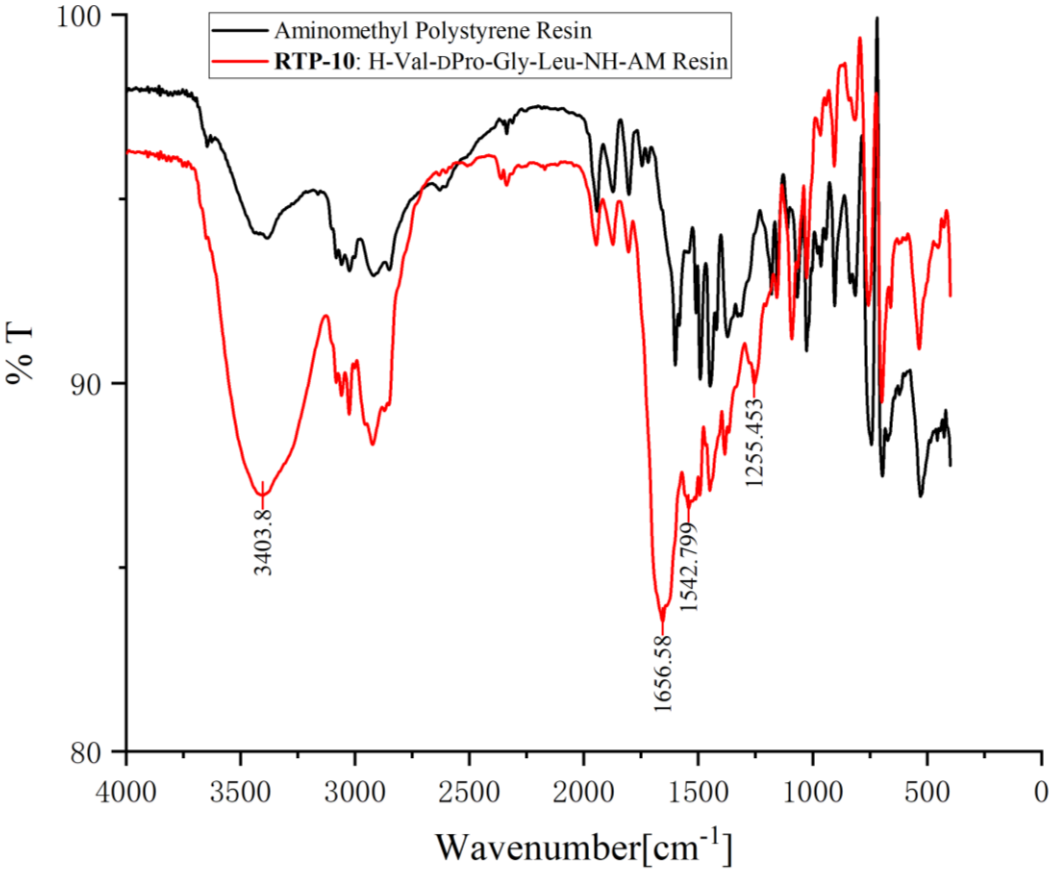

Fmoc-Rink Amide-MBHA Resin
RTP-5: H-Tle-DPro-Gly-Leu-NH-Rink Amide-MBHA Resin
% T
100
90
80
3426.942
1658.509
1506.157
1295.951
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

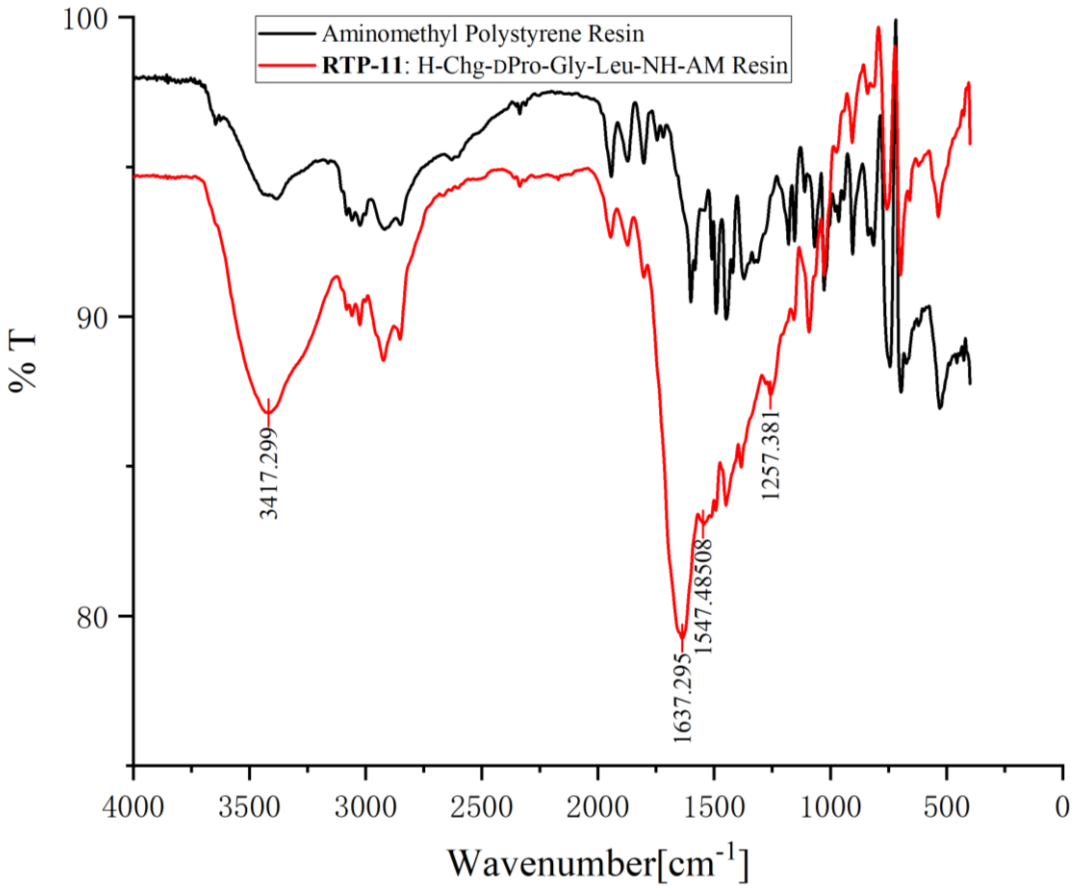

Wang Resin
RTP-6: H-Tle-DPro-Gly-Leu-O-Wang Resin
% T
100
90
80
70
60
3304.481
1671.044
1511.943
1241.953
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

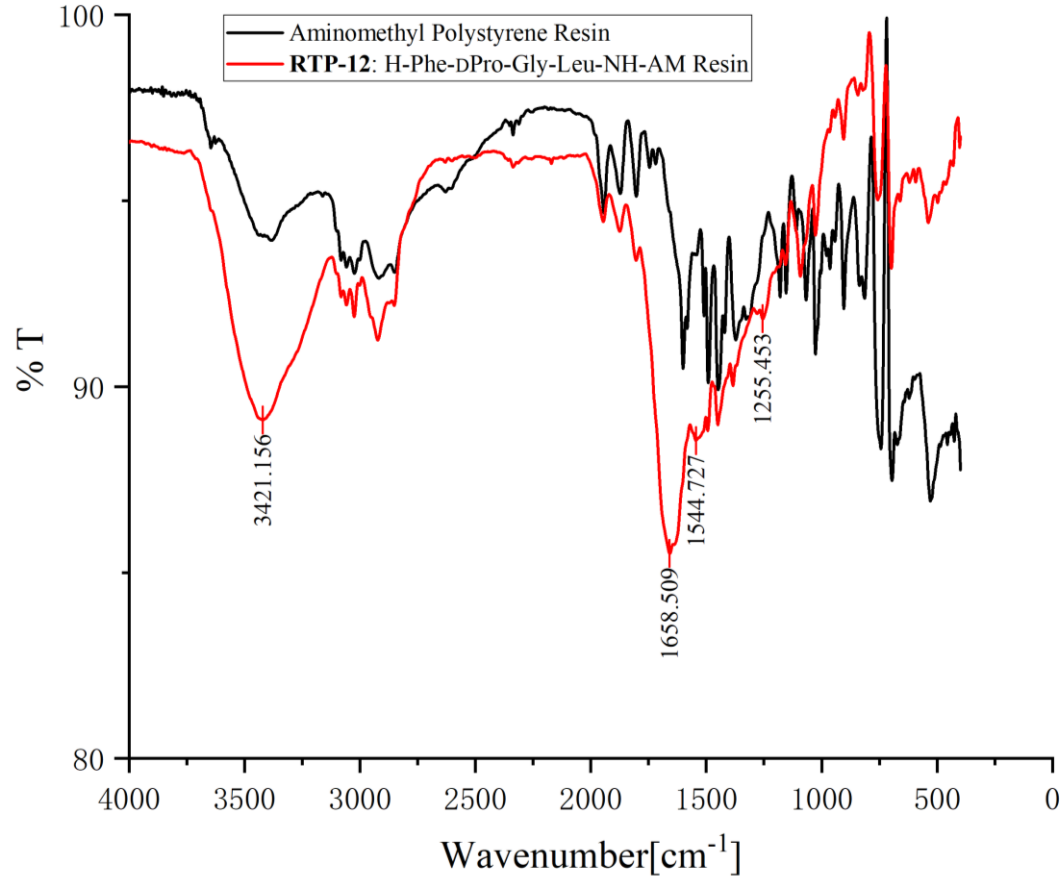

TentaGel MB-NH2
RTP-7: H-Tle-DPro-Gly-Leu-NH-TentaGel
100
90
80
% T
3391.959
1672.945
1537.247
1251.093
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

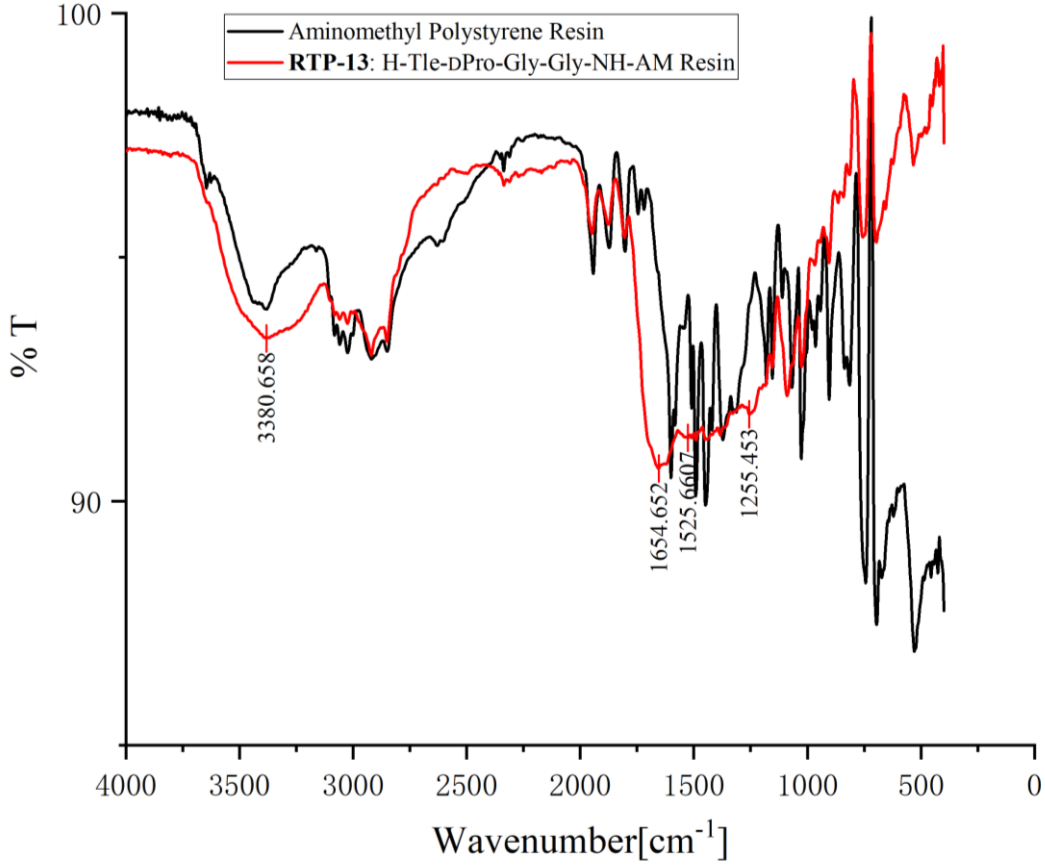

Aminomethyl Polystyrene Resin
RTP-8: H-Ala-DPro-Gly-Leu-NH-AM Resin
100
90
80
% T
3405.728
1656.58
1539.30094
1255.453
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

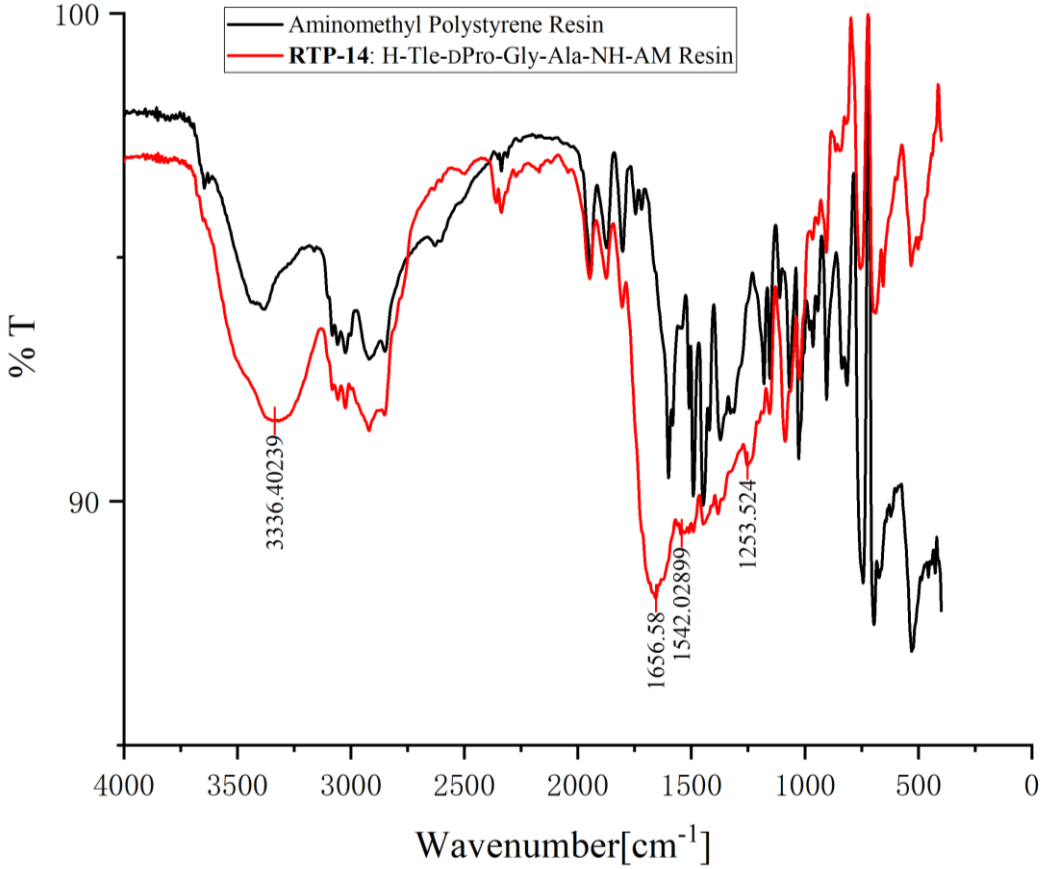

Aminomethyl Polystyrene Resin
RTP-9: H-Leu-DPro-Gly-Leu-NH-AM Resin
100
90
80
% T
3401.871
1631.51
1535.085
1253.524
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

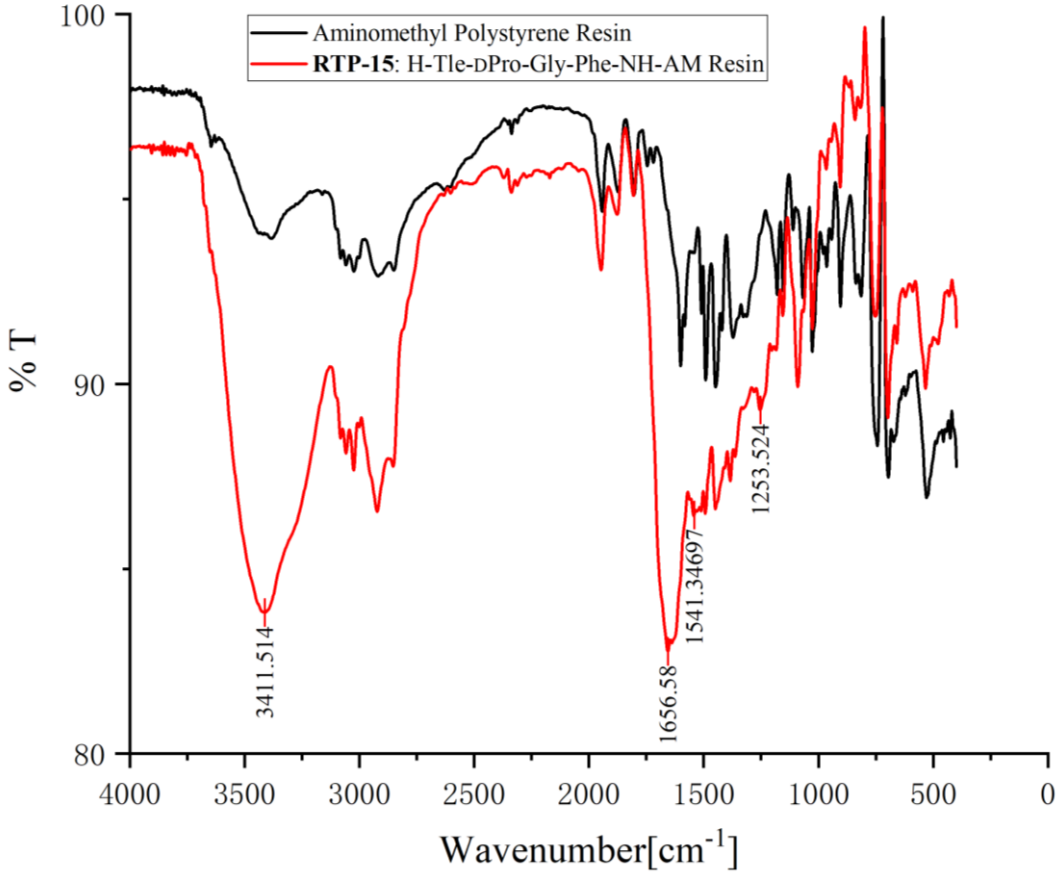

Aminomethyl Polystyrene Resin
RTP-10: H-Val-DPro-Gly-Leu-NH-AM Resin
100
90
80
% T
3403.8
1656.58
1542.799
1255.453
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

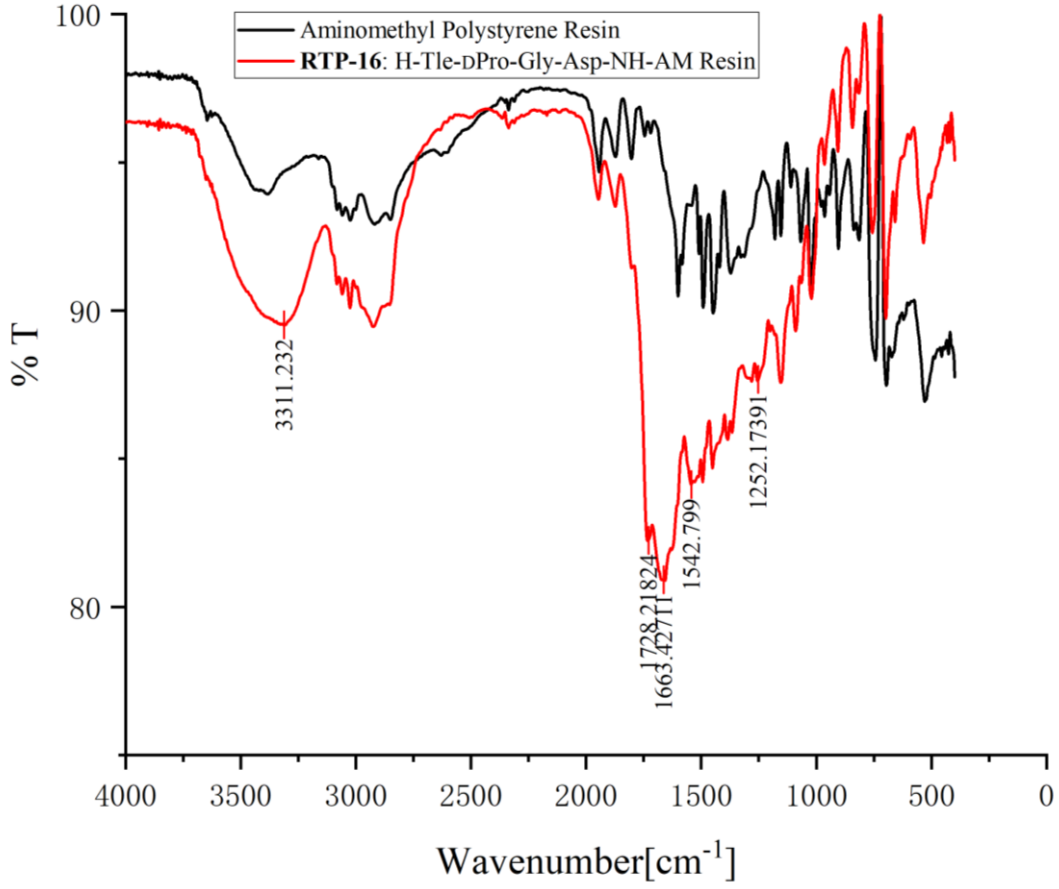

Aminomethyl Polystyrene Resin
RTP-11: H-Chg-DPro-Gly-Leu-NH-AM Resin
% T
100
90
80
3417.299
1637.295
1547.48508
1257.381
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

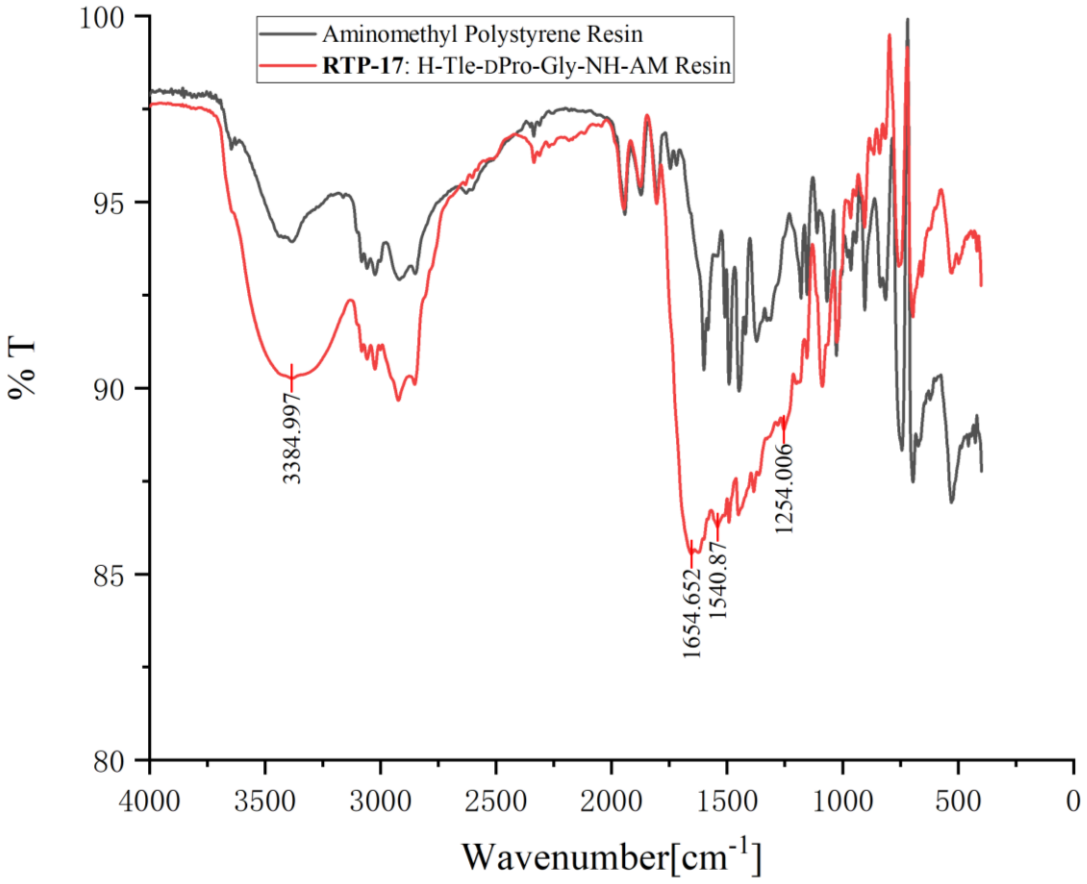

Aminomethyl Polystyrene Resin
RTP-12: H-Phe-DPro-Gly-Leu-NH-AM Resin
% T
100
90
80
3421.156
1658.509
1544.727
1255.453
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

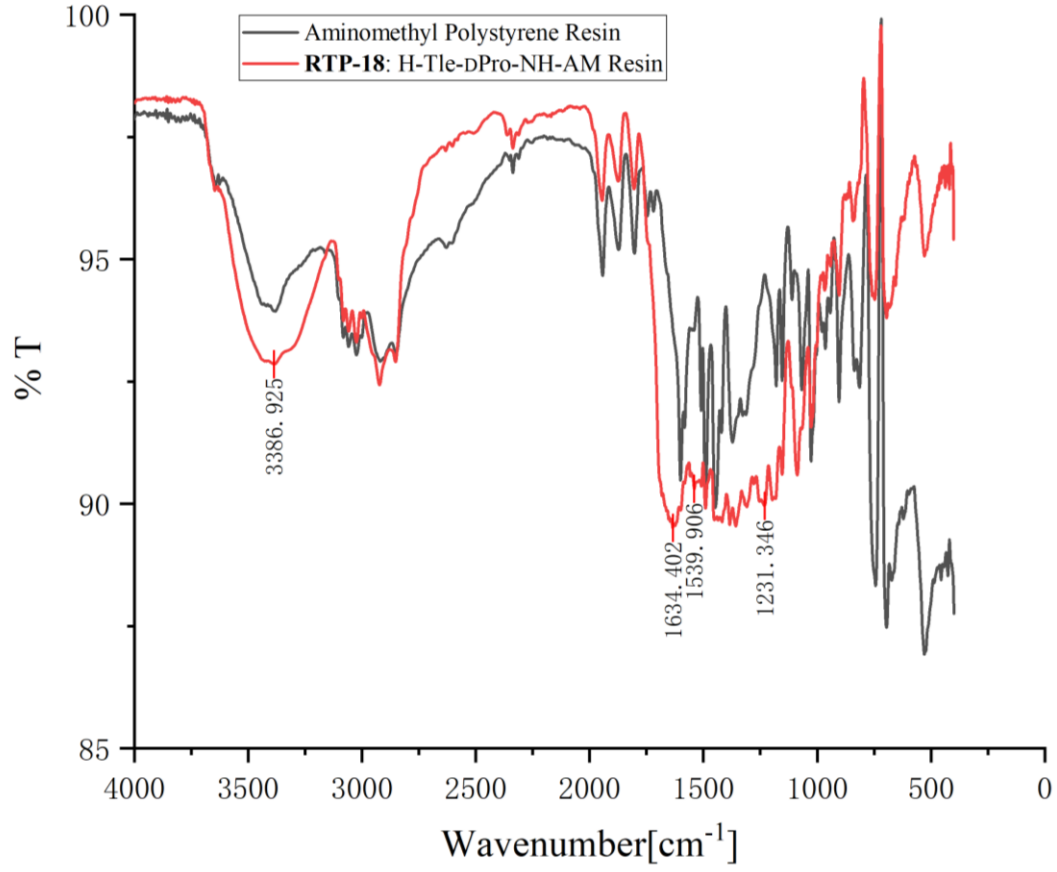

Aminomethyl Polystyrene Resin
RTP-13: H-Tle-DPro-Gly-Gly-NH-AM Resin
100
90
% T
3380.658
1654.652
1525.6607
1255.453
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

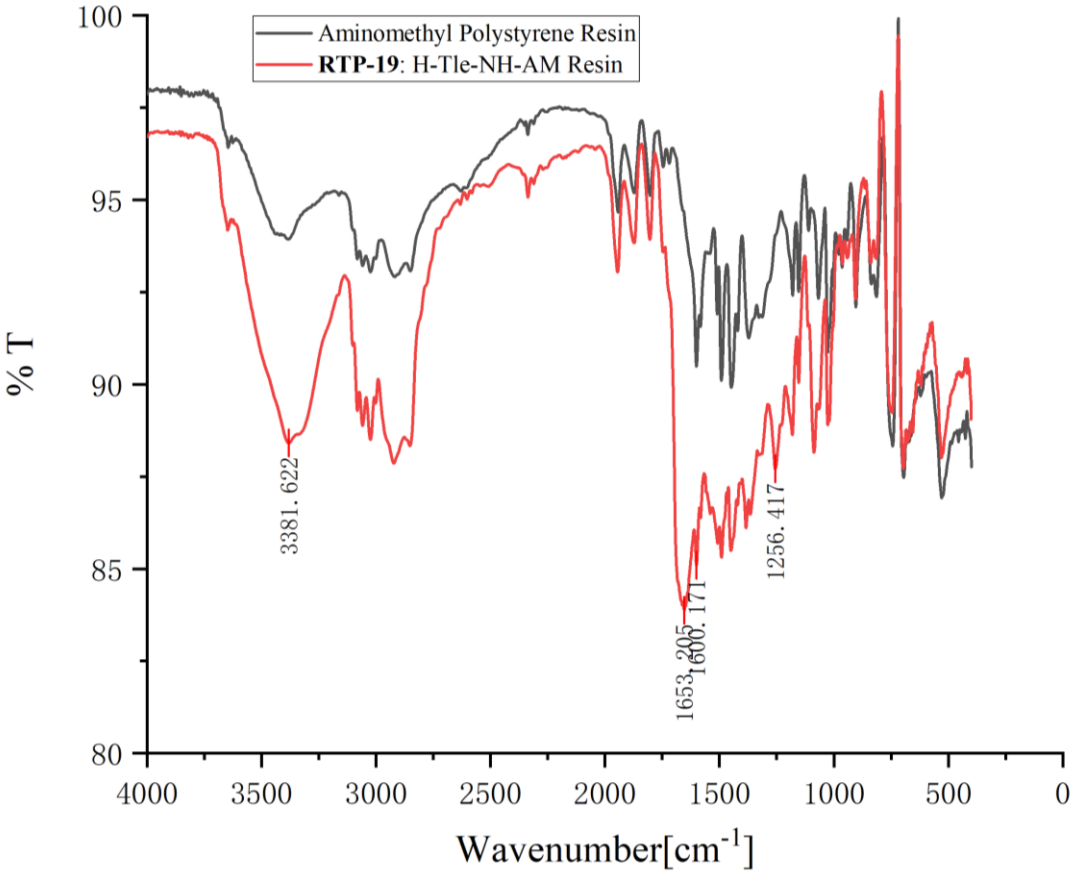

Aminomethyl Polystyrene Resin
RTP-14: H-Tle-DPro-Gly-Ala-NH-AM Resin
100
90
% T
3336.40239
1656.58
1542.02899
1253.524
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

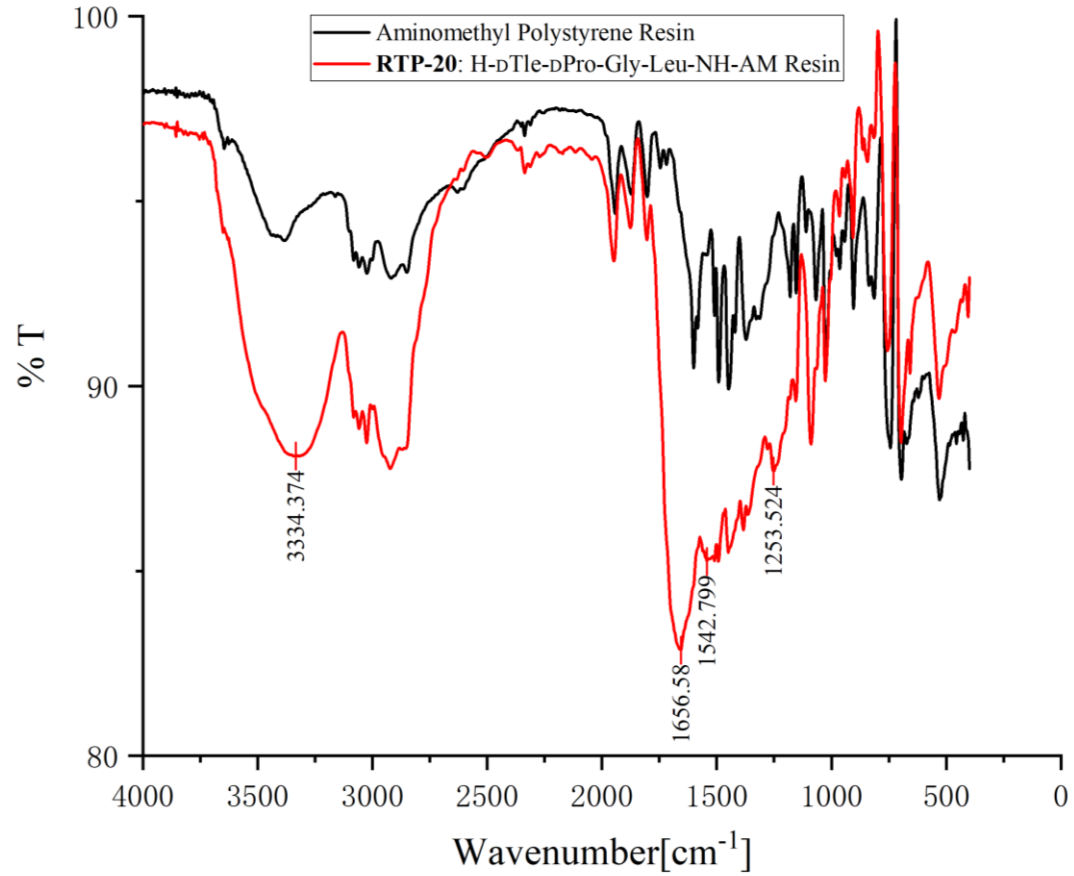

Aminomethyl Polystyrene Resin
RTP-15: H-Tle-DPro-Gly-Phe-NH-AM Resin
% T
100
90
80
3411.514
1656.58
1541.34697
1253.524
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]


Aminomethyl Polystyrene Resin
RTP-16: H-Tle-DPro-Gly-Asp-NH-AM Resin
% T
100
90
80
3311.232
1728.21824
1663.42711
1542.799
1252.17391
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

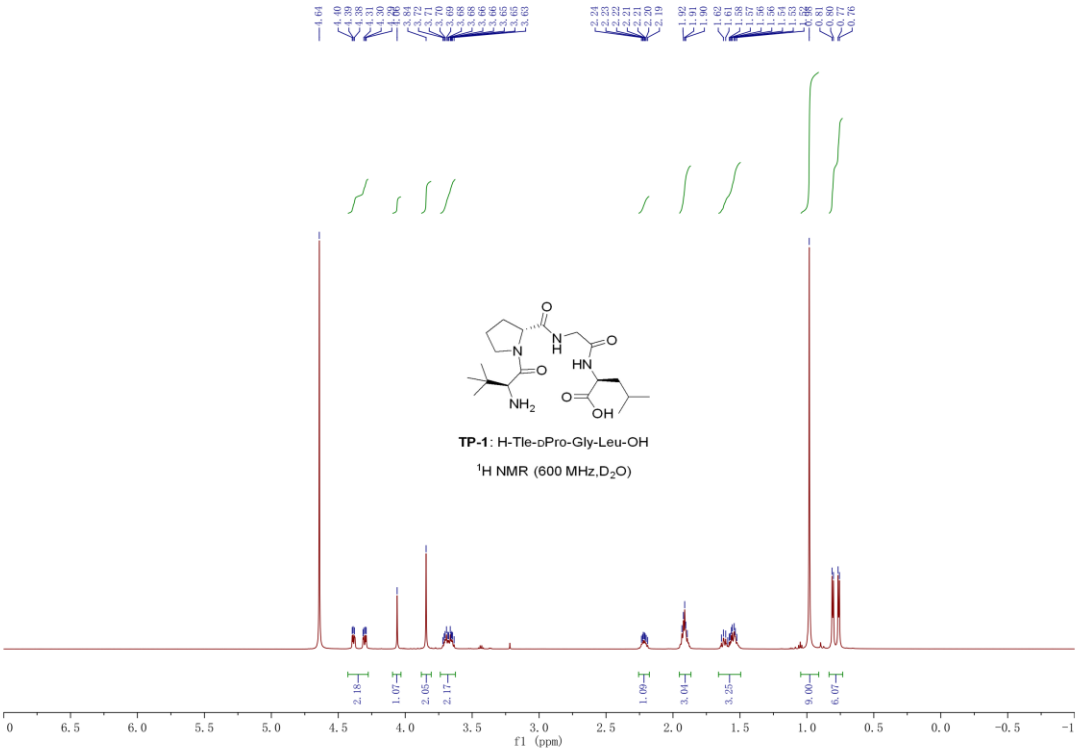

Aminomethyl Polystyrene Resin
RTP-17: H-Tle-DPro-Gly-NH-AM Resin
% T
100
95
90
85
80
3384.997
1654.652
1540.87
1254.006
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

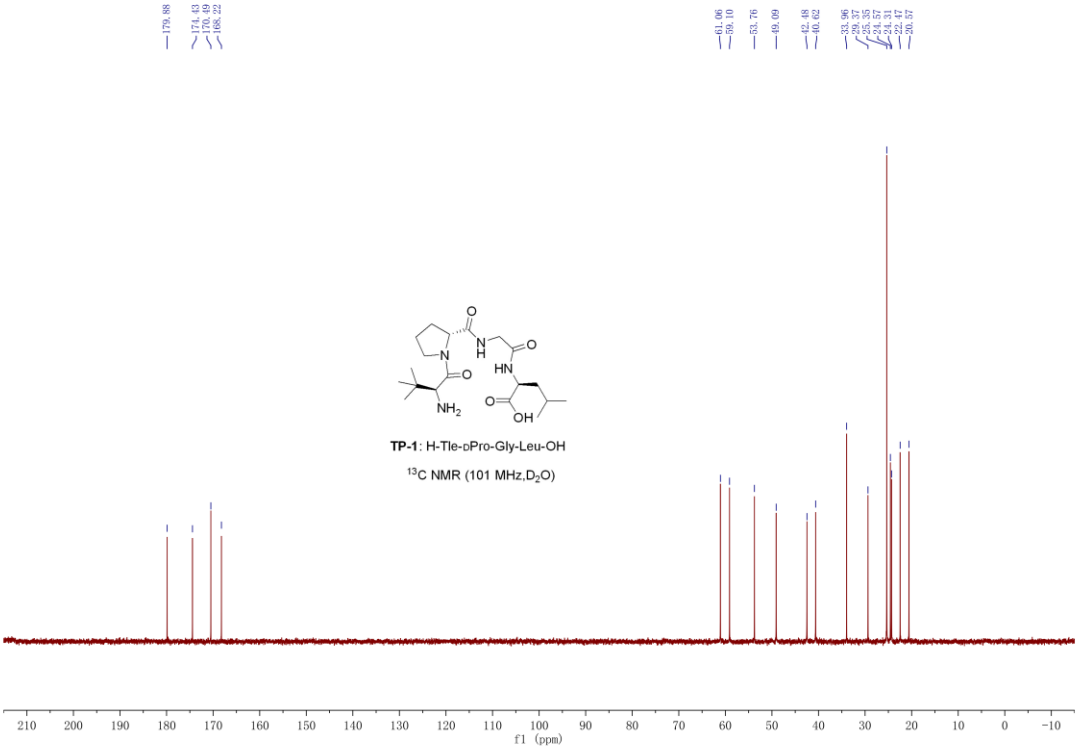

Aminomethyl Polystyrene Resin
RTP-18: H-Tle-DPro-NH-AM Resin
% T
100
95
90
85
3386. 925
1634. 402
1539. 906
1231. 346
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

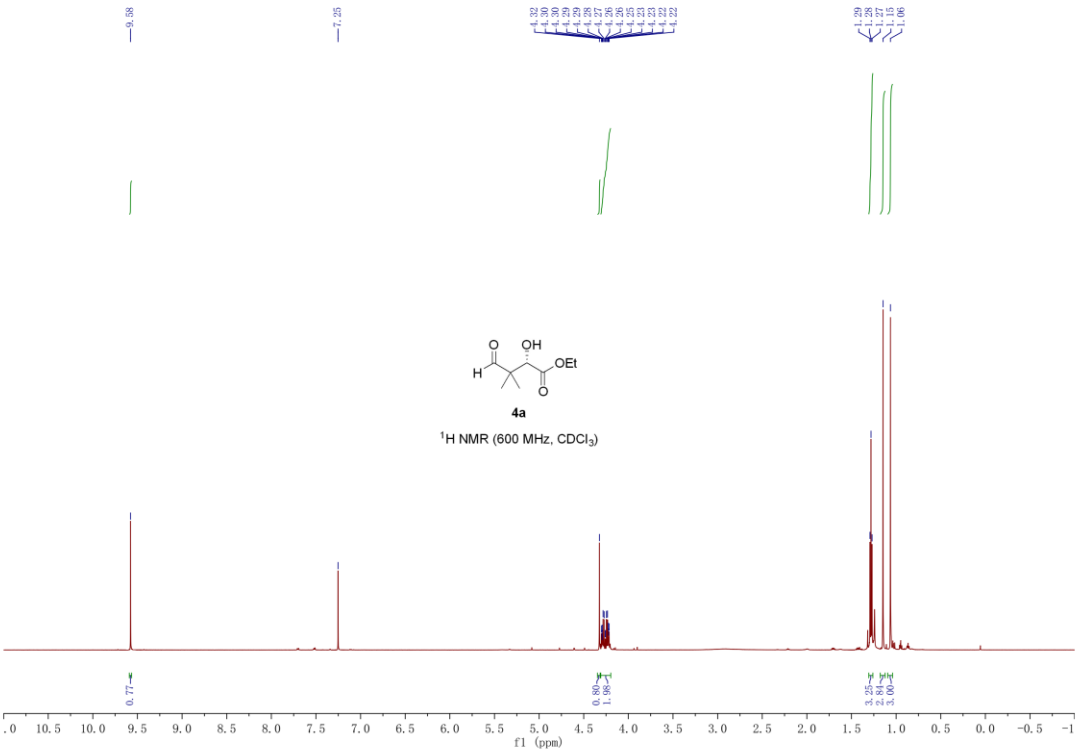

Aminomethyl Polystyrene Resin
RTP-19: H-Tle-NH-AM Resin
% T
100
95
90
85
80
3381.622
1653.205
1600.171
1256.417
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

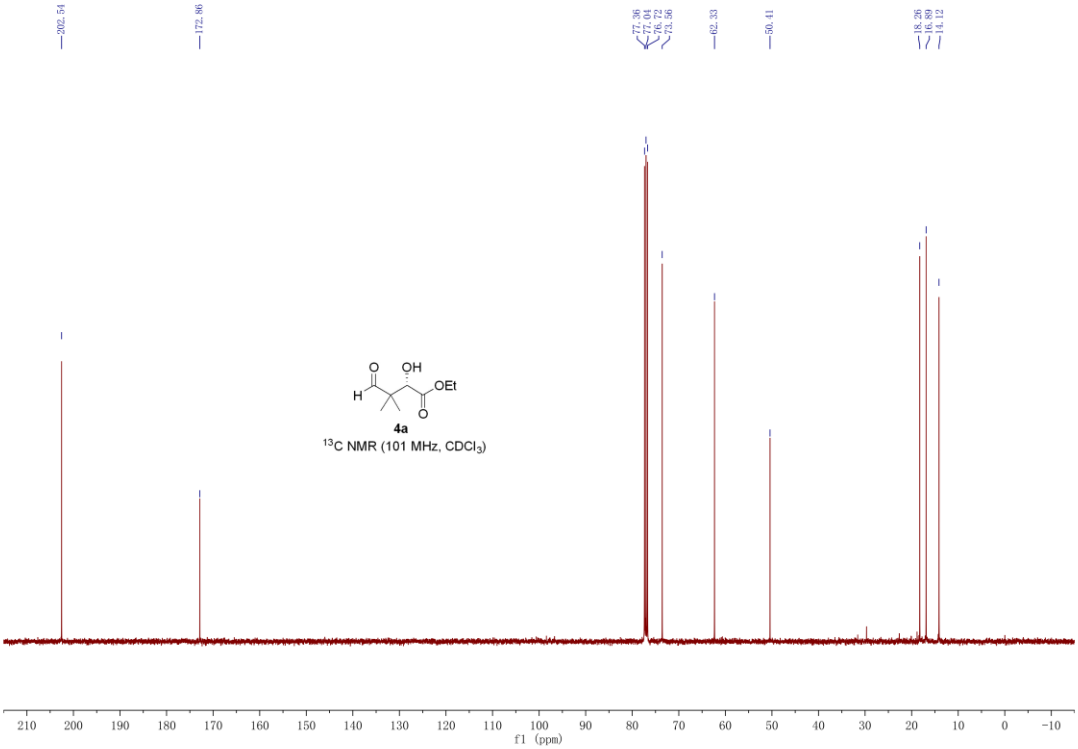

Aminomethyl Polystyrene Resin
RTP-20: H-DTle-DPro-Gly-Leu-NH-AM Resin
% T
100
90
80
3334.374
1656.58
1542.799
1253.524
4000
3500
3000
2500
2000
1500
1000
500
0
Wavenumber[cm-1]

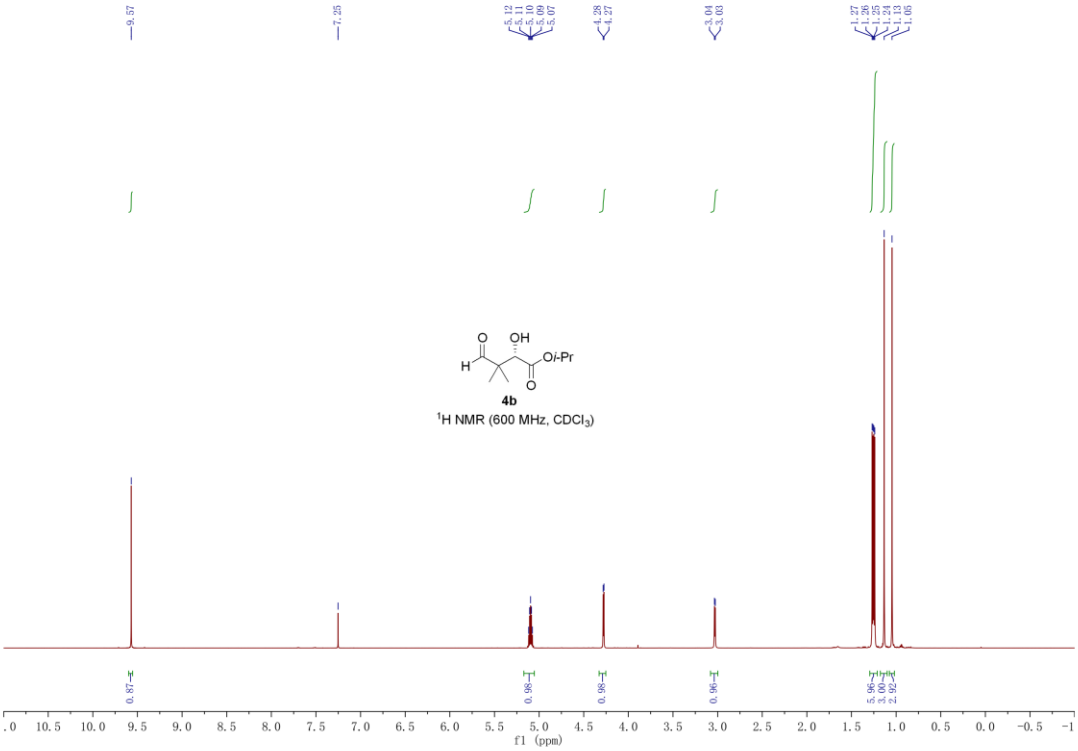
6. Copies of NMR spectra of Compounds

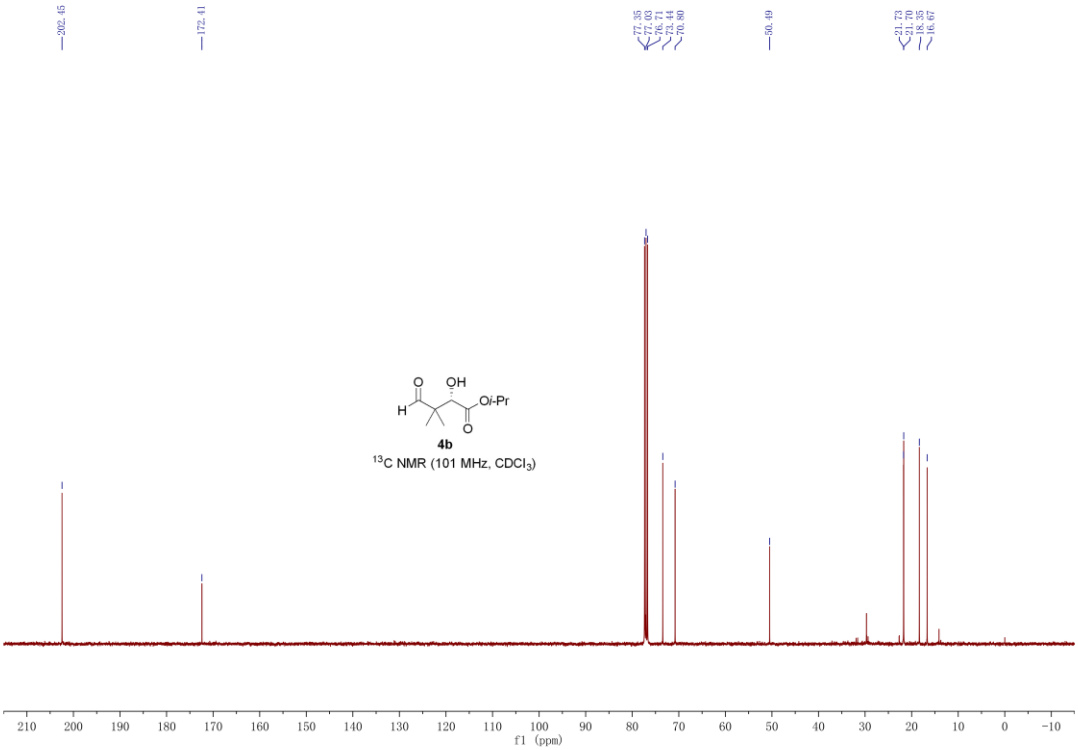


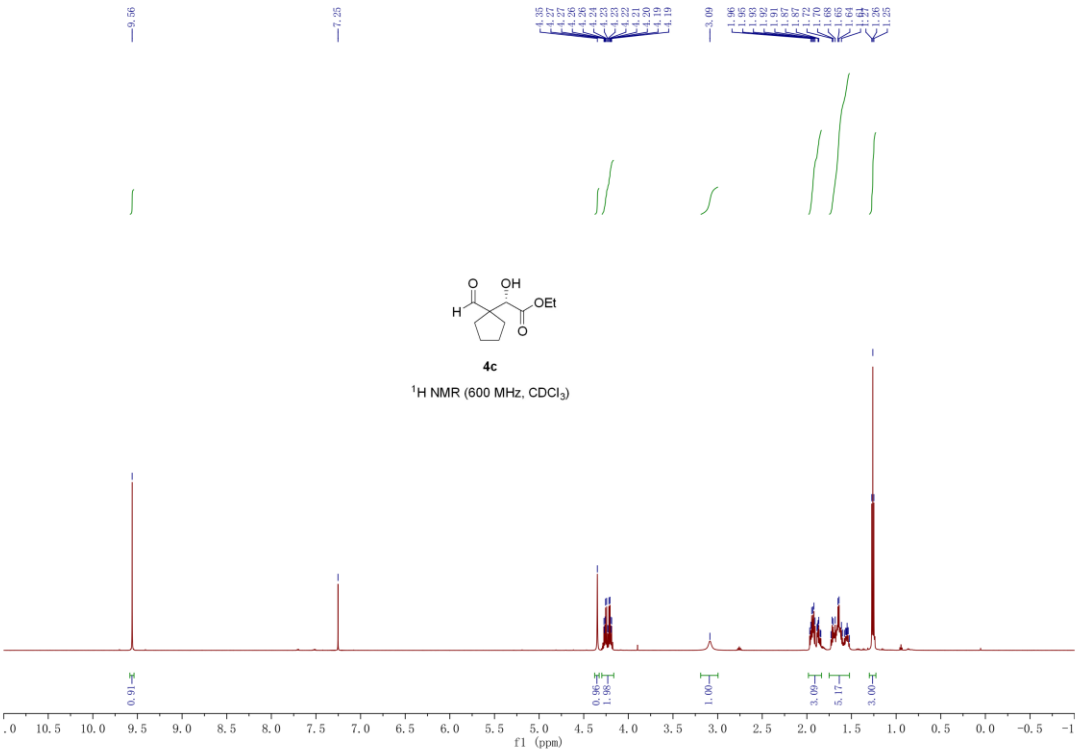


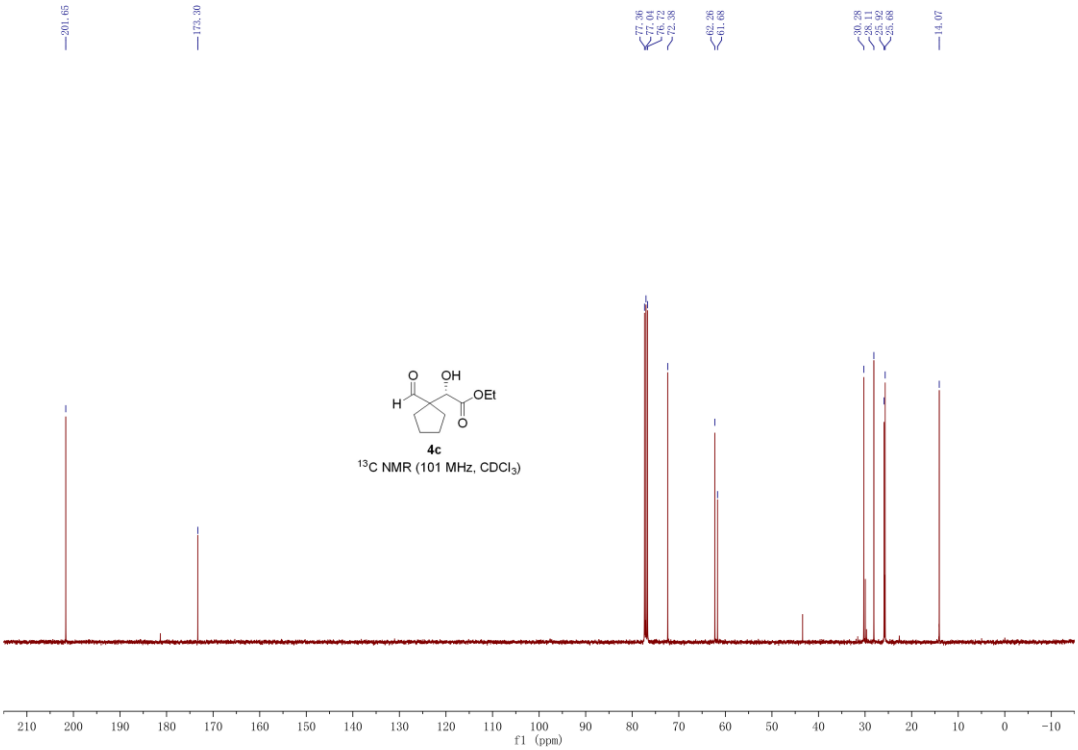

9.58
7.25
4.32
4.30
4.29
4.29
4.28
4.27
4.26
4.25
4.23
4.23
4.22
4.22
1.29
1.28
1.27
1.15
1.06
O
OH
H
OEt
O
4a
^{1}H NMR (600 MHz, CDCl$_3$)
0.77
0.80
1.98
3.25
2.84
3.00
f1 (ppm)

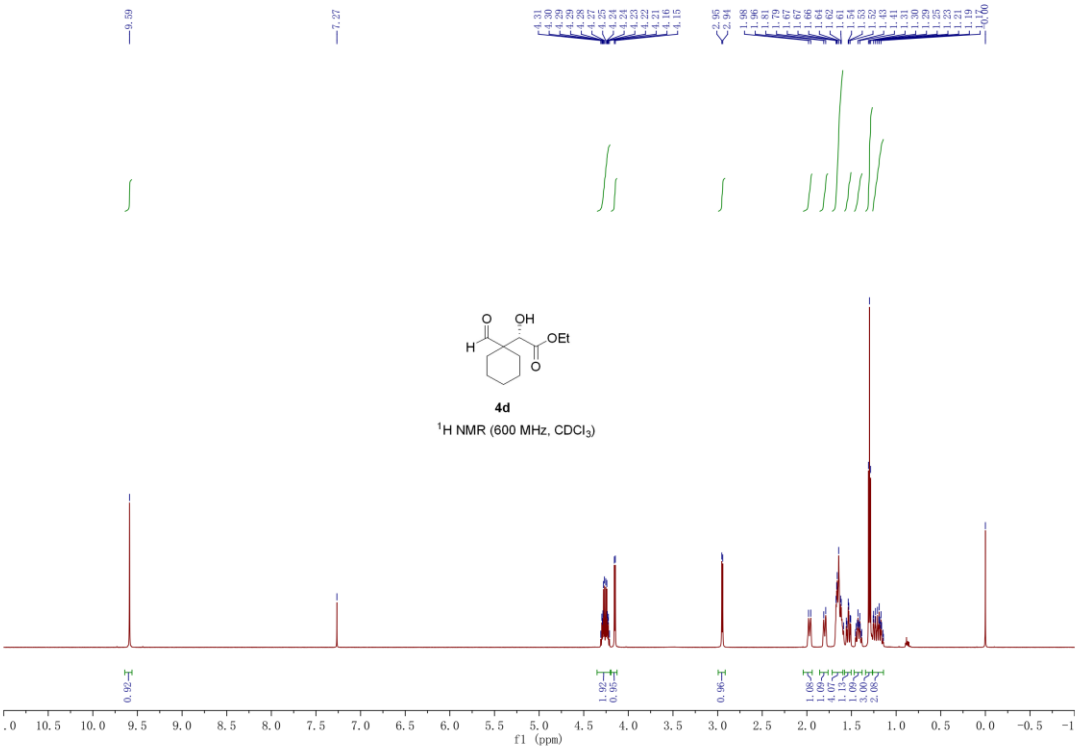

202.54
172.86
77.36
77.04
76.72
73.56
62.33
50.41
18.26
16.89
14.12
O
OH
H
OEt
O
4a
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

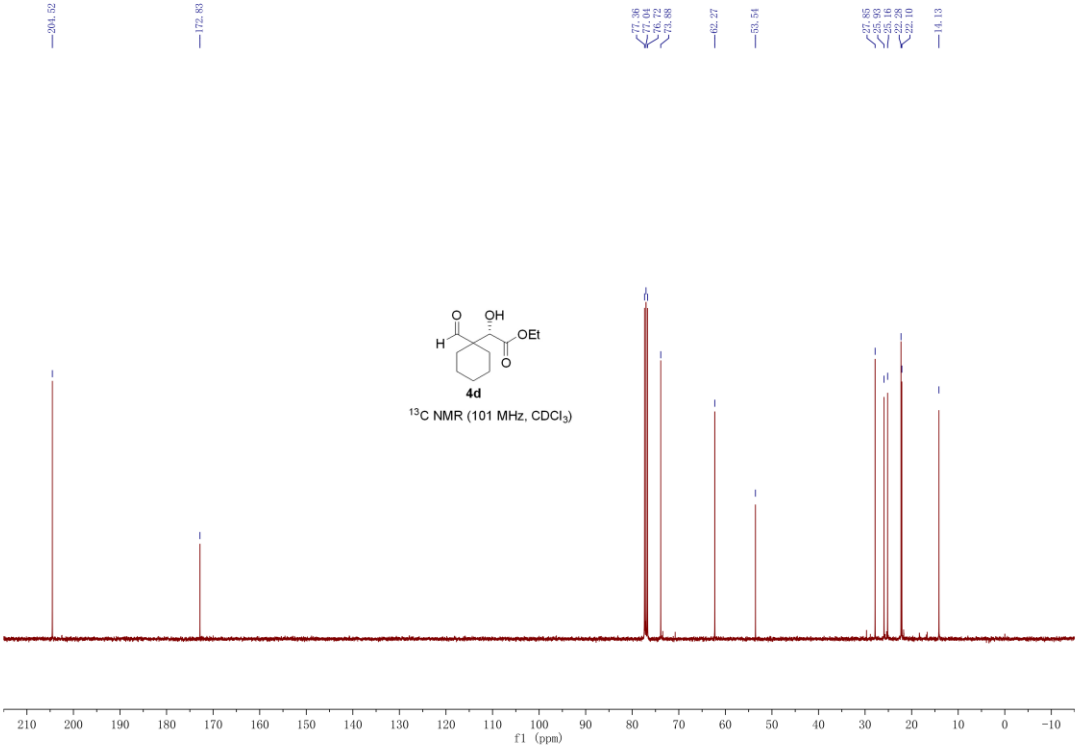

9.57
7.25
5.12
5.11
5.10
5.09
5.07
4.28
4.27
3.04
3.03
1.27
1.26
1.25
1.24
1.13
1.05
O
OH
H
O
O
O*i*-Pr
4b
^{1}H NMR (600 MHz, CDCl$_3$)
0.87
0.98
0.98
0.96
5.96
3.00
2.92
f1 (ppm)

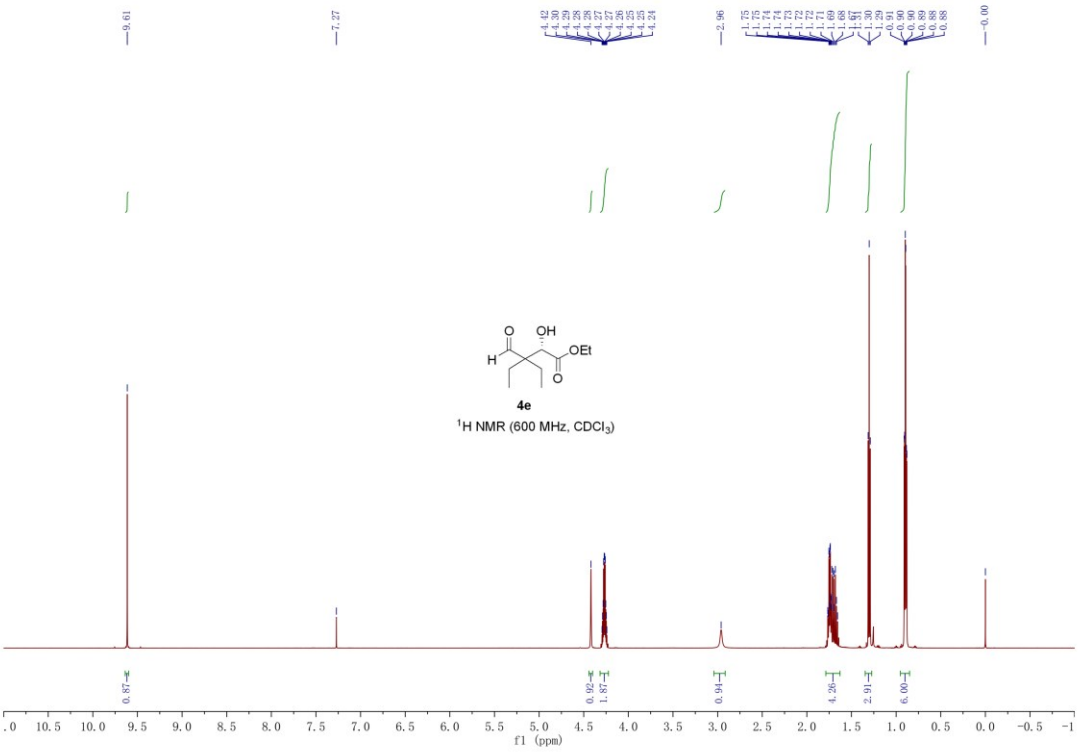

202.45
172.41
77.35
77.03
76.71
73.44
70.80
50.49
21.73
21.70
18.55
16.67
O
OH
H
O
O
O*i*-Pr
4b
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

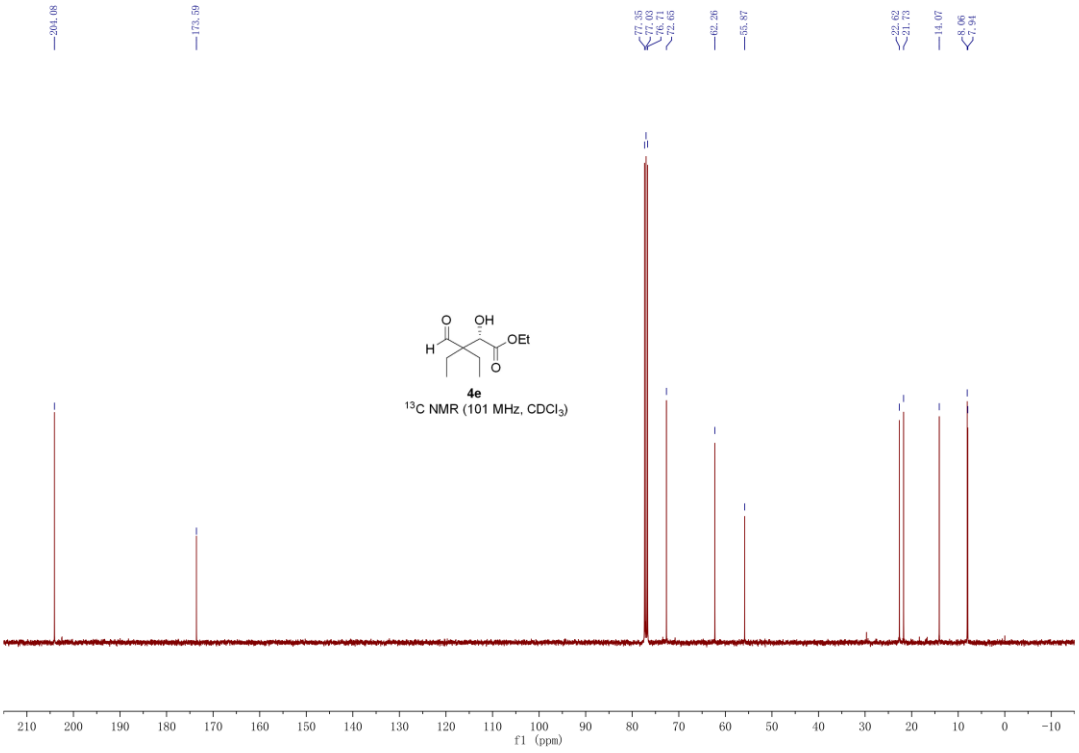

9.56
7.25
4.35
4.27
4.27
4.26
4.26
4.24
4.23
4.23
4.22
4.21
4.20
4.19
4.19
3.09
1.96
1.95
1.93
1.92
1.91
1.87
1.87
1.72
1.70
1.68
1.65
1.64
1.61
1.26
1.25
O
OH
H
OEt
O
4c
1H NMR (600 MHz, CDCl3)
0.91
0.96
1.98
1.00
3.09
5.17
3.00
f1 (ppm)

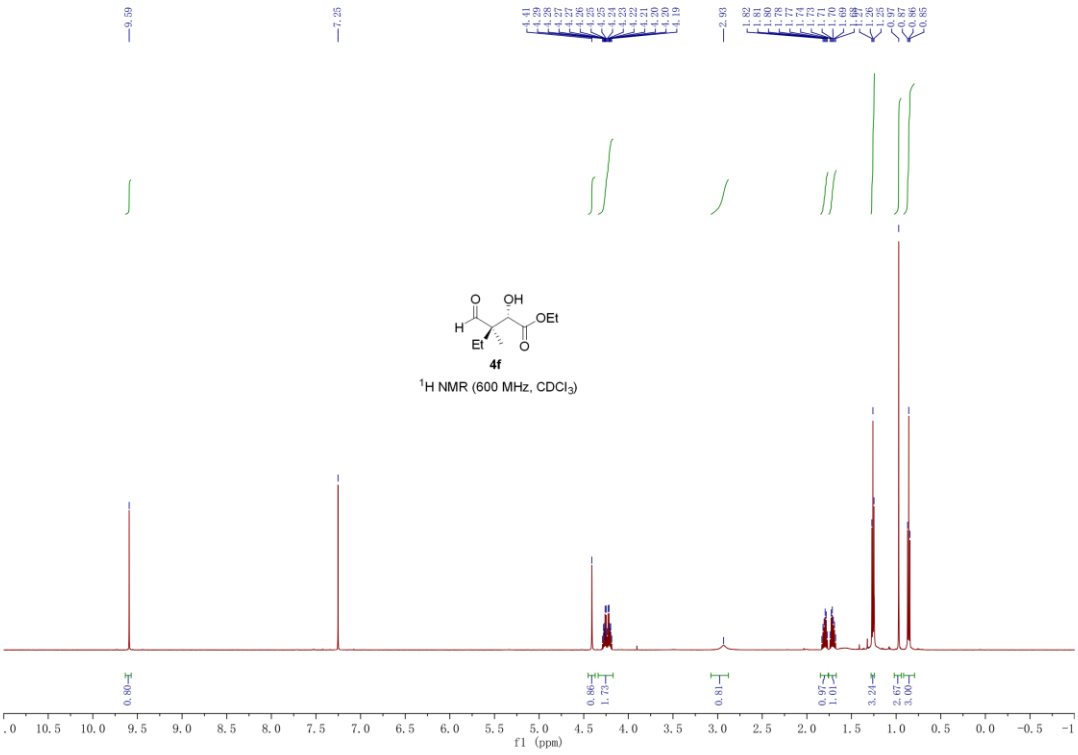

201.65
173.30
77.36
77.04
76.72
72.38
62.26
61.68
30.28
28.11
25.92
25.68
14.07
O
OH
H
OEt
O
4c
13C NMR (101 MHz, CDCl3)
f1 (ppm)

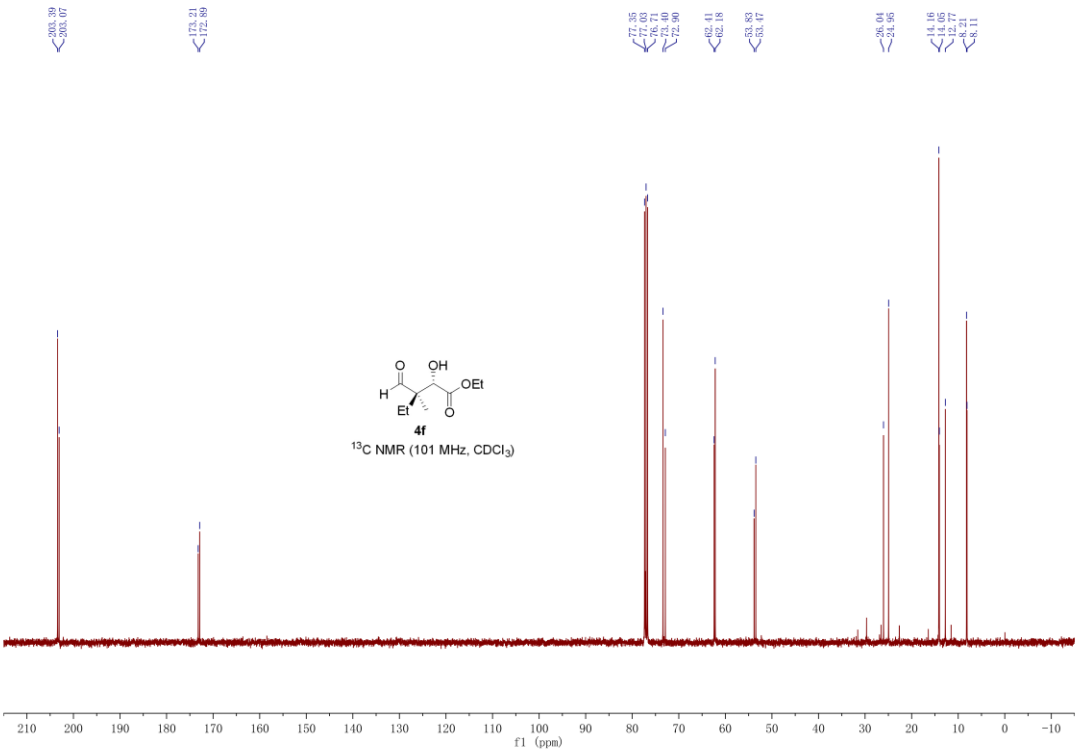

4d
^{1}H NMR (600 MHz, $CDCl_3$)
f1 (ppm)

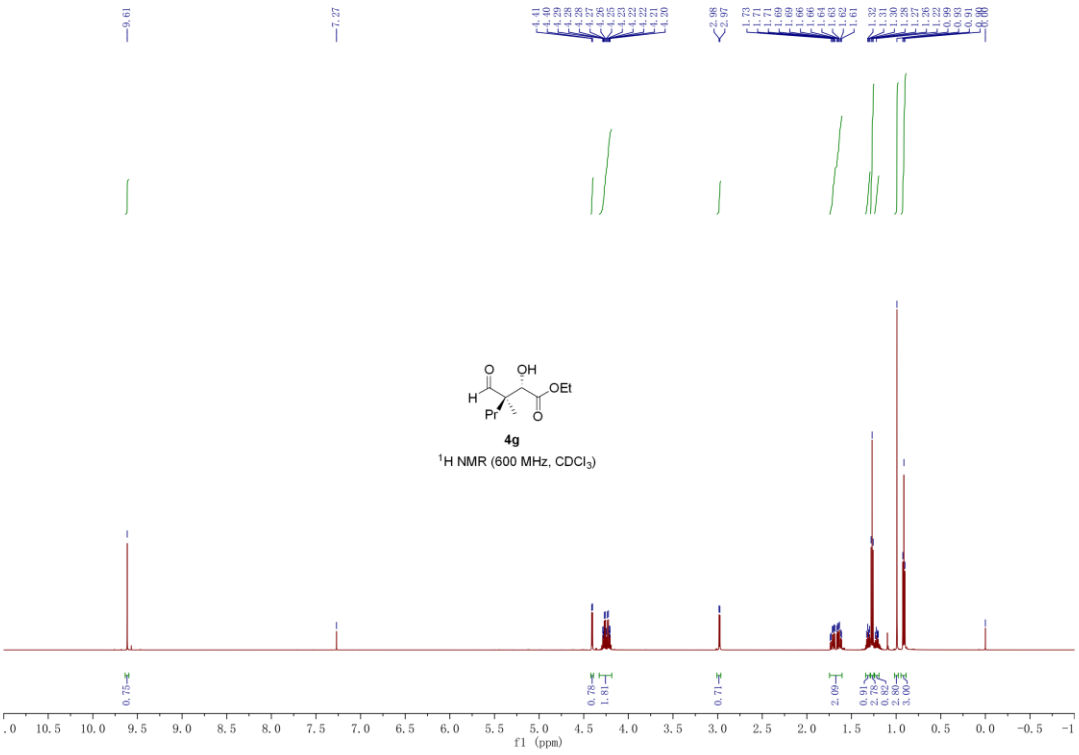

4d
^{13}C NMR (101 MHz, $CDCl_3$)
f1 (ppm)

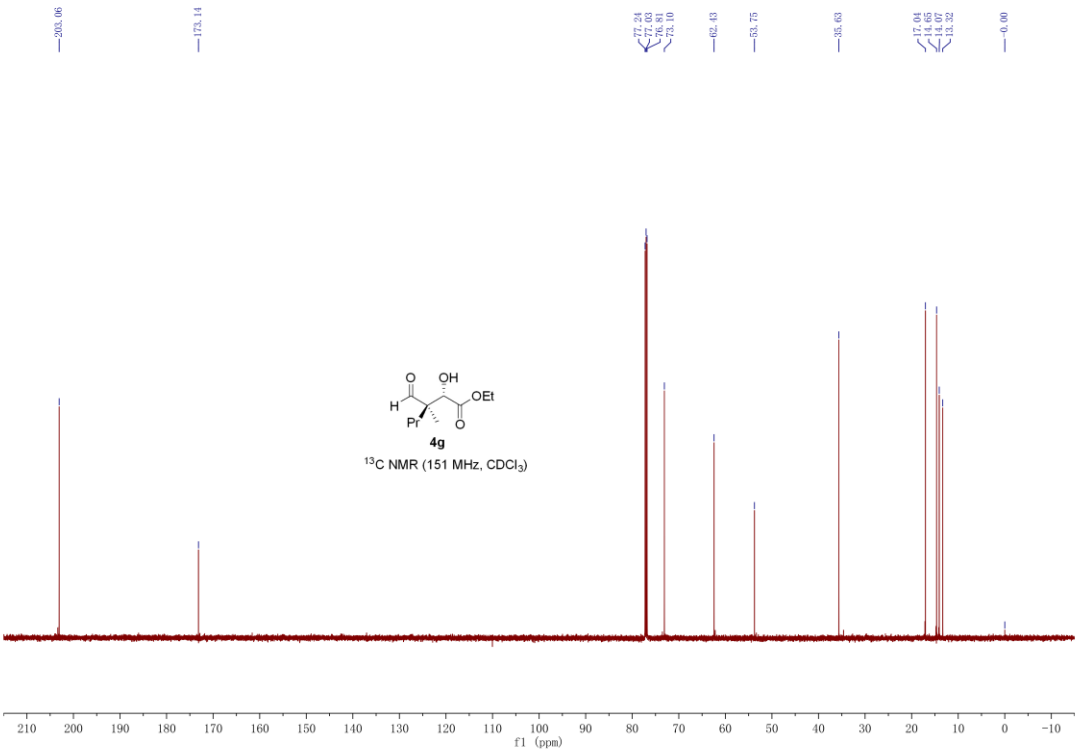

9.61
7.27
4.42
4.39
4.29
4.28
4.28
4.27
4.27
4.26
4.25
4.25
4.24
2.96
1.75
1.75
1.74
1.74
1.73
1.72
1.72
1.71
1.69
1.68
1.41
1.30
0.91
0.90
0.90
0.89
0.88
0.88
0.00
O
OH
H
OEt
O
4e
^{1}H NMR (600 MHz, CDCl$_3$)
0.87
0.92
1.87
0.94
4.26
2.91
6.00
f1 (ppm)

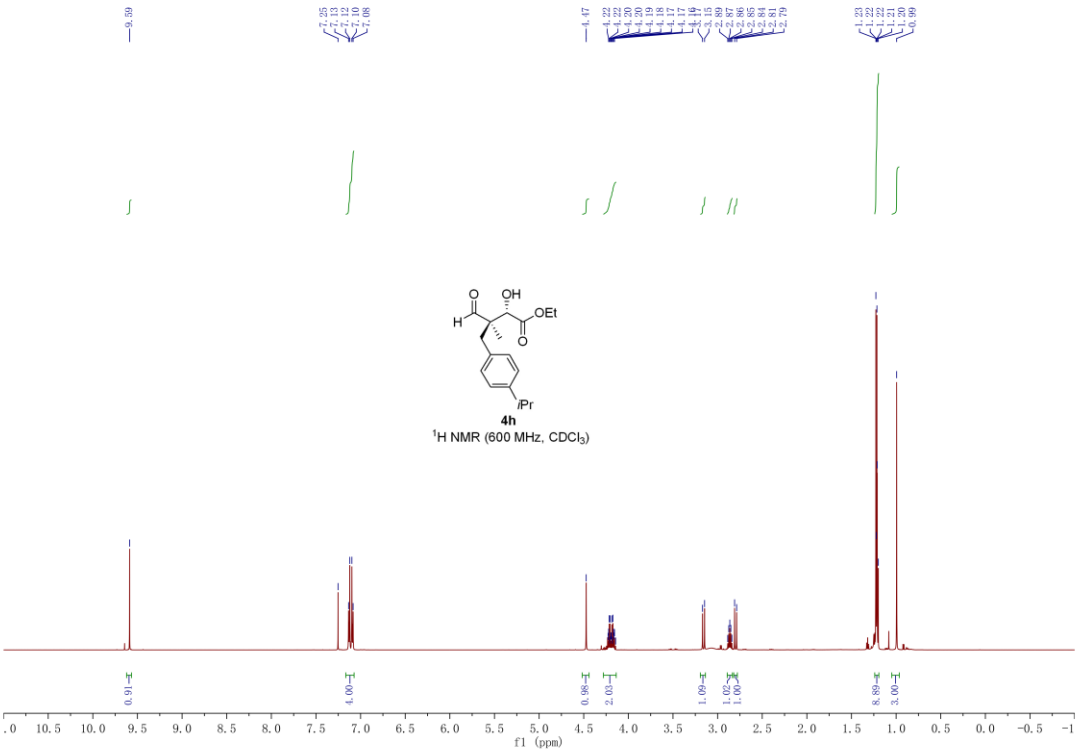

204.08
173.59
77.35
77.03
76.71
72.65
62.26
55.87
22.62
21.73
14.07
8.06
7.94
O
OH
H
OEt
O
4e
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

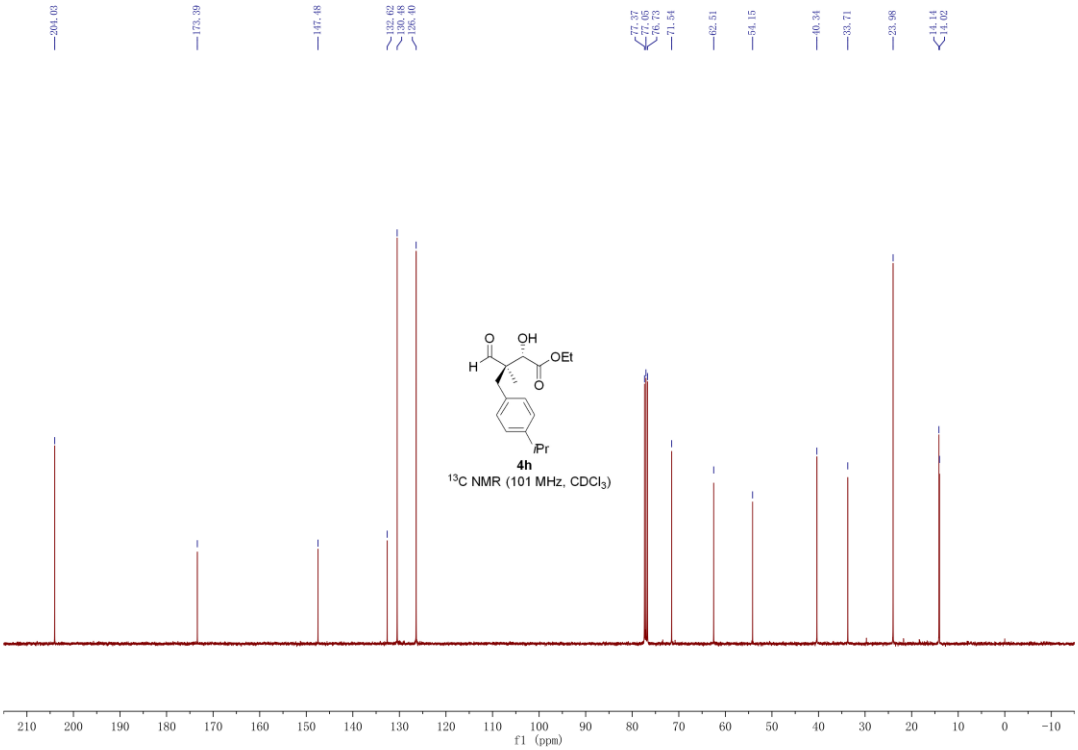

4f
^{1}H NMR (600 MHz, CDCl$_3$)
f1 (ppm)

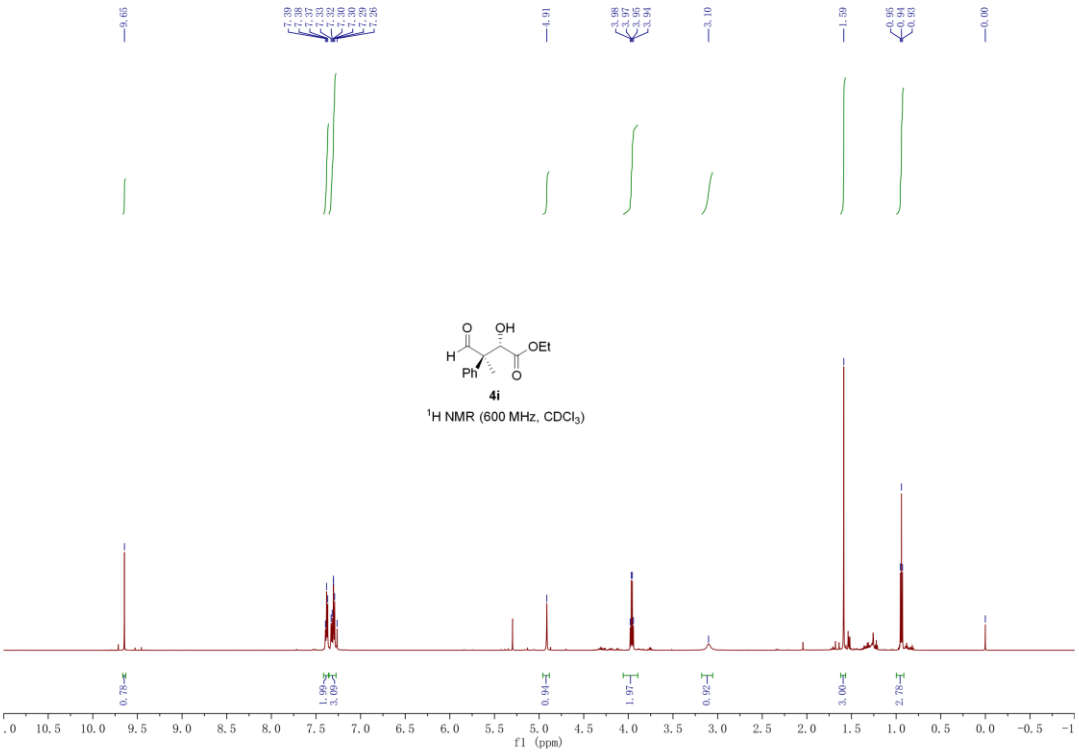

4f
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

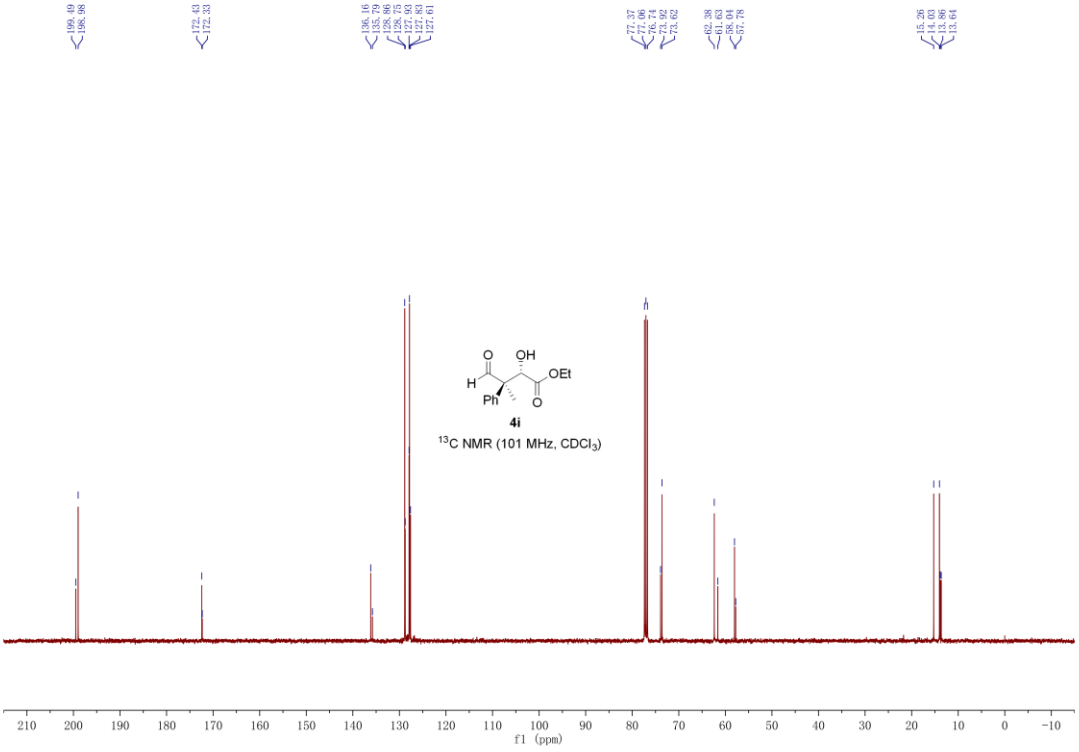

4g
^{1}H NMR (600 MHz, CDCl$_3$)
f1 (ppm)

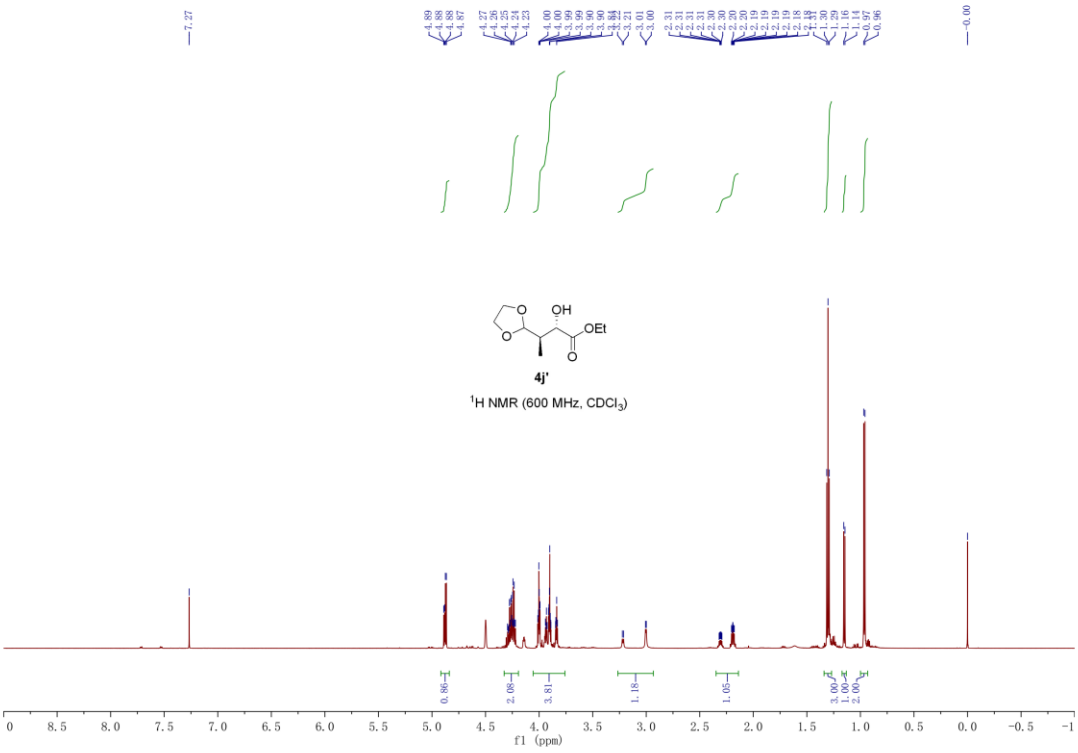

4g
^{13}C NMR (151 MHz, CDCl$_3$)
f1 (ppm)

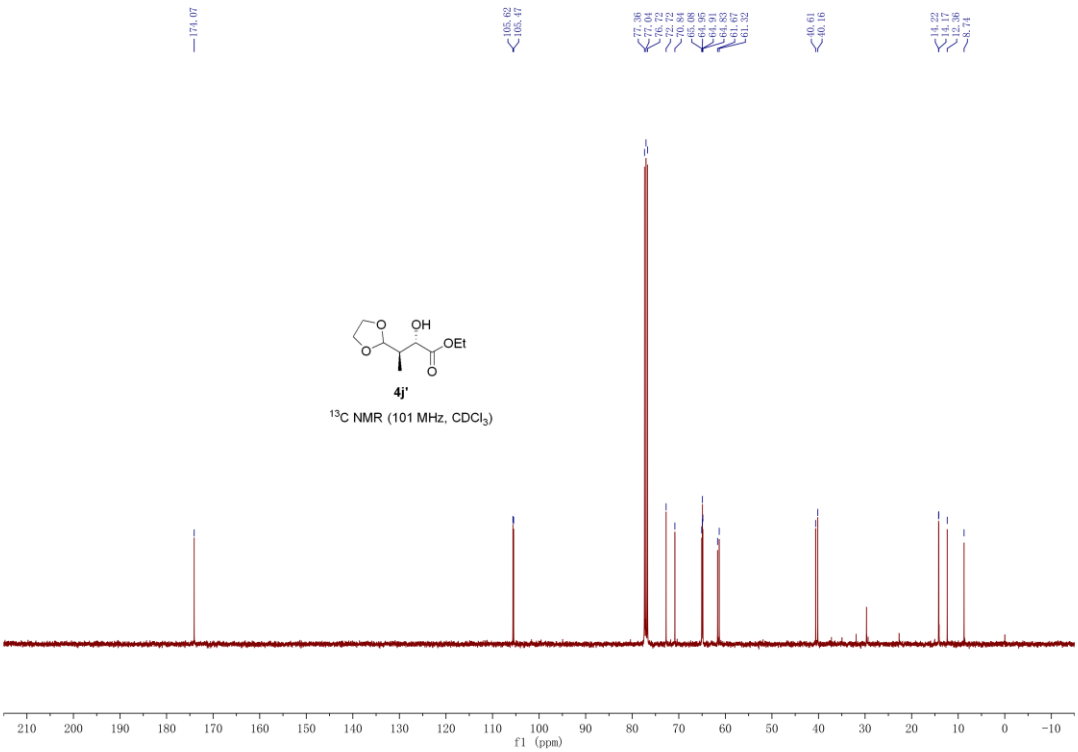

9.59
7.25
7.13
7.12
7.10
7.08
4.47
4.22
4.20
4.19
4.18
4.17
4.16
3.15
2.89
2.87
2.86
2.85
2.84
2.81
2.79
1.23
1.22
1.21
1.20
0.99
O
OH
H
OEt
O
iPr
4h
1H NMR (600 MHz, CDCl3)
0.91
4.00
0.98
2.03
1.09
1.02
1.00
8.89
3.00
f1 (ppm)

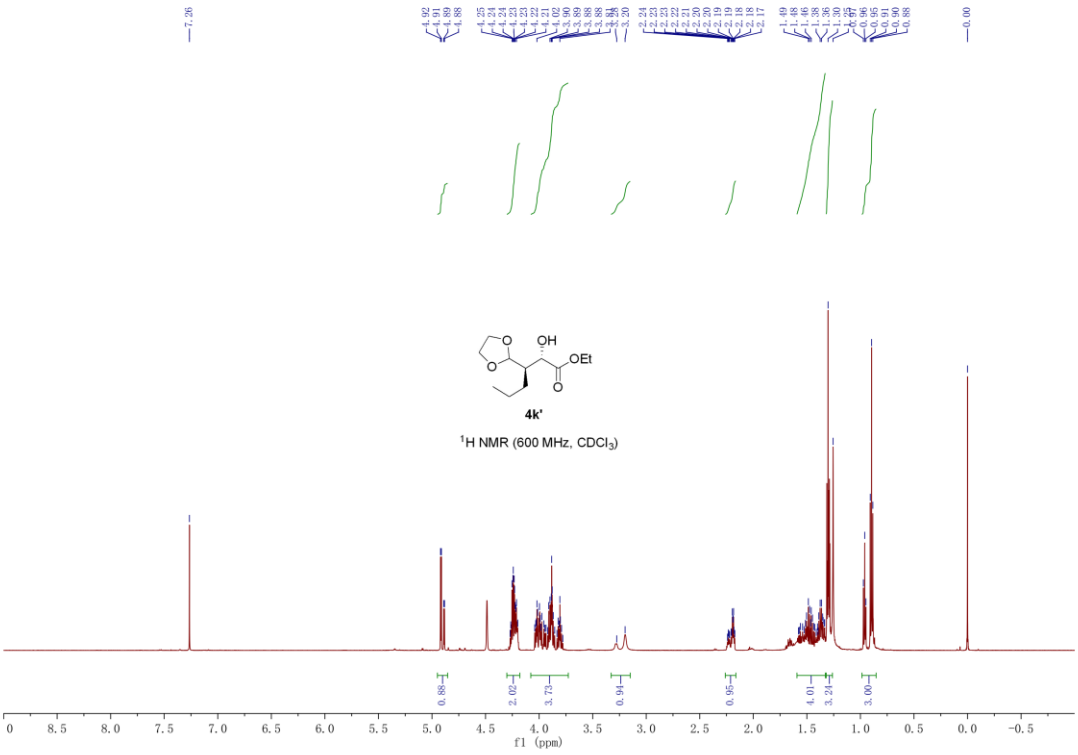

204.03
173.39
147.48
132.62
130.48
126.40
77.37
77.05
76.73
71.54
62.51
54.15
40.34
33.71
23.98
14.14
14.02
O
OH
H
OEt
O
iPr
4h
13C NMR (101 MHz, CDCl3)
f1 (ppm)

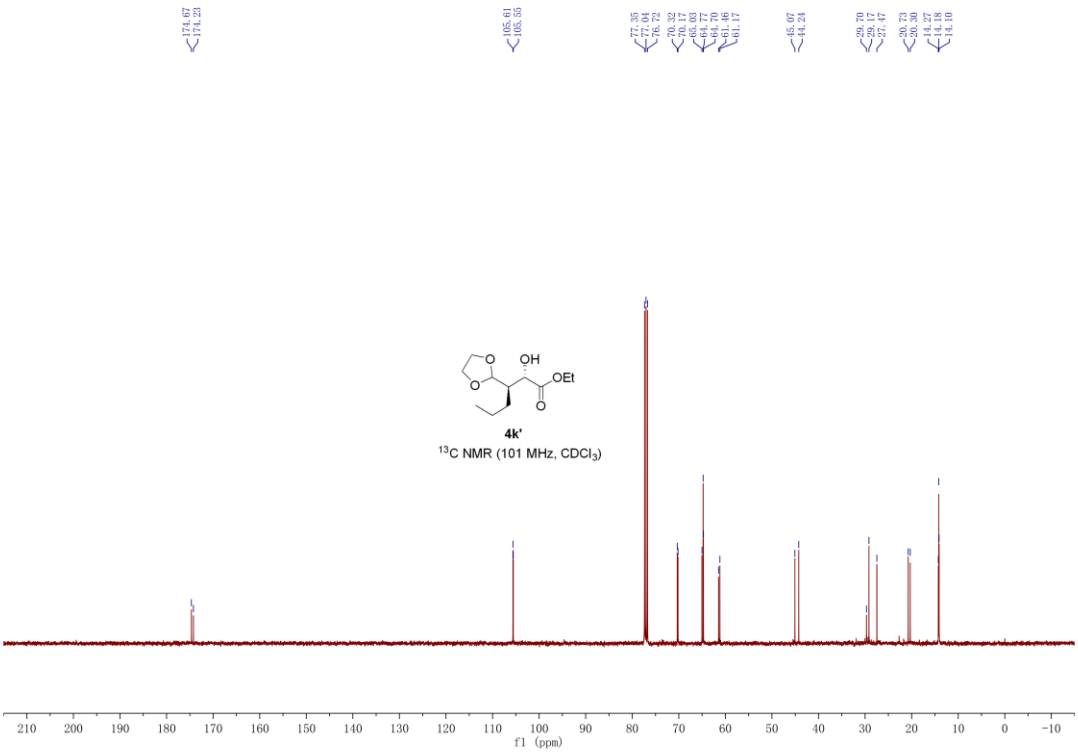

9.65
7.39
7.38
7.37
7.33
7.32
7.30
7.29
7.26
4.91
3.98
3.97
3.95
3.94
3.10
1.59
0.95
0.94
0.93
0.00
O
OH
OEt
H
Ph
O
4i
^{1}H NMR (600 MHz, CDCl$_3$)
0.78
1.99
3.09
0.94
1.97
0.92
3.00
2.78
f1 (ppm)

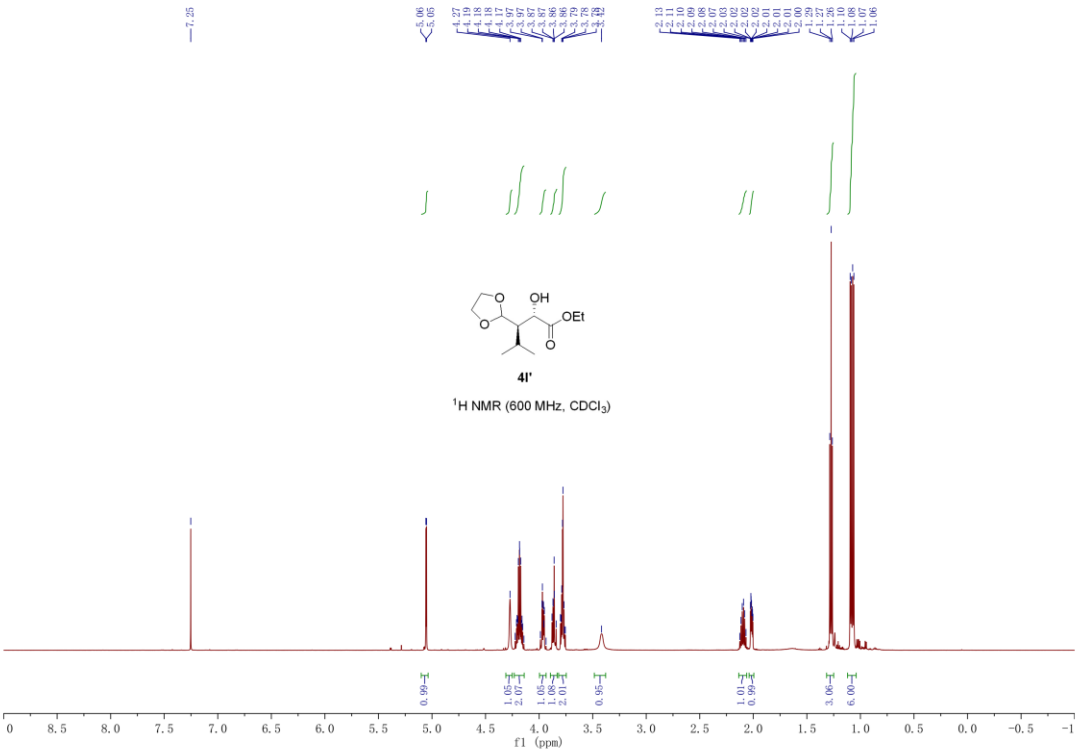

199.49
196.98
172.43
172.33
136.16
135.79
128.86
128.75
127.93
127.83
127.61
77.37
77.06
76.74
73.92
73.62
62.38
61.63
58.04
57.78
15.26
14.03
13.86
13.64
O
OH
OEt
H
Ph
O
4i
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)


OH
OEt
O
O
O
4j'
^{1}H NMR (600 MHz, CDCl$_3$)
f1 (ppm)


OH
OEt
O
O
O
4j'
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

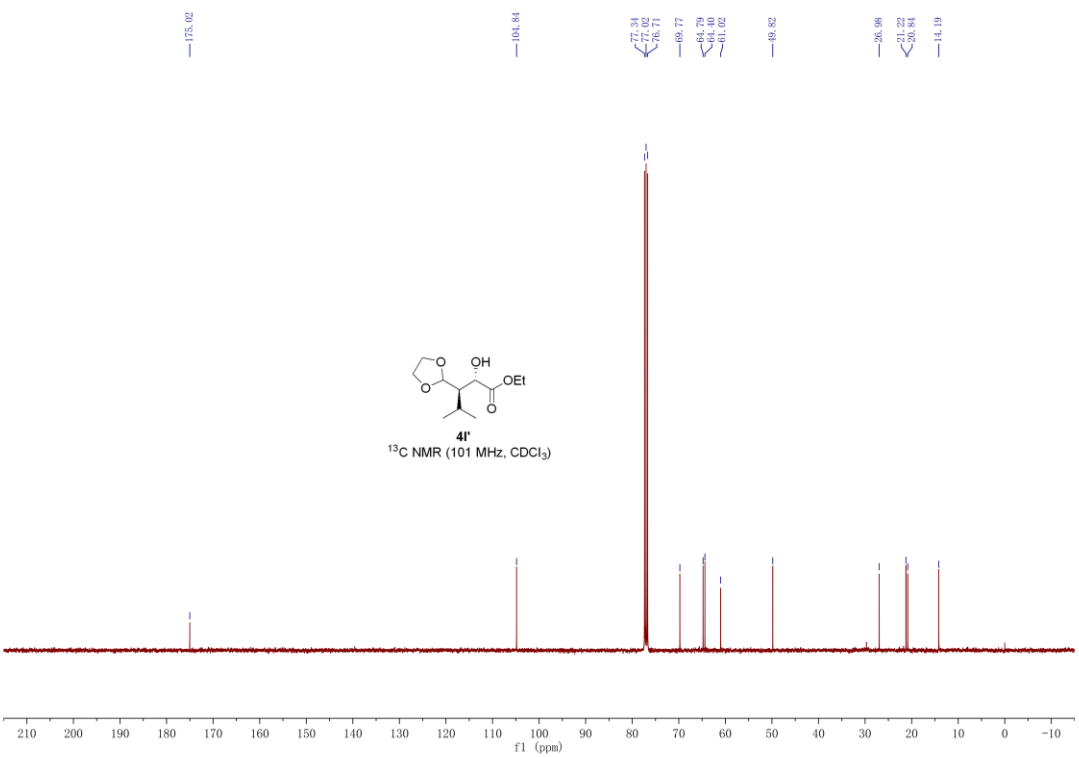

4k'
^{1}H NMR (600 MHz, $CDCl_3$)
f1 (ppm)


4k'
^{13}C NMR (101 MHz, $CDCl_3$)
f1 (ppm)



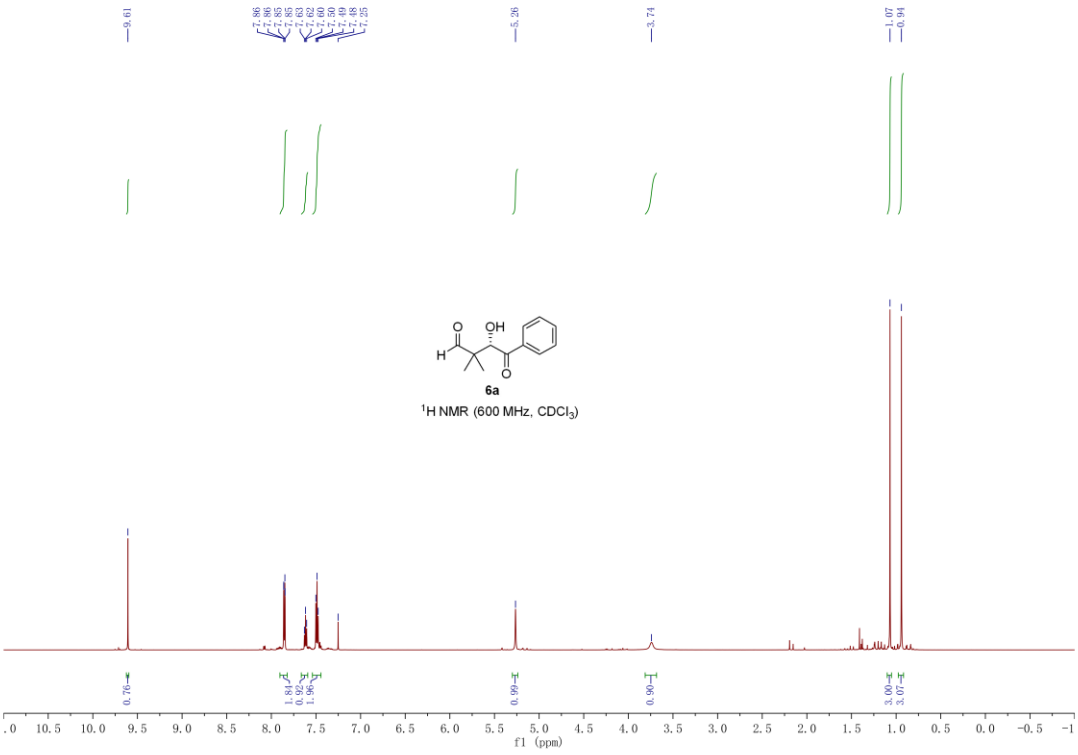
4l'

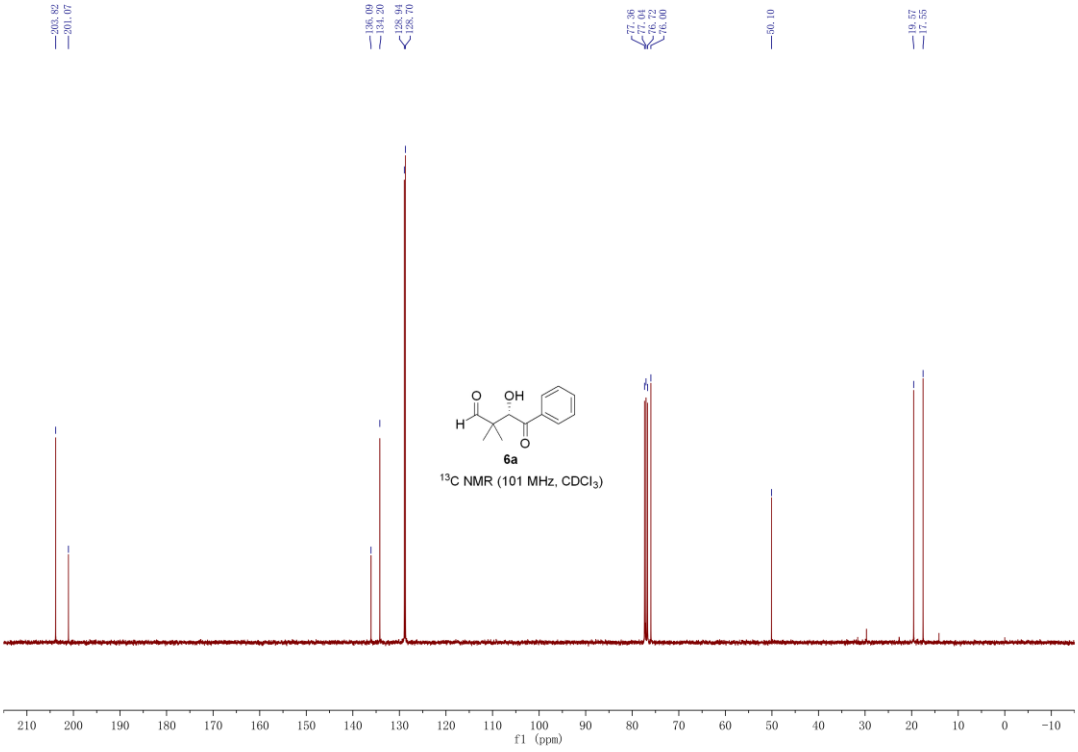
^{1}H NMR (600 MHz, $CDCl_3$)

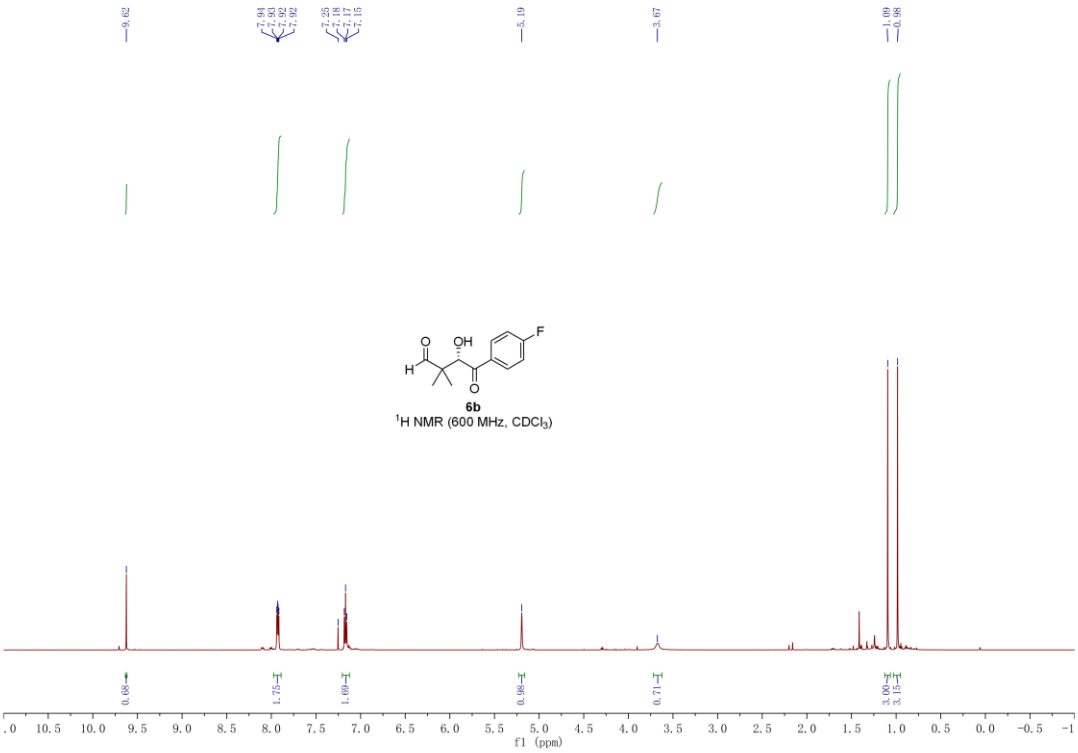


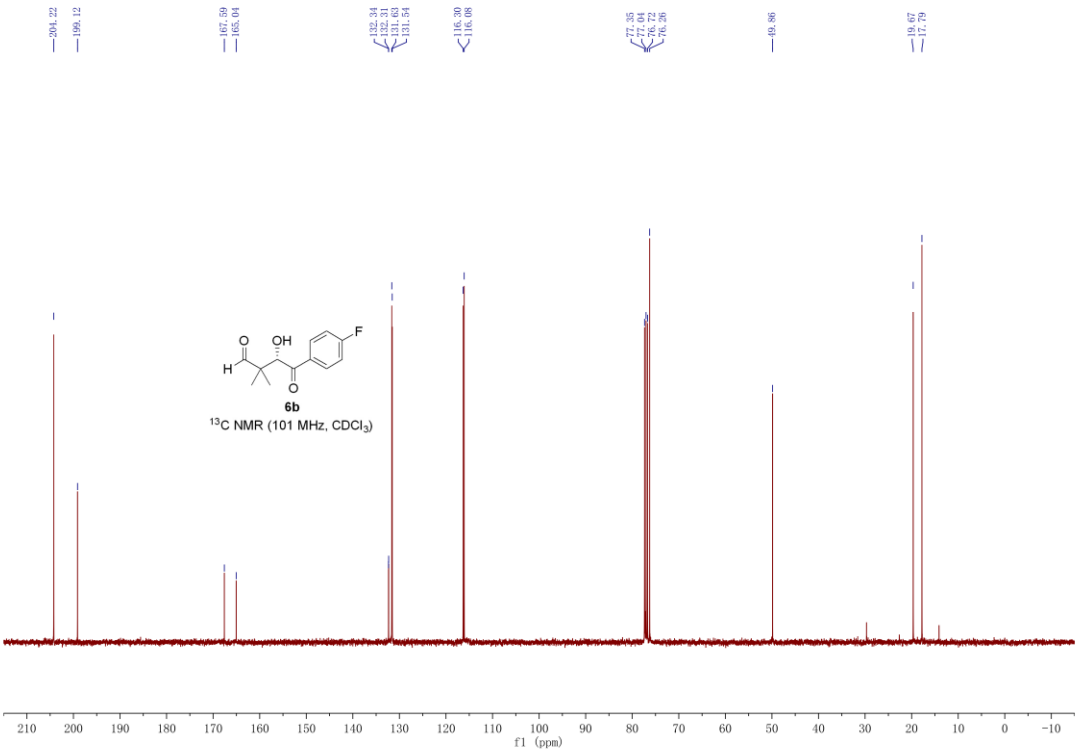
4l'

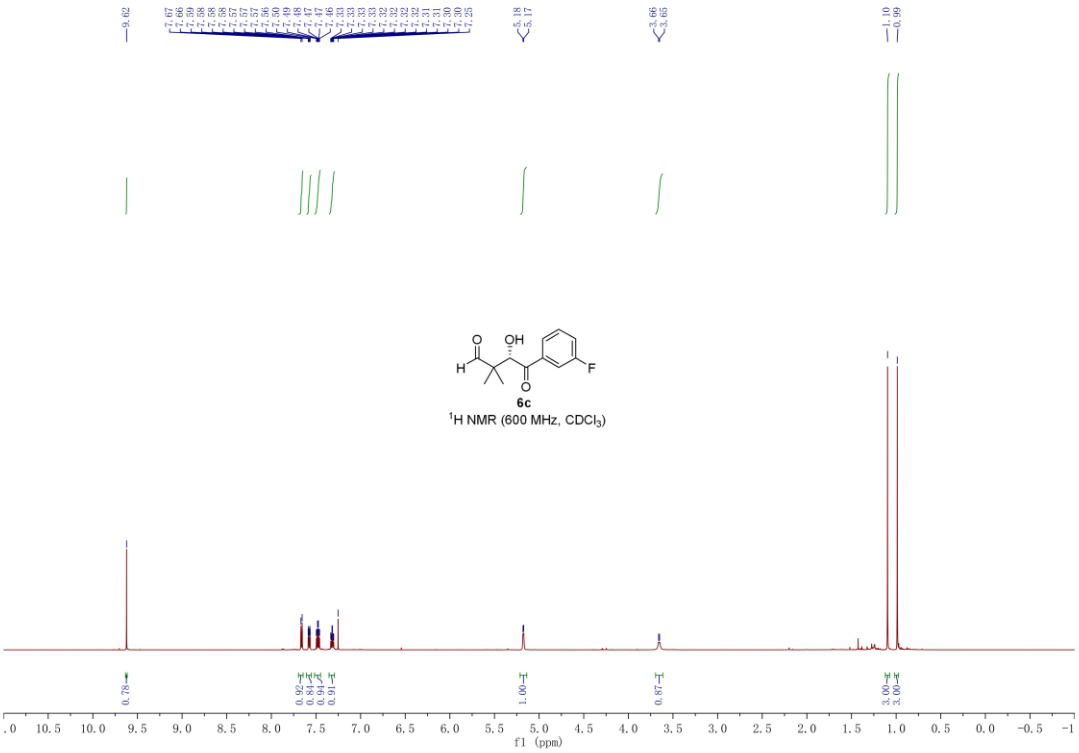
^{13}C NMR (101 MHz, $CDCl_3$)

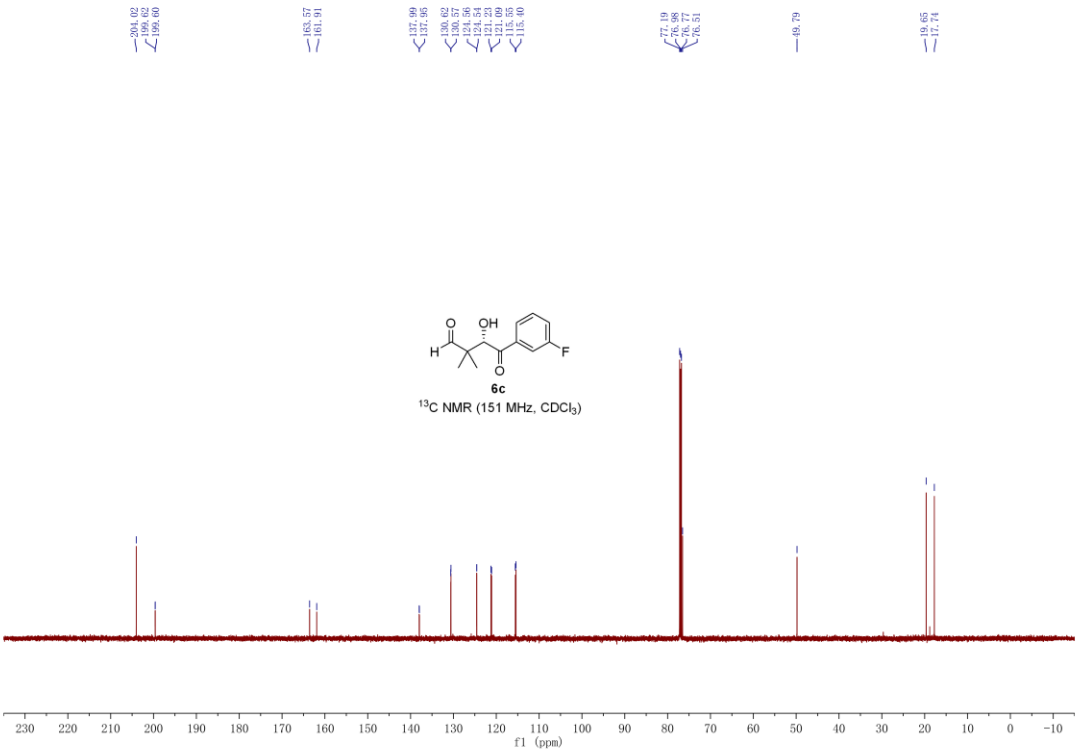

9.61
7.86
7.86
7.85
7.85
7.63
7.62
7.60
7.59
7.49
7.48
7.25
5.26
3.74
1.07
0.94
O
OH
H
O
6a
¹H NMR (600 MHz, CDCl3)
0.76
1.84
0.92
1.96
0.99
0.90
3.00
3.07
f1 (ppm)

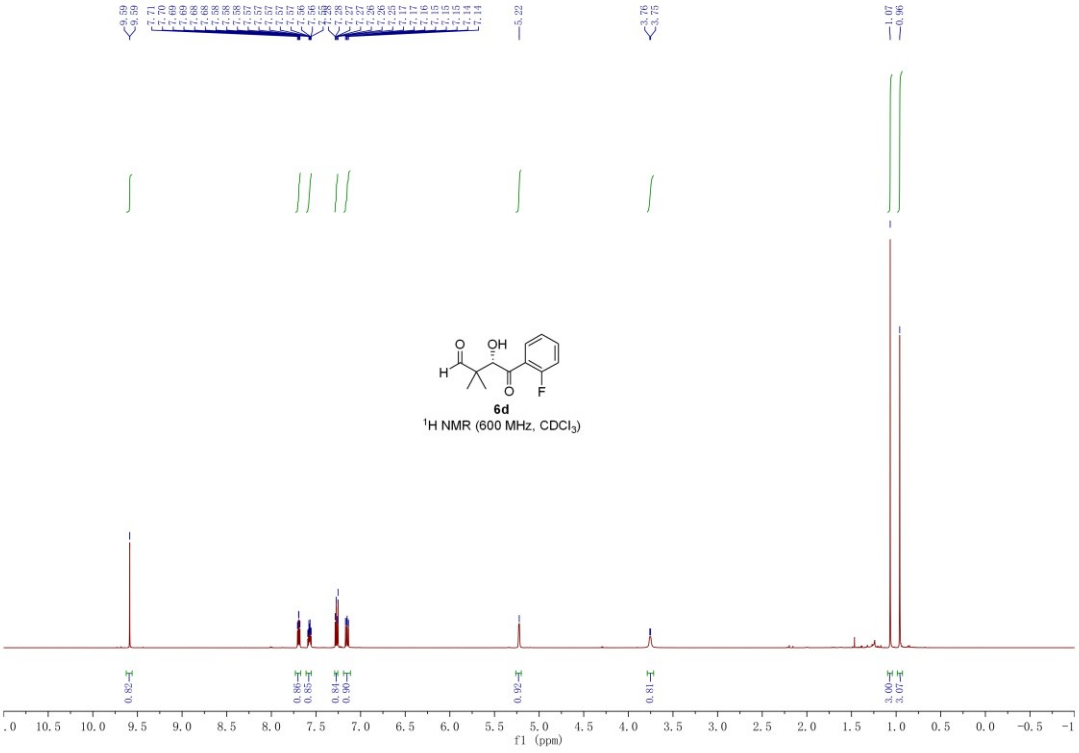

205.82
201.07
136.09
134.20
128.94
128.70
77.36
77.04
76.72
76.00
60.10
19.57
17.55
O
OH
H
O
6a
¹³C NMR (101 MHz, CDCl3)
f1 (ppm)

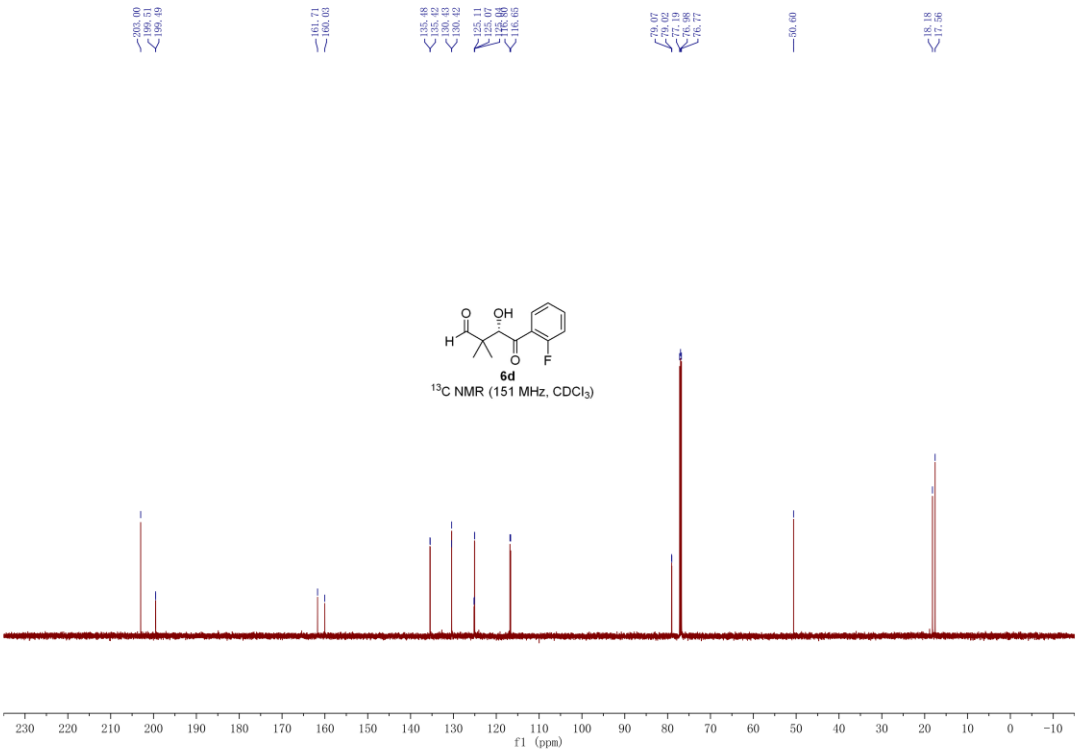

9.62
7.94
7.93
7.92
7.92
7.25
7.18
7.17
7.15
5.19
3.67
1.09
0.98
OH
O
H
O
F
6b
^{1}H NMR (600 MHz, CDCl$_3$)
0.68
1.75
1.69
0.98
0.71
3.00
3.15
f1 (ppm)

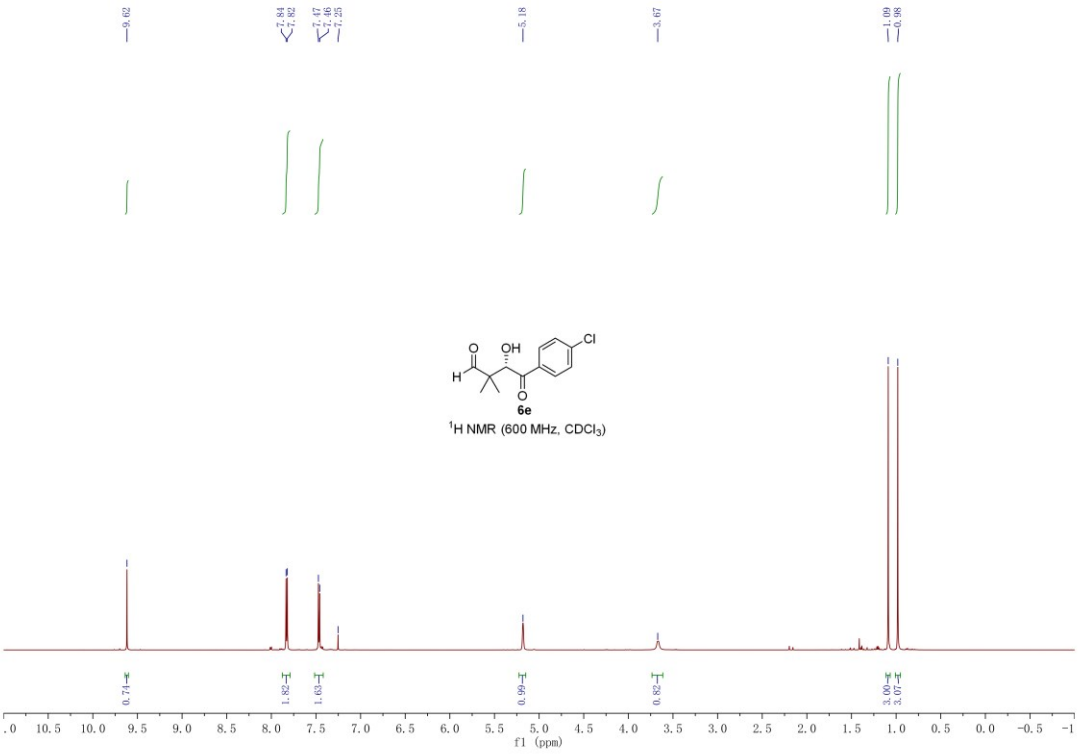

204.22
199.12
167.59
165.04
132.34
132.31
131.63
131.54
116.30
116.08
77.35
77.04
76.72
76.26
49.86
19.67
17.79
OH
O
H
O
F
6b
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

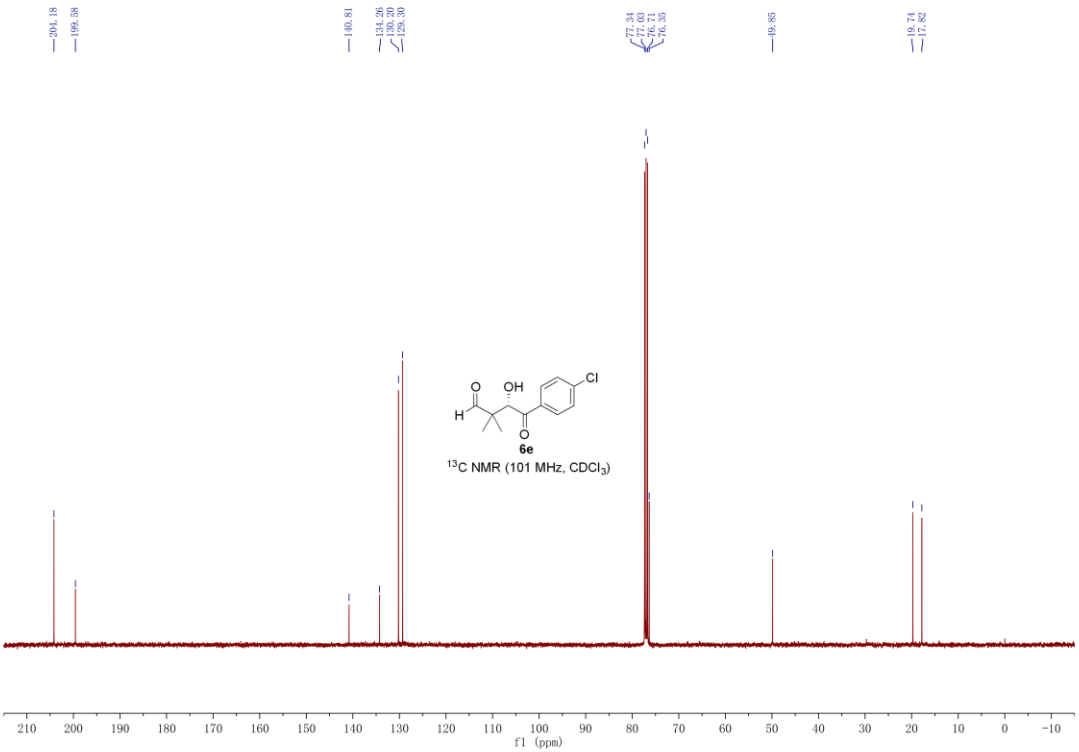

9.62
7.67
7.66
7.59
7.58
7.58
7.58
7.57
7.57
7.56
7.50
7.49
7.48
7.47
7.47
7.47
7.46
7.33
7.33
7.33
7.32
7.32
7.32
7.31
7.31
7.30
7.30
7.29
5.18
5.17
3.66
3.65
1.10
0.99
O
OH
H
F
O
6c
1H NMR (600 MHz, CDCl3)
0.78
0.92
0.84
0.94
0.91
1.00
0.87
3.00
3.00
f1 (ppm)

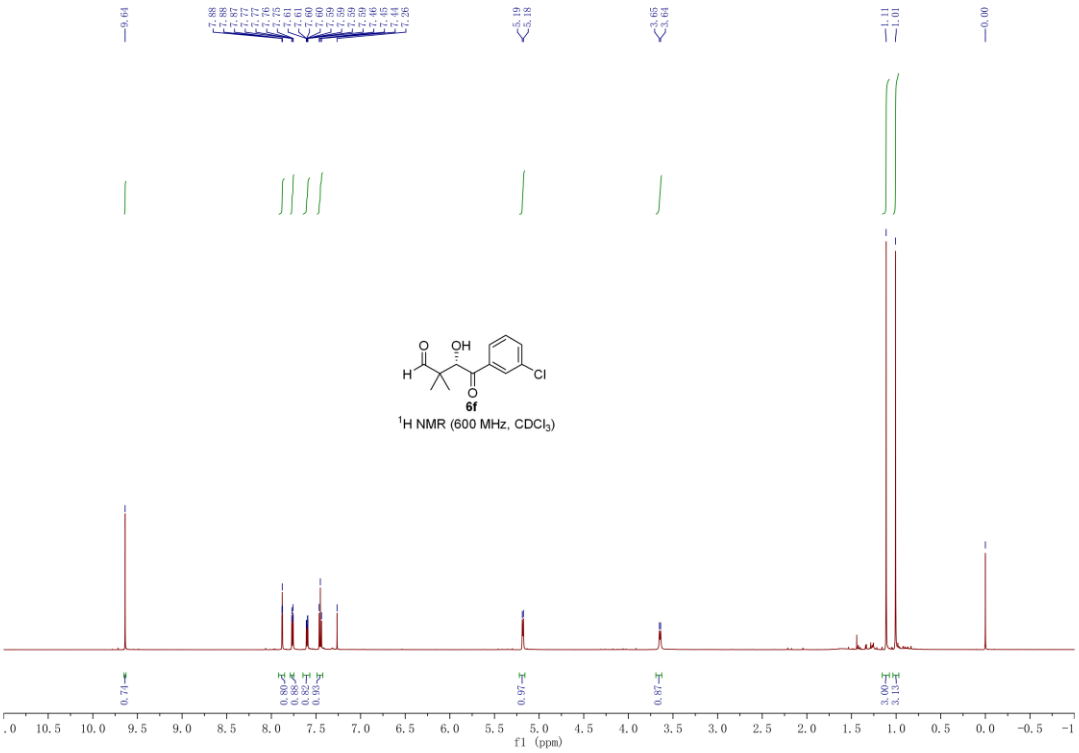

204.02
199.62
199.60
163.57
161.91
137.99
137.95
130.62
130.57
124.56
124.54
121.43
121.40
115.55
115.40
77.19
76.98
76.77
76.51
49.79
19.65
17.74
O
OH
H
F
O
6c
13C NMR (151 MHz, CDCl3)
f1 (ppm)

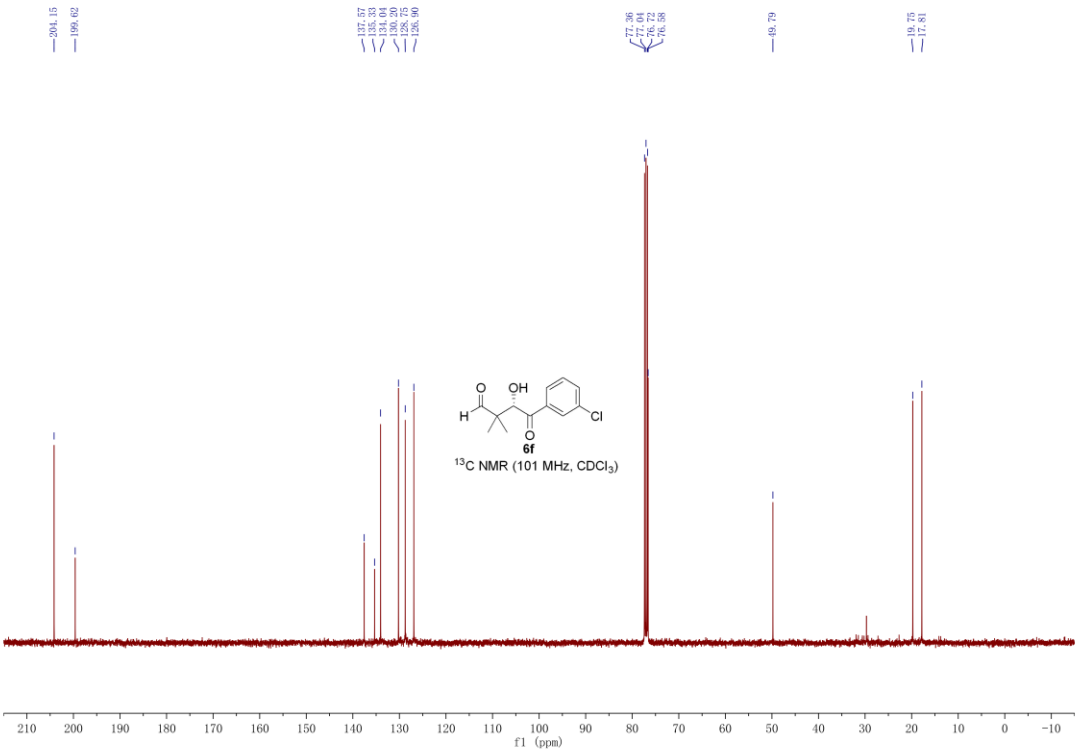

9.59
9.59
7.71
7.70
7.69
7.69
7.68
7.68
7.58
7.58
7.57
7.57
7.57
7.57
7.57
7.57
7.56
7.56
7.28
7.27
7.27
7.26
7.26
7.25
7.17
7.17
7.16
7.15
7.15
7.15
7.14
7.14
5.22
3.76
3.75
1.07
0.96
O
OH
H
O
F
6d
1H NMR (600 MHz, CDCl3)
0.82
0.86
0.85
0.84
0.90
0.92
0.81
3.00
3.07
f1 (ppm)

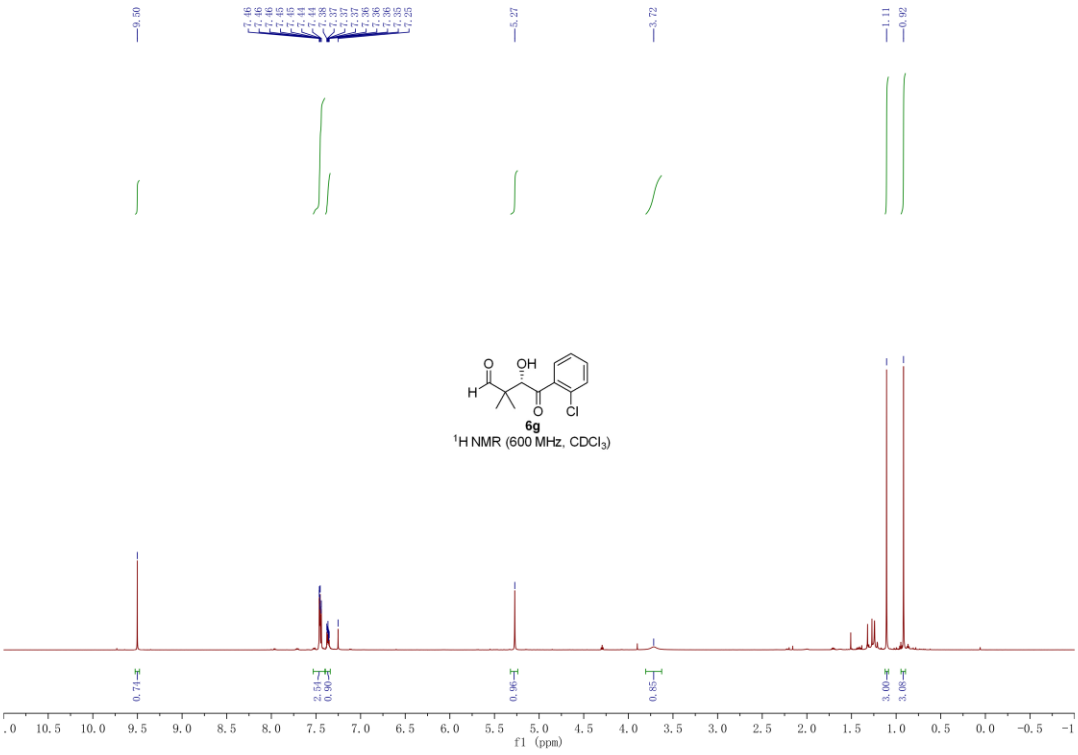

203.00
199.51
199.49
161.71
160.03
135.48
135.42
130.43
130.42
125.11
125.07
116.80
116.65
79.07
79.02
77.19
76.98
76.77
50.60
18.18
17.96
O
OH
H
O
F
6d
13C NMR (151 MHz, CDCl3)
f1 (ppm)

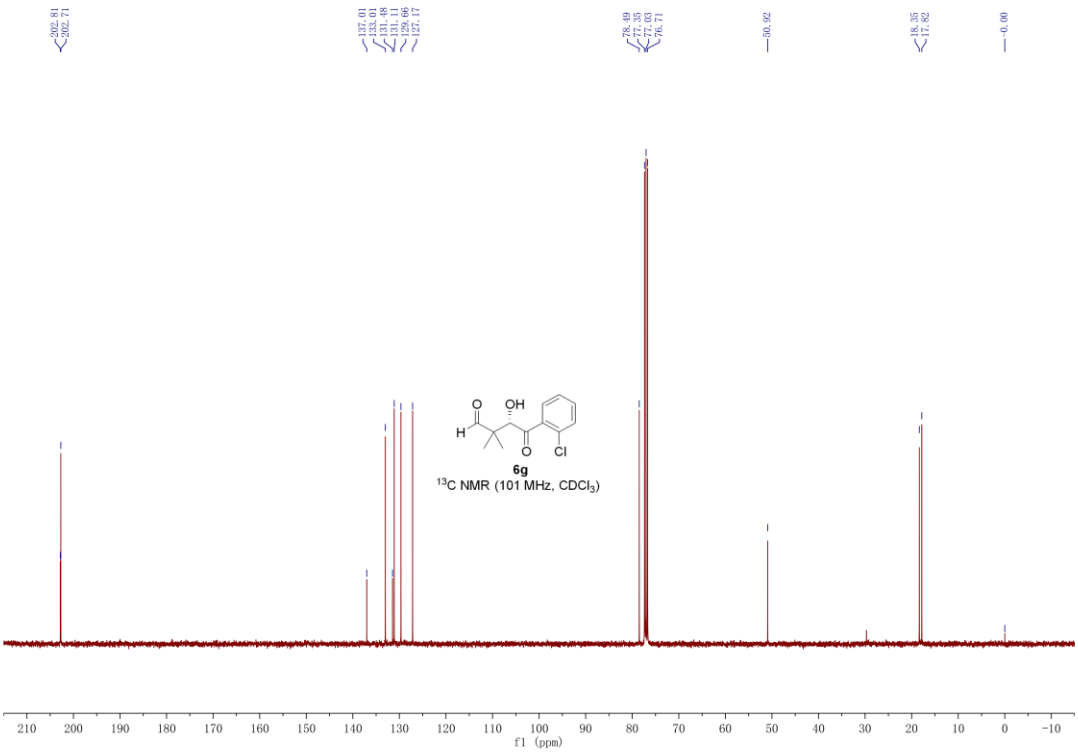

9.62
7.84
7.82
7.47
7.46
7.25
5.18
3.67
1.09
0.98
O
OH
H
O
Cl
6e
^{1}H NMR (600 MHz, CDCl$_3$)
0.74
1.82
1.63
0.99
0.82
3.00
3.07
f1 (ppm)

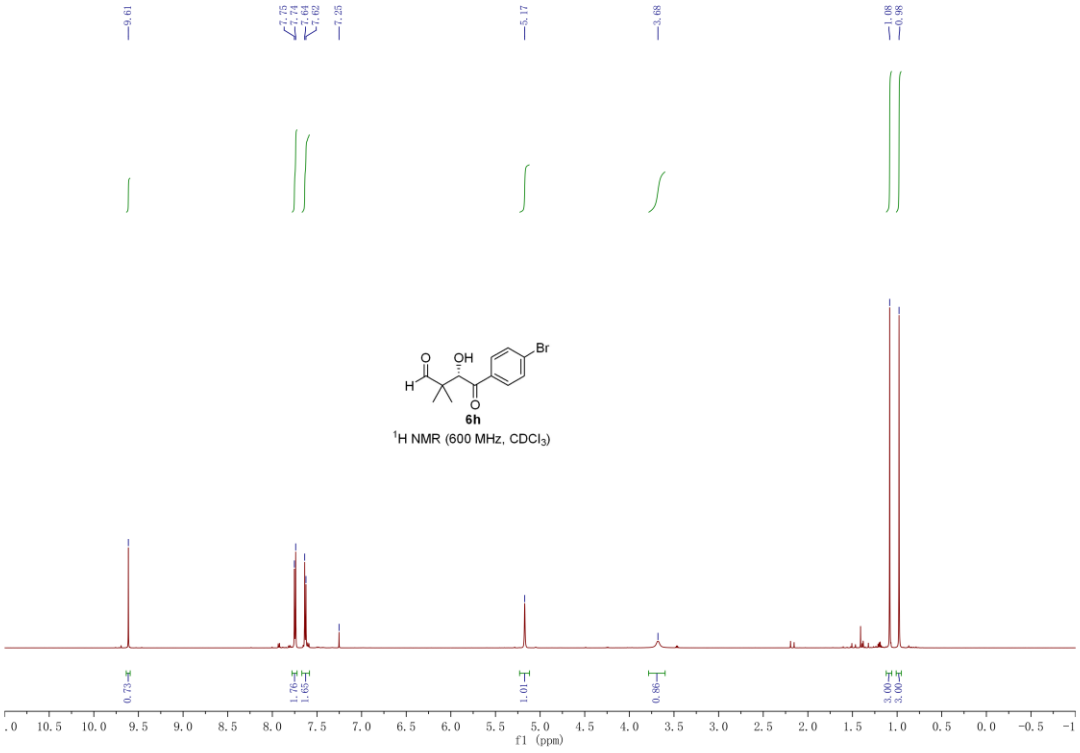

204.18
199.58
140.81
134.26
130.20
129.30
77.34
77.00
76.71
76.35
49.85
19.74
17.82
O
OH
H
O
Cl
6e
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

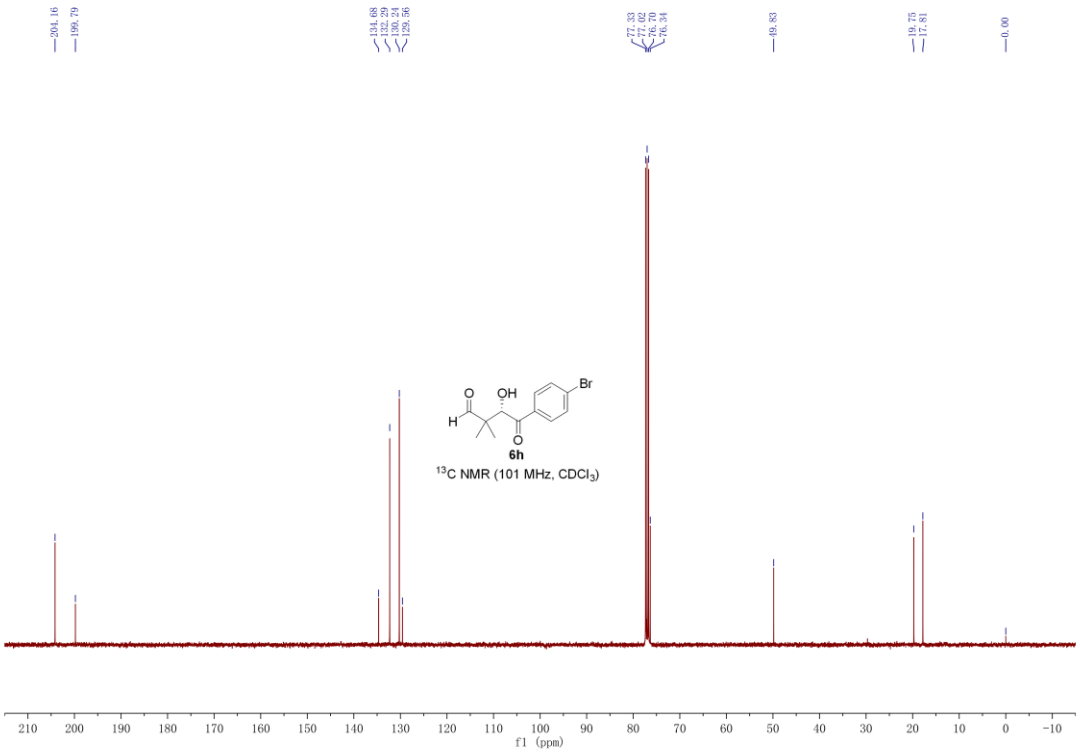

9.64
7.88
7.88
7.87
7.77
7.77
7.76
7.75
7.61
7.61
7.60
7.60
7.59
7.59
7.59
7.59
7.46
7.45
7.44
7.26
5.19
5.18
3.65
3.64
1.11
1.01
0.00
O
OH
H
Cl
O
6f
1H NMR (600 MHz, CDCl3)
0.74
0.80
0.88
0.92
0.93
0.95
0.97
0.87
3.00
3.13
f1 (ppm)

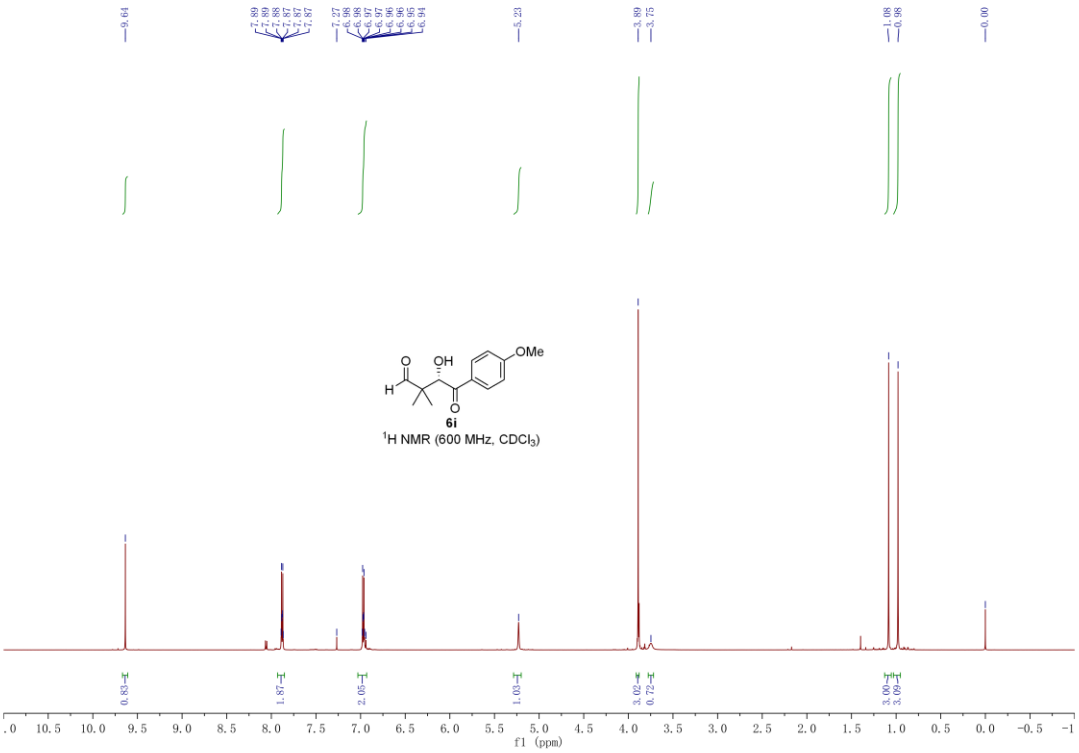

204.15
199.62
137.57
135.33
134.04
130.20
128.75
126.90
77.36
77.04
76.72
76.58
49.79
19.75
17.81
O
OH
H
Cl
O
6f
13C NMR (101 MHz, CDCl3)
f1 (ppm)

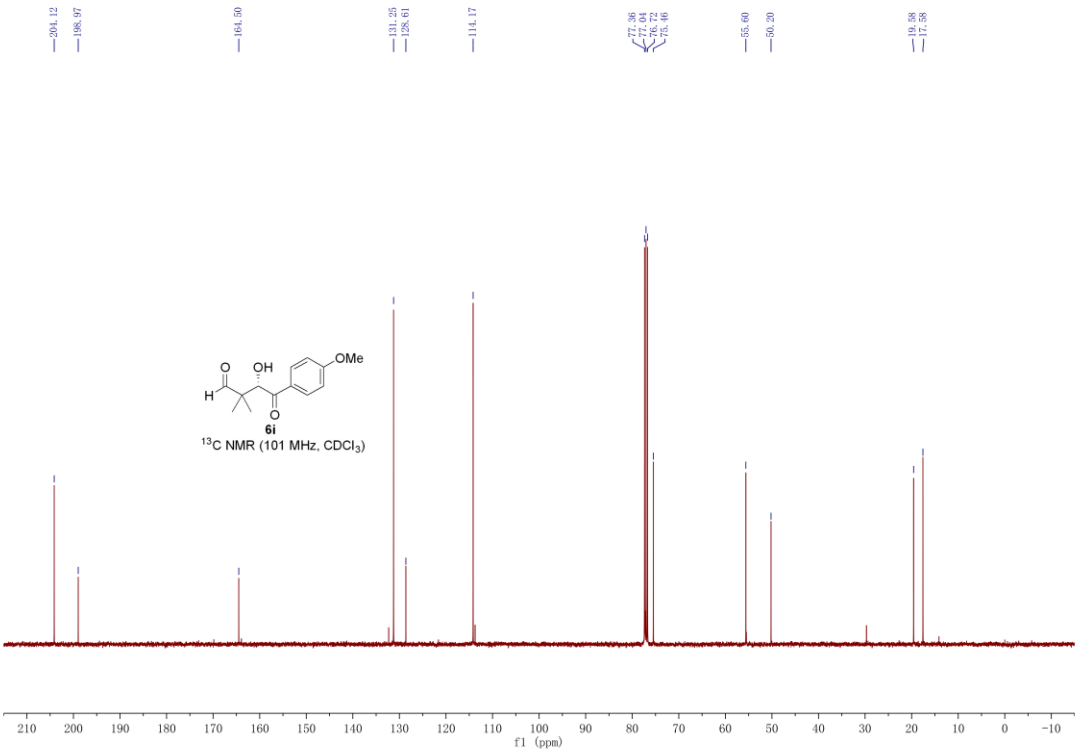

9.50
7.46
7.46
7.46
7.45
7.45
7.44
7.44
7.38
7.37
7.37
7.36
7.36
7.36
7.35
7.35
5.27
3.72
1.11
0.92
O
OH
H
O
Cl
6g
1H NMR (600 MHz, CDCl3)
0.74
2.54
0.90
0.96
0.85
3.00
3.08
f1 (ppm)

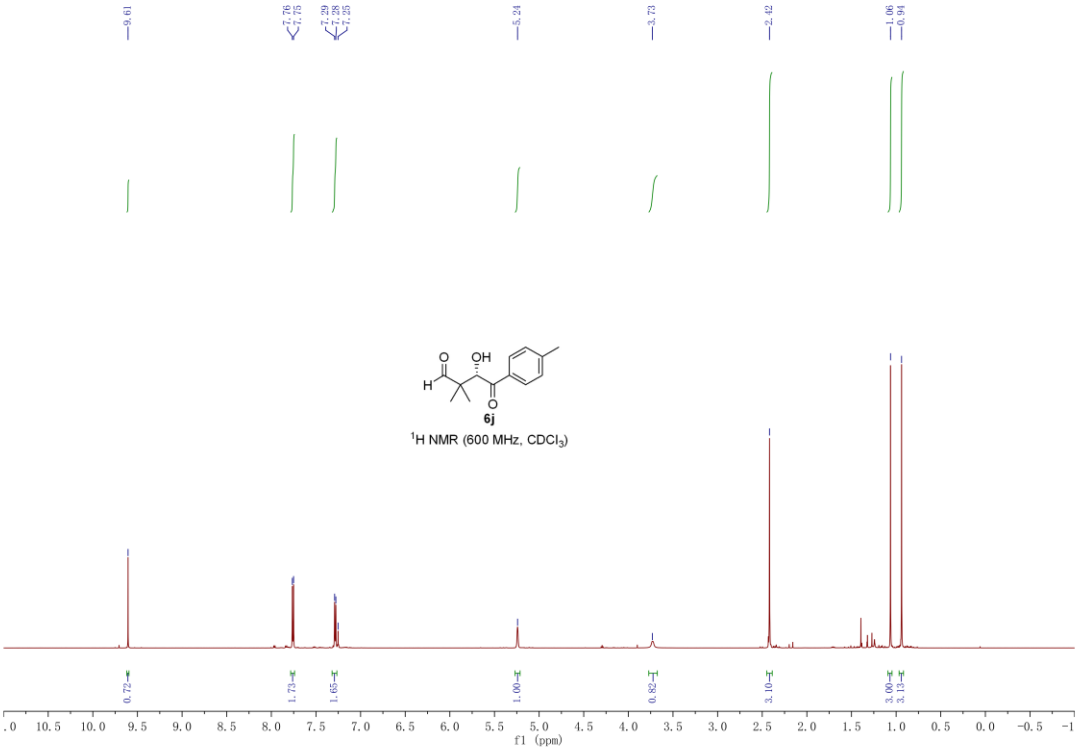

202.84
202.71
137.01
133.01
131.48
131.11
129.66
127.17
78.49
77.35
77.03
76.71
50.92
18.35
17.82
0.00
O
OH
H
O
Cl
6g
13C NMR (101 MHz, CDCl3)
f1 (ppm)

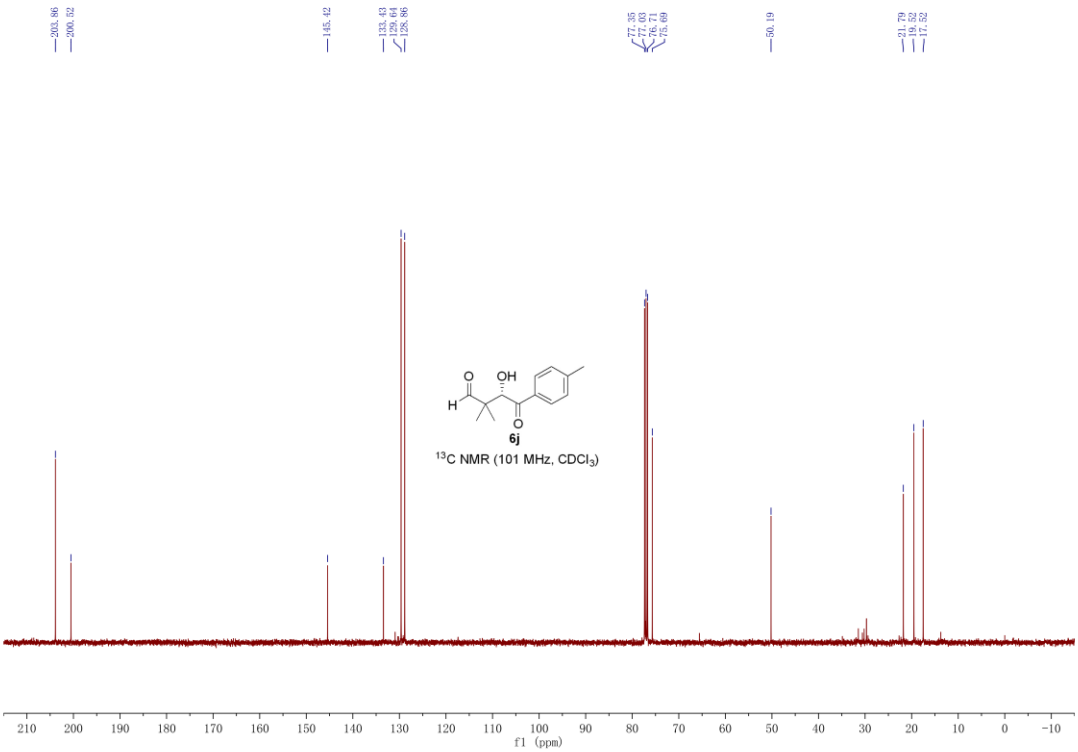

9.61
7.75
7.74
7.64
7.62
7.25
5.17
3.68
1.08
0.98
O
OH
H
Br
O
6h
^{1}H NMR (600 MHz, CDCl$_3$)
0.73
1.76
1.65
1.01
0.86
3.00
3.00
f1 (ppm)

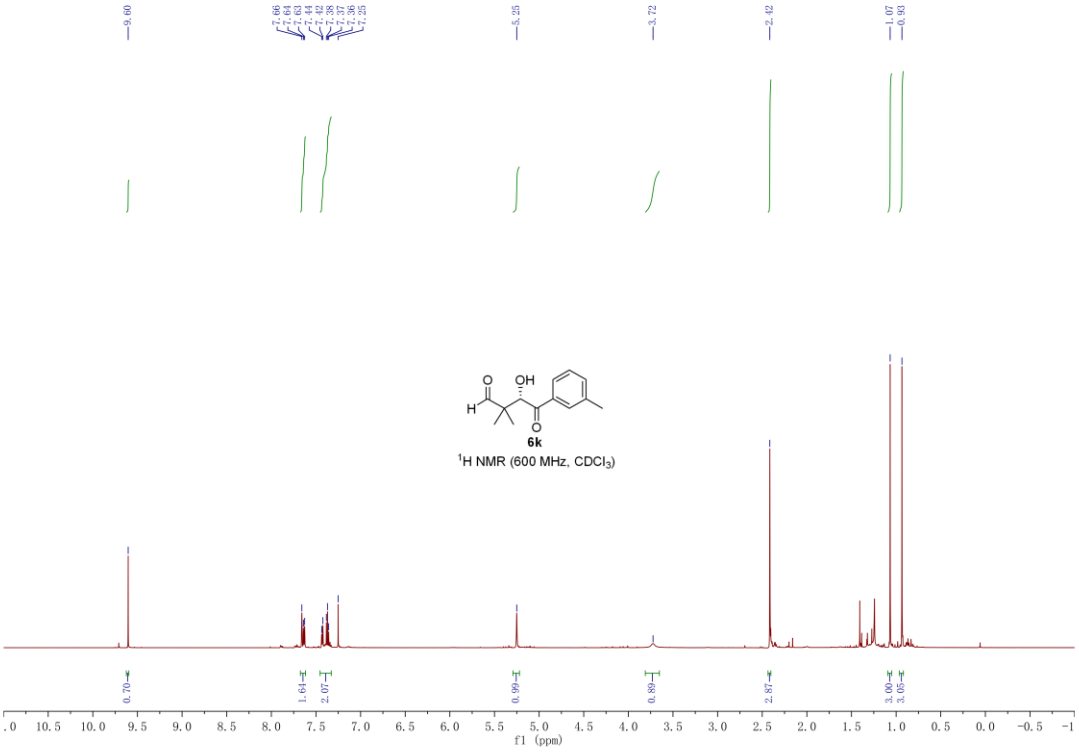

204.16
199.79
134.68
132.29
130.24
129.56
77.33
77.02
76.70
76.34
49.83
19.75
17.81
0.00
O
OH
H
Br
O
6h
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

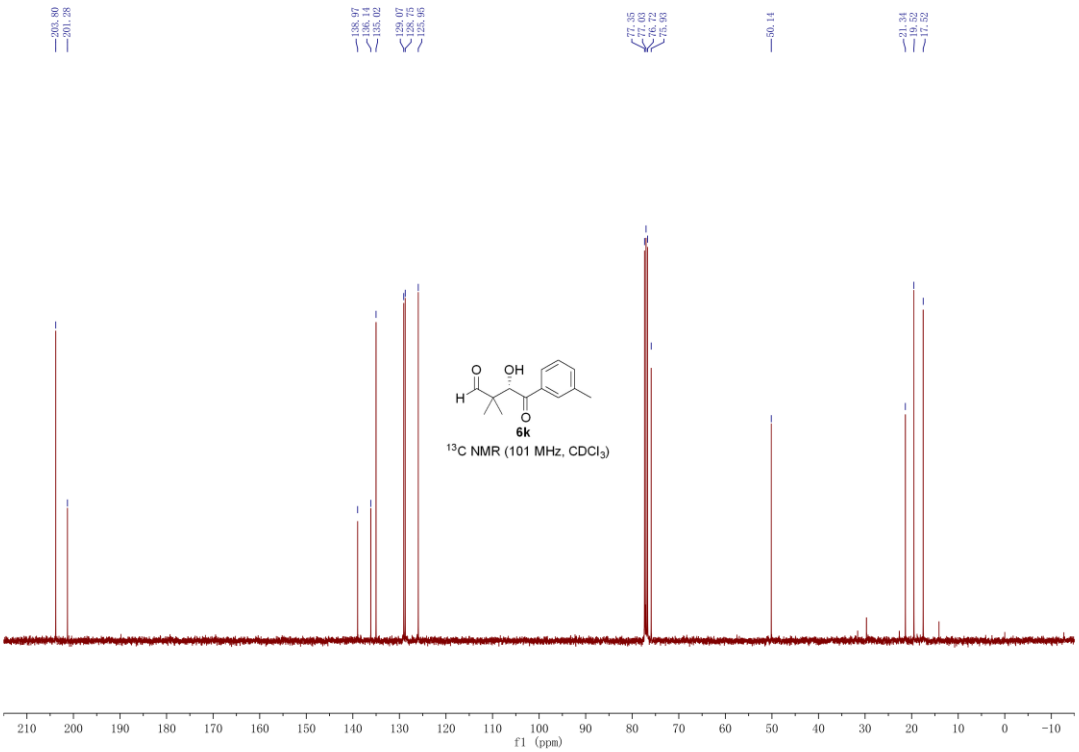

9.64
7.89
7.88
7.87
7.87
7.87
7.27
6.98
6.97
6.97
6.96
6.95
6.94
5.23
3.89
3.75
1.08
0.98
0.00
OMe
OH
O
H
O
6i
^{1}H NMR (600 MHz, CDCl$_3$)
0.83
1.87
2.05
1.03
3.02
0.72
3.00
3.09
f1 (ppm)

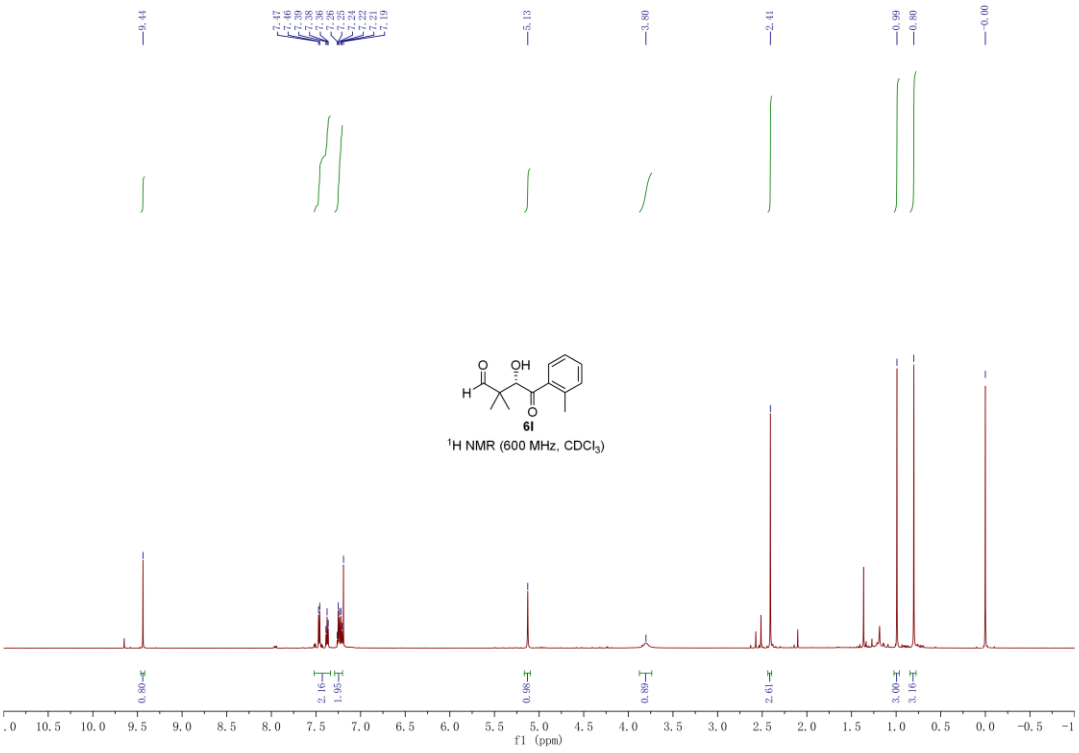

204.12
198.97
164.50
131.25
128.61
114.17
77.36
77.04
76.72
75.46
55.60
50.20
19.58
17.38
OMe
OH
O
H
O
6i
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

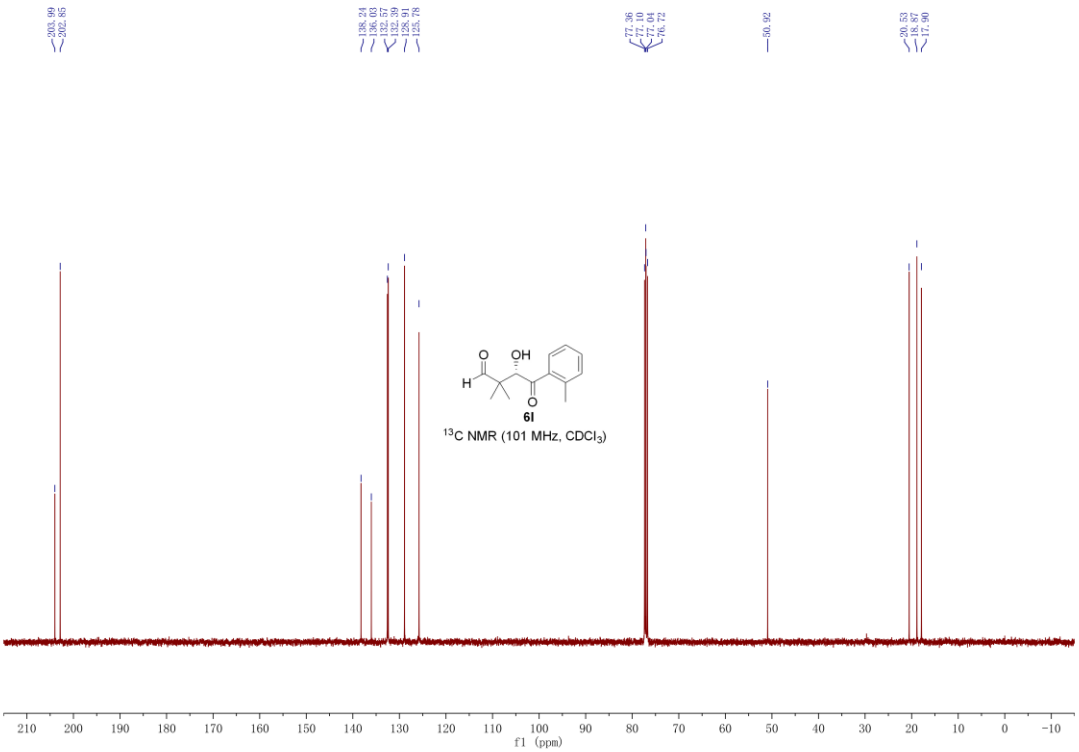

9.61
7.76
7.75
7.29
7.28
7.25
5.24
3.73
2.42
1.06
0.94
OH
O
H
O
6j
^{1}H NMR (600 MHz, CDCl$_3$)
0.72
1.73
1.65
1.00
0.82
3.10
3.00
3.13
f1 (ppm)

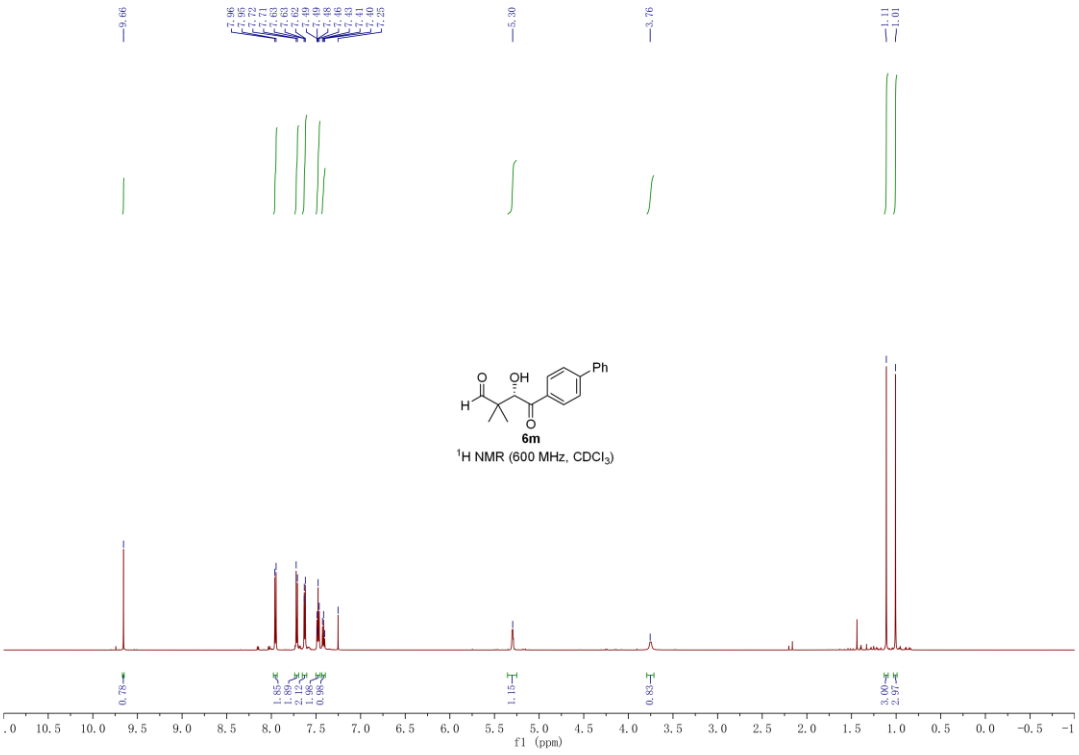

203.86
200.52
145.42
133.43
129.64
128.86
77.35
77.03
76.71
75.69
50.19
21.79
19.52
17.52
OH
O
H
O
6j
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

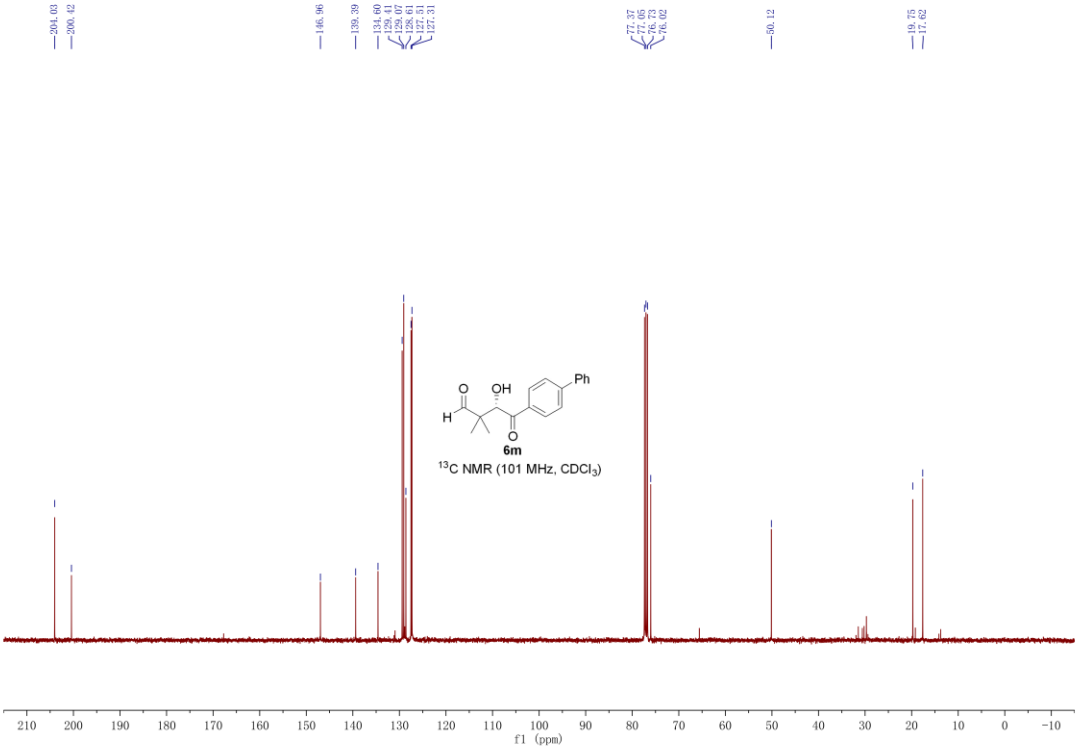

9.60
7.66
7.64
7.63
7.44
7.43
7.38
7.37
7.36
7.25
5.25
3.72
2.42
1.07
0.93
OH
O
H
O
6k
^{1}H NMR (600 MHz, CDCl$_3$)
0.70
1.64
2.07
0.99
0.89
2.87
3.00
3.05
f1 (ppm)

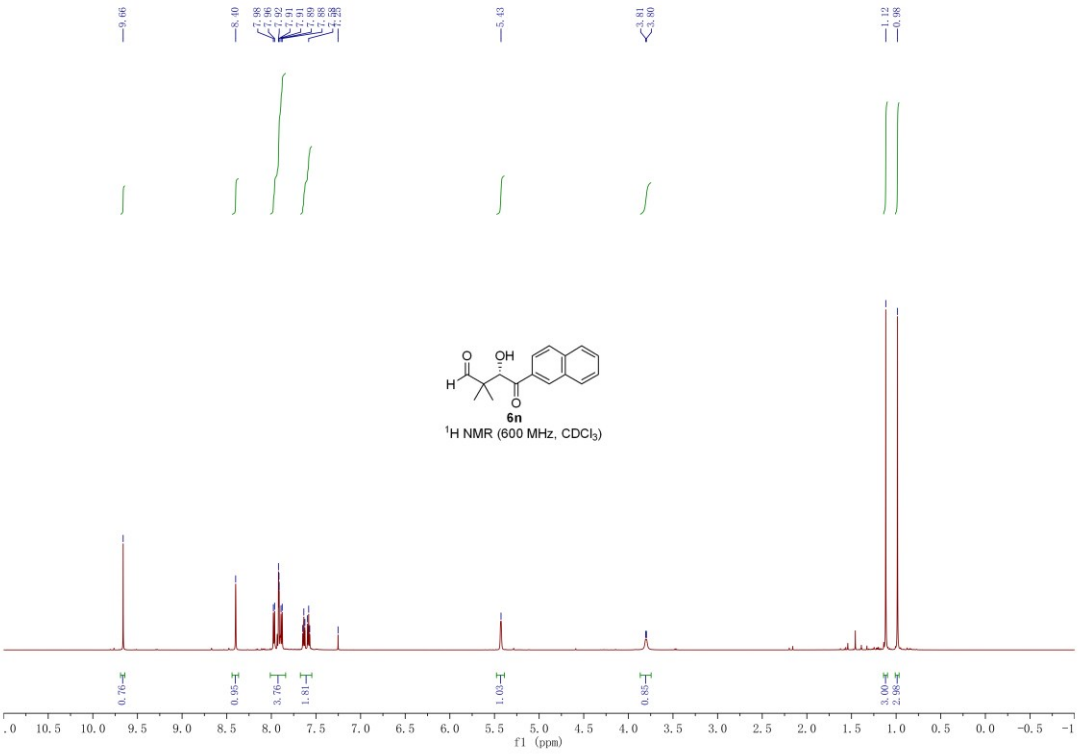

205.80
201.26
138.97
136.14
133.02
129.07
128.75
125.96
77.35
77.03
76.72
75.93
50.14
21.34
19.52
17.52
OH
O
H
O
6k
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

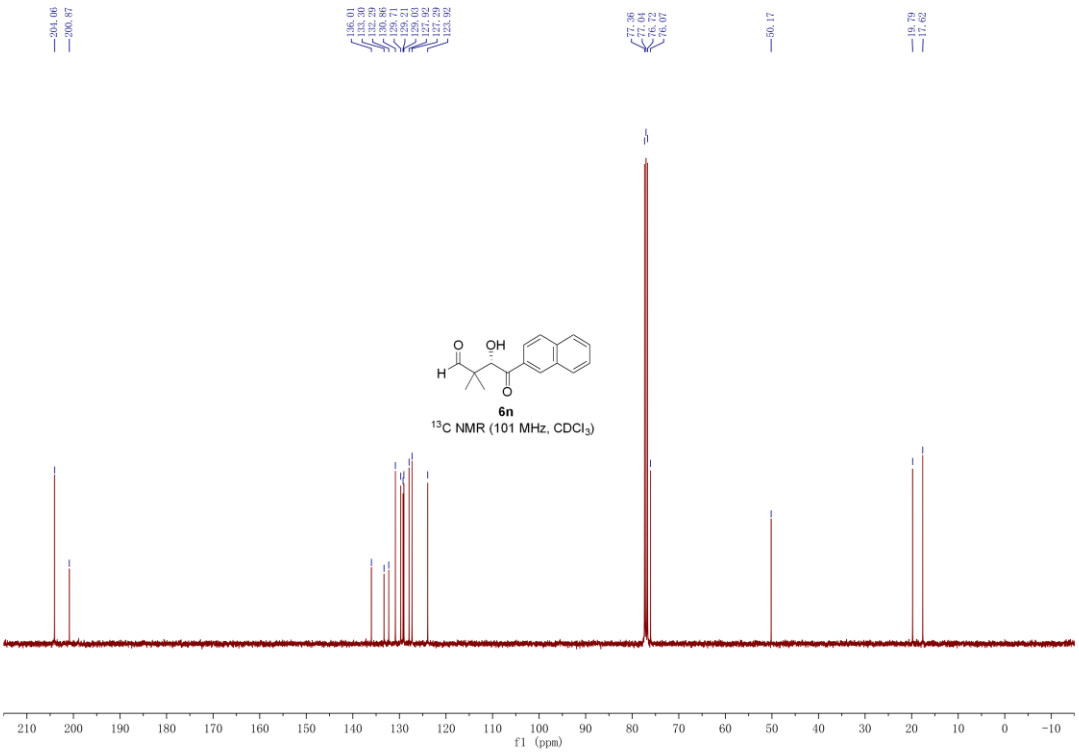

9.44
7.47
7.46
7.39
7.38
7.36
7.35
7.24
7.22
7.21
7.19
5.13
3.80
2.41
0.99
0.80
0.00
OH
O
H
O
6l
1H NMR (600 MHz, CDCl3)
0.80
2.16
1.95
0.98
0.89
2.61
3.00
3.16
f1 (ppm)

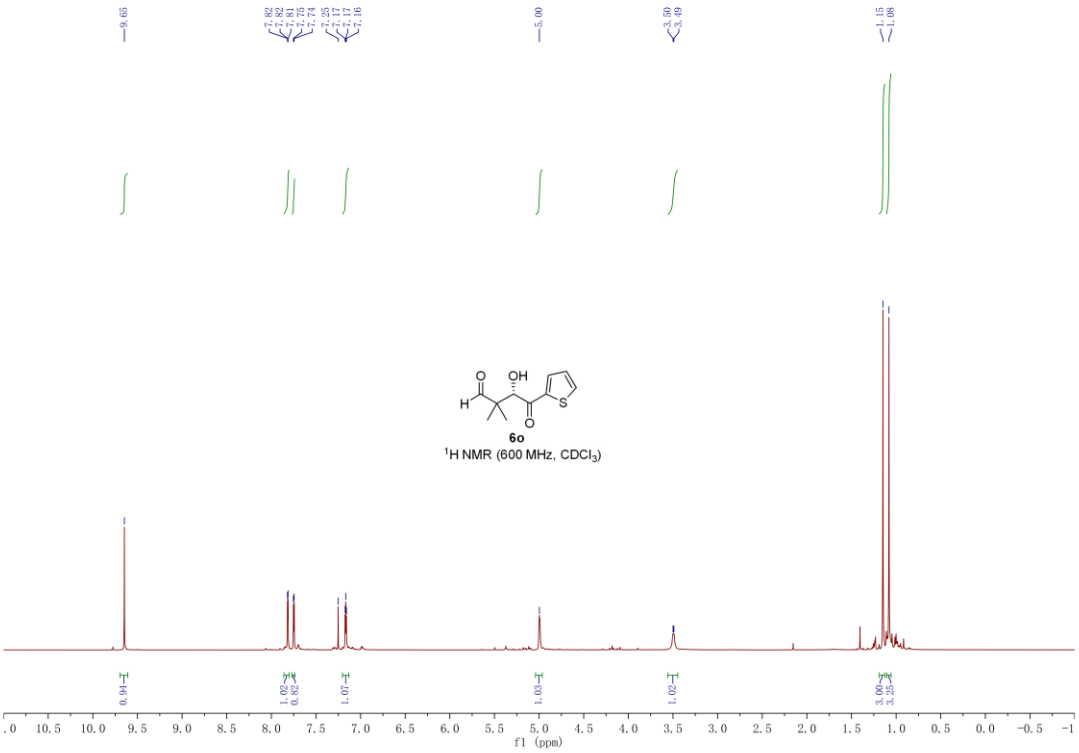

203.99
202.85
138.24
136.10
132.57
132.39
128.91
126.76
77.36
77.10
77.04
76.72
50.92
20.53
18.87
17.90
OH
O
H
O
6l
13C NMR (101 MHz, CDCl3)
f1 (ppm)

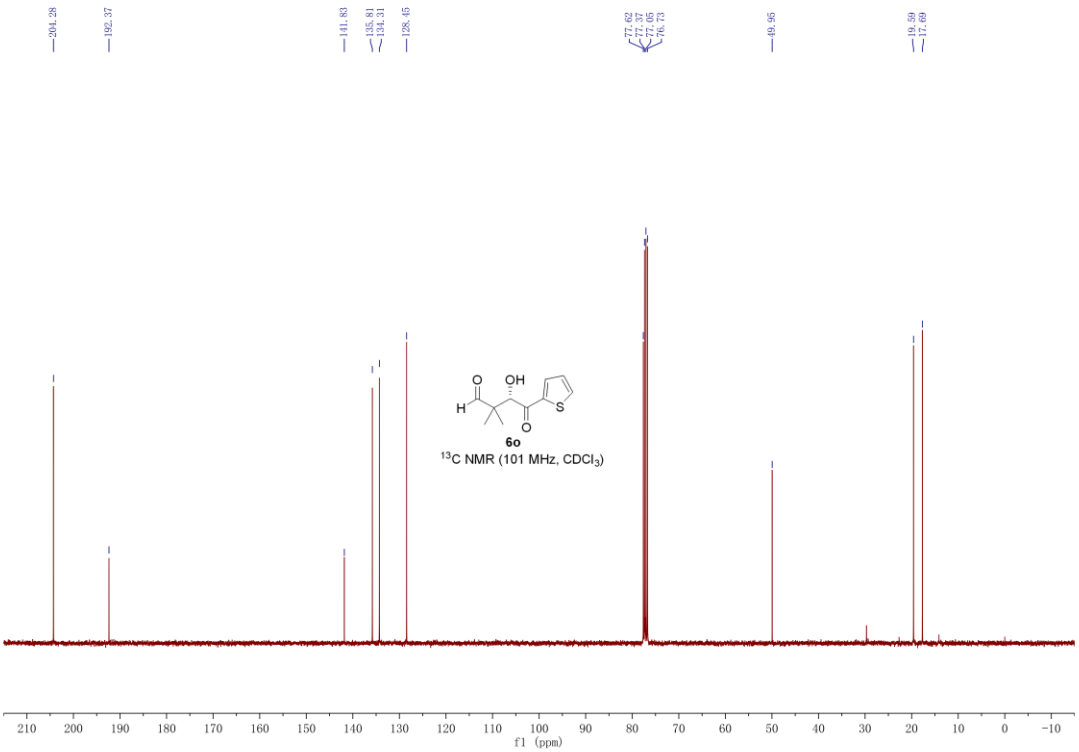

9.66
7.96
7.95
7.72
7.71
7.63
7.63
7.02
7.49
7.49
7.48
7.46
7.43
7.41
7.40
7.25
5.30
3.76
1.11
1.01
O
OH
H
O
Ph
6m
1H NMR (600 MHz, CDCl3)
0.78
1.85
1.89
2.12
1.98
0.98
1.15
0.83
3.00
2.97
f1 (ppm)

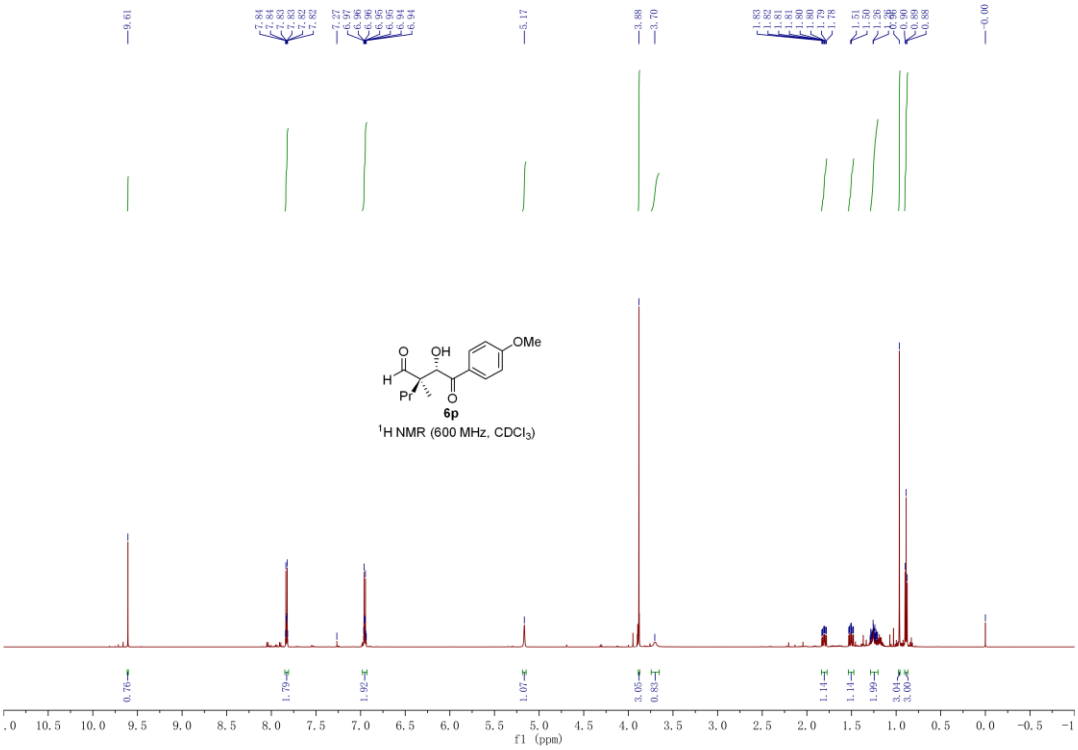

204.03
200.42
146.96
139.39
134.60
129.41
129.07
128.61
127.51
127.31
77.37
77.05
76.73
76.02
60.12
19.75
17.62
O
OH
H
O
Ph
6m
13C NMR (101 MHz, CDCl3)
f1 (ppm)

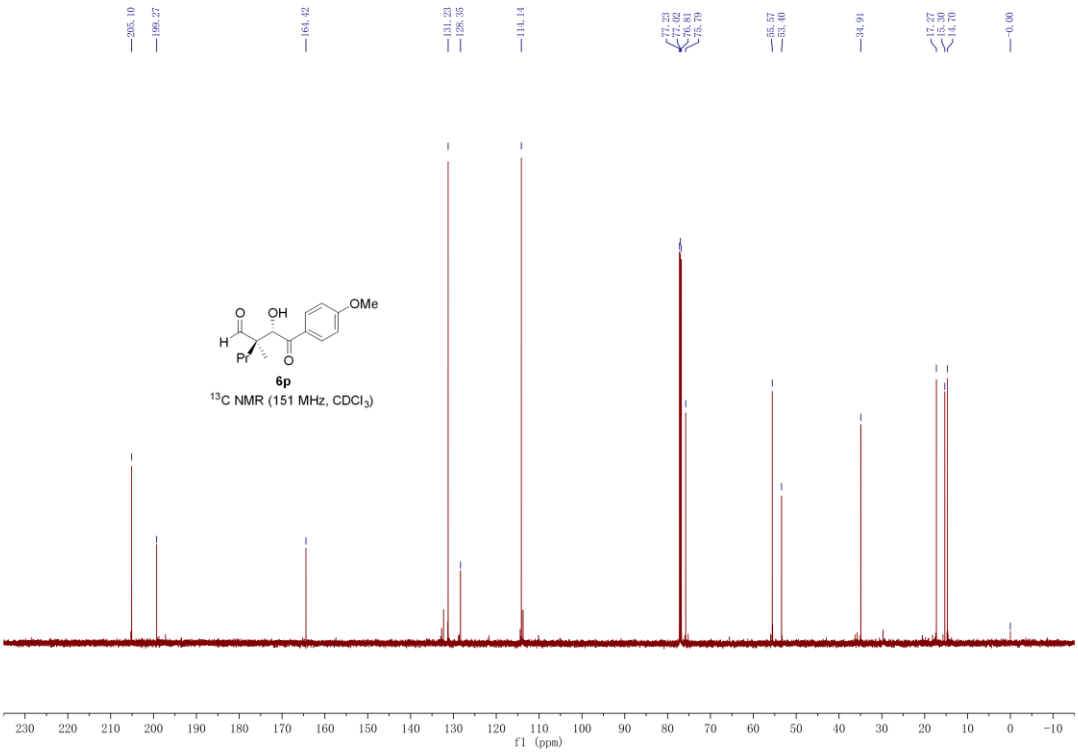

9.66
8.40
7.98
7.96
7.92
7.91
7.91
7.89
7.88
7.85
5.43
3.81
3.80
1.12
0.98
O
OH
H
O
6n
1H NMR (600 MHz, CDCl3)
0.76
0.95
3.76
1.81
1.03
0.85
3.00
2.98
f1 (ppm)

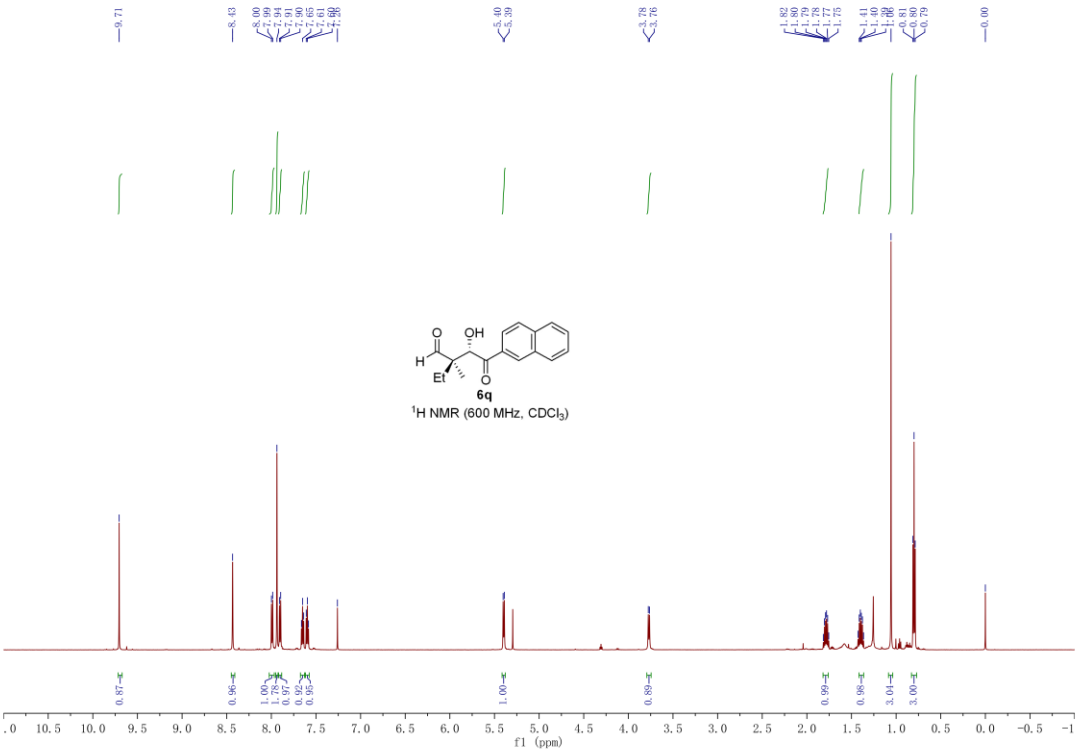

204.06
200.87
136.01
133.30
132.29
130.86
129.71
129.21
128.65
127.92
127.29
123.92
77.36
77.04
76.72
76.07
60.17
19.79
17.62
O
OH
H
O
6n
13C NMR (101 MHz, CDCl3)
f1 (ppm)

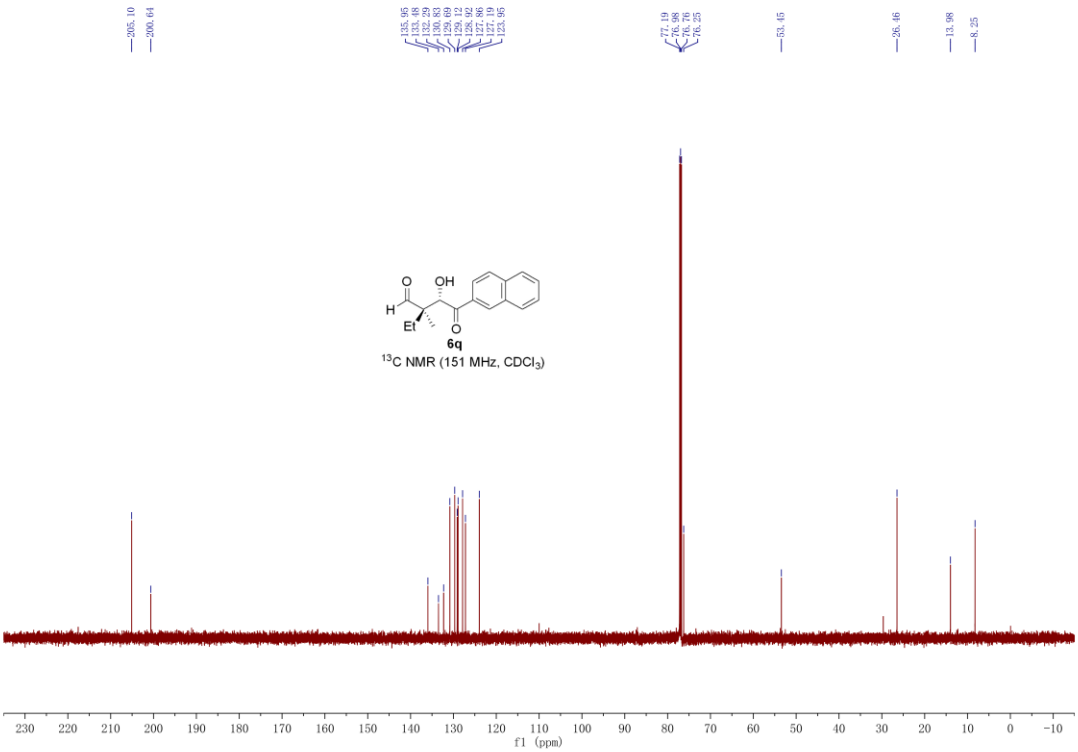

9.65
7.82
7.82
7.81
7.75
7.74
7.25
7.17
7.17
7.16
5.00
3.50
3.49
1.15
1.08
O
OH
H
S
O
6o
^{1}H NMR (600 MHz, CDCl$_3$)
0.94
1.02
0.82
1.07
1.03
1.02
3.00
3.25
f1 (ppm)

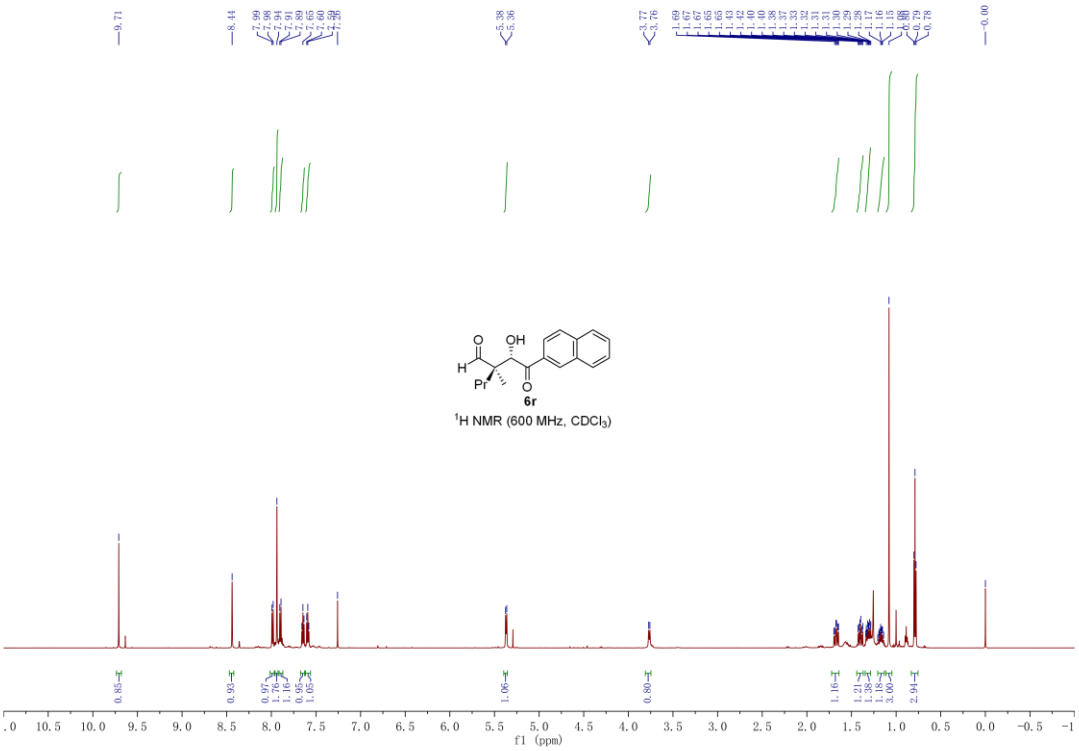

204.28
192.37
141.83
135.81
134.31
128.45
77.62
77.37
77.05
76.73
49.95
19.59
17.69
O
OH
H
S
O
6o
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

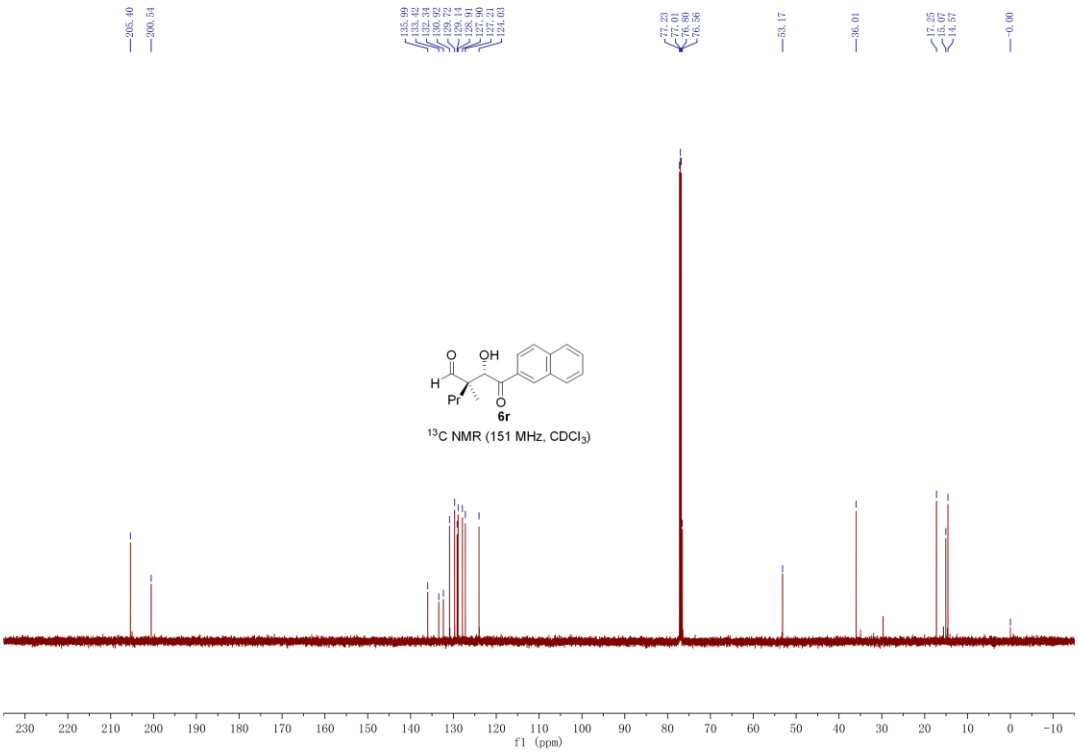

9.61
7.84
7.84
7.83
7.83
7.82
7.82
7.27
6.97
6.96
6.96
6.95
6.95
6.94
6.94
5.17
3.88
3.70
1.83
1.82
1.81
1.81
1.80
1.80
1.79
1.78
1.51
1.50
1.35
1.34
0.90
0.89
0.88
0.00
OH
O
H
Pr
O
OMe
6p
1H NMR (600 MHz, CDCl3)
0.76
1.79
1.92
1.07
3.05
0.83
1.14
1.14
1.99
3.04
3.00
f1 (ppm)

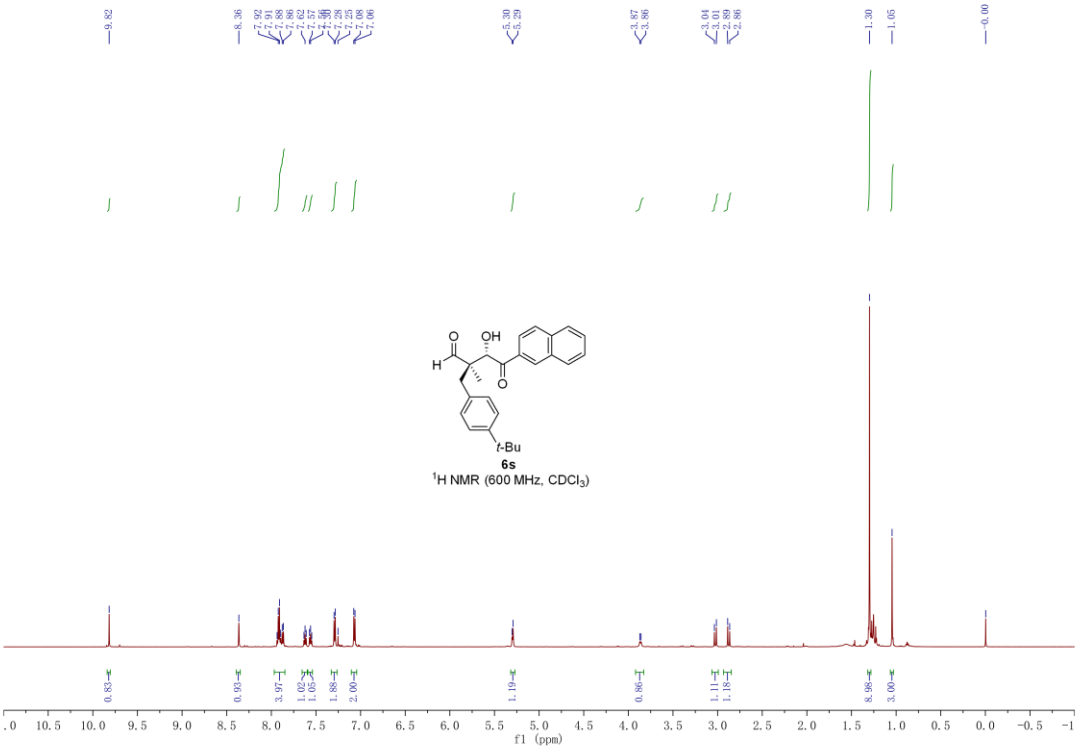

205.10
199.27
164.42
131.23
128.35
114.14
77.23
77.02
76.81
75.79
55.57
53.40
34.91
17.27
15.30
14.70
0.00
OH
O
H
Pr
O
OMe
6p
13C NMR (151 MHz, CDCl3)
f1 (ppm)

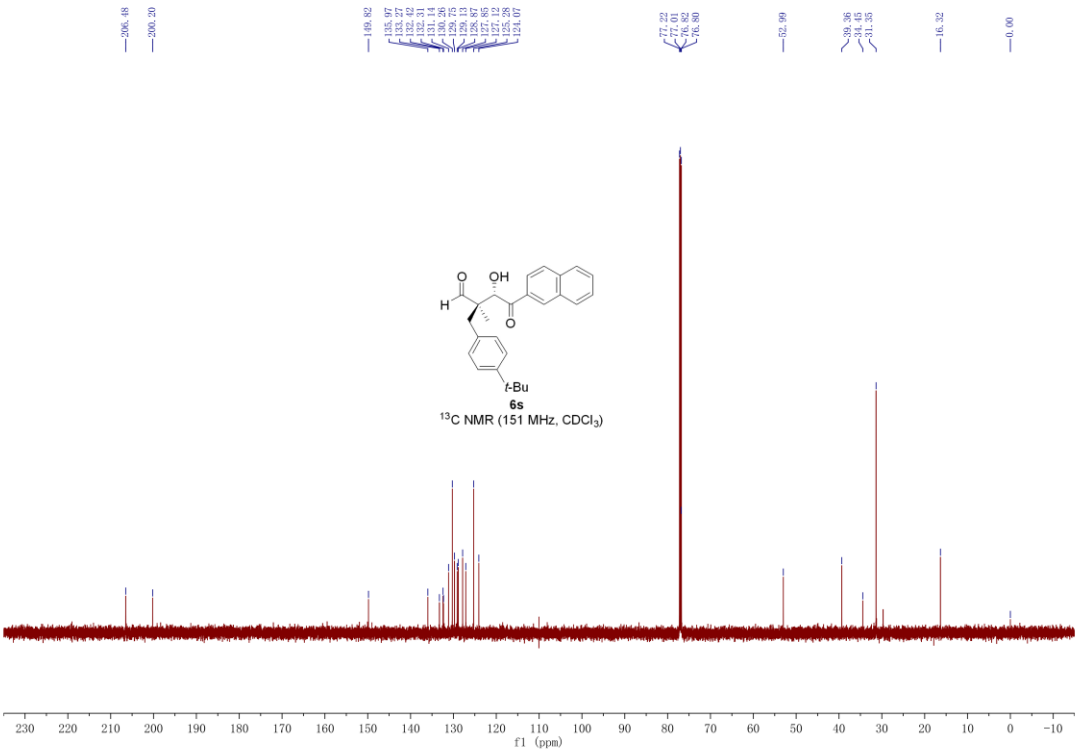

9.71
8.43
8.00
7.99
7.94
7.91
7.90
7.65
7.61
5.40
5.39
3.78
3.76
1.82
1.80
1.79
1.78
1.77
1.75
1.41
1.40
0.81
0.80
0.79
0.00
O
OH
H
Et
O
6q
^{1}H NMR (600 MHz, CDCl$_3$)
0.87
0.96
1.00
1.78
0.97
0.92
0.95
1.00
0.89
0.99
0.98
3.04
3.00
f1 (ppm)

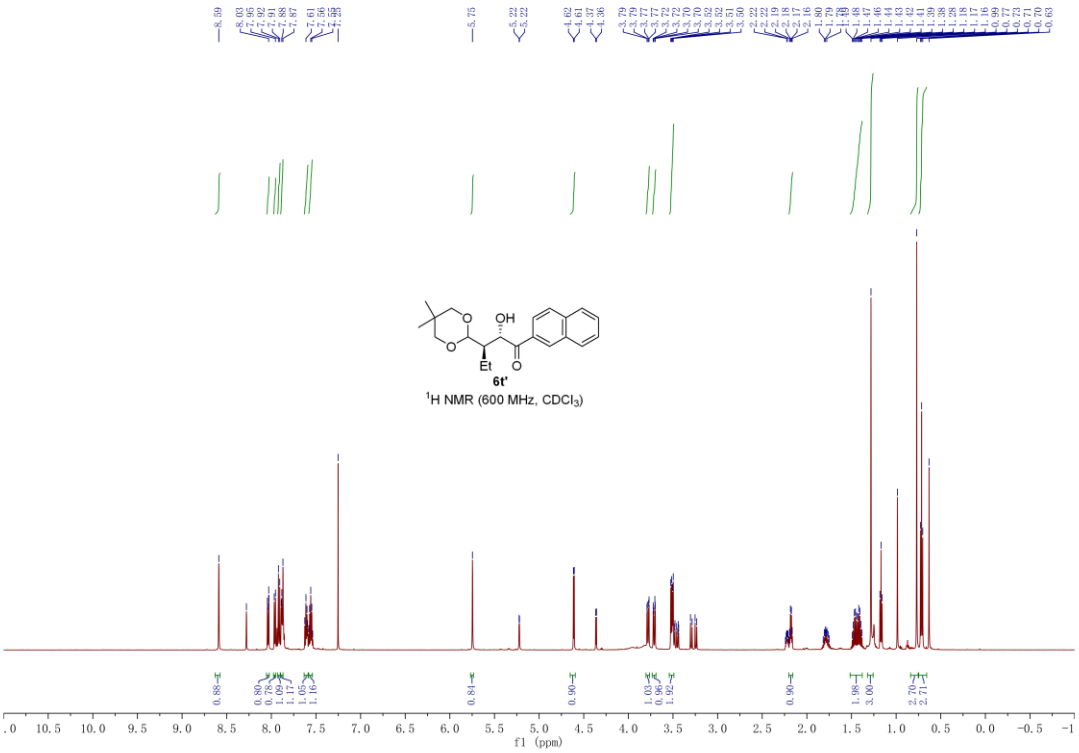

205.10
200.64
135.95
133.48
132.29
130.83
129.69
129.12
128.92
127.86
127.19
123.95
77.19
76.98
76.70
76.25
53.45
26.46
13.98
8.25
O
OH
H
Et
O
6q
^{13}C NMR (151 MHz, CDCl$_3$)
f1 (ppm)

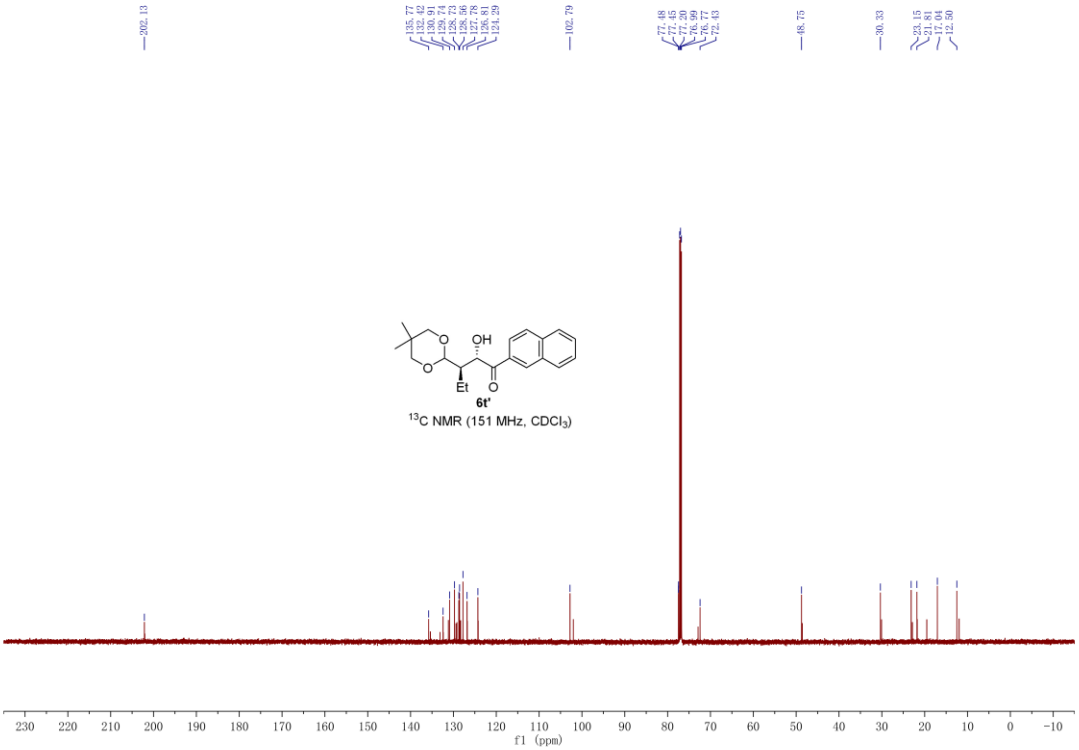

6r
^{1}H NMR (600 MHz, CDCl$_3$)
f1 (ppm)

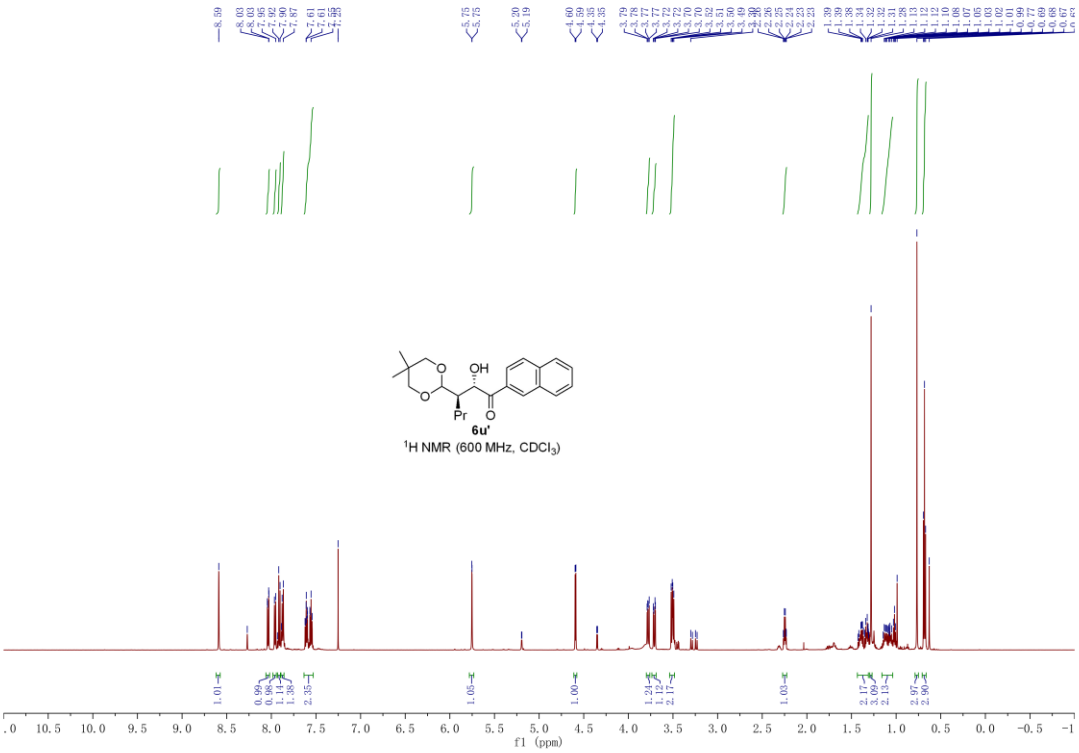

6r
^{13}C NMR (151 MHz, CDCl$_3$)
f1 (ppm)

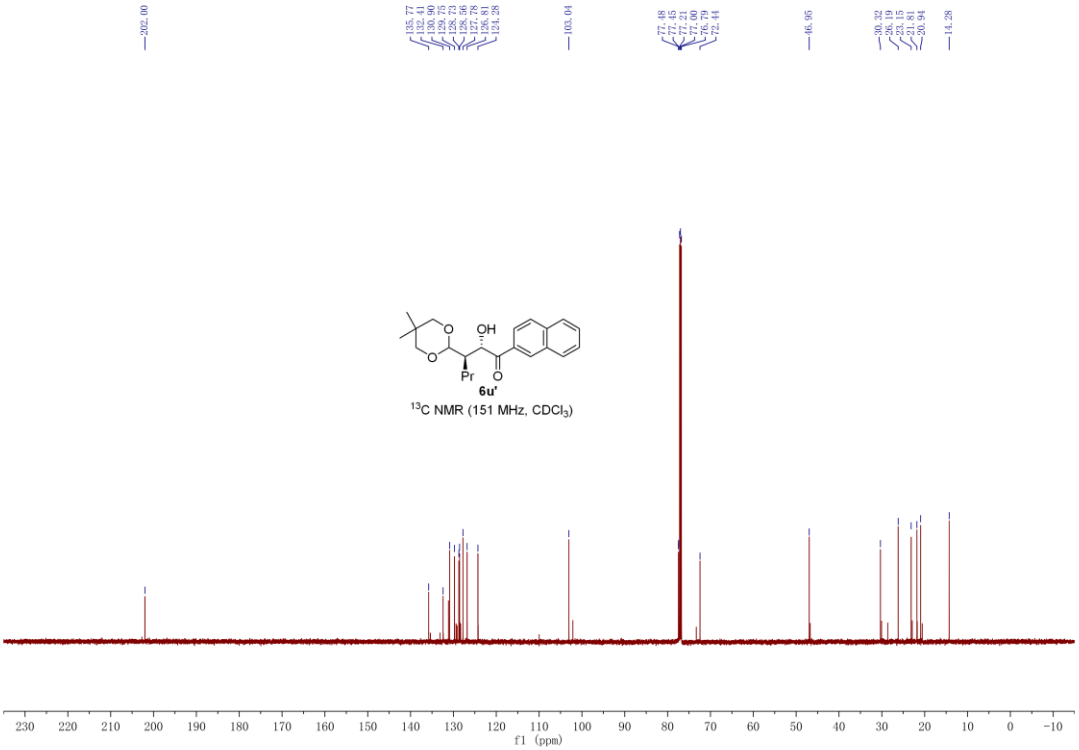

9.82
8.36
7.92
7.91
7.88
7.86
7.62
7.57
7.28
7.25
7.08
7.06
5.30
5.29
3.87
3.86
3.04
3.01
2.89
2.86
1.30
1.05
0.00
OH
O
H
O
t-Bu
6s
1H NMR (600 MHz, CDCl3)
0.83
0.93
3.97
1.02
1.05
1.88
2.00
1.19
0.86
1.11
1.18
8.98
3.00
f1 (ppm)

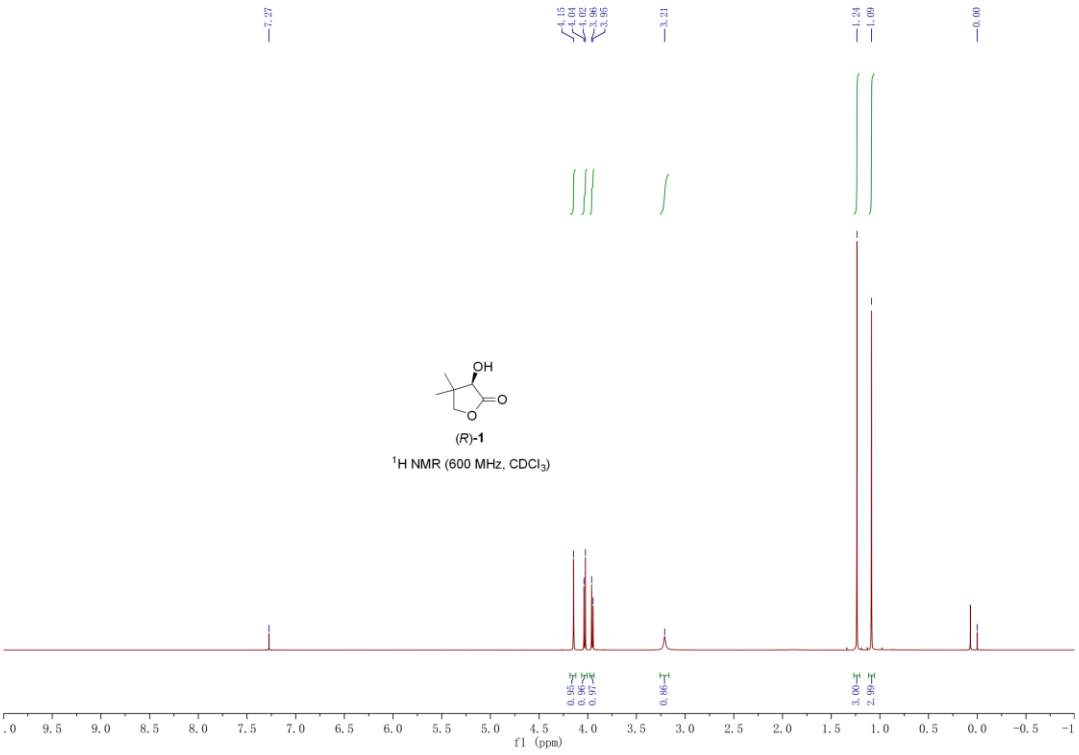

206.48
200.20
149.82
135.97
133.27
132.42
132.31
131.14
130.26
129.75
129.13
128.87
127.65
127.12
125.28
124.07
77.22
77.01
76.82
76.80
52.99
39.36
34.45
31.35
16.32
0.00
OH
O
H
O
t-Bu
6s
13C NMR (151 MHz, CDCl3)
f1 (ppm)

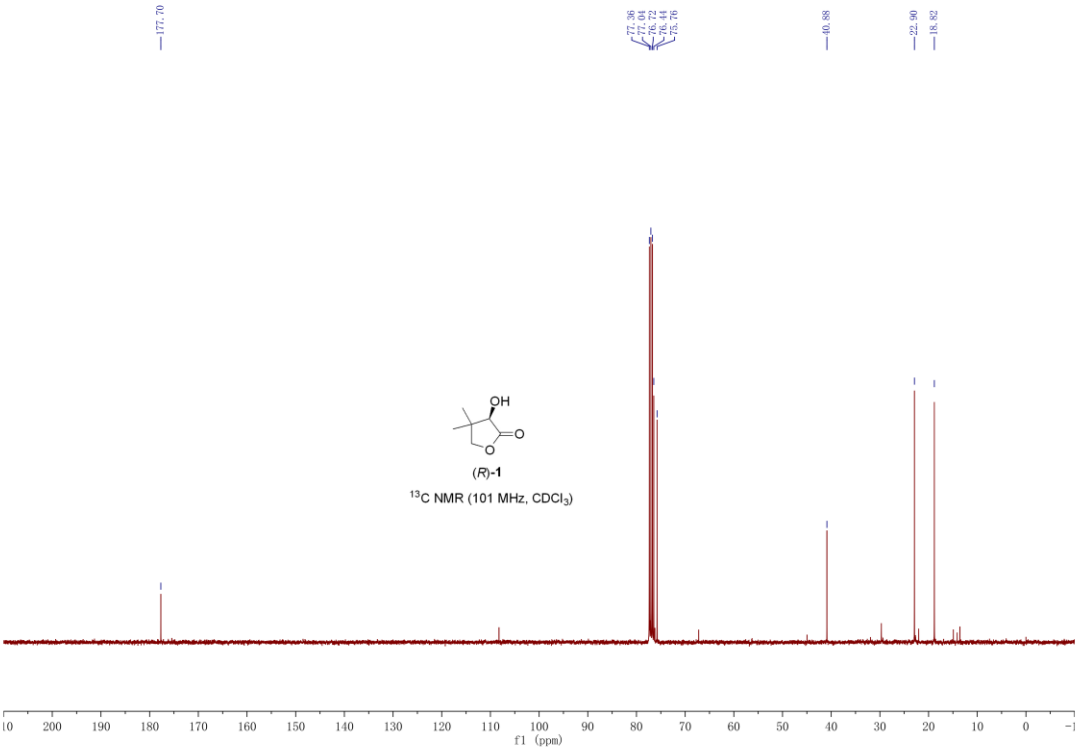

OH
Et
O
6t'
^{1}H NMR (600 MHz, CDCl$_3$)
f1 (ppm)

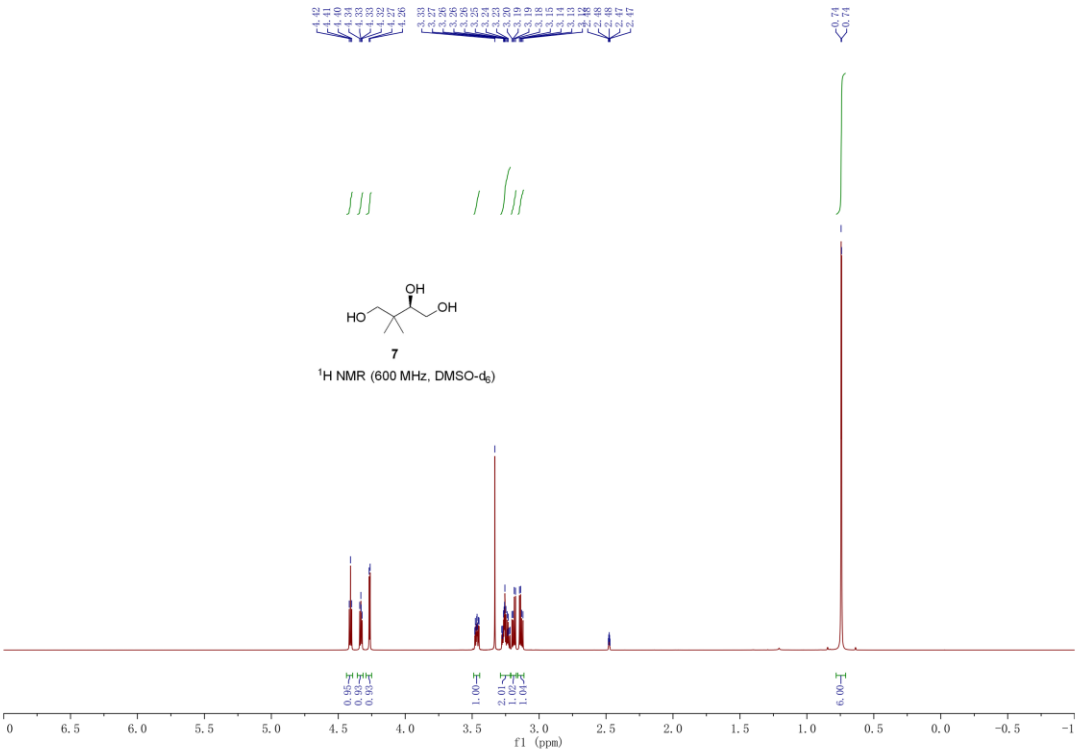

OH
Et
O
6t'
^{13}C NMR (151 MHz, CDCl$_3$)
f1 (ppm)

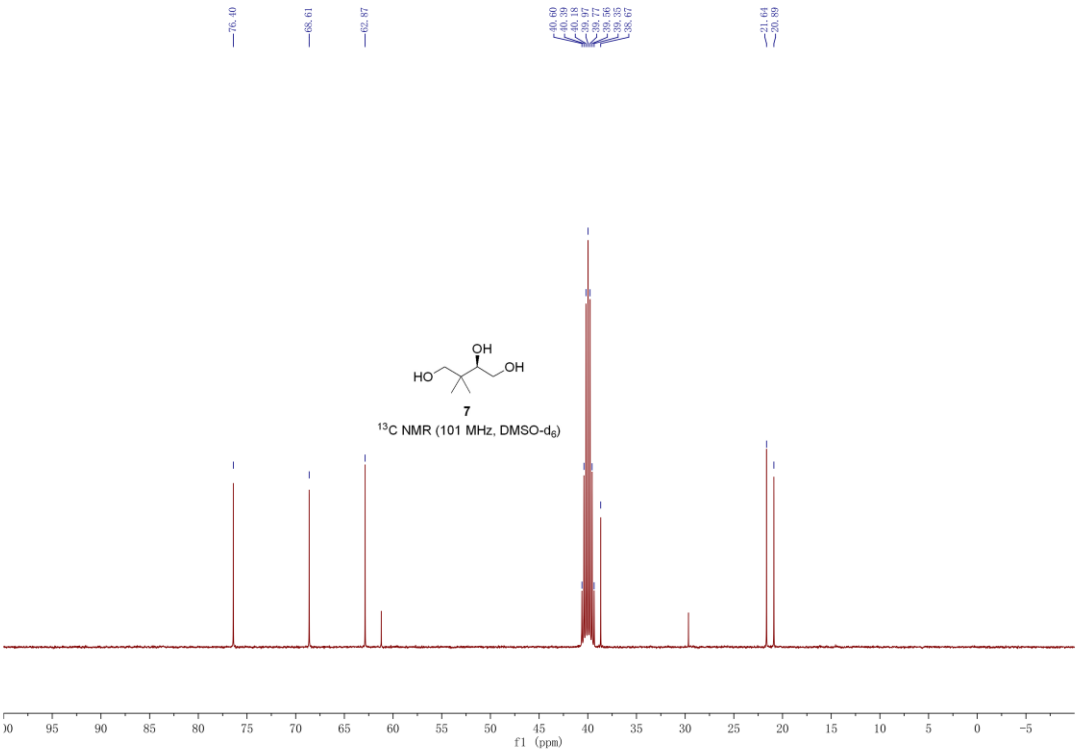

OH
O
O
Pr
O
6u'
1H NMR (600 MHz, CDCl3)
f1 (ppm)


OH
O
O
Pr
O
6u'
13C NMR (151 MHz, CDCl3)
f1 (ppm)

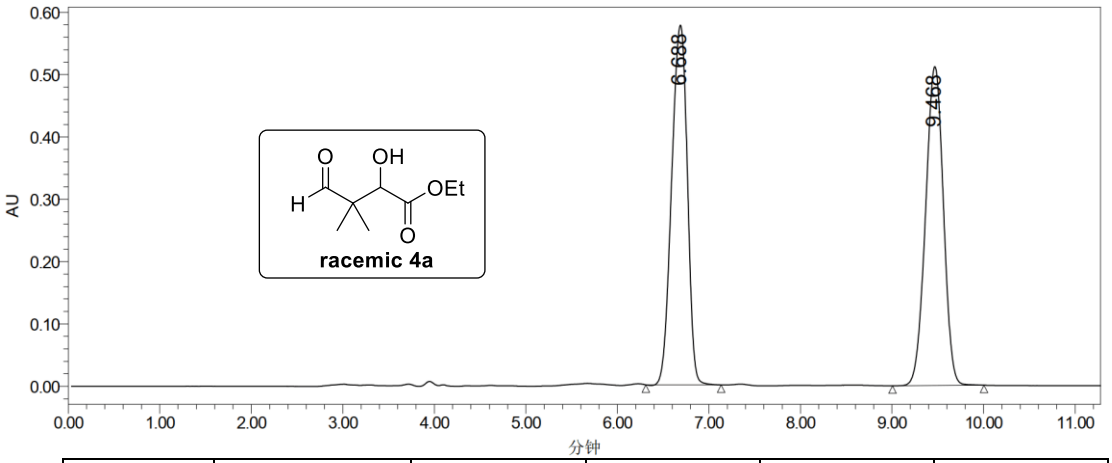

7.27
4.15
4.04
4.02
3.96
3.95
3.21
1.24
1.09
0.00
OH
O
O
(R)-1
1H NMR (600 MHz, CDCl3)
0.95
0.96
0.97
0.86
3.00
2.99
f1 (ppm)


177.70
77.36
77.04
76.72
76.44
75.76
40.88
22.90
18.82
OH
O
O
(R)-1
13C NMR (101 MHz, CDCl3)
f1 (ppm)

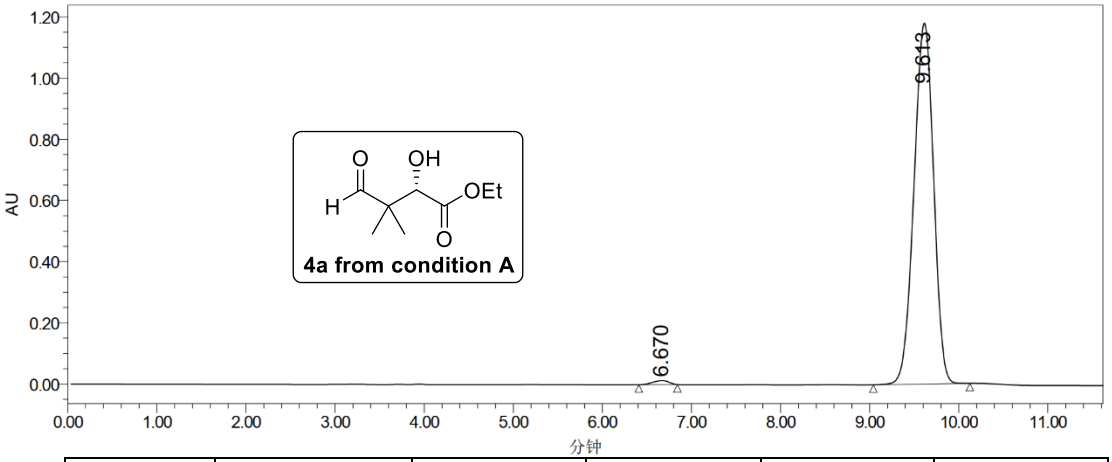

4.42
4.41
4.40
4.34
4.33
4.32
4.27
4.26
3.33
3.27
3.26
3.26
3.25
3.24
3.23
3.20
3.19
3.19
3.18
3.15
3.14
3.13
2.48
2.48
2.48
2.47
2.47
0.74
0.74
OH
HO
OH
7
1H NMR (600 MHz, DMSO-d6)
0.96
0.93
0.93
0.93
1.00
2.01
1.02
1.04
6.00
f1 (ppm)


76.40
68.61
62.87
40.60
40.39
40.18
39.97
39.77
39.56
39.35
38.67
21.64
20.89
OH
HO
OH
7
13C NMR (101 MHz, DMSO-d6)
f1 (ppm)

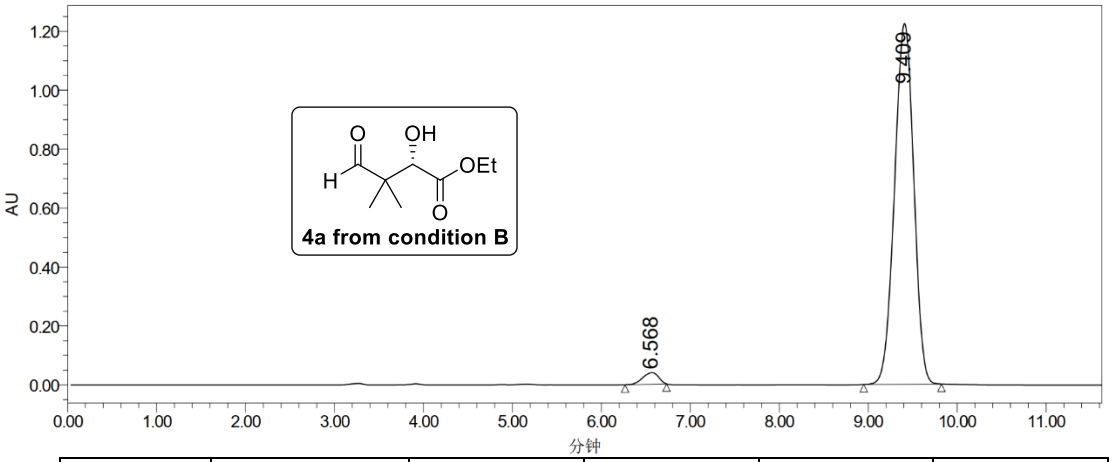
7. Copies of HPLC Chromatograms



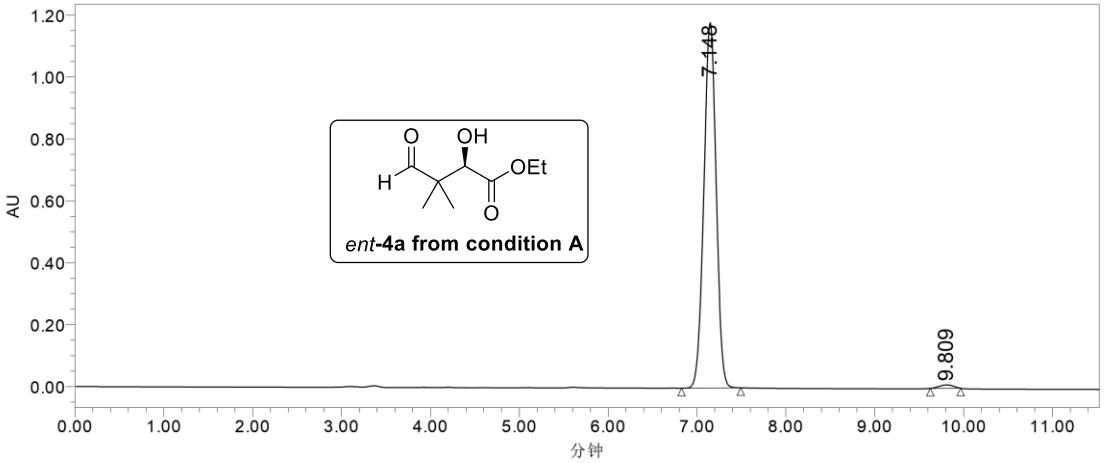
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.688	6893497	577087	49.87
2	2998(210-400)	9.468	6928813	511698	50.13



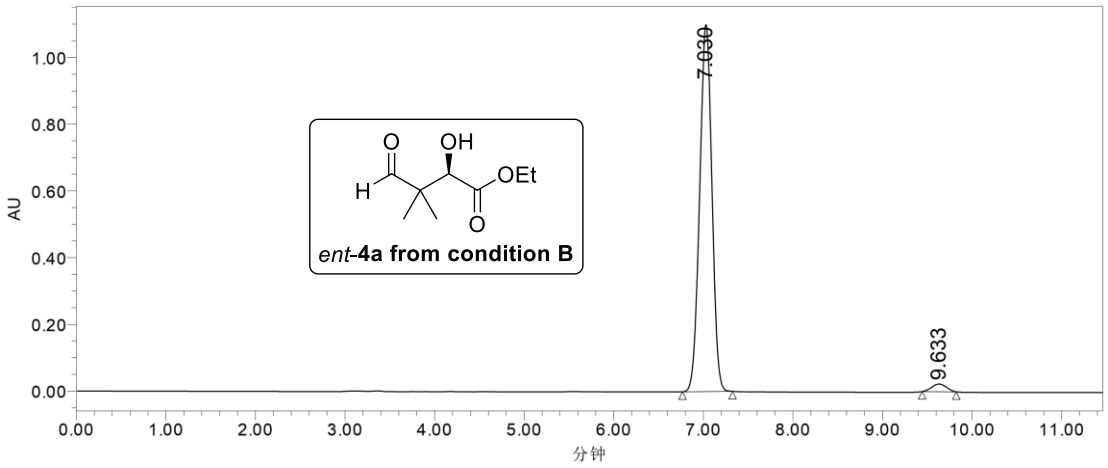
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.670	164190	13068	0.90
2	2998(210-400)	9.613	18074436	1179686	99.10



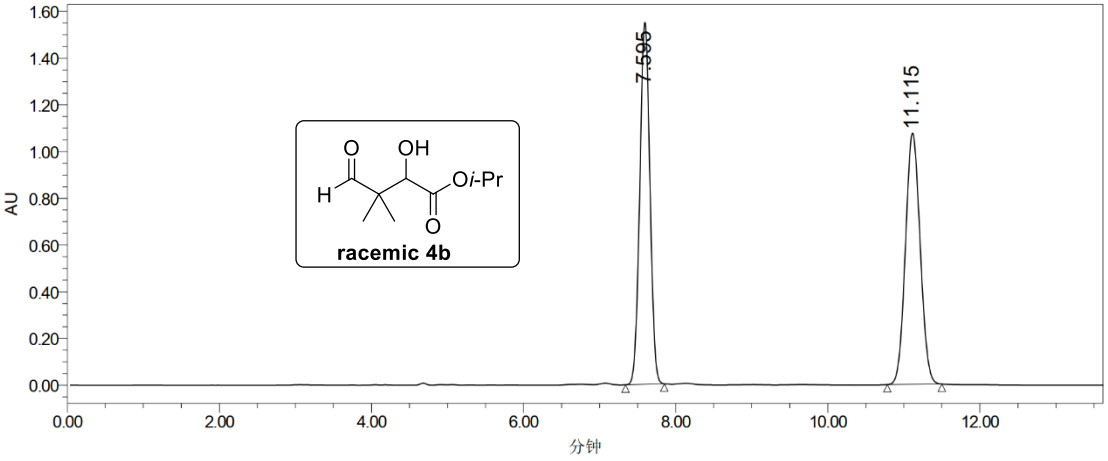
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.568	486244	39375	2.58
2	2998(210-400)	9.409	18374455	1224582	97.42



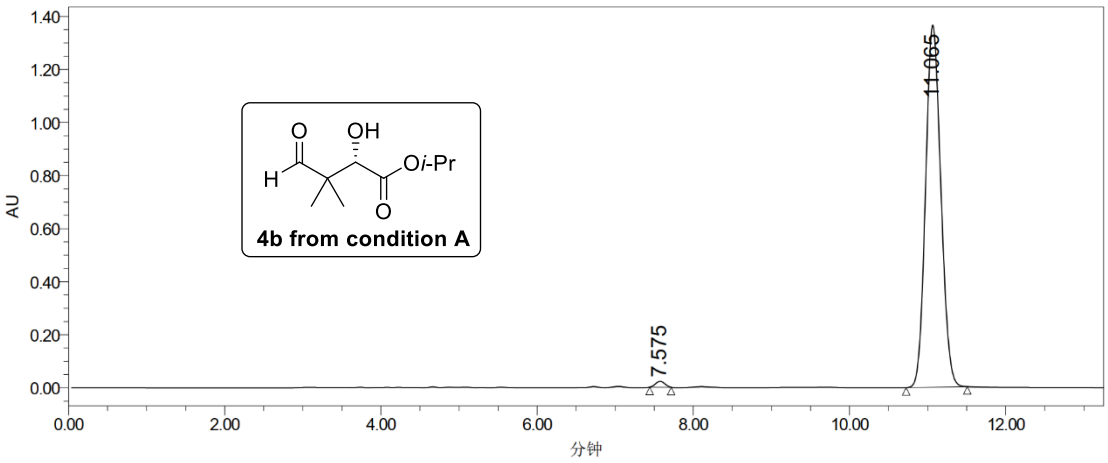
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.148	11361155	1180276	98.94
2	2998(210-400)	9.809	122242	11196	1.06



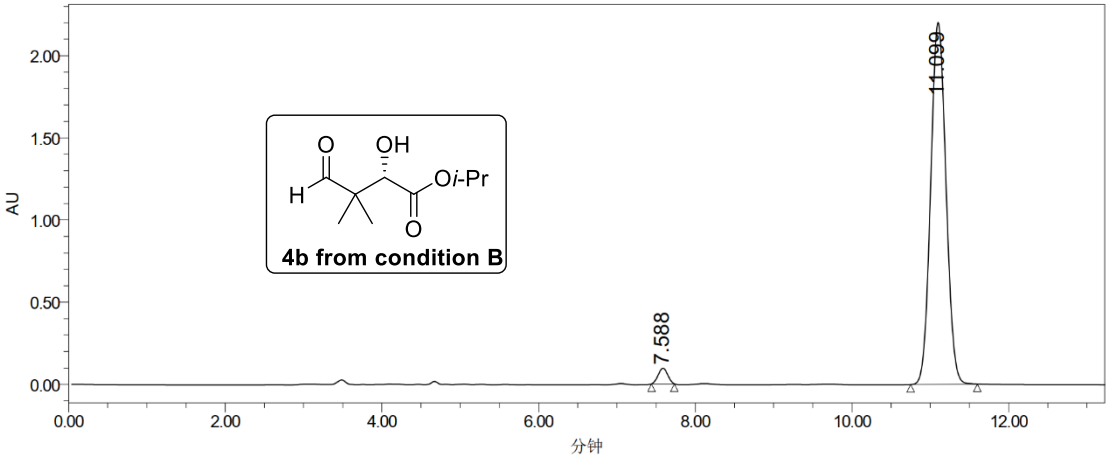
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.030	10463044	1101066	97.55
2	2998(210-400)	9.633	262829	23056	2.45



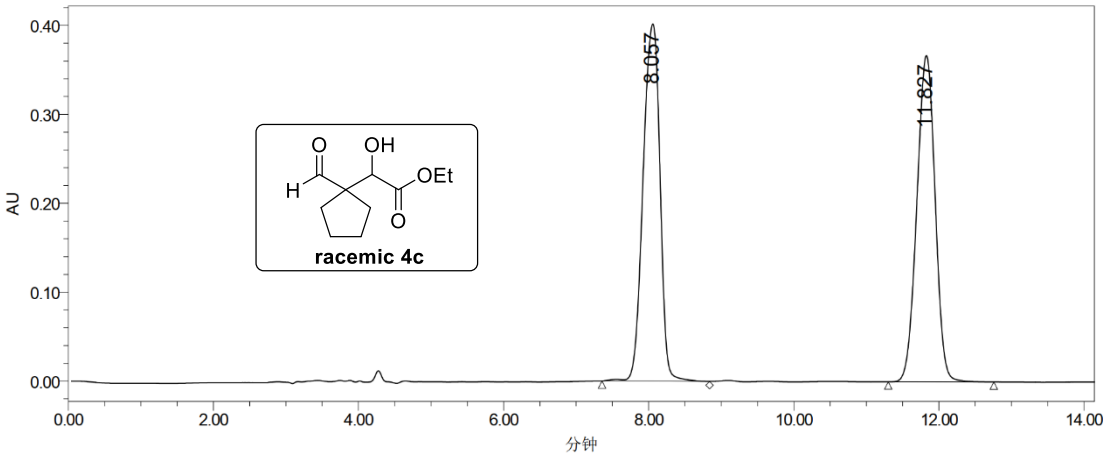
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.595	14325112	1551243	49.80
2	2998(210-400)	11.115	14441212	1075878	50.20



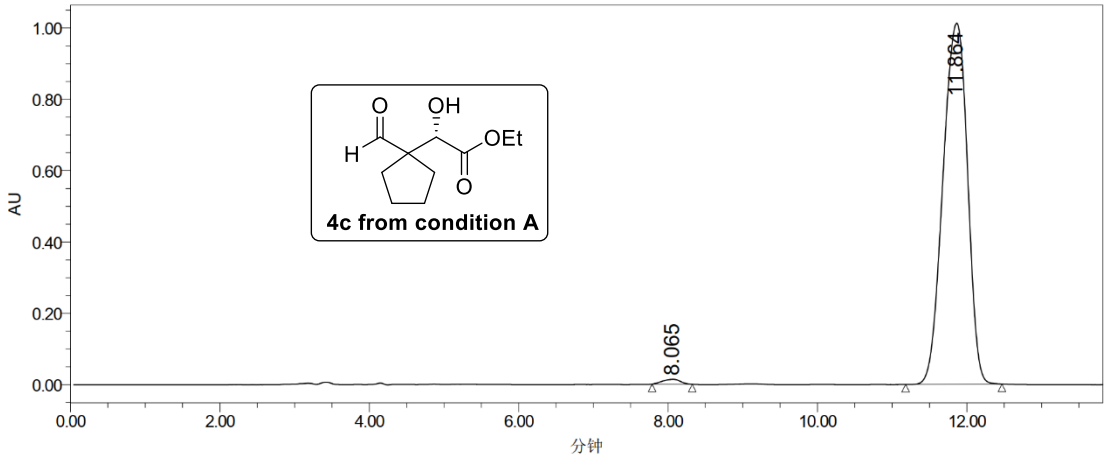
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.575	183725	22253	0.98
2	2998(210-400)	11.065	18526199	1365259	99.02



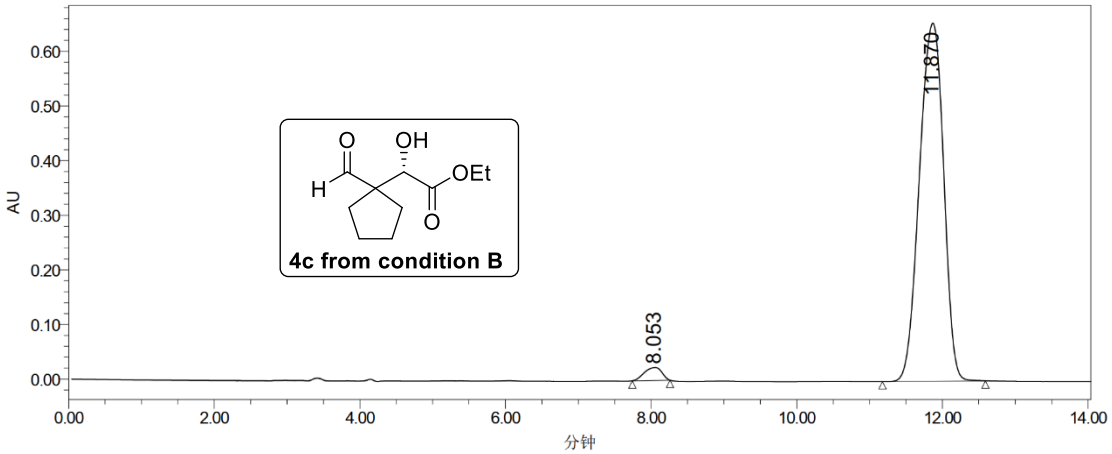
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.588	795634	94489	2.59
2	2998(210-400)	11.099	29870520	2202071	97.41



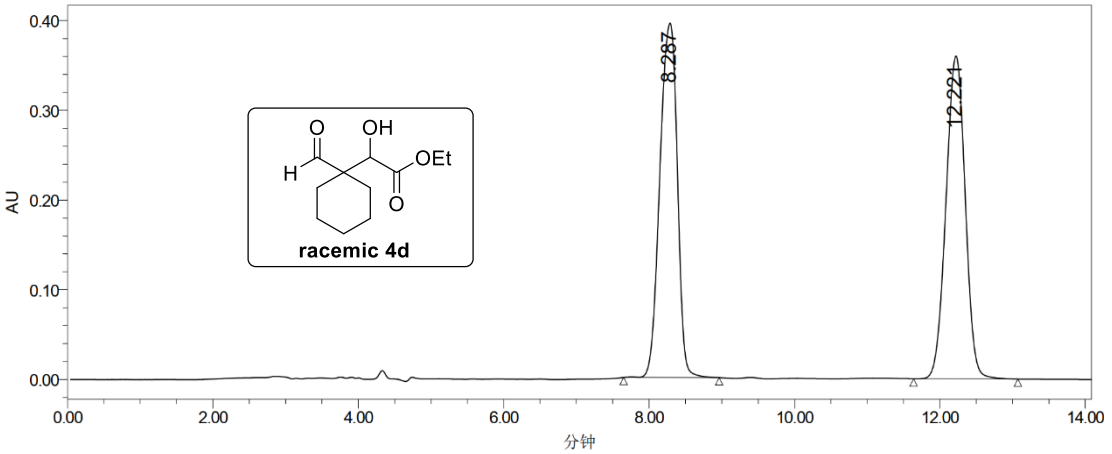
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.057	6490345	401586	50.25
2	2998(210-400)	11.827	6426742	366942	49.75



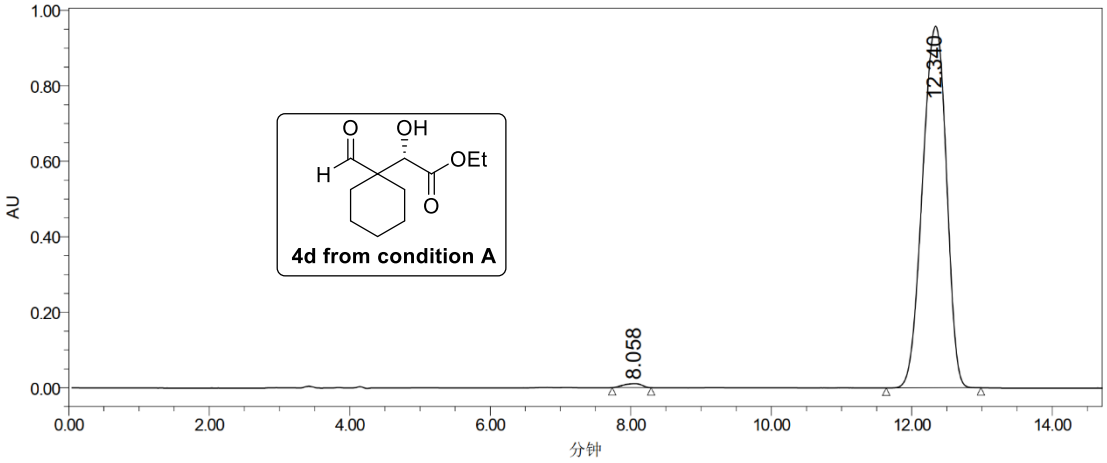
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.065	222127	13492	0.95
2	2998(210-400)	11.864	23061652	1011859	99.05



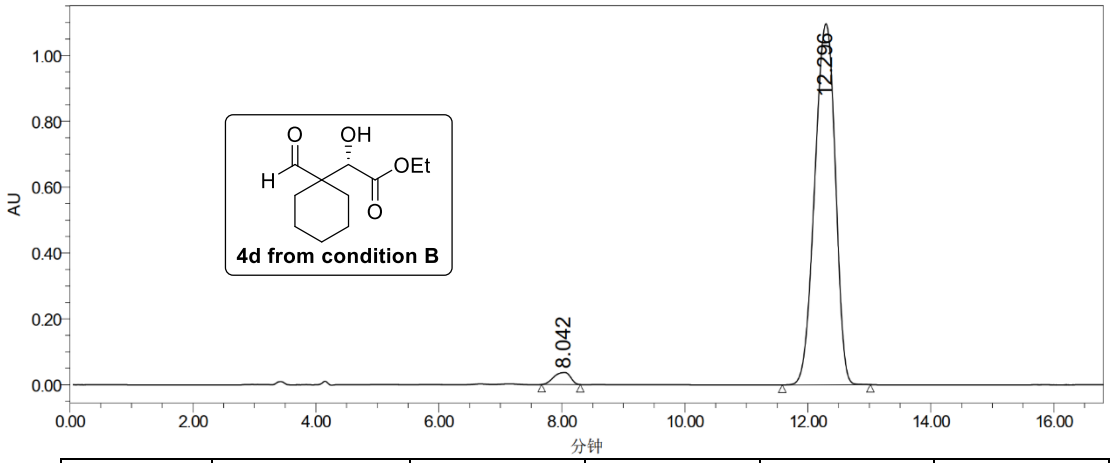
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.053	393803	23658	2.54
2	2998(210-400)	11.870	15121264	655393	97.46



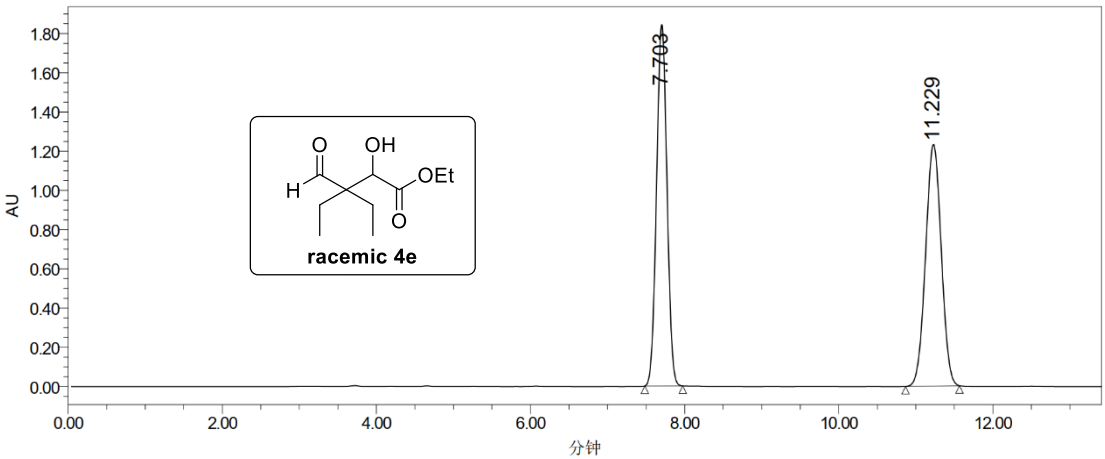
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.287	6585768	395028	49.92
2	2998(210-400)	12.221	6607887	359849	50.08



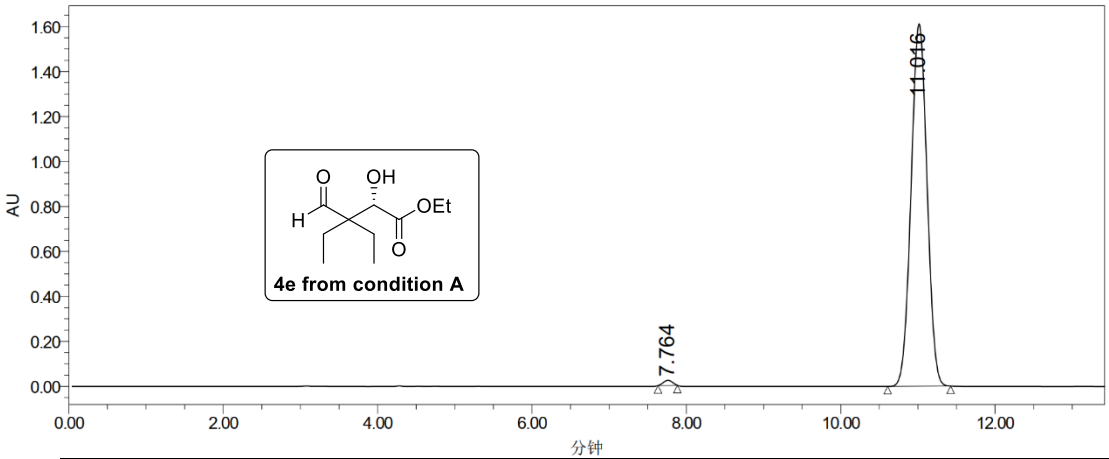
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.058	190525	10485	0.83
2	2998(210-400)	12.340	22650669	958164	99.17



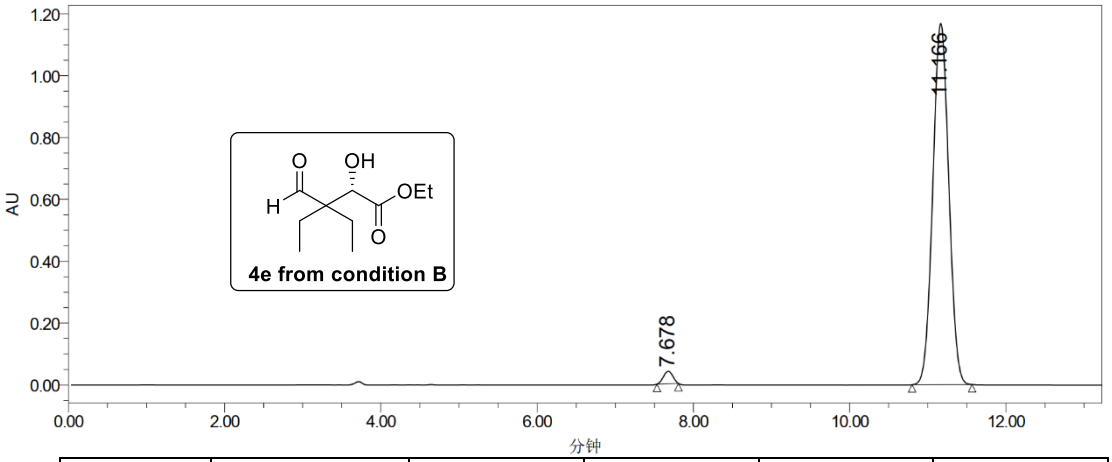
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.042	690677	36698	2.64
2	2998(210-400)	12.296	25492784	1095862	97.36



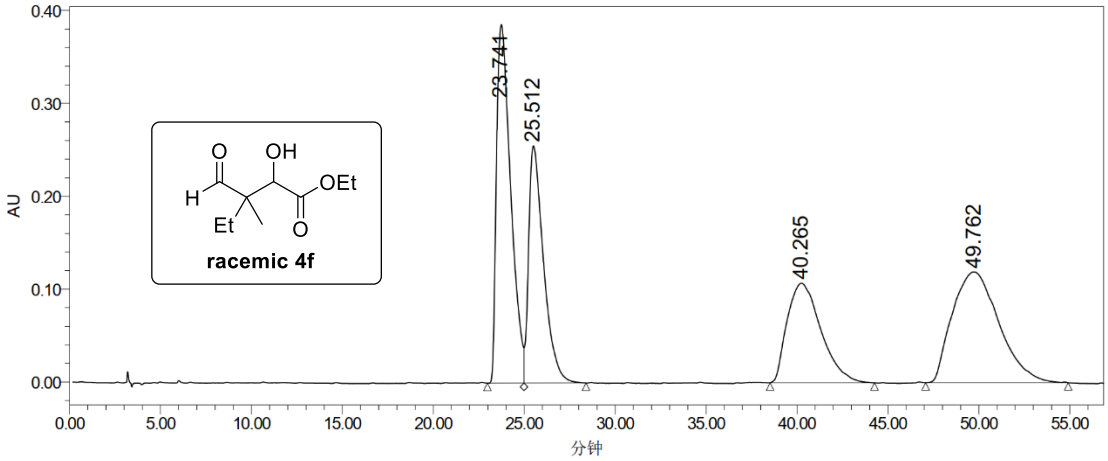
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.703	17279988	1844039	49.82
2	2998(210-400)	11.229	17404837	1232545	50.18



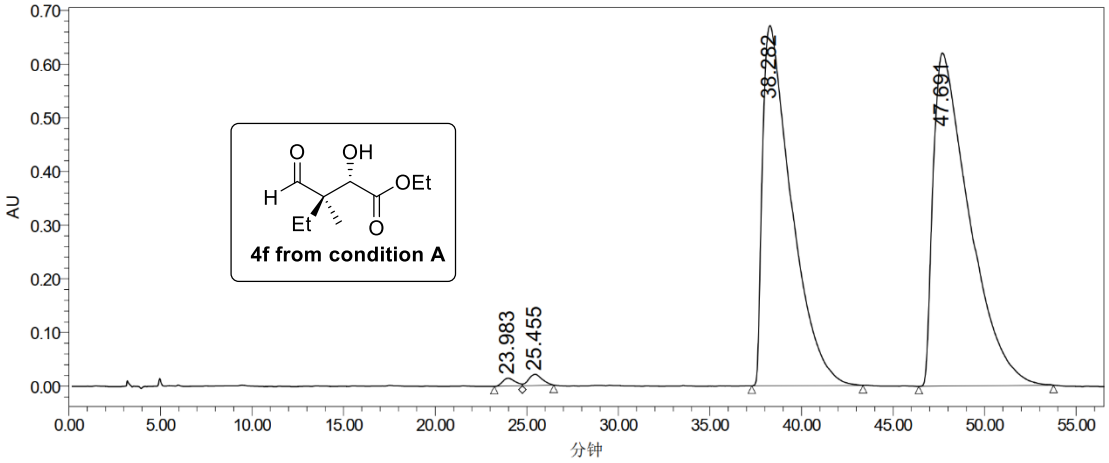
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.764	186220	22909	0.80
2	2998(210-400)	11.016	23112516	1611289	99.20



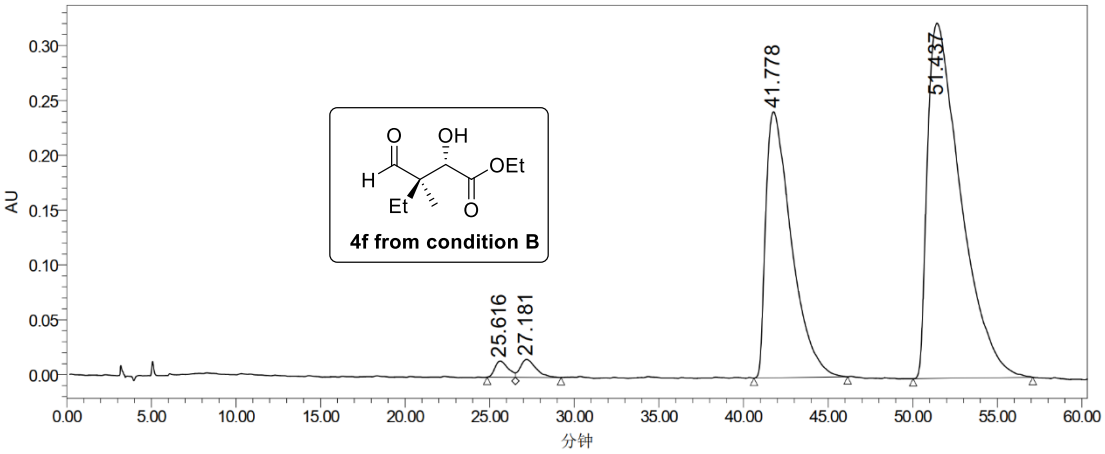
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.678	336360	40418	1.99
2	2998(210-400)	11.166	16596673	1167833	98.01



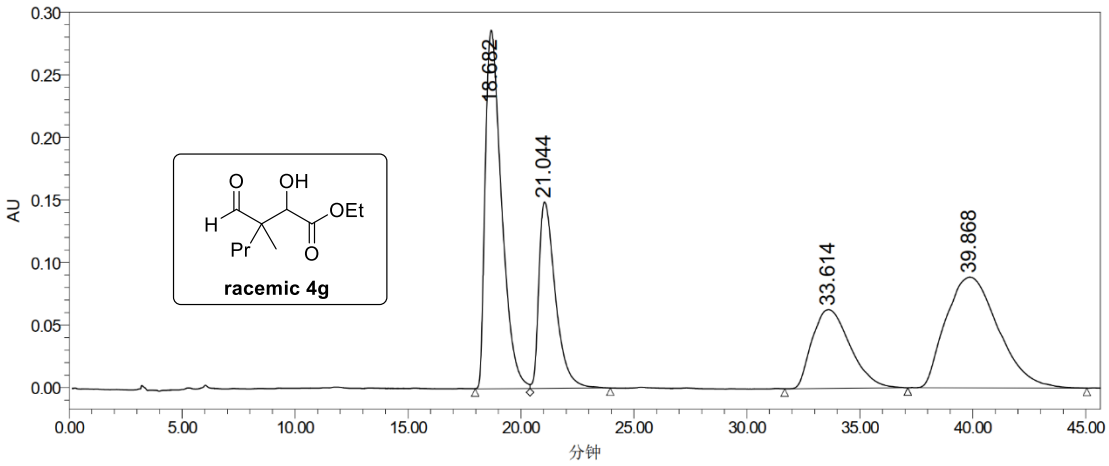
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	23.741	21039577	385932	29.73
2	2998(210-400)	25.512	14605301	255244	20.64
3	2998(210-400)	40.265	13860409	107075	19.59
4	2998(210-400)	49.762	21265062	119175	30.05



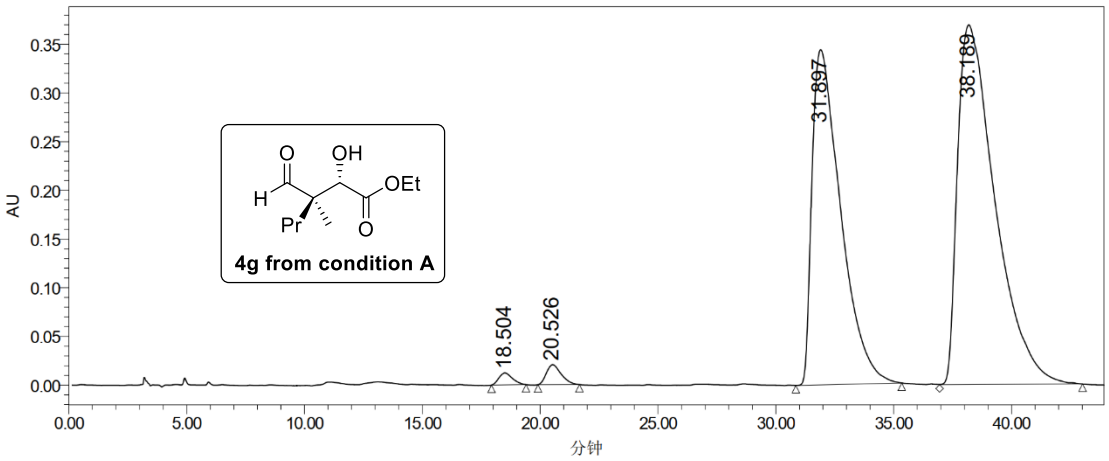
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	23.983	694998	14704	0.43
2	2998(210-400)	25.455	1057398	20748	0.66
3	2998(210-400)	38.282	73446085	670620	45.85
4	2998(210-400)	47.691	84992246	620560	53.06



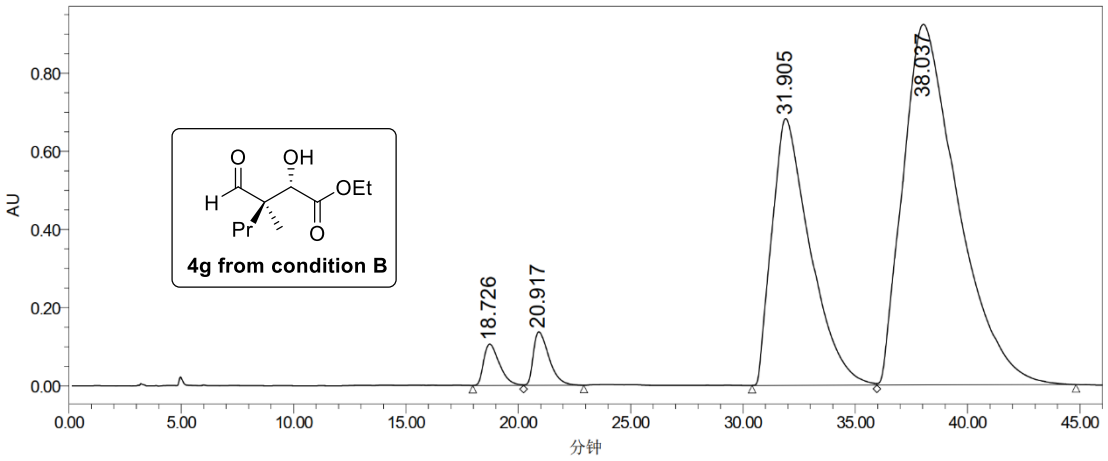
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	25.616	792173	14696	1.10
2	2998(210-400)	27.181	1043027	16341	1.45
3	2998(210-400)	41.778	25562045	242542	35.61
4	2998(210-400)	51.437	44384105	323870	61.83



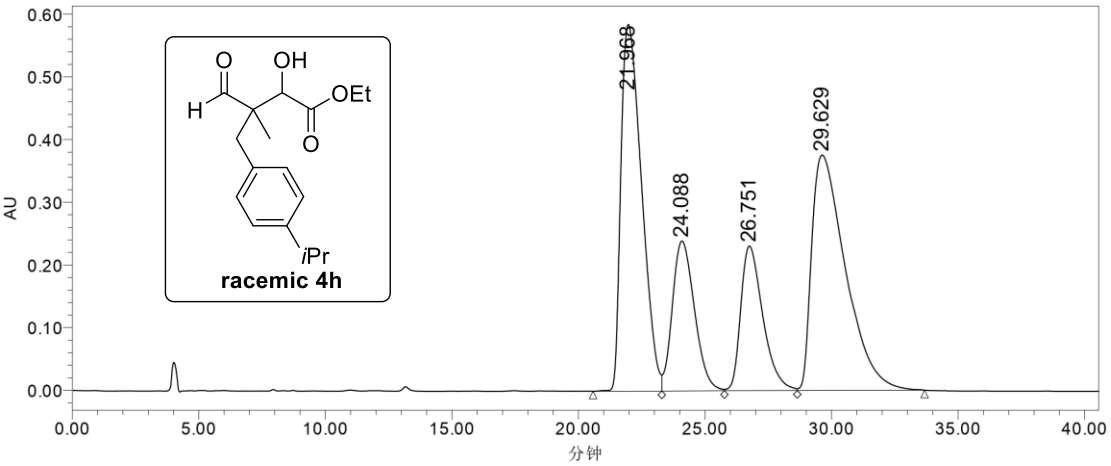
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	18.682	14391101	286396	32.99
2	2998(210-400)	21.044	7564311	148905	17.34
3	2998(210-400)	33.614	7352659	62919	16.86
4	2998(210-400)	39.868	14308145	88475	32.80



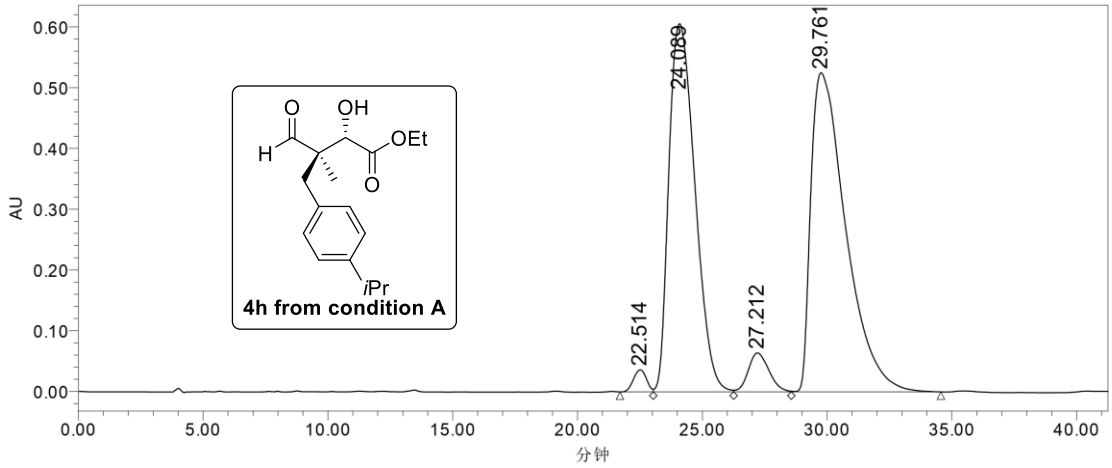
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	18.504	487249	12245	0.68
2	2998(210-400)	20.526	892599	20520	1.25
3	2998(210-400)	31.897	29123936	344316	40.83
4	2998(210-400)	38.189	40831428	369464	57.24



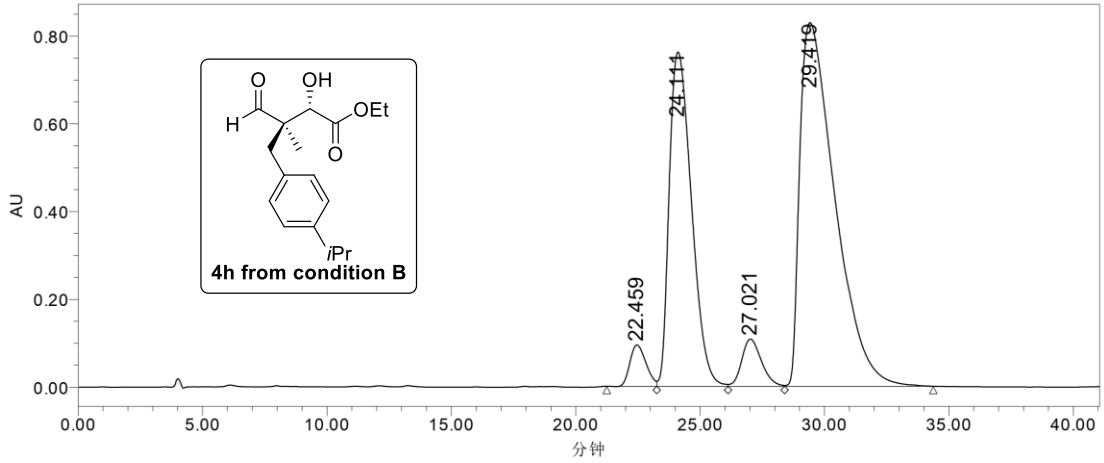
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	18.726	5130620	105595	2.06
2	2998(210-400)	20.917	6612238	136622	2.66
3	2998(210-400)	31.905	80743781	682485	32.49
4	2998(210-400)	38.037	156029279	922879	62.78



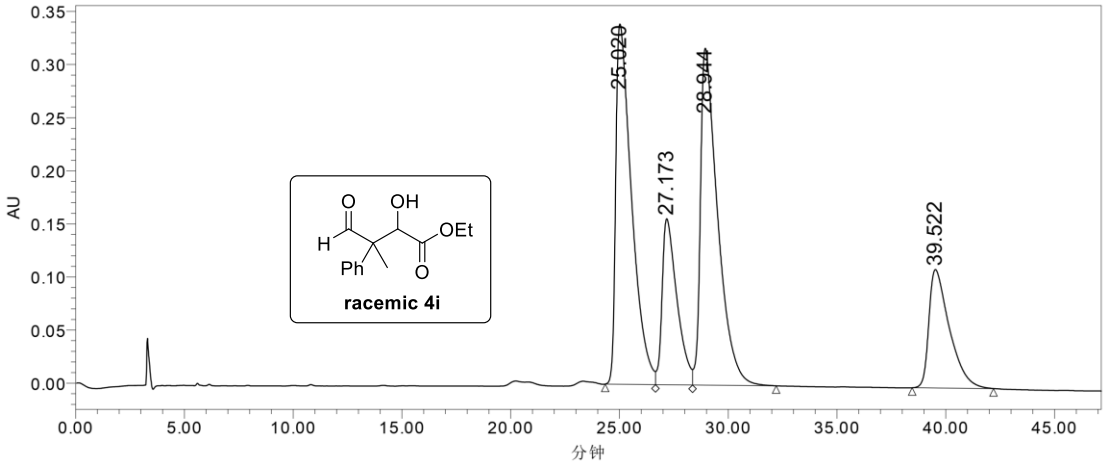
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	21.968	33224540	584774	34.38
2	2998(210-400)	24.088	14550854	239267	15.06
3	2998(210-400)	26.751	14022086	230860	14.51
4	2998(210-400)	29.629	34828068	375583	36.04



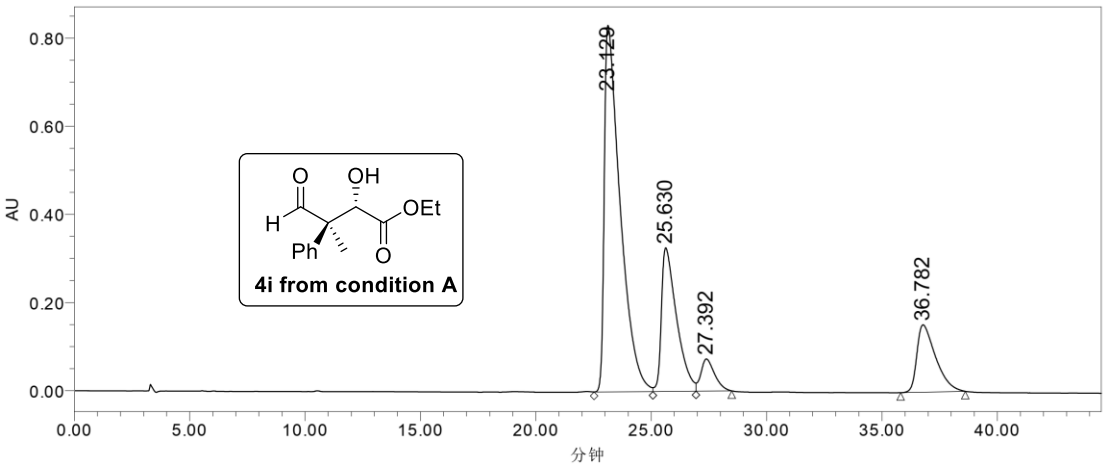
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	22.514	1313477	36343	1.31
2	2998(210-400)	24.089	44042507	605692	43.97
3	2998(210-400)	27.212	3696776	64181	3.69
4	2998(210-400)	29.761	51120550	525024	51.03



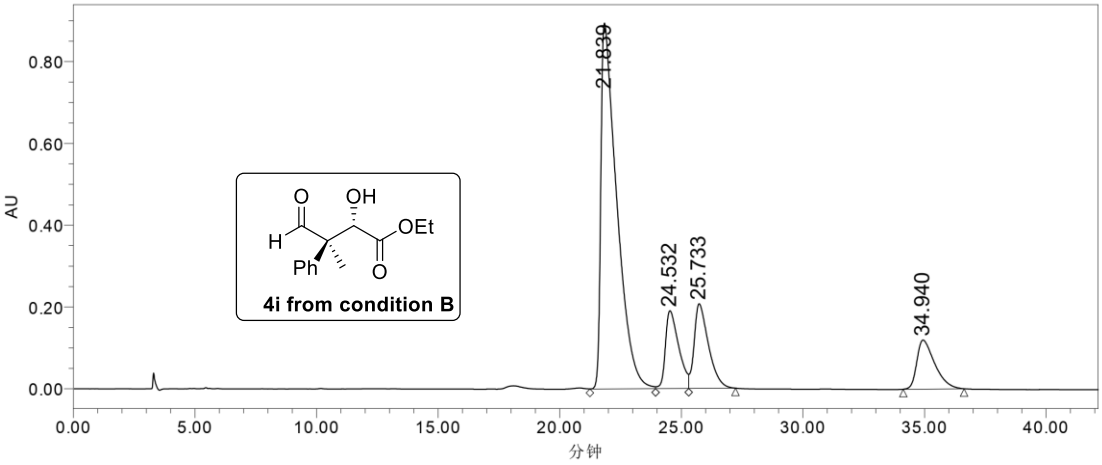
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	22.459	4225628	93825	3.05
2	2998(210-400)	24.111	47301550	761758	34.15
3	2998(210-400)	27.021	5894959	107458	4.26
4	2998(210-400)	29.419	81106683	828792	58.55



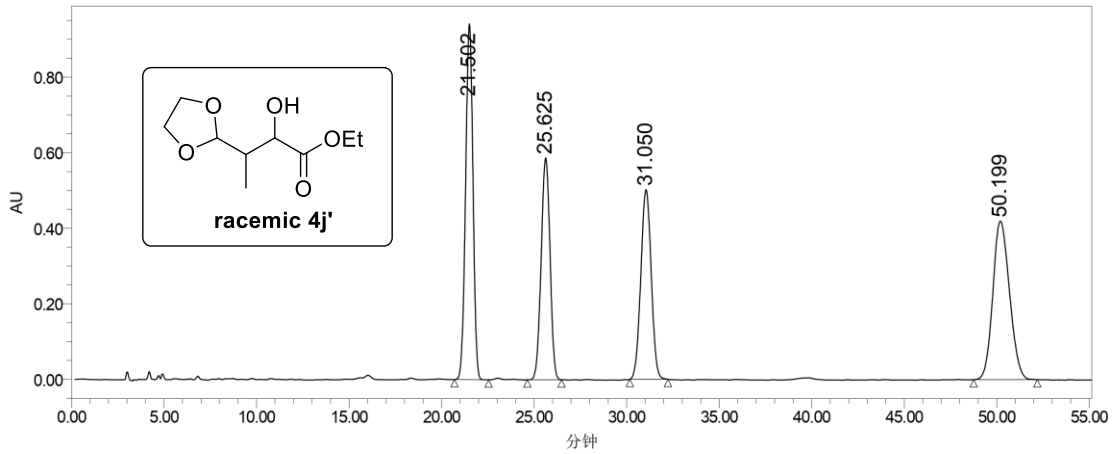
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	25.020	17204340	339167	34.82
2	2998(210-400)	27.173	7420861	156220	15.02
3	2998(210-400)	28.944	17472453	317216	35.37
4	2998(210-400)	39.522	7305092	111509	14.79



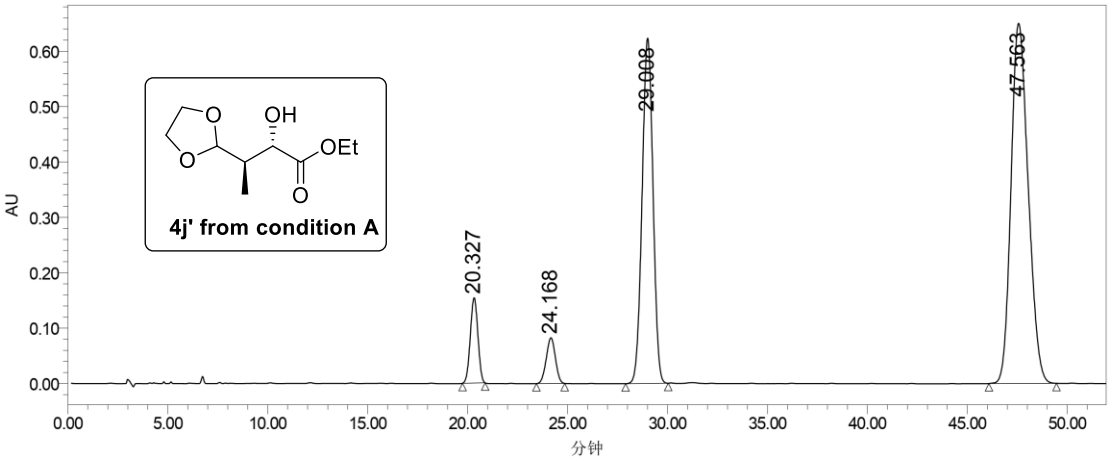
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	23.129	39920451	831709	60.04
2	2998(210-400)	25.630	14890955	325989	22.39
3	2998(210-400)	27.392	3029746	72881	4.56
4	2998(210-400)	36.782	8653378	152936	13.01



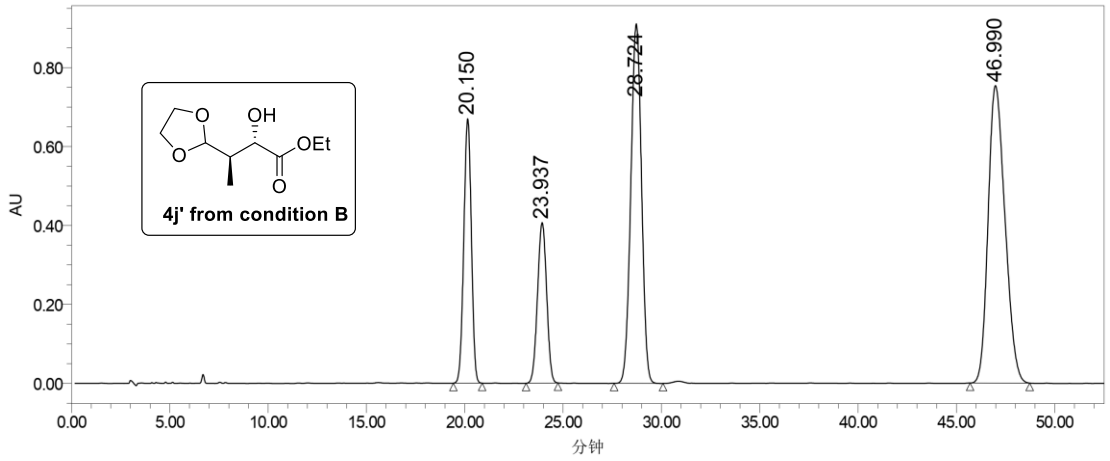
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	21.839	40745368	894517	65.11
2	2998(210-400)	24.532	7217623	190177	11.53
3	2998(210-400)	25.733	8484602	206779	13.56
4	2998(210-400)	34.940	6133123	119735	9.80



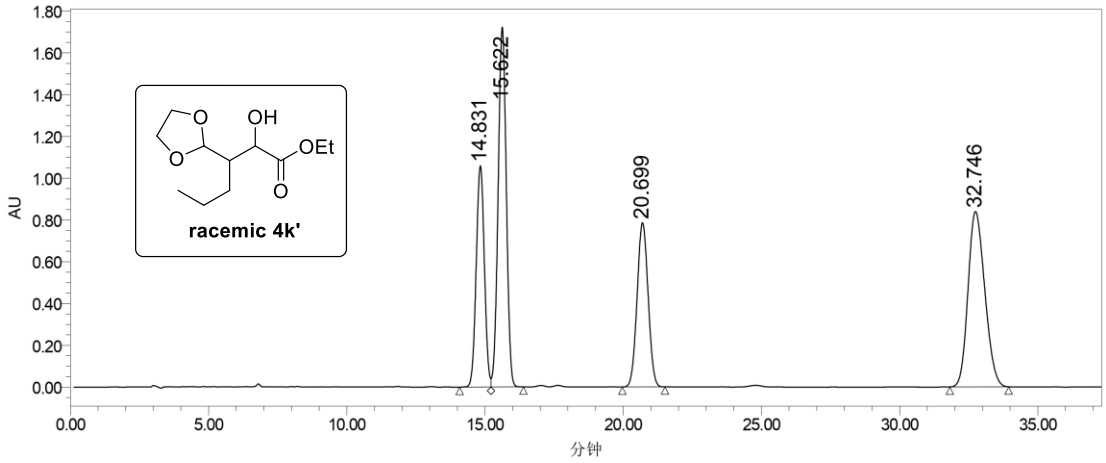
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	21.502	25924799	941754	28.37
2	2998(210-400)	25.625	19544018	587396	21.39
3	2998(210-400)	31.050	19590400	502081	21.44
4	2998(210-400)	50.199	26313424	419909	28.80



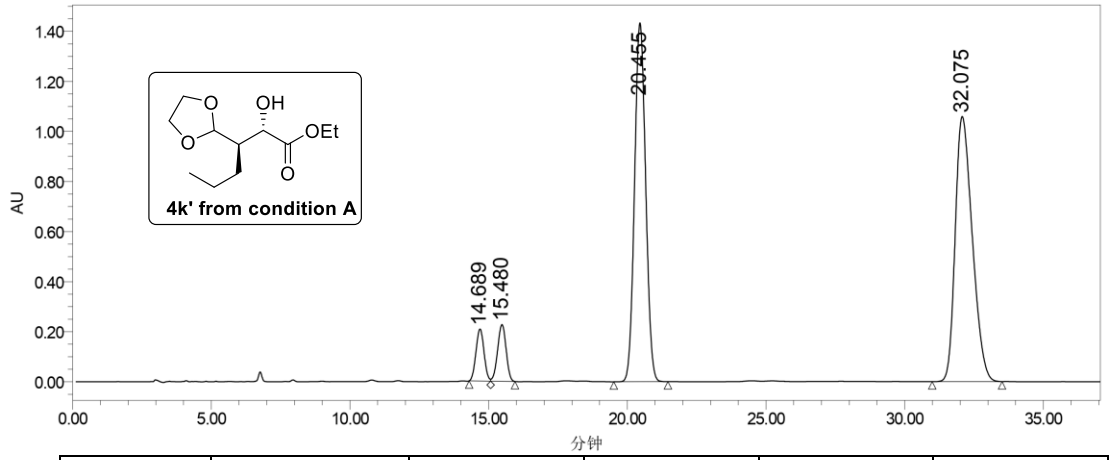
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	20.327	4053606	153843	5.87
2	2998(210-400)	24.168	2619913	82633	3.80
3	2998(210-400)	29.008	23575410	623312	34.16
4	2998(210-400)	47.563	38770796	650210	56.17



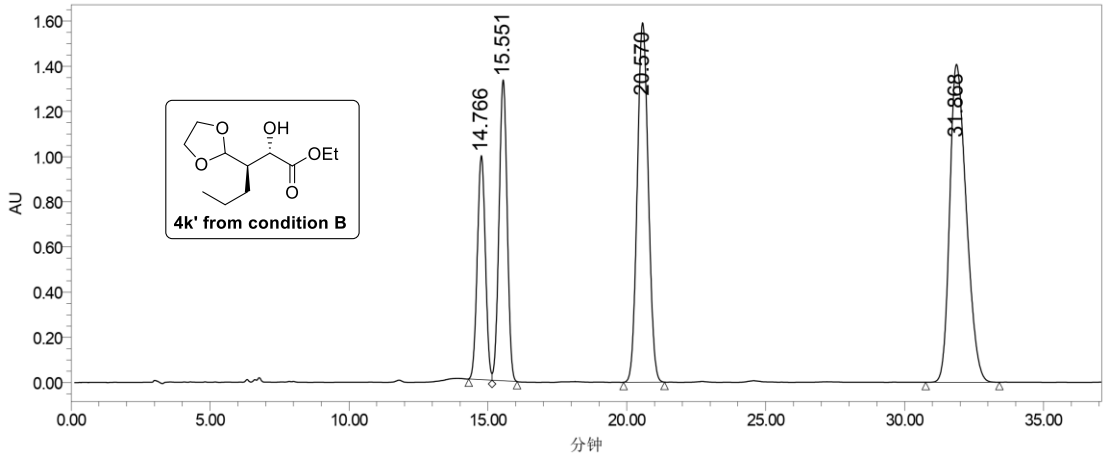
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	20.150	17890254	670204	16.21
2	2998(210-400)	23.937	13123876	406472	11.89
3	2998(210-400)	28.724	34356652	910781	31.14
4	2998(210-400)	46.990	44965735	753456	40.75



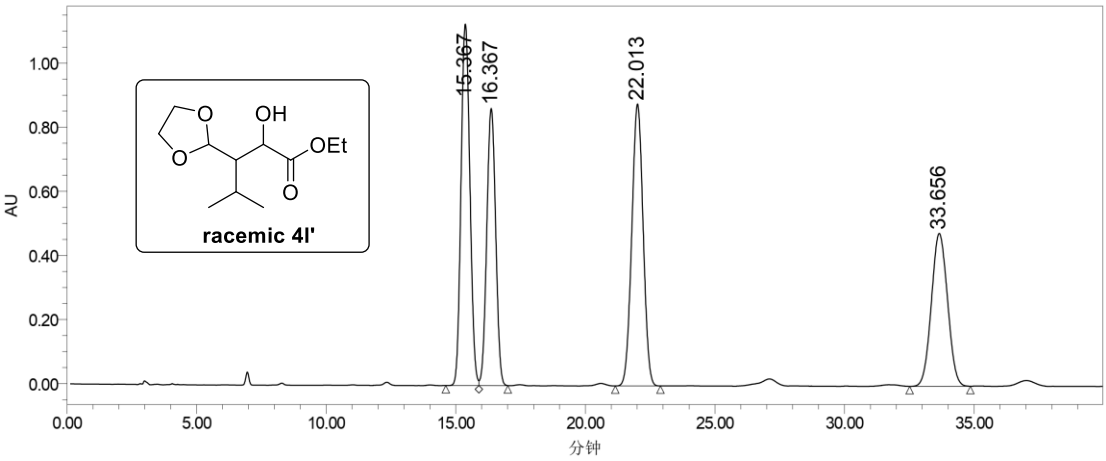
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	14.831	21730108	1059296	19.02
2	2998(210-400)	15.622	35338218	1722455	30.93
3	2998(210-400)	20.699	21729481	785793	19.02
4	2998(210-400)	32.746	35443882	839220	31.03



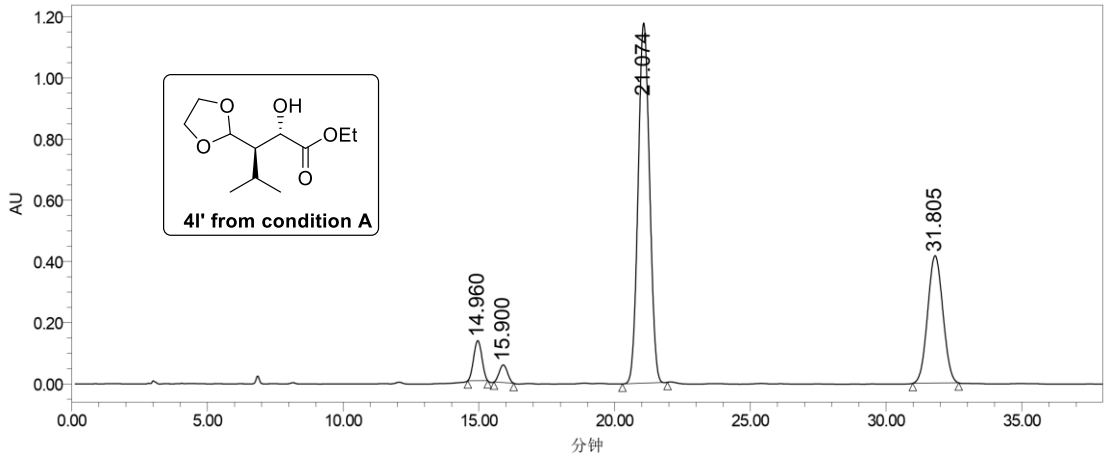
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	14.689	4178254	207507	4.44
2	2998(210-400)	15.480	4822196	226640	5.12
3	2998(210-400)	20.455	39915489	1433556	42.38
4	2998(210-400)	32.075	45268788	1059072	48.06



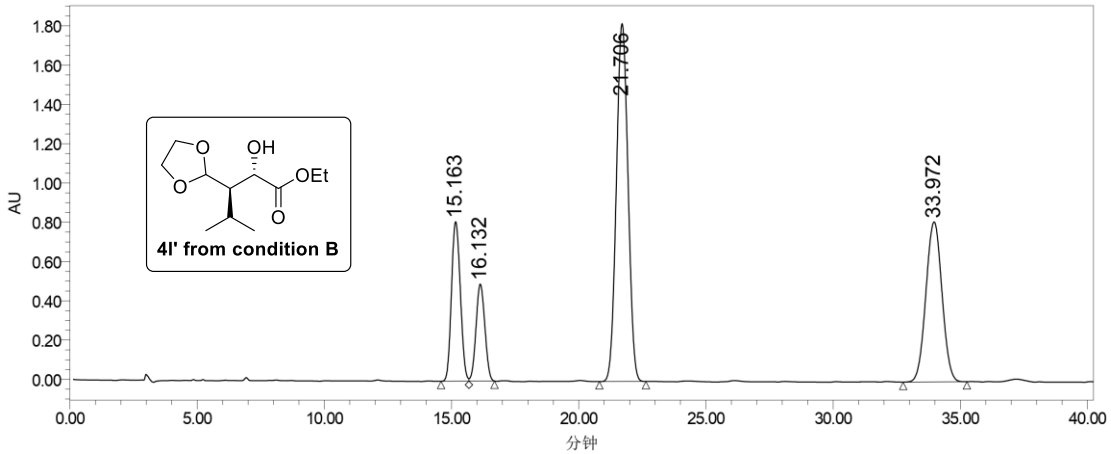
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	14.766	20019461	991364	13.38
2	2998(210-400)	15.551	27054096	1332256	18.09
3	2998(210-400)	20.570	43975632	1591520	29.40
4	2998(210-400)	31.868	58534698	1407539	39.13



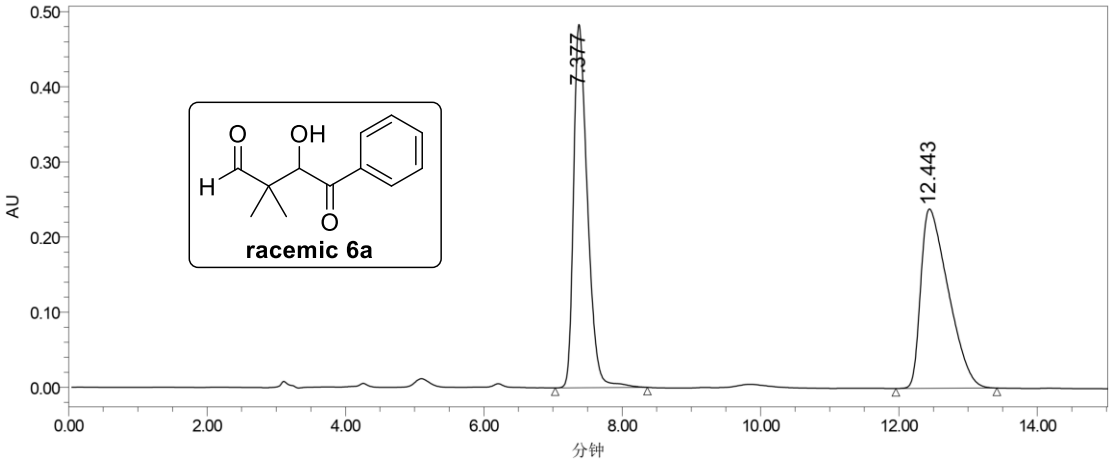
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	15.367	26797396	1127423	28.24
2	2998(210-400)	16.367	20706260	864761	21.82
3	2998(210-400)	22.013	26805555	879696	28.25
4	2998(210-400)	33.656	20589657	477125	21.70



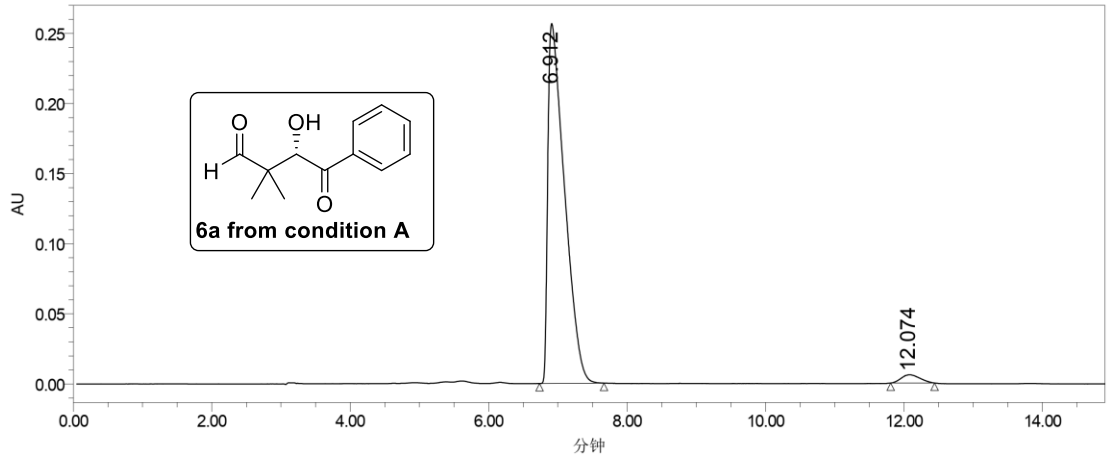
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	14.960	2741586	130870	5.02
2	2998(210-400)	15.900	1209642	57560	2.21
3	2998(210-400)	21.074	34449875	1176879	63.07
4	2998(210-400)	31.805	16221999	416458	29.70



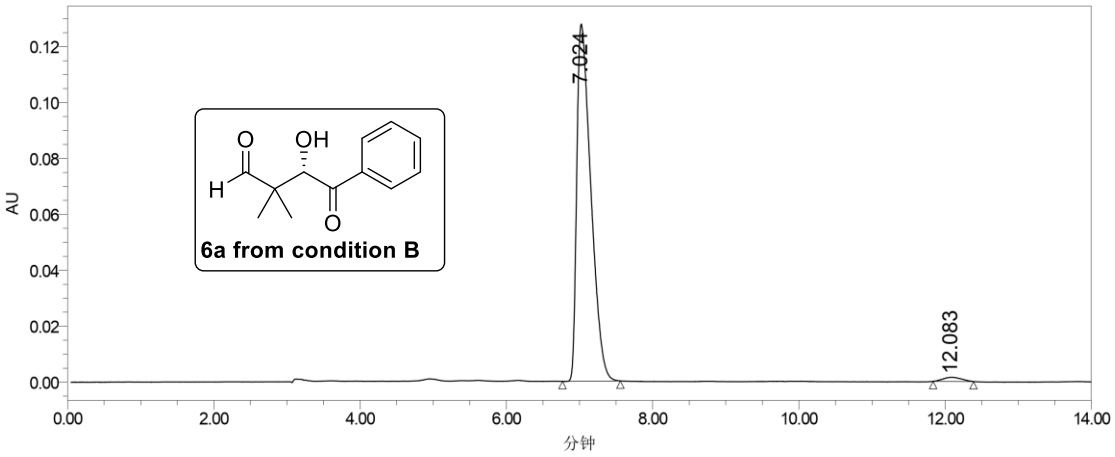
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	15.163	19715705	812361	15.71
2	2998(210-400)	16.132	11945975	494466	9.52
3	2998(210-400)	21.706	58071162	1822355	46.28
4	2998(210-400)	33.972	35732145	814452	28.48



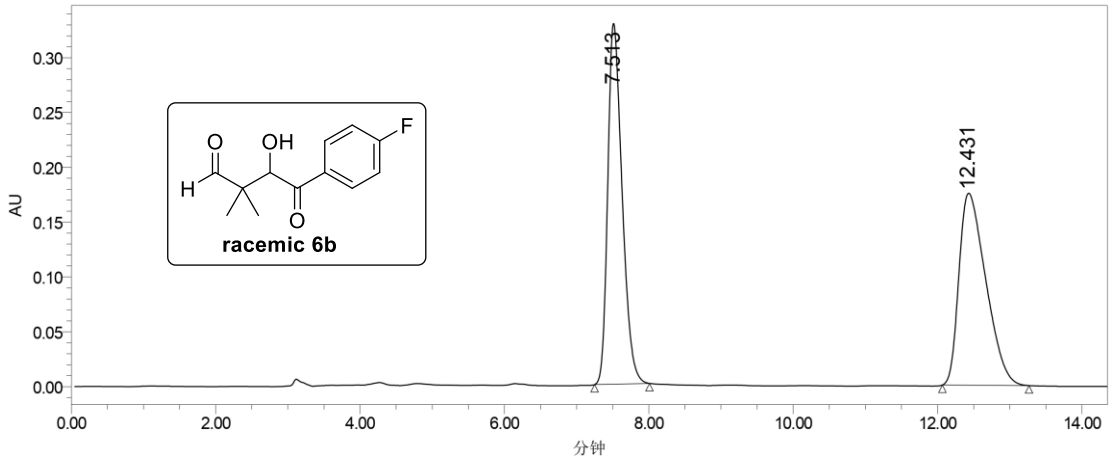
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.377	6729400	483615	50.55
2	2998(210-400)	12.443	6583745	238699	49.45



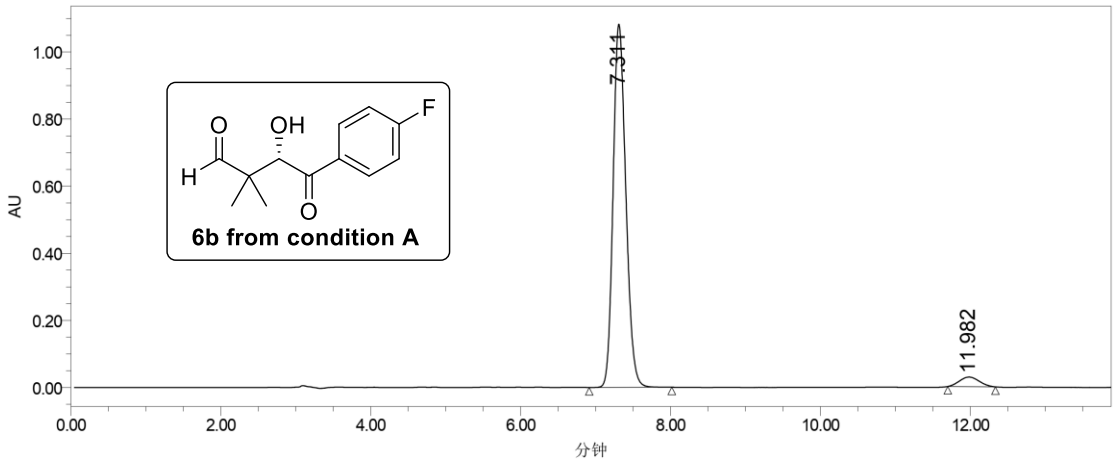
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.912	4298765	256669	97.51
2	2998(210-400)	12.074	109784	5855	2.49



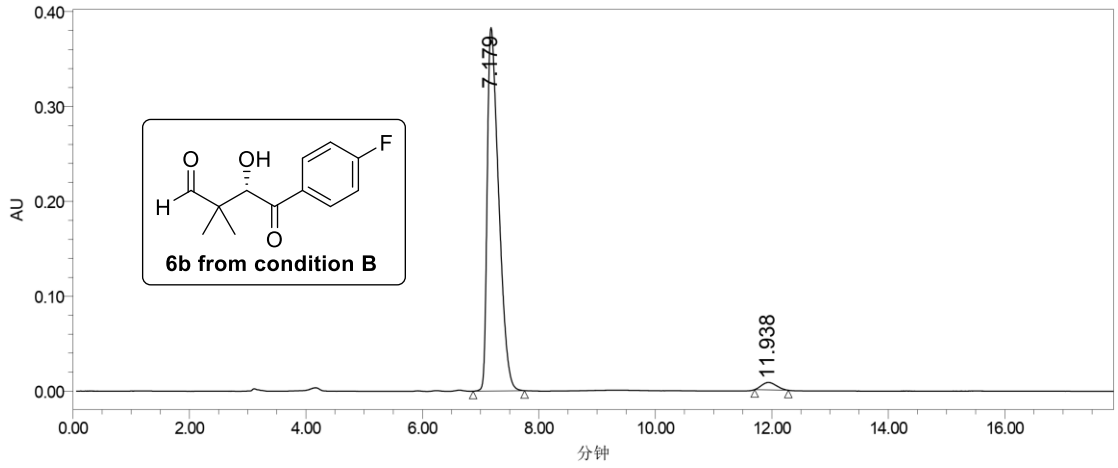
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.024	1708466	127772	98.55
2	2998(210-400)	12.083	25155	1454	1.45



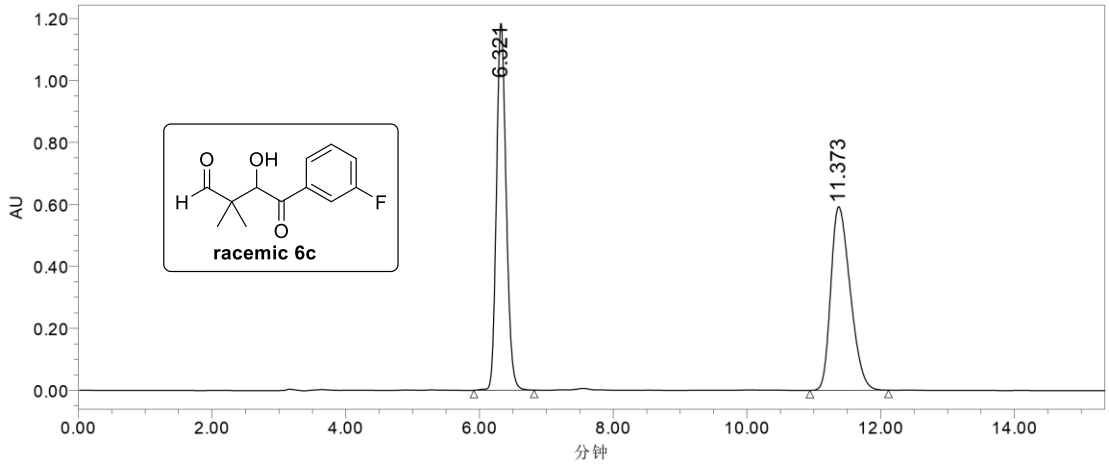
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.513	4558755	329238	50.21
2	2998(210-400)	12.431	4520822	175082	49.79



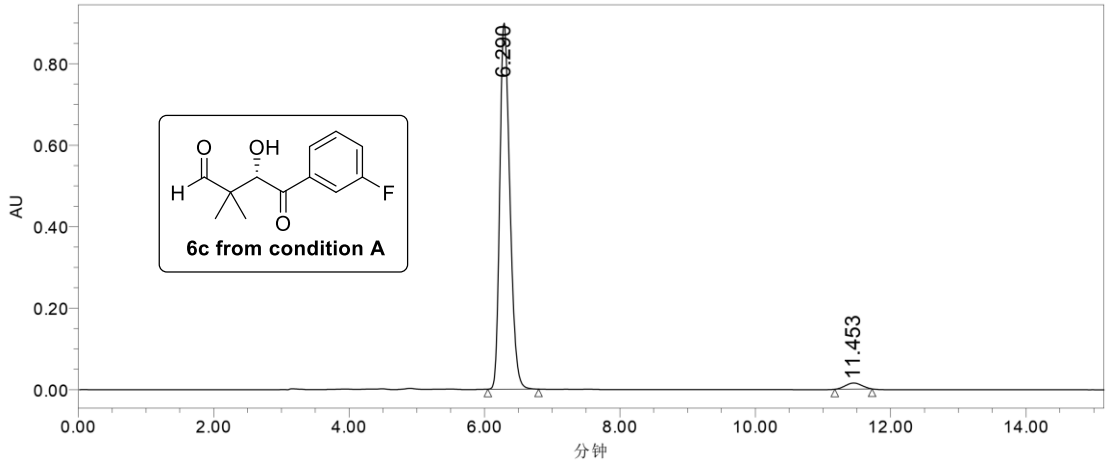
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.311	12732649	1082583	96.06
2	2998(210-400)	11.982	521711	28581	3.94



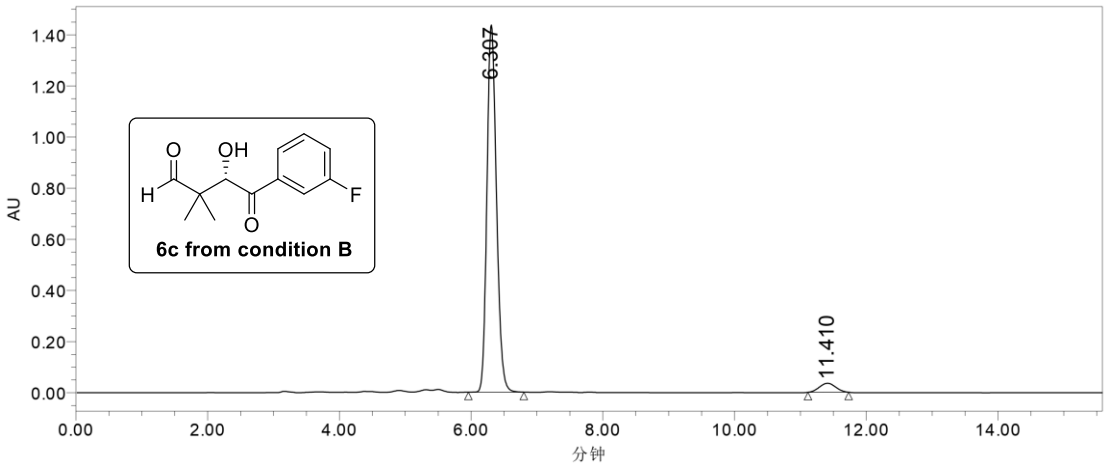
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.179	5238083	383058	97.46
2	2998(210-400)	11.938	136585	7788	2.54



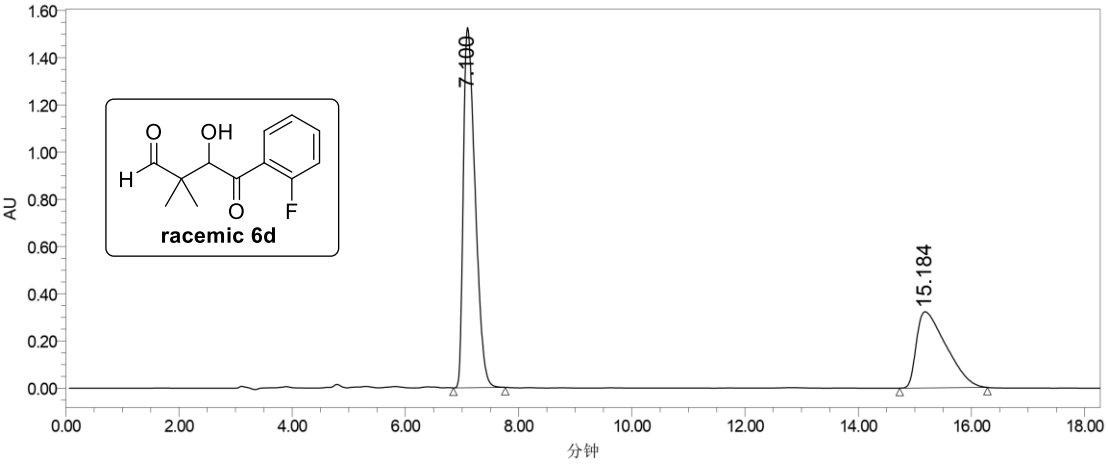
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.321	11936596	1185301	49.99
2	2998(210-400)	11.373	11943519	592403	50.01



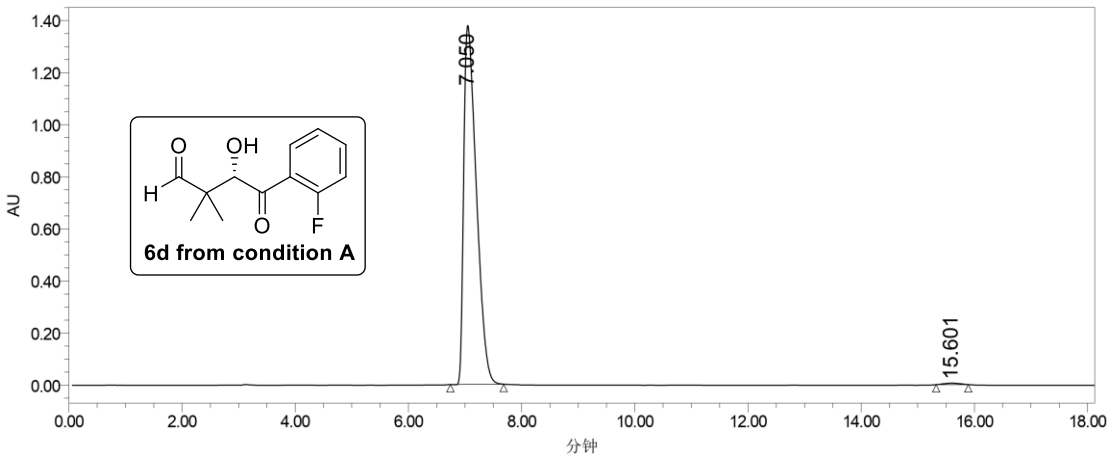
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.290	9285370	898458	97.42
2	2998(210-400)	11.453	245468	14793	2.58



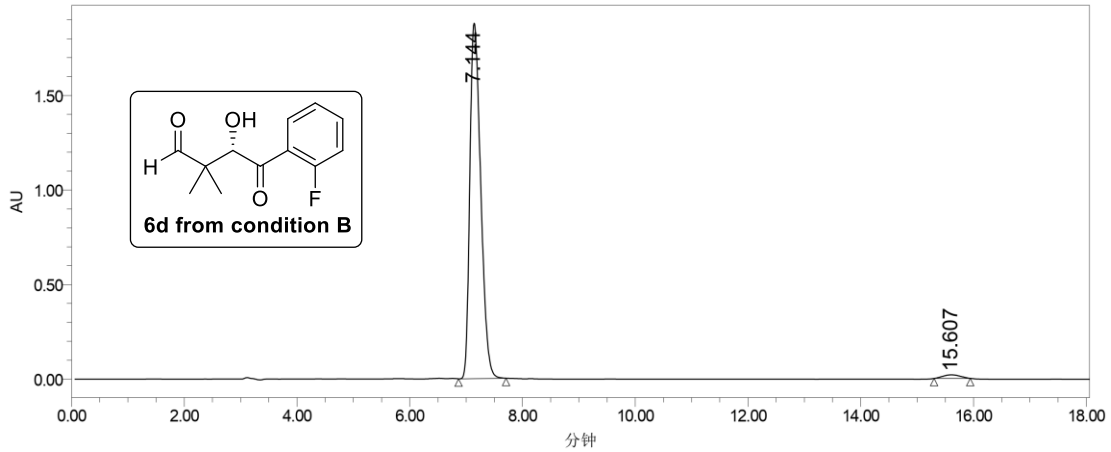
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.307	14496781	1436483	95.97
2	2998(210-400)	11.410	608359	35381	4.03



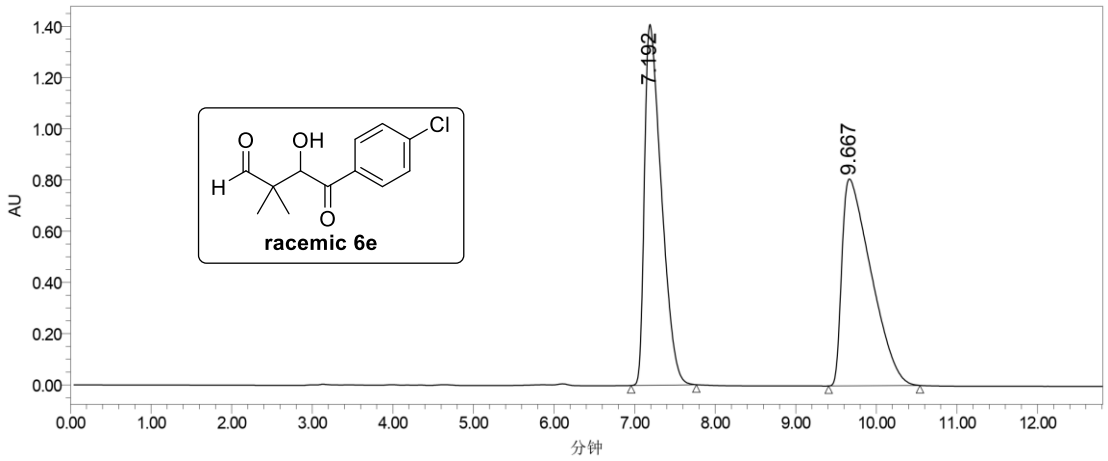
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.100	22106734	1525891	65.29
2	2998(210-400)	15.184	11751864	322695	34.71



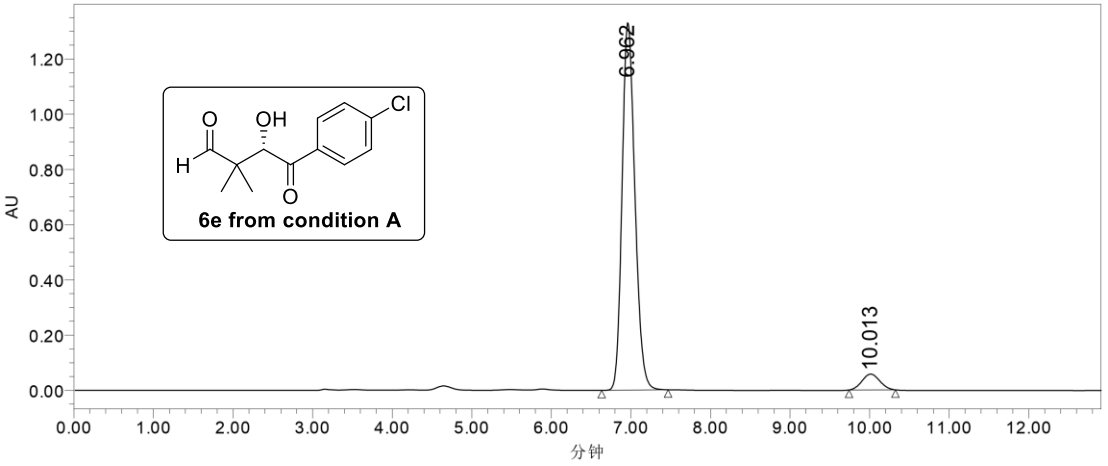
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.050	21324740	1378024	99.49
2	2998(210-400)	15.601	110285	5589	0.51



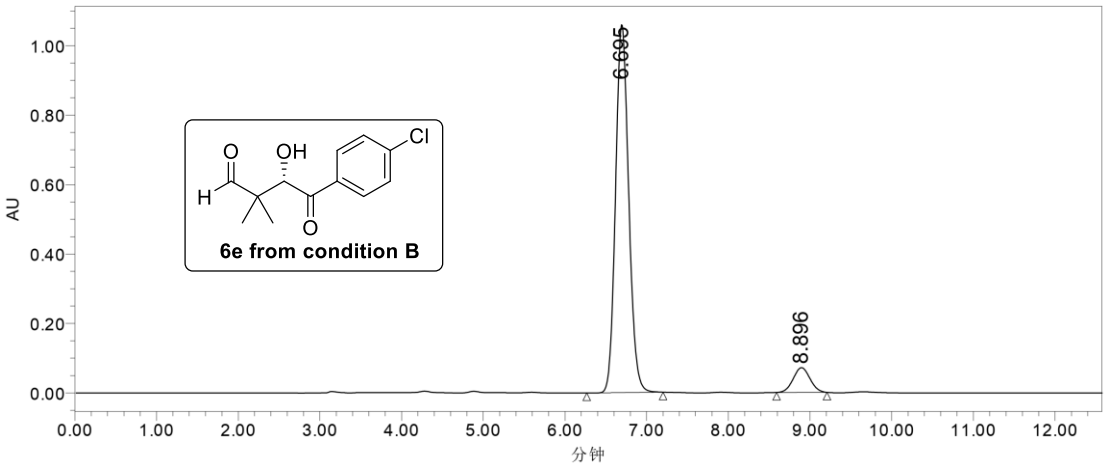
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.144	25126649	1880042	98.42
2	2998(210-400)	15.607	403273	18898	1.58



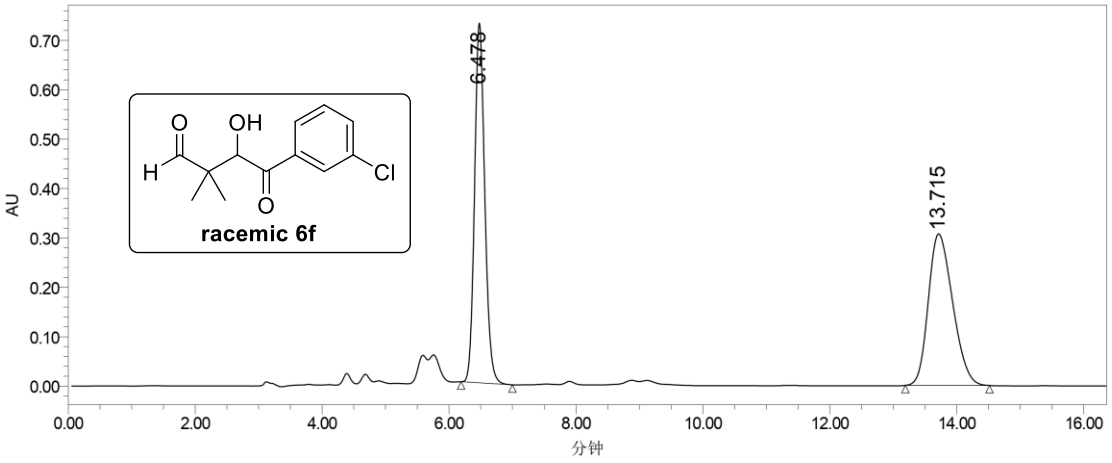
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.192	20502465	1408802	50.24
2	2998(210-400)	9.667	20308528	807545	49.76



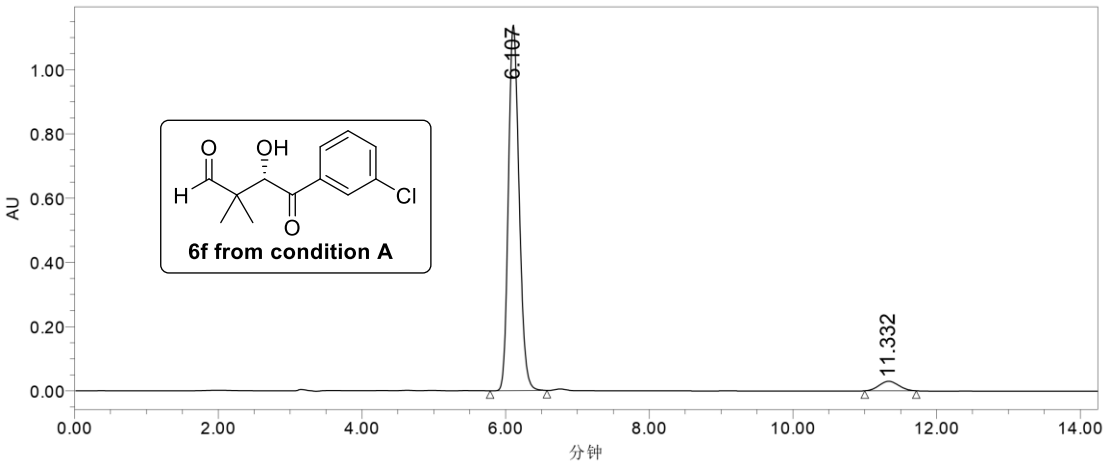
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.962	14917606	1331534	94.38
2	2998(210-400)	10.013	887579	57214	5.62



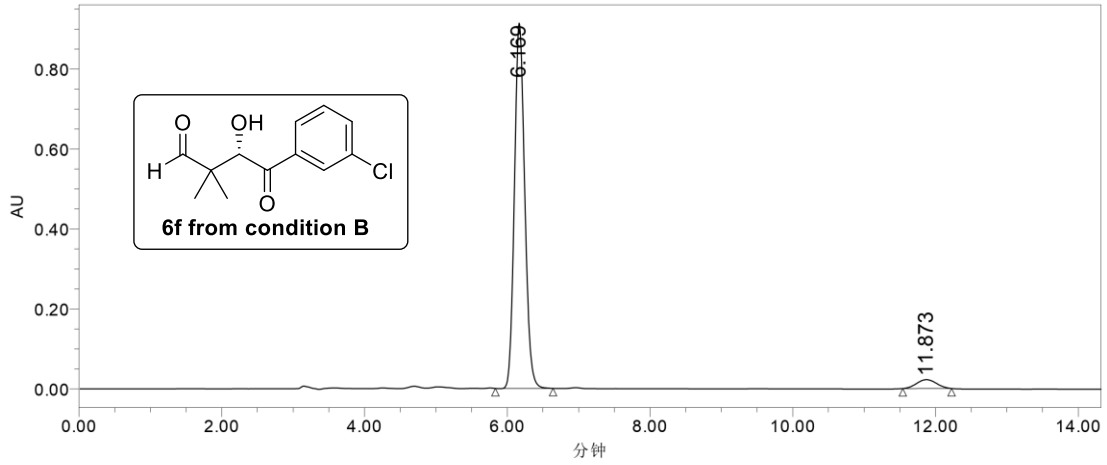
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.695	11838208	1059947	92.08
2	2998(210-400)	8.896	1018753	71167	7.92



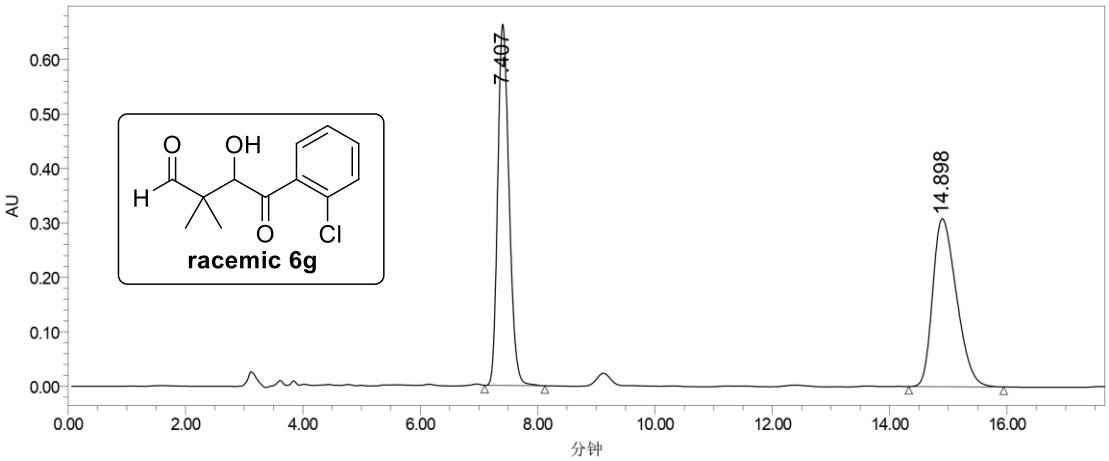
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.478	8152183	728880	49.96
2	2998(210-400)	13.715	8165532	306766	50.04



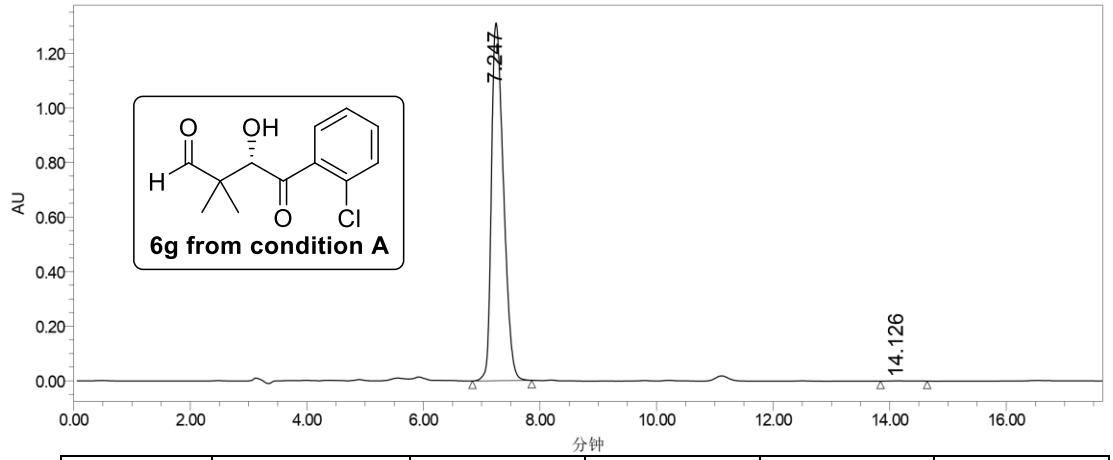
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.107	11747641	1137942	95.45
2	2998(210-400)	11.332	559857	29671	4.55



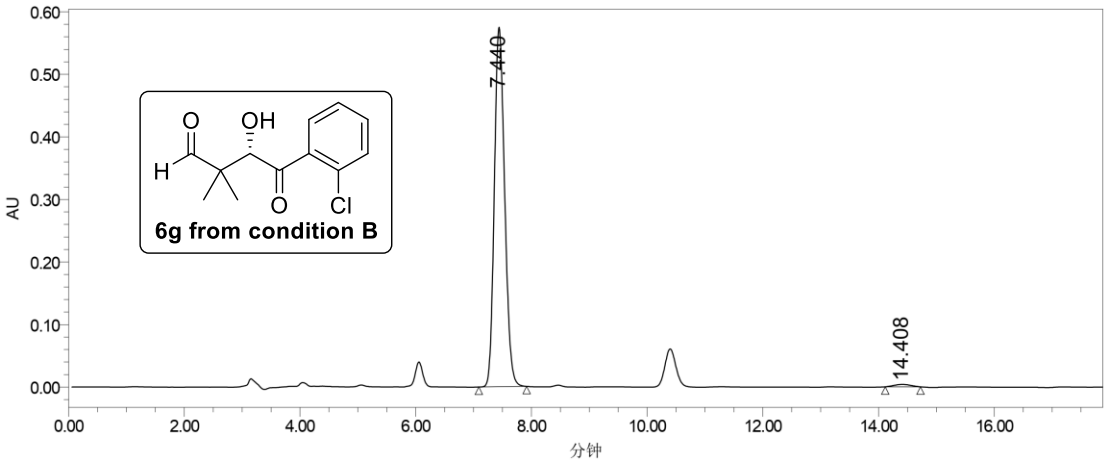
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.169	9194652	914220	95.54
2	2998(210-400)	11.873	429020	22278	4.46



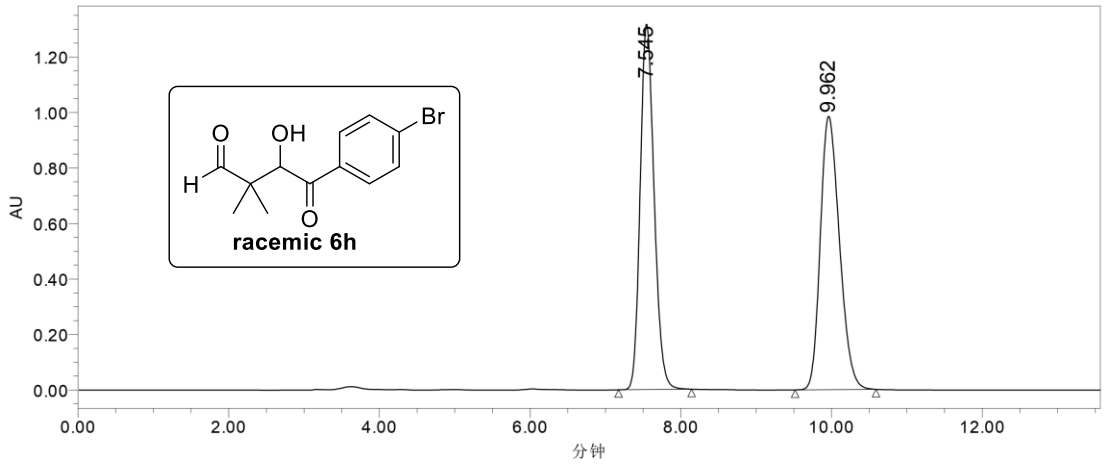
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.407	8906802	663238	50.61
2	2998(210-400)	14.898	8692503	308348	49.39



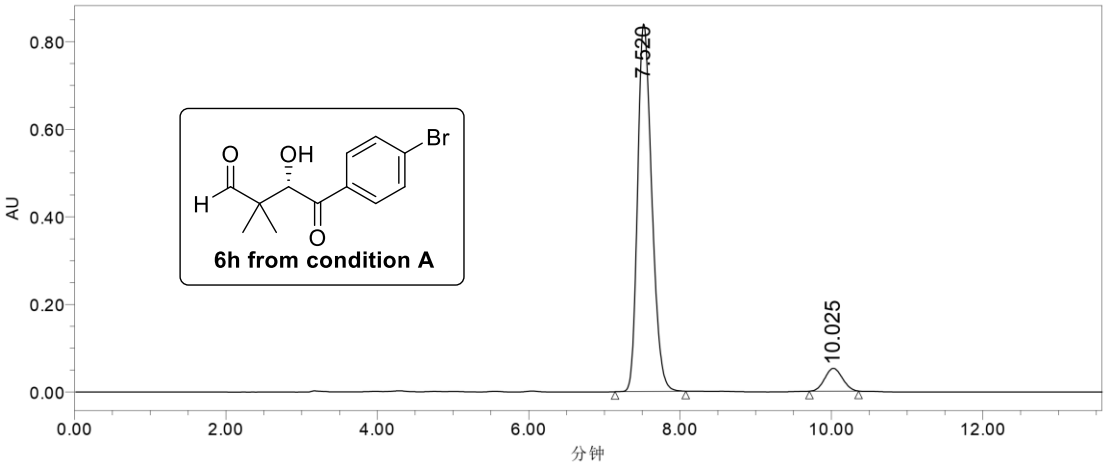
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.247	18838552	1311057	99.87
2	2998(210-400)	14.126	25346	933	0.13



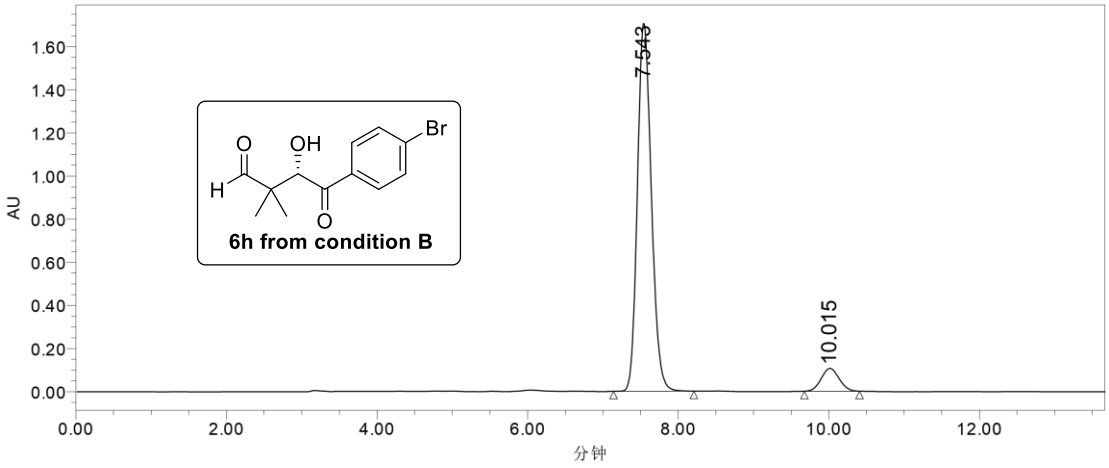
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.440	7128348	574787	98.94
2	2998(210-400)	14.408	75608	3823	1.06



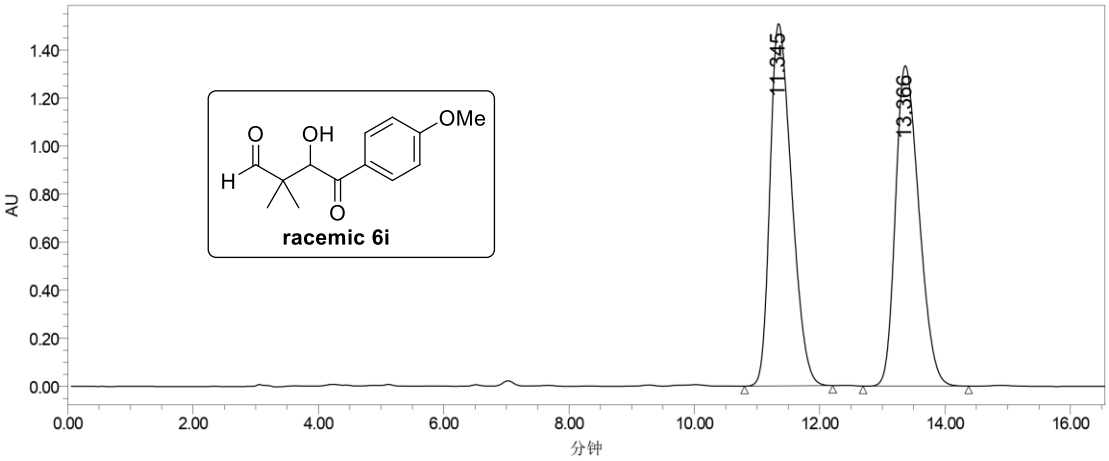
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.545	16885083	1316836	49.26
2	2998(210-400)	9.962	17389263	984500	50.74



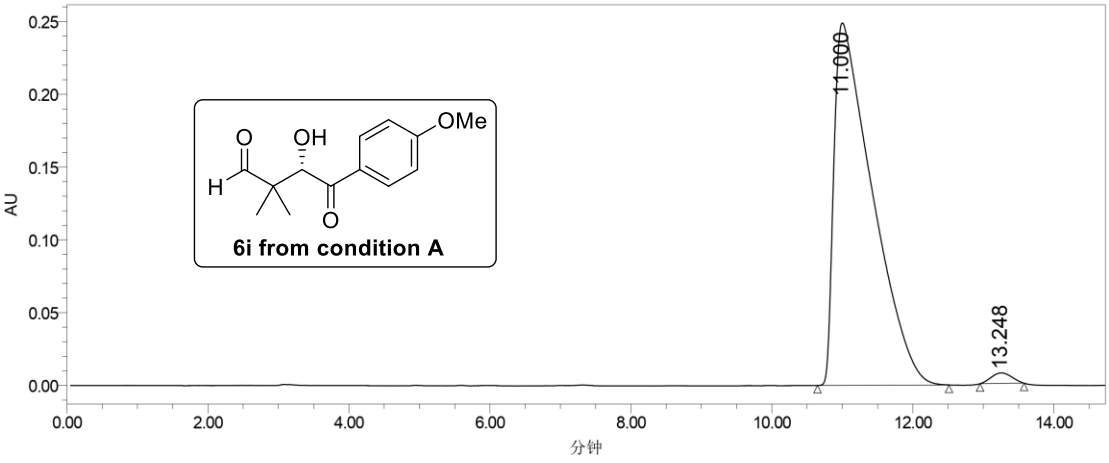
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.520	10947360	839995	92.86
2	2998(210-400)	10.025	842060	52037	7.14



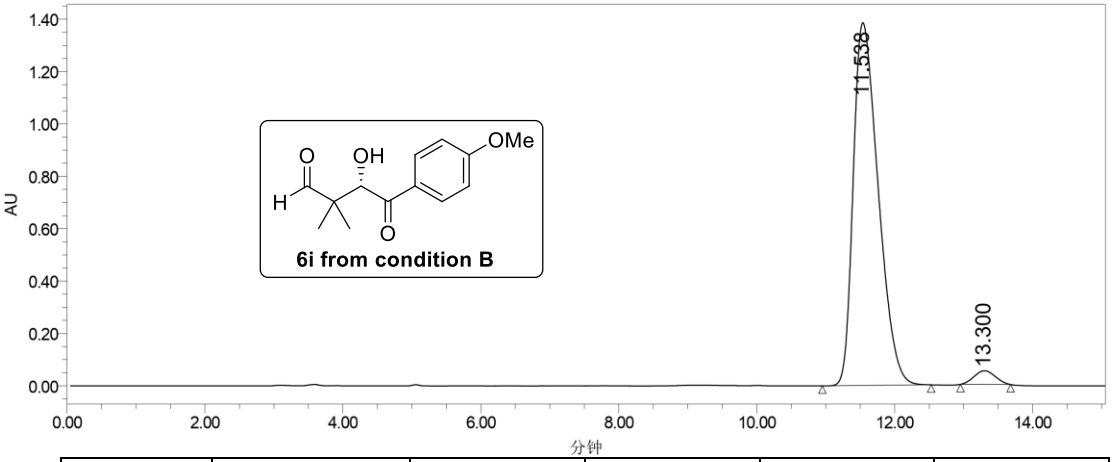
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.543	22020780	1706480	92.66
2	2998(210-400)	10.015	1744636	105806	7.34



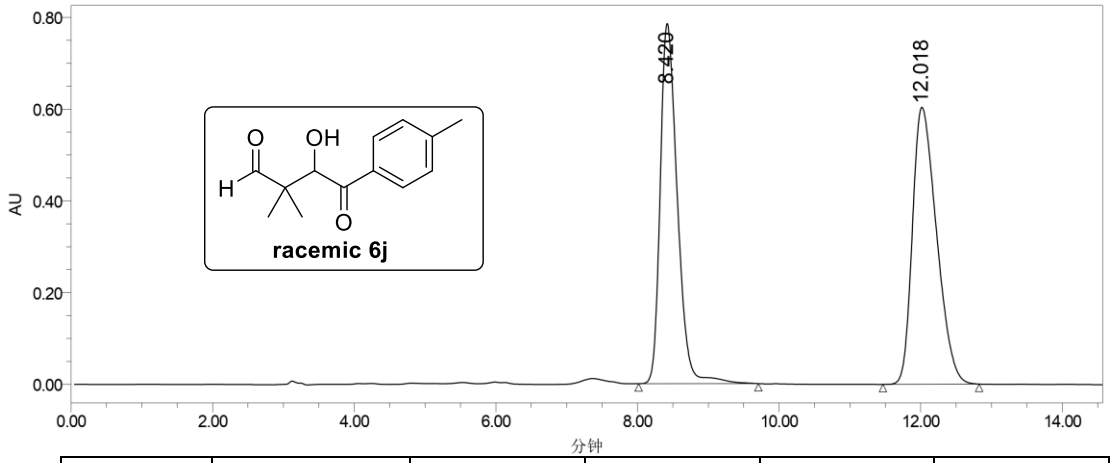
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	11.345	36246807	1507814	50.97
2	2998(210-400)	13.366	34867101	1333355	49.03



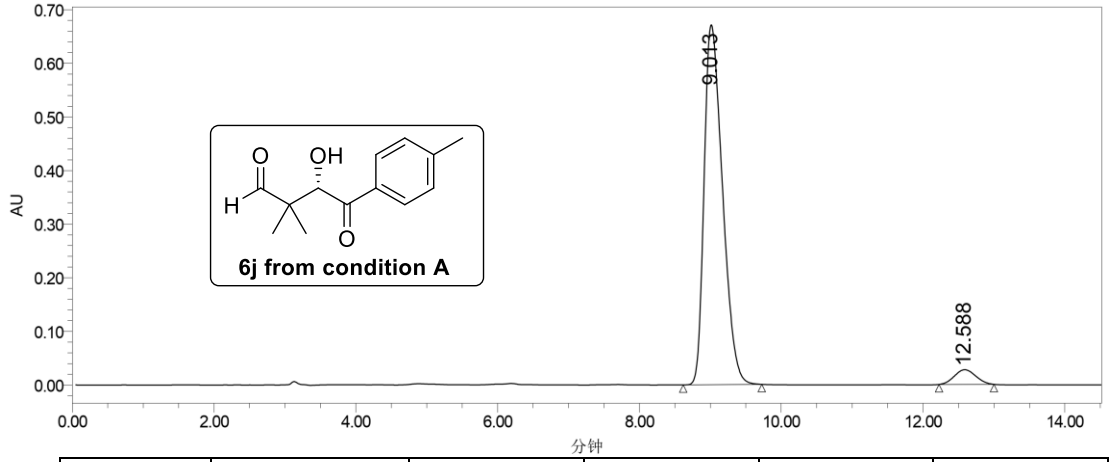
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	11.000	9400826	248922	98.47
2	2998(210-400)	13.248	146031	7229	1.53



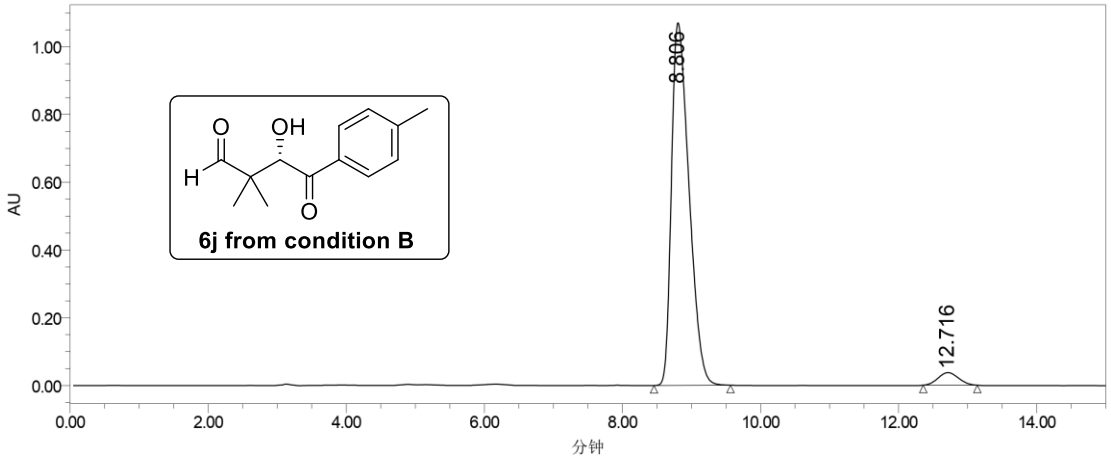
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	11.538	34818583	1385233	96.90
2	2998(210-400)	13.300	1112712	52216	3.10



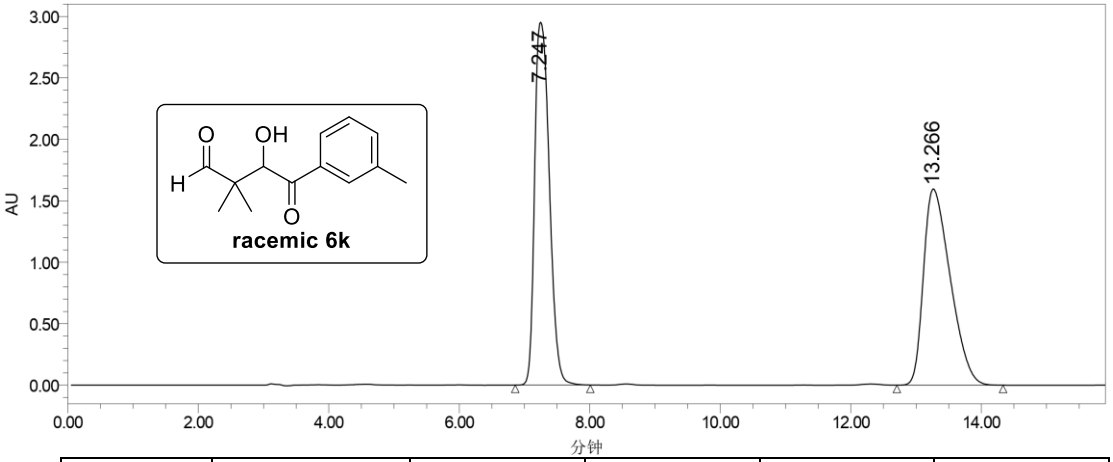
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.420	13192753	785328	47.98
2	2998(210-400)	12.018	14303390	603838	52.02



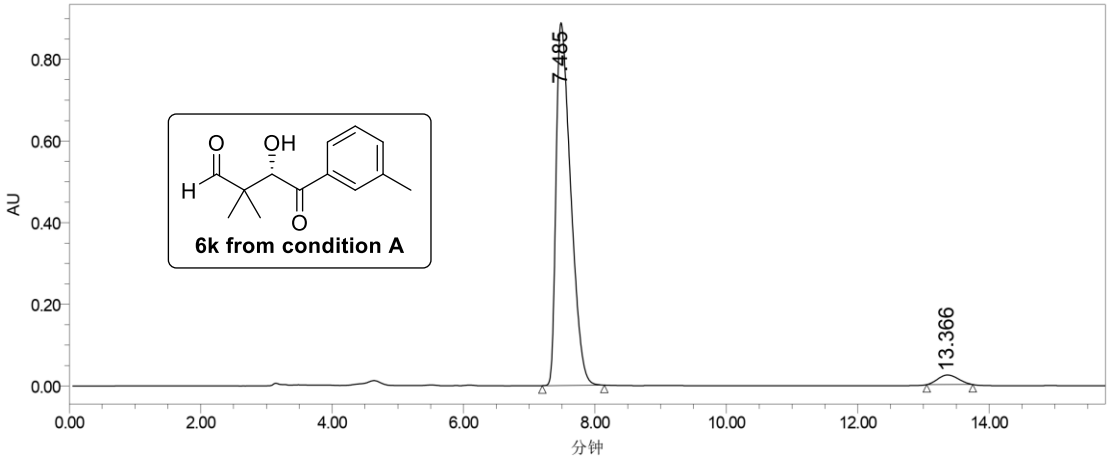
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	9.013	12088061	671414	95.48
2	2998(210-400)	12.588	571784	27358	4.52



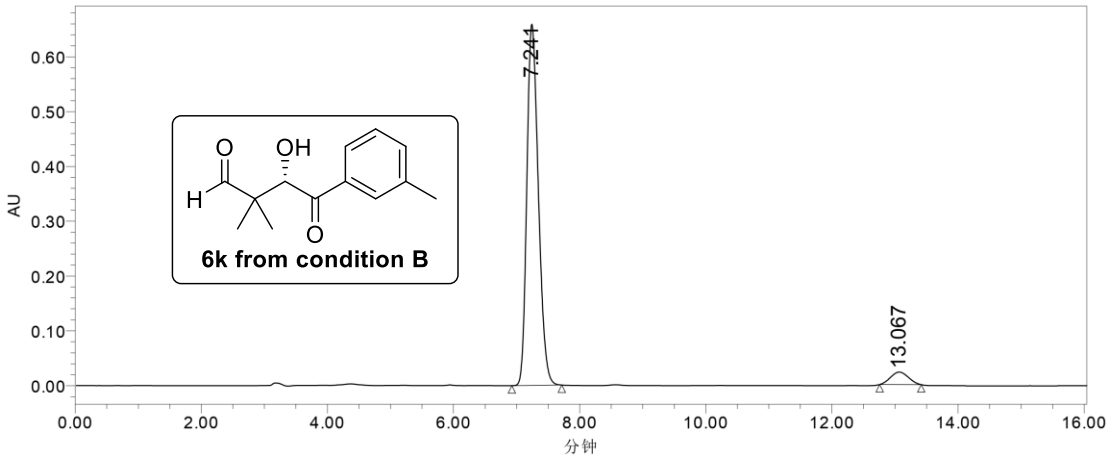
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.806	18894684	1070437	96.11
2	2998(210-400)	12.716	764726	37226	3.89



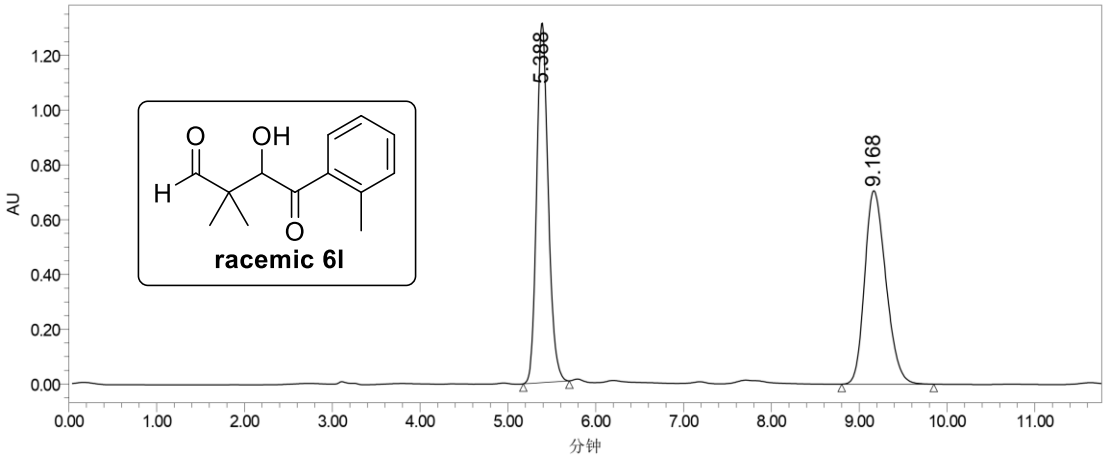
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.247	46320788	2952552	50.43
2	2998(210-400)	13.266	45524134	1596907	49.57



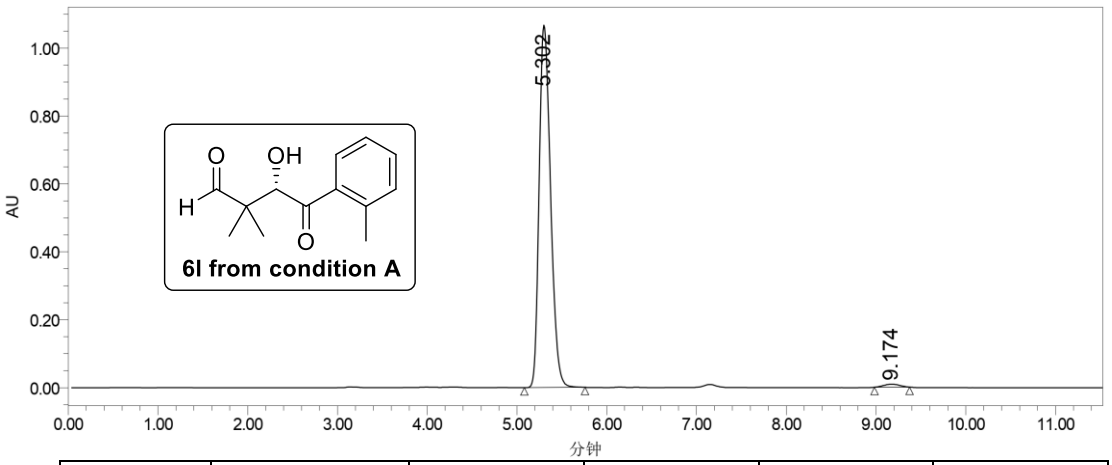
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.485	13752844	888630	96.46
2	2998(210-400)	13.366	504595	23450	3.54



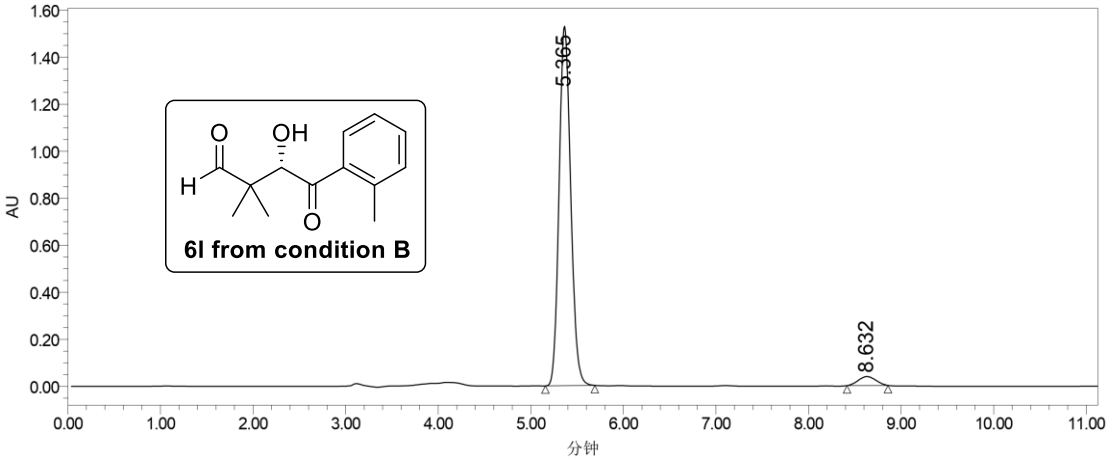
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.241	8347957	658887	94.92
2	2998(210-400)	13.067	446613	22790	5.08



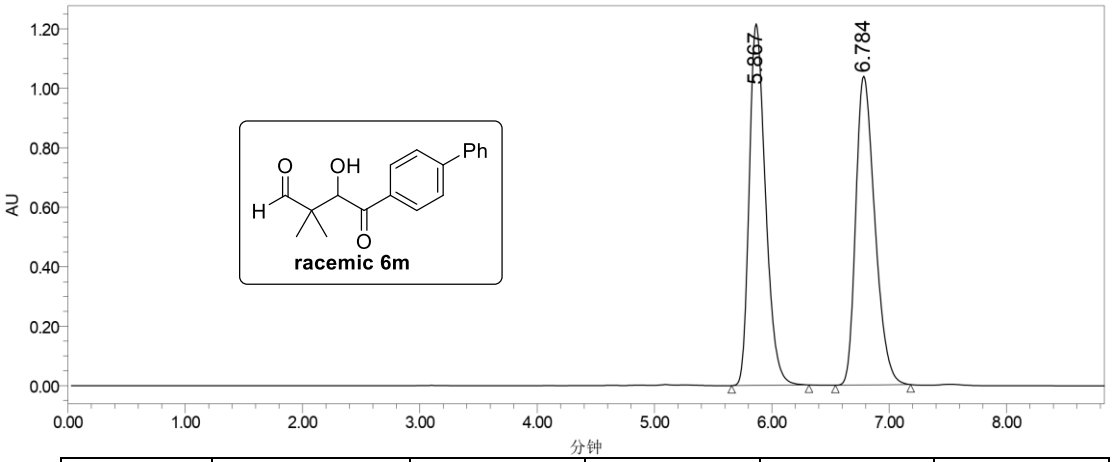
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	5.388	12022845	1314256	50.32
2	2998(210-400)	9.168	11870165	706266	49.68



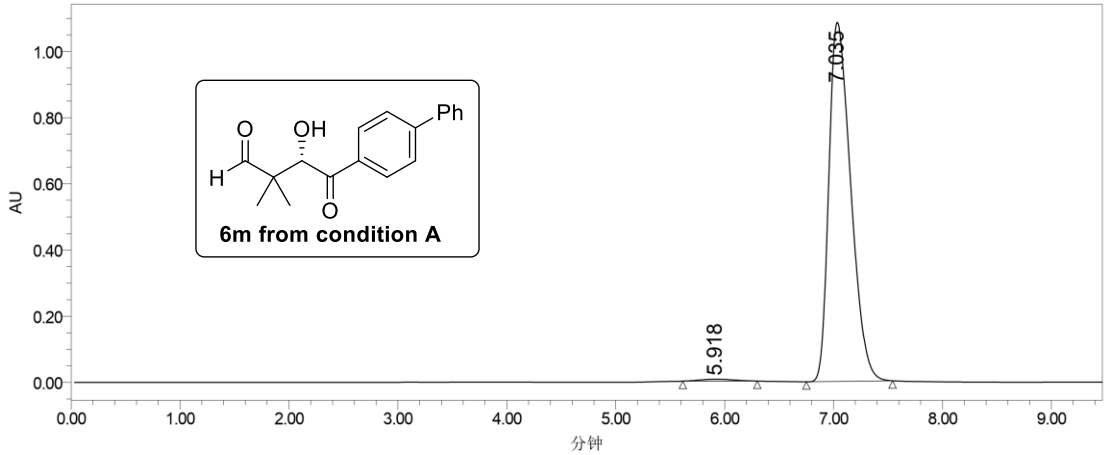
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	5.302	9405199	1067270	98.85
2	2998(210-400)	9.174	109169	8769	1.15



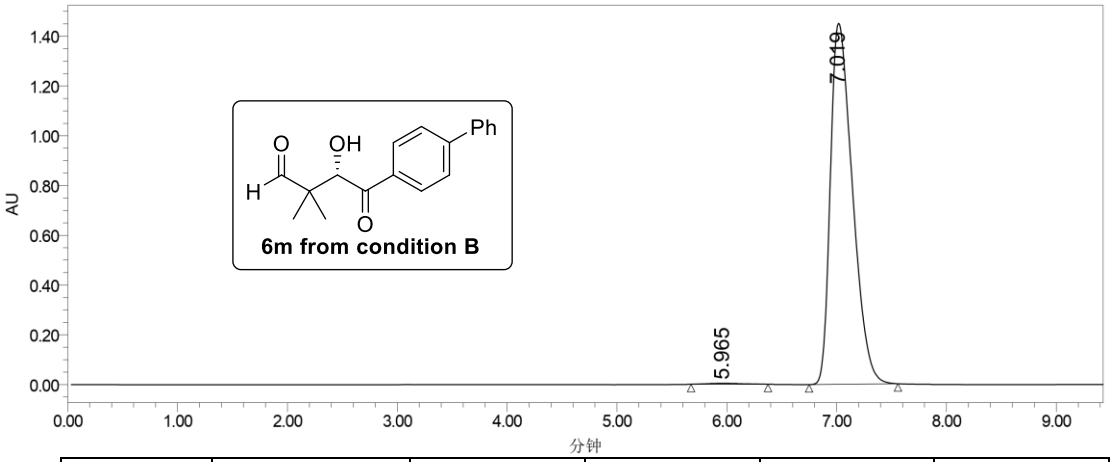
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	5.365	13310961	1529717	96.41
2	2998(210-400)	8.632	495777	37828	3.59



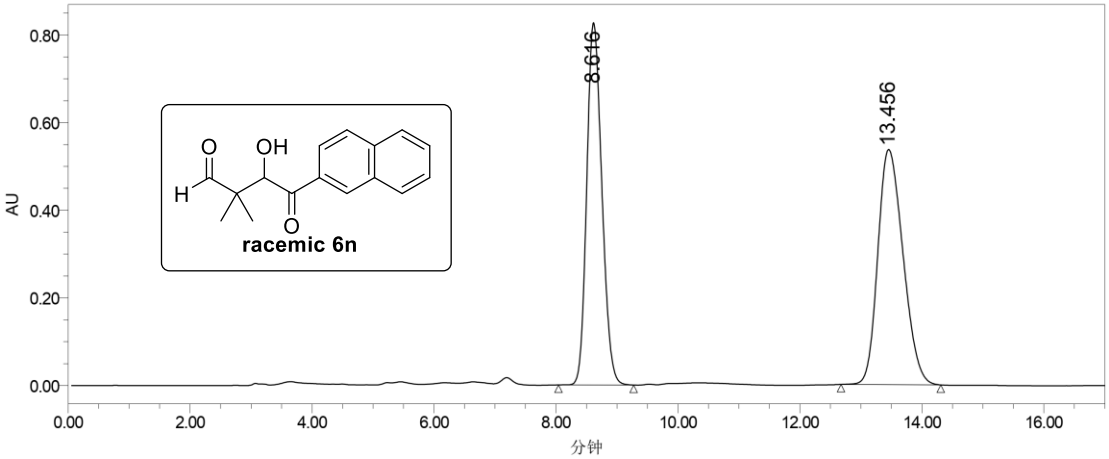
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	5.867	11728662	1215906	49.93
2	2998(210-400)	6.784	11762184	1038769	50.07



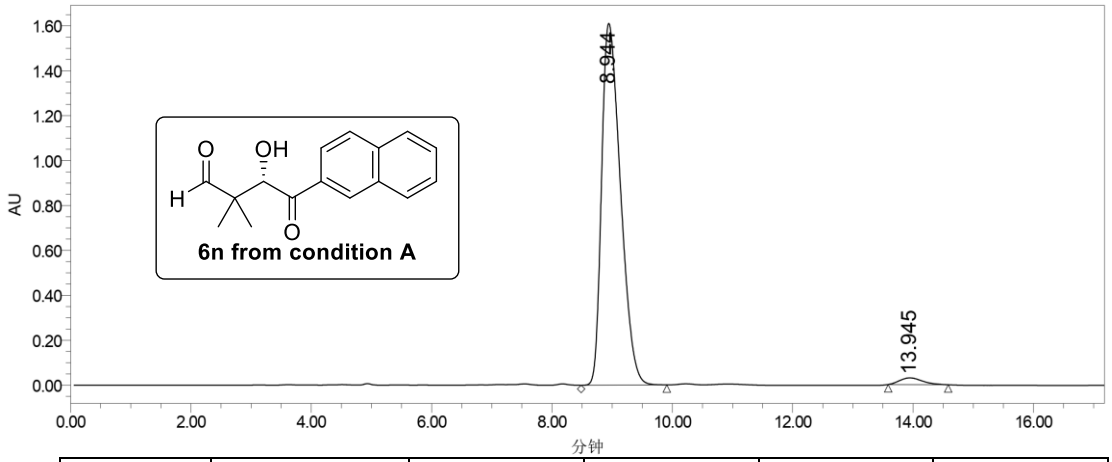
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	5.918	124994	5634	0.82
2	2998(210-400)	7.035	15121502	1085806	99.18



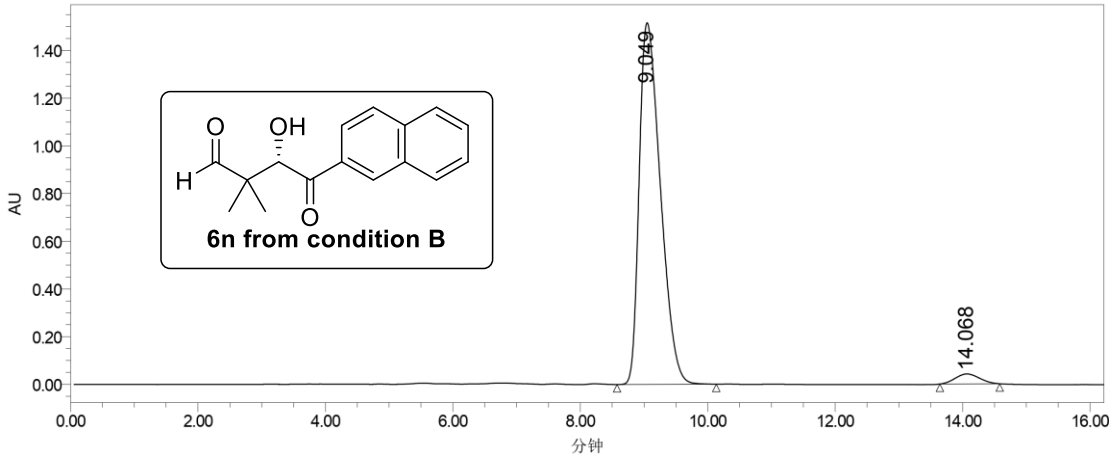
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	5.965	82147	3441	0.40
2	2998(210-400)	7.019	20256373	1451032	99.60



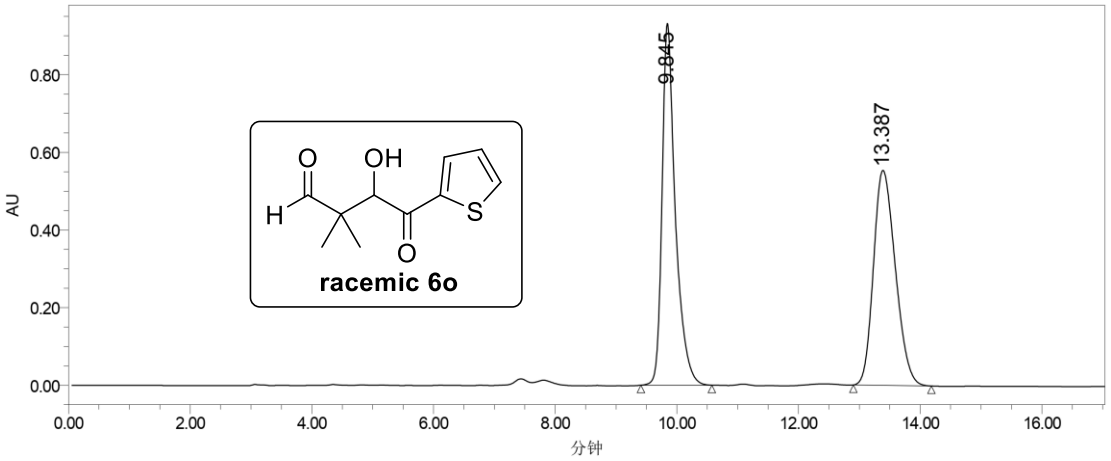
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.616	14224426	826762	47.98
2	2998(210-400)	13.456	15423873	536742	52.02



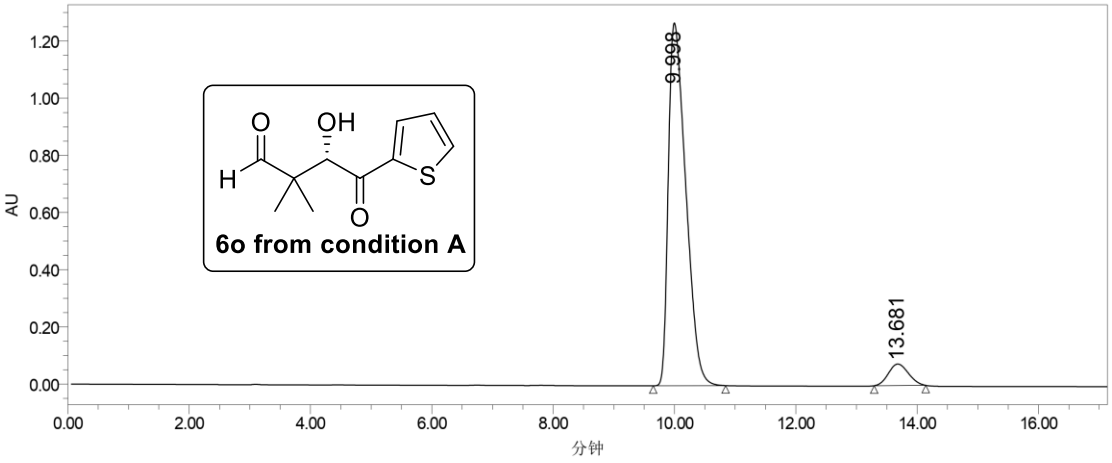
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	8.944	35108189	1610428	97.81
2	2998(210-400)	13.945	784487	29873	2.19



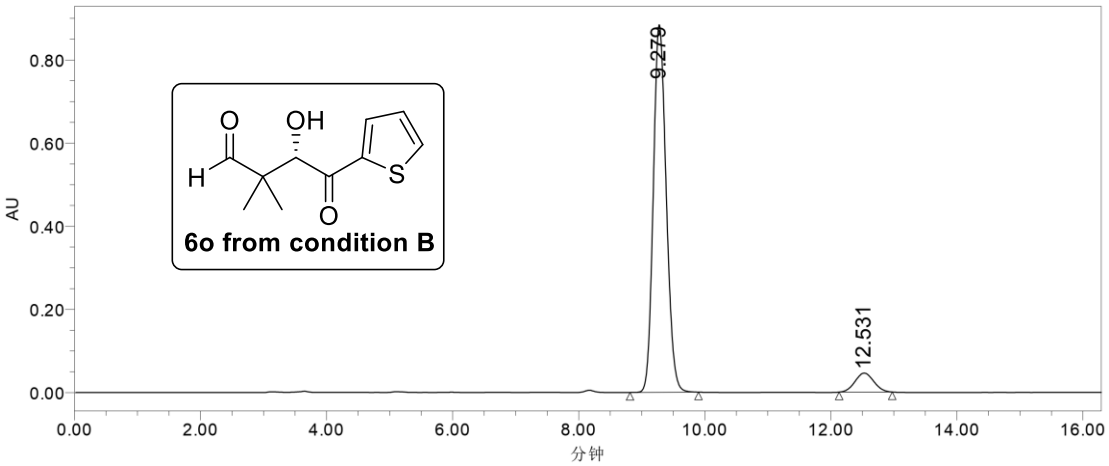
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	9.049	34989981	1515984	96.96
2	2998(210-400)	14.068	1098254	41624	3.04



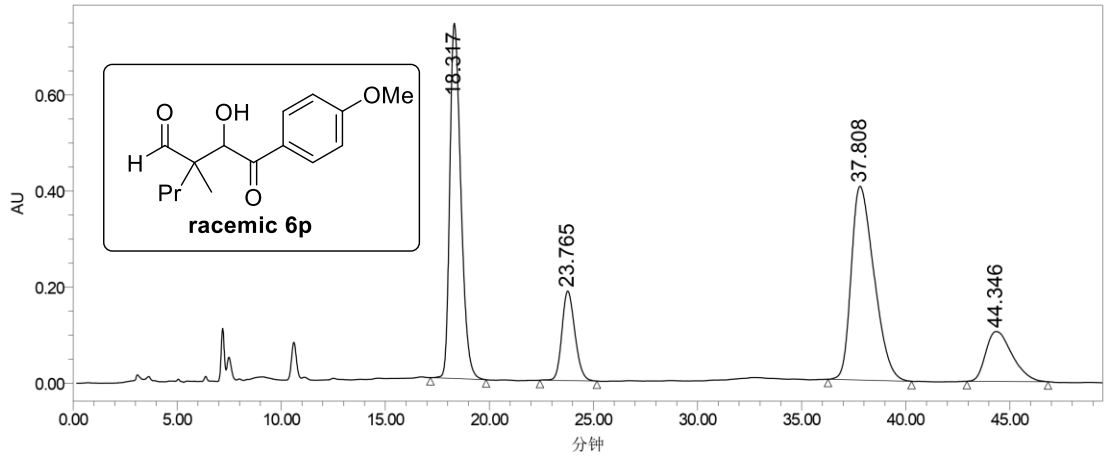
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	9.845	14110474	931891	50.53
2	2998(210-400)	13.387	13816249	553613	49.47



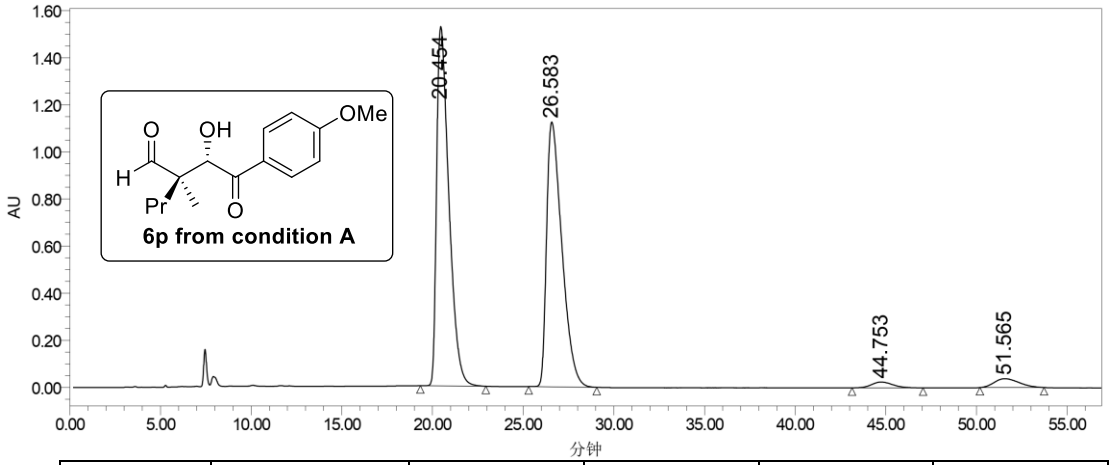
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	9.998	25259782	1268874	93.61
2	2998(210-400)	13.681	1722979	75401	6.39



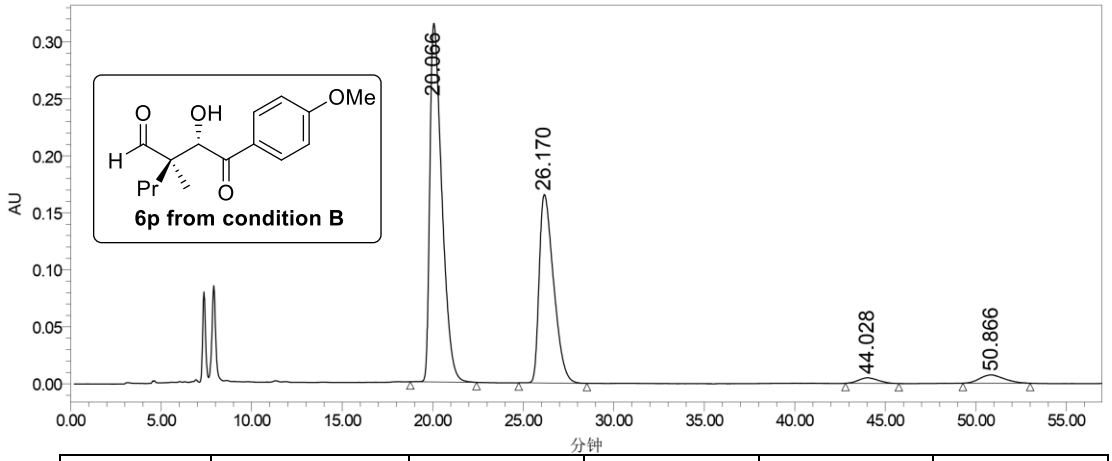
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	9.279	12613998	884555	93.04
2	2998(210-400)	12.531	943594	45674	6.96



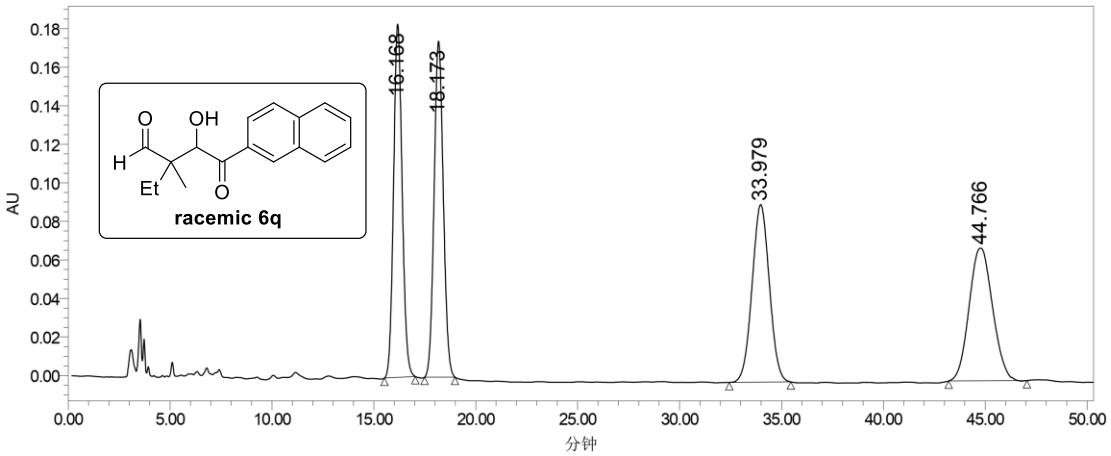
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	18.317	27303093	739451	36.24
2	2998(210-400)	23.765	8072275	186391	10.71
3	2998(210-400)	37.808	30817264	403447	40.91
4	2998(210-400)	44.346	9143852	103786	12.14



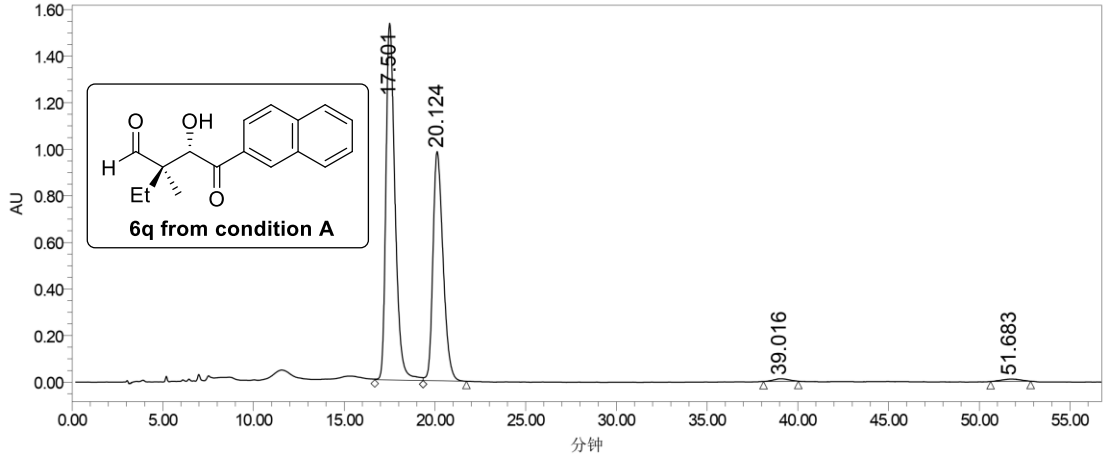
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	20.454	71597304	1525648	50.07
2	2998(210-400)	26.583	65859454	1124140	46.06
3	2998(210-400)	44.753	1965924	24519	1.37
4	2998(210-400)	51.565	3572904	37545	2.50



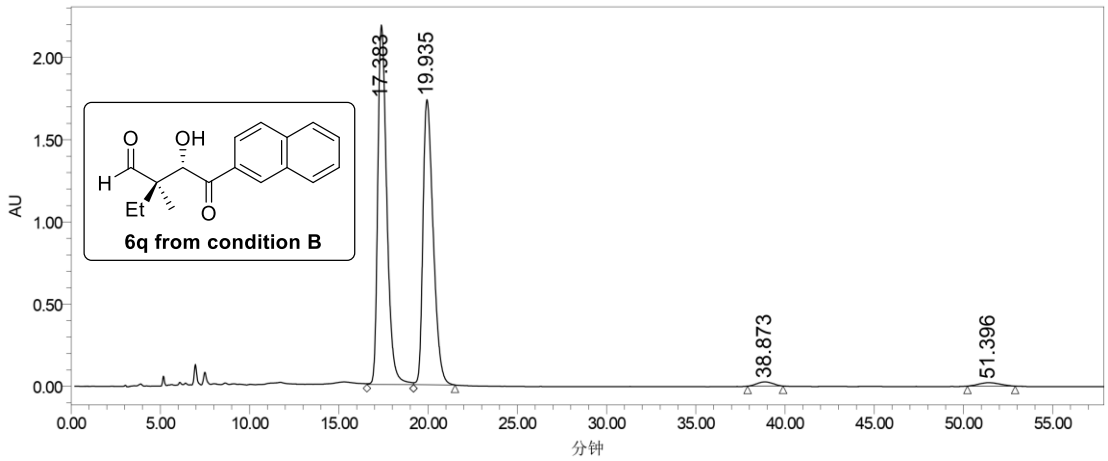
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	20.066	14458318	314522	58.64
2	2998(210-400)	26.170	9154814	165414	37.13
3	2998(210-400)	44.028	366876	4898	1.49
4	2998(210-400)	50.866	674865	7297	2.74



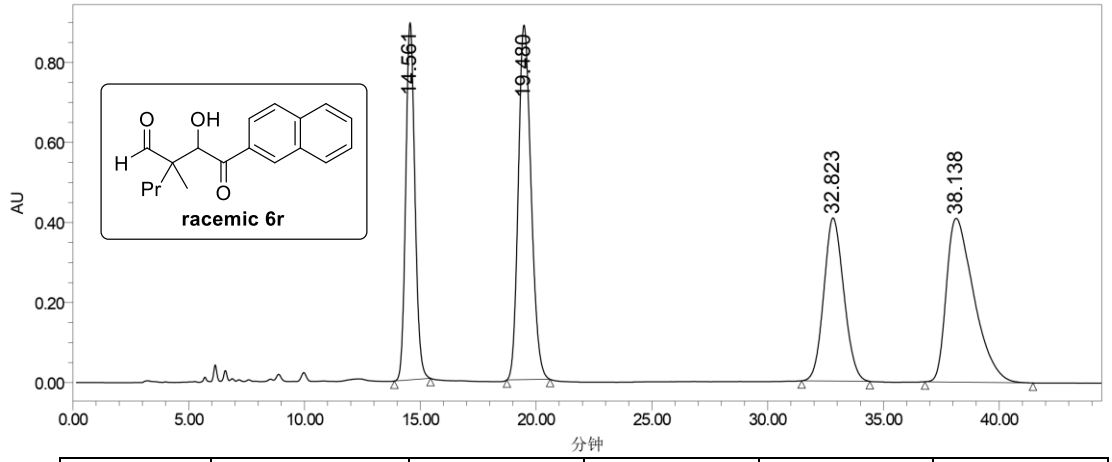
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	16.168	5355616	183308	24.62
2	2998(210-400)	18.173	5462849	174338	25.11
3	2998(210-400)	33.979	5433284	92122	24.97
4	2998(210-400)	44.766	5505255	68851	25.30



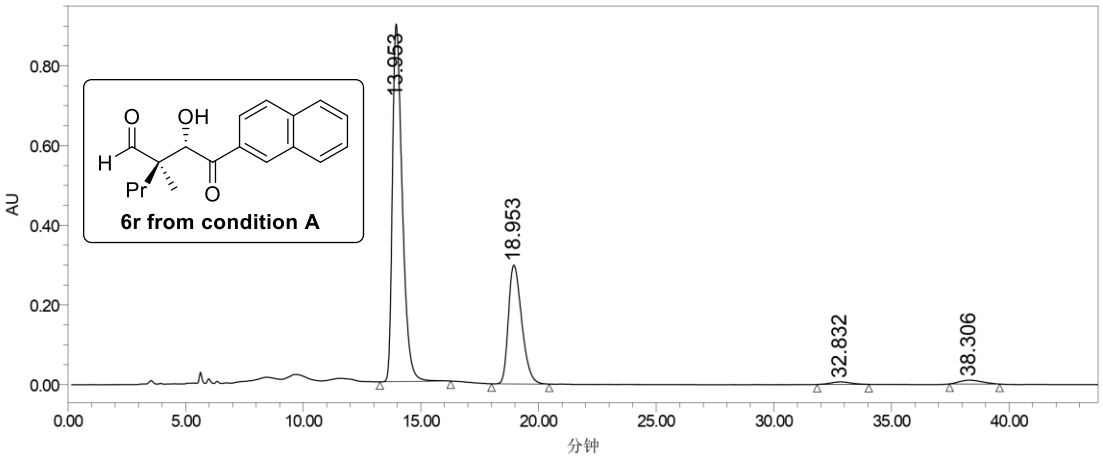
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	17.501	53560327	1531595	57.55
2	2998(210-400)	20.124	38154226	984381	40.99
3	2998(210-400)	39.016	643082	11185	0.69
4	2998(210-400)	51.683	713152	9578	0.77



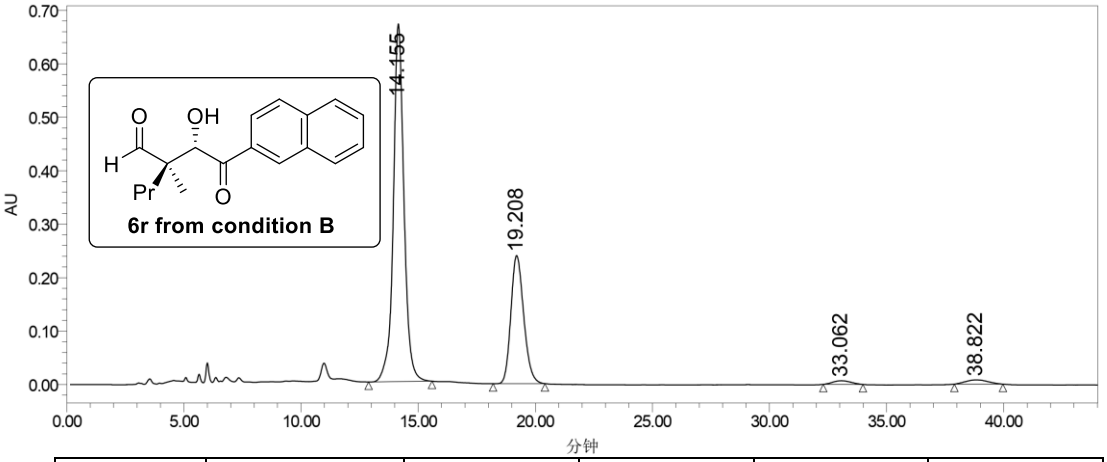
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	17.383	76905487	2186613	51.53
2	2998(210-400)	19.935	69063081	1734457	46.28
3	2998(210-400)	38.873	1562094	25577	1.05
4	2998(210-400)	51.396	1712771	21215	1.15



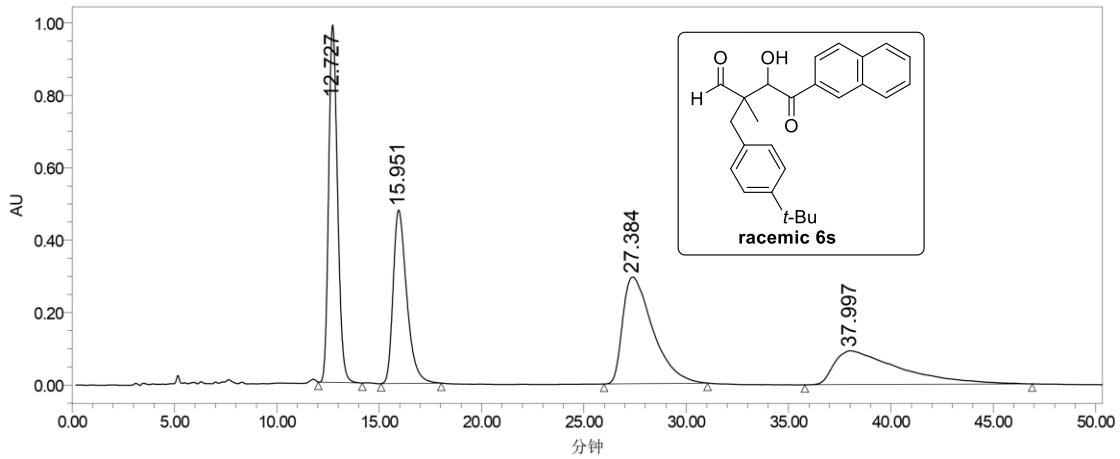
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	14.561	24459815	892814	20.96
2	2998(210-400)	19.480	33911610	886432	29.07
3	2998(210-400)	32.823	24433370	407656	20.94
4	2998(210-400)	38.138	33866099	409251	29.03



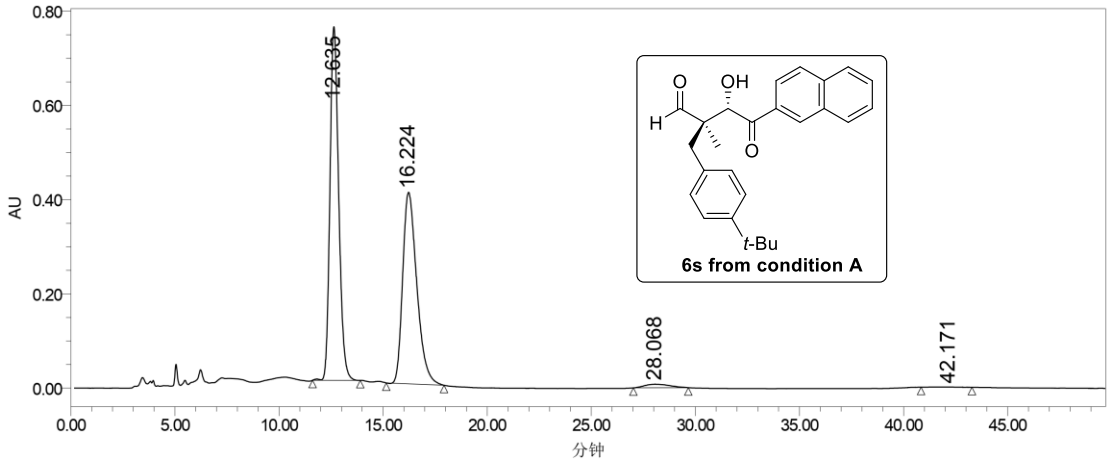
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	13.953	26137172	897308	67.25
2	2998(210-400)	18.953	11726881	298179	30.17
3	2998(210-400)	32.832	367910	6258	0.95
4	2998(210-400)	38.306	632331	9565	1.63



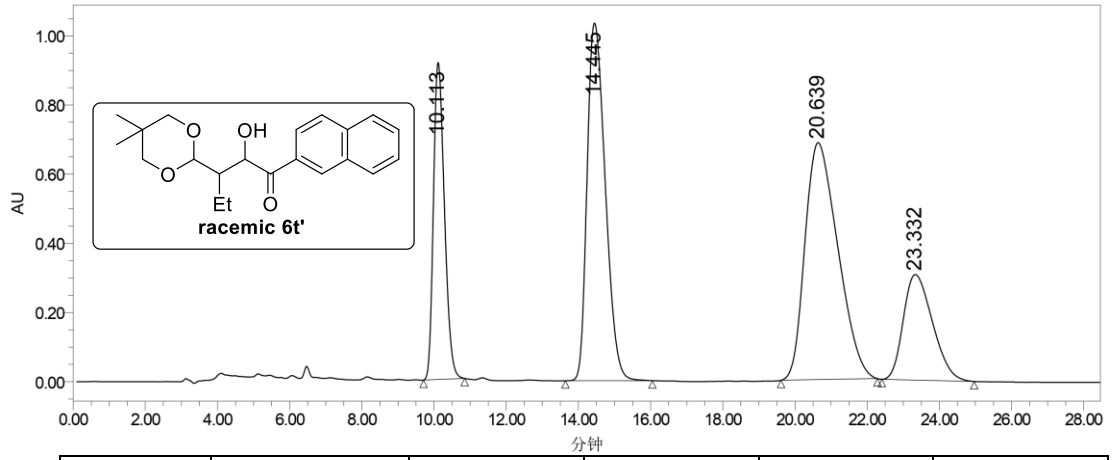
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	14.155	21055089	669335	67.75
2	2998(210-400)	19.208	9135823	239774	29.40
3	2998(210-400)	33.062	366638	6829	1.18
4	2998(210-400)	38.822	519624	7843	1.67



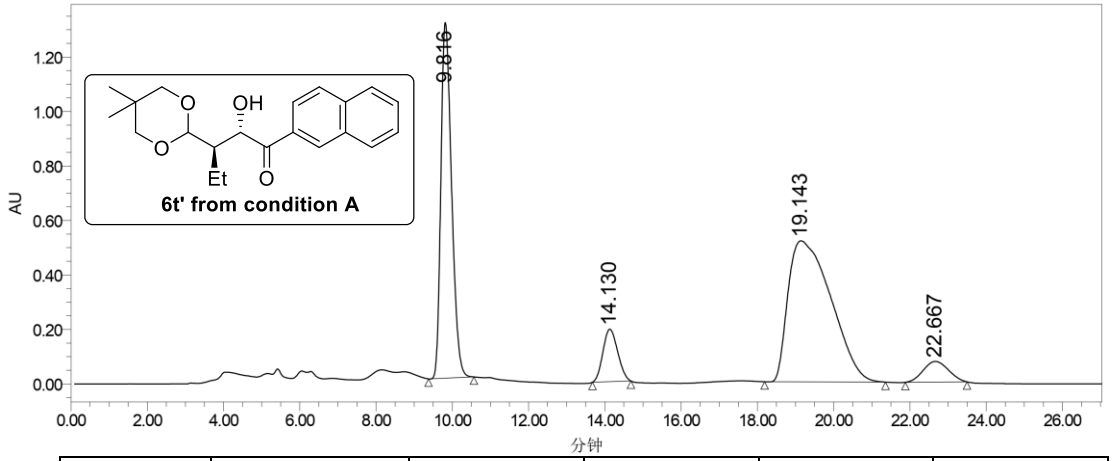
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	12.727	29702253	987138	28.93
2	2998(210-400)	15.951	22110152	479196	21.53
3	2998(210-400)	27.384	30124254	294750	29.34
4	2998(210-400)	37.997	20736036	93437	20.20



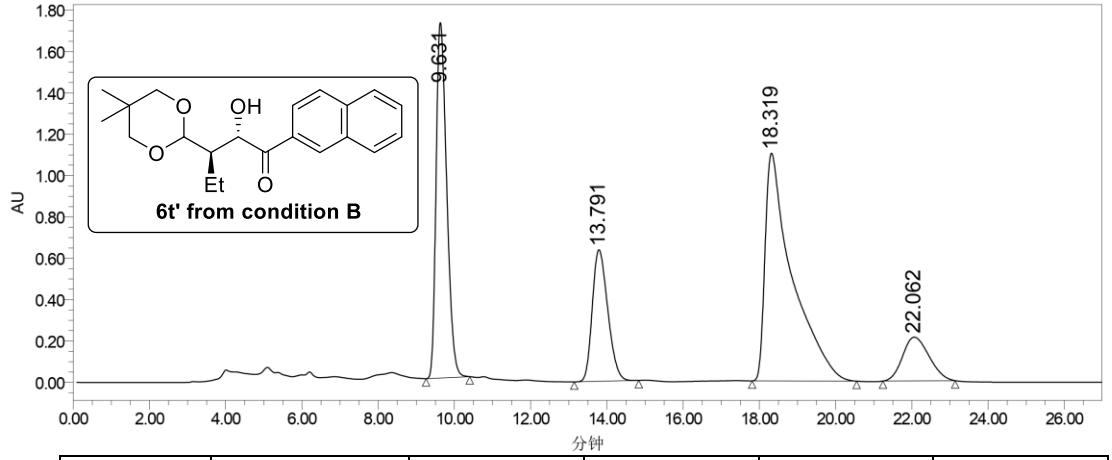
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	12.635	21833576	750492	52.36
2	2998(210-400)	16.224	19197730	406841	46.04
3	2998(210-400)	28.068	629408	7528	1.51
4	2998(210-400)	42.171	38808	584	0.09



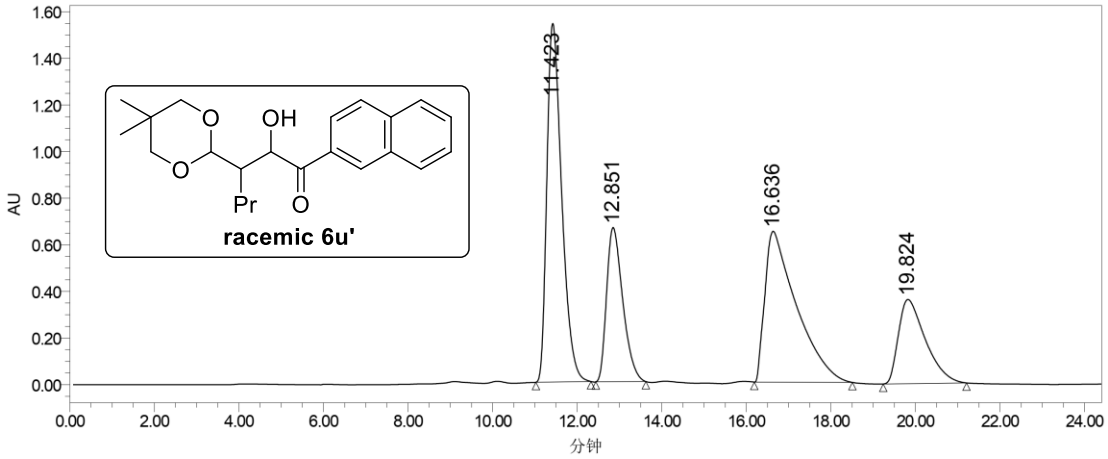
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	10.113	19399329	916593	16.73
2	2998(210-400)	14.445	36059465	1034057	31.09
3	2998(210-400)	20.639	43366941	685317	37.39
4	2998(210-400)	23.332	17152622	305092	14.79



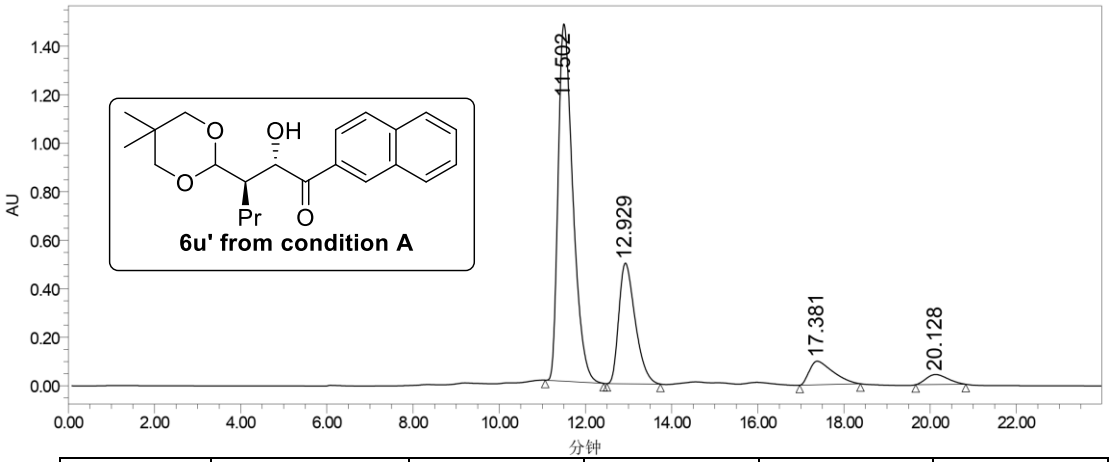
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	9.816	25354864	1305334	34.06
2	2998(210-400)	14.130	5183901	192467	6.96
3	2998(210-400)	19.143	40423829	517650	54.30
4	2998(210-400)	22.667	3476061	75776	4.67



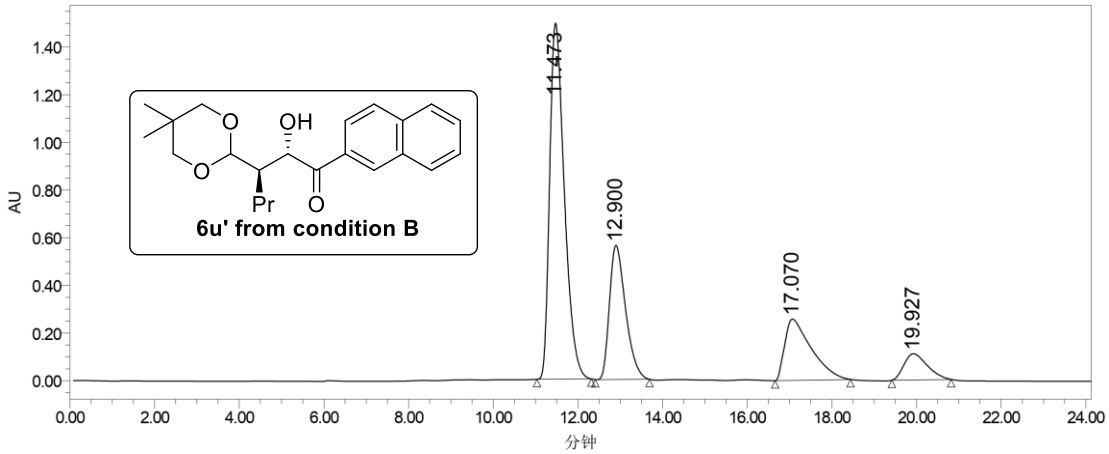
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	9.631	33144540	1719054	28.65
2	2998(210-400)	13.791	17965488	636190	15.53
3	2998(210-400)	18.319	54362817	1101229	46.99
4	2998(210-400)	22.062	10222851	211406	8.84



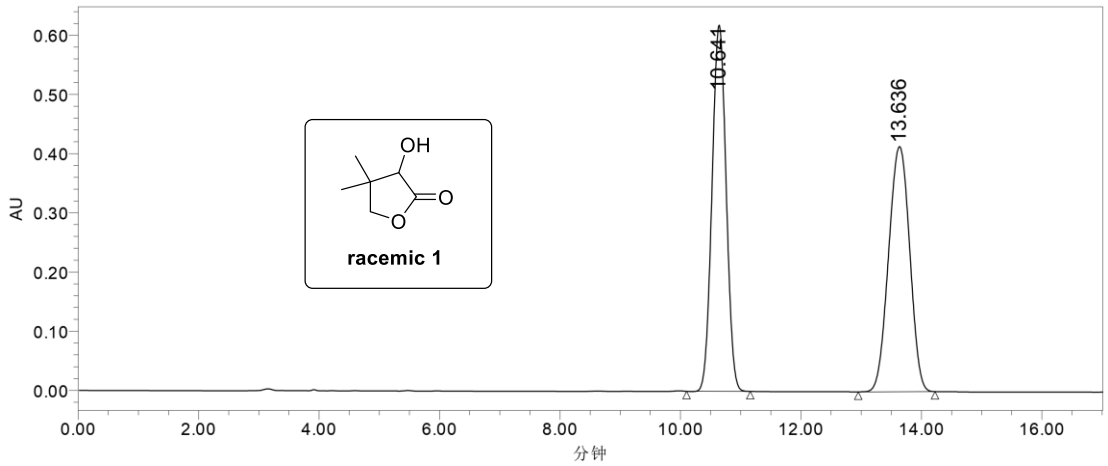
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	11.423	37537677	1538409	35.84
2	2998(210-400)	12.851	17571925	661767	16.78
3	2998(210-400)	16.636	33615902	647355	32.10
4	2998(210-400)	19.824	16008689	361263	15.29



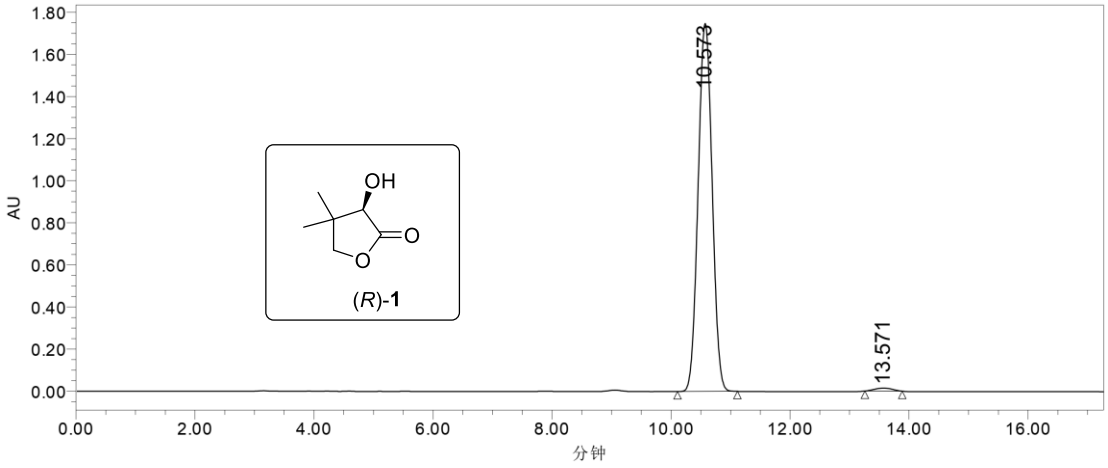
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	11.502	35053023	1472811	65.70
2	2998(210-400)	12.929	13162674	497427	24.67
3	2998(210-400)	17.381	3694819	97235	6.93
4	2998(210-400)	20.128	1440505	41176	2.70



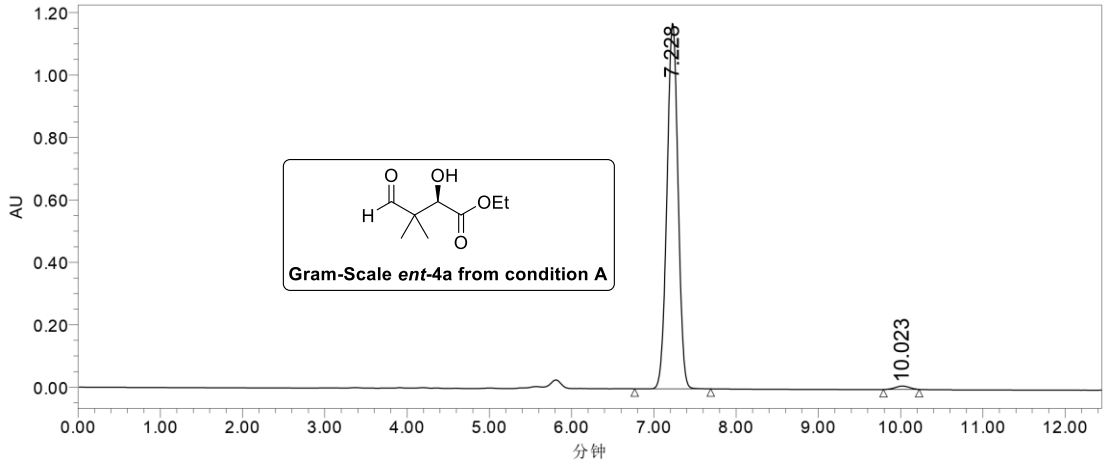
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	11.473	35973050	1494076	54.38
2	2998(210-400)	12.900	14876986	563030	22.49
3	2998(210-400)	17.070	11110884	256962	16.80
4	2998(210-400)	19.927	4189472	109724	6.33



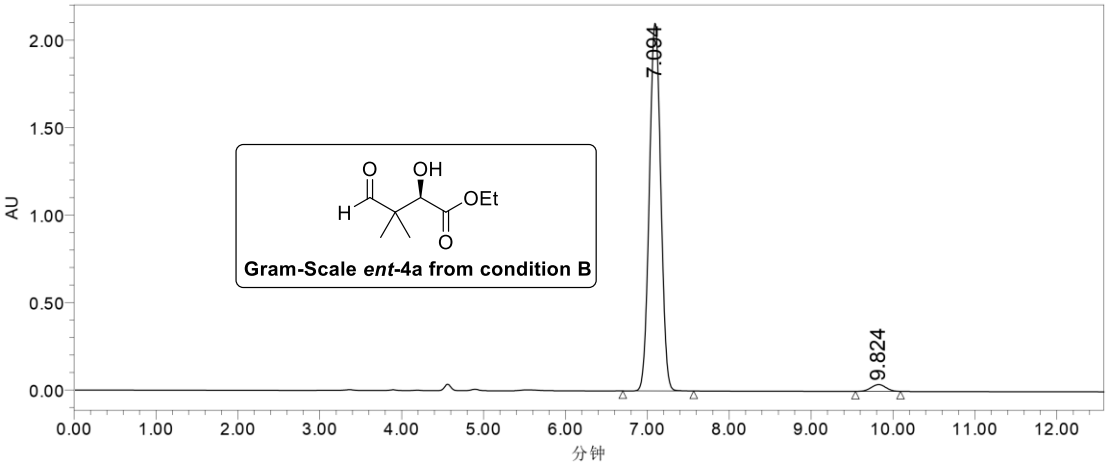
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	10.641	10073393	618713	49.97
2	2998(210-400)	13.636	10085609	414287	50.03



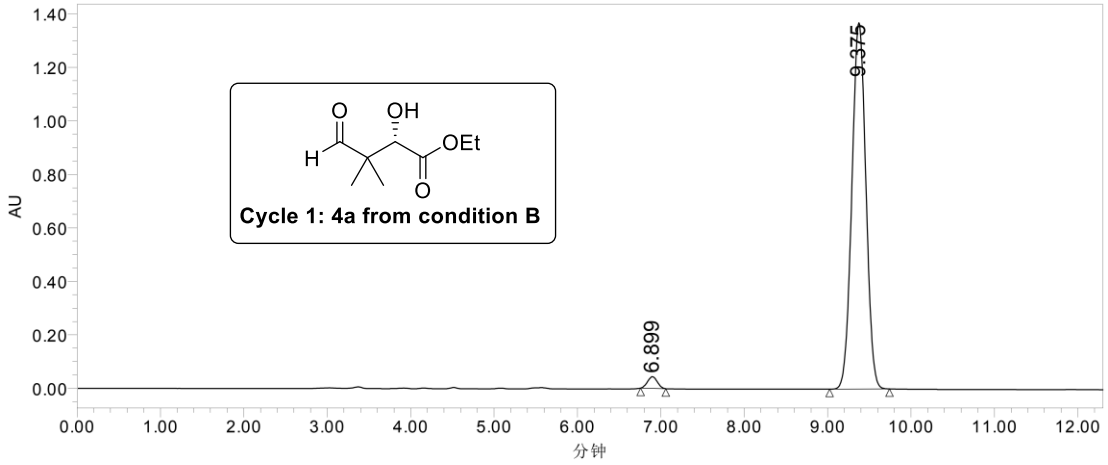
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	10.573	28604271	1747260	98.97
2	2998(210-400)	13.571	298759	14248	1.03



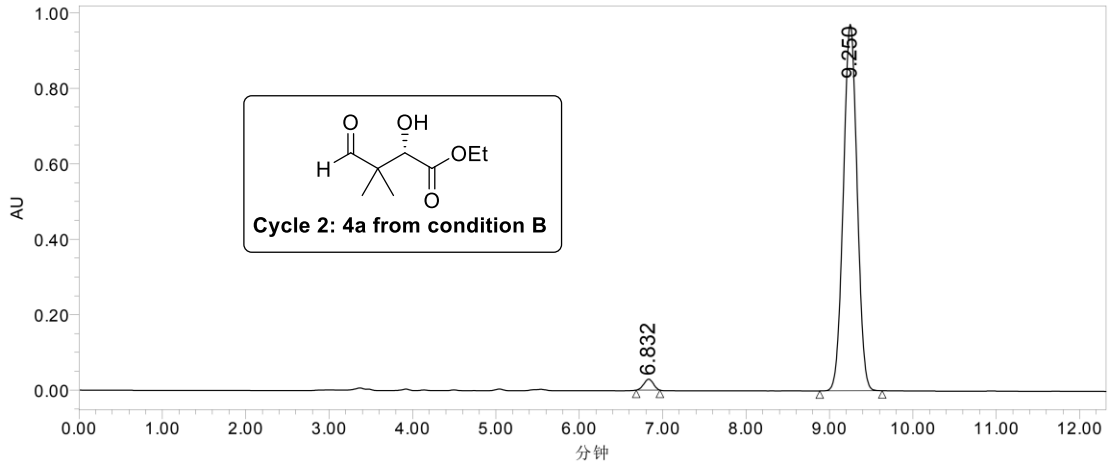
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.228	10962363	1171845	98.82
2	2998(210-400)	10.023	130574	10809	1.18



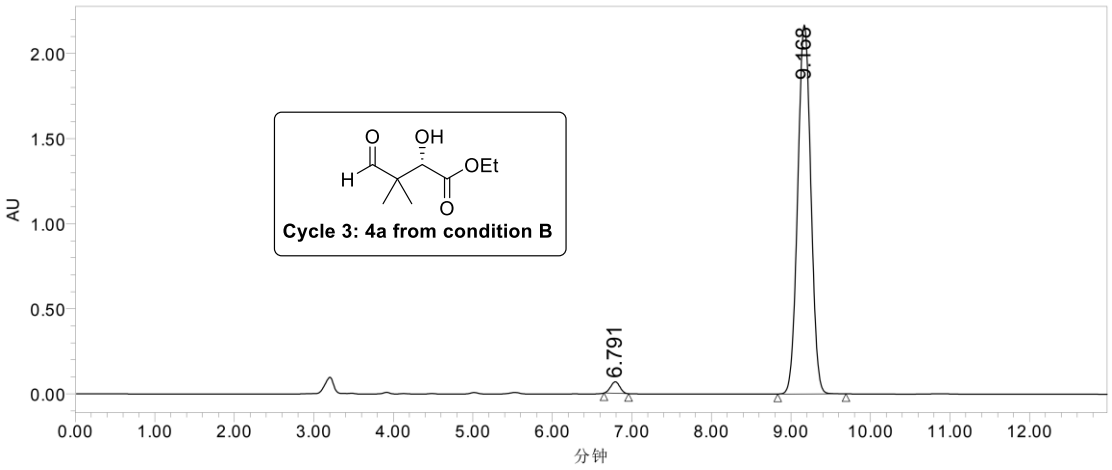
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	7.094	20391774	2103095	97.63
2	2998(210-400)	9.824	494329	39146	2.37



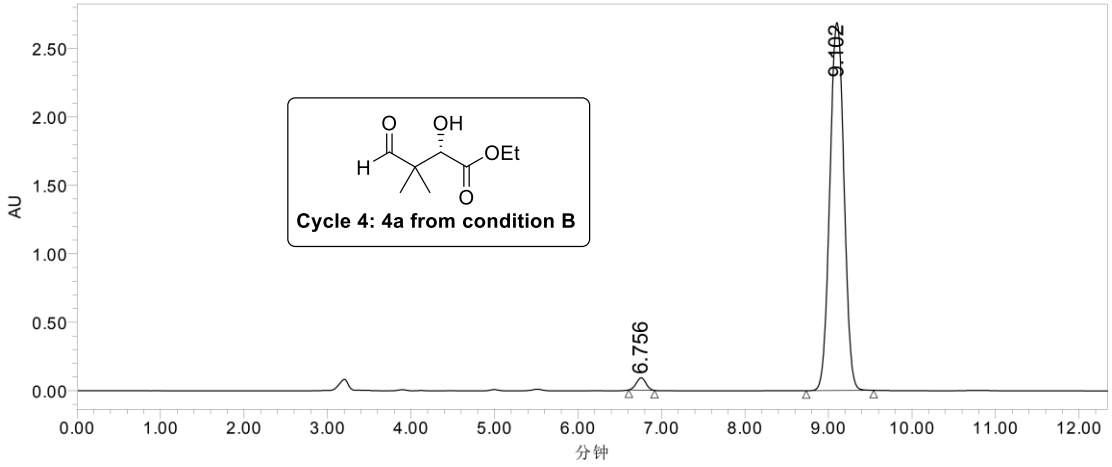
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.899	353288	43738	2.16
2	2998(210-400)	9.375	15977138	1369080	97.84



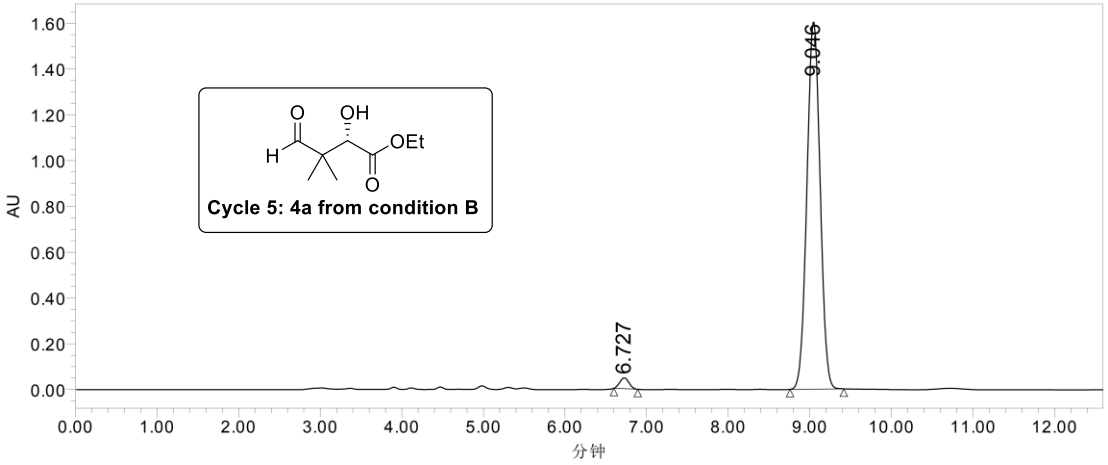
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.832	228479	28847	2.01
2	2998(210-400)	9.250	11141869	971376	97.99



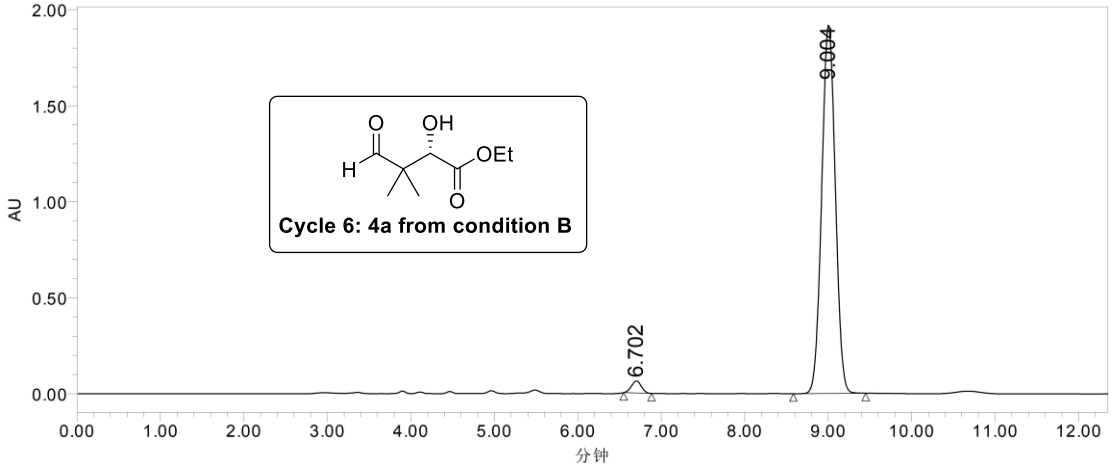
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.791	549089	68269	2.16
2	2998(210-400)	9.168	24922679	2167977	97.84



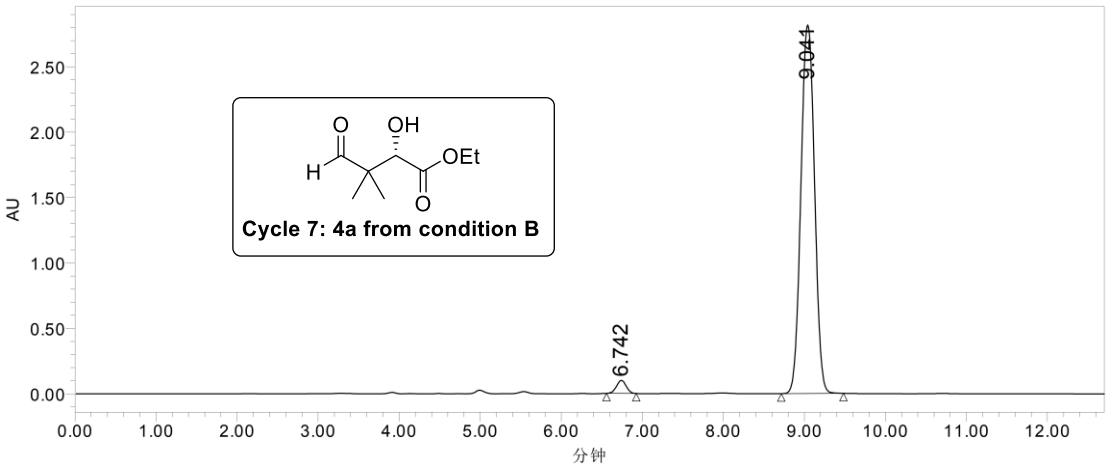
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.756	726066	89959	2.23
2	2998(210-400)	9.102	31765039	2688564	97.77



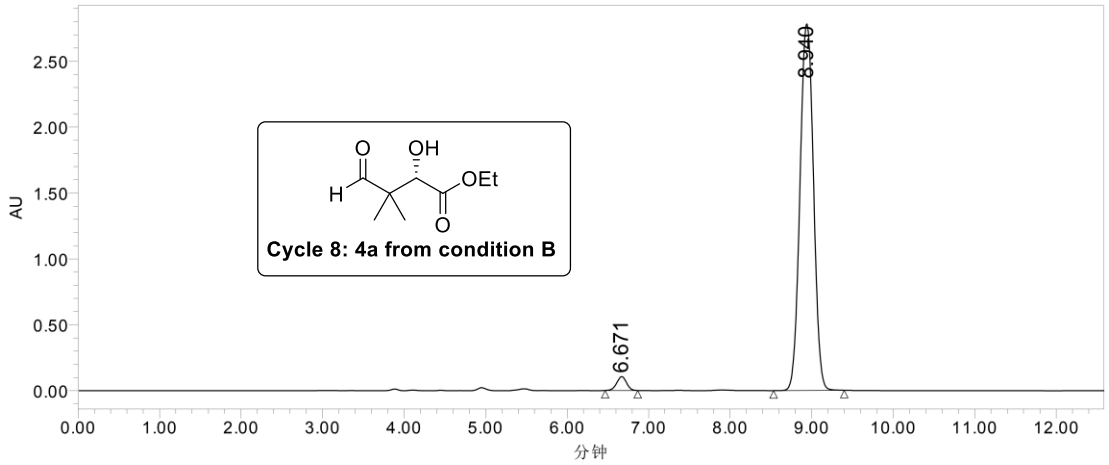
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.727	372029	47379	2.03
2	2998(210-400)	9.046	17992161	1604266	97.97



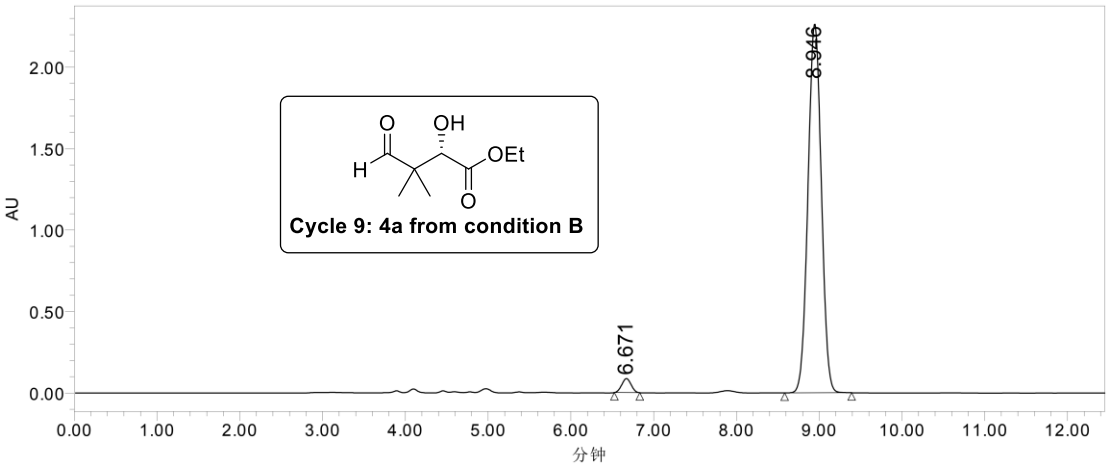
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.702	517078	62622	2.32
2	2998(210-400)	9.004	21758573	1918986	97.68



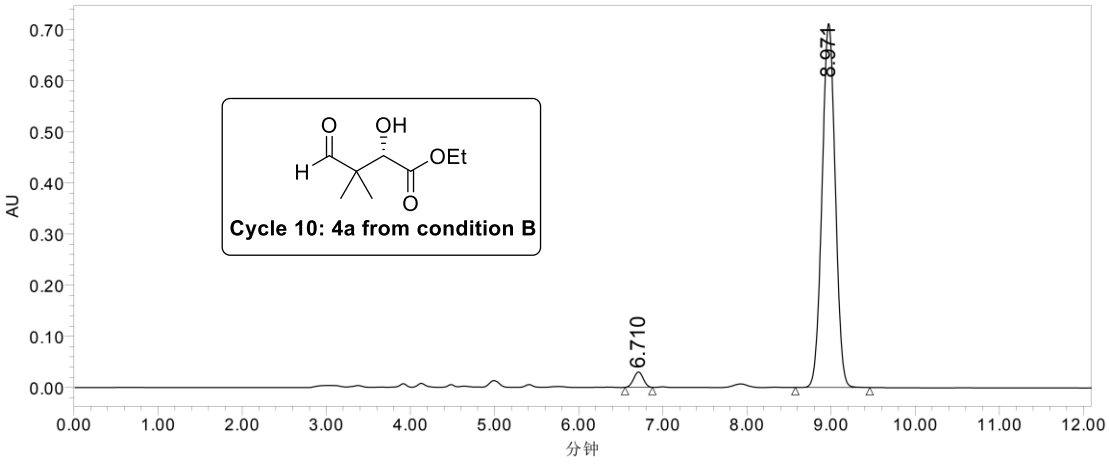
	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.742	828173	99438	2.46
2	2998(210-400)	9.041	32900548	2816293	97.54



	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.671	873880	104546	2.65
2	2998(210-400)	8.940	32066454	2781108	97.35



	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.671	680653	86658	2.62
2	2998(210-400)	8.946	25337313	2264805	97.38



	Name	Retention	Area(μV*s)	Height(μV)	% Area
1	2998(210-400)	6.710	238999	30128	2.98
2	2998(210-400)	8.971	7774892	712045	97.02

8. Reference

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