

Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Bifunctional Primary Amine-Thiourea-TfOH (BPAT·TfOH) as Chiral Phase-Transfer Catalysts:

Asymmetric Synthesis of Dihydropyrimidines

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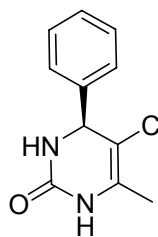
General Comments.

All reactions were performed under air atmosphere. Unless otherwise indicated, all materials were obtained from commercial sources, and used as purchased without dehydration. Element analyses were carried out on a Yanaco CHN Corder MT-3 automatic analyzer in the Analysis Center of Nankai University. ^1H NMR and ^{13}C NMR spectra were recorded in CDCl_3 or $[\text{D}_6]$ -DMSO at Bruker 400 MHz spectrometers. TMS served as internal standard ($\delta = 0$ ppm) for ^1H MNR and DMSO were used as internal standard ($\delta = 42.4$ ppm) for ^{13}C NMR; the coupling constants J are given in Hz. HPLC analyses were recorded on a chiral column (Daicel Chiralcel OD-H column, at 254 nm). Melting points were determined on a T-4 melting point apparatus. Optical rotations were recorded on a Perkin Elemer 241 Polarimeter.

General Procedure for the Biginelli reaction:

To a suspension of the catalyst BPAT (I) (0.039 g, 0.075 mmol) in saturated brine (2 mL) was added trifluoromethane sulfonic acid (0.011 g, 0.075 mmol). After stirring for 30 minutes, aldehyde **6** (0.75 mmol), urea **5** (0.030 g, 0.5 mmol), ethyl acetoacetate **7** (0.195 g, 1.5 mmol) and $t\text{-BuNH}_2\cdot\text{TFA}$ (0.009 g, 0.05 mmol) were added sequentially. The reaction mixture was stirred at room temperature for 36 hours. After completion of the reaction, the result mixture was then extracted by AcOEt and dried with anhydrous sodium sulfate. After concentrated the residue was purified by CC (silica gel, AcOEt/petroleum ether (b.p. 60-90°C) 3:2) to afford DHPMs as a white solid.

(S)-(+)-5-Ethoxycarbonyl-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1H)-one (8a):



White solid; mp 206-207 °C; $[\alpha]_D^{20} = 65^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz,

CDCl_3): δ 1.16 (t, $^3J_{\text{H,H}} = 6.8$ Hz, 3H, OCH_2CH_3), 2.34 (s, 3H, CH_3), 4.08 (q, $^3J_{\text{H,H}} =$

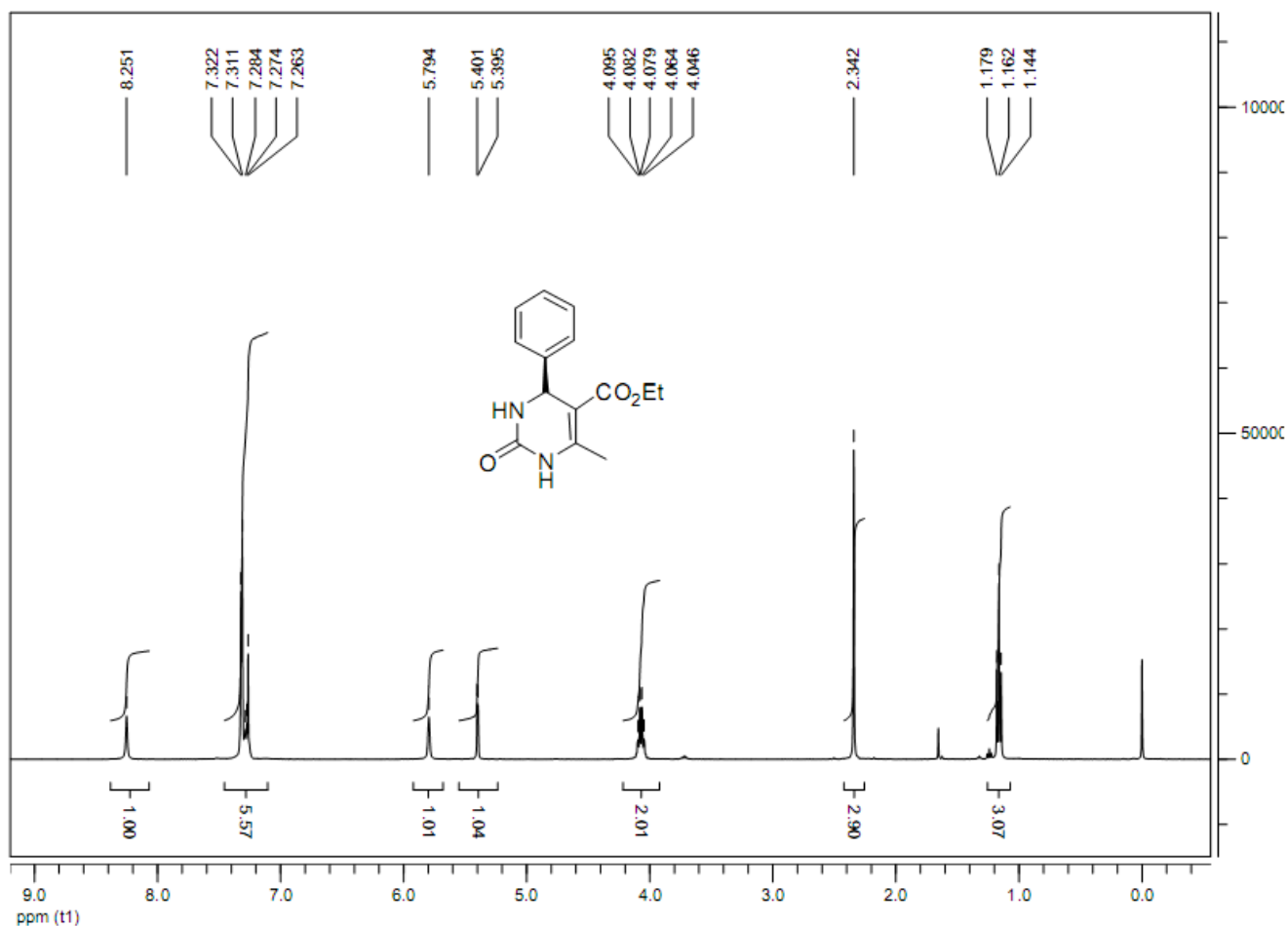
6.8 Hz, 2H, OCH_2CH_3), 5.40 (d, $^3J_{\text{H,H}} = 2.4$ Hz, 1H, CH), 5.79 (s, 1H, NH),

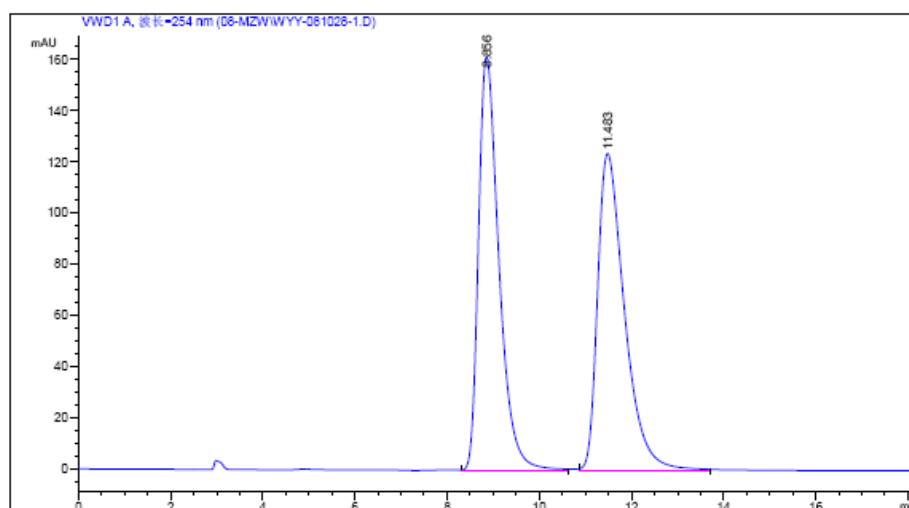
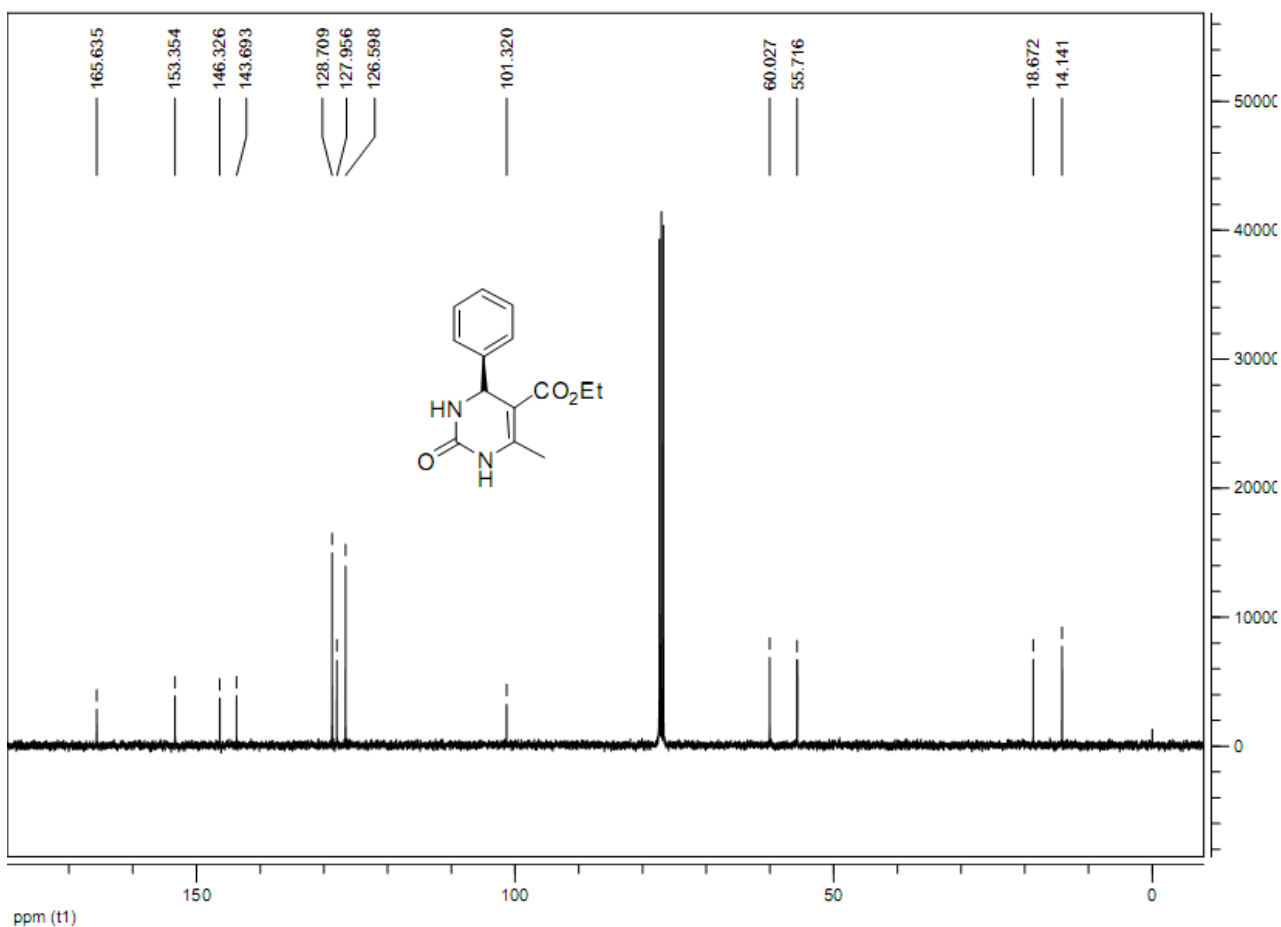
7.27-7.32 (m, 5H, Ph), 8.25 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ 14.14, 18.67, 55.72, 60.03,

101.32, 126.60, 127.99, 128.71, 143.09, 146.33, 153.35, 165.63; ESI-MS: 259.07 ($[\text{M-H}]^-$); The

enantiomeric excess was determined to be >99% by HPLC with Chiralcel OD-H column (25 cm) at

254 nm; eluent: hexane: $^i\text{PrOH}$ (85:15), flow rate = 1.0 mL/min, $t_r = 9.567$ min (major).

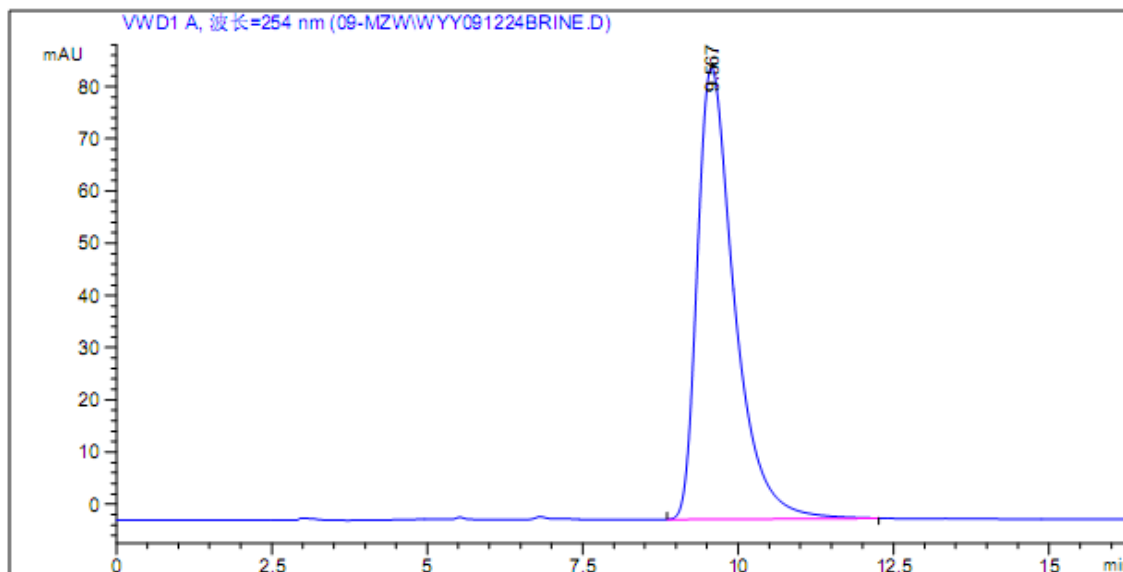




信号 1: VWD1 A, 波长=254 nm

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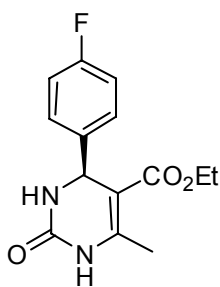
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信号 1: VWD1 A, 波长=254 nm

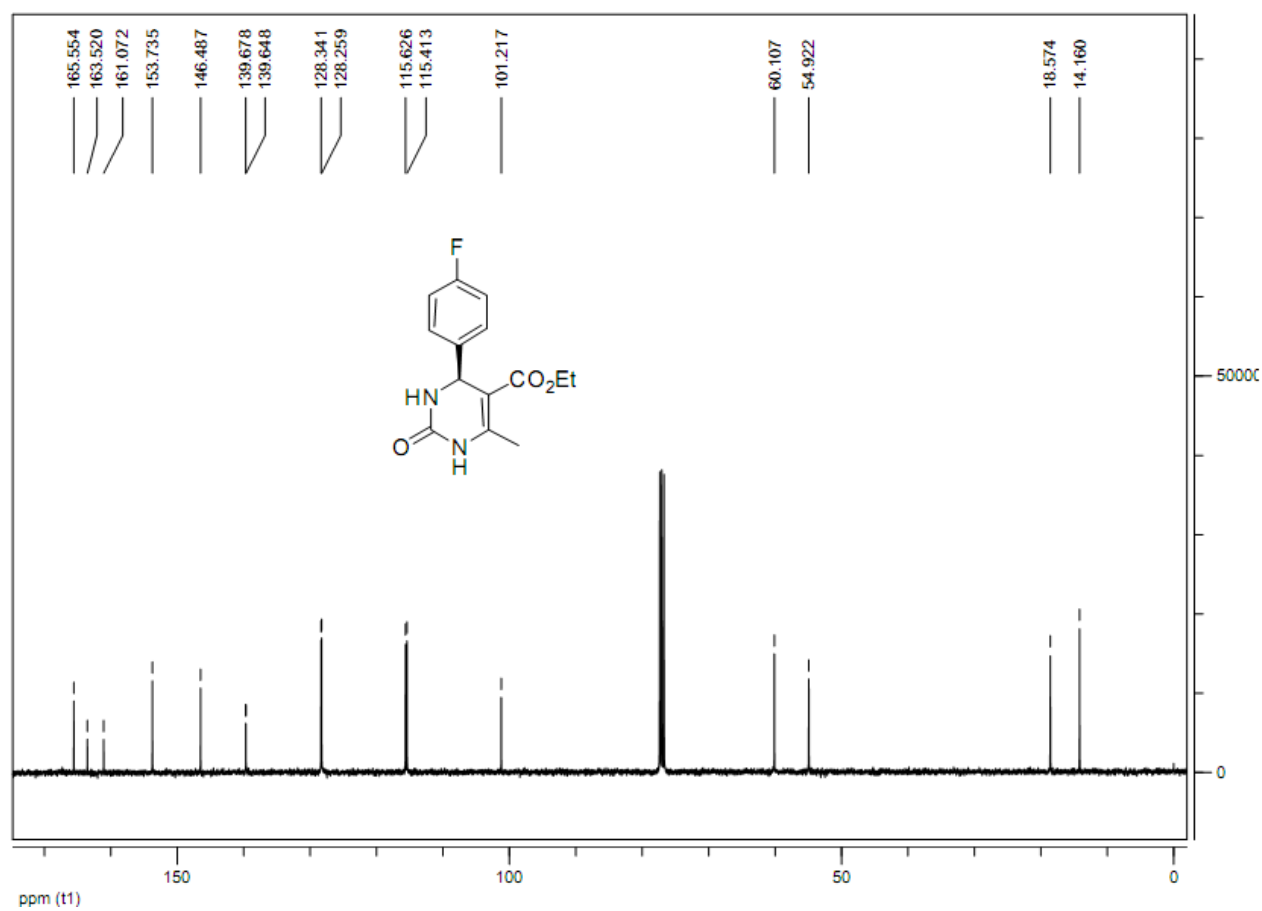
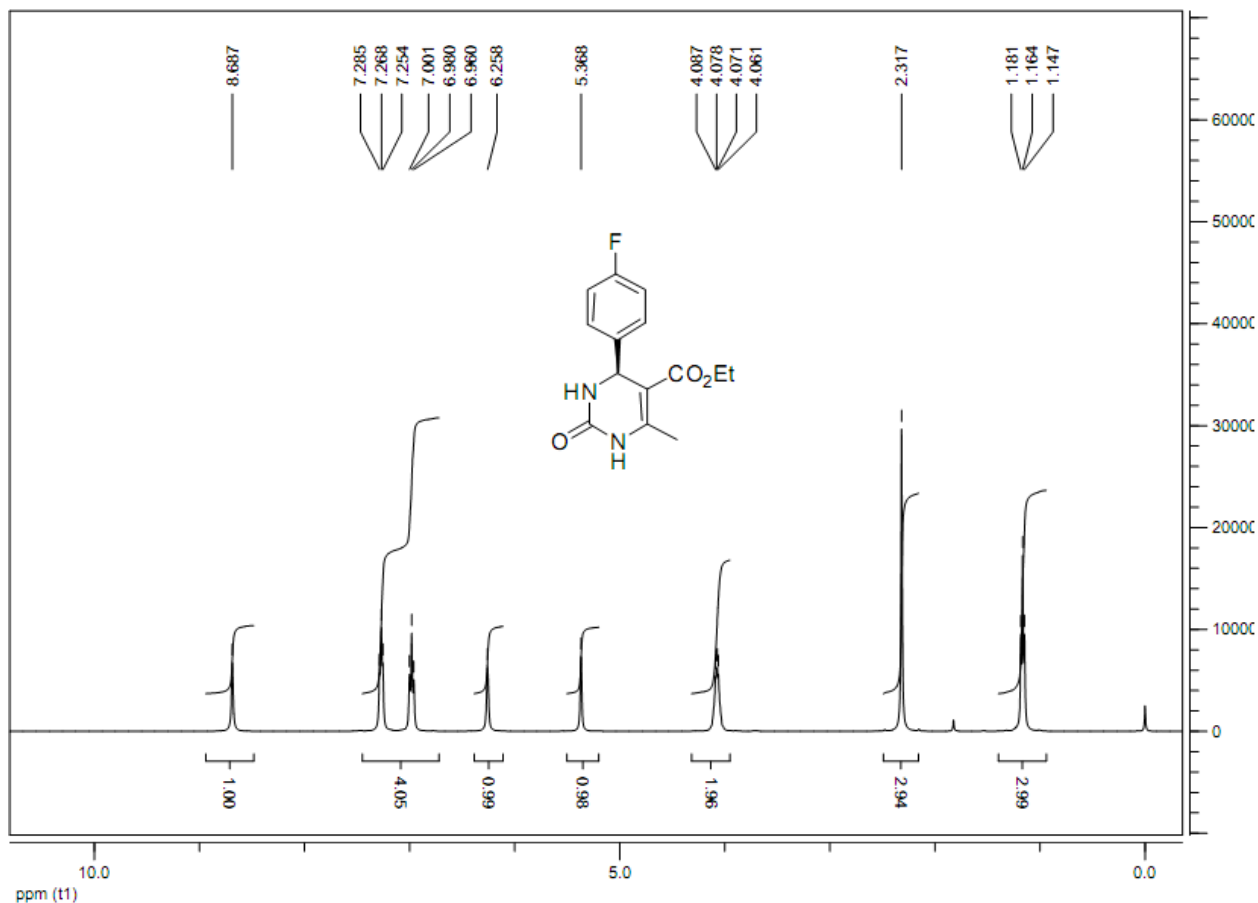
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1	9.567	BB	0.6234	3590.40283	86.60828	100.0000
总量 :				3590.40283	86.60828	

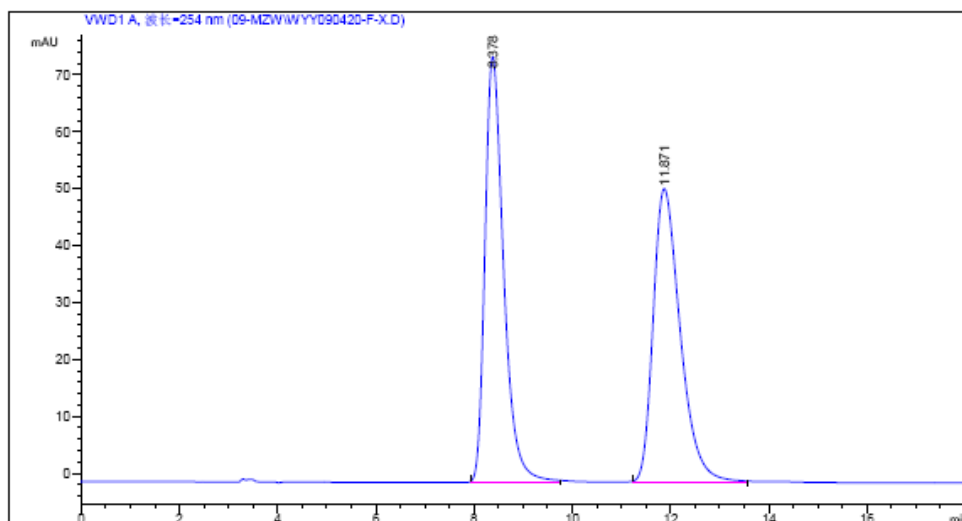
(S)-(+)-5-Ethoxycarbonyl-6-methyl-4-(4-fluorophenyl)-3,4-dihydropyrimidin-2(1H)-one (8b):



White solid; mp 191-192 °C; $[\alpha]_D^{20} = 65^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.16 (t, $^3J_{\text{H,H}} = 6.8$ Hz, 3H, OCH_2CH_3), 2.32 (s, 3H, CH_3), 4.07 (q, $^3J_{\text{H,H}} = 6.8$ Hz, 2H, OCH_2CH_3), 5.39 (d, $^3J_{\text{H,H}} = 2.4$ Hz, 1H, CH), 6.29 (s, 1H, NH), 6.99-7.29 (m, 4H, Ph), 8.69 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ 14.16,

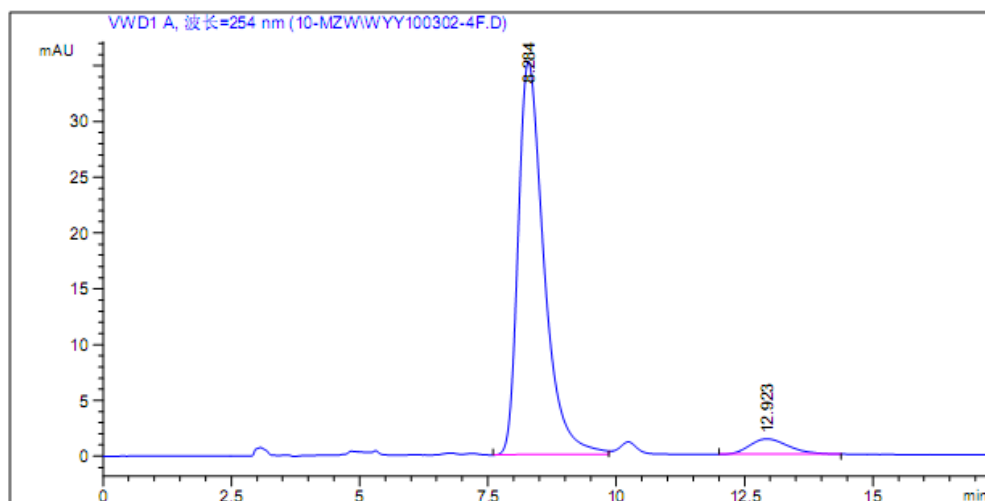
18.57, 54.92, 60.11, 101.22, 115.52, 128.30, 139.65, 146.44, 153.74, 161.07, 163.52, 165.55; ESI-MS: 277.07 ($[\text{M-H}]^-$); The enantiomeric excess was determined to be 88% by HPLC with Chiralcel OD-H column (25 cm) at 254 nm; eluent: hexane: $i\text{PrOH}$ (85:15), flow rate = 1.0 mL/min, $t_r = 8.284$ min (major), $t_r = 12.923$ min (minor).





信号 1: VWD1 A, 波长=254 nm

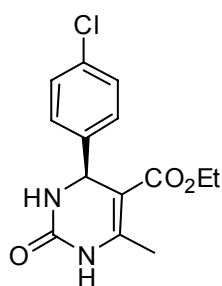
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2	11.871	BB	0.5864	1973.12341	51.54118	49.9591
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信号 1: VWD1 A, 波长=254 nm

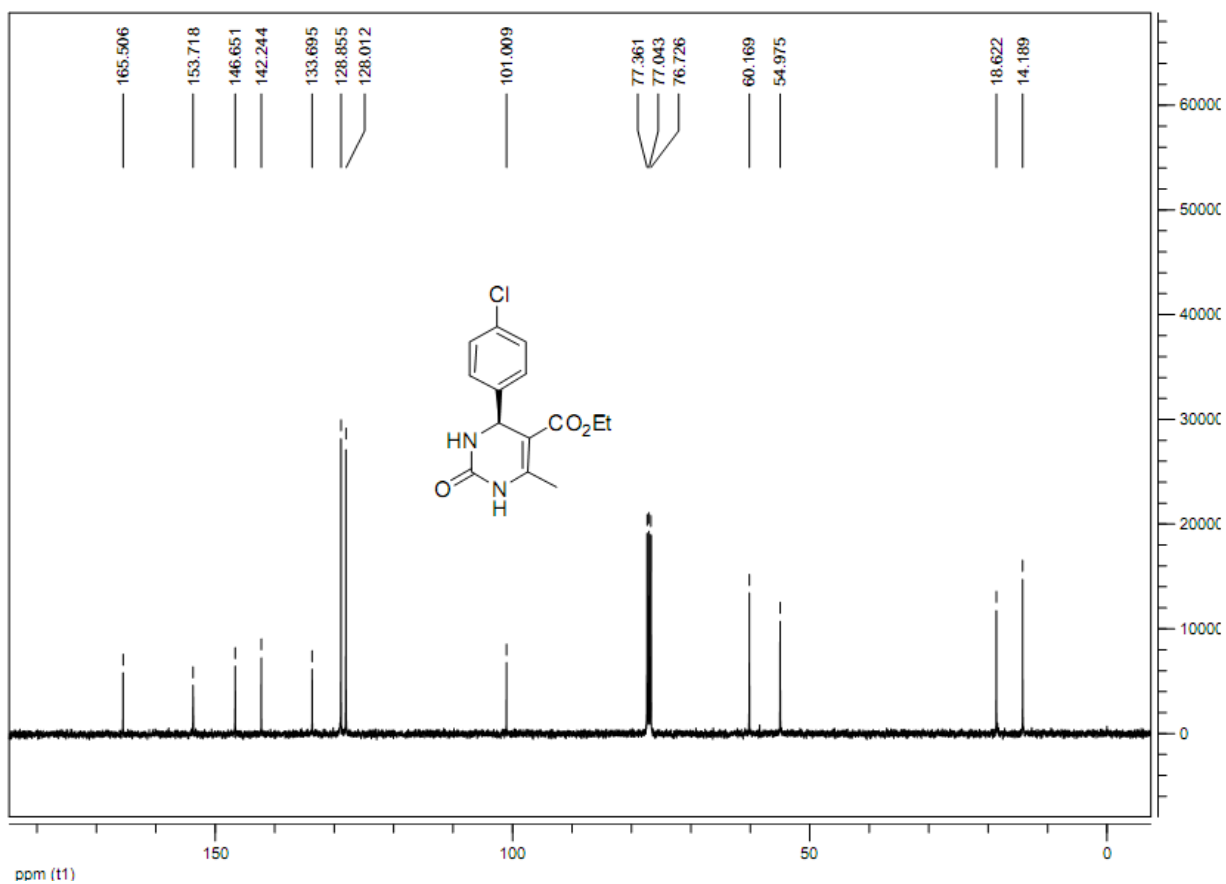
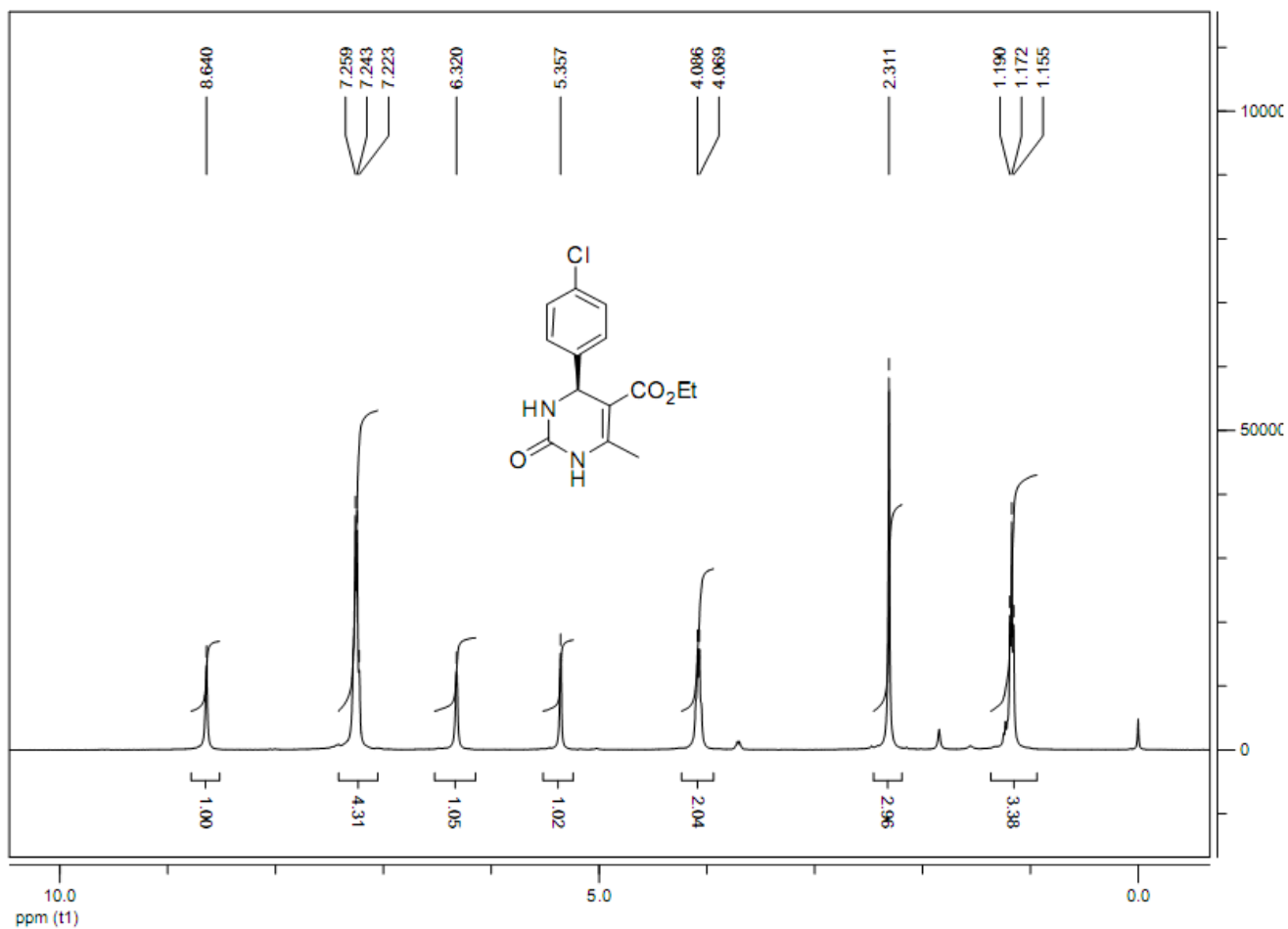
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1	8.284	VV	0.5293	1229.63232	35.24976	94.2333
2	12.923	BB	0.6611	75.24824	1.38917	5.7667
总量 :				1304.88056	36.63893	

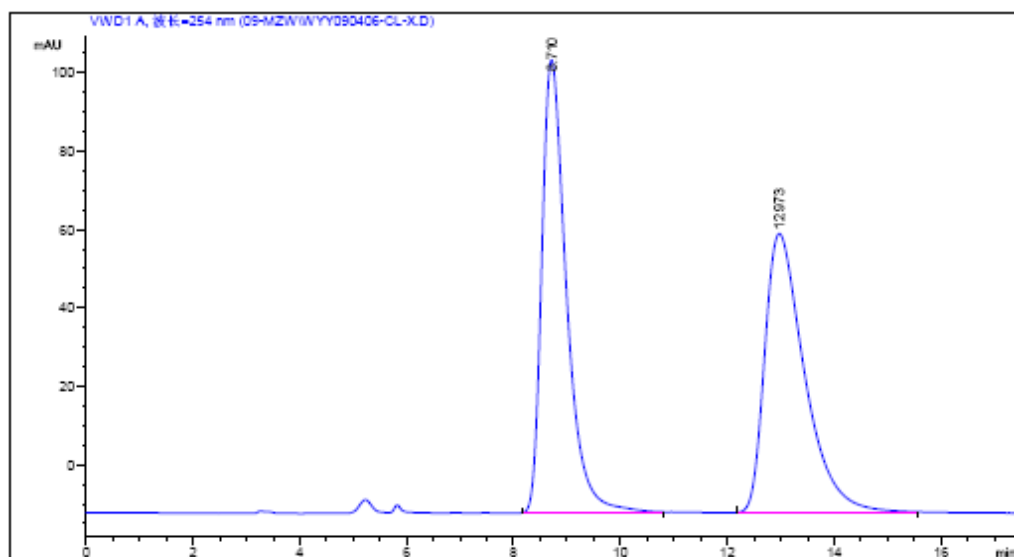
(S)-(+)-5-Ethoxycarbonyl-6-methyl-4-(4-chlorophenyl)-3,4-dihydropyrimidin-2(1H)-one (8c):



White solid; mp 210-212 °C; $[\alpha]_D^{20} = 37^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.17 (t, $^3J_{\text{H,H}} = 6.8$ Hz, 3H, OCH_2CH_3), 2.31 (s, 3H, CH_3), 4.09 (q, $^3J_{\text{H,H}} = 6.8$ Hz, 2H, OCH_2CH_3), 5.36 (s, 1H, CH), 6.32 (s, 1H, NH), 7.22-7.30 (m, 4H, Ph), 8.64 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ 14.19, 18.62, 54.98, 60.19, 101.10,

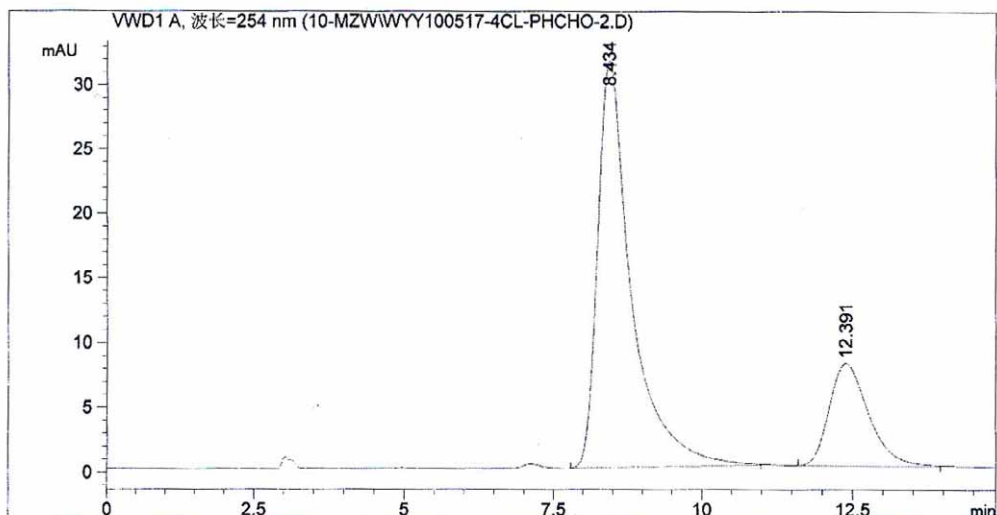
128.01, 128.87, 133.70, 142.24, 146.65, 153.72, 165.51; ESI-MS: 293.00 ($[\text{M-H}]^-$); The enantiomeric excess was determined to be 55% by HPLC with Chiralcel OD-H column (25 cm) at 254 nm; eluent: hexane: i -PrOH (85:15), flow rate = 1.0 mL/min, $t_r = 8.405$ min (major), $t_r = 13.303$ min (minor).





信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU * s	峰高 [mAU]	峰面积 %
1	8.710	BB	0.4806	3712.99561	115.27662	50.1381
2	12.973	BB	0.7805	3692.52442	71.11437	49.8619
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面积百分比报告

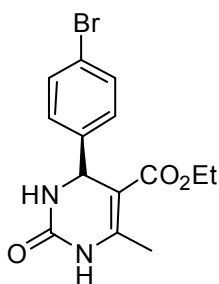
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 内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=254 nm

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1	8.434	BB	0.5761	1248.09851	31.48043	77.7798
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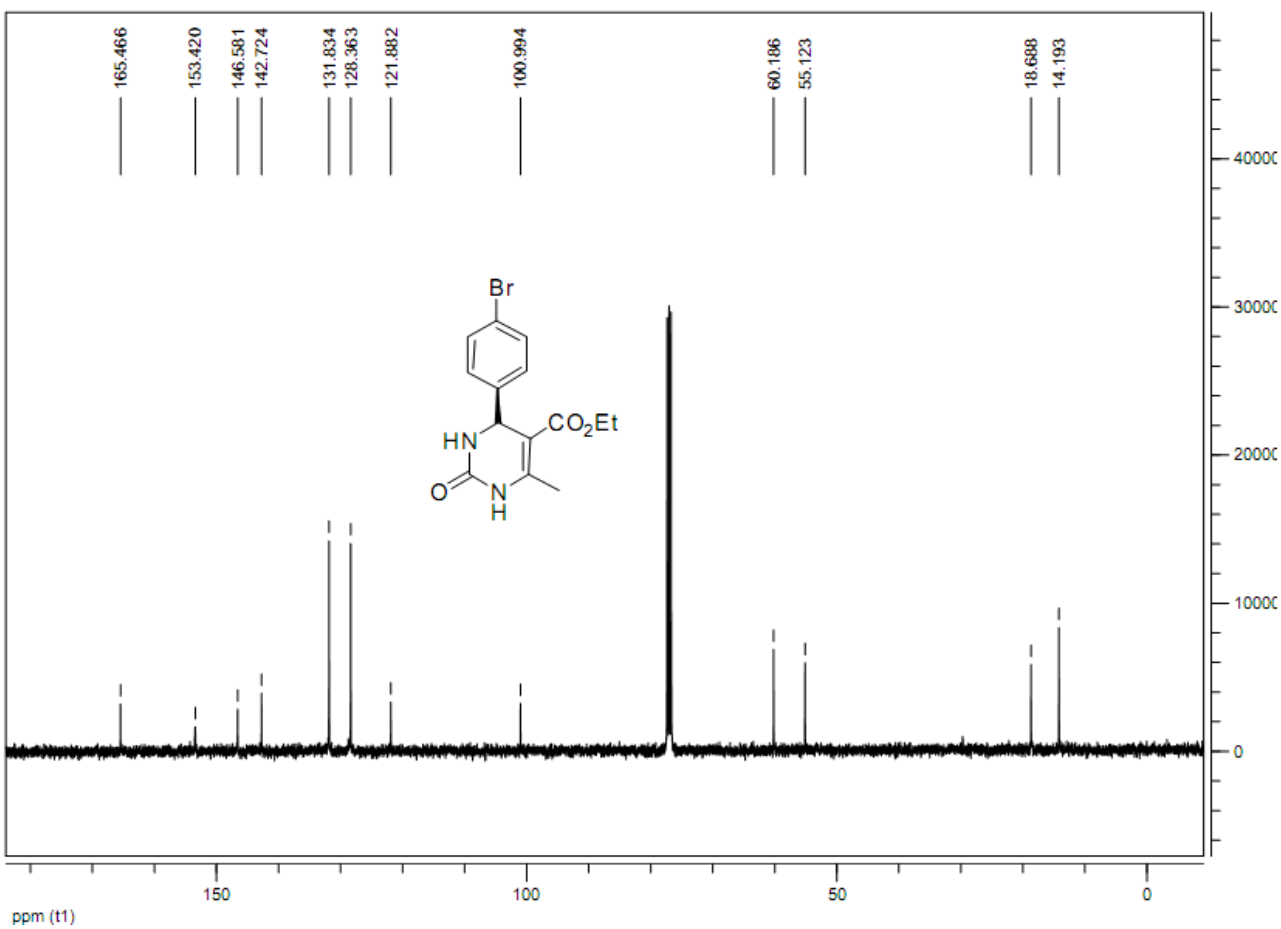
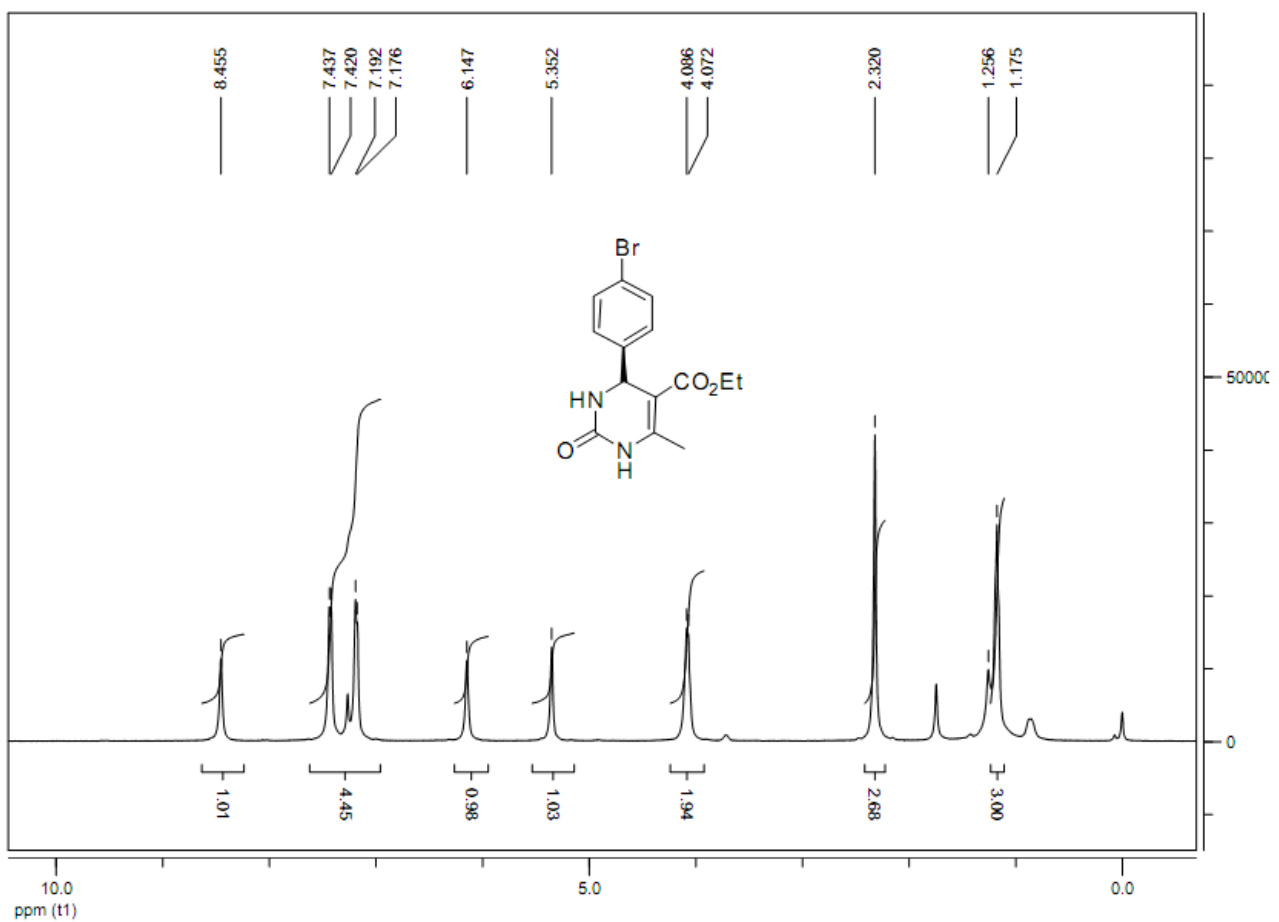
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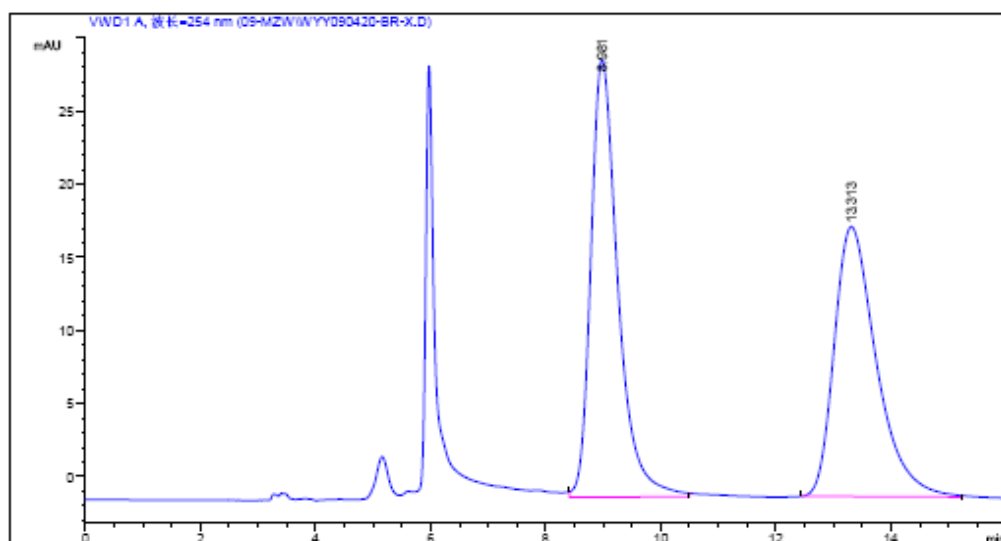
(S)-(+)-5-Ethoxycarbonyl-6-methyl-4-(4-bromophenyl)-3,4-dihydropyrimidin-2(1H)-one (8d):



White solid; mp 209-210 °C; $[\alpha]_D^{20} = 38^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.17 (t, $^3J_{\text{H,H}} = 7.2$ Hz, 3H, OCH_2CH_3), 2.33 (s, 3H, CH_3), 4.09 (q, $^3J_{\text{H,H}} = 7.2$ Hz, 2H, OCH_2CH_3), 5.35 (d, $^3J_{\text{H,H}} = 2.8$ Hz, 1H, CH), 6.15 (s, 1H, NH), 7.18-7.44 (m, 4H, Ph), 8.46 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ 14.19,

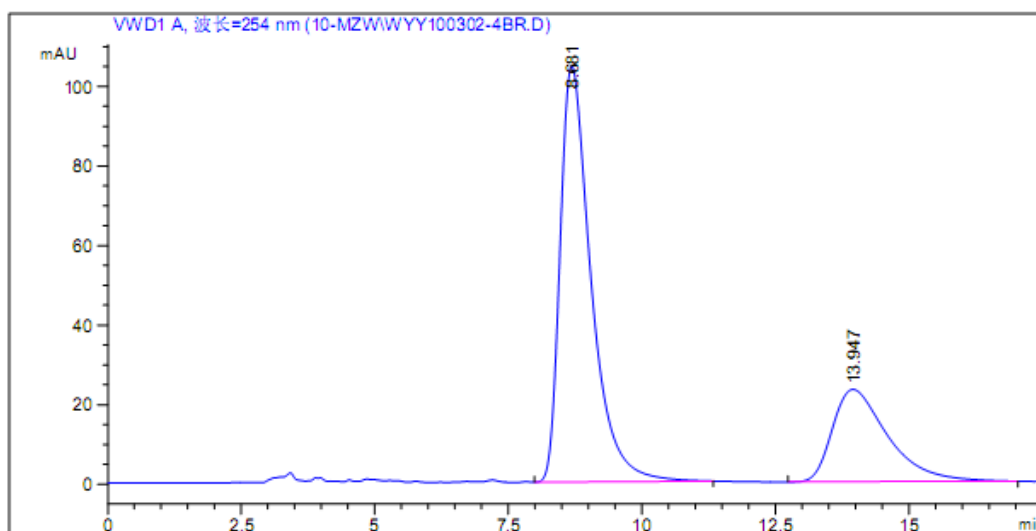
18.69, 55.12, 60.19, 100.99, 121.88, 128.38, 131.83, 142.72, 146.58, 153.42, 153.48, 165.47; ESI-MS: 336.93 ([M-H]); The enantiomeric excess was determined to be 44% by HPLC with Chiralcel OD-H column (25 cm) at 254 nm; eluent: hexane: $^i\text{PrOH}$ (85:15), flow rate = 1.0 mL/min, $t_r = 8.681$ min (major), $t_r = 13.947$ min (minor).





信号 1: VWD1 A, 波长=254 nm

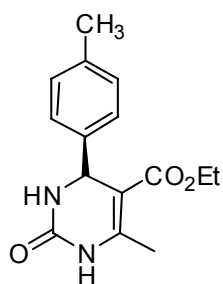
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1	8.981	BB	0.4998	981.81927	29.88554	51.1837
2	13.313	BB	0.7786	936.40894	18.44654	48.8163
总量 :				1918.22821	48.33208	



信号 1: VWD1 A, 波长=254 nm

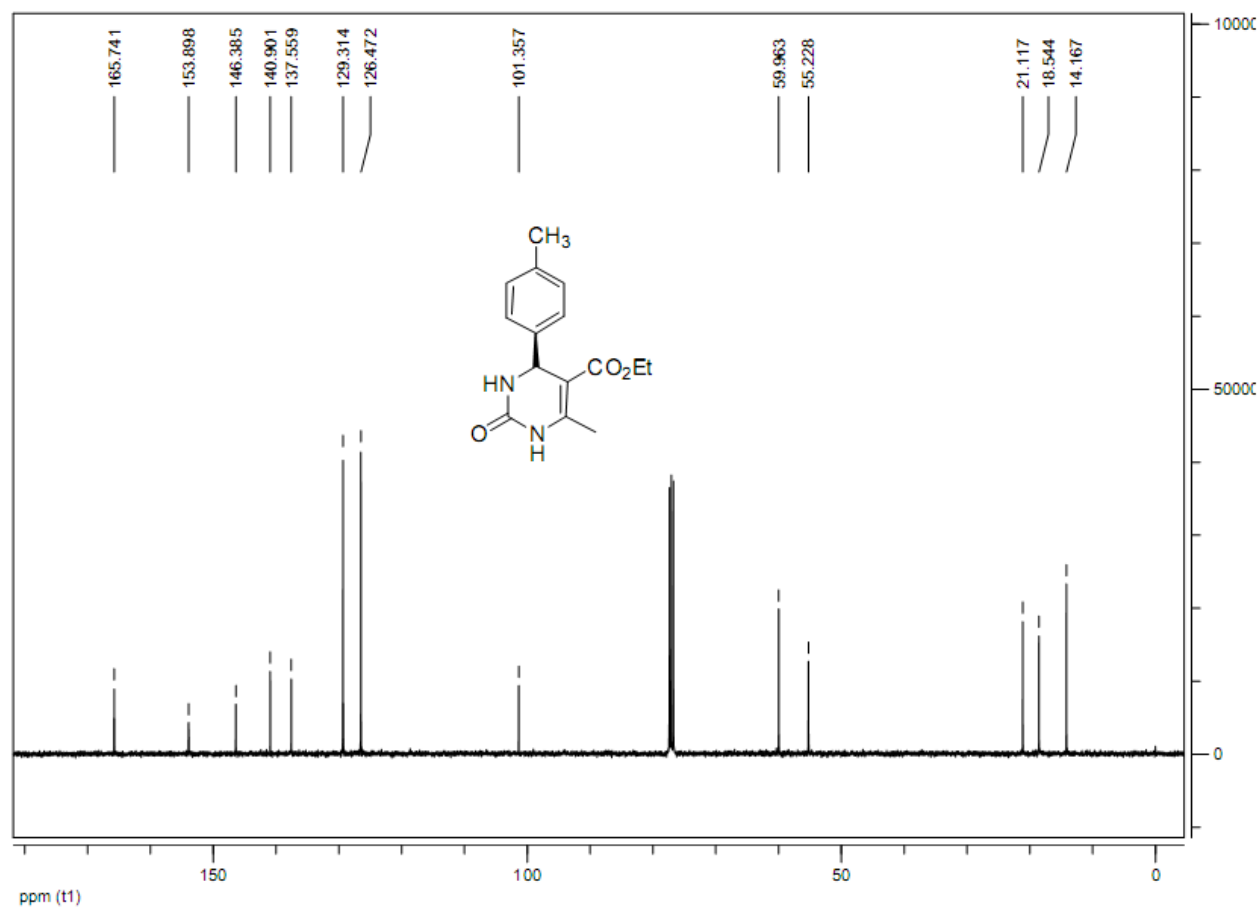
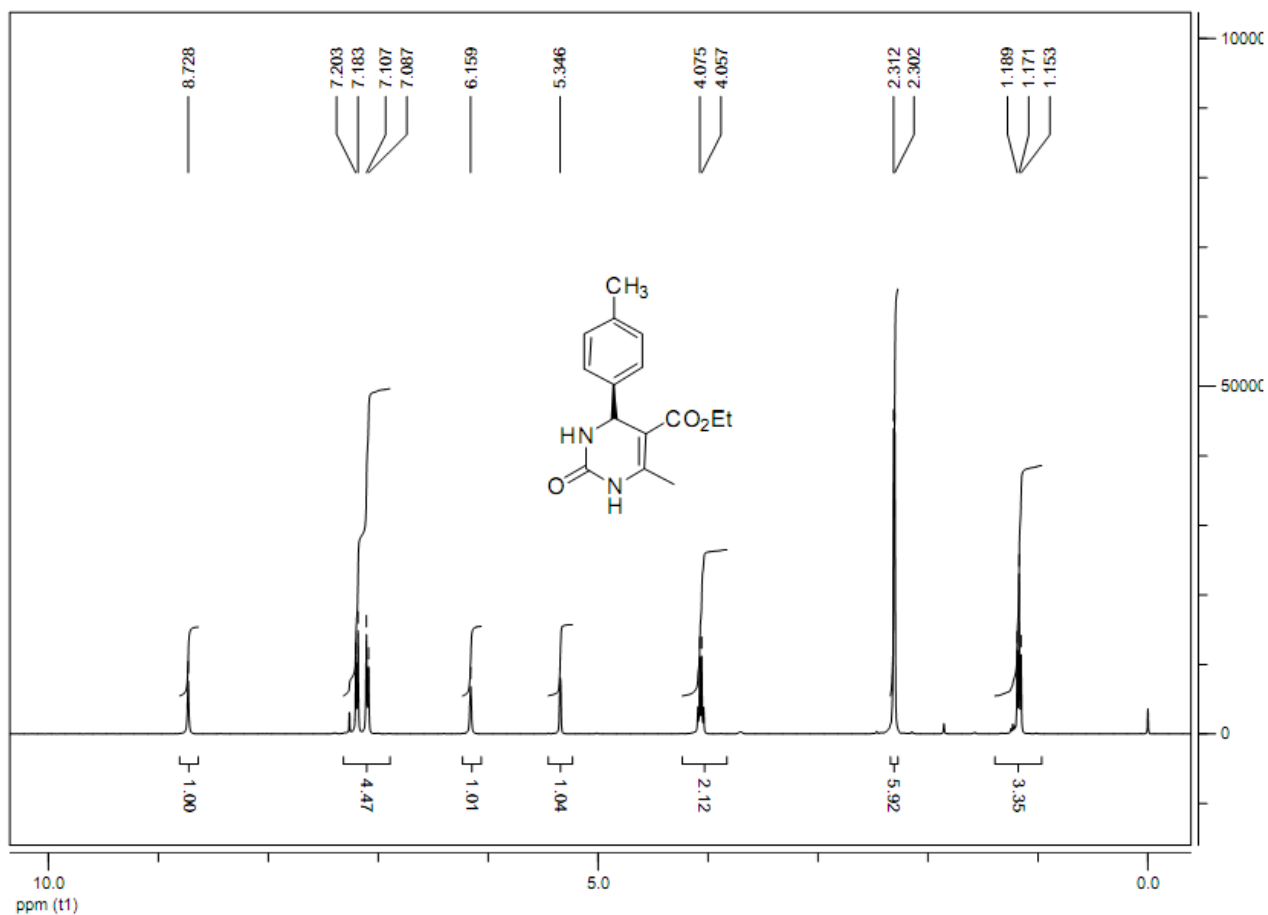
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1	8.681	VB	0.6072	4224.18750	104.77307	71.4793
2	13.947	BB	1.0956	1685.48035	23.26661	28.5207
总量 :				5909.66785	128.03969	

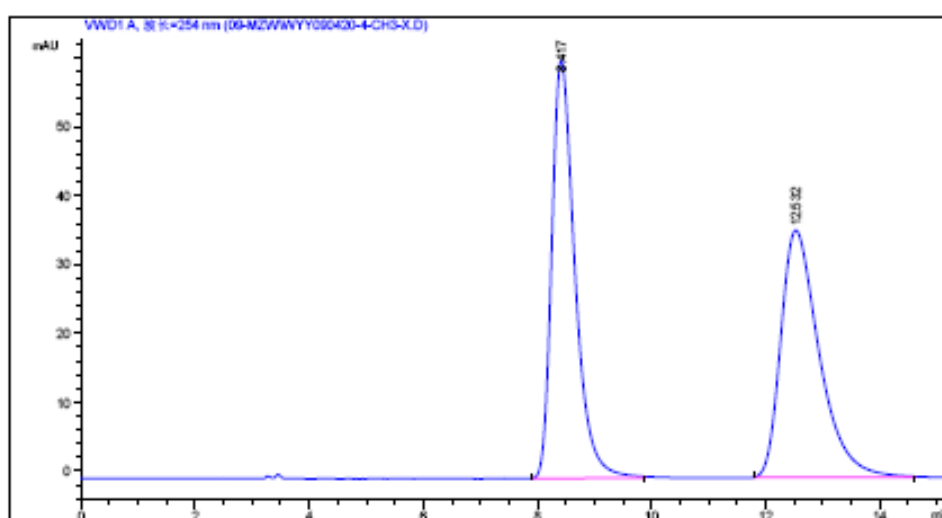
(S)-(+)-5-Ethoxycarbonyl-6-methyl-4-(4-methylphenyl)-3,4-dihydropyrimidin-2(1H)-one (8e):



White solid; mp 213-214°C; $[\alpha]_D^{20} = 56^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.17 (t, $^3J_{\text{H,H}} = 7.2$ Hz, 3H, OCH_2CH_3), 2.30 (s, 3H, CH_3), 2.31 (s, 3H, CH_3), 4.07 (q, $^3J_{\text{H,H}} = 7.2$ Hz, 2H, OCH_2CH_3), 5.35 (d, $^3J_{\text{H,H}} = 2.8$ Hz, 1H, CH), 6.16 (s, 1H, NH), 7.09-7.26 (m, 4H, Ph), 8.73 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ

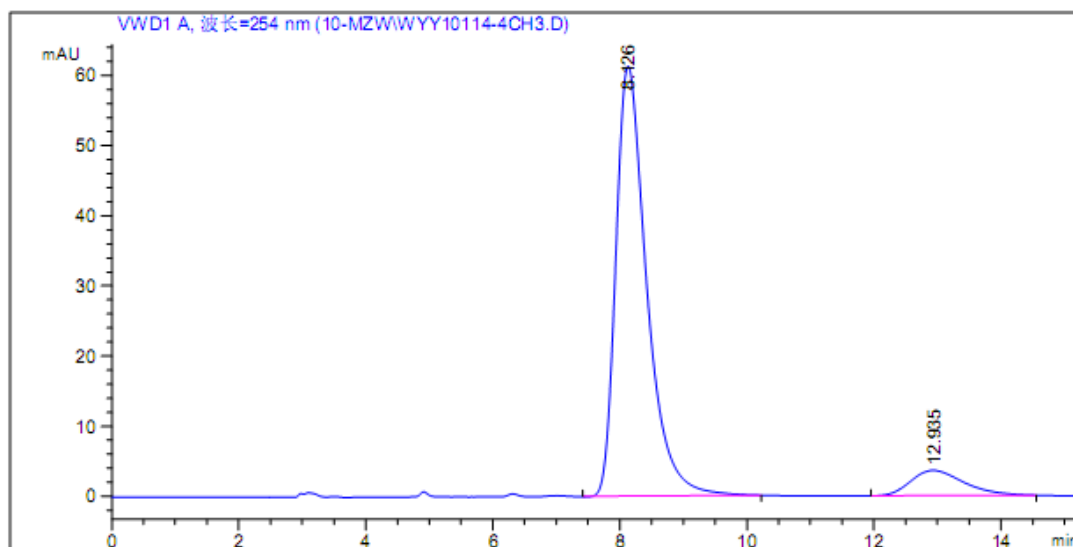
14.17, 18.54, 21.12, 55.23, 59.93, 101.36, 126.47, 129.31, 137.59, 140.90, 146.39, 153.90, 165.74; ESI-MS: 273.07 ($[\text{M-H}]^-$); The enantiomeric excess was determined to be 82% by HPLC with Chiralcel OD-H column (25 cm) at 254 nm; eluent: hexane: $^i\text{PrOH}$ (85:15), flow rate = 1.0 mL/min, $t_r = 8.126$ min (major), $t_r = 12.935$ min (minor).





信号 1: VWD1 A, 波长=254 nm

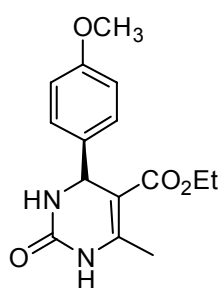
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1	8.417	BB	0.4338	1711.18677		60.67921	50.3982
2	12.532	BB	0.7101	1684.14966		35.91995	49.6018
总量 :				3395.33643		96.59916	



信号 1: VWD1 A, 波长=254 nm

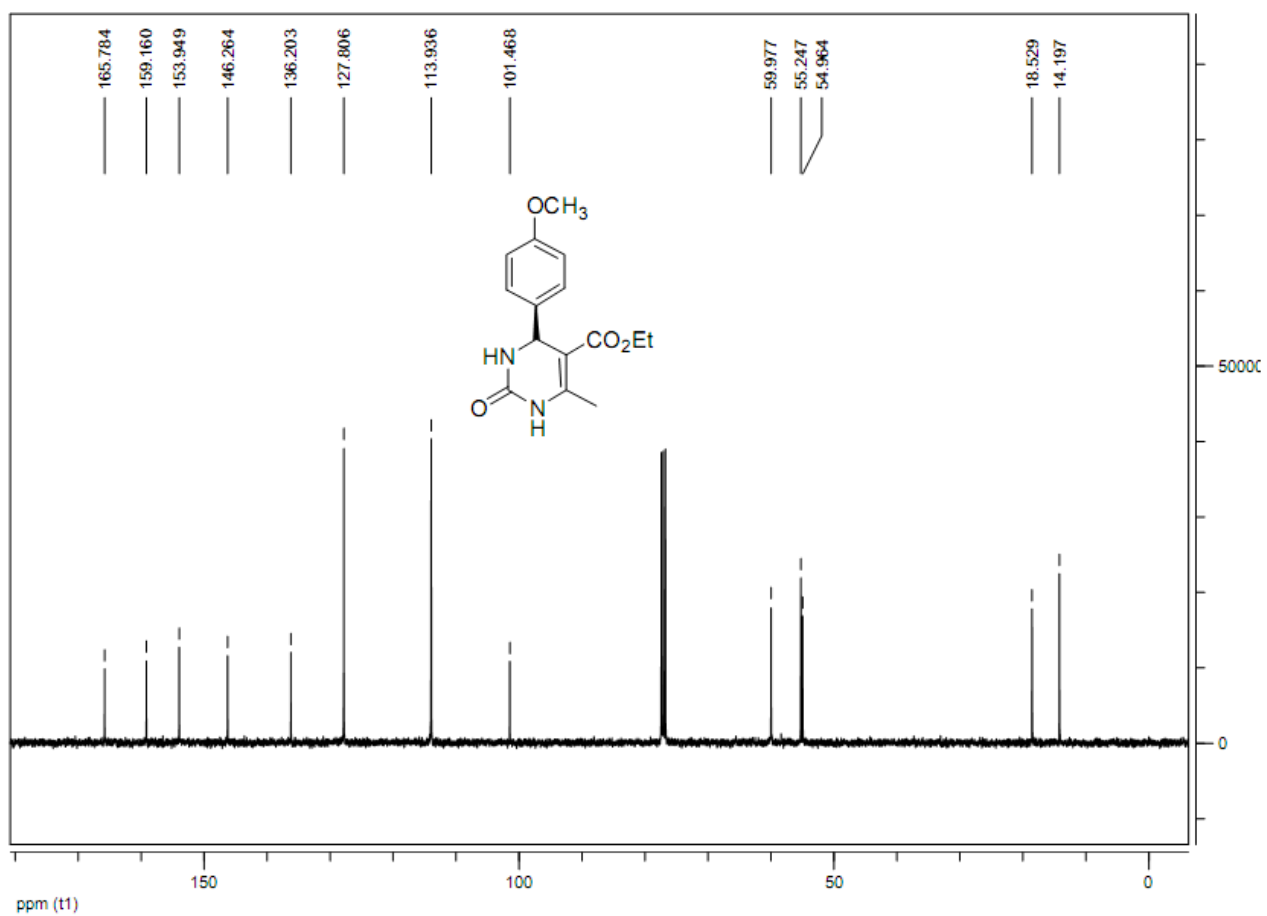
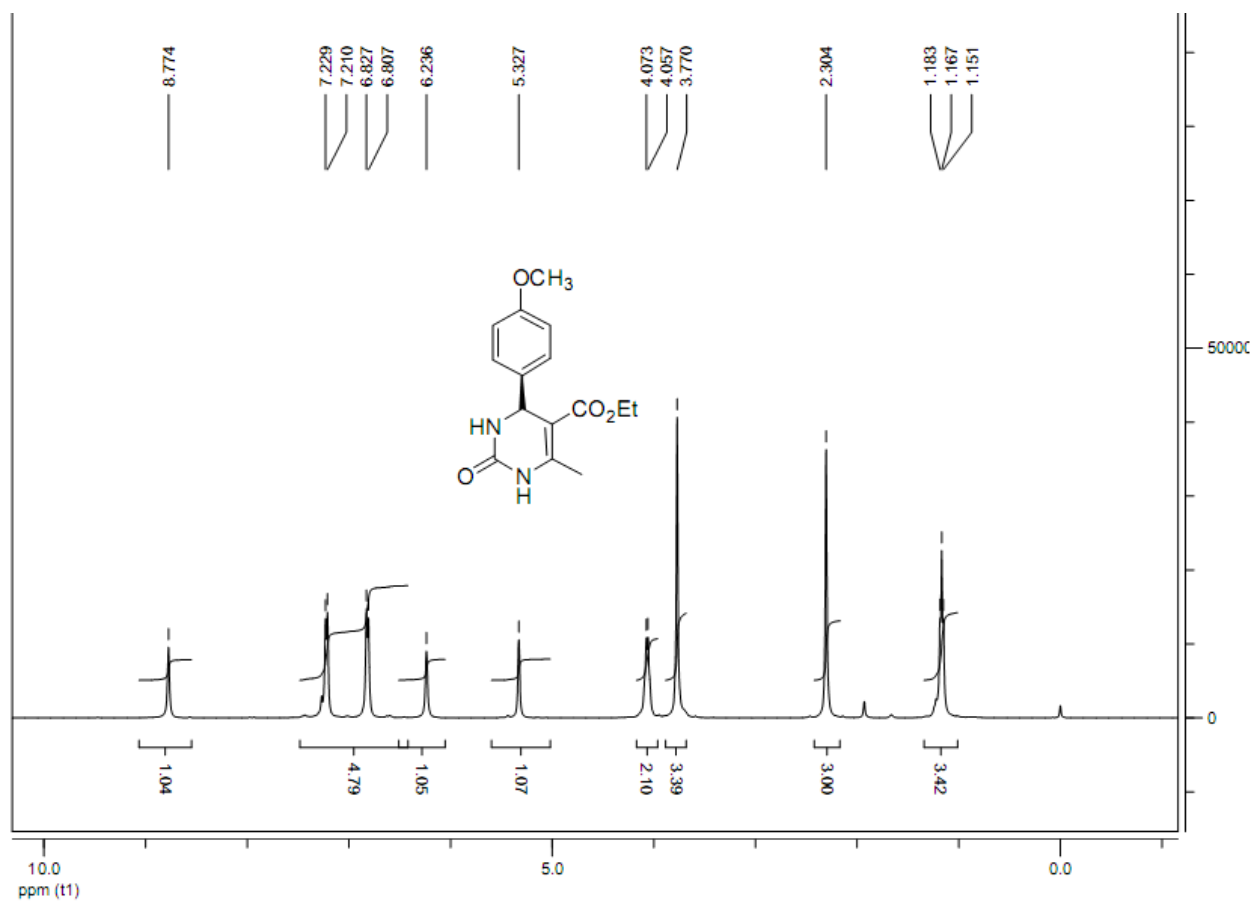
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1	8.126	VB	0.5113	2075.77246	61.34453	90.6571
2	12.935	BB	0.8529	213.92456	3.61823	9.3429
总量 :				2289.69702	64.96275	

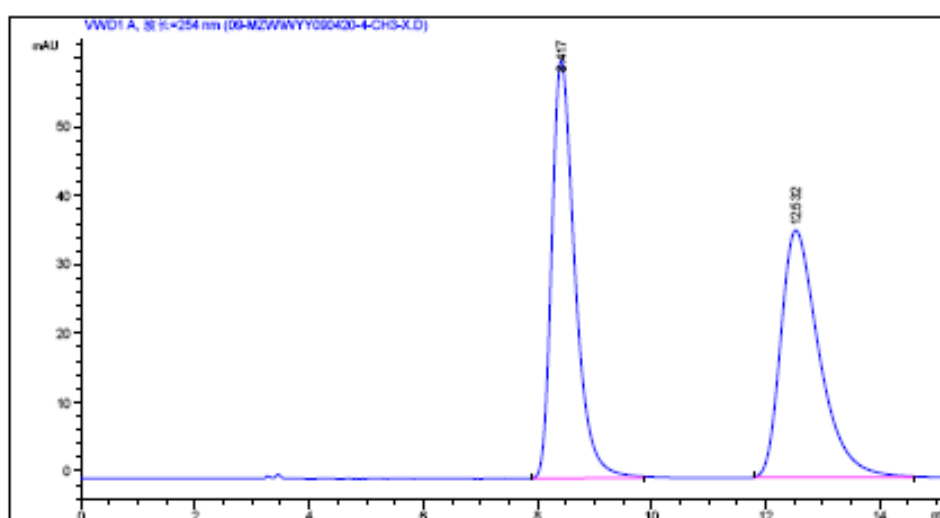
(S)-(+)-5-Ethoxycarbonyl-6-methyl-4-(4-methoxyphenyl)-3,4-dihydropyrimidin-2(1H)-one (8f):



White solid; mp 202-205 °C; $[\alpha]_D^{20} = 53^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.17 (t, $^3J_{\text{H,H}} = 7.2$ Hz, 3H, OCH_2CH_3), 2.30 (s, 3H, CH_3), 3.77 (s, 3H, OCH_3), 4.07 (q, $^3J_{\text{H,H}} = 7.2$ Hz, 2H, OCH_2CH_3), 5.33 (d, $^3J_{\text{H,H}} = 2.4$ Hz, 1H, CH), 6.24 (s, 1H, NH), 6.81-7.23 (m, 4H, Ph), 8.77 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz,

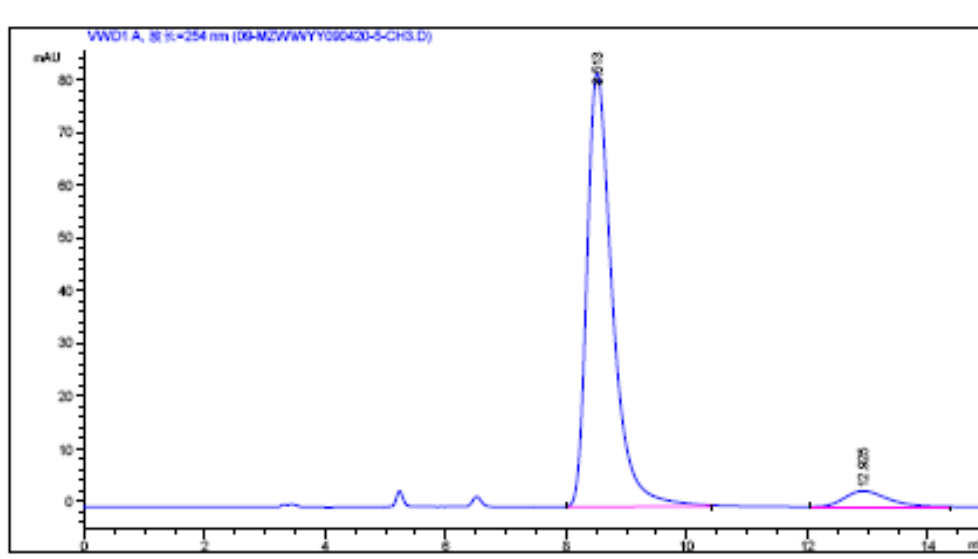
CDCl_3): δ 14.20, 18.53, 54.96, 55.25, 59.98, 101.47, 101.47, 113.94, 127.81, 136.20, 146.26, 153.95, 159.16, 165.78; ESI-MS: 289.07 ($[\text{M-H}]^-$); The enantiomeric excess was determined to be 89% by HPLC with Chiralcel OD-H column (25 cm) at 254 nm; eluent: hexane: $^i\text{PrOH}$ (85:15), flow rate = 1.0 mL/min, tr = 8.513 min (major), tr = 12.925 min





信号 1: VWD1 A, 波长=254 nm

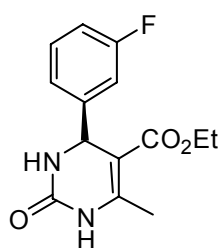
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1	8.417	BB	0.4338	1711.18677		60.67921	50.3982
2	12.532	BB	0.7101	1684.14966		35.91995	49.6018
总量 :				3395.33643		96.59916	



信号 1: VWD1 A, 波长=254 nm

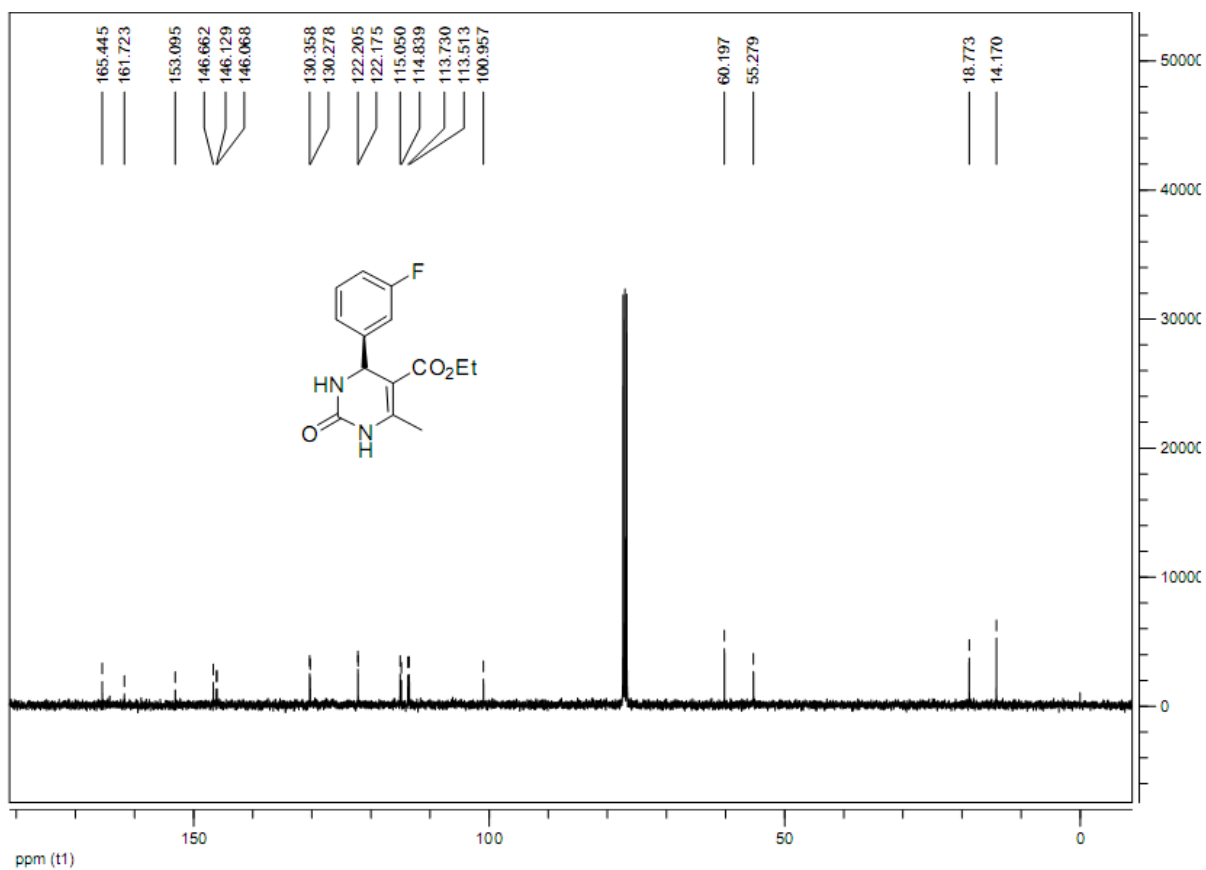
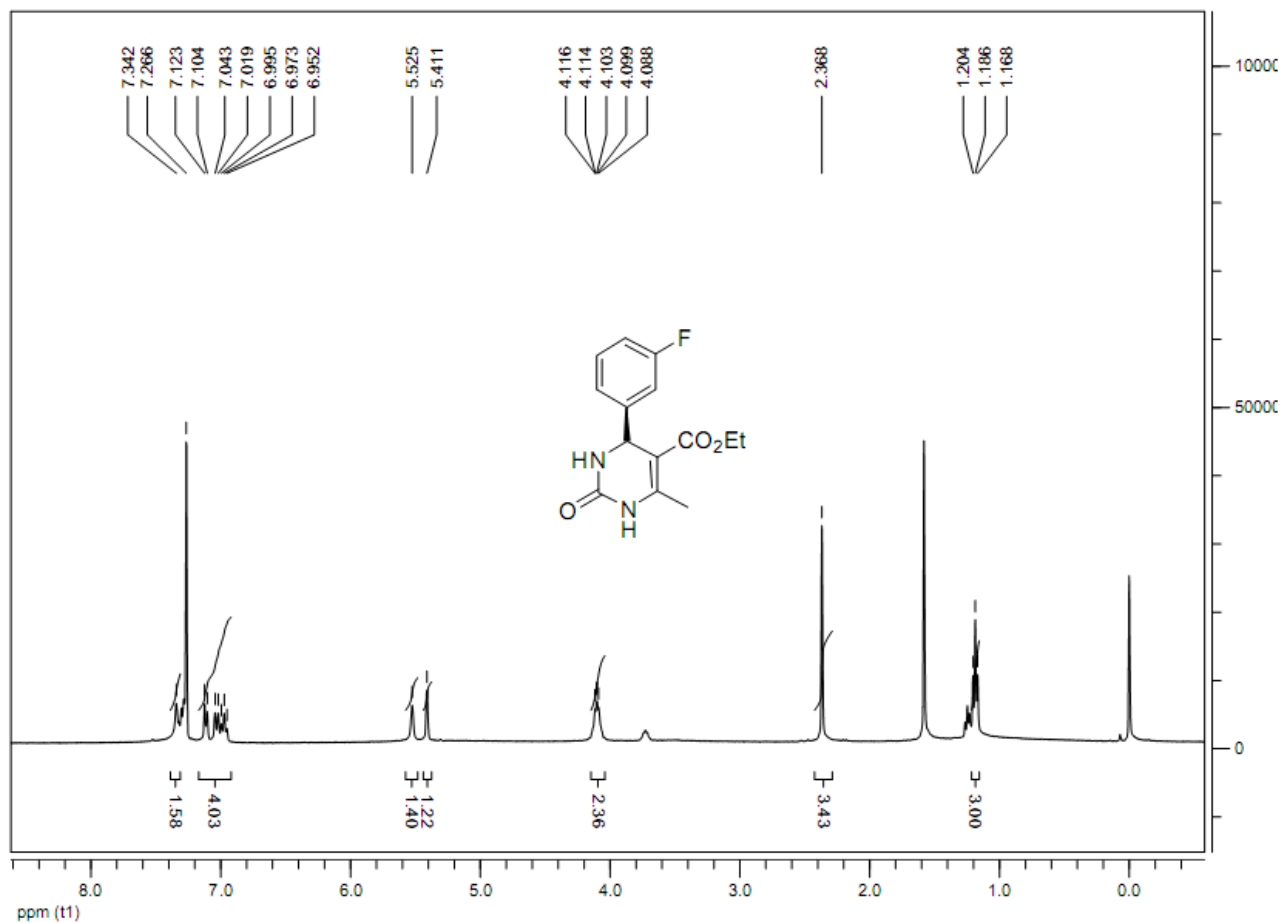
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU %	峰高 [mAU]	峰面积 %
1	8.513	BB	0.4482	2416.12036	82.08931	94.1423
2	12.925	BB	0.7570	150.33621	3.01221	5.8577
总量 :				2566.45657	85.10152	

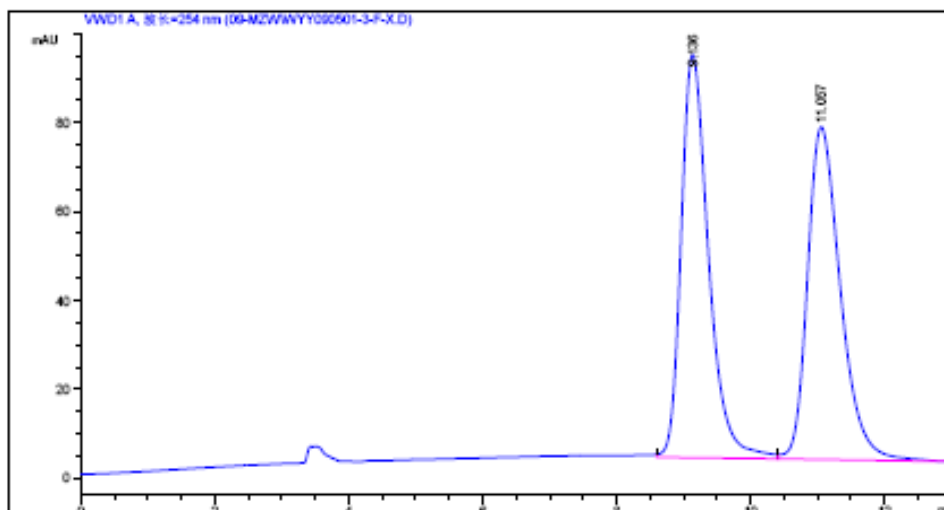
(S)-(+)-5-Ethoxycarbonyl-6-methyl-4-(3-fluorophenyl)-3,4-dihydropyrimidin-2(1H)-one (8g):



White solid; mp 212-213 °C; $[\alpha]_D^{20} = 58^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.18 (t, $^3J_{\text{H,H}} = 7.2$ Hz, 3H, OCH_2CH_3), 2.37 (s, 3H, CH_3), 4.10 (q, $^3J_{\text{H,H}} = 7.2$ Hz, 2H, OCH_2CH_3), 5.41 (s, 1H, CH), 5.53 (s, 1H, NH), 6.95-7.27 (m, 4H, Ph), 7.34(s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ 14.17, 18.77, 55.28, 60.20, 100.96,

113.61, 114.92, 122.19, 130.30, 146.09, 146.67, 153.10, 161.72, 165.45; ESI-MS: 277.07 ($[\text{M-H}]^-$); The enantiomeric excess was determined to be 87% by HPLC with Chiralpak AS-H column (25 cm) at 254 nm; eluent: hexane: $i\text{-PrOH}$ (70:30), flow rate = 1.0 mL/min, $t_r = 9.503$ min (major), $t_r = 11.811$ min (minor).

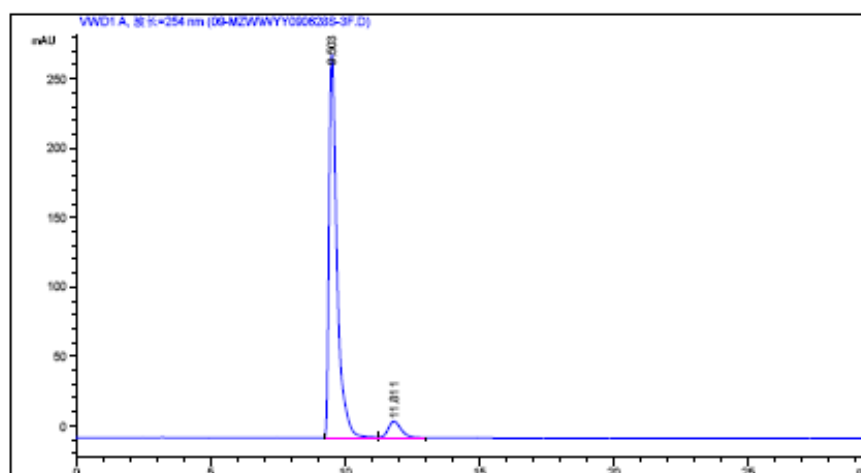




信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	9.136	BV	0.4378	2582.80127	90.49676	50.1704
2	11.057	VBA	0.5164	2565.25342	74.83933	49.8296

总量 : 5148.05469 165.33609

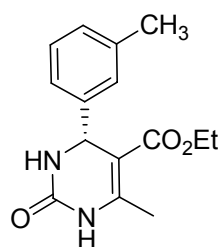


信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	9.503	BB	0.3119	5739.23096	276.62656	93.3116
2	11.811	BB	0.5295	411.37854	11.95657	6.6884

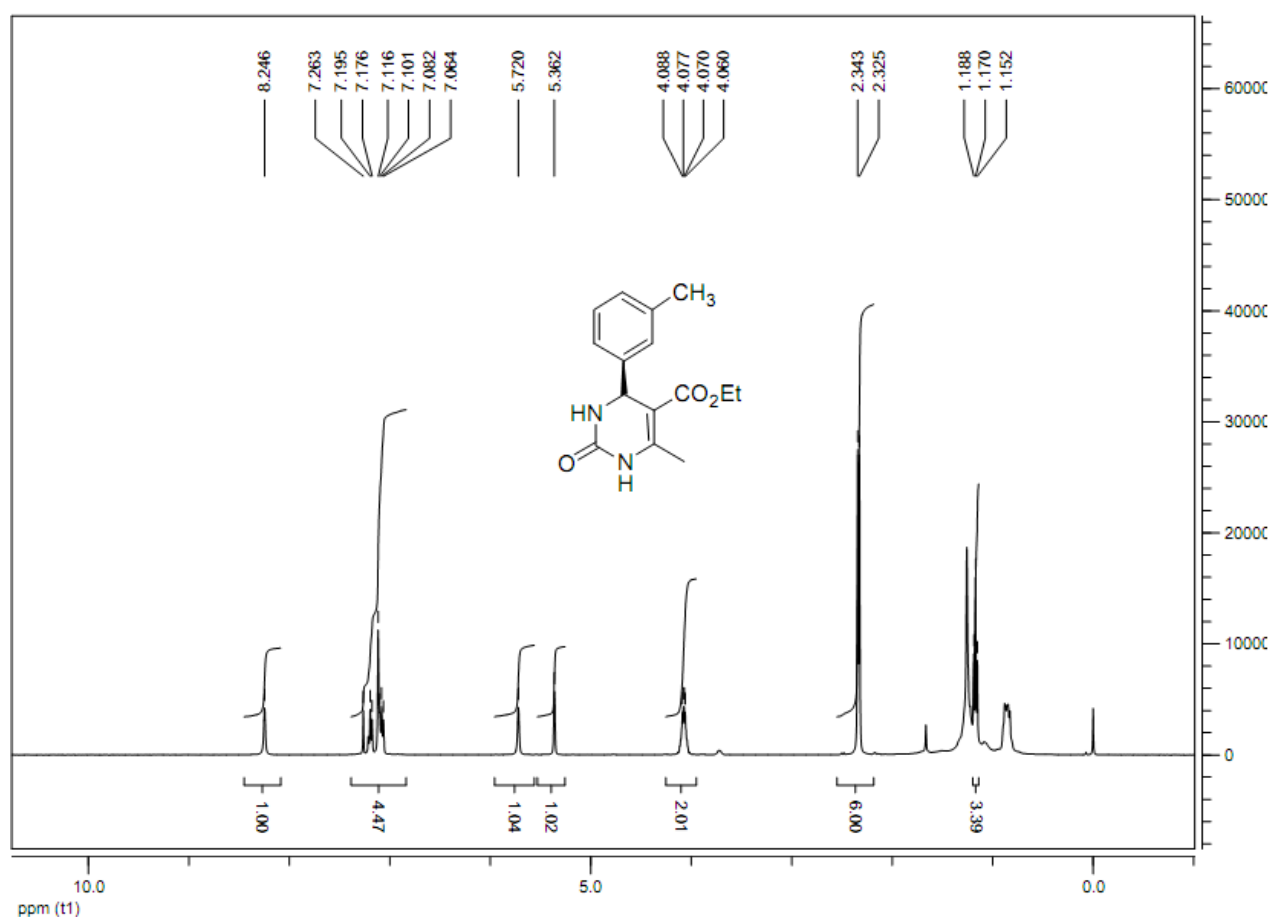
总量 : 6150.60950 288.58313

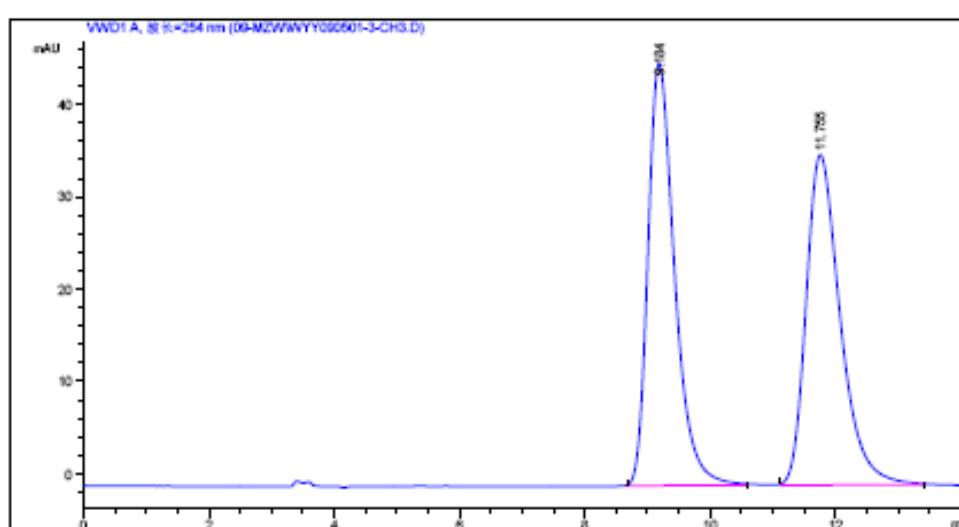
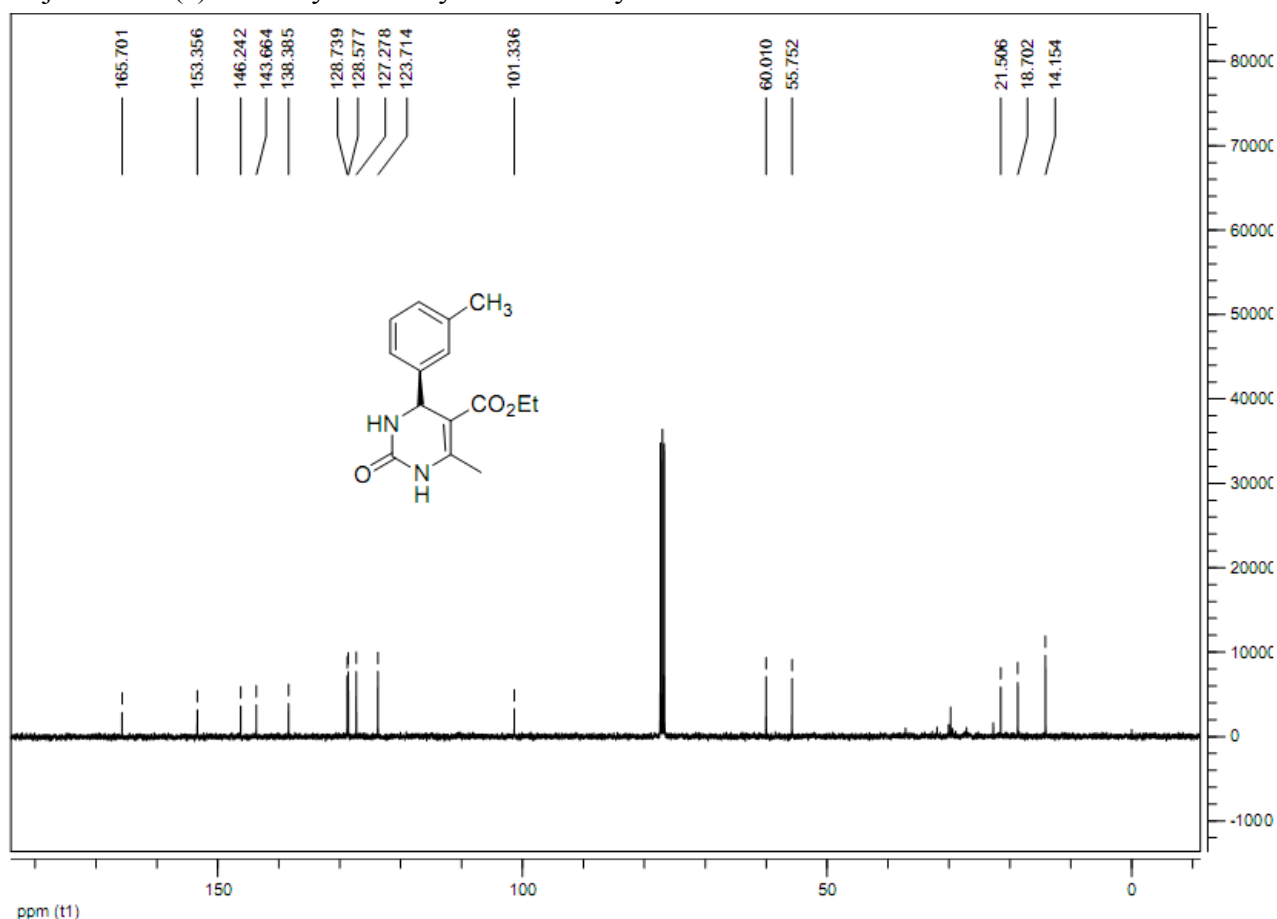
(S)-(+)-5-Ethoxycarbonyl-6-methyl-4-(3-methylphenyl)-3,4-dihydropyrimidin-2(1H)-one (8h):



White solid; mp 209-210 °C; $[\alpha]_D^{20} = 56^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.17 (t, $^3J_{H,H} = 6.8$ Hz, 3H, OCH_2CH_3), 2.33 (s, 3H, CH_3), 2.34 (s, 3H, CH_3), 4.07 (q, $^3J_{H,H} = 6.8$ Hz, 2H, OCH_2CH_3), 5.36 (s, 1H, NH), 5.72 (s, 1H, CH), 7.06-7.26 (m, 4H, Ph), 8.25 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, $[\text{D}_6]\text{DMSO}$): δ 14.15,

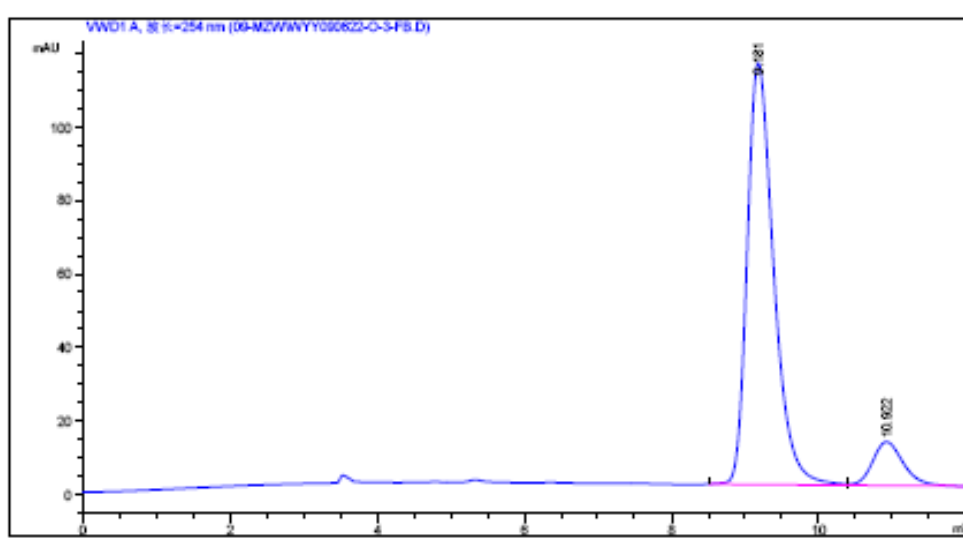
18.70, 21.51, 55.75, 60.01, 101.34, 123.71, 127.28, 128.58, 128.74, 138.39, 143.66, 146.24, 153.39, 165.70; ESI-MS: 273.13 ($[\text{M-H}]^-$); The enantiomeric excess was determined to be 80% by HPLC with Chiralcel OD-H column (25 cm) at 254 nm; eluent: hexane: $^i\text{PrOH}$ (85:15), flow rate = 1.0 mL/min, $t_r = 9.181$ min (major), $t_r = 10.922$ min (minor).





信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU]	峰高 [mAU]	峰面积 %
1	9.184	BB	0.4512	1358.03381	45.74028	50.0463
2	11.755	BB	0.5814	1355.52246	35.80646	49.9537
总量 :				2713.55627	81.54674	

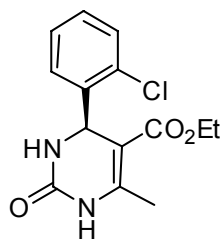


信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU	峰高 [mAU]	峰面积 %
1	9.181	BV	0.3865	2880.89209	114.57050	89.1361
2	10.922	VB	0.4484	351.12378	11.92386	10.8639

总量 : 3232.01587 126.49436

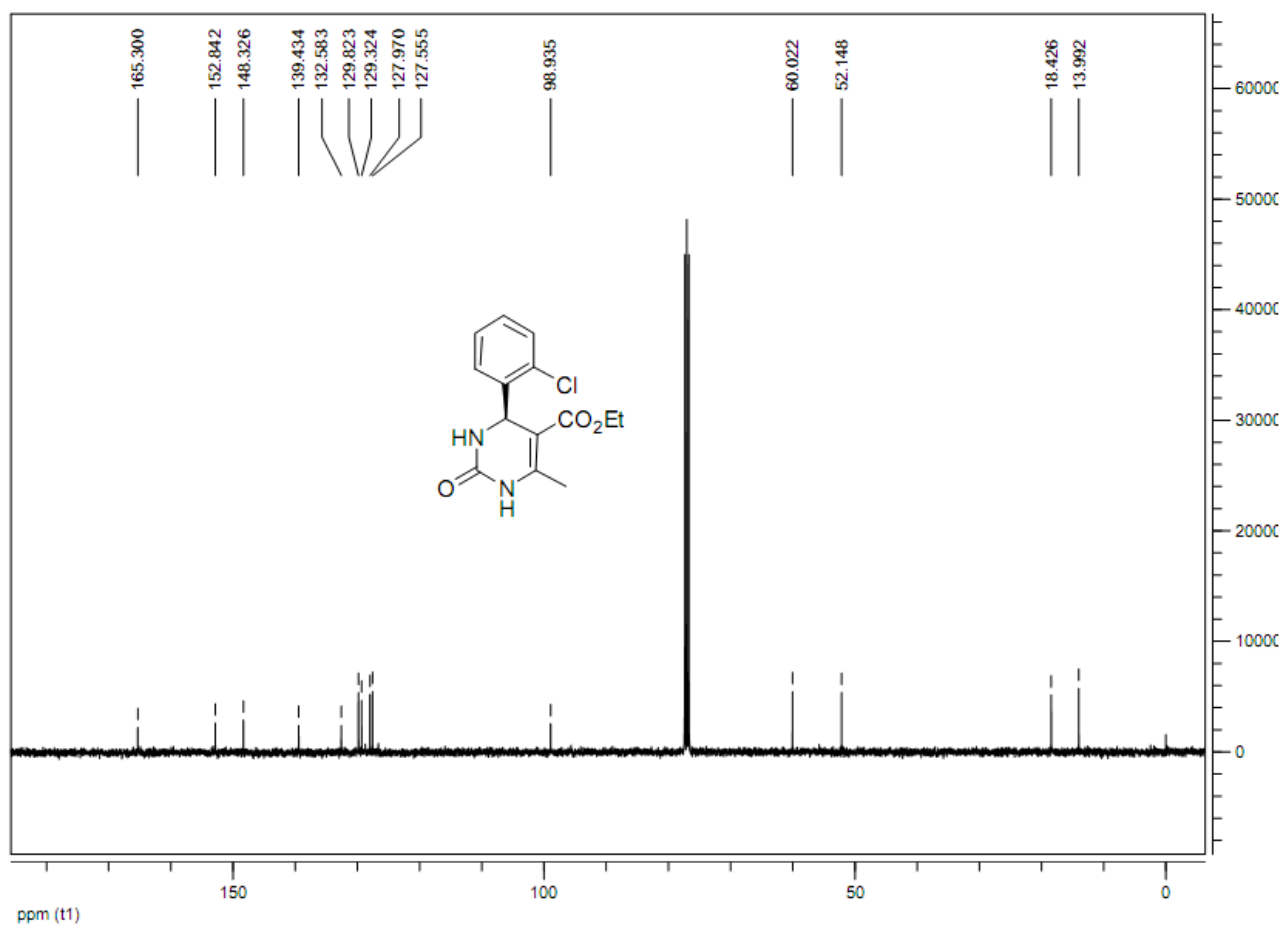
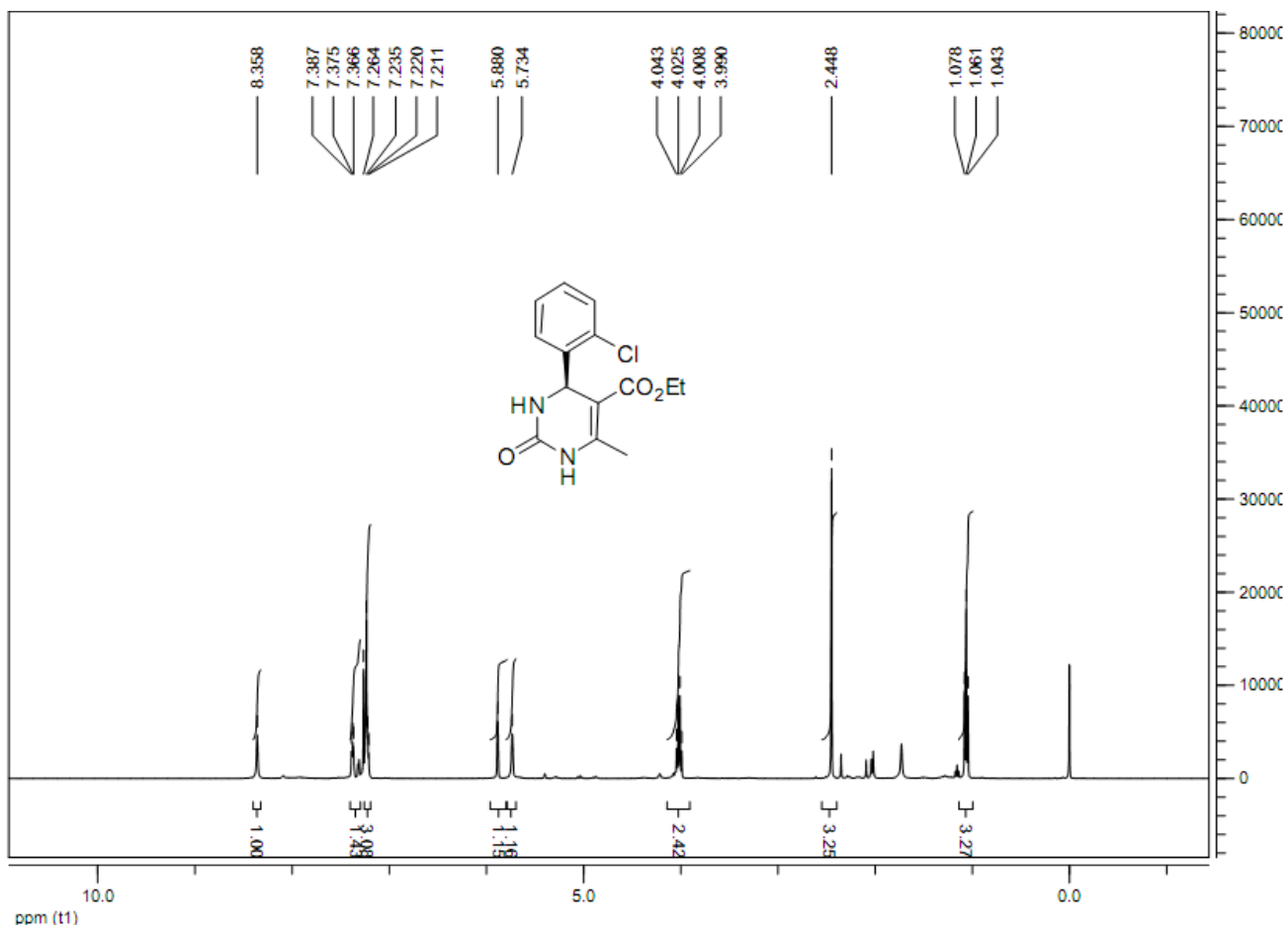
(S)-(+)-5-Ethoxycarbonyl-6-methyl-4-(2-chlorophenyl)-3,4-dihydropyrimidin-2(1H)-one (8i):

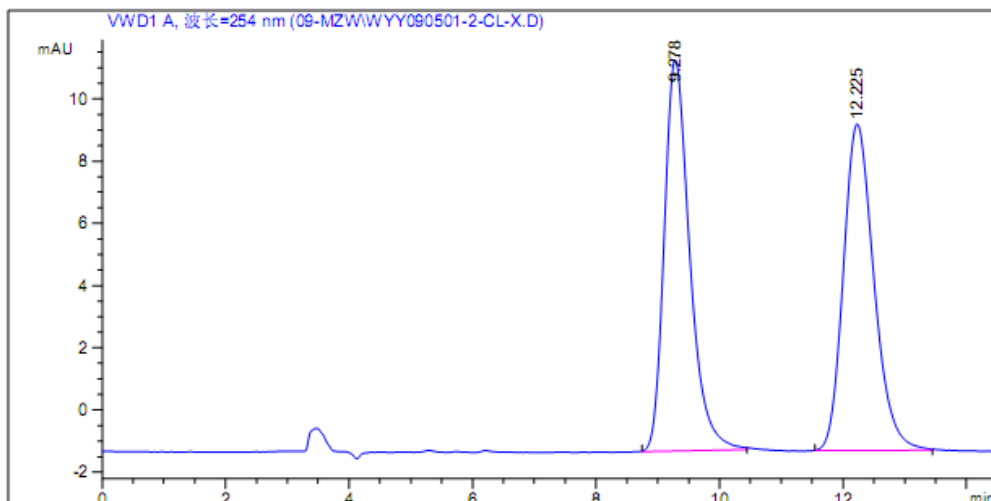


White solid; mp 228-230 °C; $[\alpha]_D^{20} = 46^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz, DMSO): δ 1.06 (t, $^3J_{H,H} = 6.8$ Hz, 3H, OCH_2CH_3), 2.45 (s, 3H, CH_3), 4.01 (q, $^3J_{H,H} = 6.8$ Hz, 2H, OCH_2CH_3), 5.73 (s, 1H, CH), 5.89 (s, 1H, NH), 7.21-7.39 (m, 4H, Ph), 8.36 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, $[\text{D}_6]\text{DMSO}$): δ 13.99, 18.43, 52.15, 60.02, 98.

94, 127.56, 127.98, 129.32, 129.82, 132.58, 139.43, 148.33, 152.84, 165.30; ESI-MS: 293.00 ($[\text{M}+\text{H}]^+$);

The enantiomeric excess was determined to be 81% by HPLC with Chiralcel OD-H column (25 cm) at 254 nm; eluent: hexane: $^i\text{PrOH}$ (85:15), flow rate = 1.0 mL/min, $t_r = 8.041$ min (major), $t_r = 11.244$ min (minor).

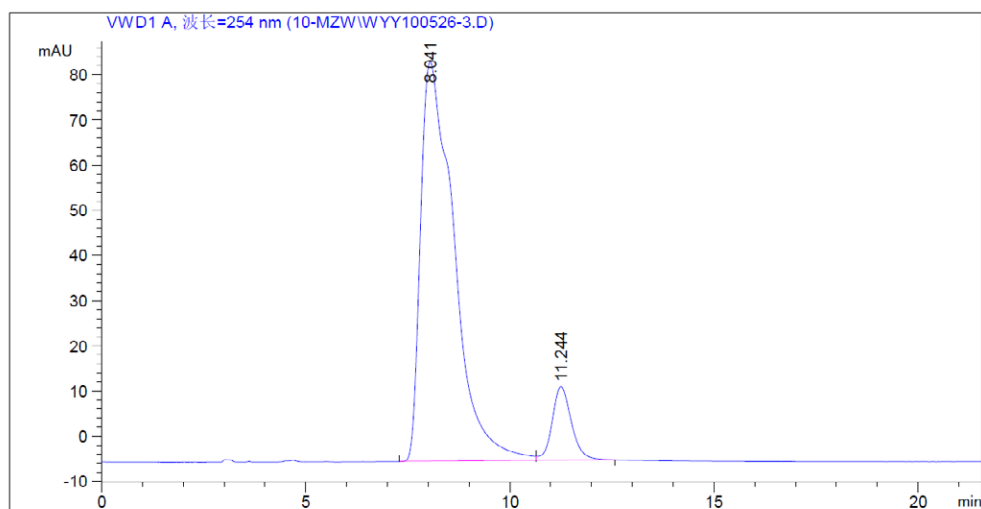




信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	9.278	BB	0.4396	360.80243	12.57282	49.9582
2	12.225	BB	0.5326	361.40637	10.49895	50.0418

总量 : 722.20880 23.07177

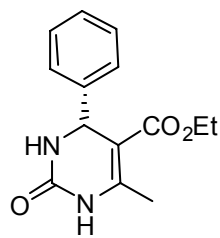


信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	8.041	BV	0.7886	5031.47412	88.48997	90.2858
2	11.244	VB	0.5033	541.35535	16.32931	9.7142

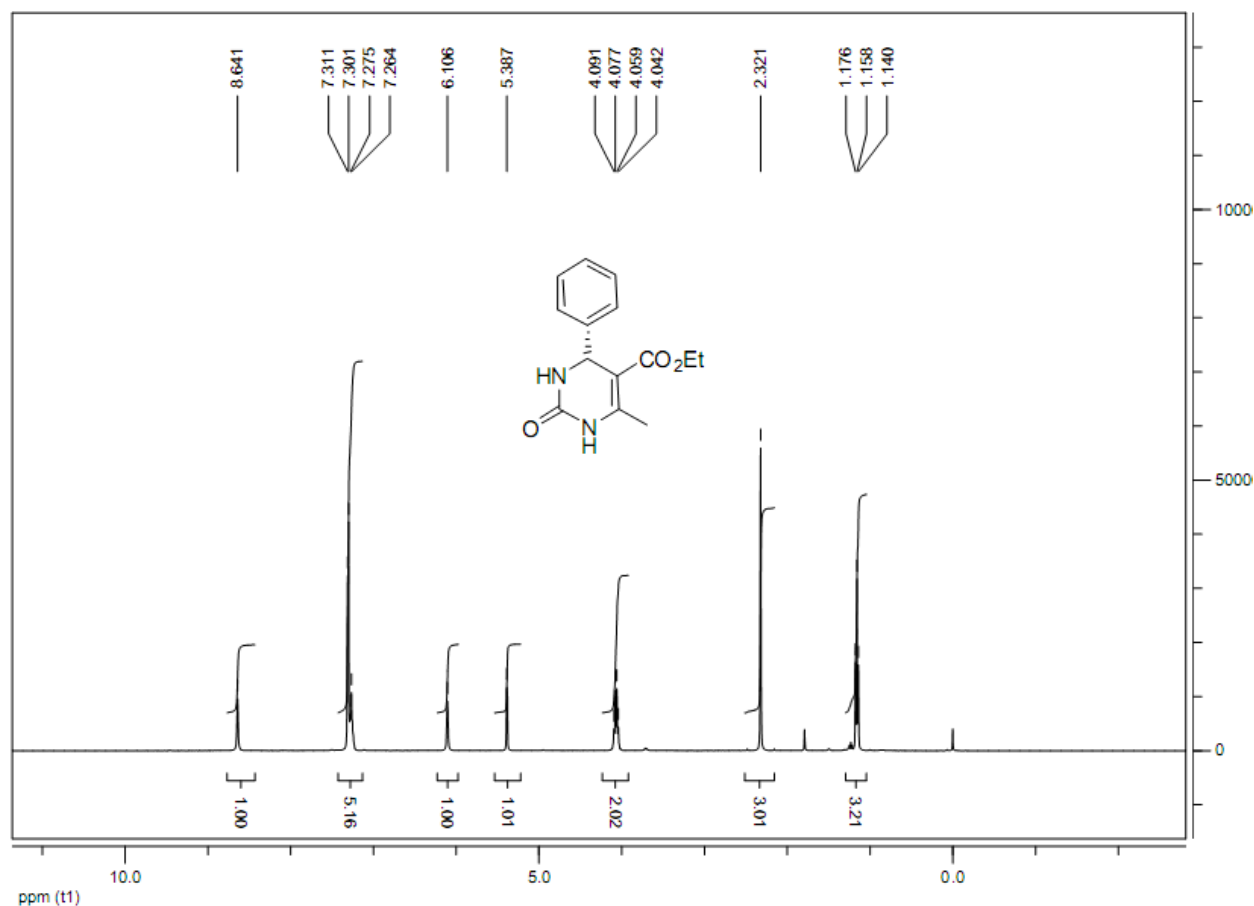
总量 : 5572.82947 104.81928

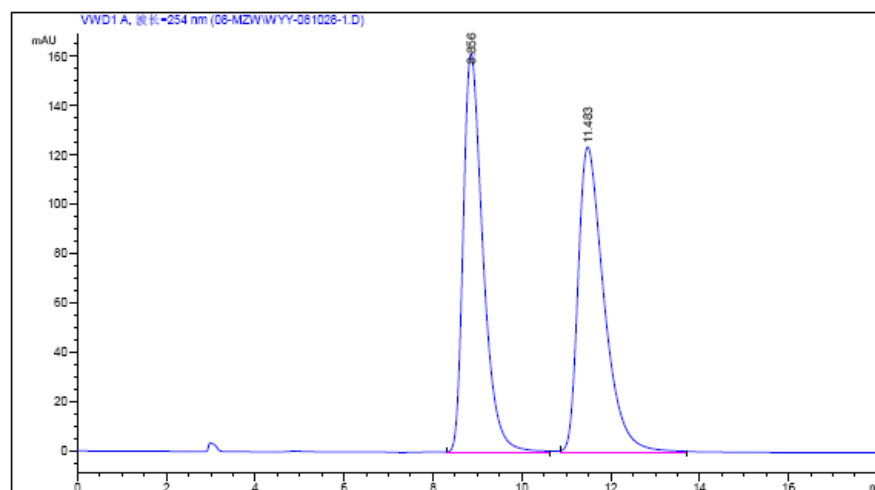
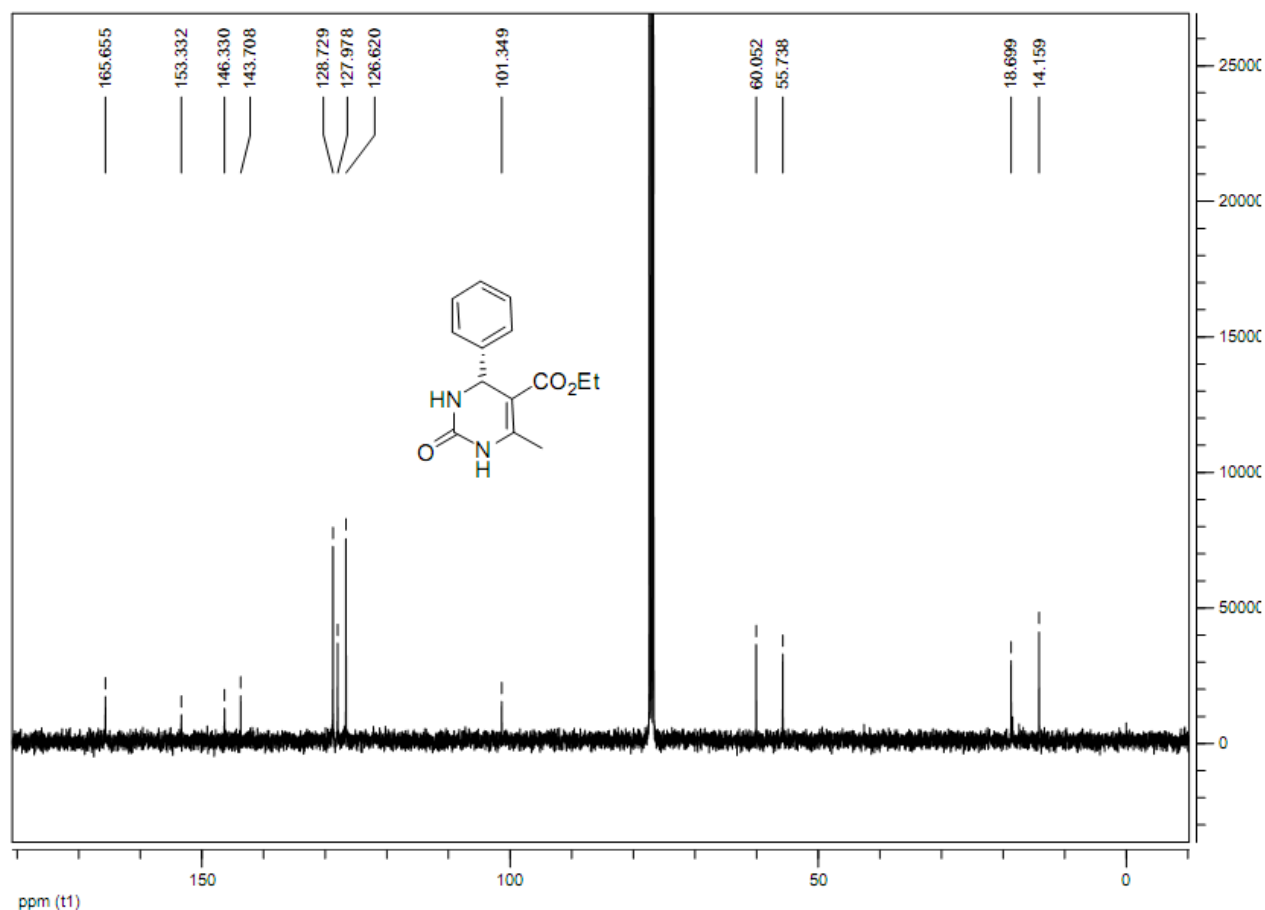
(R)-(-)-5-Ethoxycarbonyl-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1H)-one (8j) :



White solid; mp 202-204 °C; $[\alpha]_D^{20} = -64^\circ$ (c = 0.5, MeOH); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 1.16 (t, $^3J_{\text{H,H}} = 6.8$ Hz, 3H, OCH_2CH_3), 2.32 (s, 3H, CH_3), 4.07 (q, $^3J_{\text{H,H}} = 6.8$ Hz, 2H, OCH_2CH_3), 5.39(d, $^3J_{\text{H,H}} = 2.4$ Hz, 1H, CH), 6.11(s, 1H, NH), 7.26-7.31 (m, 5H, Ph), 8.64 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, CDCl_3): δ 14.16,

18.70, 55.73, 60.05, 101.35, 126.62, 127.98, 128.73, 143.71, 145.33, 153.33, 165.66. ESI-MS: 259.07 ($[\text{M-H}]^-$); The enantiomeric excess was determined to be >99% by HPLC with Chiralcel OD-H column (25 cm) at 254 nm; eluent: hexane: $^i\text{PrOH}$ (85:15), flow rate = 1.0 mL/min, $t_r = 13.091$ min (major).

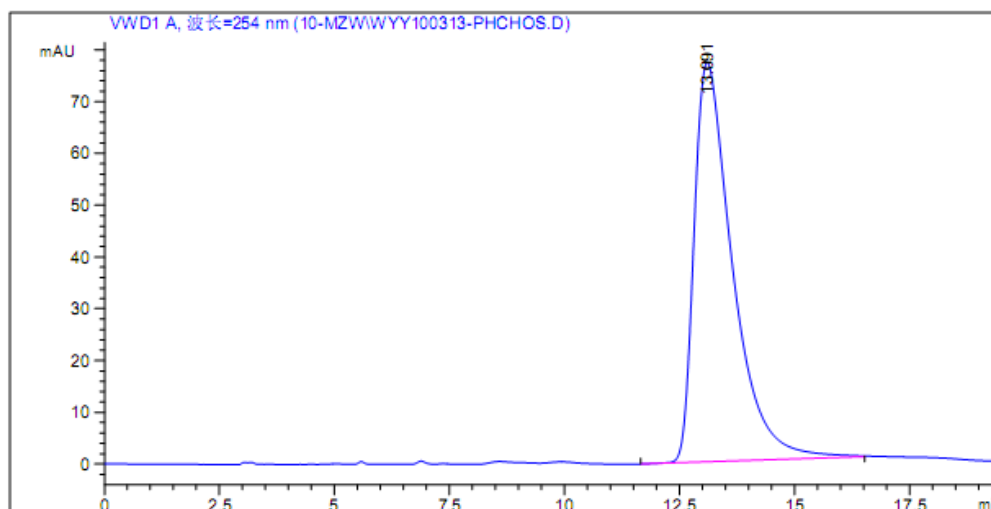




信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU * s	峰高 [mAU]	峰面积 %
1	8.856	BB	0.4766	4976.65479	161.21957	49.8219
2	11.483	BB	0.6124	5012.23193	123.73526	50.1781

总量 : 9988.88672 284.95483



信号 1: VWD1 A, 波长=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	13.091	BB	0.8694	4476.23828	77.11614	100.0000

总量 : 4476.23828 77.11614