Supplementary Information:

Highly Diasteroselective Friedel-Crafts Reaction of Indoles with *N*-*tert*-Butanesulfinylimino Esters: An Efficient and Practical Approach to Enantiomerically Enriched α-(3-Indolyl)glycines

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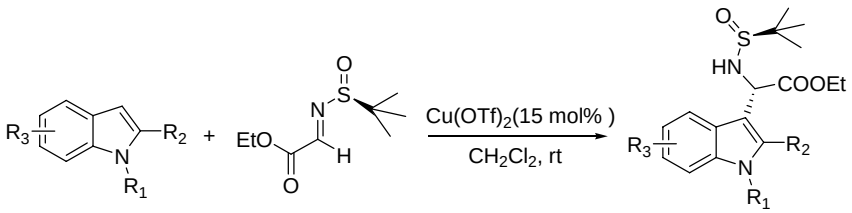
1. General

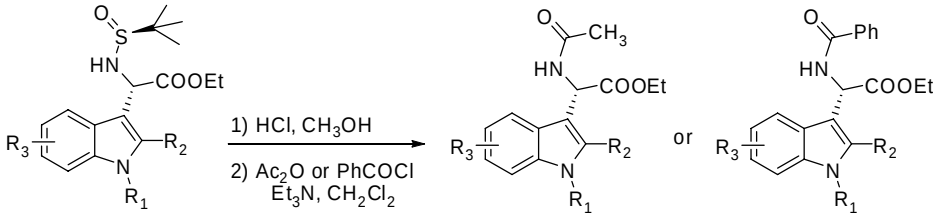
All manipulations were carried out under predried nitrogen. NMR spectra were recorded on a Varian spectrometer (300 MHz for ^{1}H, and 100 MHz for ^{13}C). Chemical shifts are reported in δ ppm referenced to an internal $SiMe_4$ standard for ^{1}H NMR and chloroform-*d* (δ 77.05) for ^{13}C NMR. HPLC was performed on a JASCO 2000 instrument by using Daicel columns.

2. General Procedure for $Cu(OTf)_2$-catalyzed Friedel-Crafts Reaction of Indoles with *N-tert*-Butanesulfinylimino Ester



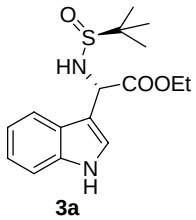
Under nitrogen atmosphere, $Cu(OTf)_2$ (0.0375 mmol, 15 mol%) was placed into a glass reaction vessel, glyoxylate imine (0.25 mmol) in 2 mL of CH_2Cl_2 and indole (0.375 mmol) were added successively. The mixture was stirred at room temperature and monitored by TLC. When the reaction was over, a saturated aq. NH_4Cl was added and the mixture was extracted with CH_2Cl_2 (10 mL×3). The combined organic phase was dried over Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel flash chromatography to afford the corresponding α-(3-indolyl)glycine product.

3. Determination of the Diastereoselectivity / Enantiomeric Excess.



The diastereoselectivities of the α-(3-indolyl)glycine products could be roughly determined by their crude ^{1}H NMR. For accuracy, enantiomeric excesses were determined by chiral HPLC analysis of their acetate or benzoate derivatives after the removal of sulfinyl. α-(3-Indolyl)glycines **3b**, **3d**, **3g** and **3k** were converted to the corresponding benzoate, all others were converted to their acetate. The HPLC reference compound for **3e**, **3g** was a mixture of related products consisting of *R* and *S* enantiomers. Others were obtained by the same Friedel-Crafts reaction of indoles with *N-tert*-butanesulfinylimino ester catalyzed by $AgNO_3$.

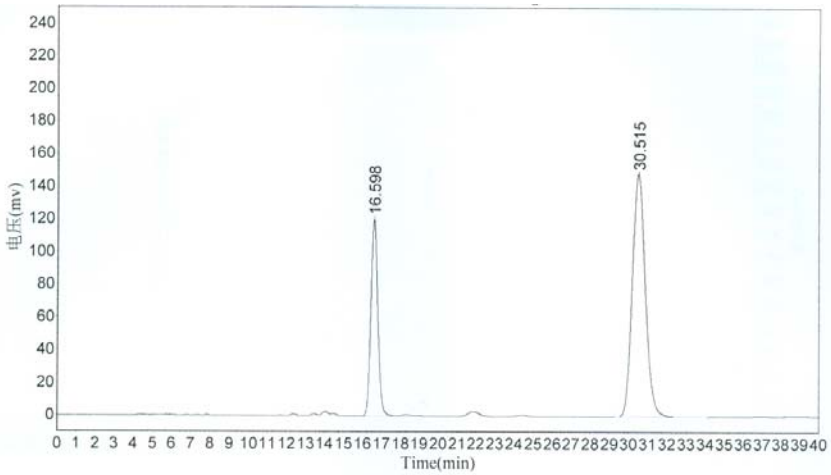
4. Characterization and HPLC of the Obtained Chiral α-(3-Indolyl)glycines



3a

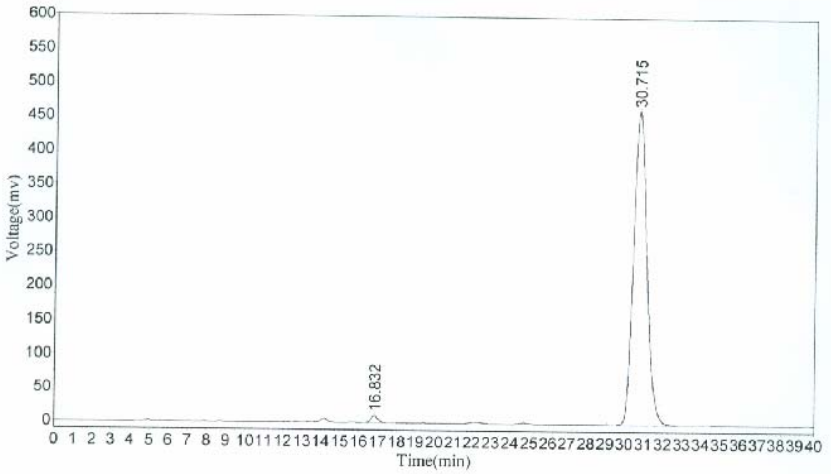
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.17 (t, *J* = 6.9 Hz, 3H), 1.21 (s, 9H), 4.09-4.26 (m, 2H), 4.53 (d, *J* = 4.5 Hz, 1H), 5.3 (d, *J* = 4.8 Hz, 1H), 7.10 (t, *J* = 6.9 Hz, 1H), 7.16-7.21 (m, 2H), 7.35 (d, *J* = 8.1 Hz, 1H), 7.62 (d, *J* = 7.8 Hz, 1H), 8.92 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) 13.9, 22.6, 54.3, 55.7, 61.9, 111.0, 111.5, 119.4, 119.7, 122.3, 124.3, 125.5, 136.6, 171.9. EI-MS (m/z, %): 322 (M^+), 249, 202 (100), 193, 174, 143; HRMS (EI) for $C_{16}H_{22}N_2O_3S$: calcd 322.1351, found 322.1344.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 85/15; flow = 0.7 mL/min; Retention time: 16.8 min, 30.7 min (maj).



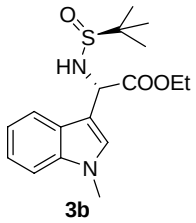
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		16.598	120168.070	3046574.250	30.2311
2		30.515	148393.109	7031040.000	69.7689
Total			268561.180	10077614.250	100.0000



Results

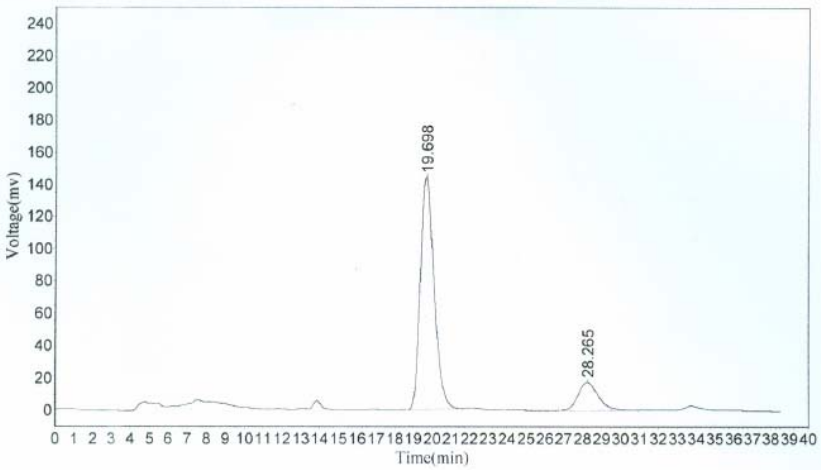
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		16.832	10184.643	264985.594	1.1647
2		30.715	463787.688	22486460.000	98.8353
Total			473972.330	22751445.594	100.0000



3b

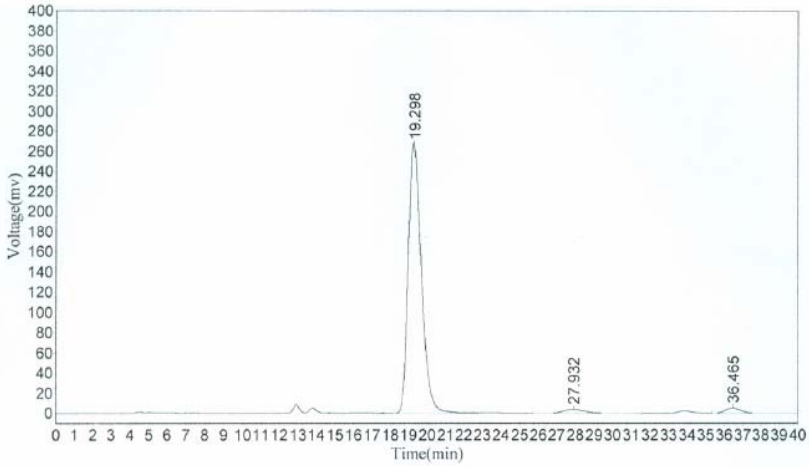
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.18-1.22 (m, 12H), 3.77 (s, 3H), 4.07-4.31 (m, 2H), 4.48 (d, *J* = 4.8 Hz, 1H), 5.29 (d, *J* = 4.8 Hz, 1H), 7.08-7.13 (m, 2H), 7.21-7.32 (m, 2H), 7.62 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 13.4, 22.6, 32.8, 54.1, 55.6, 61.9, 109.4, 109.9, 119.5, 119.7, 122.0, 126.0, 128.4, 137.3, 171.9; EI-MS (m/z, %): 336 (M^+), 263, 231, 216 (100), 188, 157; HRMS (EI) for $C_{17}H_{24}N_2O_3S$: calcd 336.1508, found 336.1505.

HPLC: Chiracel AS-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 80/20; flow = 0.7 mL/min; Retention time: 19.3 min (maj), 27.9 min.



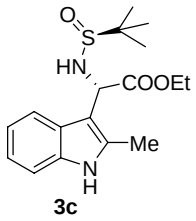
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		19.698	144353.891	7449333.500	85.1571
2		28.265	17379.508	1298417.750	14.8429
Total			161733.398	8747751.250	100.0000



Results

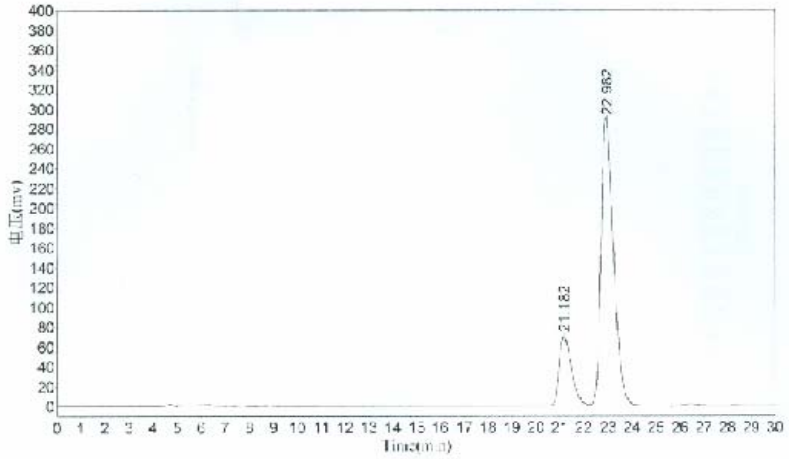
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		19.298	267462.188	13289919.000	95.5196
2		27.932	3745.210	311171.406	2.2365
3		36.465	5377.732	312197.125	2.2439
Total			276585.130	13913287.531	100.0000



3c

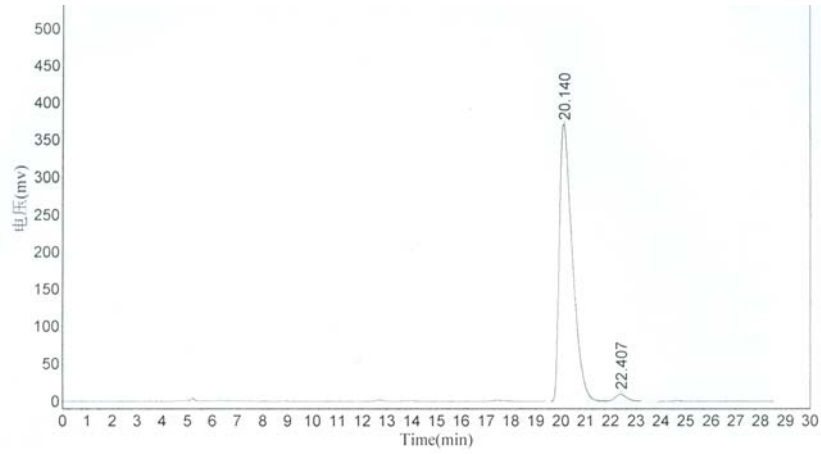
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.14 (t, *J* = 7.5 Hz, 3H), 1.16 (s, 9H), 2.46 (s, 3H), 4.03-4.27 (m, 2H), 4.53 (d, *J* = 1.5 Hz, 1H), 5.3 (d, *J* = 2.4 Hz, 1H), 7.03 (t, *J* = 6.9 Hz, 1H), 7.11 (t, *J* = 7.2 Hz, 1H), 7.25 (d, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 7.5 Hz, 1H), 8.20 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) : 11.7, 14.0, 22.6, 52.6, 55.4, 61.9, 106.6, 110.4, 118.9, 119.7, 121.3, 126.6, 134.7, 135.2, 171.8; EI-MS (m/z, %): 336 (M^+), 263, 217, 216 (100), 188, 170, 158; HRMS (EI) for $C_{17}H_{24}N_2O_3S$: calcd 336.1508, found 336.1511.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 90/10; flow = 0.7 mL/min; Retention time: 20.1 min (maj), 22.4 min.



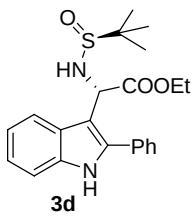
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		21.182	69906.430	2500696.500	19.1738
2		22.982	290284.313	10541543.000	80.8262
Total			360190.742	13042239.500	100.0000



Results

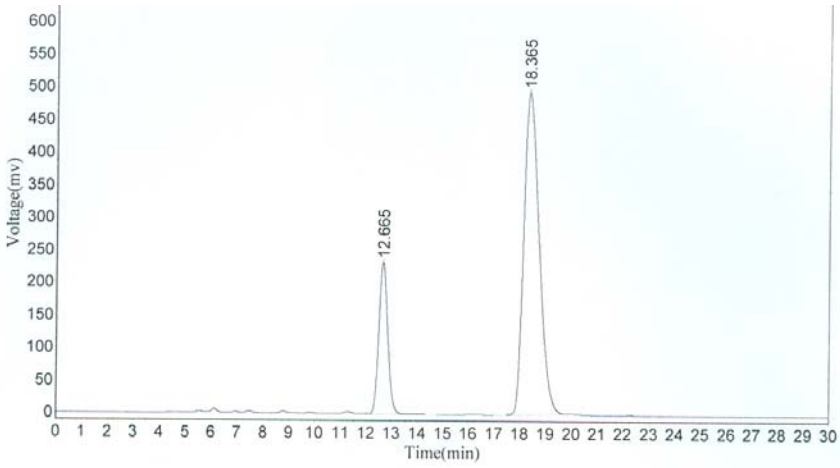
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		20.140	371607.781	13469749.000	97.8932
2		22.407	8887.570	289883.219	2.1068
Total			380495.352	13759632.219	100.0000



3d

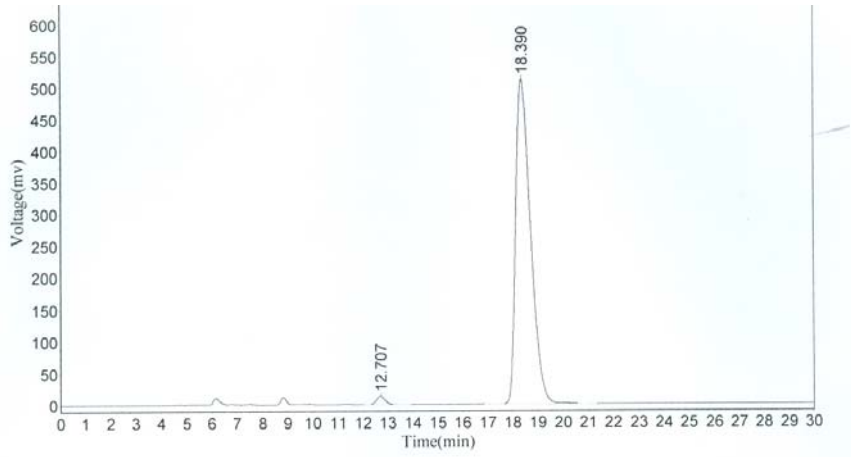
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.10 (t, *J* = 7.2 Hz, 3H), 1.13 (s, 9H), 4.00-4.22 (m, 2H), 4.60 (d, *J* = 2.7 Hz, 1H), 5.45 (d, *J* = 2.7 Hz, 1H), 7.08-7.23 (m, 2H), 7.36-7.50 (m, 4H), 7.66-7.74 (m, 3H), 8.40 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 13.9, 22.5, 53.3, 55.5, 61.9, 107.1, 111.1, 120.0, 120.1, 122.4, 126.8, 128.5, 128.7, 128.9, 131.8, 135.8, 138.2, 171.7. EI-MS (m/z, %): 398 (M^+), 325, 278 (100), 219, 204, 193; HRMS (EI) for $C_{22}H_{26}N_2O_3S$: calcd 398.1664, found 398.1672.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 70/30; flow = 0.7 mL/min; Retention time: 12.7 min, 18.4 min (maj).



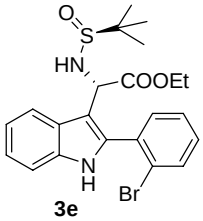
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.665	232569.359	5640813.000	21.4165
2		18.365	494197.594	20697848.000	78.5835
Total			726766.953	26338661.000	100.0000



Results

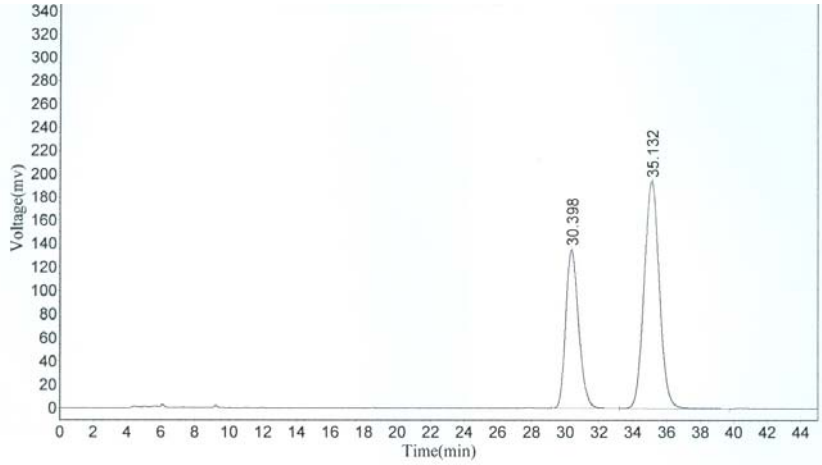
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.707	13133.595	314791.906	1.4719
2		18.390	512485.656	21071930.000	98.5281
Total			525619.251	21386721.906	100.0000



3e

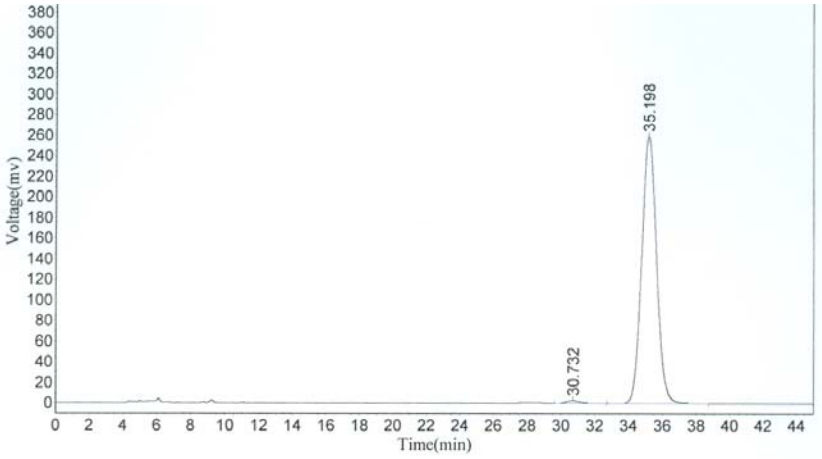
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12 (t, *J* = 7.2 Hz, 3H), 1.14 (s, 9H), 4.00-4.19 (m, 2H), 4.52 (d, *J*=2.4 Hz, 1H), 5.12 (d, *J* = 2.4 Hz, 1H), 7.10-7.46 (m, 5H), 7.67-7.72 (m, 3H), 8.36 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) 14.0, 22.6, 53.3, 55.5, 62.0, 109.2, 111.1, 120.1, 120.3, 122.7, 124.2, 125.7, 127.6, 130.7, 132.4, 133.1, 133.2, 135.6, 136.5, 171.5; EI-MS (m/z, %): 476 (M^+), 405, 356 (100), 299, 284; HRMS (EI) for $C_{22}H_{25}BrN_2O_3S$: calcd 476.0769, found 476.0767.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 90/10; flow = 0.7 mL/min; Retention time: 30.7 min, 35.2 min (maj).



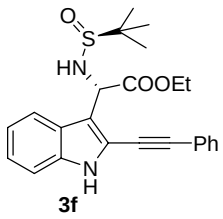
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		30.398	134744.547	7204382.500	37.1389
2		35.132	193893.000	12194083.000	62.8611
Total			328637.547	19398465.500	100.0000



Results

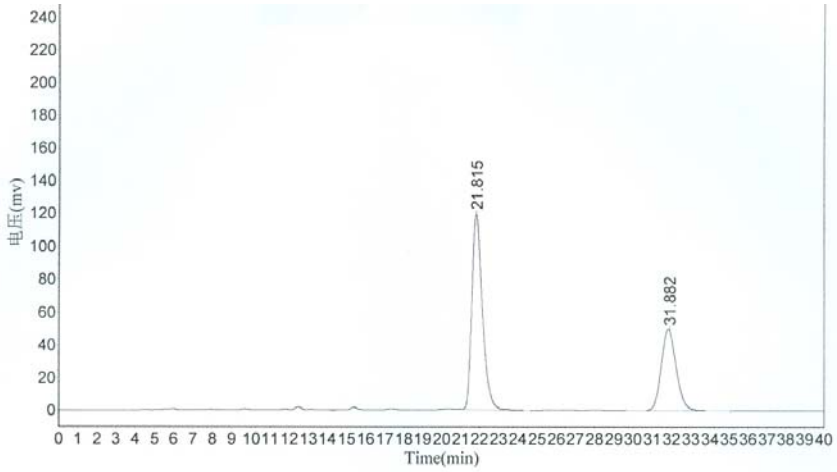
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		30.732	2524.275	143499.297	0.8941
2		35.198	258790.016	15906081.000	99.1059
Total			261314.290	16049580.297	100.0000



3f

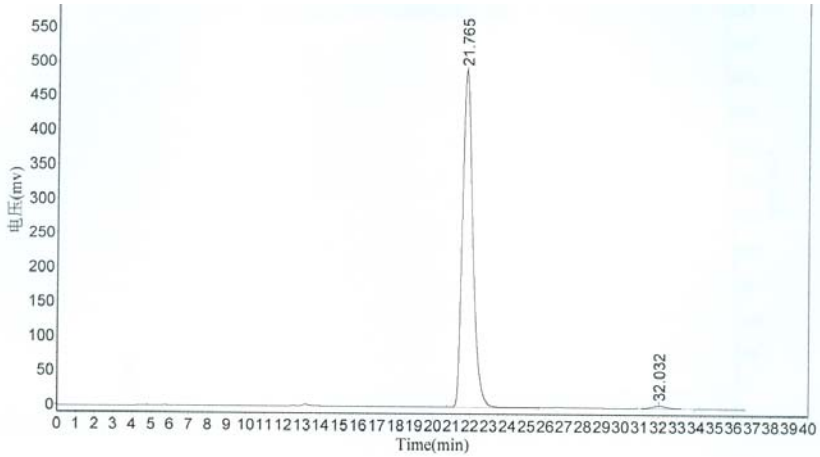
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.12-1.17 (m, 12H), 4.11-4.27 (m, 2H), 4.72 (d, *J* = 3.6 Hz, 1H), 5.58 (d, *J* = 3.6 Hz, 1H), 7.10 (t, *J* = 7.2 Hz, 1H), 7.22-7.37 (m, 5H), 7.56-7.61 (m, 3H), 8.43 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 22.6, 53.5, 55.8, 62.2, 79.6, 96.4, 111.1, 116.8, 119.0, 119.5, 120.7, 122.2, 123.9, 125.4, 128.4, 128.8, 131.6, 135.9, 171.4; EI-MS (m/z, %): 422 (M^+), 349, 303, 302 (100), 244. HRMS (EI) for $C_{24}H_{26}N_2O_3S$: calcd 422.1664, found 422.1663.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 85/15; flow = 0.7 mL/min; Retention time: 21.8 min (maj), 32.0 min.



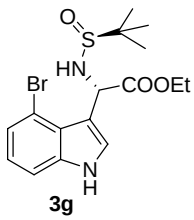
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		21.815	117907.414	4652515.000	63.3515
2		31.882	49627.023	2691451.500	36.6485
Total			167534.438	7343966.500	100.0000



Results

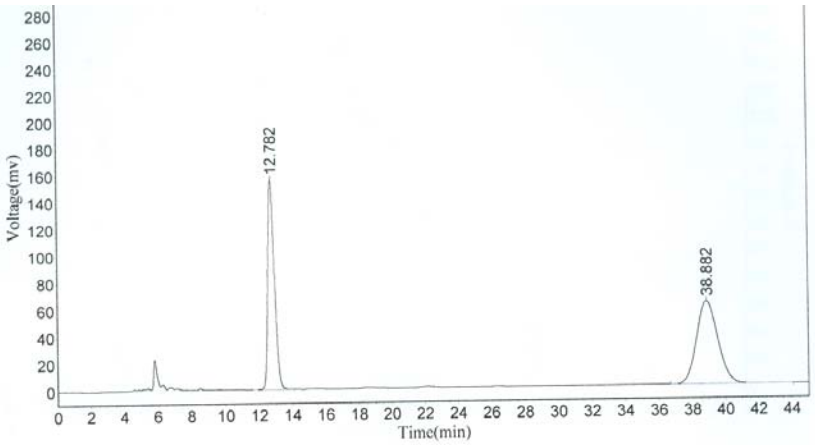
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		21.765	490179.094	18890426.000	98.7910
2		32.032	4298.667	231177.500	1.2090
Total			494477.760	19121603.500	100.0000



3g

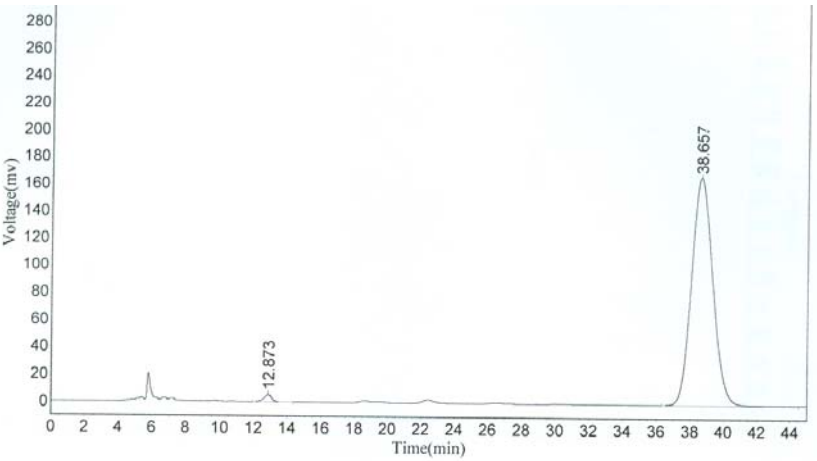
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.20-1.25 (m, 12H), 4.17-4.29 (m, 2H), 4.41 (bs, 1H), 5.86 (bs, 1H), 7.01 (t, *J* = 7.8 Hz, 1H), 7.26-7.35 (m, 3H), 9.46 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 22.7, 53.8, 56.1, 61.9, 111.1, 111.9, 113.5, 123.1, 124.3, 124.5, 126.1 (bs), 138.0, 172.6; EI-MS (m/z, %): 400 (M^+), 282 (100), 281, 210. HRMS (EI) for $C_{16}H_{21}BrN_2O_3S$: calcd 400.0456, found 400.0454.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 70/30; flow = 0.7 mL/min; Retention time: 12.9 min, 38.6 min (maj).



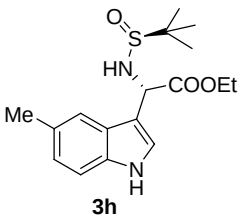
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.782	156162.469	4280112.500	43.9831
2		38.882	61328.648	5451161.500	56.0169
Total			217491.117	9731274.000	100.0000



Results

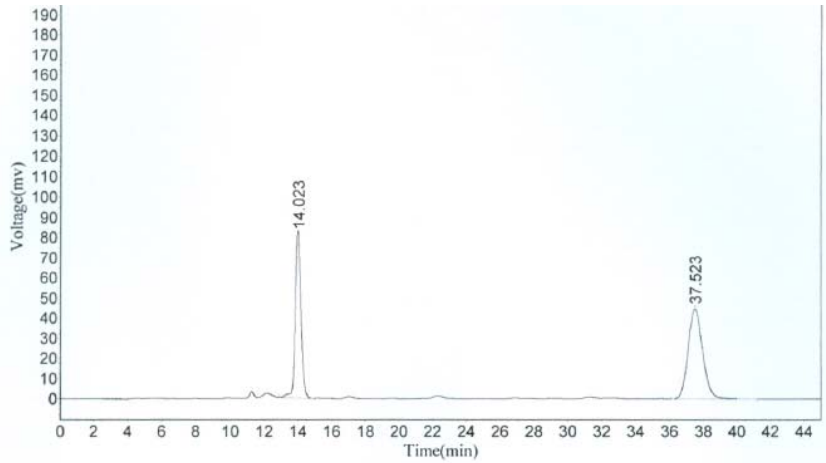
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.873	5378.042	174413.500	1.1695
2		38.657	167484.781	14738697.000	98.8305
Total			172862.823	14913110.500	100.0000



3h

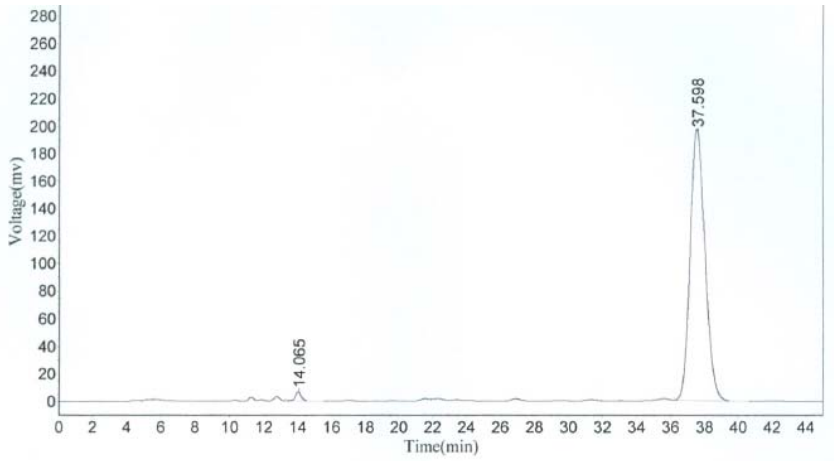
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.19 (t, *J* = 7.2 Hz, 3H), 1.21 (s, 9H), 2.42 (s, 3H), 4.10-4.27 (m, 2H), 4.49 (d, *J* = 4.5 Hz, 1H), 5.28 (d, *J* = 4.5 Hz, 1H), 7.01 (d, *J* = 8.4 Hz, 1H), 7.15 (d, *J* = 2.4 Hz, 1H), 7.24 (d, *J* = 8.1 Hz, 1H), 7.42 (s, 1H), 8.57 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 13.4, 21.5, 22.6, 54.4, 55.7, 61.9, 110.8, 111.1, 119.1, 124.0, 124.2, 125.8, 129.1, 134.9, 171.9; EI-MS (m/z, %): 336 (M^+), 263, 231, 217, 216 (100), 188, 158, 157, 142. HRMS (EI) for $C_{17}H_{24}N_2O_3S$: calcd 336.1508, found 336.1512.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 85/15; flow = 0.7 mL/min; Retention time: 14.1 min, 37.6 min (maj).



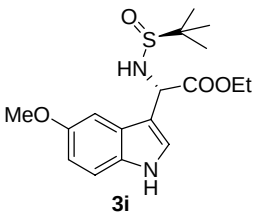
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.023	82686.898	1958422.875	42.2437
2		37.523	44652.035	2677584.750	57.7563
Total			127338.934	4636007.625	100.0000



Results

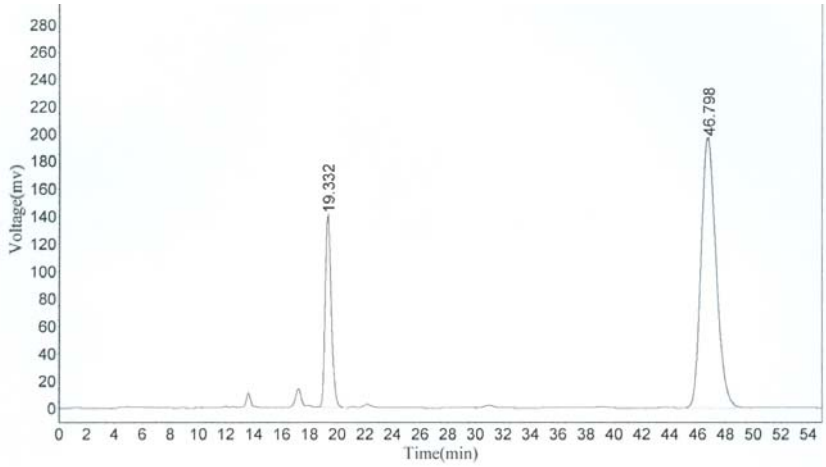
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.065	6507.499	172117.141	1.3930
2		37.598	197941.813	12183512.000	98.6070
Total			204449.311	12355629.141	100.0000



3i

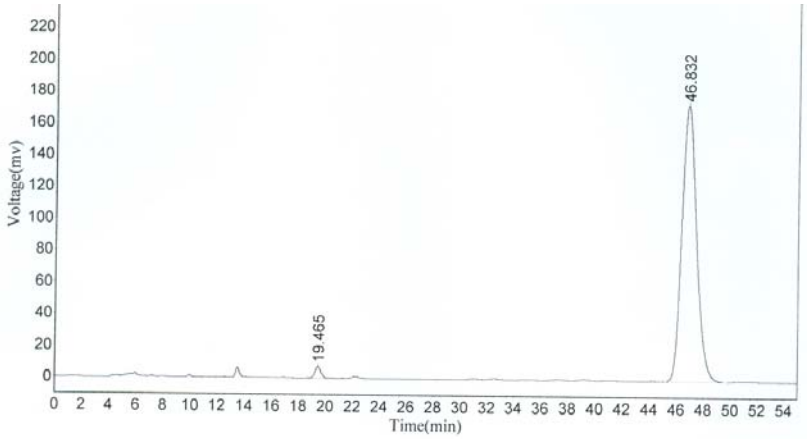
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.19 (t, *J* = 7.5 Hz, 3H), 1.22 (s, 9H), 3.82 (s, 3H), 4.12-4.27 (m, 2H), 4.49 (d, *J* = 4.8 Hz, 1H, NH), 5.28 (d, *J* = 4.8 Hz, 1H), 6.84 (dd, *J* = 9.0, 2.4 Hz, 1H), 7.08 (d, *J* = 2.4 Hz, 1H), 7.16 (d, *J* = 2.4 Hz, 1H), 7.24 (s, 1H), 8.67 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 22.6, 54.3, 55.7, 62.0, 101.1, 110.8, 112.2, 112.8, 124.8, 126.0, 131.7, 154.1, 171.8; EI-MS (m/z, %): 353 (M^++H), 352 (M^+), 279, 232 (100), 223, 204, 173. HRMS (EI) for $C_{17}H_{24}N_2O_4S$: calcd 352.1457, found 352.1464.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 85/15; flow = 0.7 mL/min; Retention time: 19.5 min, 46.8 min (maj).



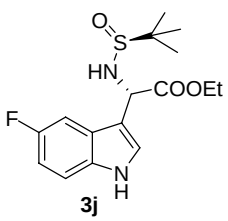
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		19.332	139864.594	4360103.500	22.5759
2		46.798	196718.922	14953010.000	77.4241
Total			336583.516	19313113.500	100.0000



Results

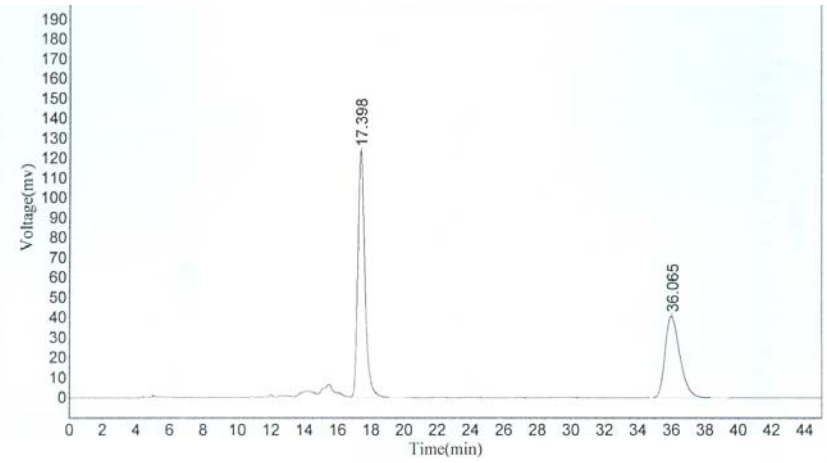
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		19.465	7112.225	228375.000	1.7058
2		46.832	173563.938	13159646.000	98.2942
Total			180676.163	13388021.000	100.0000



3j

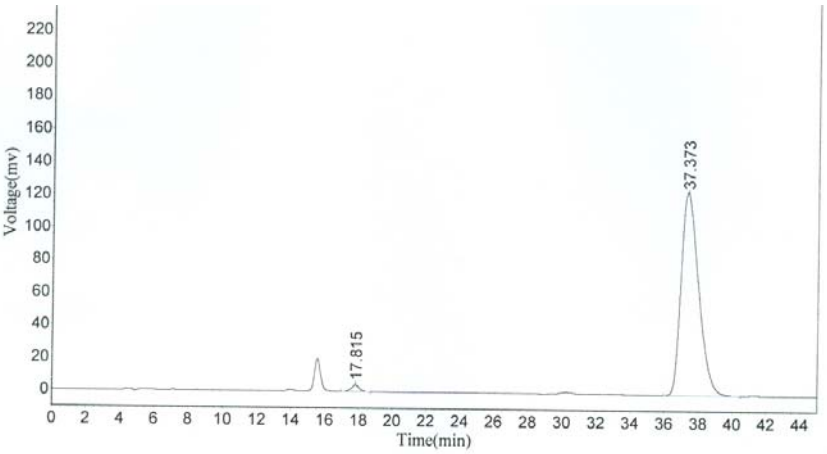
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.19 (t, *J* = 7.2 Hz, 3H), 1.22 (s, 9H), 4.11-4.28 (m, 2H), 4.50 (d, *J* = 4.2 Hz, 1H), 5.26 (d, *J* = 4.2 Hz, 1H), 6.95 (td, *J* = 8.7, 2.4 Hz, 1H), 7.25-7.31 (m, 3H), 8.73 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 22.6, 54.1, 55.8, 62.1, 104.4, 104.7, 110.8, 111.1, 111.4, 112.1, 112.2, 125.8, 126.0, 133.1, 156.6, 159.0, 171.5; EI-MS (m/z, %): 340 (M^+), 267, 220 (100), 211, 192, 161. HRMS (EI) for $C_{16}H_{21}FN_2O_3S$: calcd 340.1257, found 340.1252.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 87/13; flow = 0.7 mL/min; Retention time: 17.8 min, 37.4 min (maj).



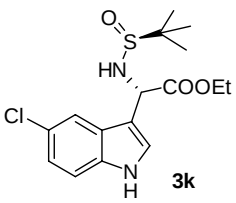
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		17.398	123410.086	3682645.500	59.5834
2		36.065	40534.703	2498009.250	40.4166
Total			163944.789	6180654.750	100.0000



Results

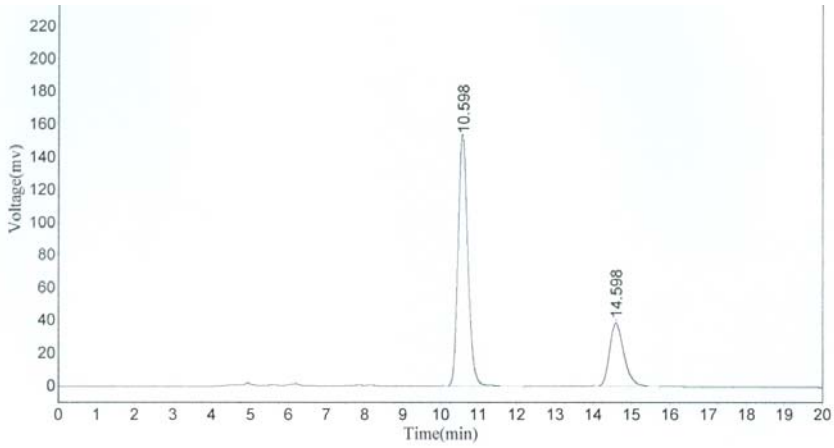
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		17.815	3938.029	121967.398	1.3529
2		37.373	124512.352	8893079.000	98.6471
Total			128450.381	9015046.398	100.0000



3k

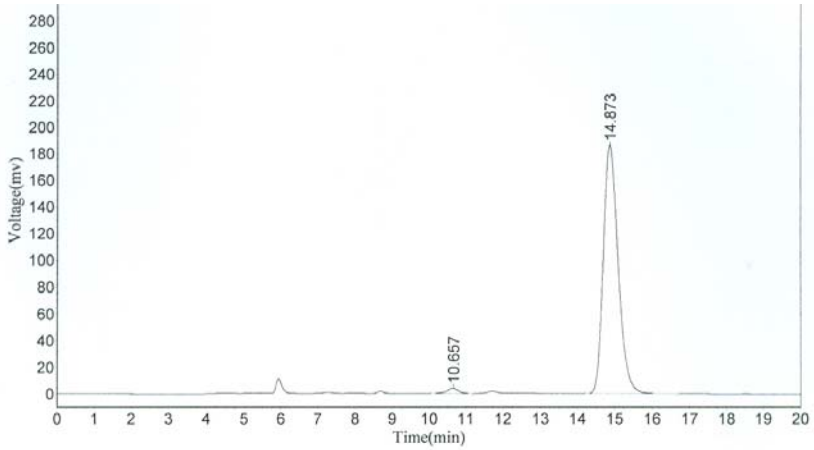
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.20 (t, *J* = 7.2 Hz, 3H), 1.23 (s, 9H), 4.12-4.28 (m, 2H), 4.50 (d, *J* = 4.2 Hz, 1H), 5.26 (d, *J* = 4.2 Hz, 1H), 7.14 (dd, *J* = 8.7, 2.1 Hz, 1H), 7.23 (d, *J* = 2.7 Hz, 1H), 7.27 (d, *J* = 8.7 Hz, 1H), 7.61 (d, *J* = 1.8 Hz, 1H), 8.74 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 22.6, 54.0, 55.8, 62.2, 111.1, 112.5, 119.2, 122.8, 125.6, 125.7, 126.5, 135.0, 171.4; EI-MS (m/z, %): 356 (M^+), 283, 236 (100); HRMS (EI) for $C_{16}H_{21}ClN_2O_3S$: calcd 356.0961, found 356.0954.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 75/25; flow = 0.7 mL/min; Retention time: 10.6 min, 14.9 min (maj).



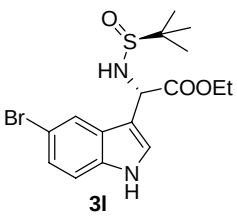
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		10.598	153561.016	2789328.000	73.0489
2		14.598	38703.078	1029109.688	26.9511
Total			192264.094	3818437.688	100.0000



Results

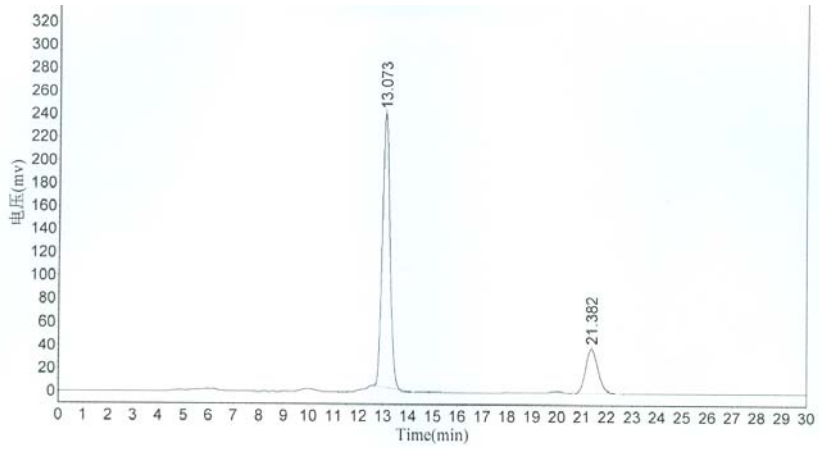
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		10.657	3896.431	86264.242	1.6419
2		14.873	185956.969	5167575.500	98.3581
Total			189853.400	5253839.742	100.0000



3l

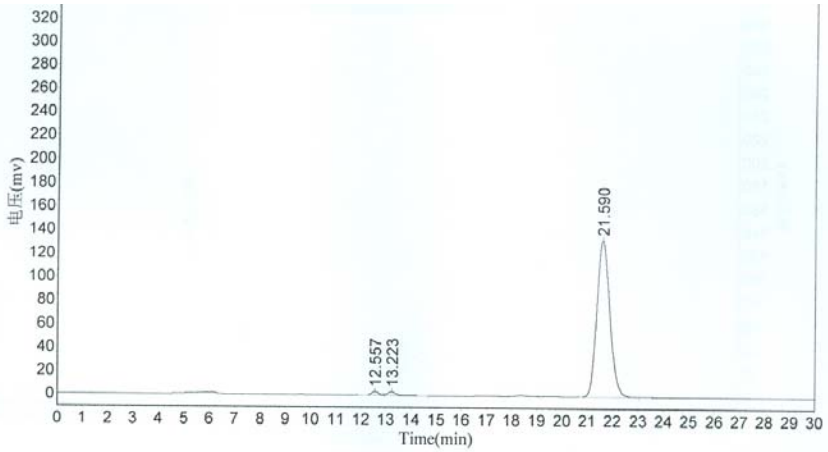
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.19 (td, *J* = 6.9, 1.5 Hz, 3H), 1.23 (s, 9H), 4.11-4.26 (m, 2H), 4.51 (d, *J* = 4.5 Hz, 1H), 5.26 (d, *J* = 4.5 Hz, 1H), 7.18-4.29 (m, 3H), 7.77 (s, 1H), 9.12 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 13.4, 22.6, 54.1, 55.9, 61.2, 110.7, 113.0, 113.1, 122.2, 125.3, 125.6, 127.1, 135.3, 171.5; EI-MS (m/z, %): 403, 283, 280 (100), 271, 254, 223; HRMS (EI) for $C_{16}H_{22}BrN_2O_3S$ (M^+ + H): calcd 401.0535, found 401.0530.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 85/15; flow = 0.7 mL/min; Retention time: 13.2 min, 21.6 min (maj).



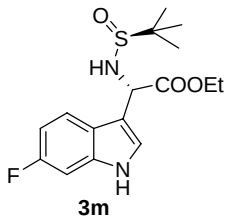
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		13.073	241367.313	5342728.000	80.2052
2		21.382	38374.344	1318597.750	19.7948
Total			279741.656	6661325.750	100.0000



Results

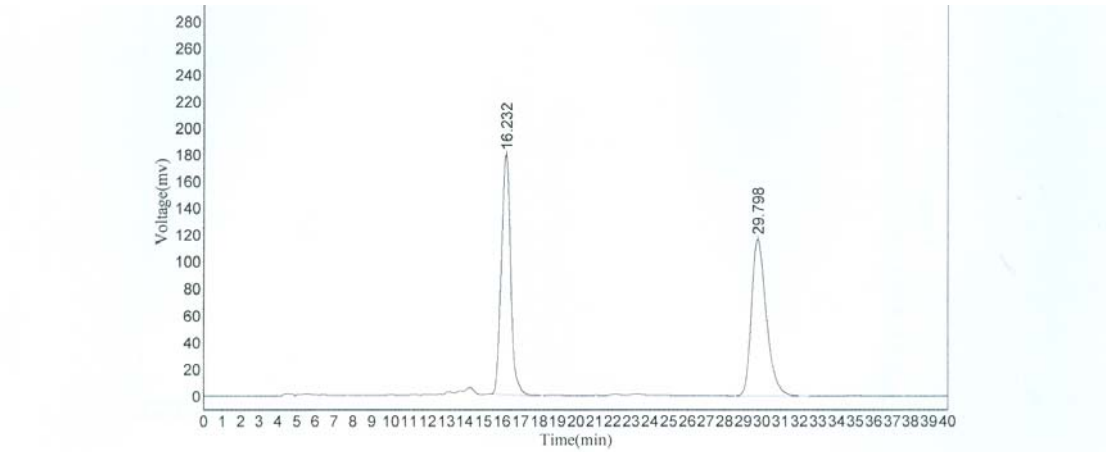
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.557	3325.312	70378.852	1.4350
2		13.223	3098.902	71445.898	1.4568
3		21.590	133100.500	4762598.000	97.1082
Total			139524.714	4904422.750	100.0000



3m

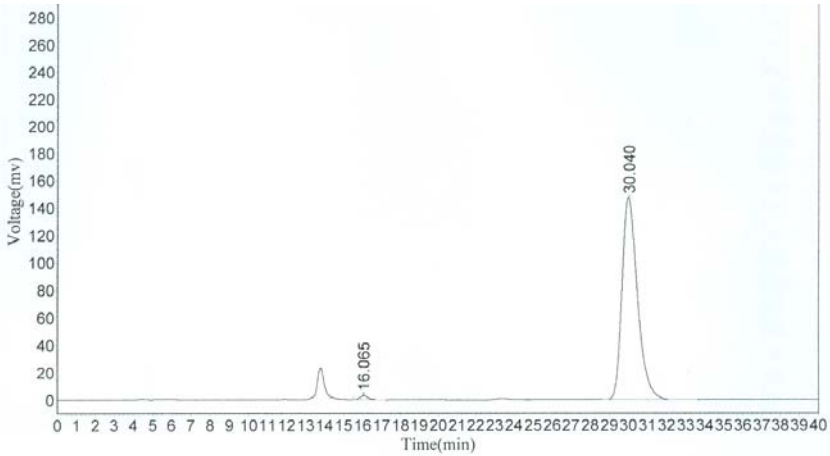
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.18 (t, *J* = 7.2 Hz, 3H), 1.21 (s, 9H), 4.11-4.27 (m, 2H), 4.50 (d, *J* = 4.2 Hz, 1H), 5.28 (d, *J* = 4.2 Hz, 1H), 6.87 (td, *J* = 9.0, 2.1 Hz, 1H), 7.04 (dd, *J* = 9.6, 2.4 Hz, 1H), 7.19 (d, *J* = 9, 2.7 Hz, 1H), 7.52-7.57 (m, 1H), 8.71 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 22.6, 54.1, 55.7, 62.1, 97.7, 97.9, 108.6, 108.9, 111.5, 120.3, 120.4, 122.0, 124.6, 136.6, 136.7, 158.9, 161.2, 171.6; EI-MS (m/z, %): 340 (M^+), 267, 220 (100), 211, 192, 161; HRMS (EI) for $C_{16}H_{21}FN_2O_3S$: calcd 340.1257, found 340.1262.

HPLC: Chiracel AD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 85/15; flow = 0.7 mL/min; Retention time: 16.1 min, 30.0 min (maj).



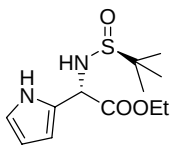
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		16.232	179400.781	6375770.500	49.6004
2		29.798	116566.594	6478512.500	50.3996
Total			295967.375	12854283.000	100.0000



Results

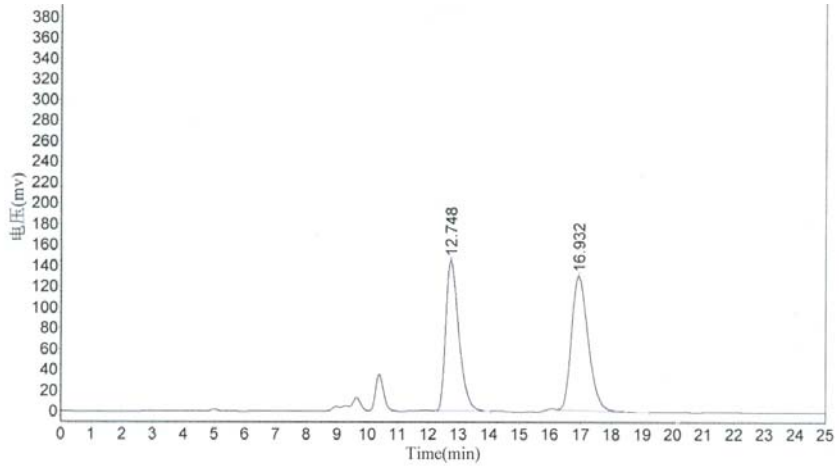
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		16.065	2806.167	83157.797	0.9909
2		30.040	147116.047	8308992.000	99.0091
Total			149922.214	8392149.797	100.0000



5a

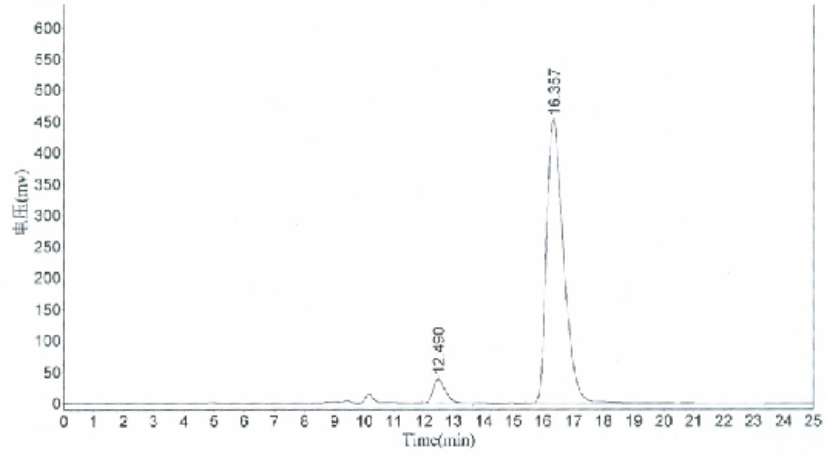
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.26-1.31 (m, 12H), 4.18-4.31 (m, 2H), 4.43 (d, *J* = 6.3 Hz, 1H), 5.15 (d, *J* = 6.3 Hz, 1H), 6.15-6.17 (m, 2H), 6.78 (d, *J* = 2.1 Hz, 1H), 8.71 (bs, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 22.6, 54.6, 56.3, 62.2, 107.7, 108.6, 118.8, 126.1, 170.5. EI-MS (m/z, %): 272 (M^+), 167, 152 (100), 96; HRMS (EI) for $C_{12}H_{20}N_2O_3S$: calcd 272.1195, found 272.1182.

HPLC: Chiracel OD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 90/10; flow = 0.7 mL/min; Retention time: 12.5 min, 16.4 min (maj).



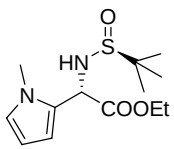
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.748	144660.797	4351853.500	46.1859
2		16.932	130720.453	5070622.500	53.8141
Total			275381.250	9422476.000	100.0000



Results

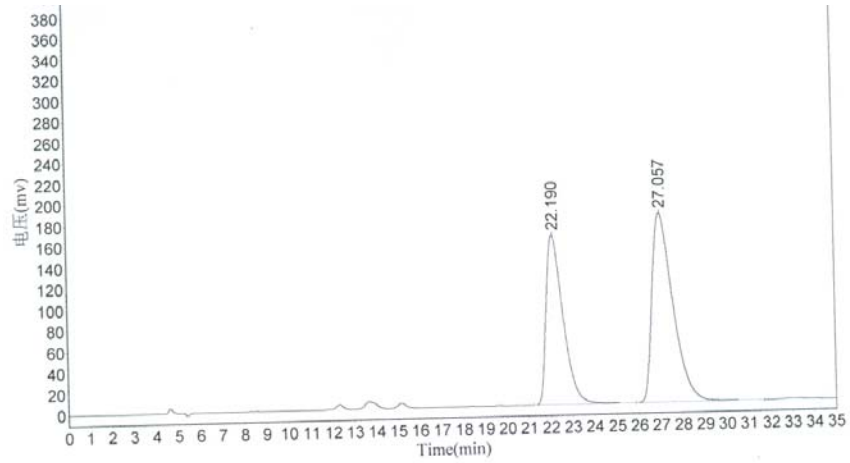
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.490	38556.027	1116841.000	5.8716
2		16.357	453398.281	17904280.000	94.1284
Total			491954.309	19021121.000	100.0000



5b

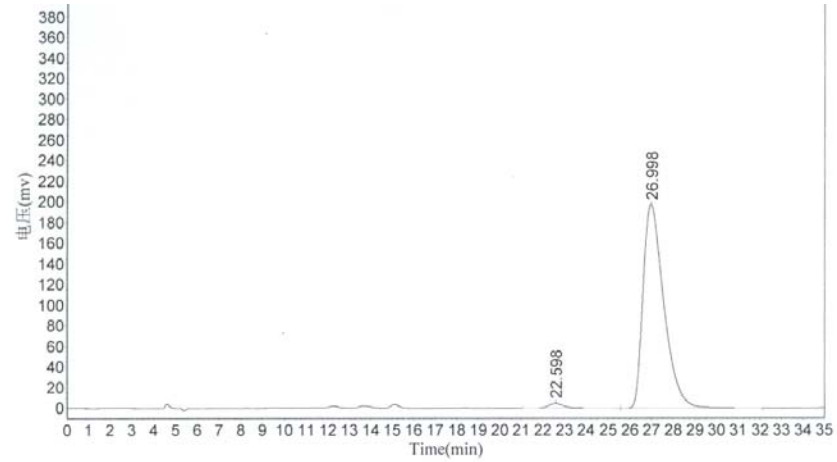
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.24 (s, 9H), 1.27 (t, *J* = 7.2 Hz, 3H), 3.61 (s, 3H), 4.19-4.33 (m, 2H), 4.38 (d, *J* = 6.0 Hz, 1H), 5.07 (d, *J* = 5.4 Hz, 2H), 6.06-6.11 (m, 2H), 6.63 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 14.0, 22.6, 34.1, 54.0, 56.0, 62.2, 107.1, 109.6, 124.0, 126.6, 170.7; EI-MS (m/z, %): 286 (M^+), 181, 166 (100), 152, 138, 107; HRMS (EI) for $C_{13}H_{22}N_2O_3S$: calcd 286.1351, found 286.1344.

HPLC: Chiracel OD-H Column (250 mm); detected at 254 nm; n-hexane / i-propanol = 90/10; flow = 0.7 mL/min; Retention time: 22.6 min, 27.0 min (maj).



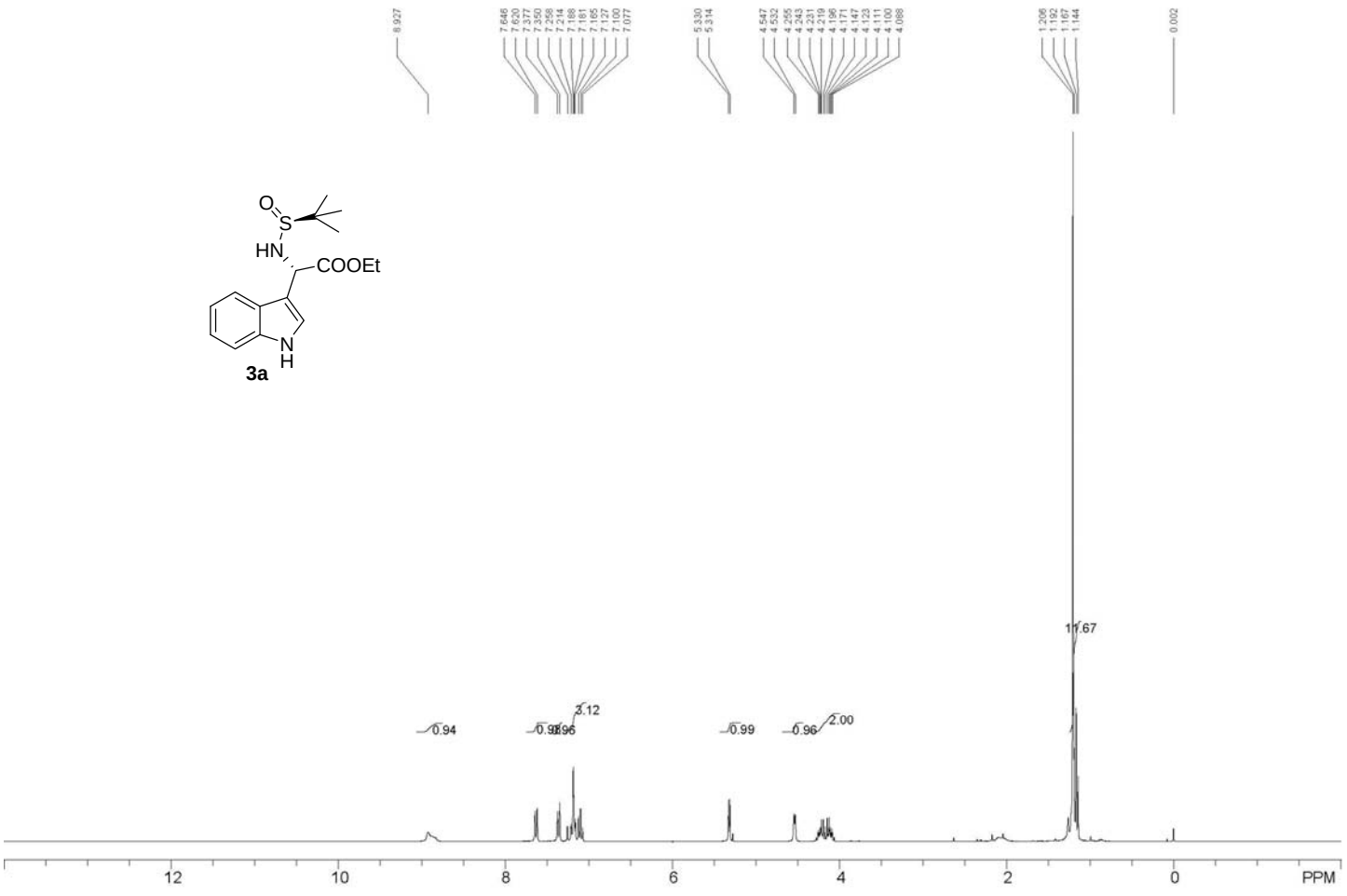
Results

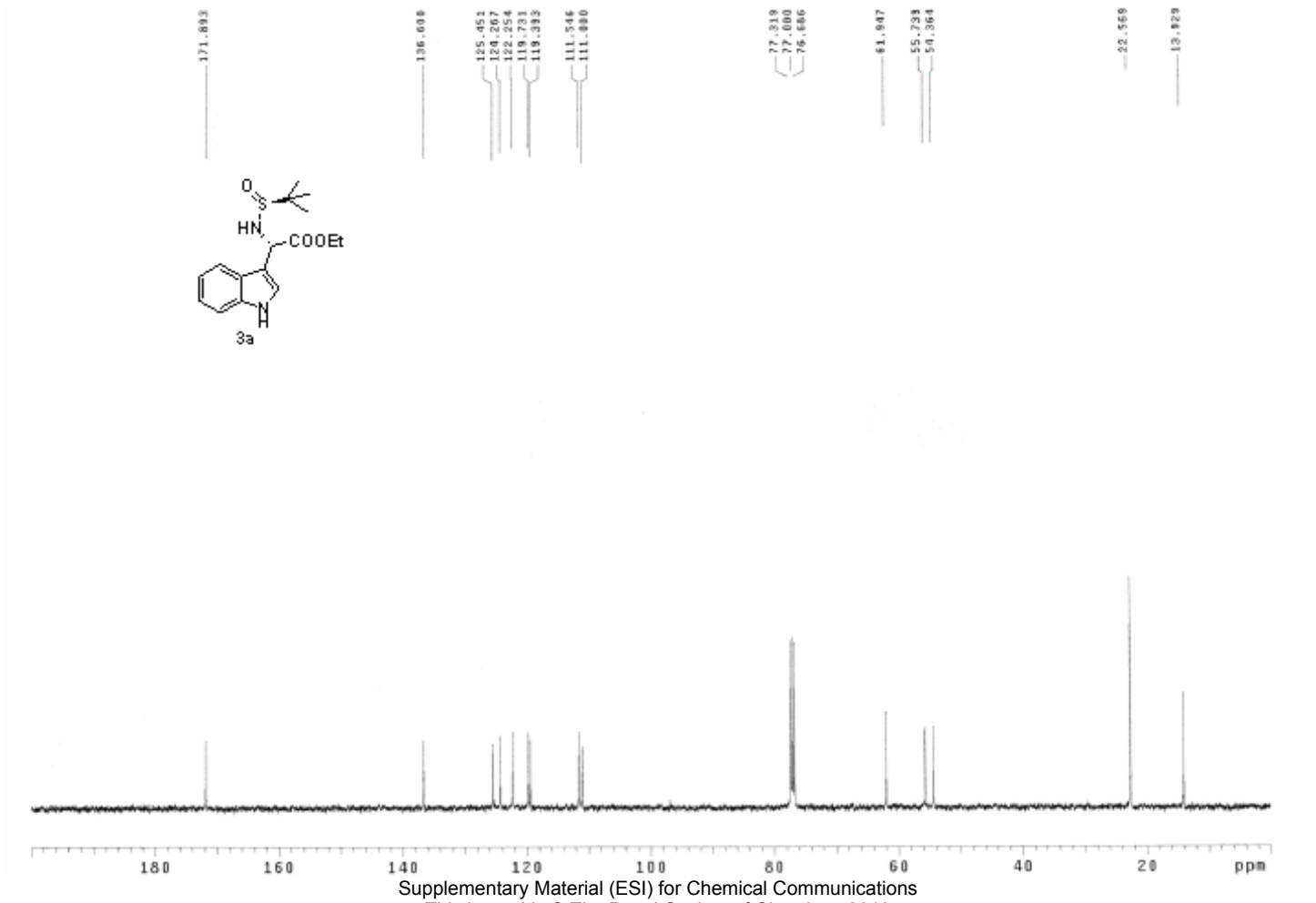
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		22.190	161589.625	8645908.000	42.0976
2		27.057	180116.297	11891875.000	57.9024
Total			341705.922	20537783.000	100.0000

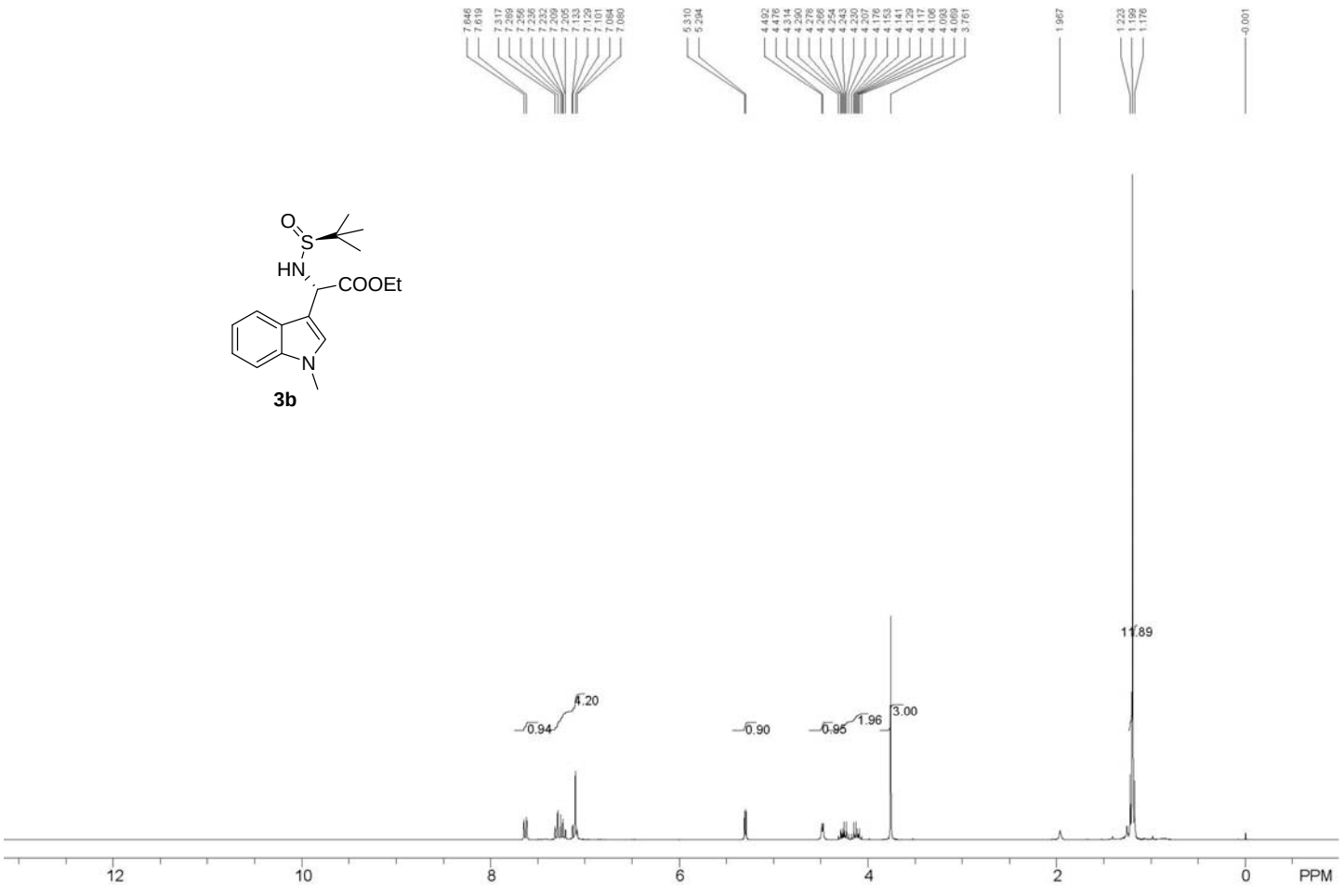


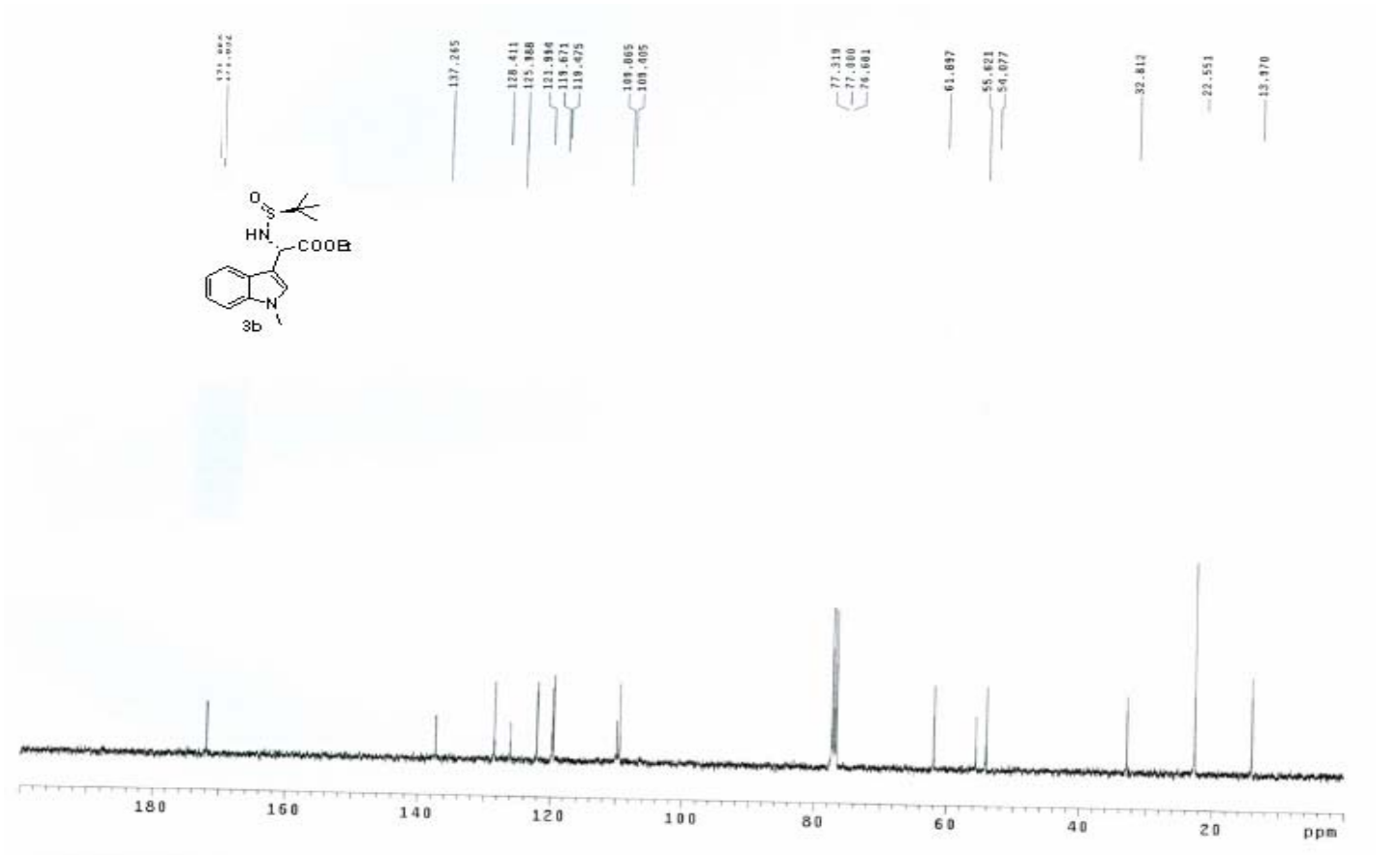
Results

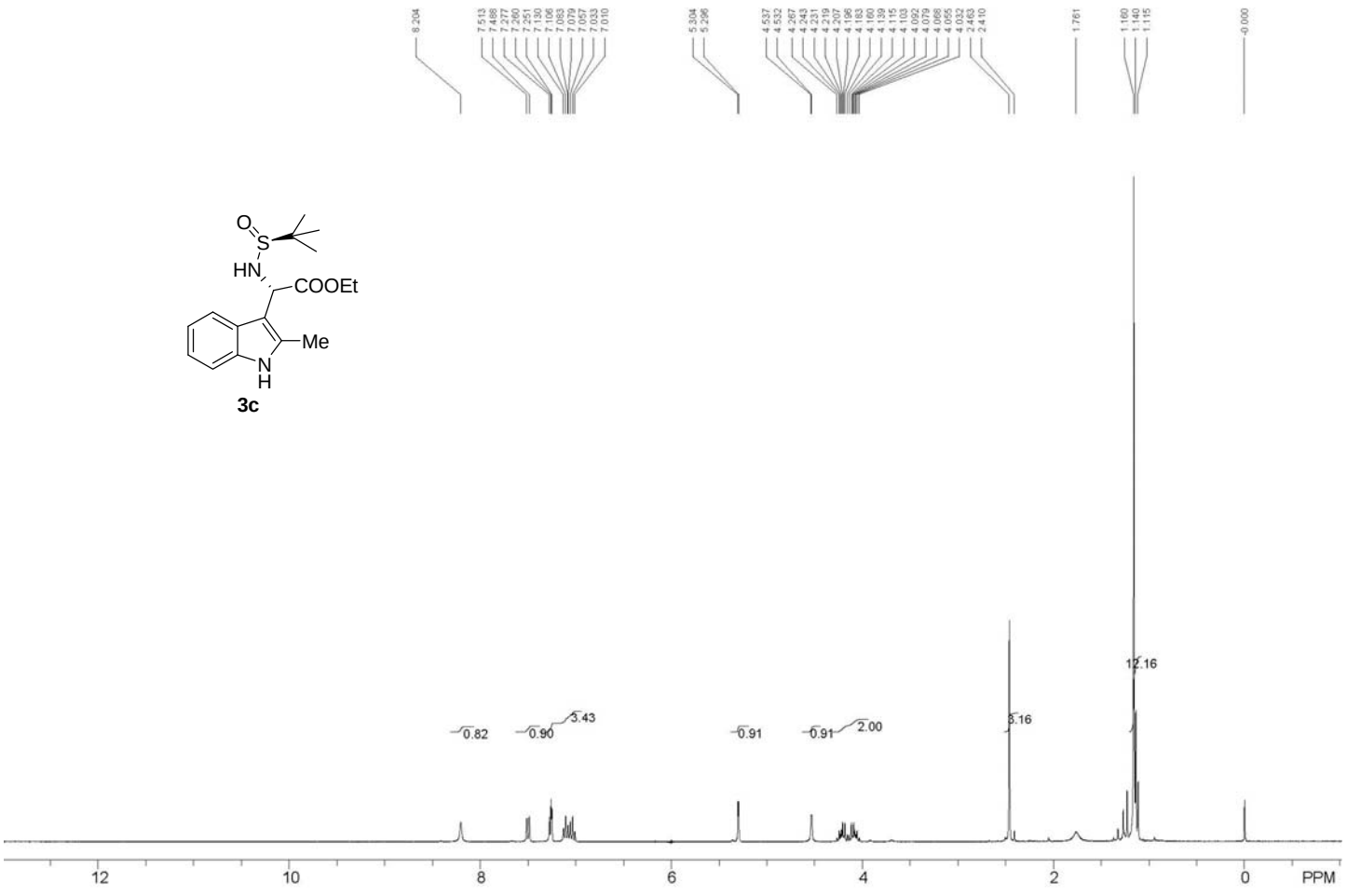
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		22.598	4488.818	218749.219	1.6402
2		26.998	197803.359	13118042.000	98.3598
Total			202292.178	13336791.219	100.0000

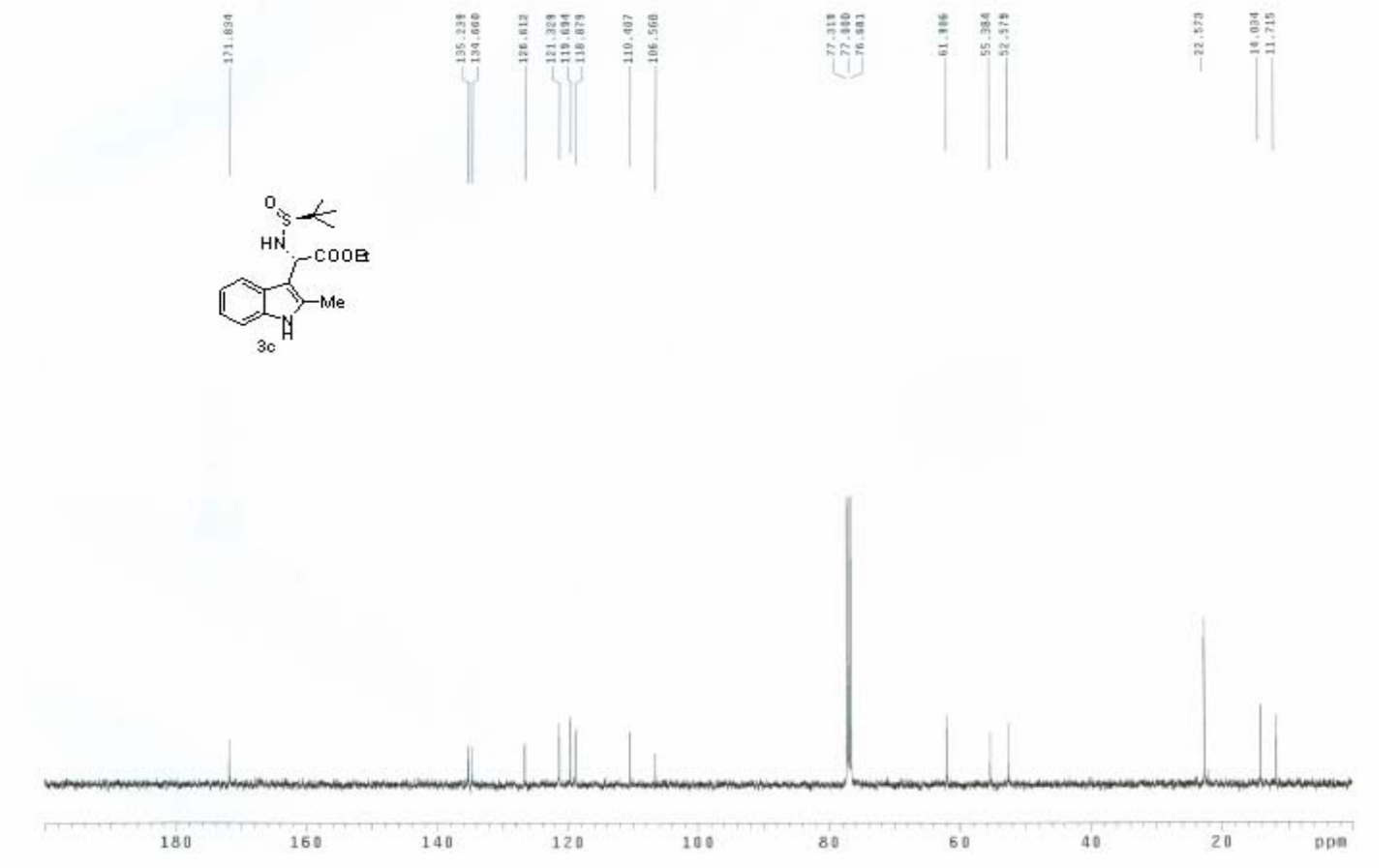


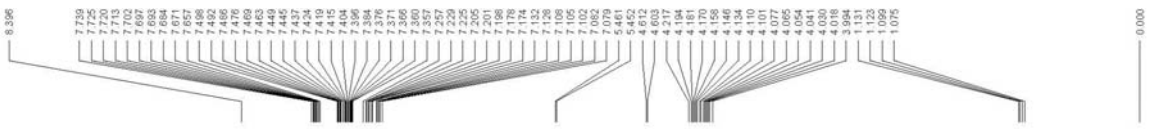




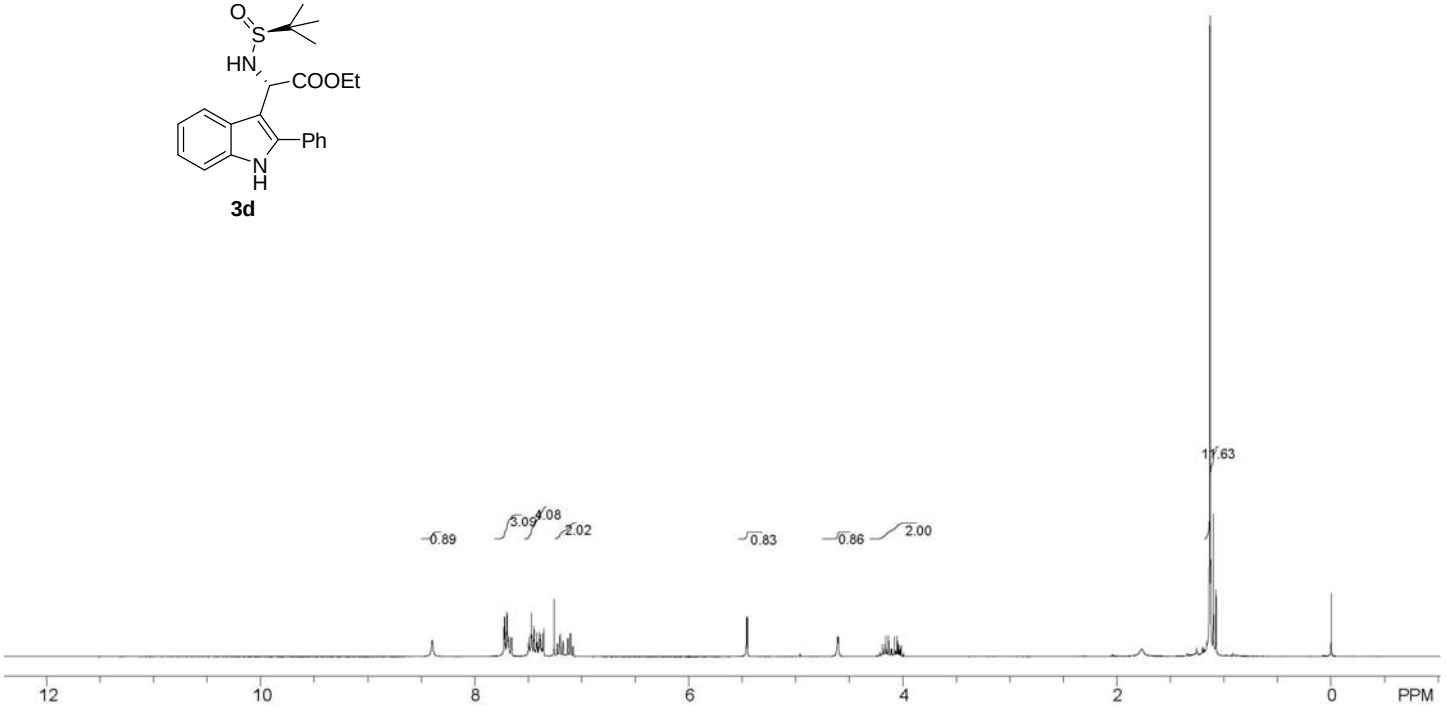


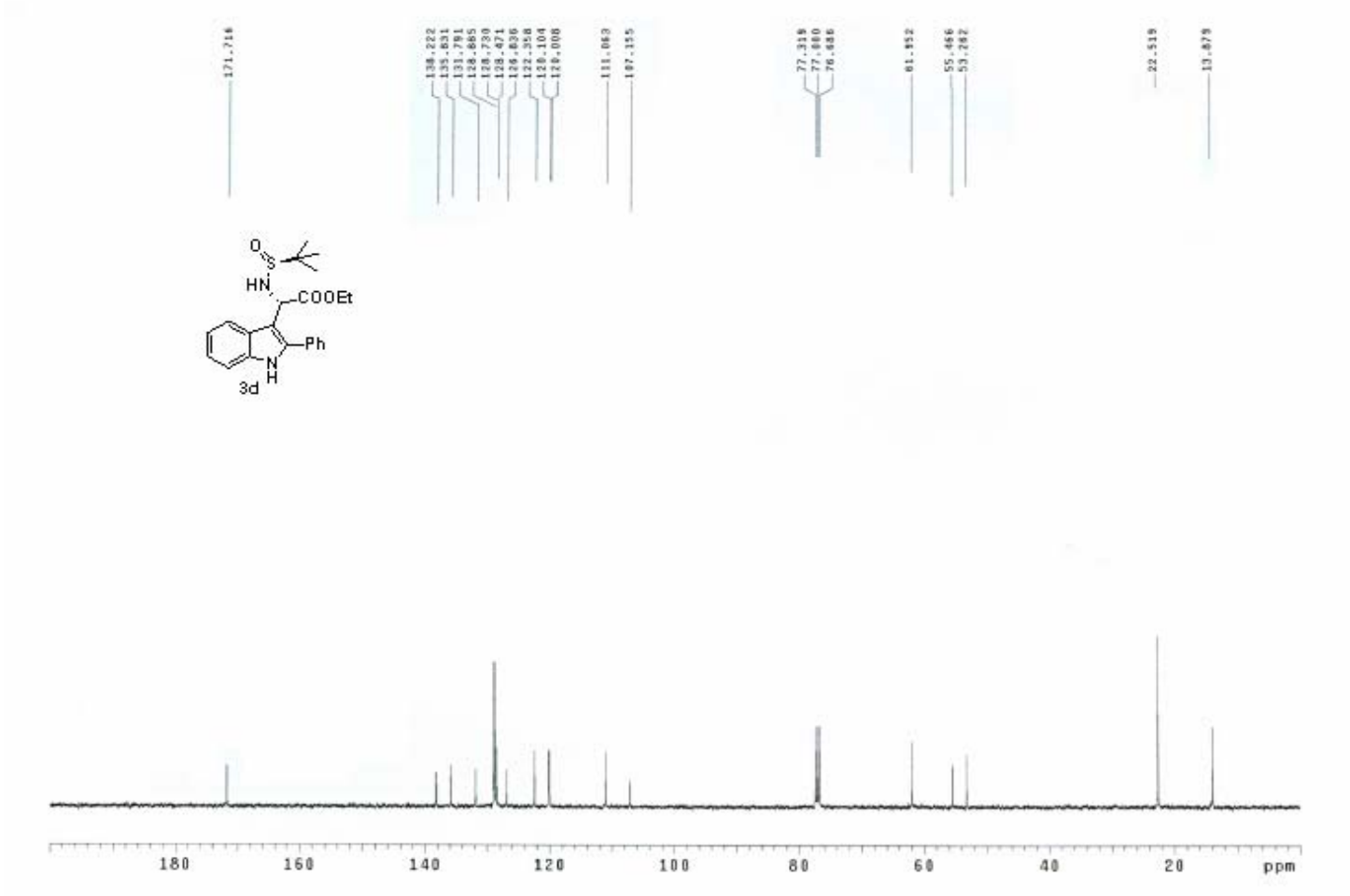


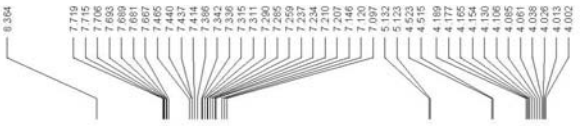


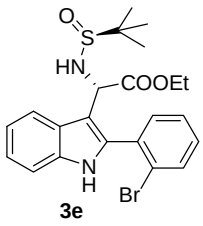


3d

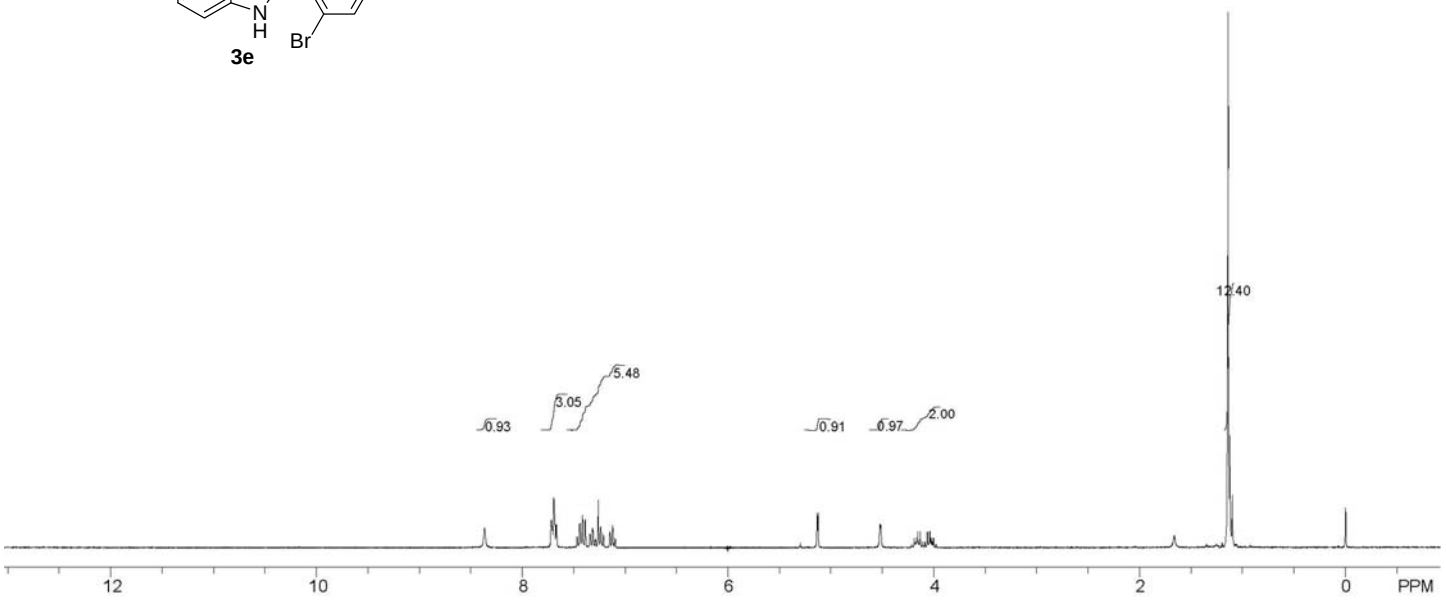


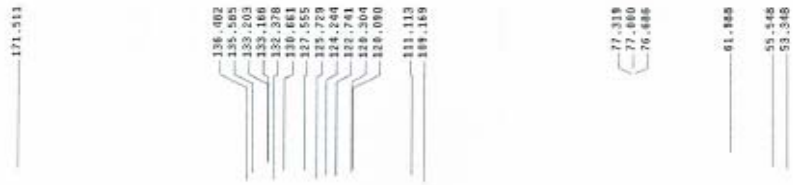


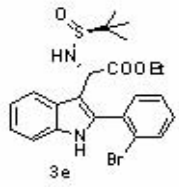




3e







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