Supporting Information

for

The Catalytic Asymmetric Direct α-Alkylation of Amino Esters by Aldehydes *via* Imine Activation

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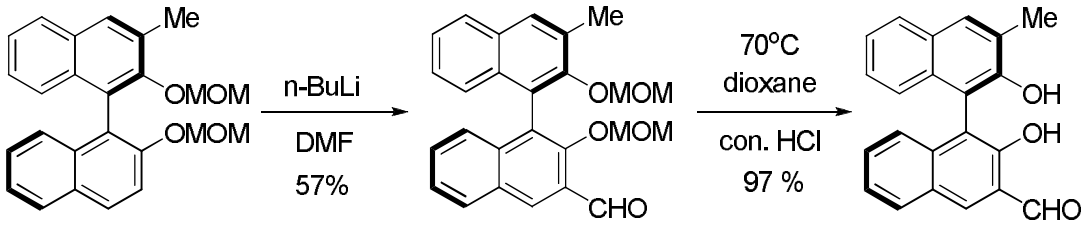
General data

Unless otherwise noted, commercial reagents were used as received and all reactions were carried out directly in air atmosphere. All reactions were monitored by TLC with silica gel coated plates. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance 300 spectrometer. HRMS (Bio TOF Q) spectra were recorded on P-SIMS-Gly of Bruker Daltonics Inc. Chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard. Proton signal multiplicities are given as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad) or a combination of them. *J*-values are in Hz. Enantiomer ratios were determined by HPLC with chiral columns (Chiralpak IA and AD-H were purchased from Daicel Industries).Optical rotations were measured on a Perkin Elmer 341 Polarimeter at $\lambda = 589$ nm. The 3-indolylmethanols [1] and α-amino esters [2] were prepared according to the literatures. The chiral aldehydes **4a-h**[3], **5a**[4], compounds **5.1b-j**[5-7] and **5k**[8]were prepare according to the reported methods.

1. The representative procedure for the preparation of catalyst.



5.1d **5.2d** **5d**

The preparation of 5.2d

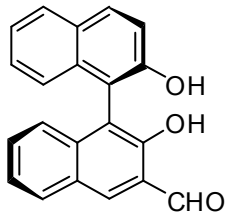
1.6 M *n*-BuLi in hexane (1.8 mL, 3 mmol) was added to a solution of **5.1d**（1.2 g, 3 mmol） in THF (20 mL) at -45 °C and the mixture was stirred for 5 minutes. Then the mixture was warmed to room temperature and stirred for 0.5 h. After cooled to -45°C, DMF (2.1 mL, 3 mmol) was added and the resulting solution was warmed to room temperature and stirred for additional 0.5 h. The reaction mixture was quenched by water, washed with saturated NH_4Cl and extracted with EtOAc. The combined organic phase was dried over Na_2SO_4 and concentrated under reduced pressure. The pure product **5.2d** (711 mg, 57%) was obtained through silica gel column chromatography separation.

The preparation of chiral aldehyde 5d

Concentrated HCl (1 mL) was added to a solution of **5.2d** (710 mg, 1.7 mmol) in dioxane (20

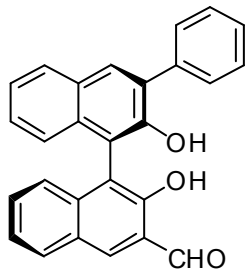
mL) slowly, and then the mixture was heated to 70 ºC and stirred for 2 h. The solvent was evaporated in vacuo. The residue was dissolved in EtOAc and washed with saturated $NaHCO_3$. The organic phase was dried over Na_2SO_4 and concentrated under reduced pressure. The crude residue was purified by silica gel chromatography, affording the pure compound **5d** in 97% yield (541 mg).

The chiral aldehydes **5b-j** were prepared in according to the following procedure of **5d**. The analytic data of **5a-j** were listed below:



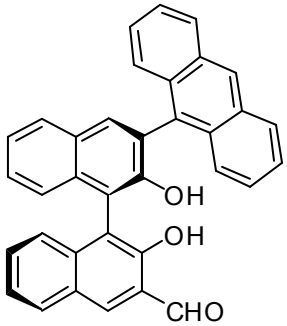
5a

5a was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +156.0 (c = 0.1, CH_3CH_2OH); mp 230 ^oC; ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.64 (s, 1H), 10.19 (s, 1H), 8.38 (s, 1H), 7.86-8.03 (m, 3H), 7.42-7.45 (m, 2H), 7.23-7.38 (m, 4H), 7.05-7.08 (m, 1H), 4.97 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 196.62, 154.26, 151.41, 139.10, 137.61, 133.31, 131.17, 130.40, 130.01, 129.18, 128.29, 127.70, 126.69, 124.98, 124.87, 124.36, 123.47, 122.02, 117.71, 115.05, 113.11; HRMS (ESI): calcd. for $C_{21}H_{14}NaO_3$(M^++Na): 337.0835, found: 337.0840.



5b

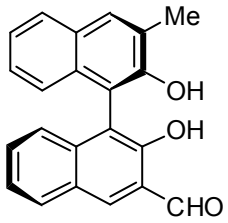
5b was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +57.0 (c = 0.1, CH_3CH_2OH); mp 117^oC; ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.64 (s, 1H), 10.19 (s, 1H), 8.37 (s, 1H), 7.88-8.02 (m, 3H), 7.70-7.73 (m, 2H), 7.25-7.52 (m, 8H), 7.08-7.11 (m, 1H), 5.22 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm)196.58, 154.16, 148.81, 138.84, 137.60, 137.47, 132.94, 130.99, 130.42, 129.95, 129.54, 129.12, 128.57, 128.33, 127.69, 126.66, 125.03, 124.73, 124.29, 123.82, 122.07, 115.95, 114.02; HRMS (ESI): calcd. for $C_{27}H_{19}O_3$ (M^++H): 391.1329, found: 391.1324.



5c

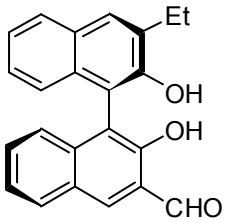
5c was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +67.0 (c = 0.1, CH_3CH_2OH); mp 253^oC; ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.64 (s, 1H),10.16 (s, 1H), 8.58 (s, 1H), 8.32 (s, 1H), 8.06-8.10 (m, 2H), 7.96-8.00 (m, 2H), 7.85-7.92 (m, 3H), 7.34-7.50 (m, 9H), 7.25-7.30 (m, 1H), 4.80 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ (ppm) 196.36, 154.44, 152.60, 137.71, 136.42,

134.11, 133.75, 132.27, 131.44, 131.40, 130.69, 130.60, 130.46, 128.76, 128.66, 128.62, 128.46, 127.88, 127.34, 126.87, 126.70, 126.06, 125.87, 125.58, 125.51, 124.72, 124.37, 123.76, 123.47, 116.91, 114.32, 79.52; HRMS (ESI): calcd. for $C_{35}H_{23}O_3$ (M^++H): 491.1642, found: 491.1656.



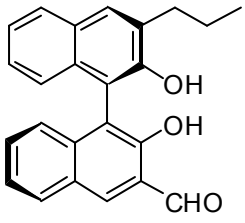
5d

5d was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +94.0 (c = 0.1, CH_3CH_2OH); mp 238°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.63 (s, 1H), 10.18 (s, 1H), 8.37 (s, 1H), 7.99-8.02 (m, 1H), 7.77-7.81 (m, 2H), 7.41-7.45 (m, 2H), 7.16-7.32 (m, 3H), 6.98-7.00 (m, 1H), 4.98 (s, 1H), 2.52 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 196.55, 154.36, 150.69, 139.11, 137.68, 132.05, 131.17, 129.99, 129.90, 129.17, 127.74, 127.50, 126.69, 125.67, 125.03, 124.88, 124.11, 123.39, 122.06, 115.16, 112.48, 17.01; HRMS (ESI): calcd. for $C_{22}H_{16}NaO_3$ (M^++Na): 351.0992, found: 351.0994.



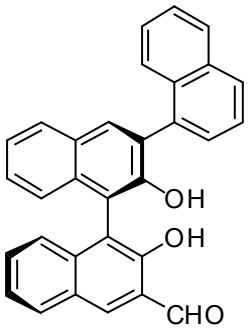
5e

5e was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +118.0 (c = 0.1, CH_3CH_2OH); mp 210°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.62 (s, 1H),10.21 (s, 1H), 8.39 (s, 1H), 8.00-8.03 (m, 1H), 7.82-7.84 (m, 1H), 7.78 (s, 1H),7.42-7.46 (m, 2H), 7.28-7.33 (m, 1H), 7.16-7.24 (m, 2H), 6.98-7.01 (m, 1H), 4.97 (s, 1H), 2.88-2.95 (q, *J* = 7.5 Hz, 2H), 1.40 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 196.51, 154.39, 150.39, 139.08, 137.70, 132.47, 131.89, 131.17, 129.98, 129.23, 128.03, 127.74, 127.70, 125.69, 125.03, 124.88, 124.04, 123.34, 122.08, 115.20 , 23.73, 13.67; HRMS (ESI): calcd. for $C_{23}H_{19}O_3$ (M^++H): 343.1329, found: 343.1339.



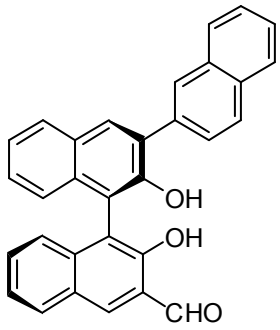
5f

5f was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +84.0 (c = 0.1, CH_3CH_2OH); mp 135°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.61 (s, 1H), 10.21 (s, 1H), 8.40 (s, 1H), 8.01-8.04 (m, 1H), 7.81-7.83 (m, 1H), 7.76 (s, 1H), 7.43-7.46 (m, 2H), 7.27-7.32 (m, 1H), 7.16-7.24 (m, 2H), 6.97-7.00 (m, 1H), 4.94 (s, 1H), 2.84-2.89 (m, 2H), 1.58-1.78 (m, 2H), 1.05 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 196.63, 154.43, 150.61, 139.16, 137.75, 132.07, 131.21, 131.17, 130.07, 129.23, 129.11, 127.79, 127.76, 125.76, 125.02, 124.88, 124.16, 123.41, 122.08, 115.29, 112.78, 32.91, 22.81, 14.17; HRMS (ESI): calcd. for $C_{24}H_{20}NaO_3$ (M^++Na): 379.1305, found: 379.1310.



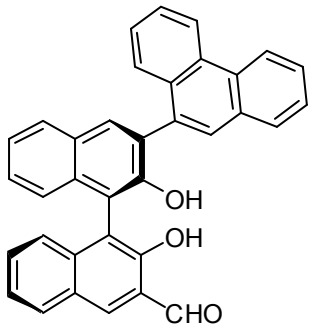
5g

5g was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +51.0 (c = 0.1, CH_3CH_2OH); mp 286°C; ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.63-10.65 (m, 1H), 10.17-10.17 (m, 1H), 8.33 (s, 1H), 7.78-8.01 (m, 6H), 7.57-7.71 (m, 2H), 7.29-7.54 (m, 7H), 7.18-7.25 (m, 1H), 4.98 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 196.61, 154.10, 149.51, 138.61, 137.67, 134.93, 133.68, 133.39, 132.07, 131.21, 130.81, 129.89, 128.97, 128.67, 128.27, 128.18, 127.88, 127.59, 126.72, 126.60, 126.18, 126.01, 125.69, 125.44, 125.16, 124.57, 124.47, 123.84, 122.09, 116.44, 113.96; HRMS (ESI): calcd. for $C_{31}H_{20}NaO_3$ (M^++Na): 463.1305, found: 463.1308.



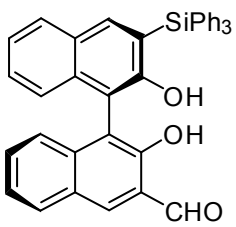
5h

5h was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +17.0 (c = 0.1, CH_3CH_2OH); mp 231°C; ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.67 (s, 1H), 10.22 (s, 1H), 8.39 (s, 1H), 8.19 (s, 1H), 8.01-8.07 (m, 2H), 7.84-7.98 (m, 5H), 7.42-7.54 (m, 4H), 7.29-7.40 (m, 3H), 7.11-7.14 (m, 1H), 5.30 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 196.60, 154.19, 148.98, 138.89, 137.61, 135.05, 133.41, 133.01, 132.66, 131.05, 130.73, 130.37, 129.98, 129.20, 128.37, 128.10, 128.07, 127.70, 127.62, 126.73, 126.25, 126.16, 125.03, 124.76, 124.32, 123.88, 122.07, 115.86, 114.13; HRMS (ESI): calcd. for $C_{31}H_{21}O_3$ (M^++H): 441.1485, found: 441.1484.



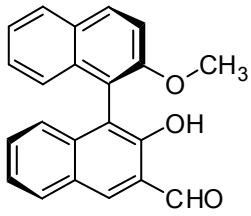
5i

5i was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +46.0 (c = 0.1, CH_3CH_2OH); mp 212°C; ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.66-10.68 (m, 1H), 10.20-10.21 (m, 1H), 8.74-8.81 (m, 2H), 8.37 (s, 1H), 7.83-8.06 (m, 6H), 7.33-7.72 (m, 9H), 7.19-7.22 (m, 1H), 5.01-5.02 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 196.60, 154.13, 149.67, 138.64, 137.66, 133.44, 131.38, 131.21, 130.86, 129.99, 129.89, 129.15, 128.83, 128.68, 128.28, 127.59, 127.03, 126.93, 126.82, 126.74, 126.56, 125.17, 124.93, 124.60, 124.49, 123.87, 122.96, 122.76, 122.51, 122.03, 116.20, 113.98; HRMS (ESI): calcd. for $C_{35}H_{23}O_3$ (M^++H): 491.1642, found: 491.1658.



5j

5j was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +32.0 (c = 0.1, $CHCl_3$); mp 244°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.62 (s, 1H), 10.18 (s, 1H), 8.35 (s, 1H), 7.98-8.00 (m, 1H), 7.89 (s, 1H), 7.68-7.74 (m, 7H), 7.33-7.47 (m, 11H), 7.28-7.29 (m, 3H), 7.10-7.12 (m, 1H), 5.08(s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 196.48, 155.28, 154.31, 141.24, 138.92, 137.54, 136.26, 134.75, 134.38, 131.02, 129.97, 129.39, 128.92, 128.84, 127.72, 127.47, 124.97, 124.81, 124.20, 123.34, 123.13, 122.06, 115.25, 112.78; HRMS (ESI): calcd. for $C_{39}H_{28}NaO_3Si$ (M^++Na): 595.1700, found: 595.1691.



5k

5k was obtained as a yellow solid after flash chromatography; $[\alpha]_D^{20}$ = +144.0 (c = 0.1, CH_3CH_2OH); mp 228°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 10.44 (s, 1H),10.19 (s, 1H), 8.31 (s, 1H), 7.96-8.03 (m, 2H), 7.87-7.90 (m, 1H), 7.46-7.50 (m, 1H), 7.31-7.40 (m, 3H), 7.23-7.28 (m, 1H), 7.12-7.17 (m, 2H), 3.80 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 196.55, 154.36, 150.69, 139.11, 137.68, 132.05, 131.17, 129.99, 129.90, 129.17, 127.74, 127.50, 126.69, 125.67, 125.03, 124.88, 124.11, 123.39, 122.06, 115.16, 112.48, 17.01; HRMS (ESI): calcd. for $C_{22}H_{16}NaO_3$ (M^++Na): 351.0992, found: 351.0992.

References:

1. Guo, Q.-X.; Peng, Y.-G.; Zhang, J.-W.; Song, L.; Feng, Z.; Gong, L.-Z. *Org. Lett*. **2009**, *11*, 4620.
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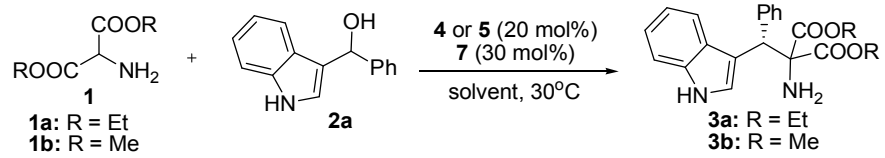
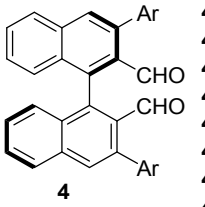
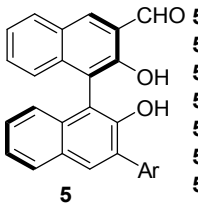
2. The optimization of reaction conditions.

Table S1: Screening of catalysts and the optimization of reaction conditions.[a]

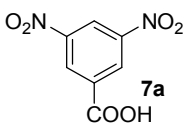


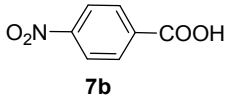


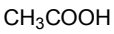
4a: Ar = Ph;
4b: Ar = 2-naphthyl;
4c: Ar = 4-^{t}BuC$_6$H$_4$;
4d: Ar = 3,5-2FC$_6$H$_3$
4e: Ar = 3,5-2MeOC$_6$H$_3$;
4f: Ar = 2-MeC$_6$H$_4$
4g: Ar = 3-MeC$_6$H$_4$;
4h: Ar = 4-MeC$_6$H$_4$

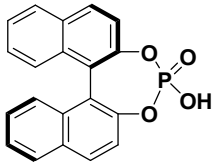


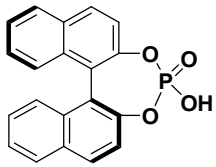
5a: Ar = H; **5b**: Ar = Ph;
5c: Ar = 9-Anthracene;
5d: Ar = Me; **5e**: Ar = Et;
5f: Ar = ^{n}-Pr; **5g**: Ar = 1-Naphthyl
5h: Ar = 2-Naphthyl;
5i: Ar = 9-Phenathrene;
5j: Ar = Ph$_3$Si

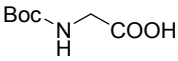


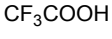


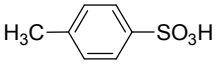












7g

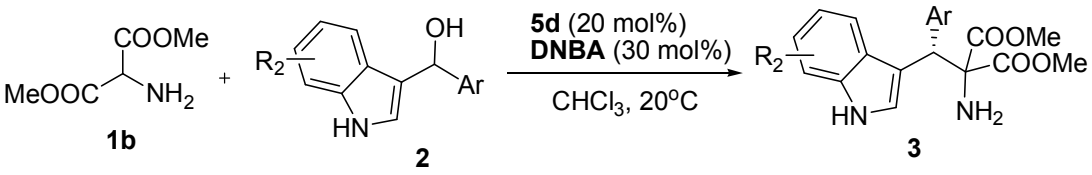
7h

entry	Cat.	1	7	solvent	time (h)	yield (%)[b]	ee (%)[c]
1	**4a**	**1a**	**7a**	$CHCl_3$	24	24	50
2	**4b**	**1a**	**7a**	$CHCl_3$	30	37	55
3	**4c**	**1a**	**7a**	$CHCl_3$	40	42	64
4	**4d**	**1a**	**7a**	$CHCl_3$	30	32	66
5	**4e**	**1a**	**7a**	$CHCl_3$	40	32	52
6	**4f**	**1a**	**7a**	$CHCl_3$	72	24	48
7	**4g**	**1a**	**7a**	$CHCl_3$	72	37	54
8	**4h**	**1a**	**7a**	$CHCl_3$	72	29	56
9	**5a**	**1a**	**7a**	$CHCl_3$	4	66	71
10	**5b**	**1a**	**7a**	$CHCl_3$	6	57	81
11	**5b**	**1b**	**7a**	$CHCl_3$	4	57	82
12	**5b**	**1b**	**7a**	$CHCl_3$	4	65	84[d]
13	**5b**	**1b**	**7b**	$CHCl_3$	30	74	73[d]
14	**5b**	**1b**	**7c**	$CHCl_3$	42	71	65[d]
15	**5b**	**1b**	**7d**	$CHCl_3$	17	81	44[d]
16	**5b**	**1b**	**7e**	$CHCl_3$	65	70	13[d]
17	**5b**	**1b**	**7f**	$CHCl_3$	73	28	39[d]
18	**5b**	**1b**	**7g**	$CHCl_3$	48	48	24[d]
19	**5b**	**1b**	**7h**	$CHCl_3$	48	37	13[d]

20	**5b**	**1b**	**7a**	CH_2Cl_2	15	48	81[d]
21	**5b**	**1b**	**7a**	toluene	15	51	76[d]
22	**5b**	**1b**	**7a**	Et_2O	15	NR[e]	NR
23	**5b**	**1b**	**7a**	benzene	9	41	75[d]
24	**5b**	**1b**	**7a**	$ClCH_2CH_2Cl$	9	31	75[d]
25	**5b**	**1b**	**7a**	CCl_4	9	14	67[d]
26	**5b**	**1b**	**7a**	THF	9	NR	NR
27	**5c**	**1a**	**7a**	$CHCl_3$	5	61	77
28	**5d**	**1b**	**7a**	$CHCl_3$	6	64	85
29	**5e**	**1b**	**7a**	$CHCl_3$	6	60	84[d]
30	**5f**	**1b**	**7a**	$CHCl_3$	6	65	83[d]
31	**5g**	**1a**	**7a**	$CHCl_3$	5	68	77
32	**5h**	**1a**	**7a**	$CHCl_3$	5	62	77
33	**5i**	**1a**	**7a**	$CHCl_3$	5	55	76
34	**5j**	**1a**	**7a**	$CHCl_3$	9	53	82
35	**5d**	**1b**	**7a**	$CHCl_3$	6	77	86[d]
36	**5d**	**1b**	**7a**	$CHCl_3$	6	72	87[f]
37	**5d**	**1b**	**7a**	$CHCl_3$	9	68	86[g]
38	**5d**	**1b**	**7a**	$CHCl_3$	12	55	85[h]

[a] for entries 1-8: **1** (0.2 mmol), **2a** (0.1 mmol), **4** or **5** (0.02 mmol), $CHCl_3$ (1 mL), 30°C; for entries 9-38: **1** (0.4 mmol), **2a** (0.2 mmol),**4 or 5** (0.04 mmol), $CHCl_3$ (2 mL), 30°C. [b] Yield of isolated product. [c] determined by chiral HPLC analysis. [d] at 20°C. [e] NR = no reaction. [f] Using 50 mol% DNBA. [g] Using 15 mol% **5d**. [h] Using 10 mol% **5d**.

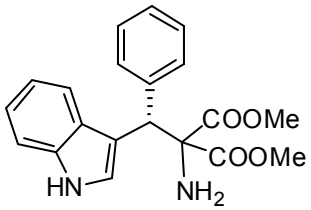
3. The general procedure for the Asymmetric reaction.



General procedure:

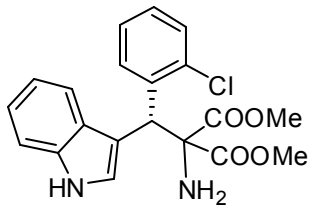
A reaction tube was charged with catalyst **5d** (13.1 mg, 0.04 mmol), **DNBA** (12.7 mg, 0.06 mmol), **1b** (47 uL, 0.4 mmol), **2** (0.2 mmol) and chloroform (2 mL).The solution was stirred at 20°C until the reaction completed (monitored by TLC). One drop of Et_3N was added to the mixture. The solvent was concentrated and the residue was subjected to silica gel column chromatography to afford the desired products **3** with using ethyl acetate/petroleum as eluents. The analytic data of

pure **3b-3u** were listed below:



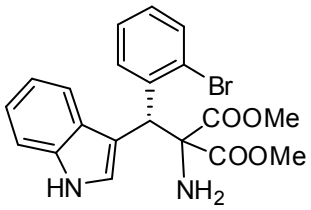
3b

3b was obtained as a white solid (54 mg, 77%); the enantiomeric excess was determined to be 86% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 80/20, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 14.78 min, t_R (minor) 20.41 min; $[\alpha]_D^{20}$ = +76.0 (c = 0.1, CH_3CH_2OH); mp 155°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.07 (s, 1H), 7.76 (m, 1H), 7.45-7.50 (m, 3H), 7.29-7.31 (m, 1H), 7.09-7.23 (m, 4H), 6.99-7.20 (m, 1H), 5.46 (s, 1H), 3.71 (s, 3H), 3.52 (s, 3H), 2.09 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.54, 170.81, 139.49, 135.49, 129.53, 128.21, 127.37, 127.06, 123.50, 121.82, 119.21, 118.83, 114.34, 111.02, 70.96, 53.24, 53.19, 46.92; HRMS(ESI): calcd. for $C_{20}H_{21}N_2O_4$ (M^++H): 353.1496, found: 353.1497.



3c

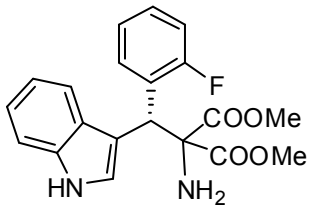
3c was obtained as a white solid (53 mg, 68%); the enantiomeric excess was determined to be 92% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 7.00 min, t_R (minor) 8.16 min; $[\alpha]_D^{20}$ = -40.0 (c = 0.1, CH_3CH_2OH); mp 131°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.06-8.12 (m, 2H), 7.66-7.69 (m, 1H), 7.57-7.58 (m, 1H), 7.29-7.31 (m, 2H), 7.05-7.18 (m, 4H), 5.97 (s, 1H), 3.57 (s, 3H), 3.67 (s, 3H), 2.37 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.43, 170.99, 138.28, 135.34, 134.15, 130.73, 129.29, 127.87, 127.21, 126.62, 123.81, 121.96, 119.45, 119.06, 114.12, 111.02, 69.72, 53.31, 53.22, 41.71; HRMS(ESI): calcd. for $C_{20}H_{20}ClN_2O_4$ (M^++H): 387.1106, found: 387.1109.



3d

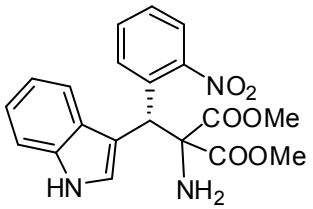
3d was obtained as a red solid (55 mg, 64%); the enantiomeric excess was determined to be 96% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 90/10, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 22.34 min, t_R (minor) 31.25min; $[\alpha]_D^{20}$ = -36.0 (c = 0.1, CH_3CH_2OH); mp 118°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.18-8.21 (m, 1H), 8.09 (s, 1H), 7.73-7.75 (m, 1H), 7.48-7.51 (m, 2H), 7.00-7.29 (m, 5H),5.95 (s, 1H), 3.66 (s, 3H), 3.57 (s, 3H), 2.16 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm)171.54, 170.89, 140.22, 135.37, 132.74, 130.65, 128.17, 127.28, 127.17, 125.36, 124.08, 121.94, 119.45, 119.32, 113.84, 111.11, 69.78, 53.37, 53.24, 44.62; HRMS(ESI): calcd.

for $C_{20}H_{20}BrN_2O_4$ (M^++H): 431.0601, found: 431.0601.



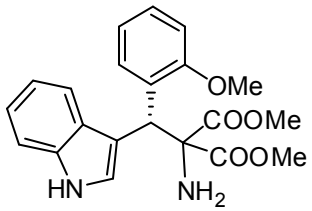
3e

3e was obtained as a white solid (46 mg, 62%); the enantiomeric excess was determined to be 91% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 80/20, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 8.70 min, t_R (minor) 10.27 min; $[\alpha]_D^{20}$ = +8.0 (c = 0.1, CH_3CH_2OH); mp 131°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.13 (s, 1H), 7.74-7.79 (m, 1H), 7.66 (m, 1H), 7.56-7.59 (m, 1H), 7.24-7.27 (m, 1H), 6.94-7.16 (m, 5H),5.80 (s, 1H), 3.70 (s, 3H), 3.52 (s, 3H), 2.26 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.27, 170.91, 162.03, 158.78, 135.28, 131.30, 128.35, 127.19, 123.76, 123.47, 121.98, 119.39, 118.70, 115.02, 114.27, 110.85, 69.91, 53.23, 53.16, 37.90; HRMS (ESI): calcd. for $C_{20}H_{20}FN_2O_4$ (M^++H): 371.1402, found: 371.1397.



3f

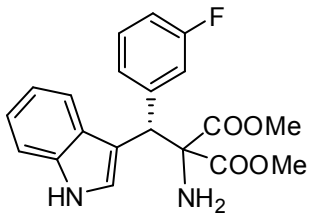
3f was obtained as a yellow solid (39 mg, 49%); the enantiomeric excess was determined to be 95% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 7.18 min, t_R (minor) 8.25 min; $[\alpha]_D^{20}$ = -156.0 (c = 0.1, CH_3CH_2OH); mp 164°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.35 (d, J = 9 Hz, 1H), 8.13 (s, 1H), 7.84 (d, J = 9 Hz, 1H), 7.67-7.72 (m, 2H), 7.44-7.49 (m, 1H), 7.30-7.33 (m, 2H), 7.11-7.19 (m, 2H), 6.19 (s, 1H), 3.63 (s, 3H), 3.52 (s, 3H), 2.35 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 170.83, 170.72, 150.44, 135.26, 134.29, 131.91, 131.87, 127.50, 127.00, 124.11, 124.08, 122.16, 119.71, 119.35, 112.94, 110.99, 69.97, 53.46, 53.44, 39.16; HRMS (ESI): calcd. for $C_{20}H_{20}N_3O_6$ (M^++H): 398.1347, found: 398.1332.



3g

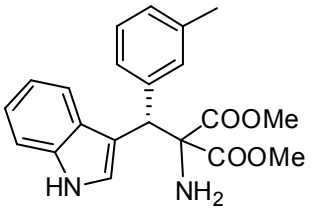
3g was obtained as a white solid (45 mg, 59%); the enantiomeric excess was determined to be 82% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 7.63 min, t_R (minor) 12.62 min; $[\alpha]_D^{20}$ = +55.0 (c = 0.1, CH_3CH_2OH); mp 179°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.03 (s, 1H), 7.64-7.68 (m, 2H), 7.52-7.55 (m, 1H), 7.26-7.29 (m, 1H), 6.99-7.13 (m, 3H), 6.80-6.84 (m, 2H), 5.91 (s, 1H), 3.86 (s, 3H), 3.66 (s, 3H), 3.52 (s, 3H), 2.05 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.63, 171.45, 156.83, 135.32, 130.63, 128.01, 127.82, 127.51, 123.34, 121.69, 120.27, 119.07, 118.98, 115.63, 110.71, 110.01, 69.69, 55.59, 52.95, 52.87, 37.92;

HRMS (ESI): calcd. for $C_{21}H_{23}N_2O_5(M^+H)$: 383.1601, found: 383.1604.



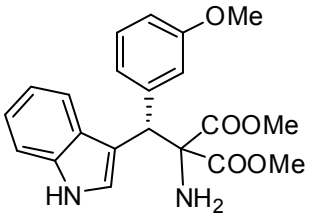
3h

3h was obtained as a red solid (65 mg, 88%); the enantiomeric excess was determined to be 87% by HPLC analysis on Daicel Chirapak AD-H column(hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 7.81 min, t_R (minor) 9.58 min; $[\alpha]_D^{20}$ = +49.0 (c = 0.1, CH_3CH_2OH); mp 40°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.16 (s, 1H), 7.70-7.71 (m, 1H), 7.49-7.51 (m, 1H), 7.10-7.30 (m, 5H), 7.01-7.06 (m, 1H), 6.83-6.89 (m, 1H), 5.45 (s, 1H), 3.72 (s, 3H), 3.50 (s, 3H), 2.26 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.18, 170.54, 164.19, 160.94, 142.34, 135.42, 129.49, 127.19, 125.15, 123.44, 121.98, 119.36, 118.66, 116.64, 114.03, 110.98, 70.73, 53.25, 53.19, 46.48; HRMS (ESI): calcd. for $C_{20}H_{20}FN_2O_4$ (M^+H): 371.1402, found: 371.1394.



3i

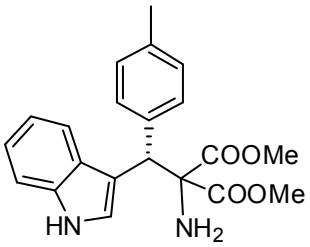
3i was obtained as a white solid (31 mg, 42%); the enantiomeric excess was determined to be 84% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 6.33 min, t_R (minor) 9.85 min; $[\alpha]_D^{20}$ = +61.0 (c = 0.1, CH_3CH_2OH); mp 142°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.13 (s, 1H), 7.75 (m, 1H), 7.48-7.51 (m, 1H), 7.23-7.29 (m, 3H), 7.09-7.14 (m, 2H), 6.96-7.04 (m, 2H), 5.43 (s, 1H), 3.71 (s, 3H), 3.50 (s, 3H), 2.26 (s, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.62, 170.73, 139.26, 137.61, 135.47, 130.19, 128.03, 127.84, 127.43, 126.48, 123.41, 121.77, 119.16, 118.85, 114.50, 110.90, 70.97, 53.17, 53.09, 46.82, 21.51; HRMS(ESI): calcd. for $C_{21}H_{23}N_2O_4$ (M^+H): 367.1652, found: 367.1642.



3j

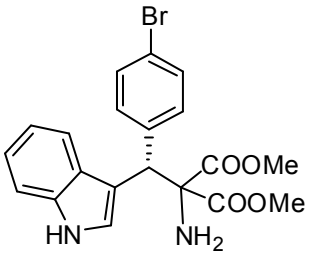
3j was obtained as a white solid (59 mg, 77%); the enantiomeric excess was determined to be 87% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 7.78 min, t_R (minor) 11.73 min; $[\alpha]_D^{20}$ = +63.0 (c = 0.1, CH_3CH_2OH); mp 144°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.30 (s, 1H), 7.68 (s, 1H), 7.49-7.52 (m, 1H), 7.21-7.23 (m, 1H), 6.98-7.15 (m, 5H), 6.68-6.70 (m, 1H), 5.44 (s, 1H), 3.69 (s, 6H), 3.47 (s, 3H), 2.22 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.53, 170.65, 159.28, 141.05, 135.44, 129.07, 127.37, 123.42, 121.90, 121.79, 119.18, 118.80, 115.65, 114.34, 111.95, 110.90, 70.90, 55.05, 53.19, 53.16,

46.78; HRMS (ESI): calcd. for $C_{21}H_{23}N_2O_5$ (M^++H): 383.1601, found: 383.1587.



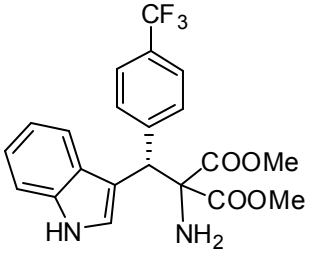
3k

3k was obtained as a white solid (39 mg, 53%); the enantiomeric excess was determined to be 77% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 8.45 min, t_R (minor) 10.55 min; $[\alpha]_D^{20}$ = +58.0 (c = 0.1, CH_3CH_2OH); mp 48°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.19 (s, 1H), 7.69-7.70 (m, 1H), 7.46-7.48 (m, 1H), 7.31-7.34 (m, 2H), 7.23 (m, 1H), 6.97-7.11 (m, 4H), 5.44 (s, 1H), 3.71 (s, 3H), 3.49 (s, 3H), 2.24 (s, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.61, 170.75, 136.51, 136.27, 135.48, 129.34, 128.90, 127.39, 123.28, 121.76, 119.15, 118.86, 114.64, 110.87, 70.96, 53.13, 53.11, 46.50, 20.98; HRMS (ESI): calcd. for $C_{21}H_{23}N_2O_4$ (M^++H): 367.1652, found: 367.1654.



3l

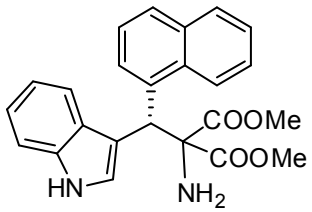
3l was obtained as a white solid (65 mg, 76%); the enantiomeric excess was determined to be 87% by HPLC analysis on Daicel Chirapak AD-H column(hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 9.03 min, t_R (minor) 10.24 min; $[\alpha]_D^{20}$ = +21.0 (c = 0.1, CH_3CH_2OH); mp 84°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.13 (s, 1H), 7.66-7.67 (m, 1H), 7.45-7.48 (m, 1H), 7.25-7.40 (m, 5H), 7.10-7.15 (m, 1H), 7.00-7.05 (m, 1H),5.41 (s, 1H), 3.71 (s, 3H), 3.51 (s, 3H), 2.23 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.08, 170.62, 138.77, 135.49, 131.33, 131.24, 127.10, 123.46, 122.02, 121.02, 119.39, 118.67, 113.85, 111.09, 70.64, 53.35, 53.31, 53.27, 46.31; HRMS (ESI): calcd. for $C_{20}H_{20}BrN_2O_4$ (M^++H): 431.0601, found: 431.0590.



3m

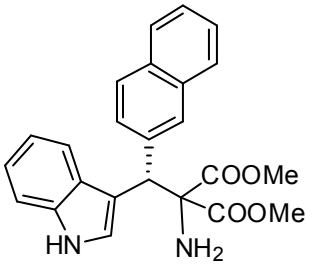
3m was obtained as a red solid (64 mg, 76%); the enantiomeric excess was determined to be 88% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 80/20, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 7.98 min, t_R (minor) 9.96 min; $[\alpha]_D^{20}$ = +20.0 (c = 0.1, CH_3CH_2OH); mp 48°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.13 (s, 1H),7.65-7.70 (m, 3H), 7.47-7.49 (m, 3H), 7.30-7.33 (m, 1H), 7.12-7.17 (m, 1H), 7.02-7.07 (m, 1H), 5.51 (s, 1H), 3.71 (s, 3H), 3.53 (s, 3H), 2.27 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 170.95, 170.55, 143.93, 135.42, 129.89, 127.04, 124.96, 123.46, 122.11, 119.48, 118.59, 113.78, 111.02, 70.54, 53.34, 53.26, 46.53;

HRMS (ESI): calcd. for $C_{21}H_{20}F_3N_2O_4$ (M^++H): 421.1370, found: 421.1364.



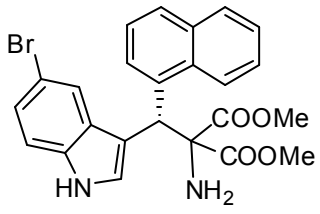
3n

3n was obtained as a white solid (54 mg, 67%); the enantiomeric excess was determined to be 89% by HPLC analysis on Daicel Chirapak AD-H column(hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 9.09 min, t_R (minor) 11.42 min; $[\alpha]_D^{20}$ = -44.0 (c = 0.1, CH_3CH_2OH); mp 85°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.35-8.38 (m, 1H), 8.19-8.21 (m, 1H), 8.03 (s, 1H), 7.79-7.82 (m, 1H), 7.68-7.70 (m, 1H), 7.28-7.58 (m, 6H), 7.09-7.14 (m, 1H), 6.99-7.04 (m, 1H), 6.31 (s, 1H), 3.66 (s, 3H), 3.35 (s, 3H), 2.37 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.81, 171.06, 136.87, 135.65, 133.86, 131.77, 128.94, 127.39, 127.01, 126.31, 126.00, 125.30, 125.22, 124.52, 123.30, 121.87, 119.42, 118.88, 114.65, 111.20, 70.27, 53.43, 52.95, 40.83; HRMS (ESI): calcd. for $C_{24}H_{23}N_2O_4$ (M^++H): 403.1652, found: 403.1656.



3o

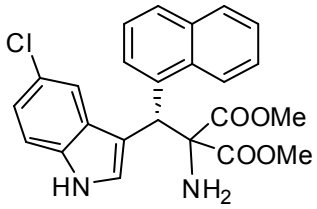
3o was obtained as a white solid (71 mg, 88%) yield after flash chromatography and the enantiomeric excess was determined to be 80% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 10.00 min, t_R (minor) 13.80 min; $[\alpha]_D^{20}$ = +29.0 (c = 0.1, CH_3CH_2OH); mp 61°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.22 (s, 1H), 7.96 (s, 1H), 7.67-7.76 (m, 4H), 7.49-7.60 (m, 2H), 7.35-7.40 (m, 2H), 7.20-7.24 (m, 1H), 7.04-7.09 (m, 1H), 6.95-7.00 (m, 1H), 5.65 (s, 1H), 3.66 (s, 3H), 3.52 (s, 3H), 2.27 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.49, 170.88, 137.22, 135.57, 133.30, 132.52, 128.25, 128.01, 127.82, 127.77, 127.53, 127.38, 125.91, 125.74, 123.64, 121.91, 119.30, 118.89, 114.33, 111.04, 71.08, 53.34, 53.24, 47.04; HRMS (ESI): calcd. for $C_{24}H_{23}N_2O_4$ (M^++H): 403.1652, found: 403.1655.



3p

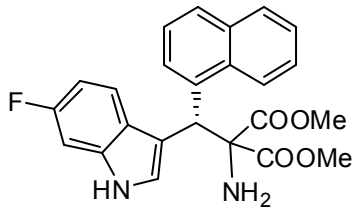
3p was obtained as a yellow solid (83 mg, 86%); the enantiomeric excess was determined to be 82% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 5.79 min, t_R (minor) 7.05 min; $[\alpha]_D^{20}$ = -85.0 (c = 0.1, CH_3CH_2OH); mp 86°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.22-8.30 (m, 2H), 8.07 (s, 1H), 7.69-7.83 (m, 3H), 7.37-7.53 (m,

4H), 7.14-7.23 (m, 2H), 6.22 (s, 1H), 3.70 (s, 3H), 3.37 (s, 3H), 2.28 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.76, 170.82, 136.53, 134.18, 133.86, 131.61, 128.98, 128.56, 127.53, 126.20, 126.00, 125.35, 125.23, 124.73, 123.08, 121.21, 114.16, 112.83, 112.75, 70.07, 53.56, 53.02, 40.56; HRMS (ESI): calcd. for $C_{24}H_{22}BrN_2O_4$ (M^++H): 481.0757, found: 481.0762.



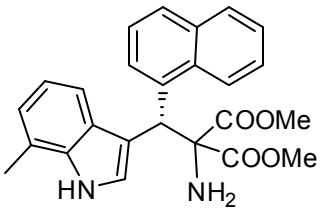
3q

3q was obtained as a yellow solid (73 mg, 84%); the enantiomeric excess was determined to be 79% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 5.85 min, t_R (minor) 7.00 min; $[\alpha]_D^{20}$ = -66.0 (c = 0.1, CH_3CH_2OH); mp 175°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.21-8.28 (m, 2H), 8.11 (s, 1H), 7.76-7.78 (m, 1H), 7.66-7.68 (m, 1H), 7.57 (s, 1H), 7.33-7.48 (m, 4H), 6.99-7.05 (m, 2H), 6.21 (s, 1H), 3.66 (s, 3H), 3.33 (s, 3H), 2.34 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm)171.68, 170.83, 136.43, 133.89, 133.85, 131.62, 128.96, 127.98, 127.52, 126.15, 126.12, 126.00, 125.31, 125.20, 123.06, 122.20, 118.19, 114.44, 112.22, 70.08, 53.48, 52.98, 40.53; HRMS (ESI): calcd. for $C_{24}H_{22}ClN_2O_4$ (M^++H): 437.1263, found: 437.1267.



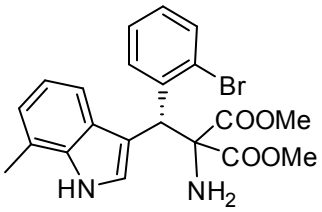
3r

3r was obtained as a white solid (41 mg, 49%); the enantiomeric excess was determined to be 94% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 7.90 min, t_R (minor) 9.24 min; $[\alpha]_D^{20}$ = -16.0 (c = 0.1, CH_3CH_2OH); mp 216°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.34-8.37 (m, 1H), 8.13-8.16 (m, 1H), 8.01 (s, 1H), 7.81-7.83 (m, 1H), 7.69-7.71 (m, 1H), 7.39-7.55 (m, 5H), 6.95-6.58 (m, 1H), 6.74-6.80 (m, 1H), 6.26 (s, 1H), 3.66 (s, 3H), 3.35 (s, 3H), 2.28 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.45, 170.88, 161.35, 158.21, 136.19, 133.81, 131.64, 128.95, 127.49, 126.53, 125.96, 125.23, 124.42, 123.04, 119.72, 115.30, 108.36, 108.03, 97.43, 97.09, 70.13, 53.36, 52.91, 40.58; HRMS (ESI): calcd. for $C_{24}H_{22}FN_2O_4$ (M^++H): 421.1558, found: 421.1562.



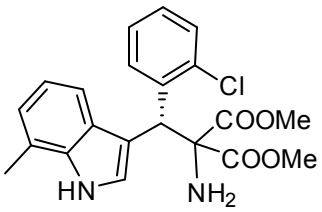
3s

3s was obtained as a white solid (27 mg, 33%); the enantiomeric excess was determined to be 92% by HPLC analysis on Daicel Chirapak AD-H column(hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 6.49 min, t_R (minor) 8.15 min; $[\alpha]_D^{20}$ = -33.0 (c = 0.1, CH_3CH_2OH); mp 166°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.33-8.35 (m, 1H), 8.20-8.22 (m, 1H), 7.90 (s, 1H), 7.76-7.79 (m, 1H), 7.65-7.68 (m, 1H), 7.34-7.47 (m, 5H), 6.87-6.96 (m, 2H), 6.28 (s, 1H), 3.64 (s, 3H), 3.33 (s, 3H), 2.32 (s, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm)171.75, 170.99, 136.90, 135.15, 133.83, 131.75, 128.88, 127.28, 126.56, 126.25, 125.91, 125.28, 125.13, 124.12, 123.29, 122.43, 120.20, 119.62, 116.64, 115.28, 70.18, 53.39, 52.88, 40.87, 16.42; HRMS (ESI): calcd. for $C_{25}H_{25}N_2O_4$ (M^++H): 417.1809, found: 417.1809.



3t

3t was obtained as a white solid (38 mg, 43%); the enantiomeric excess was determined to be 98% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 80/20, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 7.50 min, t_R (minor) 9.78 min; $[\alpha]_D^{20}$ = -23.0 (c = 0.1, CH_3CH_2OH); mp 162°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.19-8.21 (m, 1H), 8.03 (s, 1H), 7.57-7.60 (m, 1H), 7.42-7.48 (m, 2H), 7.16-7.23 (m, 1H), 6.86-7.02 (m, 4H), 5.93 (s, 1H), 3.64 (s, 3H), 3.57 (s, 3H), 2.37 (s, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.51, 170.85, 140.24, 134.91, 132.68, 130.63, 128.07, 127.22, 126.73, 125.32, 123.67, 122.51, 120.11, 119.66, 117.10, 114.49, 69.71, 53.34, 53.18, 44.70, 16.46; HRMS (ESI): calcd. for $C_{21}H_{22}BrN_2O_4$ (M^++H): 445.0757, found: 445.0760.

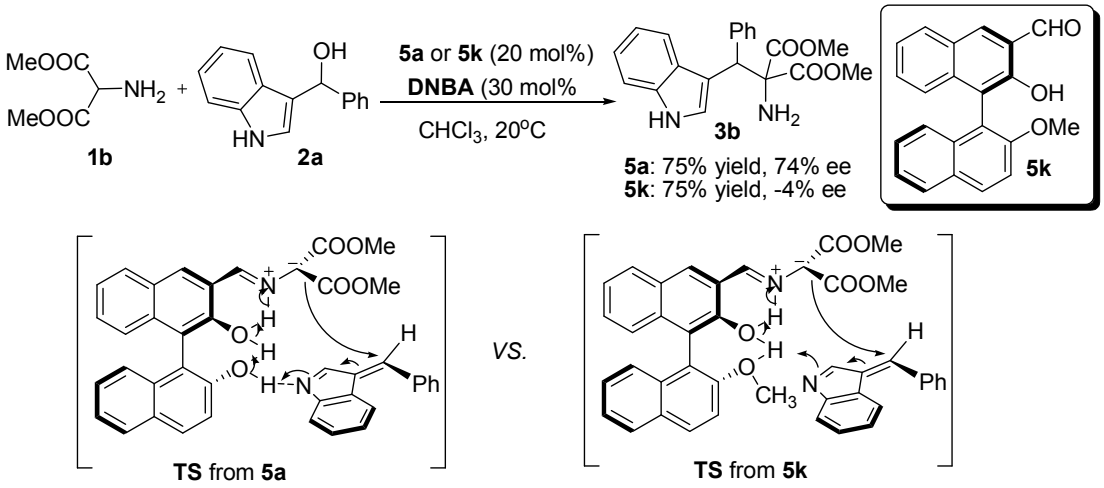


3u

3u was obtained as a white solid (54 mg, about 67%); the enantiomeric excess was determined to be 95% by HPLC analysis on Daicel Chirapak AD-H column(hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 5.89 min, t_R (minor) 7.16 min; $[\alpha]_D^{20}$ = -12.0 (c = 0.1, CH_3CH_2OH); mp 58°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.11-8.14 (m, 1H), 8.02 (s, 1H), 7.52-7.54 (m, 2H), 7.29-7.31 (m, 1H), 6.93-7.18 (m, 4H), 5.97 (s, 1H), 3.67 (s, 3H), 3.59 (s, 3H), 2.42 (s, 3H), 2.16 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 171.35, 170.93, 138.22, 134.87, 134.11, 130.72, 129.23, 127.78, 126.77, 126.56, 123.34, 122.54, 119.99, 119.66, 116.86, 114.85, 69.64, 53.29, 53.15,

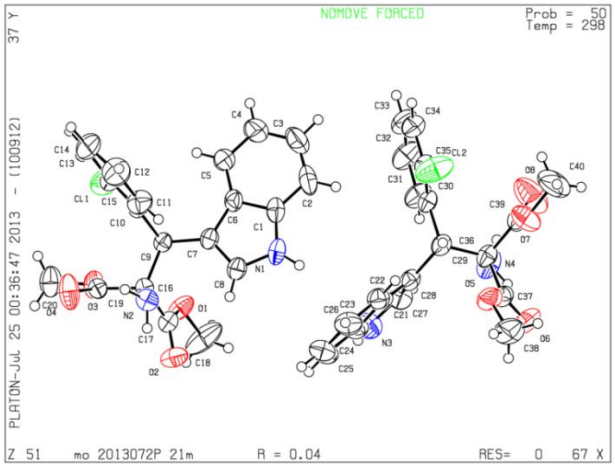
41.81, 16.48; HRMS (ESI): calcd. for $C_{21}H_{22}ClN_2O_4$ (M^++H): 401.1263, found: 401.1260.

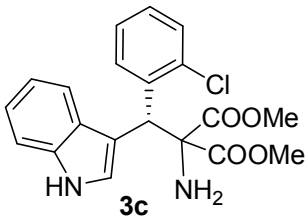
4. Control experiment for mechanism investigation.



5. Determination of the absolute configuration of compound 3c.

The absolute configuration of **3c** was established by X-ray single crystal analysis, using the anomalous dispersion of Cl atom.



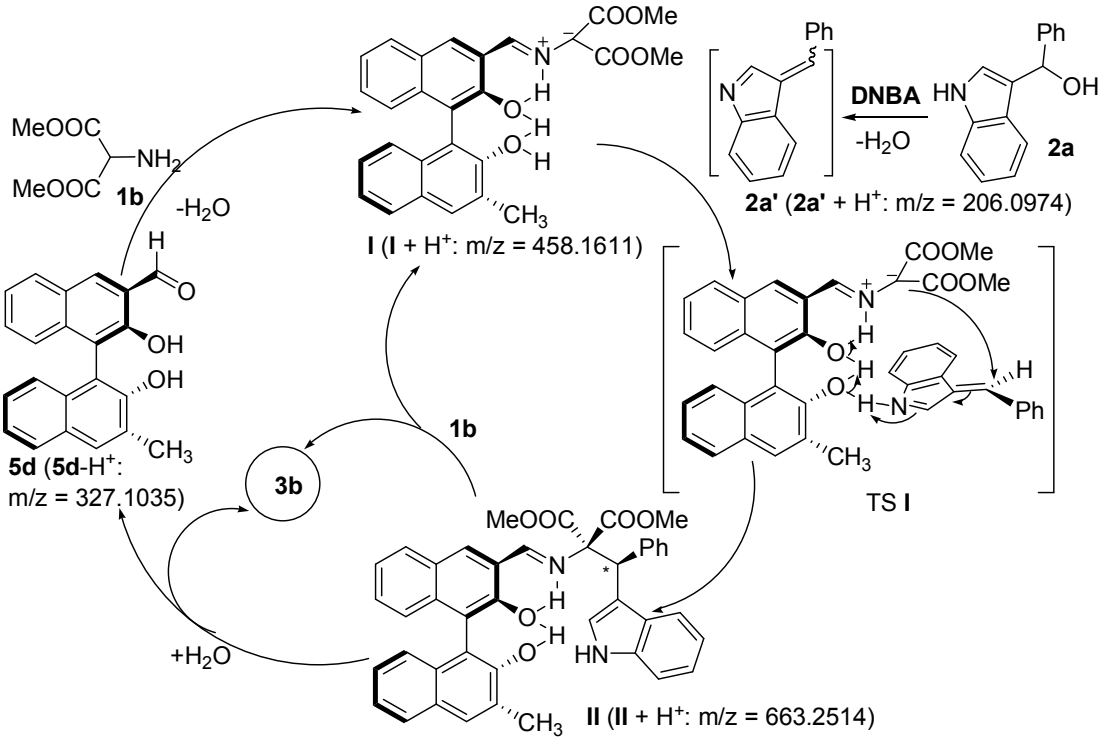


(*R*)-dimethyl 2-amino-2-((2-chloro-phenyl)(1*H*-indol-3-yl)methyl)malonate

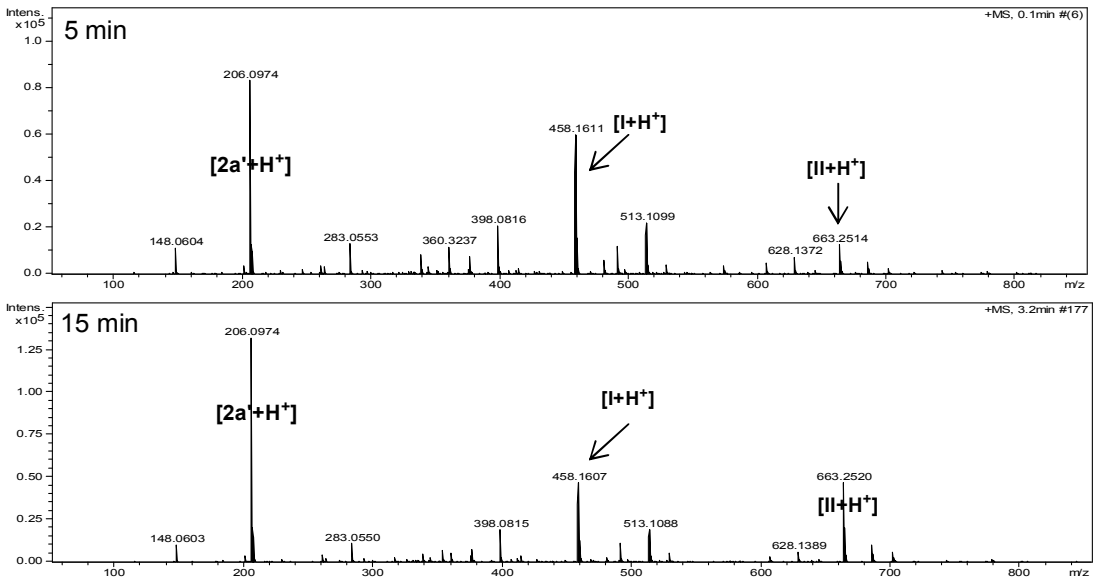
Chemical Formula	$C_{20}H_{19}ClN_2O_4$
Formula weight	386.82
Temperature	298(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P 21
a, Å	11.1925(12)
b, Å	8.1206(9)
c, Å	21.465(2)
α, °	90.00

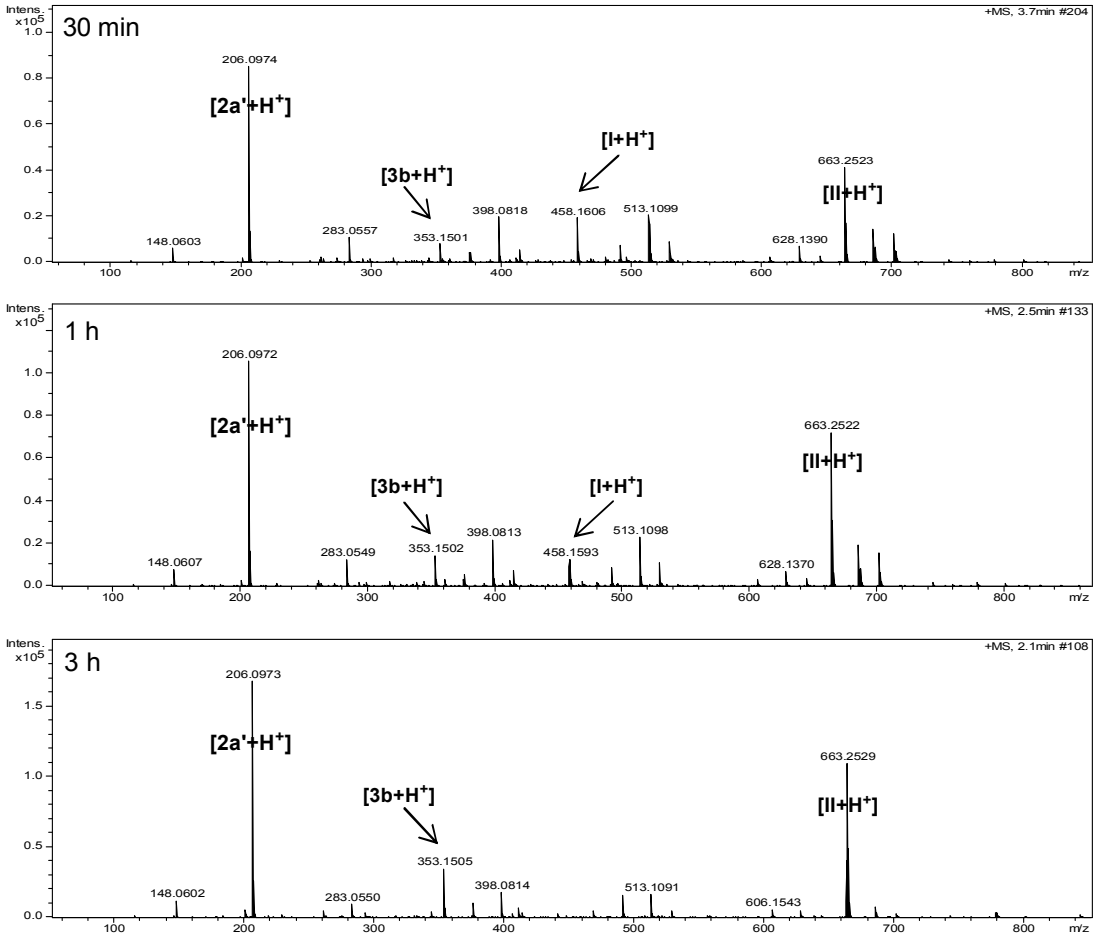
β, °	101.402(3)
γ, °	90.00
V, Å^3	1912.5(4)
Flack	-0.01(7)
Z, Calculated density	4, 1.343 Mg/m^3

6. Reaction process monitored by HRMS.

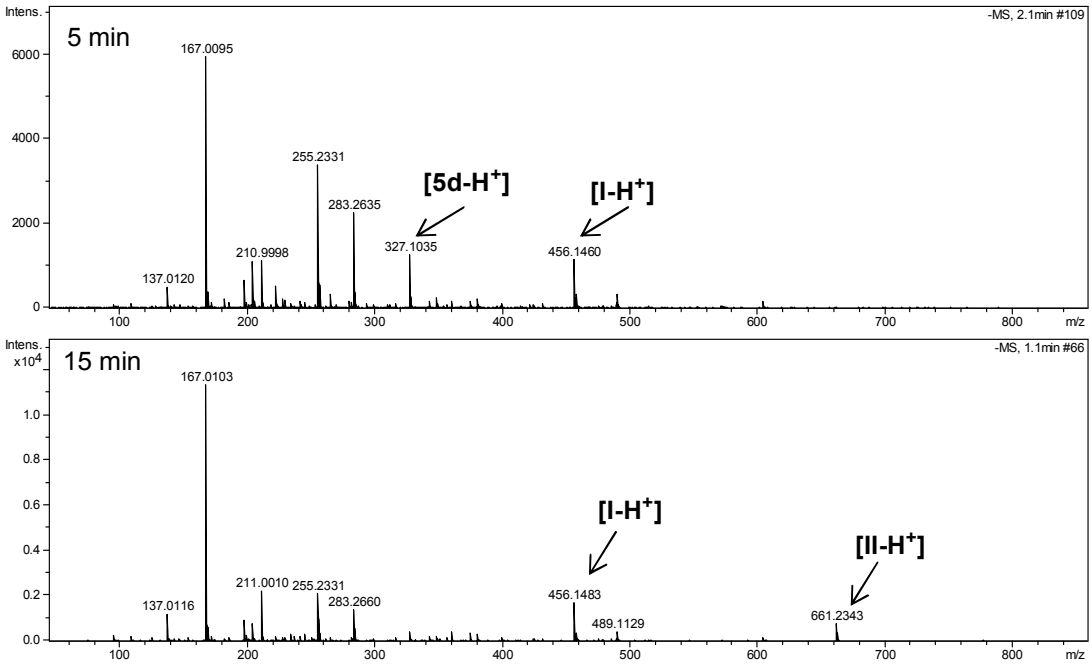


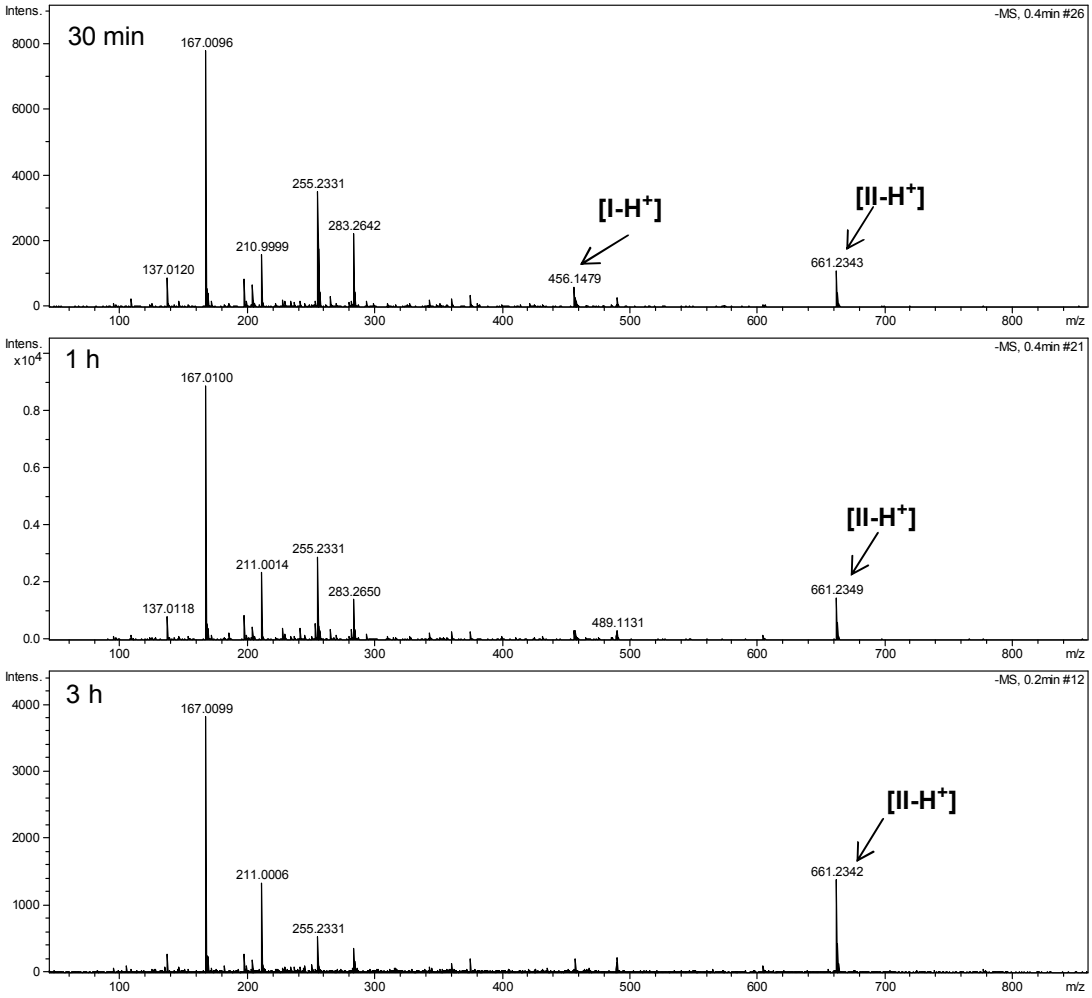
(a) In positive-ion mode



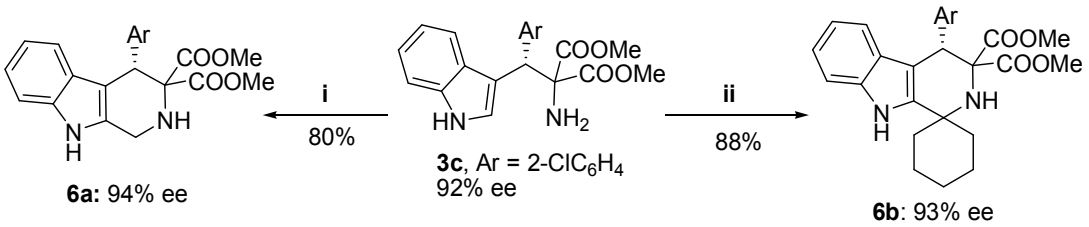


(b) In negative-ion model





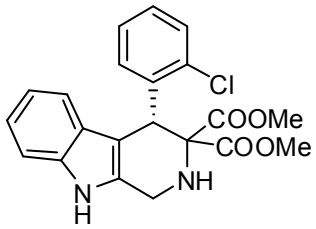
7. The Pictet-Spengler reactions between 3c and carbonyl compounds.



Procedure i：A reaction tube was charged with catalyst **TFA** (6.7 uL, 0.1 mmol), 4Å MS (94 mg)，**3c** (38.7 mg, 0.1 mmol), CH_2O (13.8 uL, 0.5 mmol) and MeOH (1 mL). The solution was stirred at 40°C until the reaction completed (monitored by TLC). One drop of Et_3N was added to the mixture. Then the solution was diluted with water and extracted with EtOAc. The combined organic phase was dried over Na_2SO_4, concentrated and subjected to silica gel column chromatography to afford the desired product **6a** (80% yield, 94% ee) with using ethyl acetate/petroleum as eluents.

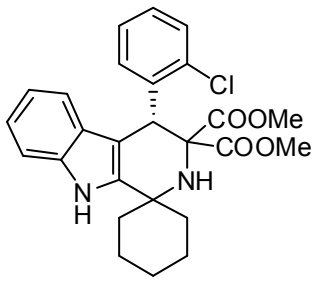
Procedure ii：A reaction tube was charged with catalyst **TFA** (2 uL, 0.03 mmol), **3c** (38.7

mg, 0.1 mmol), cyclohexanone (20.6 uL, 0.2 mmol) and CH_2Cl_2 (1 mL). The solution was stirred at 30°C until the reaction completed (monitored by TLC). One drop of Et_3N was added to the mixture. The solvent was concentrated and the residue was subjected to silica gel column chromatography to afford the desired product **6b** (88% yield, 93% ee) with using ethyl acetate/petroleum as eluents.



6a

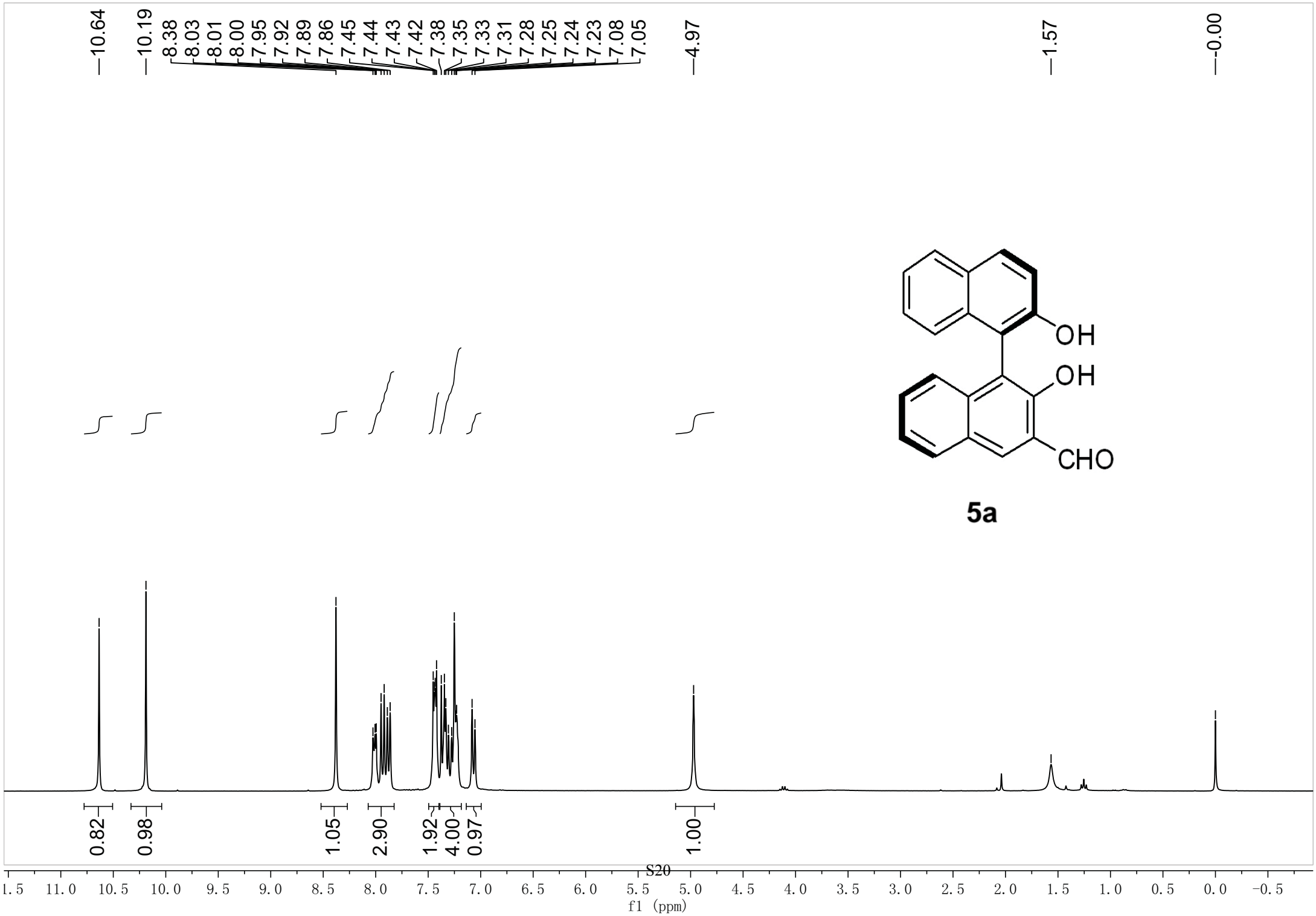
6a was obtained as a white solid (32 mg, 80%); the enantiomeric excess was determined to be 94% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 6.28 min, t_R (minor) 5.38 min; $[\alpha]_D^{20}$ = +386.0 (c = 0.1, CH_3CH_2OH); mp 245°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.87 (s, 1H), 7.62-7.65 (s, 1H), 7.35-7.38 (m, 2H), 7.22-7.25 (m, 1H), 6.96-7.11 (m, 4H), 5.88 (s, 1H), 4.08-4.31 (m, 2H), 3.73 (s, 3H), 3.51 (s, 3H), 2.50 (s, 2H); ^{13}C NMR (75 MHz, DMSO): δ (ppm)170.27, 168.79, 138.64, 136.31, 133.07, 132.18, 132.08, 128.89, 128.72, 127.02, 126.33, 121.26, 118.93, 117.50, 111.45, 109.60, 70.92, 53.35, 52.43, 39.71, 37.18; HRMS (ESI): calcd. for $C_{21}H_{20}ClN_2O_4$ (M^++H): 399.1106, found: 399.1102.

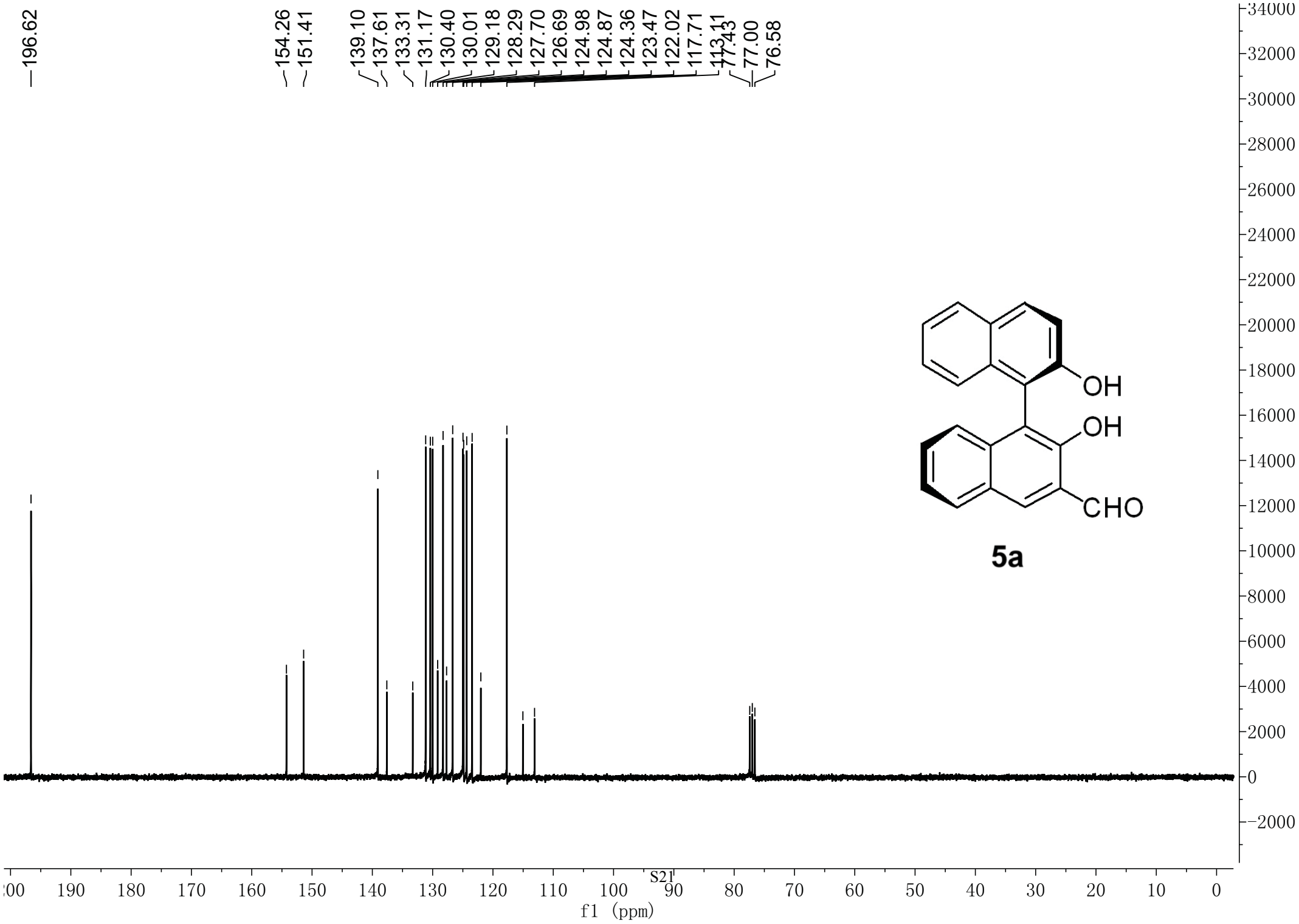


6b

6b was obtained as a white solid (42 mg, 90%); the enantiomeric excess was determined to be 93% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 70/30, flow rate 1 mL/min, T = 30°C), UV 254 nm, t_R (major) 3.87 min, t_R (minor) 4.96 min; $[\alpha]_D^{20}$ = +283.0 (c = 0.1, CH_3CH_2OH); mp 240°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.74 (m, 1H), 7.33-7.50 (m, 3H), 7.26-7.28 (m, 1H), 6.96-7.12 (m, 4H), 5.85 (s, 1H), 3.68 (s, 3H), 3.46 (s, 3H), 1.81-2.06 (m, 8H), 1.58-1.67 (m, 1H), 1.25-1.39 (m, 1H); ^{13}C NMR (75 MHz, DMSO): δ (ppm) 171.88, 169.19, 139.14, 138.35, 136.33, 132.98, 131.64, 129.00, 128.66, 127.25, 125.98, 121.41, 118.88, 117.76, 111.41, 108.24, 79.49, 68.04, 52.71, 52.41, 51.91, 36.80, 34.71, 25.13, 21.53, 21.02; HRMS (ESI): calcd. for $C_{26}H_{28}ClN_2O_4$ (M^++H): 467.1732, found: 467.1729.

8. The spectrums of ^{1}H NMR, ^{13}C NMR and HPLC.


10.64
10.19
8.38
8.03
8.01
8.00
7.95
7.92
7.89
7.86
7.45
7.44
7.43
7.42
7.38
7.35
7.33
7.31
7.28
7.25
7.24
7.23
7.08
7.05
4.97
1.57
0.00
OH
OH
CHO
5a
0.82
0.98
1.05
2.90
1.92
4.00
0.97
1.00
f1 (ppm)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124.98
124.87
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76.58
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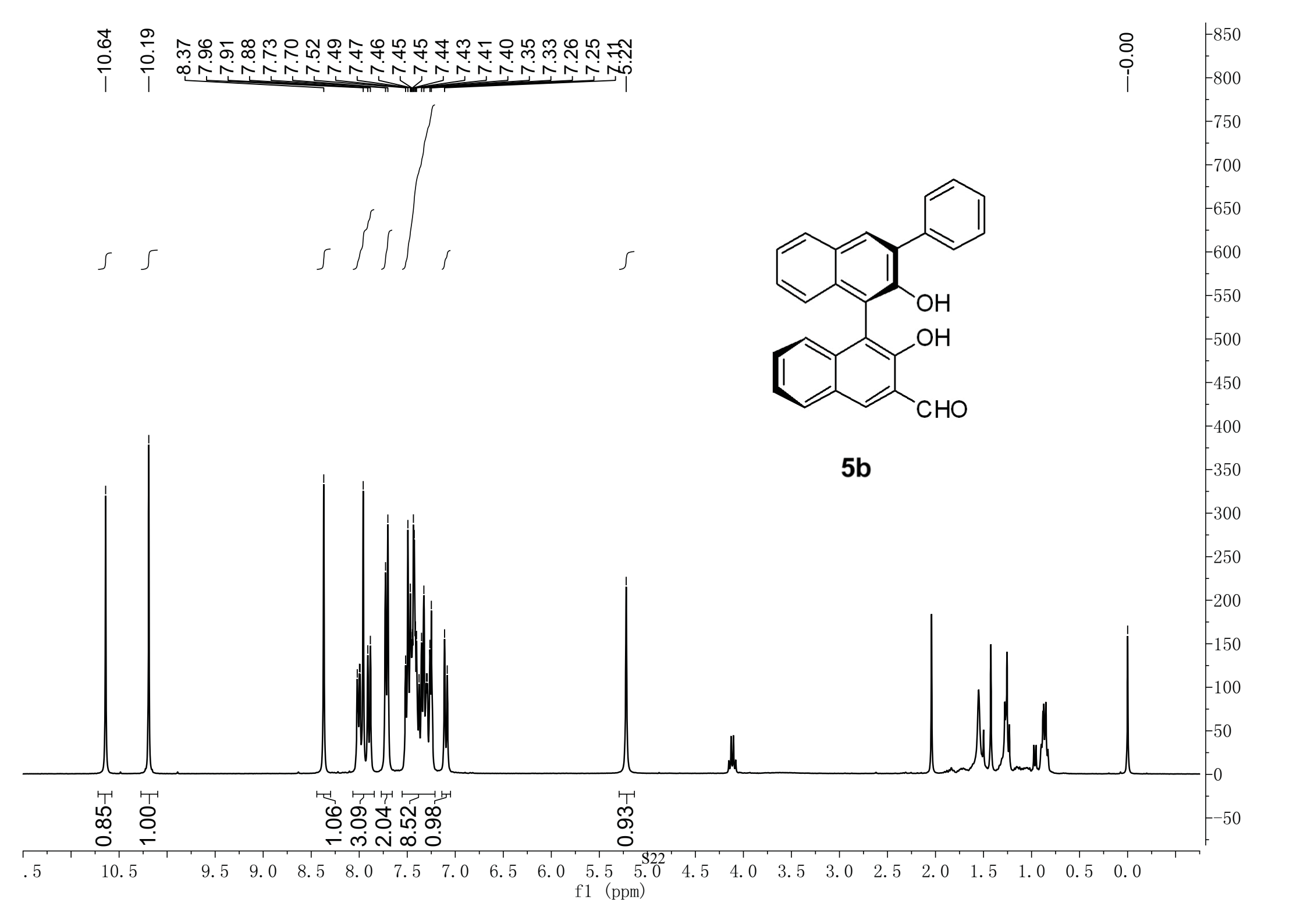
OH
CHO
5a
f1 (ppm)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7.45
7.44
7.43
7.41
7.40
7.35
7.33
7.26
7.25
7.11
5.22
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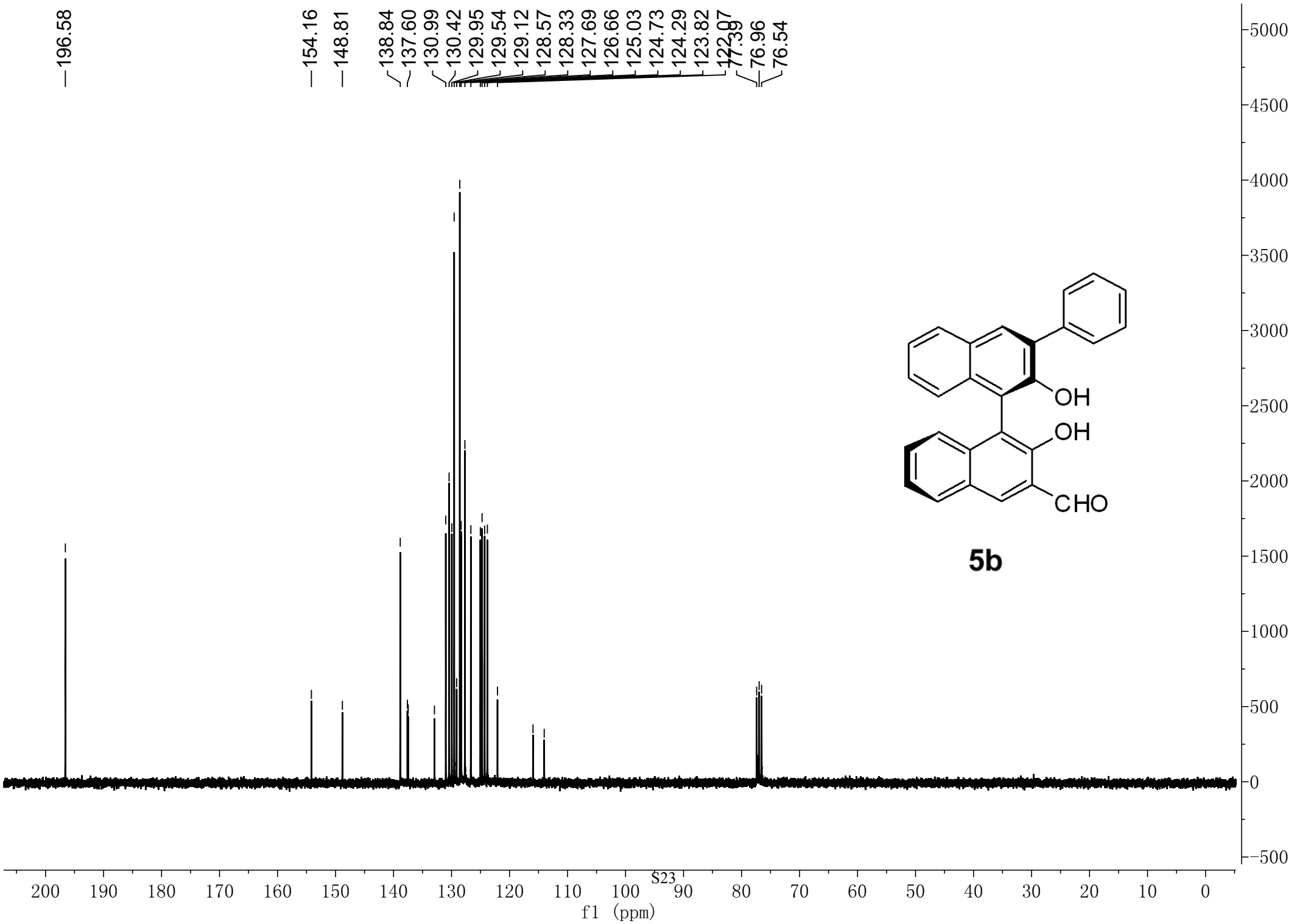
f1 (ppm)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126.66
125.03
124.73
124.29
123.82
122.07
77.39
76.96
76.54
OH
OH
CHO
5b
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
−500
200
190
180
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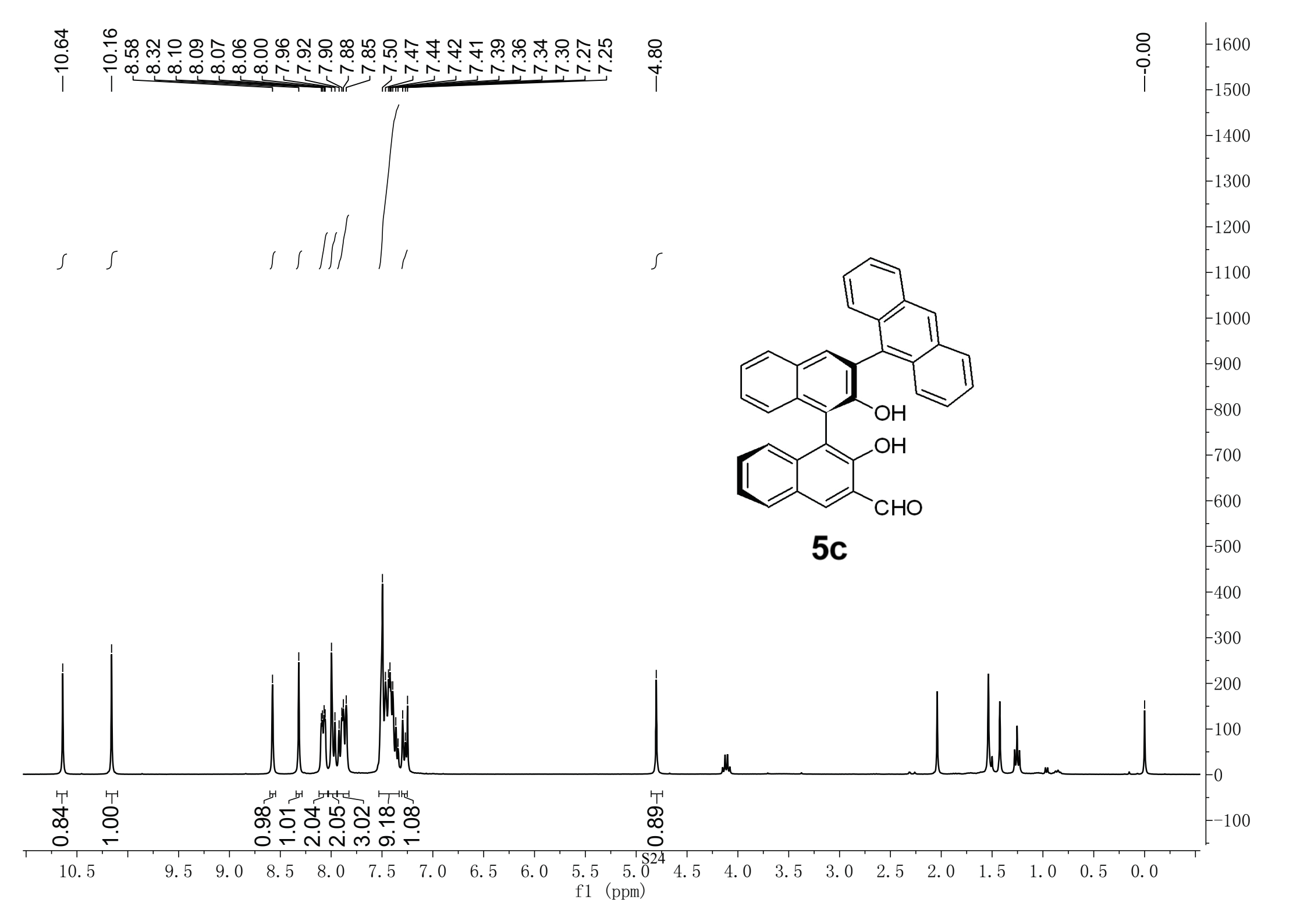
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7.85
7.50
7.47
7.44
7.42
7.41
7.39
7.36
7.34
7.30
7.27
7.25
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OH
CHO
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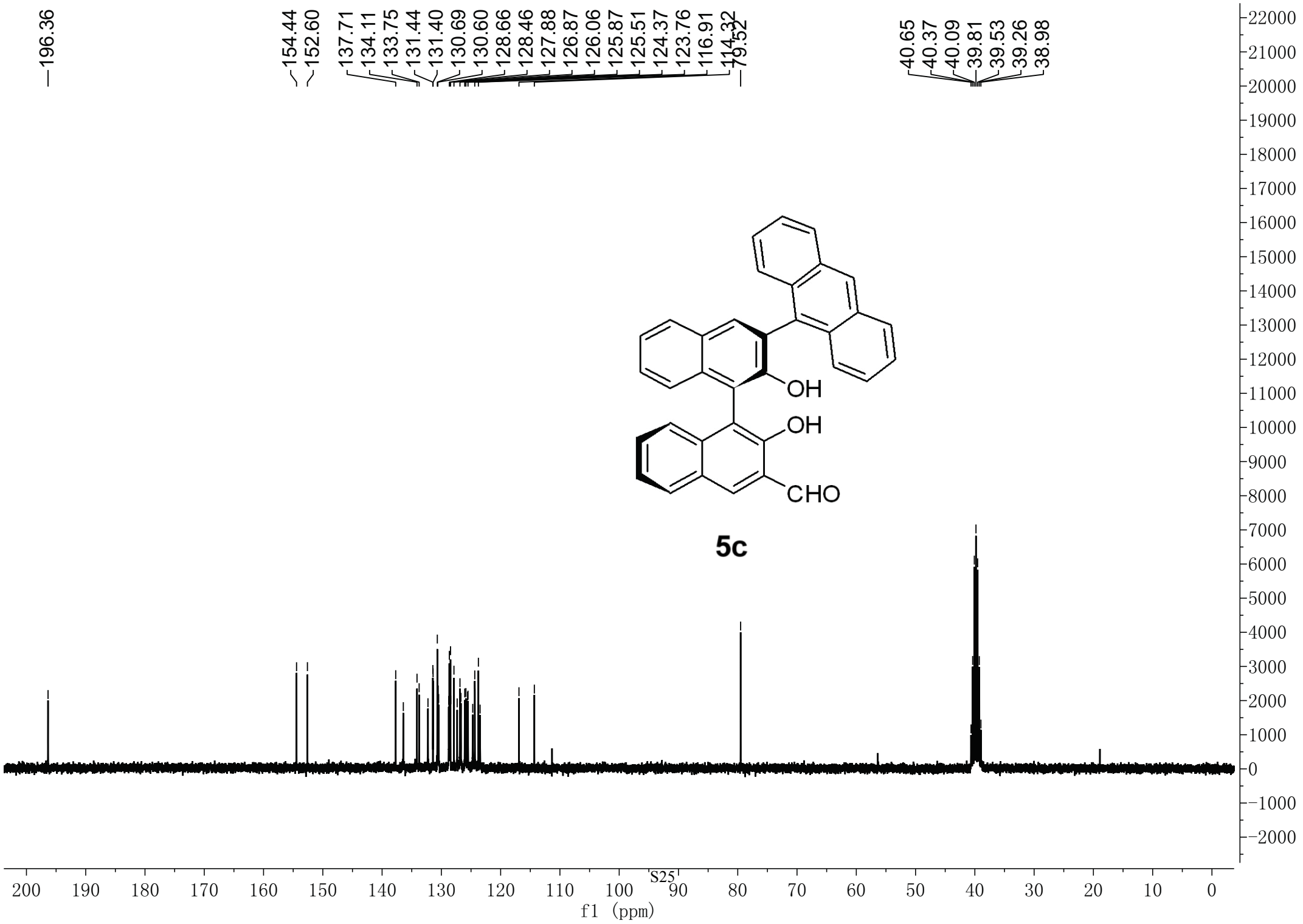
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125.51
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114.32
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38.98
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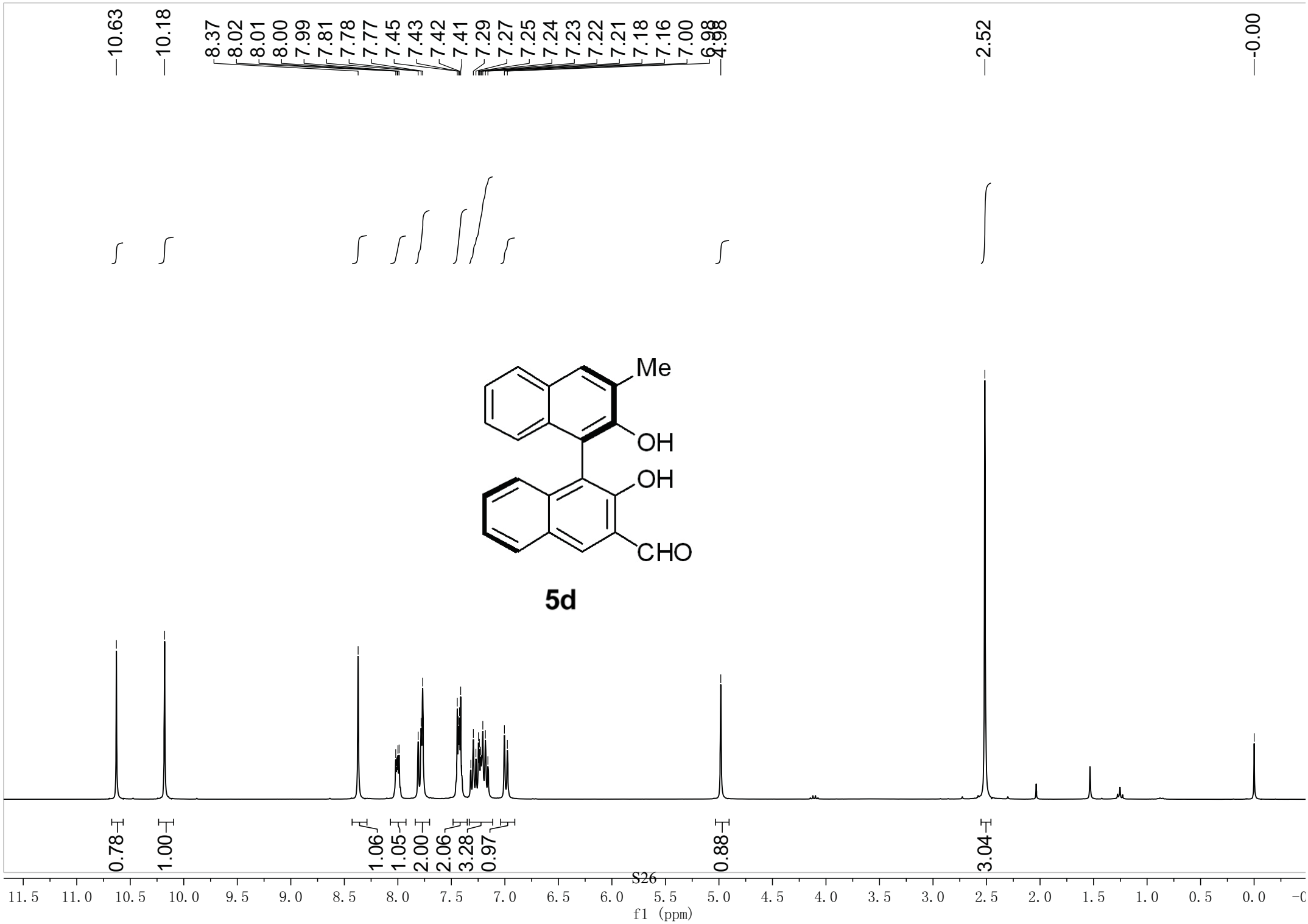
OH
CHO
5c
f1 (ppm)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7.42
7.41
7.29
7.27
7.25
7.24
7.23
7.22
7.21
7.18
7.16
7.00
6.98
4.98
2.52
0.00
Me
OH
OH
CHO
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1.06
1.05
2.00
2.06
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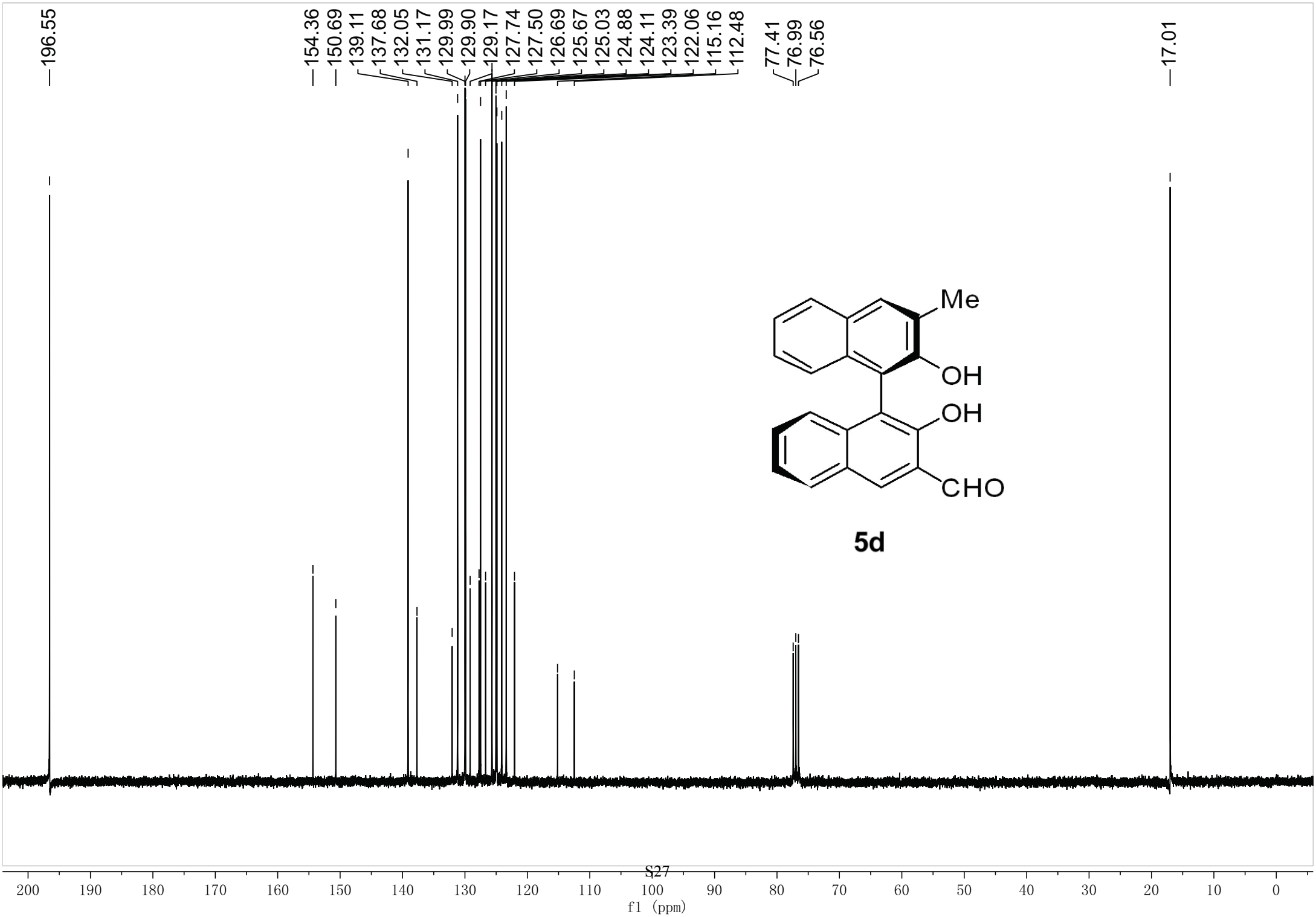
3.04
f1 (ppm)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125.67
125.03
124.88
124.11
123.39
122.06
115.16
112.48
77.41
76.99
76.56
17.01
Me
OH
OH
CHO
5d
200
190
180
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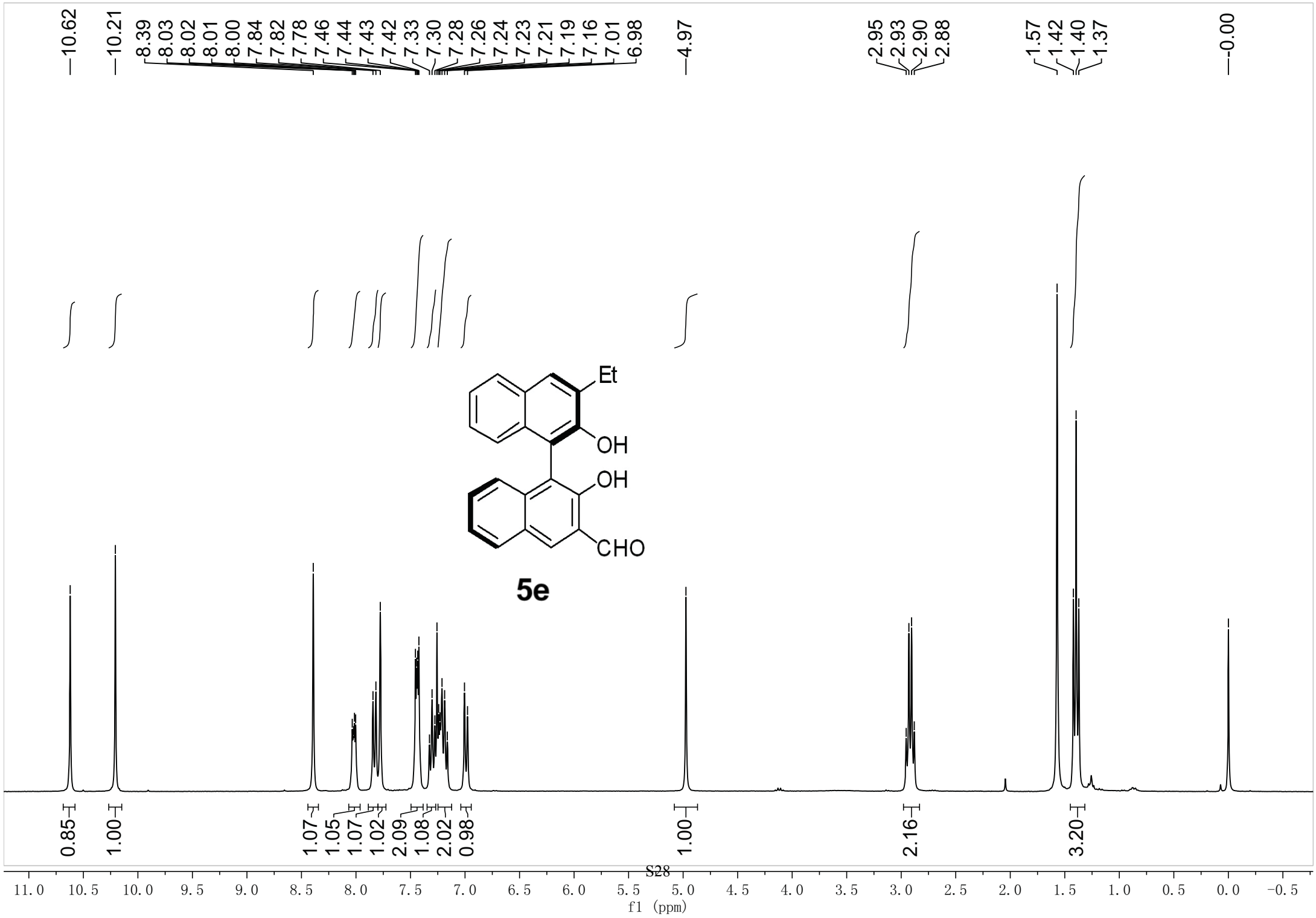
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.43
7.42
7.33
7.30
7.28
7.26
7.24
7.23
7.21
7.19
7.16
7.01
6.98
4.97
2.95
2.93
2.90
2.88
1.57
1.42
1.40
1.37
0.00
Et
OH
OH
CHO
5e
0.85
1.00
1.07
1.05
1.07
1.02
2.09
1.08
2.02
0.98
1.00
2.16
3.20
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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2.5
2.0
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0.5
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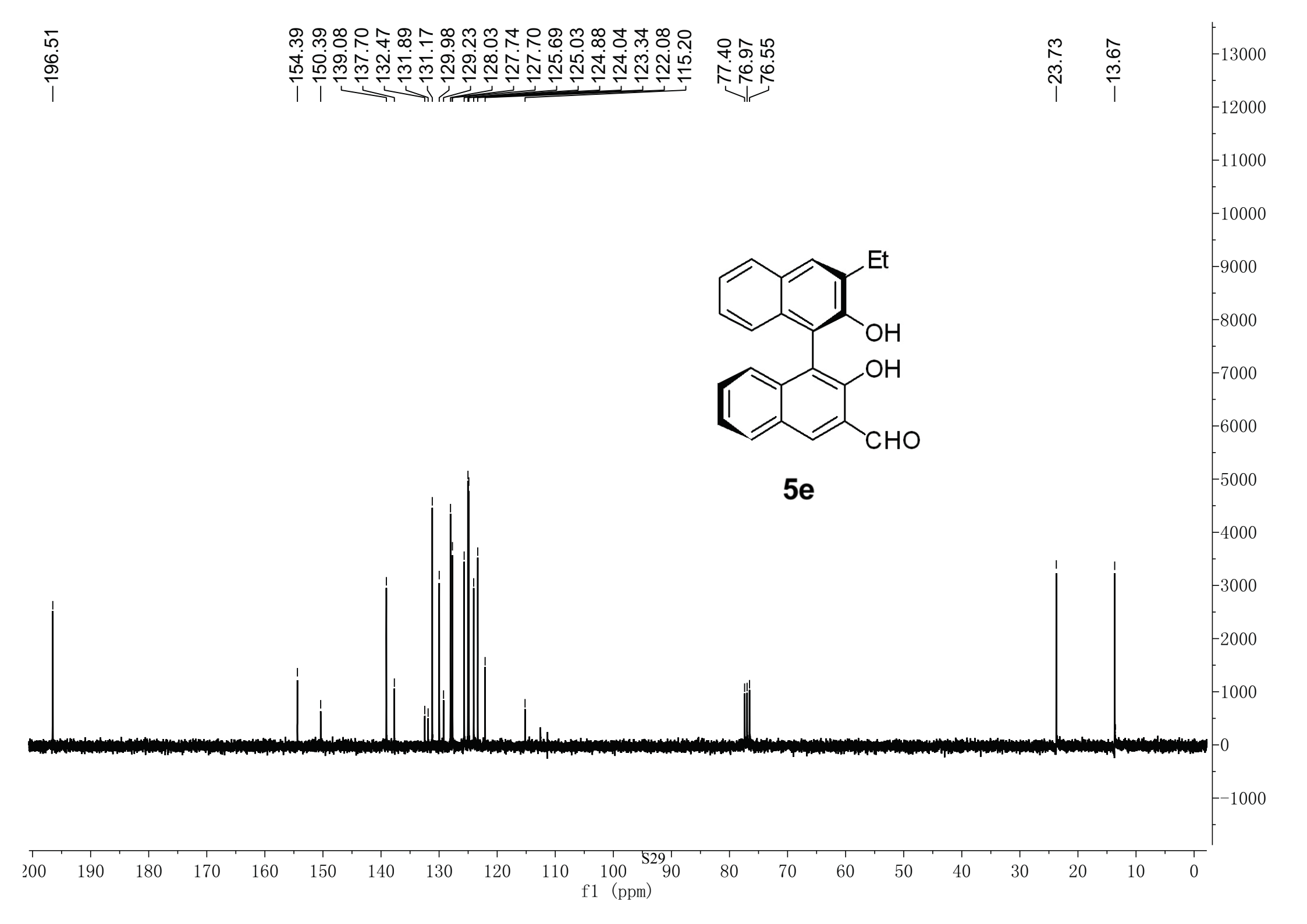
-0.5
f1 (ppm)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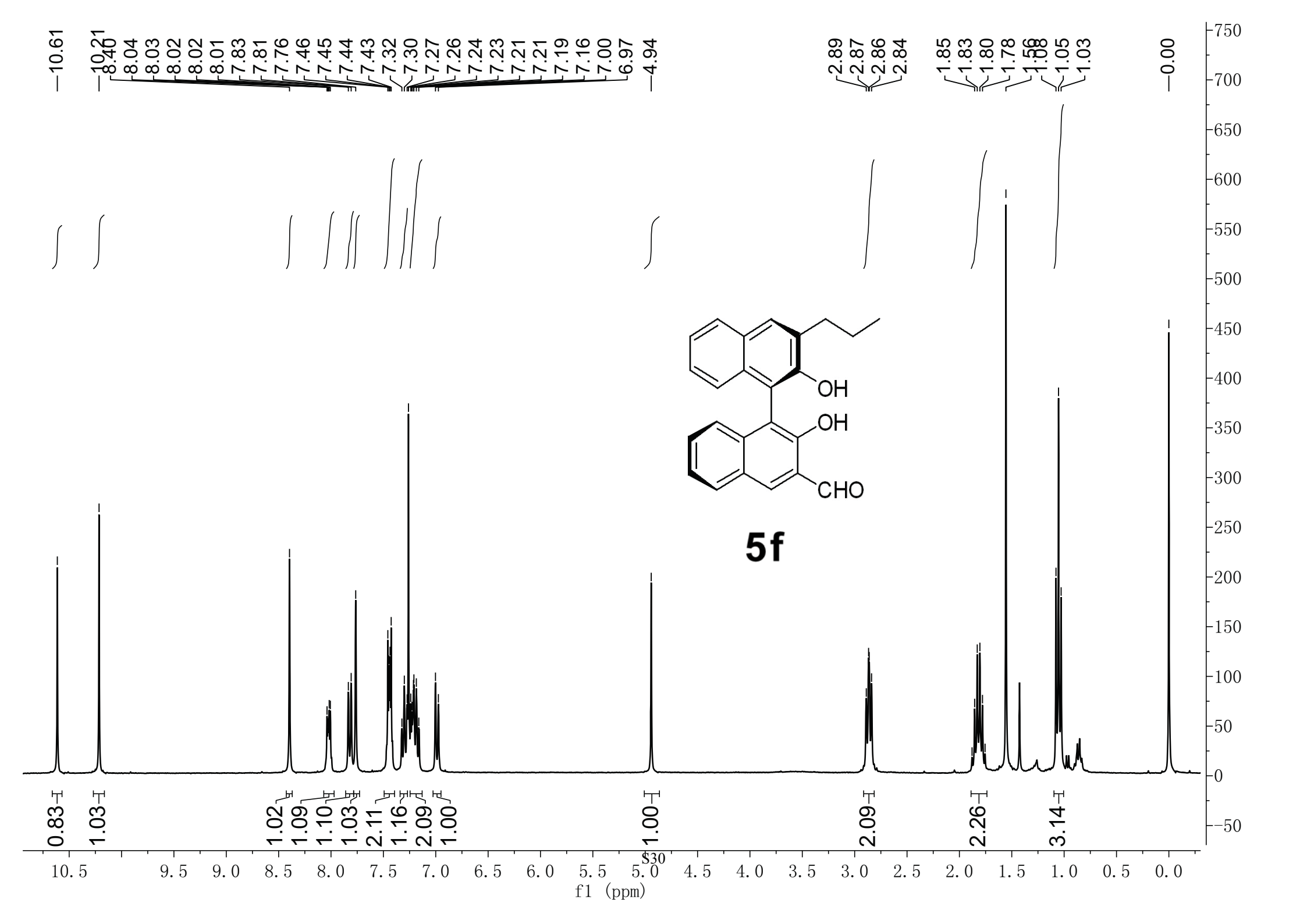
129.23
128.03
127.74
127.70
125.69
125.03
124.88
124.04
123.34
122.08
115.20
77.40
76.97
76.55
23.73
13.67
Et
OH
OH
CHO
5e
f1 (ppm)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7.81
7.76
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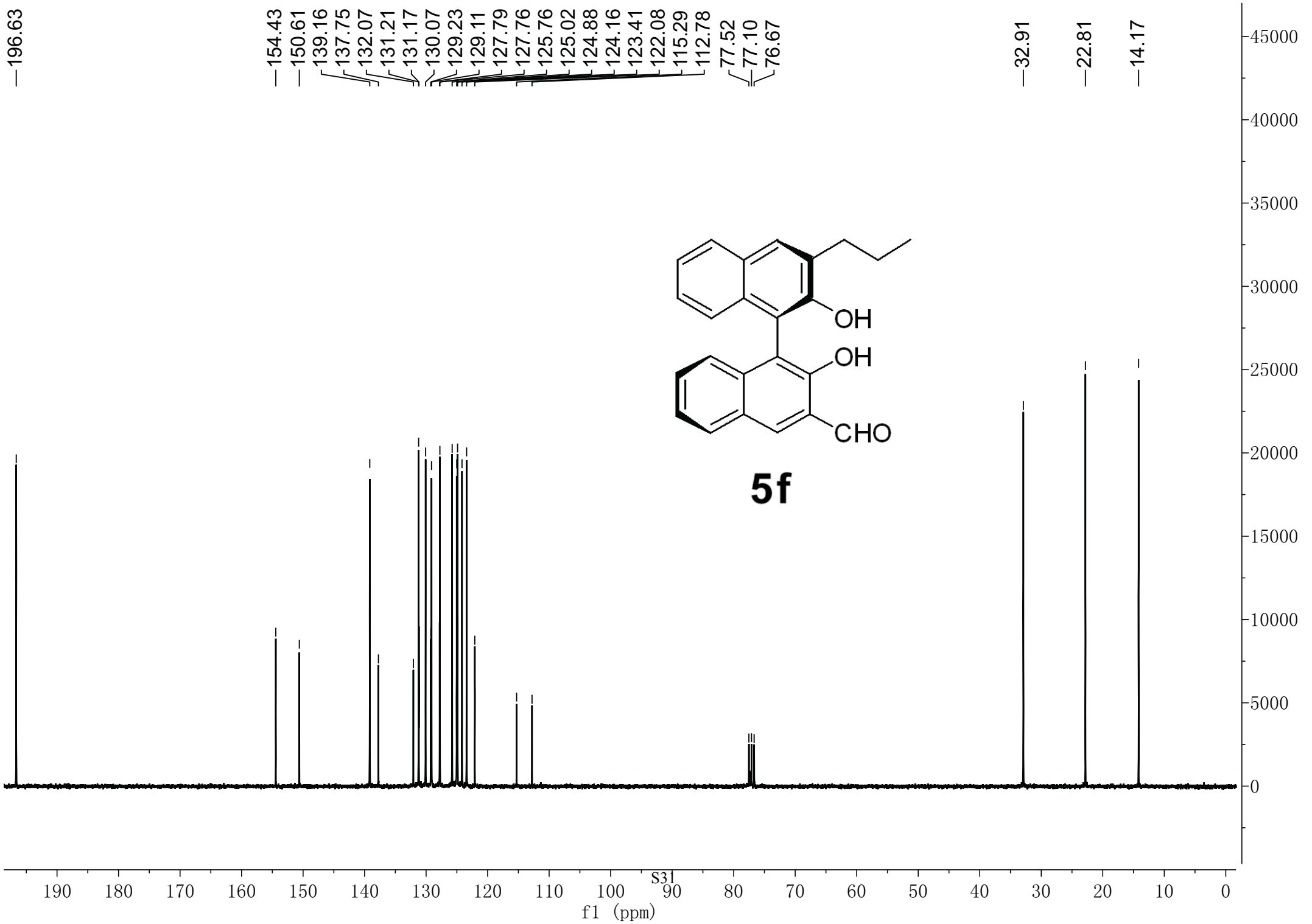
7.45
7.44
7.43
7.32
7.30
7.27
7.26
7.24
7.23
7.21
7.21
7.19
7.16
7.00
6.97
4.94
2.89
2.87
2.86
2.84
1.85
1.83
1.80
1.78
1.56
1.05
1.03
0.00
OH
OH
CHO
5f
0.83
1.03
1.02
1.09
1.10
1.03
2.11
1.16
2.09
1.00
1.00
2.09
2.26
3.14
f1 (ppm)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127.76
125.76
125.02
124.88
124.16
123.41
122.08
115.29
112.78
77.52
77.10
76.67
32.91
22.81
14.17
OH
OH
CHO
5f
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
190
180
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130
120
110
100
90
80
70
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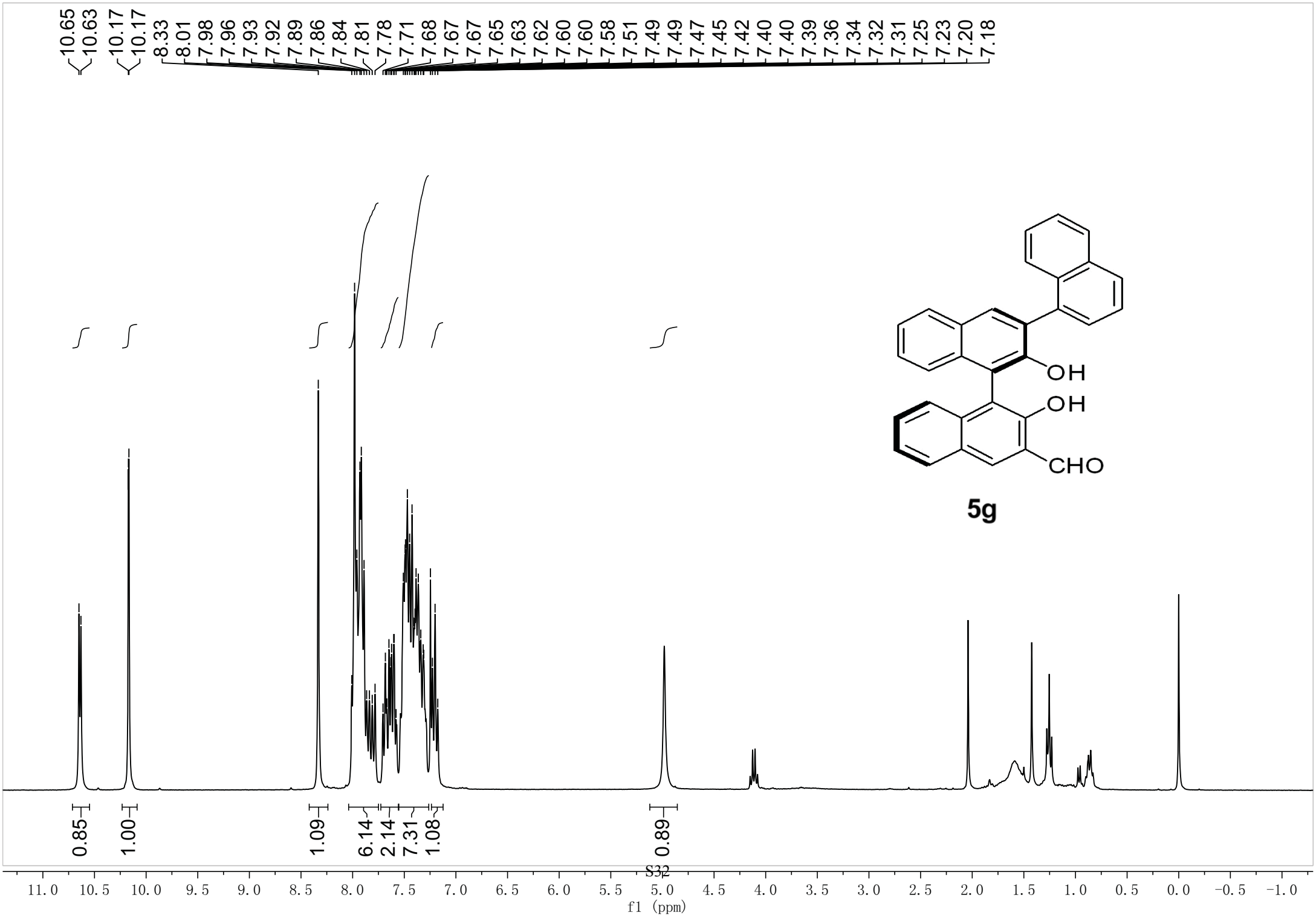
50
40
30
20
10
0
f1 (ppm)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7.92
7.89
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7.84
7.81
7.78
7.71
7.68
7.67
7.67
7.65
7.63
7.62
7.60
7.60
7.58
7.51
7.49
7.49
7.47
7.45
7.42
7.40
7.40
7.39
7.36
7.34
7.32
7.31
7.25
7.23
7.20
7.18
OH
OH
CHO
5g
0.85
1.00
1.09
6.14
2.14
7.31
1.08
0.89
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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2.5
2.0
1.5
1.0
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0.0
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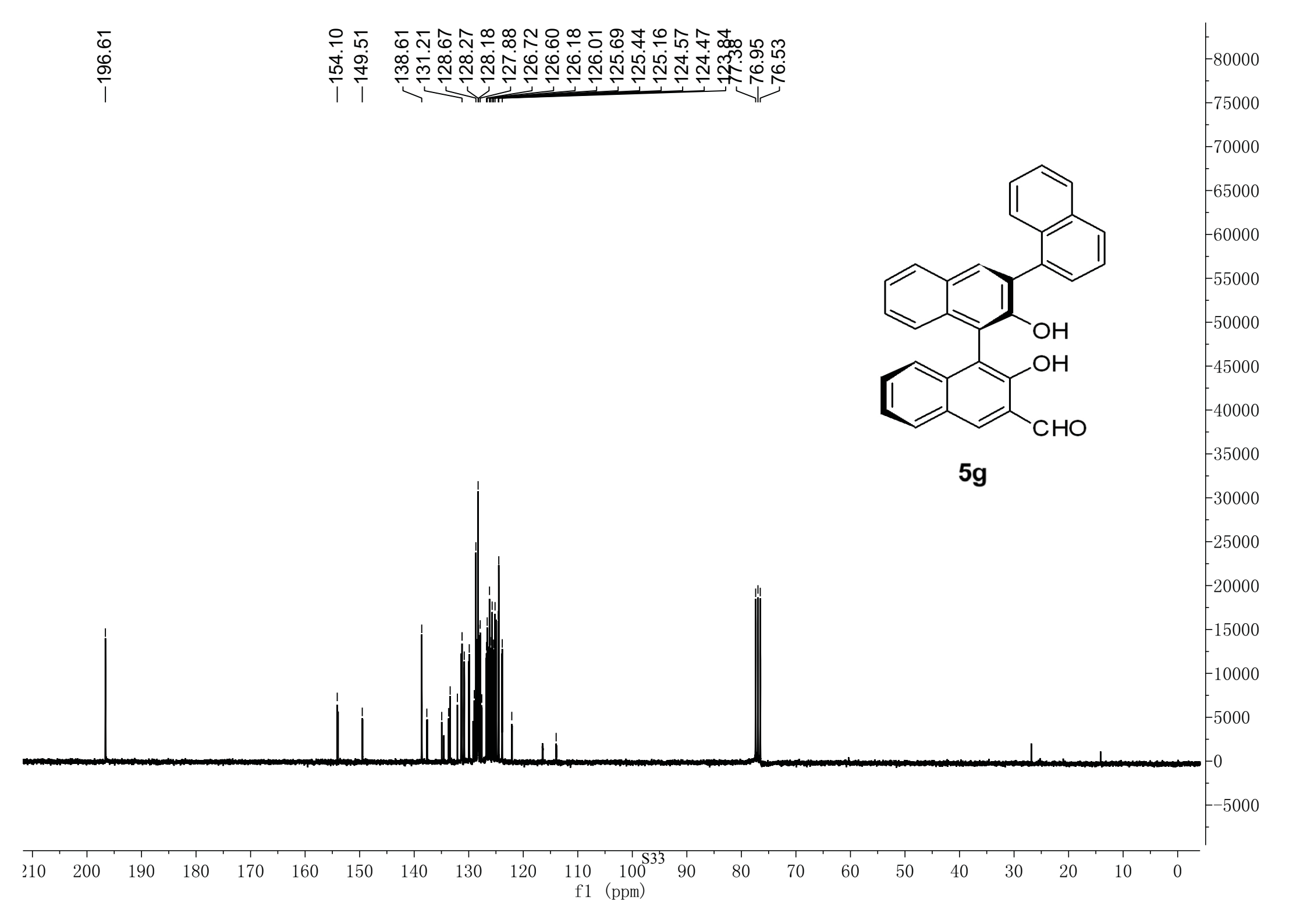
-1.0
f1 (ppm)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126.18
126.01
125.69
125.44
125.16
124.57
124.47
123.84
77.38
76.95
76.53
OH
OH
CHO
5g
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
210
200
190
180
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120
110
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50
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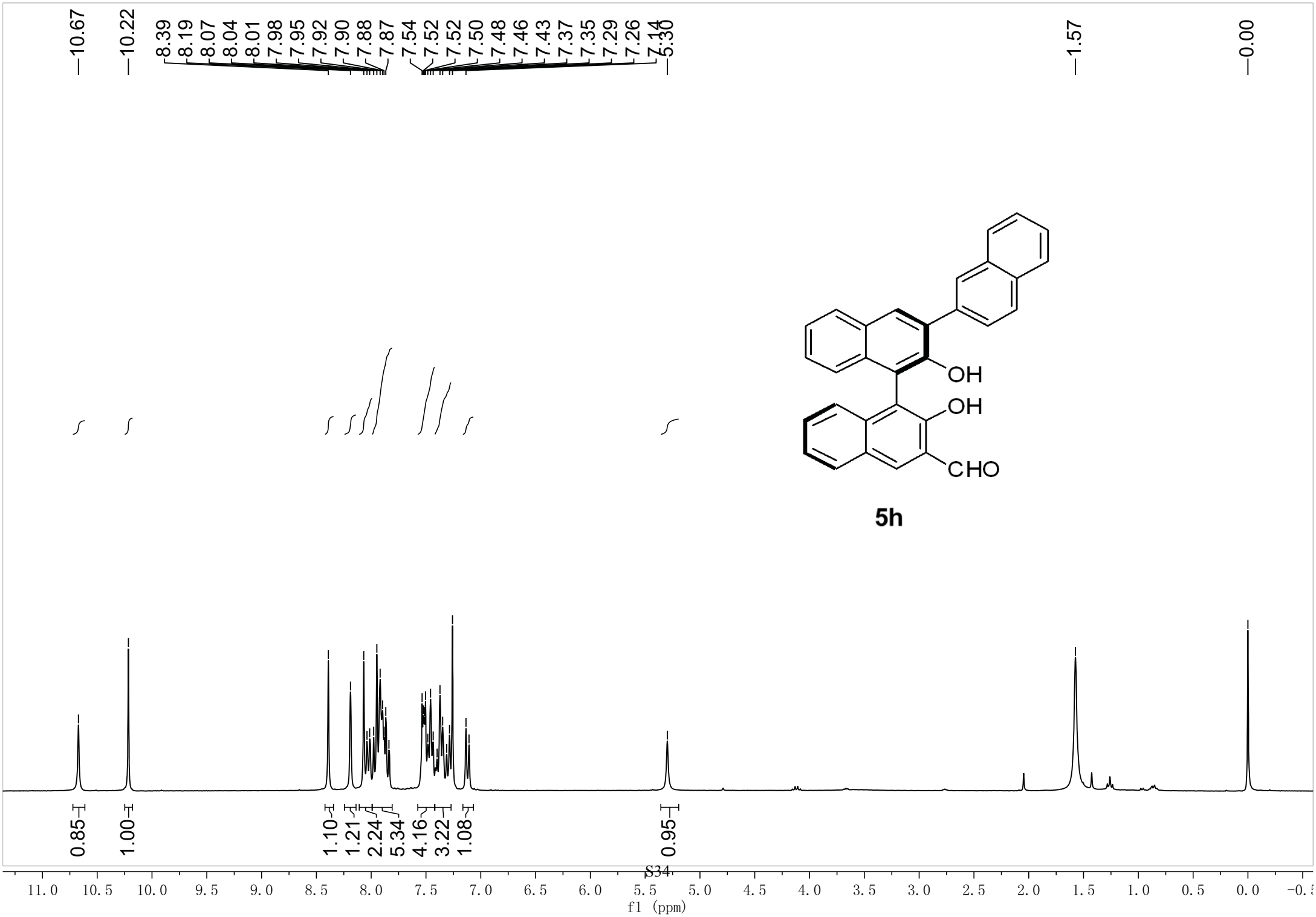
10
0
f1 (ppm)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7.87
7.54
7.52
7.52
7.50
7.48
7.46
7.43
7.37
7.35
7.29
7.26
7.14
5.30
1.57
0.00
OH
OH
CHO
5h
0.85
1.00
1.10
1.21
2.24
5.34
4.16
3.22
1.08
0.95
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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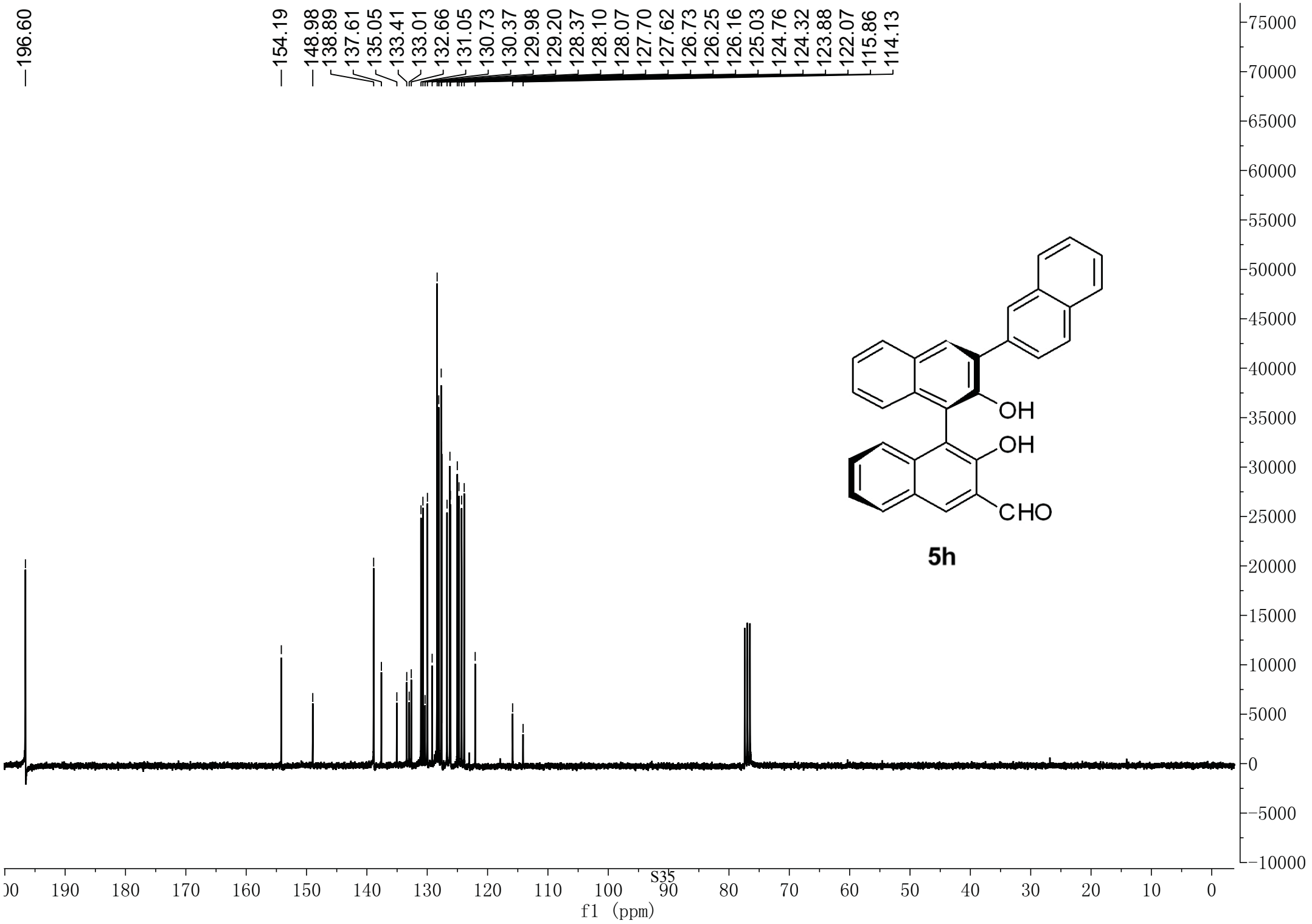
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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131.05
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130.37
129.98
129.20
128.37
128.10
128.07
127.70
127.62
126.73
126.25
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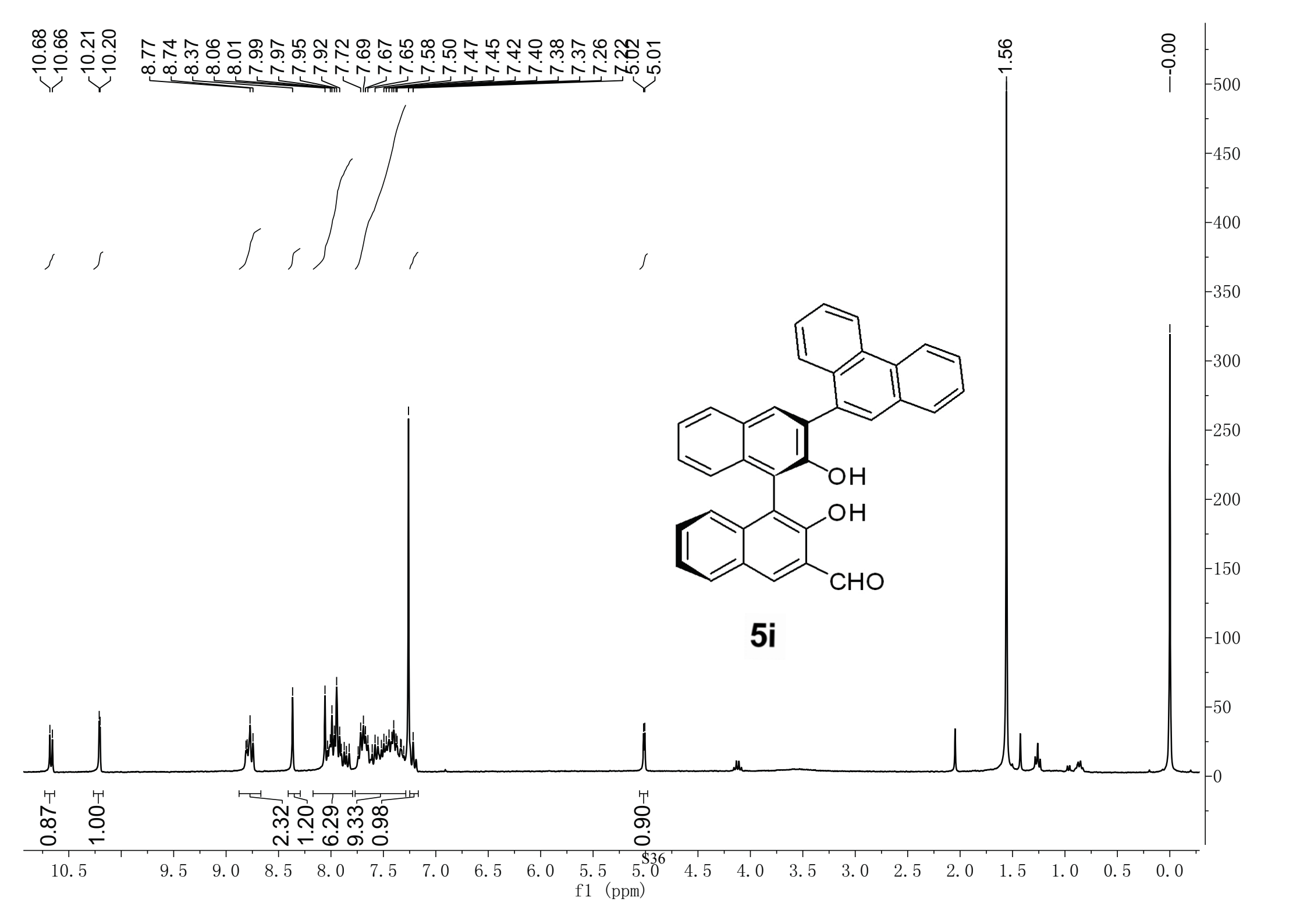
124.76
124.32
123.88
122.07
115.86
114.13
OH
OH
CHO
5h
f1 (ppm)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7.95
7.92
7.72
7.69
7.67
7.65
7.58
7.50
7.47
7.45
7.42
7.40
7.38
7.37
7.26
7.22
5.02
5.01
1.56
0.00
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OH
CHO
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1.20
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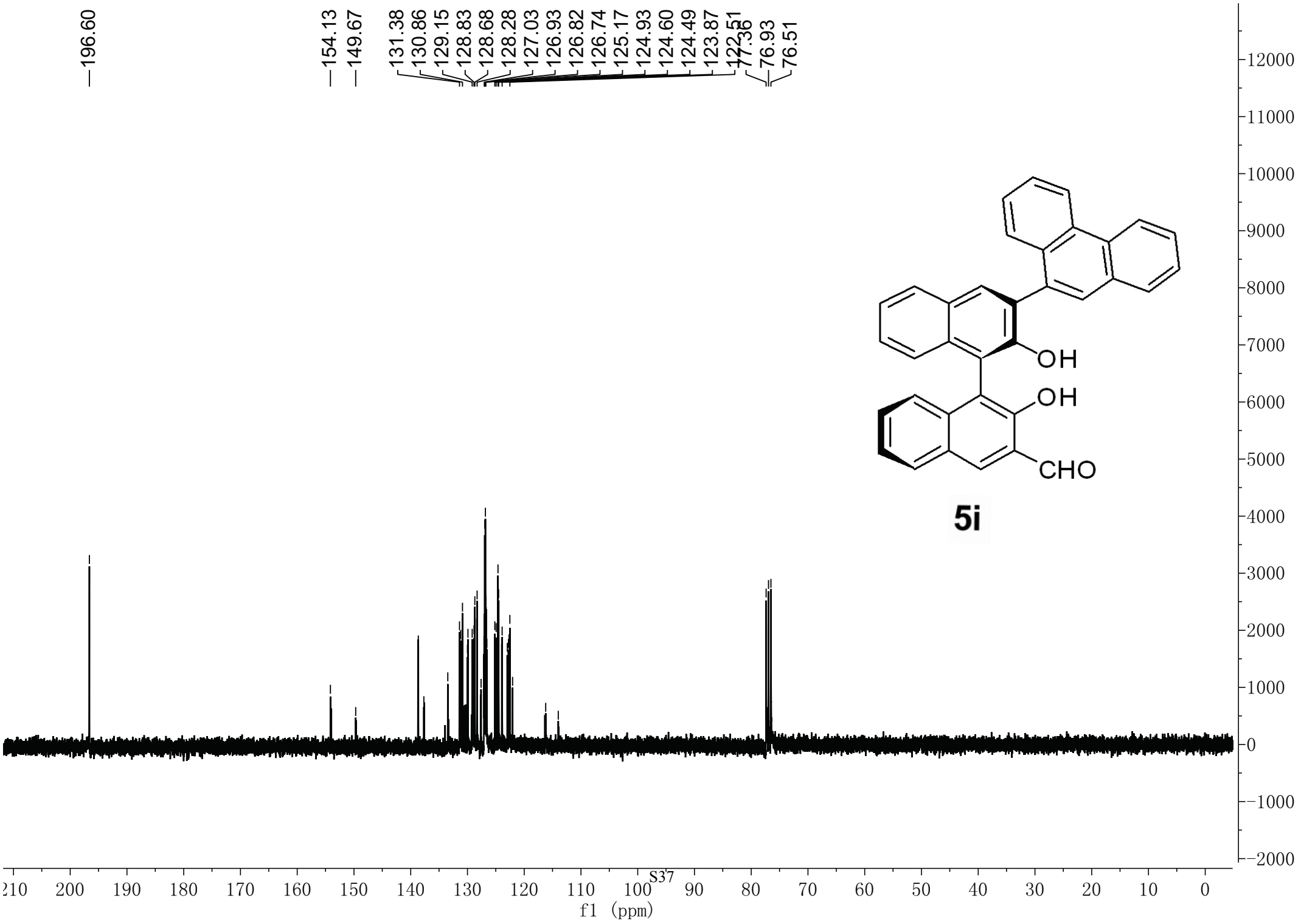
0.90
f1 (ppm)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124.93
124.60
124.49
123.87
122.51
77.36
76.93
76.51
OH
OH
CHO
5i
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
210
200
190
180
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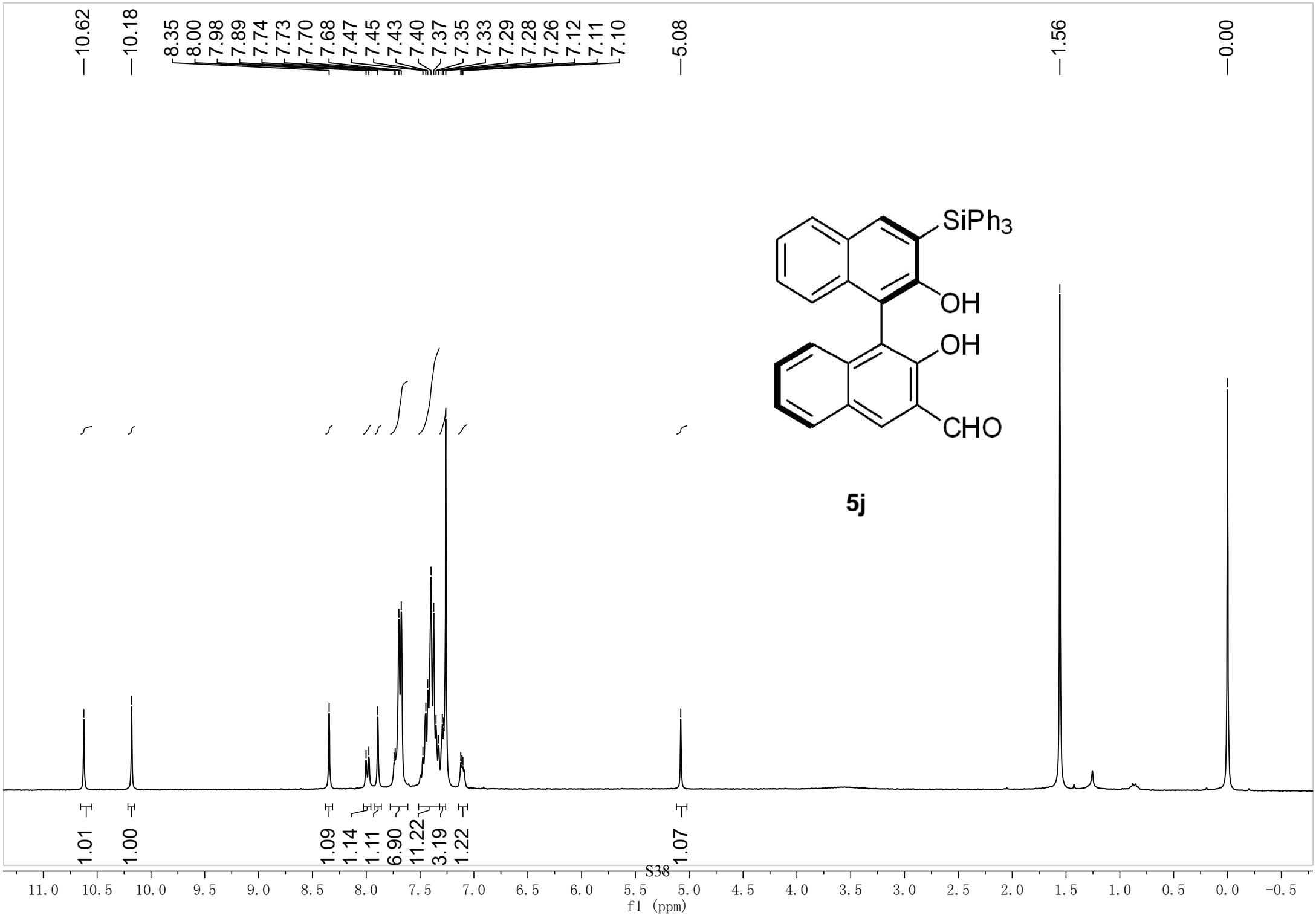
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.45
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7.40
7.37
7.35
7.33
7.29
7.28
7.26
7.12
7.11
7.10
5.08
1.56
0.00
SiPh3
OH
OH
CHO
5j
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1.00
1.09
1.14
1.11
6.90
11.22
3.19
1.22
1.07
11.0
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9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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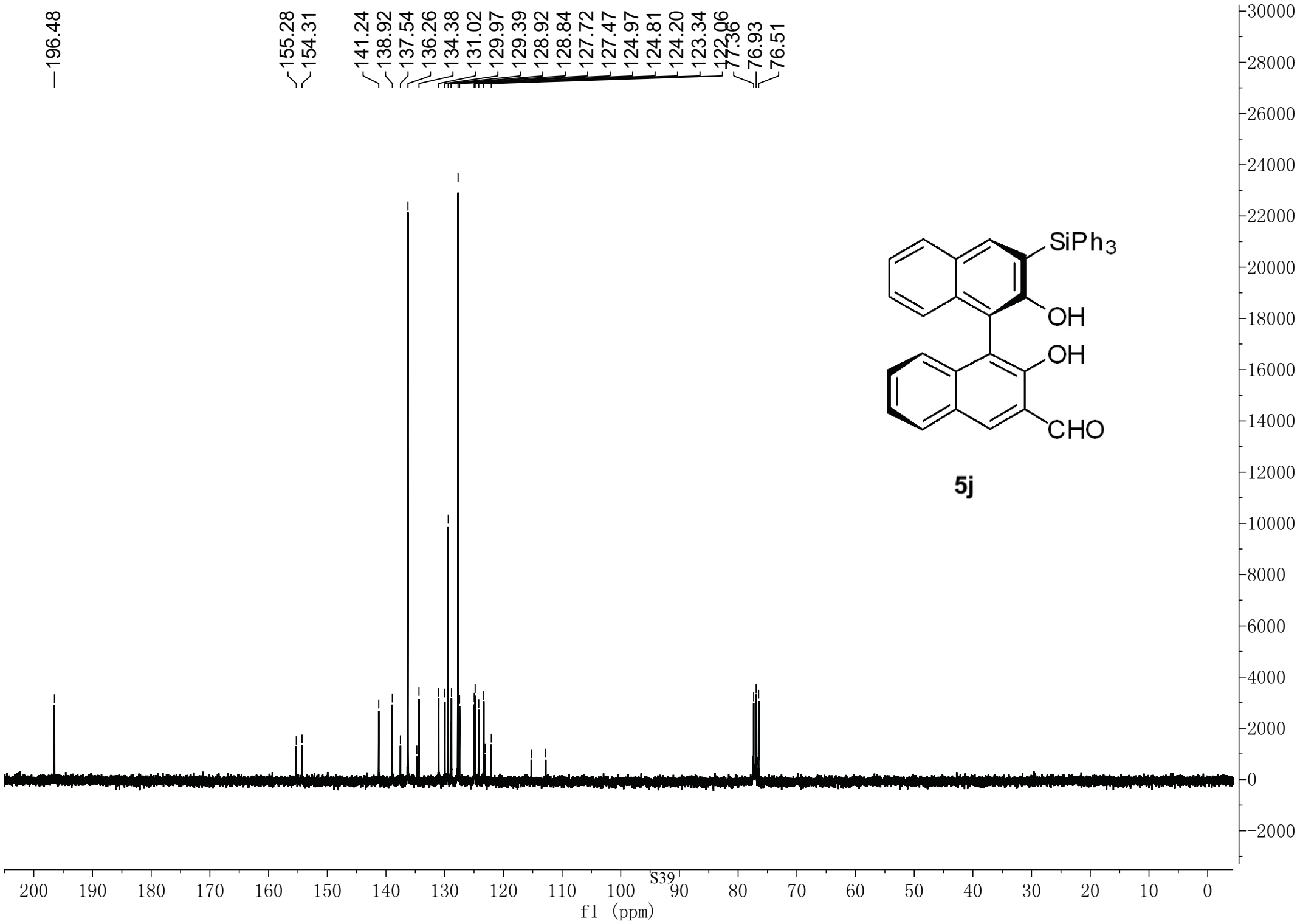
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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127.72
127.47
124.97
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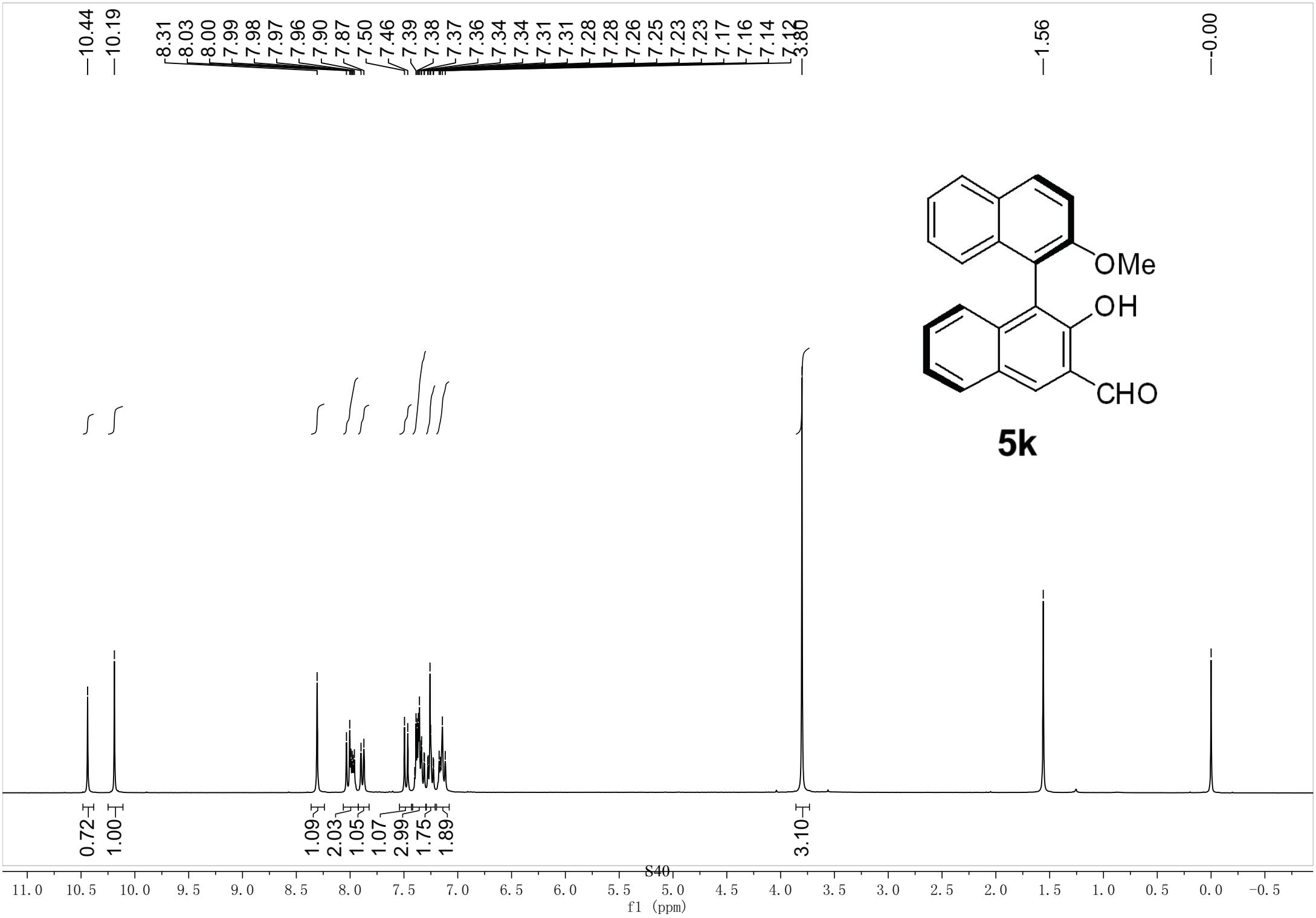
123.34
122.06
77.36
76.93
76.51
SiPh3
OH
OH
CHO
5j
f1 (ppm)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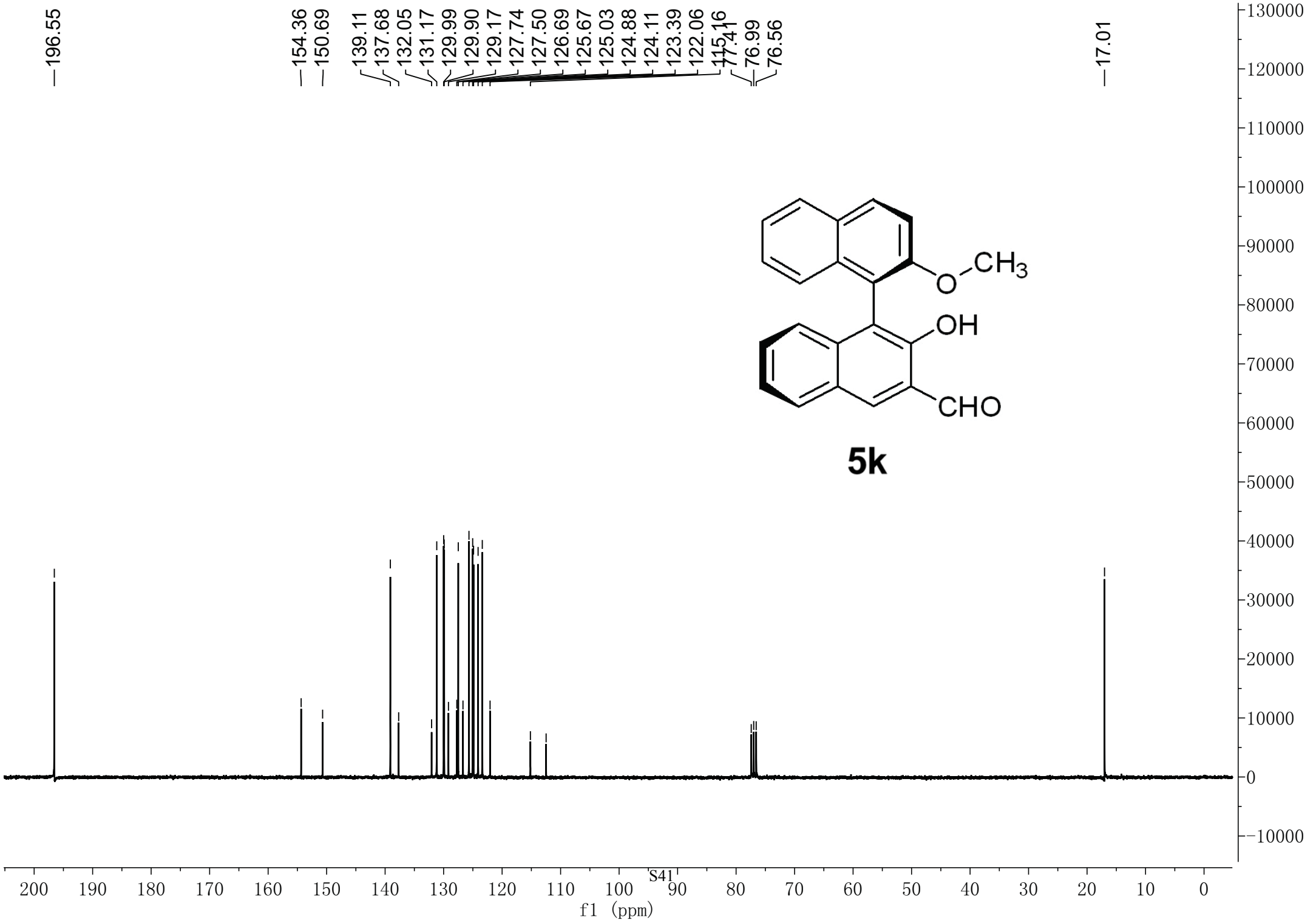
7.46
7.39
7.38
7.37
7.36
7.34
7.34
7.31
7.31
7.28
7.28
7.26
7.25
7.23
7.23
7.17
7.16
7.14
7.12
3.80
1.56
0.00
OMe
OH
CHO
5k
0.72
1.00
1.09
2.03
1.05
1.07
2.99
1.75
1.89
3.10
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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127.74
127.50
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125.67
125.03
124.88
124.11
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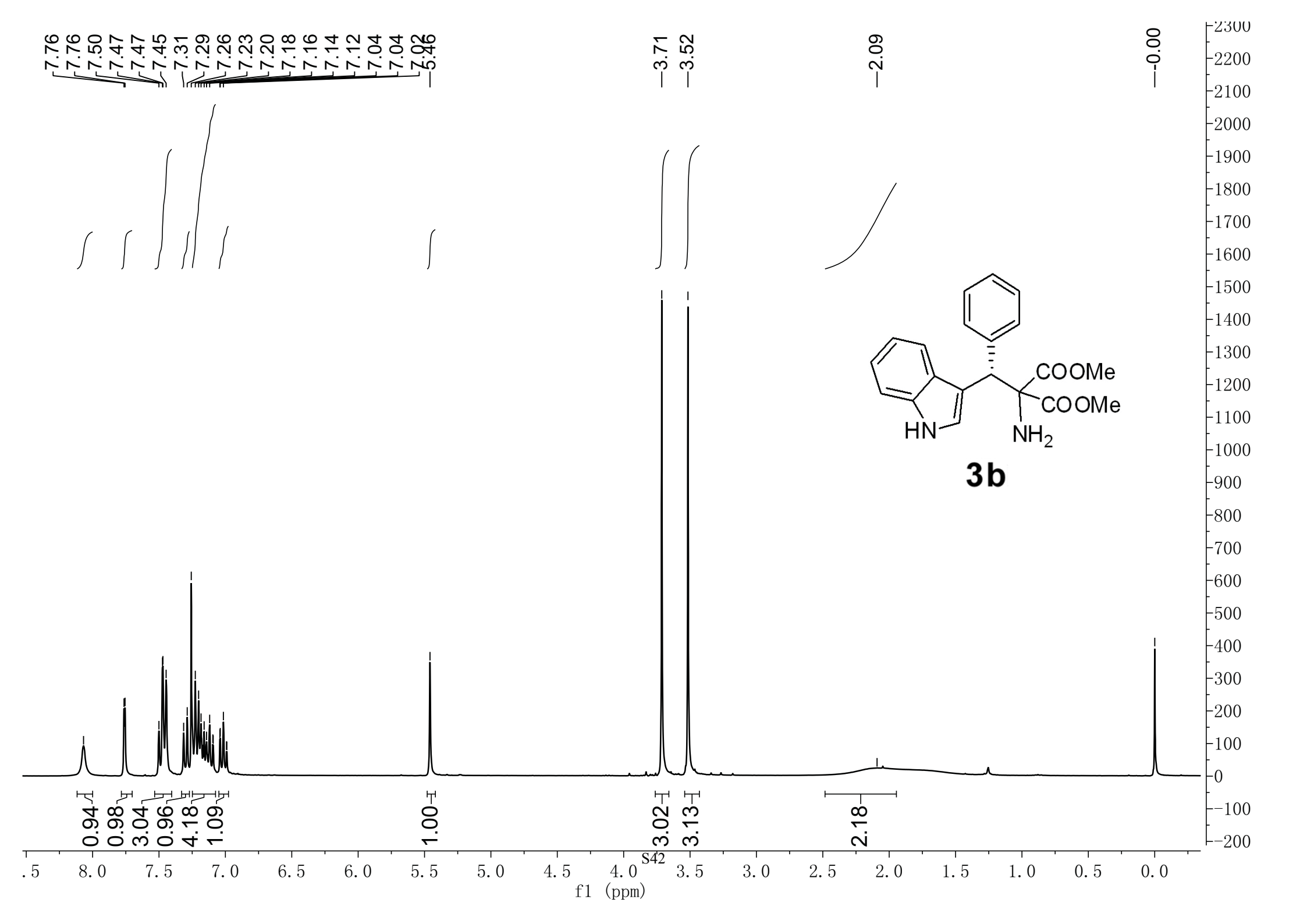
115.16
77.41
76.99
76.56
17.01
O
CH3
OH
CHO
5k
f1 (ppm)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7.31
7.29
7.26
7.23
7.20
7.18
7.16
7.14
7.12
7.04
7.04
7.02
5.46
3.71
3.52
2.09
0.00
COOMe
COOMe
HN
NH2
3b
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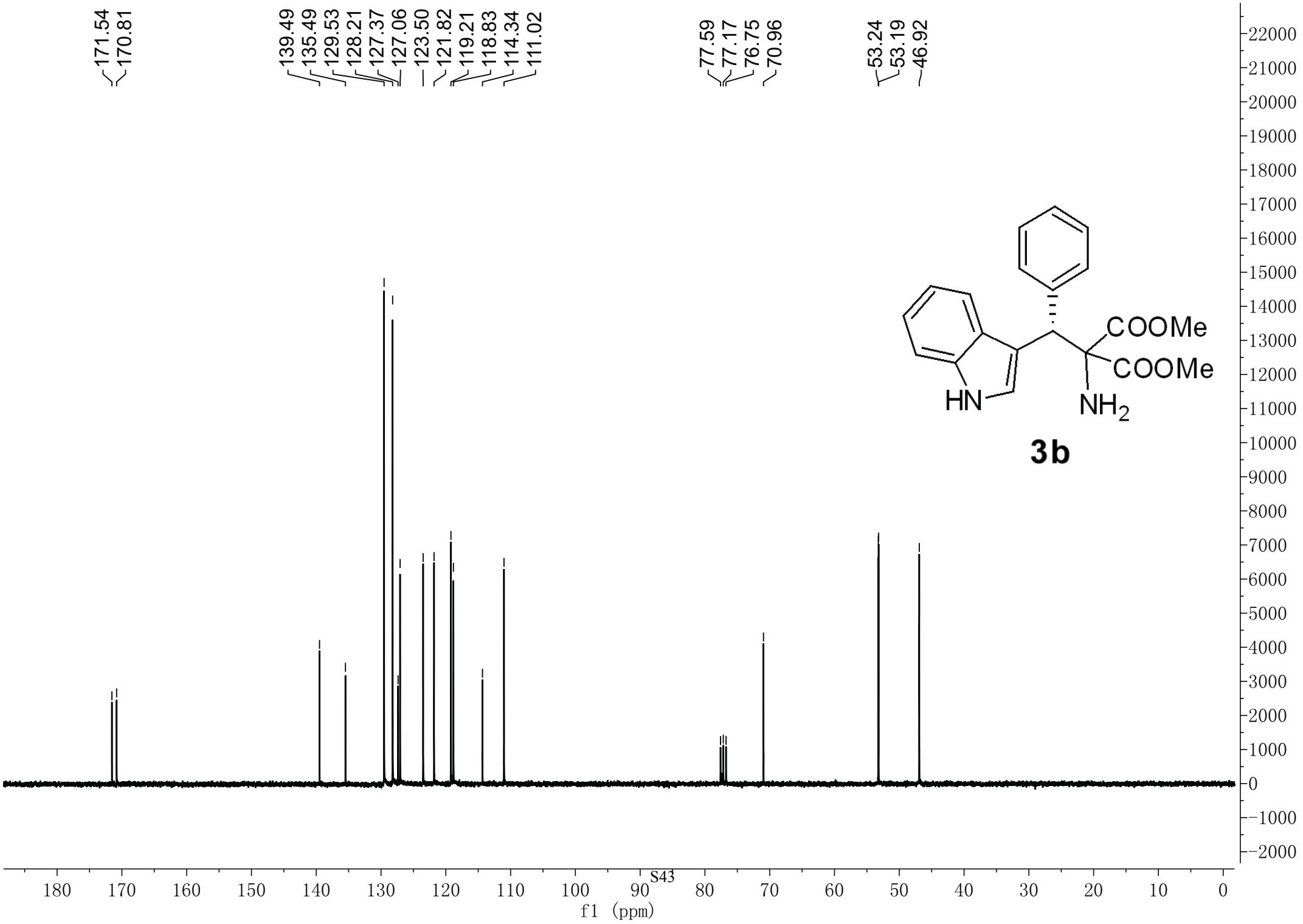
3.04
0.96
4.18
1.09
1.00
3.02
3.13
2.18
f1 (ppm)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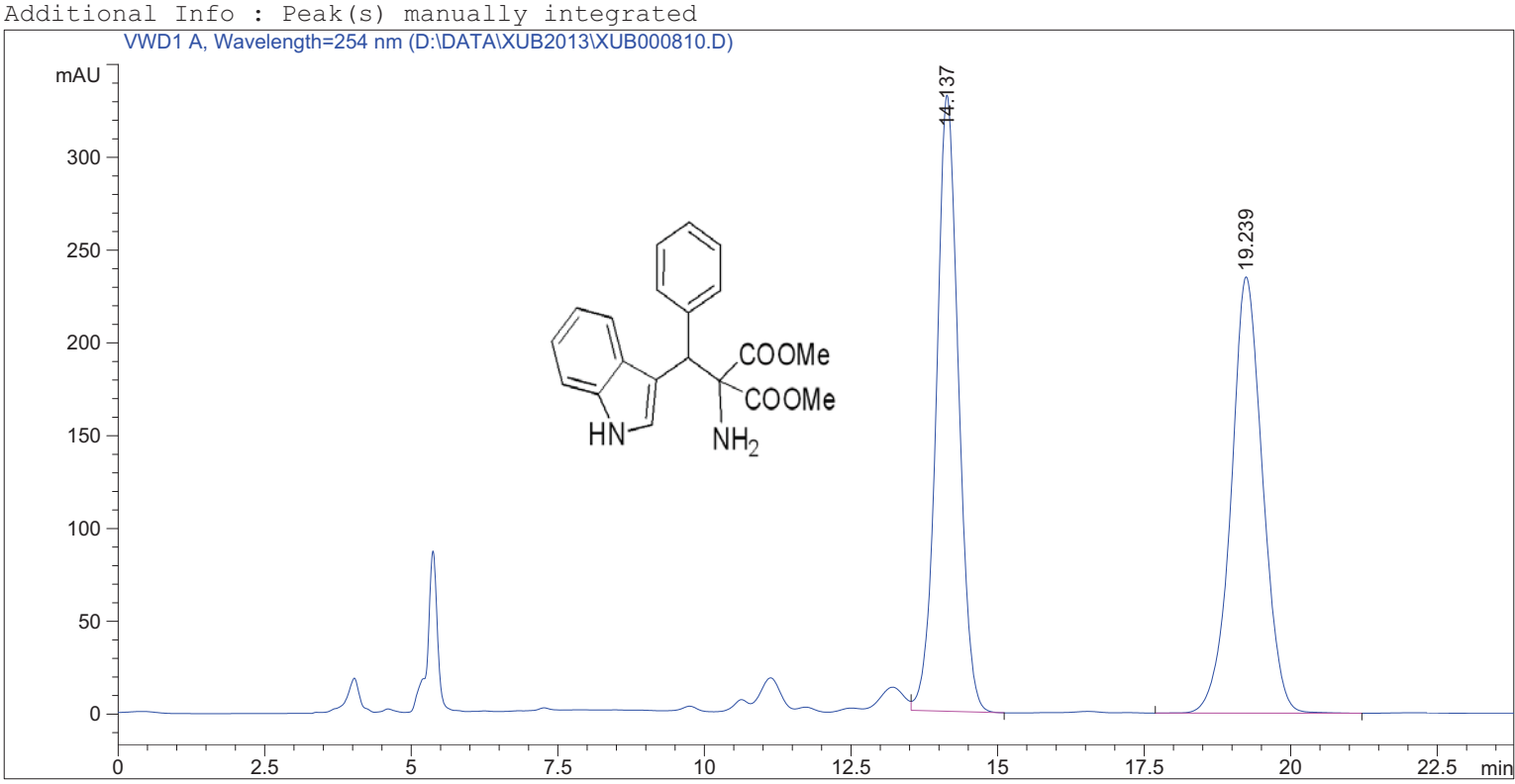
127.06
123.50
121.82
119.21
118.83
114.34
111.02
77.59
77.17
76.75
70.96
53.24
53.19
46.92
COOMe
COOMe
HN
NH2
3b
f1 (ppm)

Data File D:\DATA\XUB2013\XUB000810.D
Sample Name: SLL2013-4-17

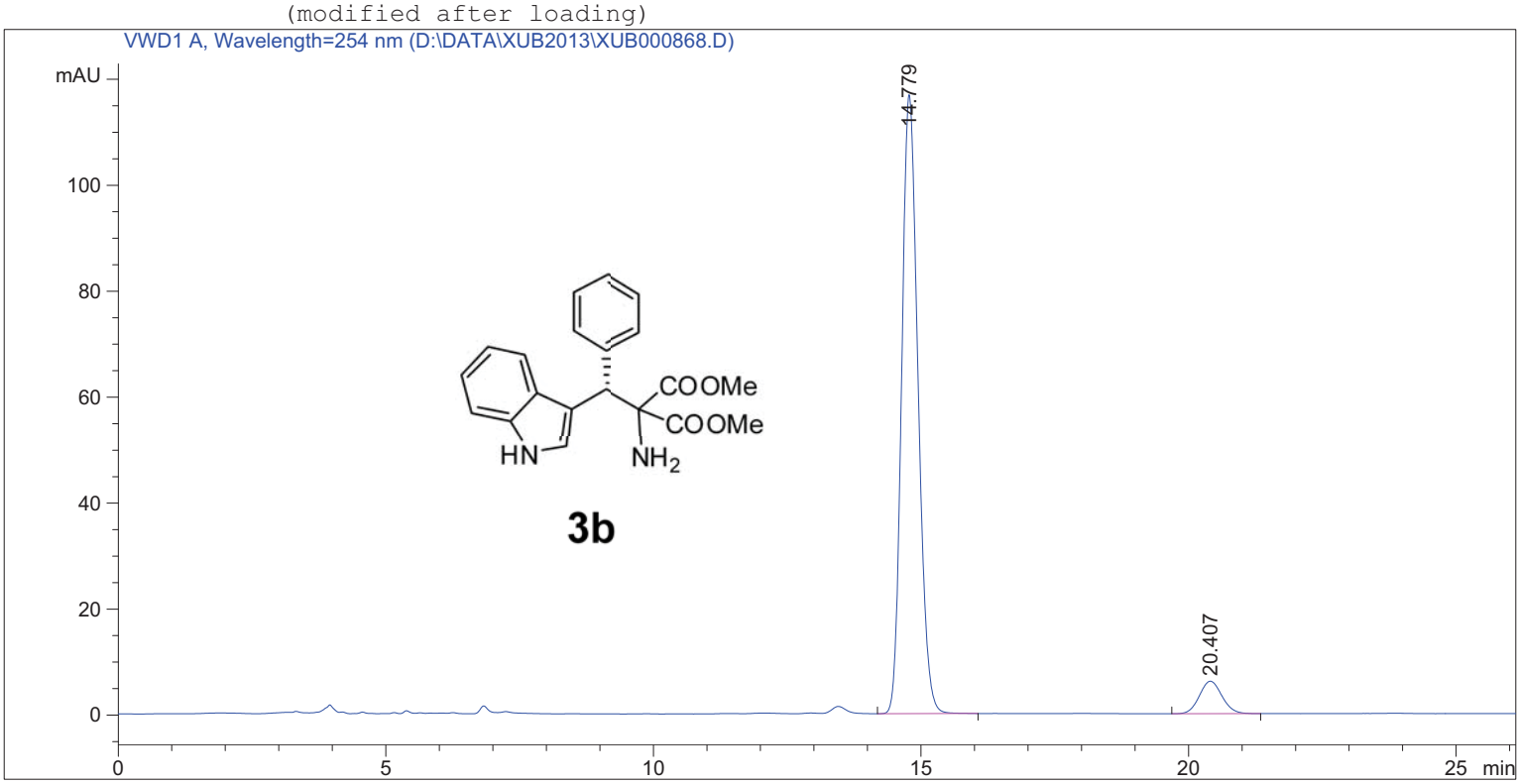
```
================================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 4/19/2013 11:43:18 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_20_1.M
Last changed    : 4/19/2013 11:42:56 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/8/2013 8:57:22 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



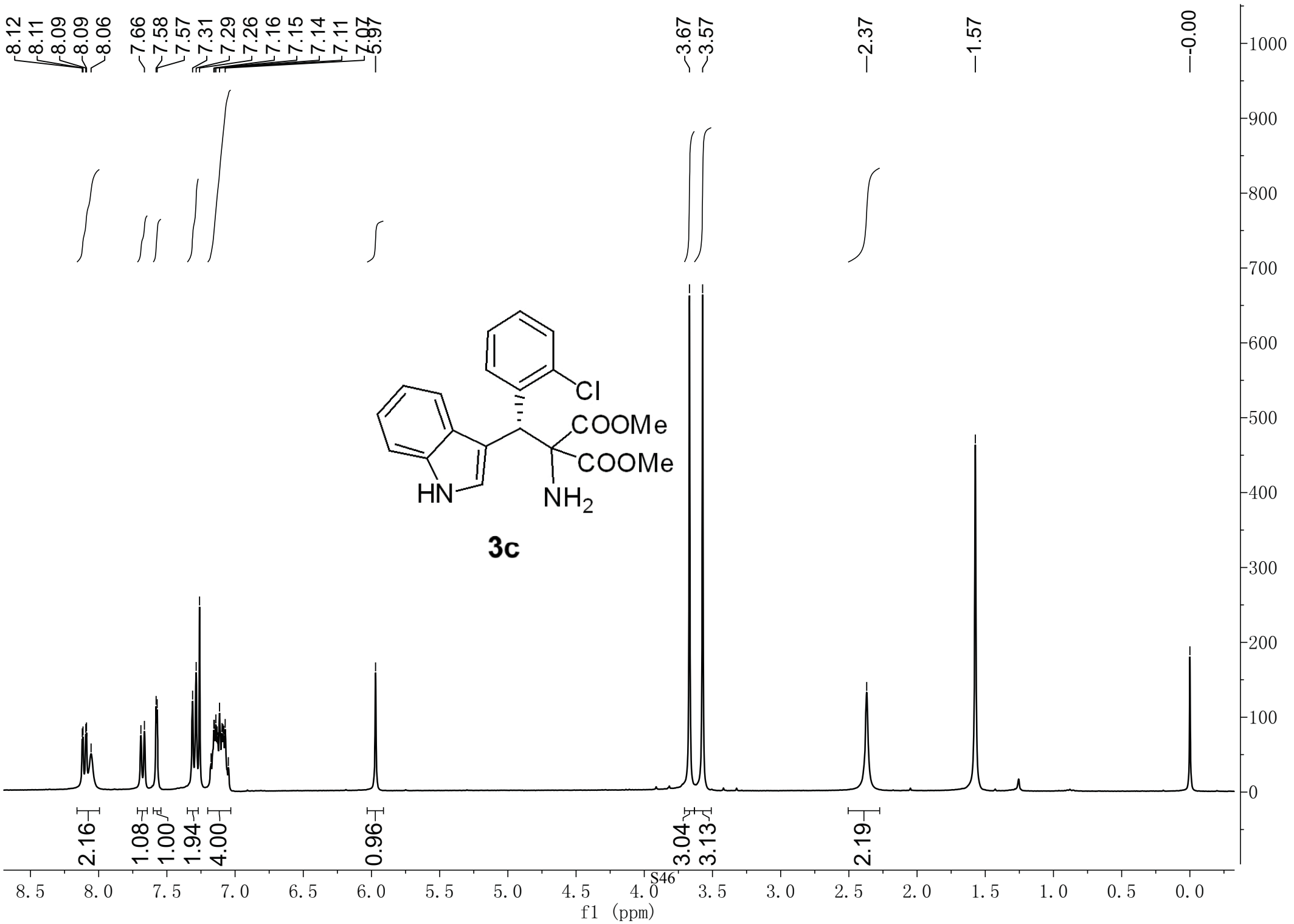

```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.137 VB    0.4006 8703.84961  331.93762  49.7863
   2  19.239 BB    0.5623 8778.56152  235.10524  50.2137

Totals :                  1.74824e4   567.04286


================================================================================
                          *** End of Report ***
```

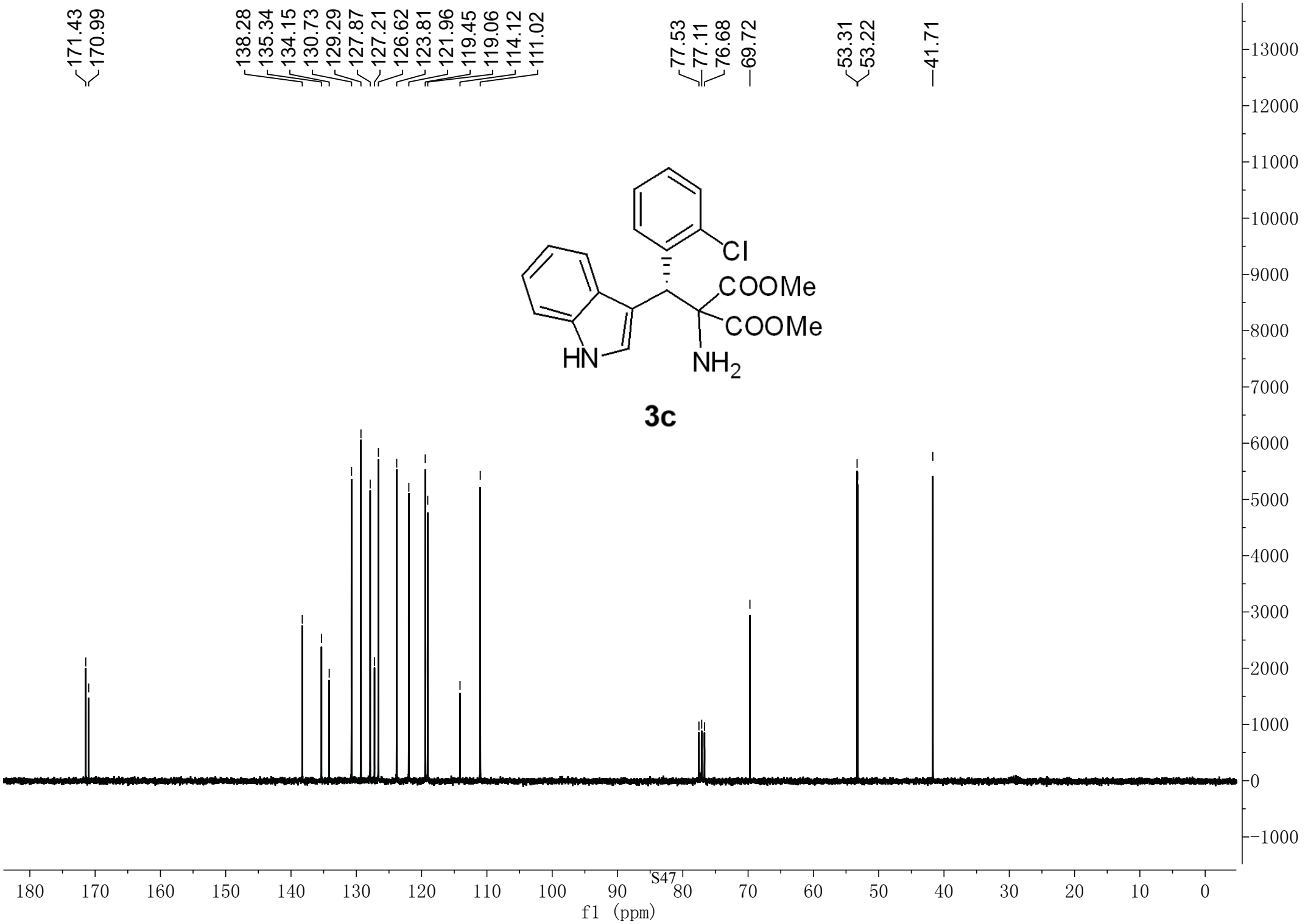
Data File D:\DATA\XUB2013\XUB000868.D
Sample Name: XUB2013-5-1-2

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/3/2013 9:43:19 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_20_1.M
Last changed    : 5/3/2013 9:42:54 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/8/2013 8:57:56 AM by LNF
                  (modified after loading)
```



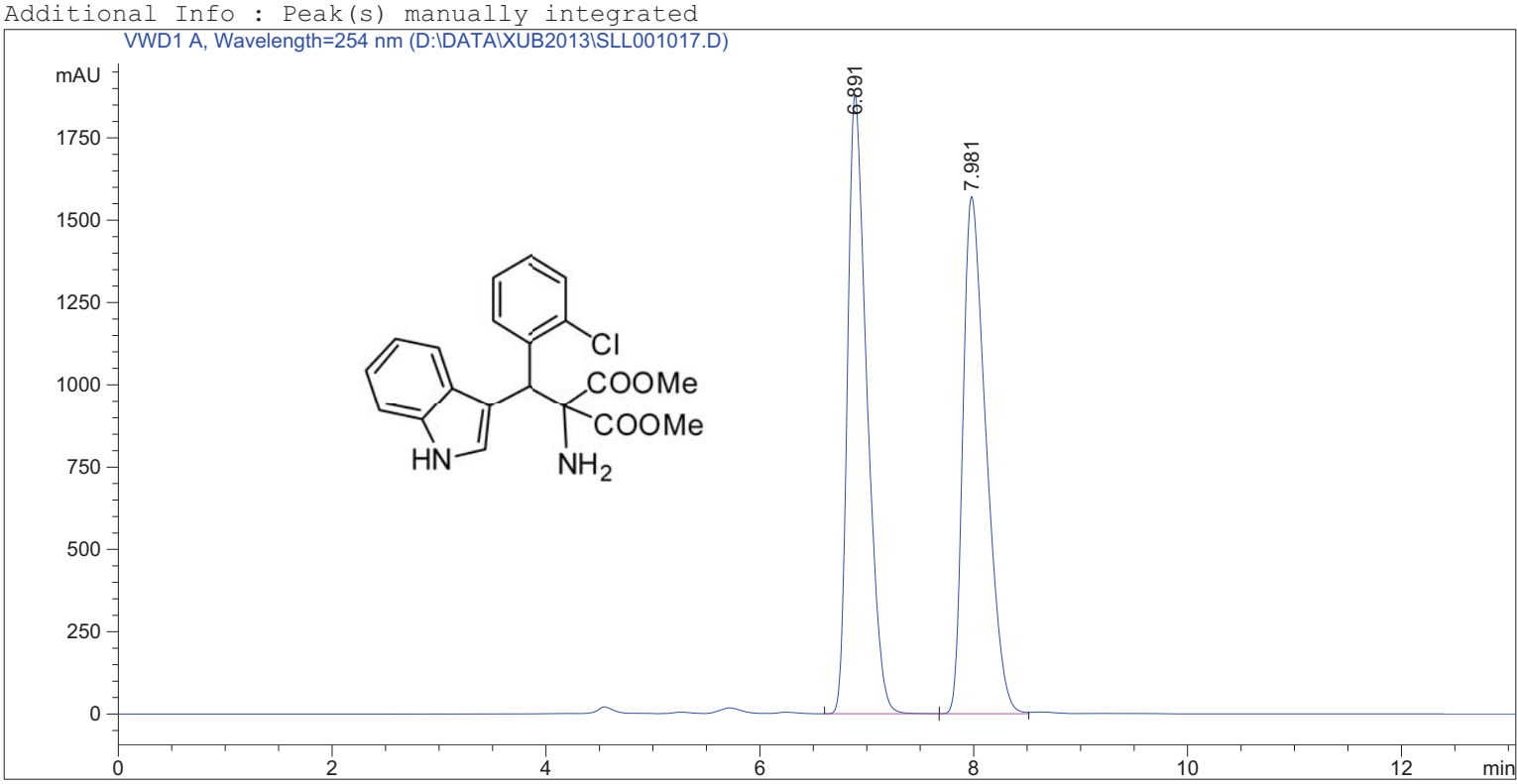

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.779 BB    0.3285 2472.99829  116.80853  93.2104
   2  20.407 BB    0.4503  180.13809    6.13452   6.7896

Totals :                  2653.13638  122.94305

==========================================================================
                          *** End of Report ***
```


8.12
8.11
8.09
8.09
8.06
7.66
7.58
7.57
7.31
7.29
7.26
7.16
7.15
7.14
7.11
7.07
5.97
3.67
3.57
2.37
1.57
0.00
Cl
COOMe
COOMe
HN
NH2
3c
2.16
1.08
1.00
1.94
4.00
0.96
3.04
3.13
2.19
f1 (ppm)


171.43
170.99
138.28
135.34
134.15
130.73
129.29
127.87
127.21
126.62
123.81
121.96
119.45
119.06
114.12
111.02
77.53
77.11
76.68
69.72
53.31
53.22
41.71
Cl
COOMe
COOMe
HN
NH2
3c
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001017.D
Sample Name: SLL2013-6-23-1-0

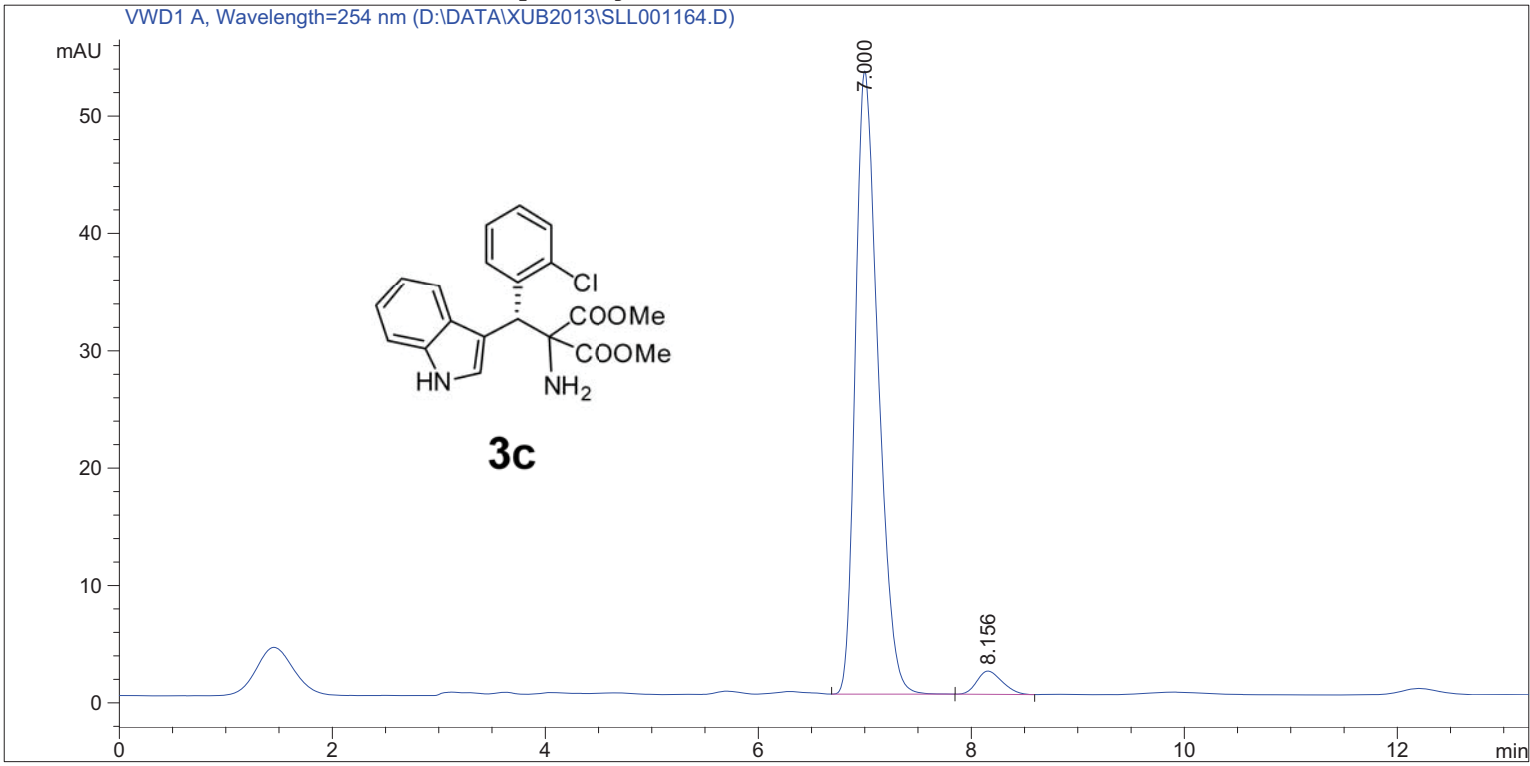
```
================================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/26/2013 10:09:29 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 6/26/2013 10:08:58 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 8/8/2013 3:22:33 PM by xub
Additional Info : Peak(s) manually integrated
```



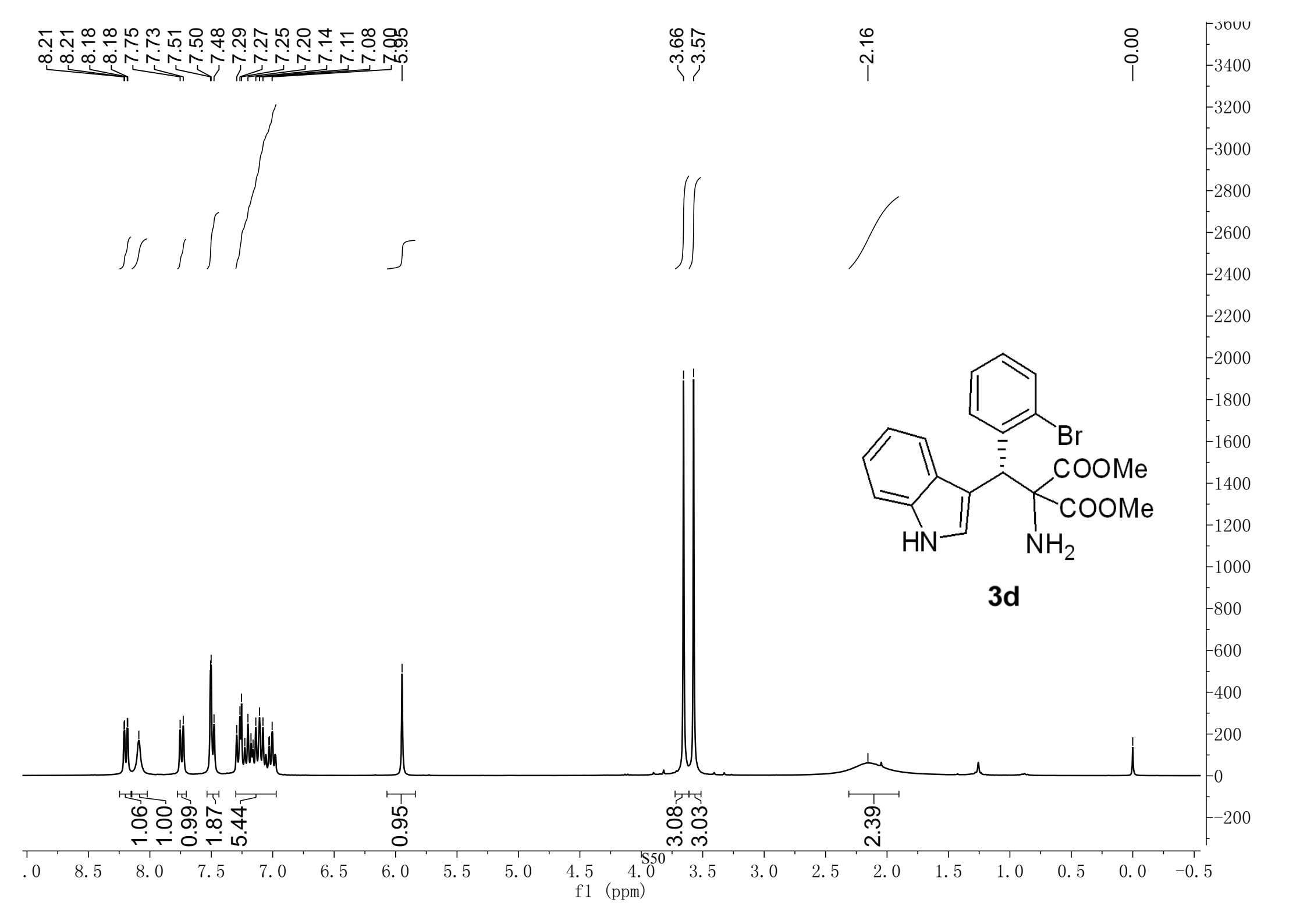

```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.891 BB    0.1873 2.36172e4  1880.49500  50.0661
   2   7.981 BV    0.2259 2.35549e4  1570.83105  49.9339

Totals :                  4.71721e4  3451.32605

================================================================================
                          *** End of Report ***
```

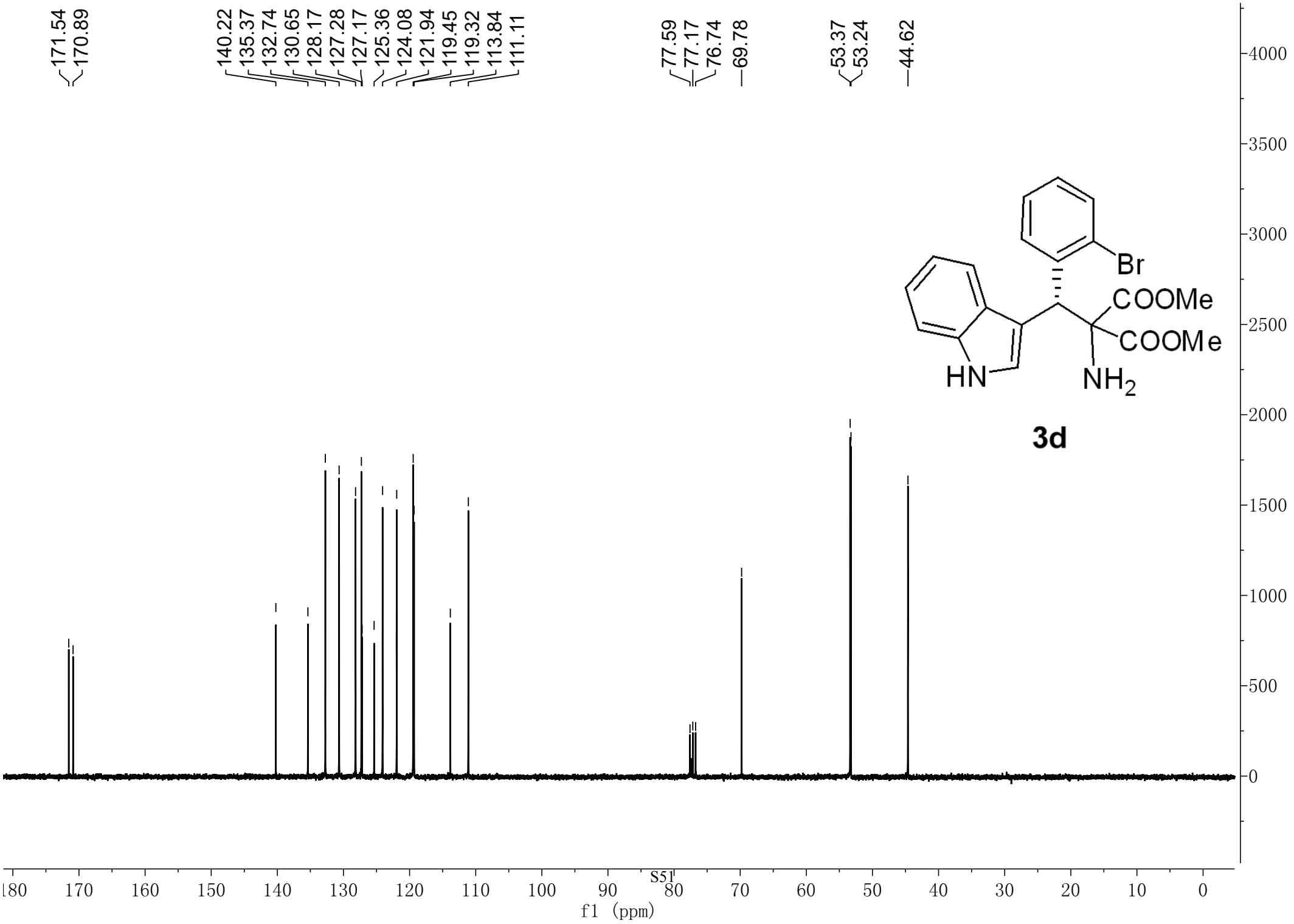
Data File D:\DATA\XUB2013\SLL001164.D
Sample Name: xub-2013-8-2-2

```
==========================================================================
Acq. Operator    : xub
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 8/6/2013 10:50:29 AM
                                                  Inj Volume : No inj
Acq. Method      : D:\METHOD\AD_30_1.M
Last changed     : 8/6/2013 10:49:30 AM by xub
Analysis Method : D:\METHOD\AD_30_1.M
Last changed     : 8/8/2013 3:22:33 PM by xub
Additional Info : Peak(s) manually integrated
```



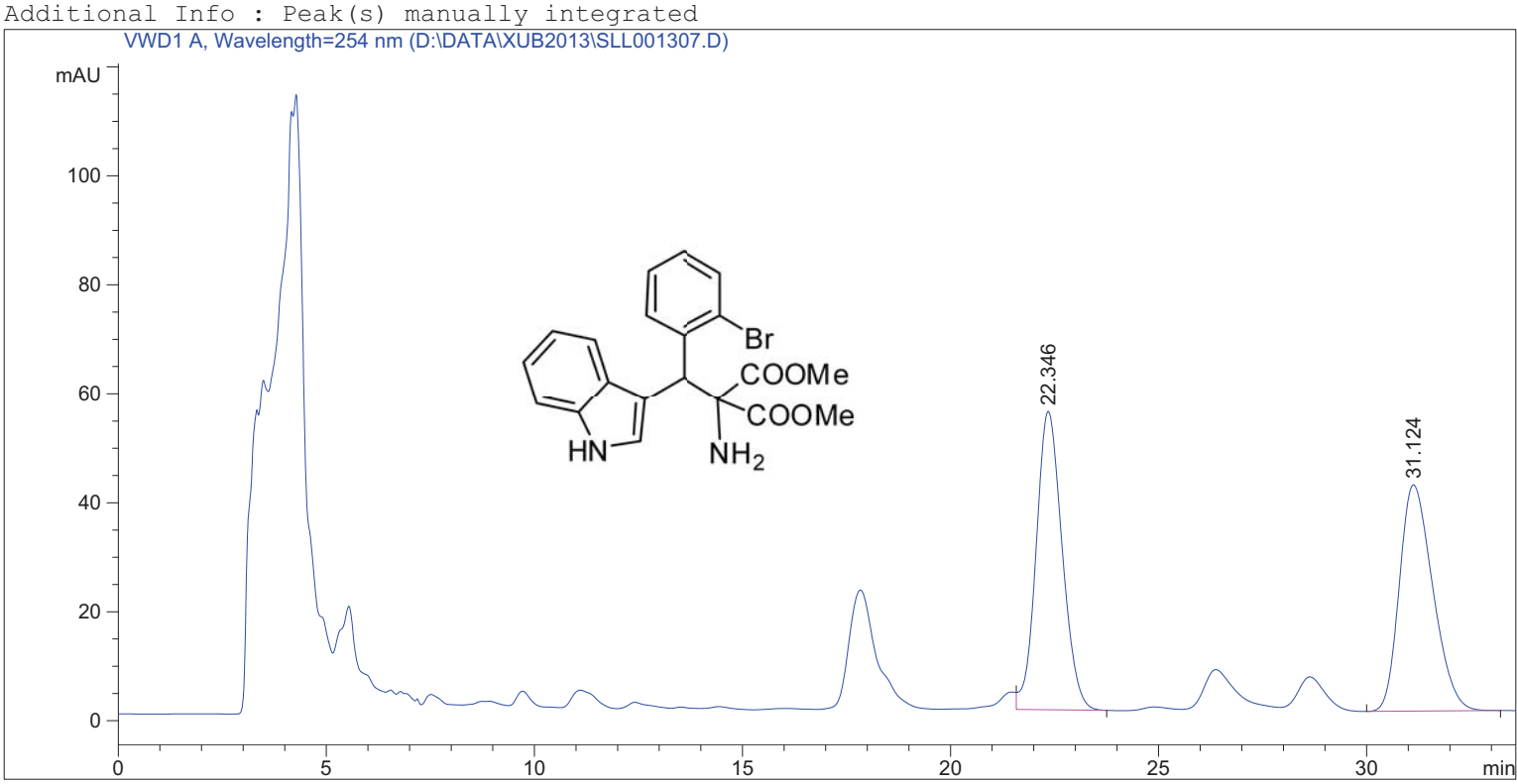

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.000 BB    0.2260  789.91754   53.10199  96.0174
   2   8.156 BB    0.2522   32.76384    1.98555   3.9826

Totals :                   822.68138   55.08755


==========================================================================
                          *** End of Report ***
```


8.21
8.21
8.18
8.18
7.75
7.73
7.51
7.50
7.48
7.29
7.27
7.25
7.20
7.14
7.11
7.08
7.00
5.95
3.66
3.57
2.16
0.00
Br
COOMe
COOMe
HN
NH2
3d
1.06
1.00
0.99
1.87
5.44
0.95
3.08
3.03
2.39
f1 (ppm)


171.54
170.89
140.22
135.37
132.74
130.65
128.17
127.28
127.17
125.36
124.08
121.94
119.45
119.32
113.84
111.11
77.59
77.17
76.74
69.78
53.37
53.24
44.62
Br
COOMe
COOMe
HN
NH2
3d
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001307.D
Sample Name: SLL2013-6-23-2-0

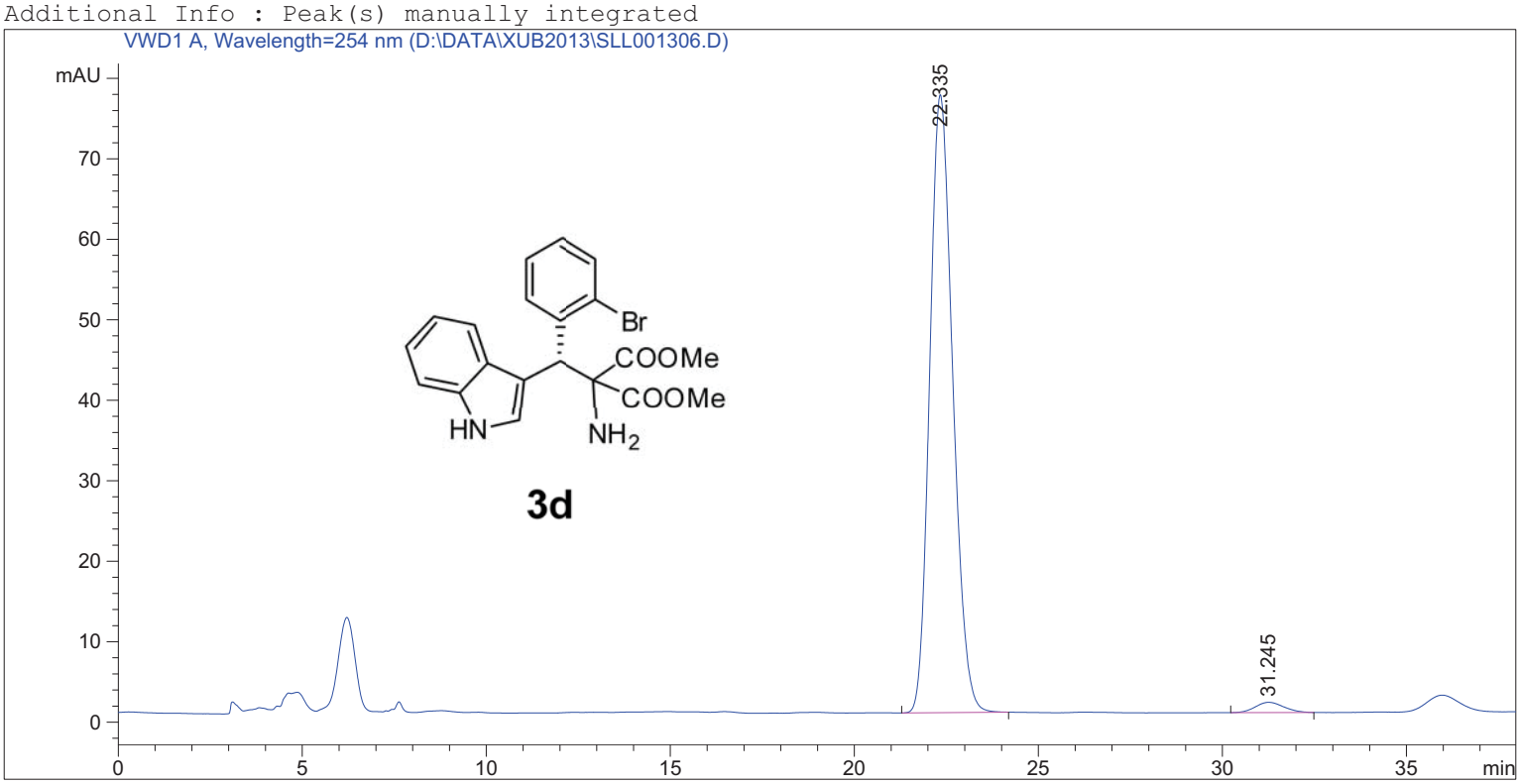
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 10/6/2013 11:24:00 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_10_1.M
Last changed    : 10/6/2013 11:23:31 AM by LNF
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/6/2013 11:58:14 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



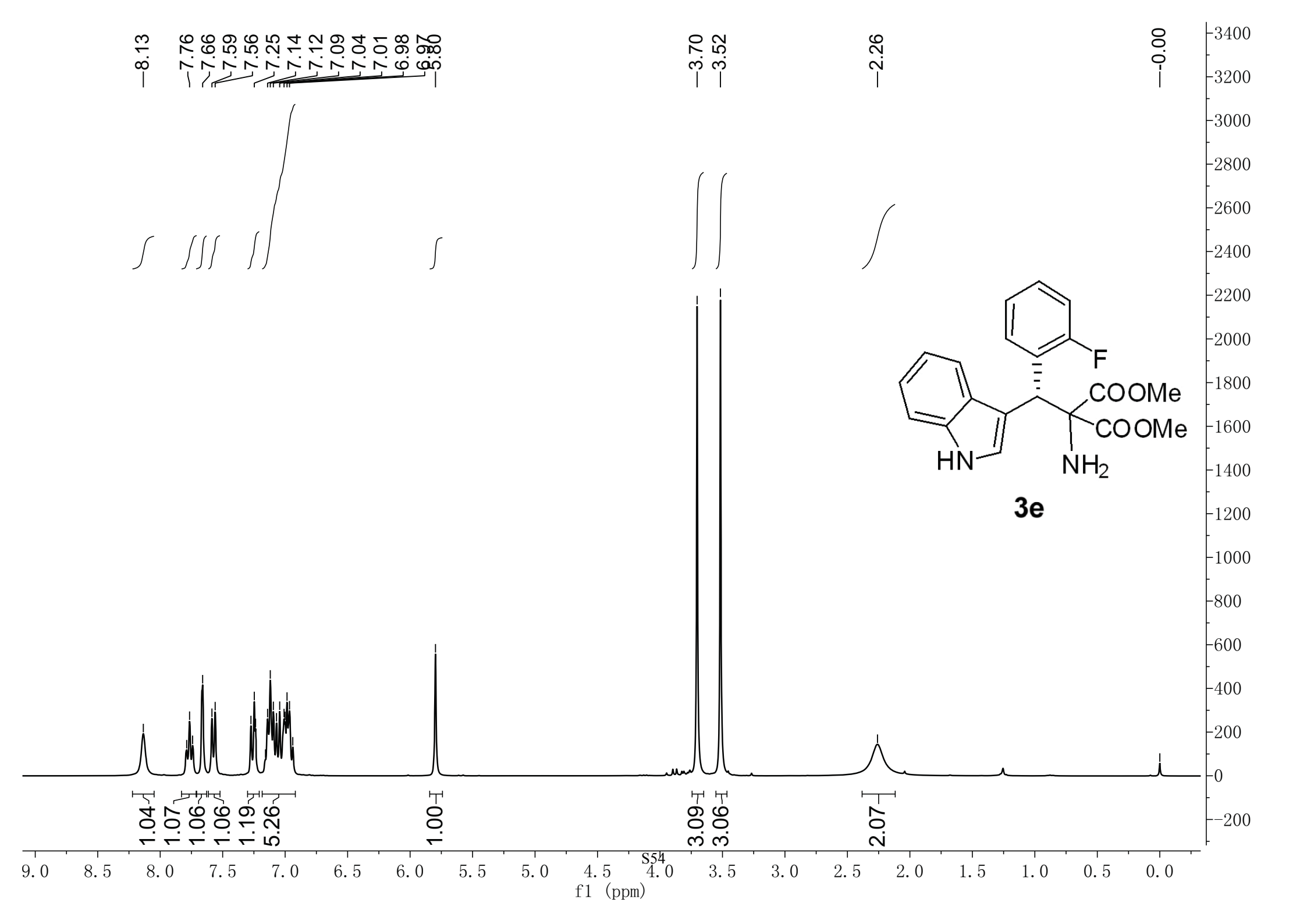

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  22.346 VB    0.6693 2382.89331   54.75752  50.7084
   2  31.124 BB    0.8647 2316.31104   41.53075  49.2916

Totals :                  4699.20435   96.28827

==========================================================================
                          *** End of Report ***
```

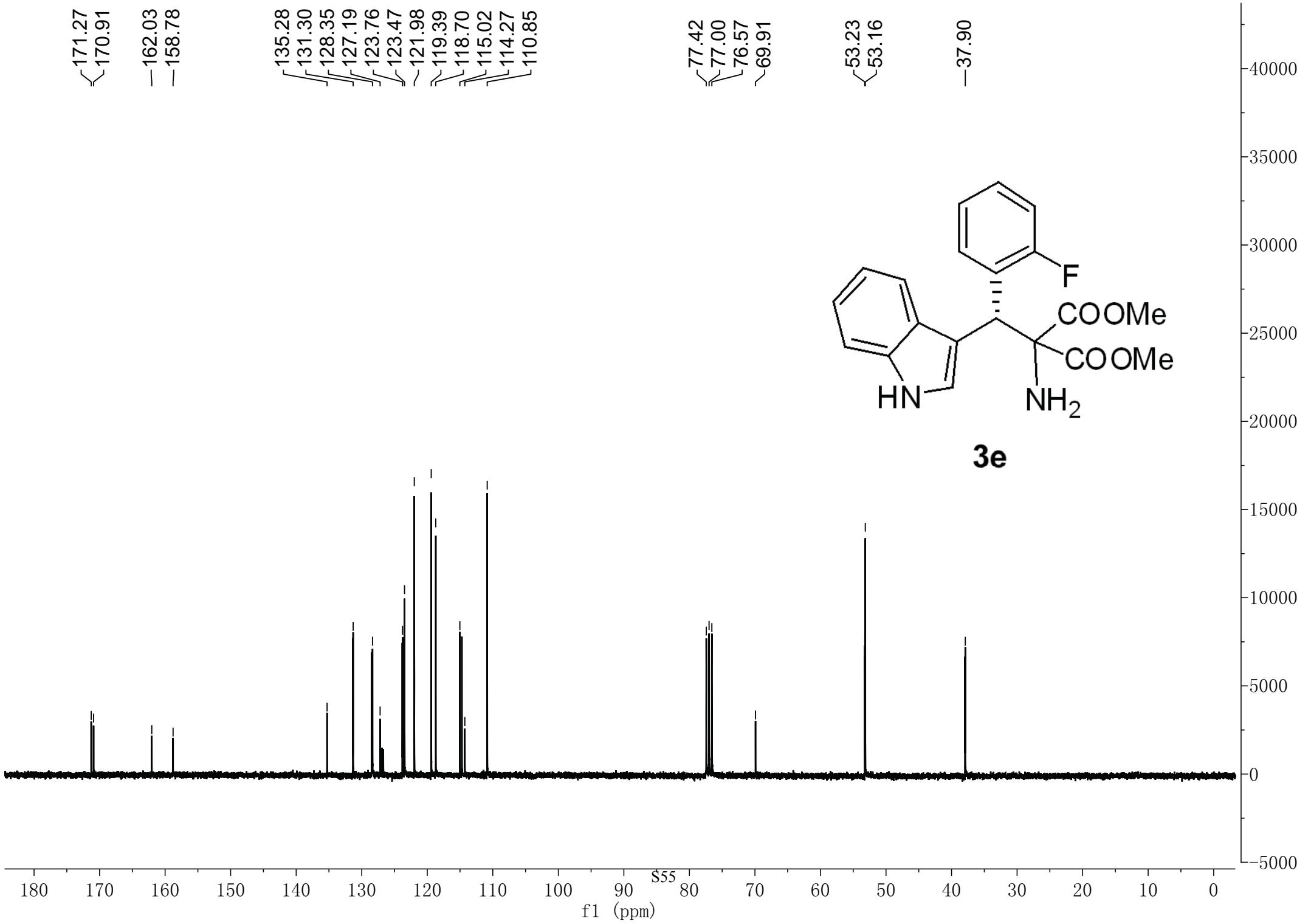
Data File D:\DATA\XUB2013\SLL001306.D
Sample Name: SLL2013-6-27-2

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 10/6/2013 10:45:28 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_10_1.M
Last changed    : 10/6/2013 10:44:54 AM by LNF
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/6/2013 11:24:44 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



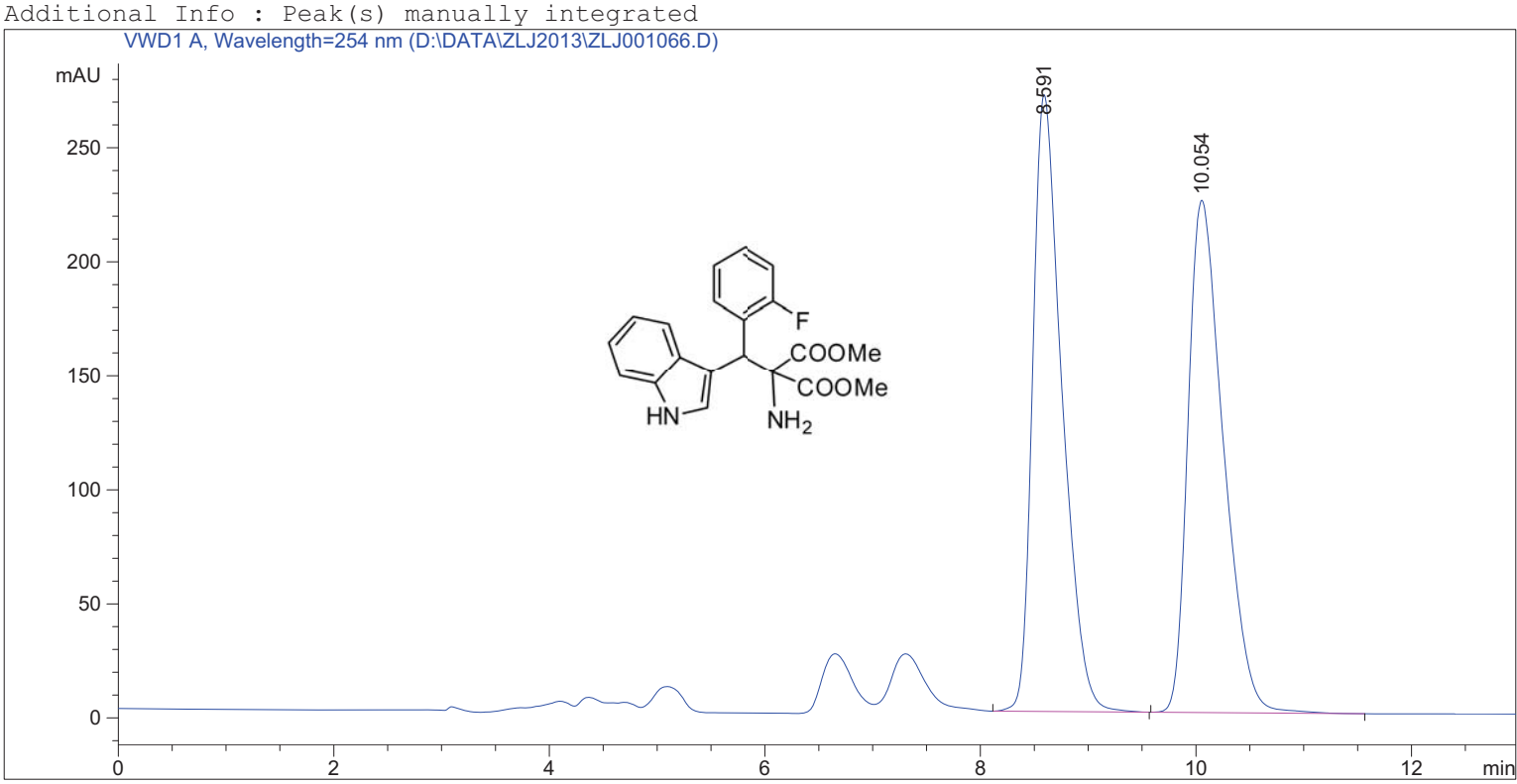

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  22.335 BB    0.6717 3337.86548   76.77780  98.0064
   2  31.245 BB    0.6372   67.89854    1.26936   1.9936

Totals :                  3405.76402   78.04716

==========================================================================
                          *** End of Report ***
```


8.13
7.76
7.66
7.59
7.56
7.25
7.14
7.12
7.09
7.04
7.01
6.98
6.97
5.80
3.70
3.52
2.26
0.00
F
COOMe
COOMe
HN
NH2
3e
1.04
1.07
1.06
1.06
1.19
5.26
1.00
3.09
3.06
2.07
f1 (ppm)


171.27
170.91
162.03
158.78
135.28
131.30
128.35
127.19
123.76
123.47
121.98
119.39
118.70
115.02
114.27
110.85
77.42
77.00
76.57
69.91
53.23
53.16
37.90
F
COOMe
COOMe
HN
NH2
3e
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Data File D:\DATA\ZLJ2013\ZLJ001066.D
Sample Name: ZLJ-7-2-2-0

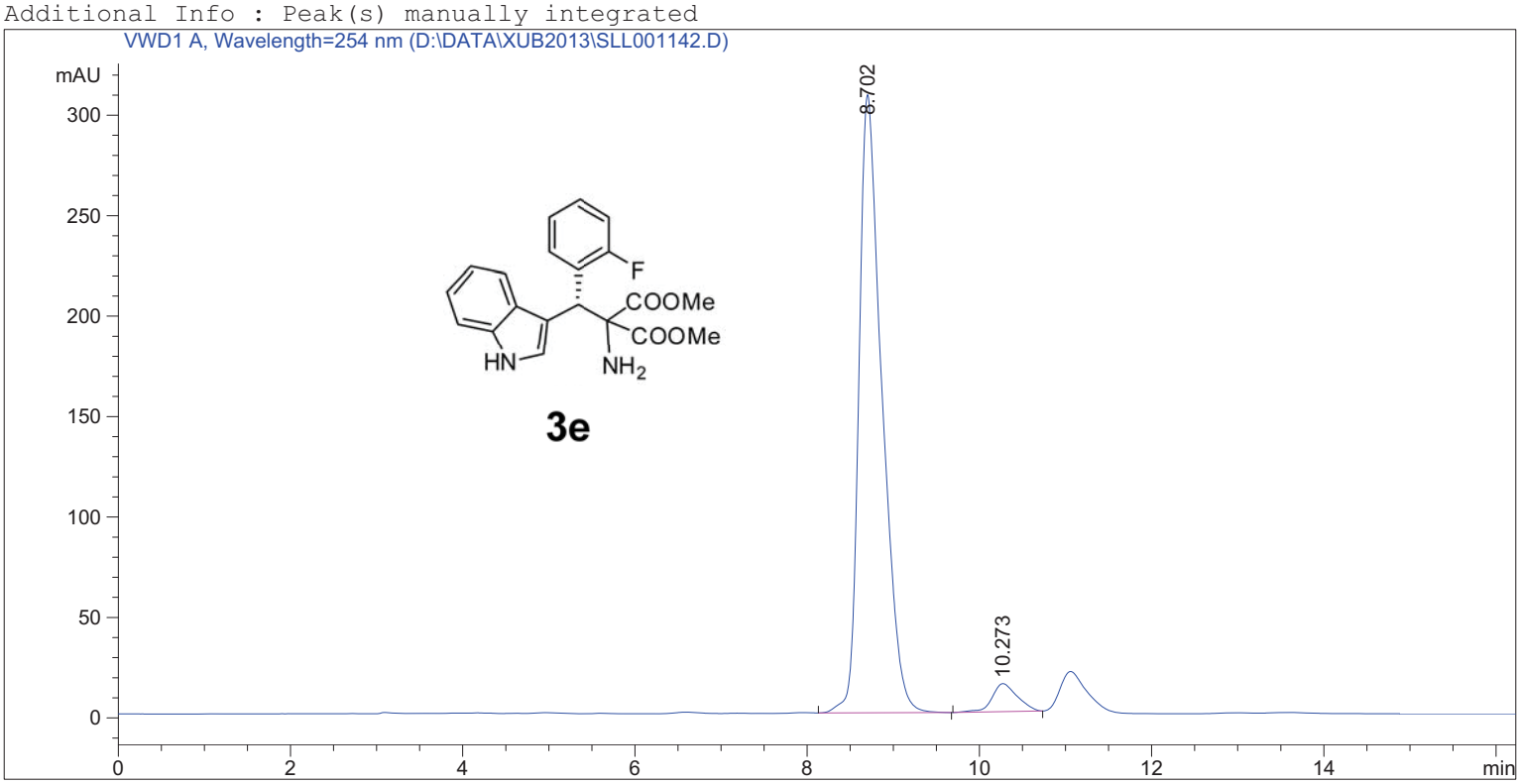
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/9/2013 4:19:03 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\IA_20_1.M
Last changed    : 11/22/2012 9:07:52 AM by LNF
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:30:09 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



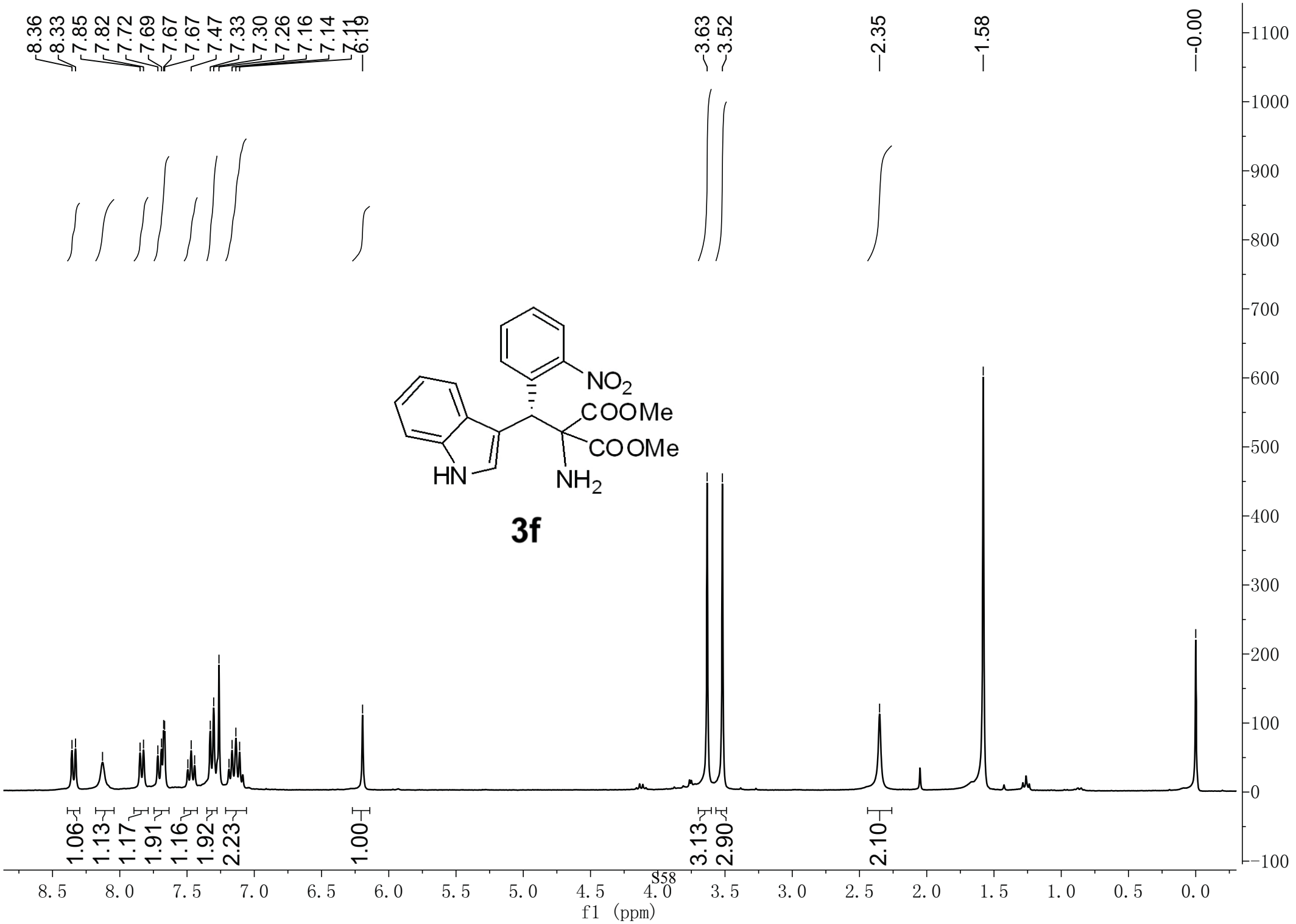

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.591 BB    0.2815 5107.94629  270.44263  49.9923
   2  10.054 BB    0.3425 5109.52393  224.63335  50.0077

Totals :                  1.02175e4   495.07597

==========================================================================
                          *** End of Report ***
```

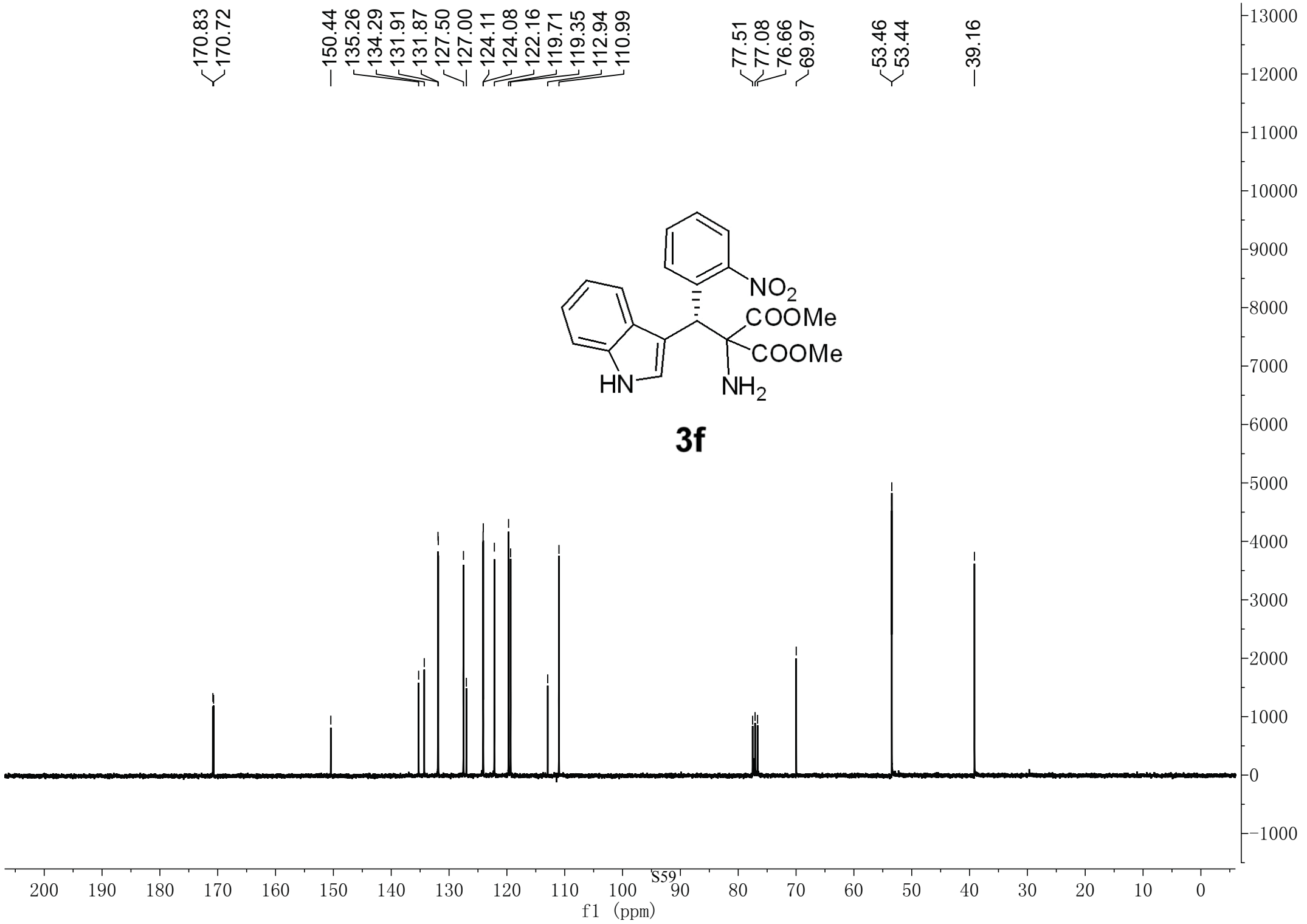
Data File D:\DATA\XUB2013\SLL001142.D
Sample Name: SLL2013-7-30-2

```
==========================================================================
Acq. Operator   : xub
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 8/1/2013 3:45:01 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\IA_20_1.M
Last changed    : 8/1/2013 3:42:00 PM by xub
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:30:09 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



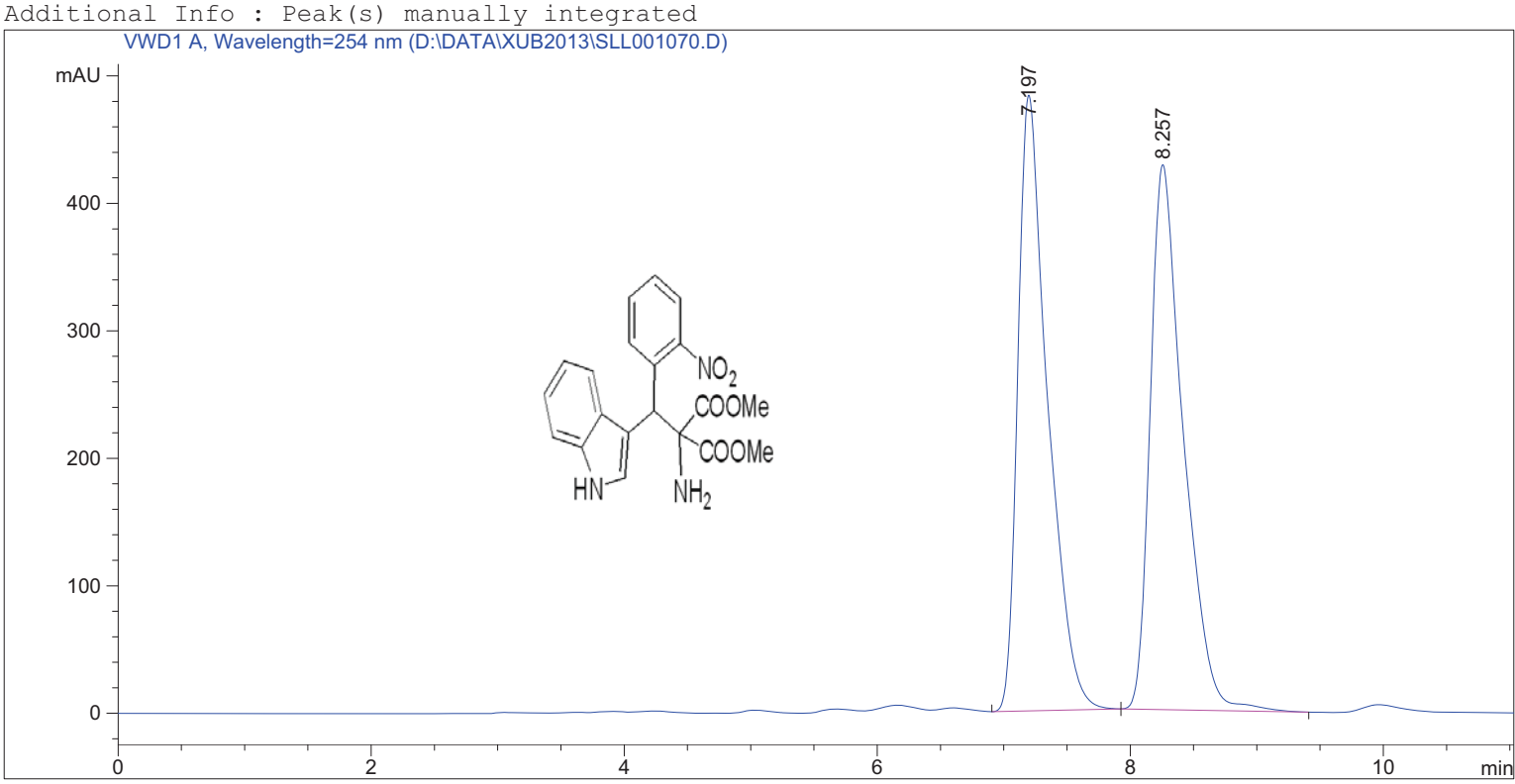

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.702 BB    0.2720 5713.98242  307.62079  95.2568
   2  10.273 BB    0.2986  284.52390   13.98118   4.7432

Totals :                  5998.50632  321.60197

==========================================================================
                          *** End of Report ***
```


8.36
8.33
7.85
7.82
7.72
7.69
7.67
7.67
7.47
7.33
7.30
7.26
7.16
7.14
7.11
6.19
3.63
3.52
2.35
1.58
0.00
NO2
COOMe
COOMe
HN
NH2
3f
1.06
1.13
1.17
1.91
1.16
1.92
2.23
1.00
3.13
2.90
2.10
f1 (ppm)


170.83
170.72
150.44
135.26
134.29
131.91
131.87
127.50
127.00
124.11
124.08
122.16
119.71
119.35
112.94
110.99
77.51
77.08
76.66
69.97
53.46
53.44
39.16
NO2
COOMe
COOMe
HN
NH2
3f
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001070.D
Sample Name: SLL2013-7-3-2-0

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/15/2013 10:41:48 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/15/2013 10:39:22 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:35:21 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

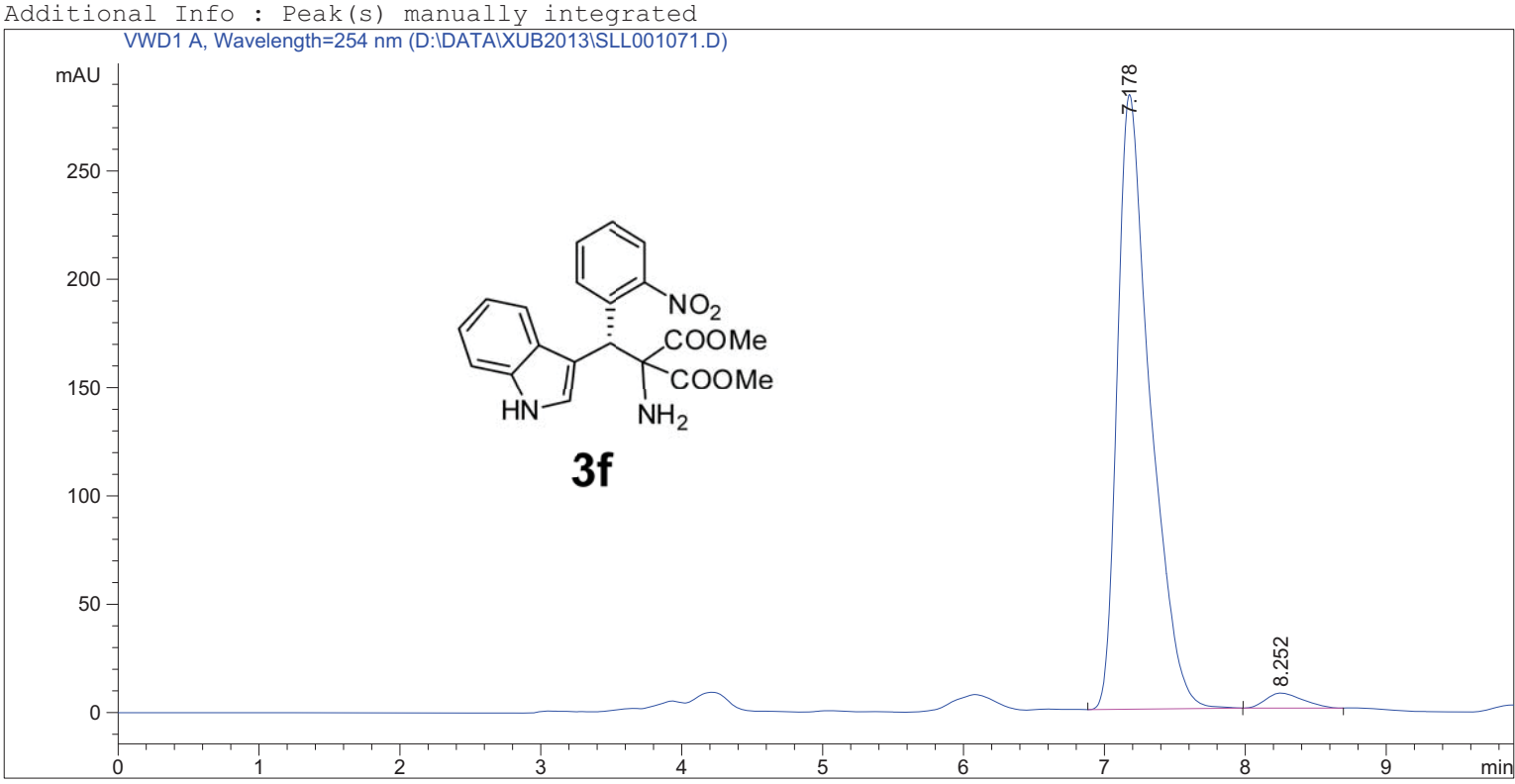



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.197 BB    0.2376 7906.85352  483.02295  50.3175
   2   8.257 BB    0.2669 7807.06787  427.49213  49.6825

Totals :                  1.57139e4   910.51508


==========================================================================
                          *** End of Report ***
```

Data File D:\DATA\XUB2013\SLL001071.D
Sample Name: SLL2013-7-7-1

==========

Acq. Operator : LNF
Location : Vial 1
Injection Date : 7/15/2013 10:53:37 AM
Acq. Method : AD_30_1.M
Analysis Method : D:\METHOD\AD_30_1.M
Last changed : 9/29/2013 9:35:21 AM by LNF
(modified after loading)
Additional Info : Peak(s) manually integrated



==========

Area Percent Report

==========

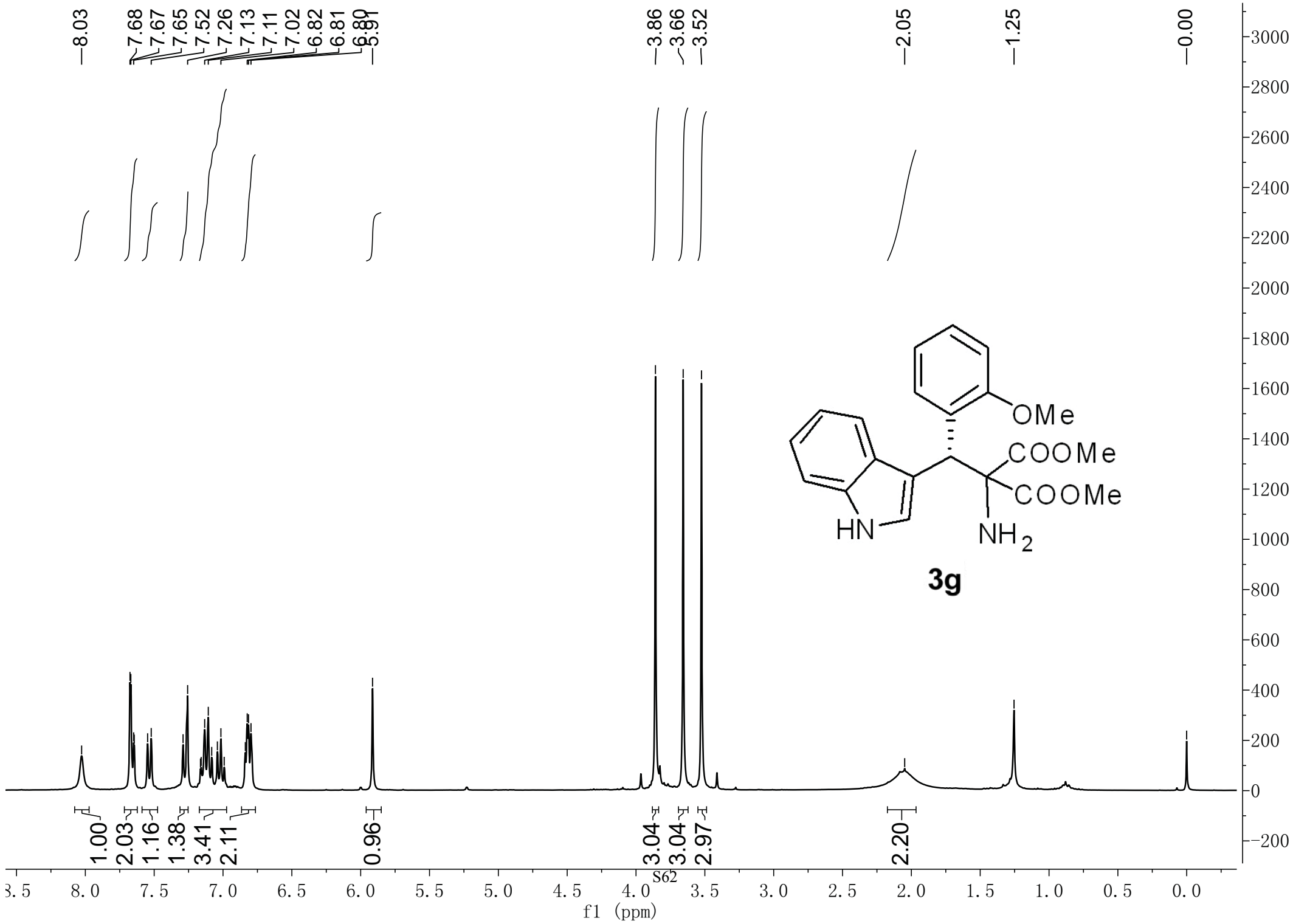
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

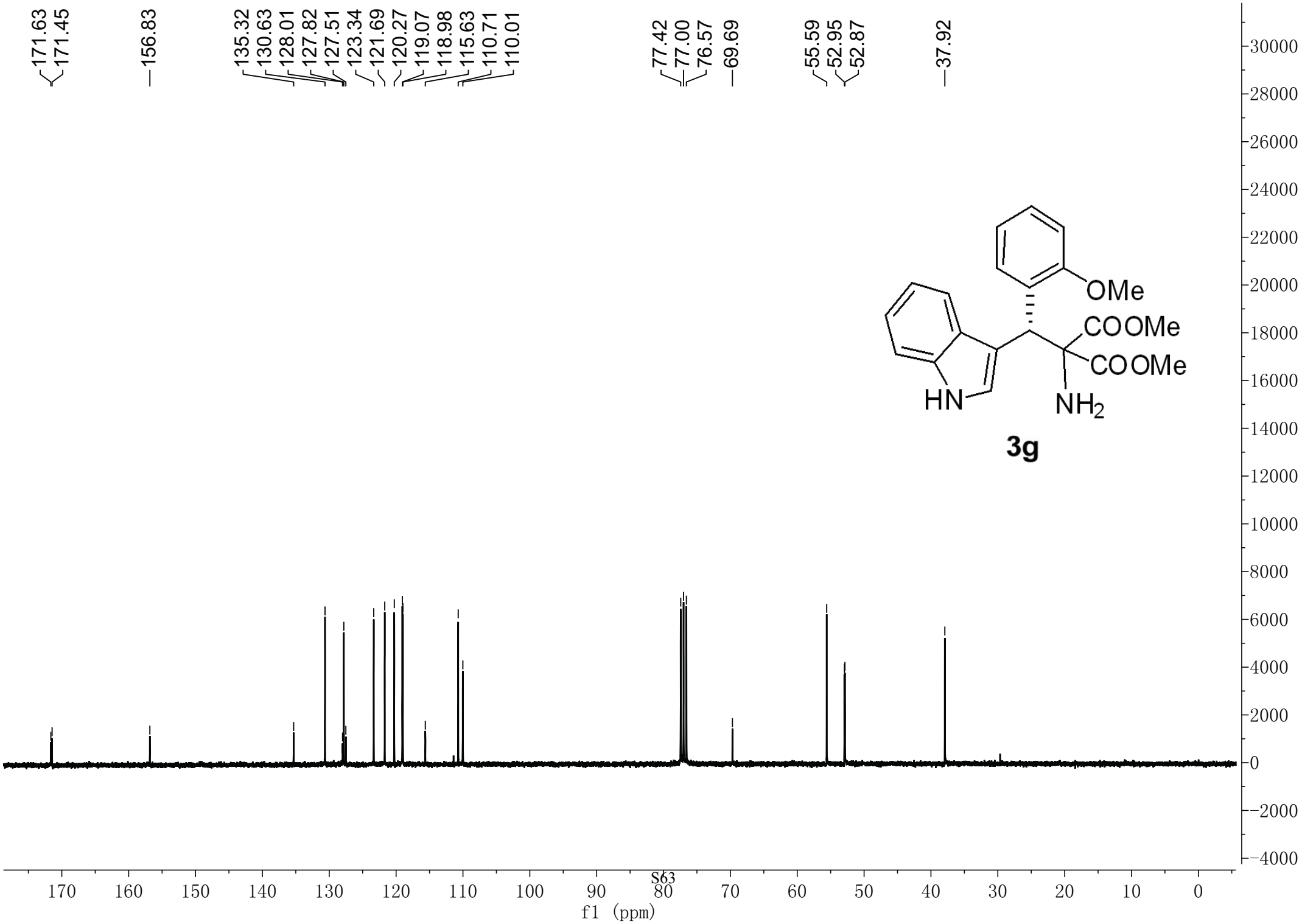
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.178	BB	0.2368	4591.36768	283.76300	97.3220
2	8.252	BB	0.2758	126.34003	6.96057	2.6780
Totals :				4717.70771	290.72357	

==========

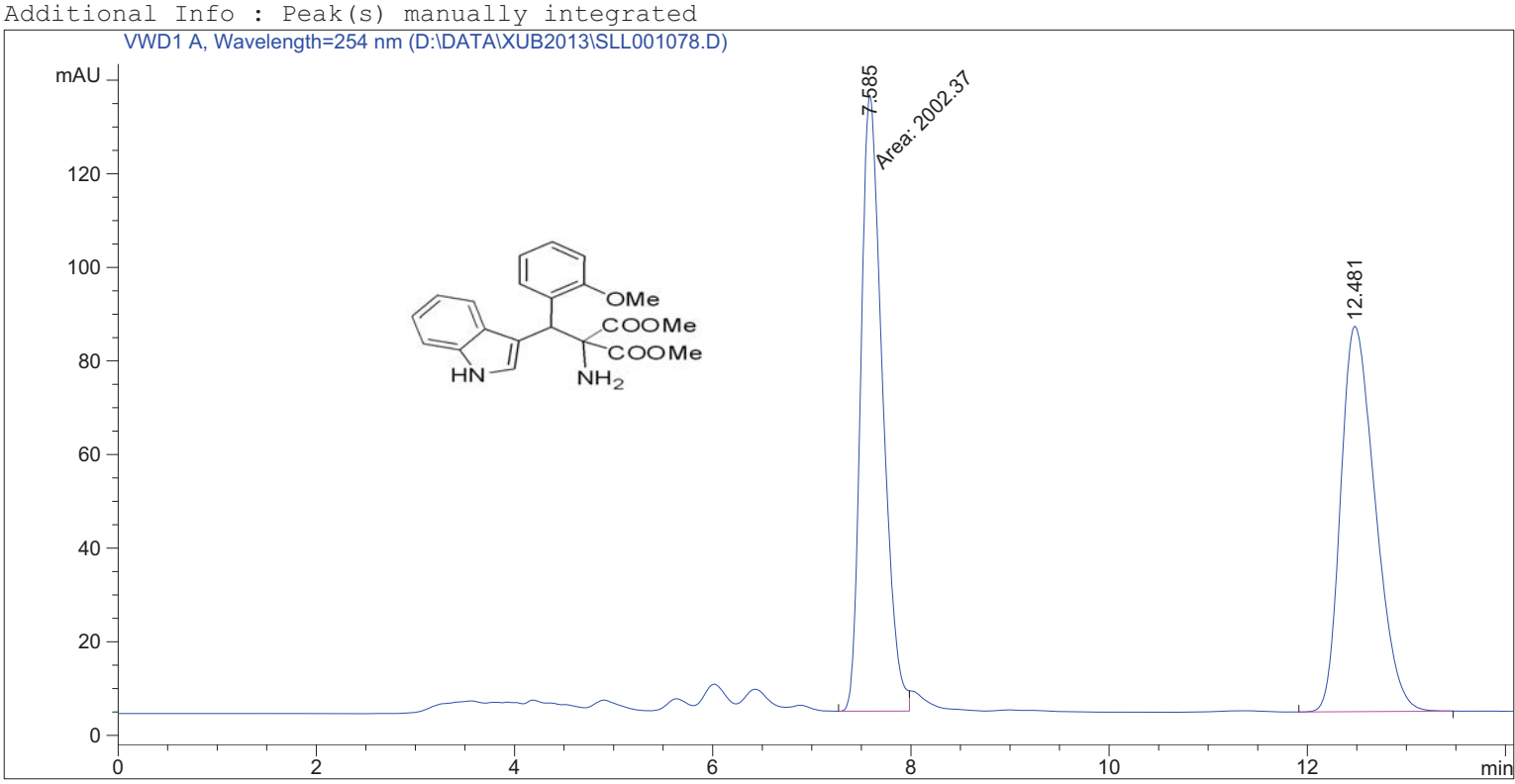
*** End of Report ***


8.03
7.68
7.67
7.65
7.52
7.26
7.13
7.11
7.02
6.82
6.81
6.80
5.91
3.86
3.66
3.52
2.05
1.25
0.00
OMe
COOMe
COOMe
HN
NH2
3g
1.00
2.03
1.16
1.38
3.41
2.11
0.96
3.04
3.04
2.97
2.20
f1 (ppm)


171.63
171.45
156.83
135.32
130.63
128.01
127.82
127.51
123.34
121.69
120.27
119.07
118.98
115.63
110.71
110.01
77.42
77.00
76.57
69.69
55.59
52.95
52.87
37.92
OMe
COOMe
COOMe
HN
NH2
3g
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001078.D
Sample Name: SLL2013-7-17-1-0

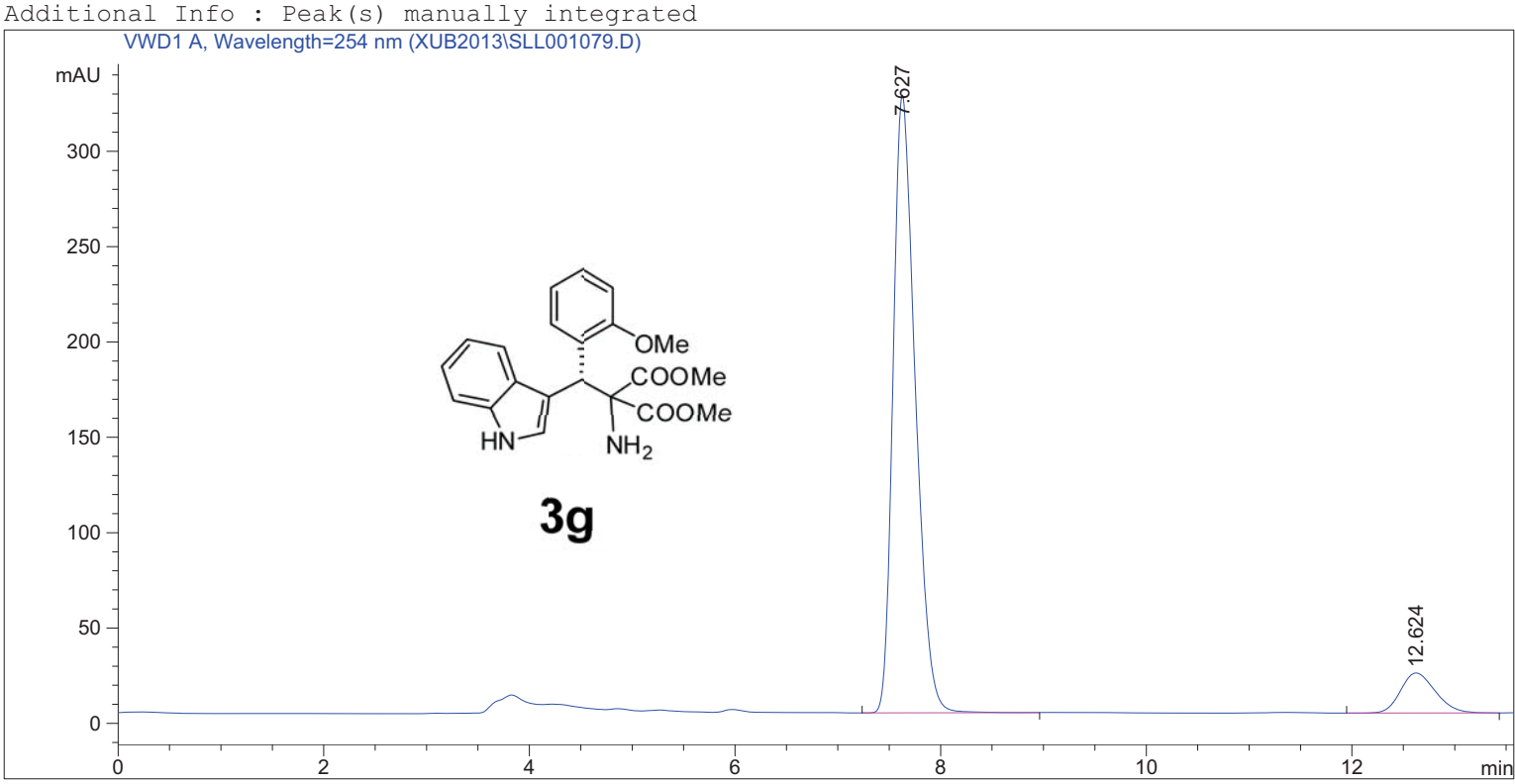
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/18/2013 10:21:03 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/18/2013 10:18:17 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:22:42 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



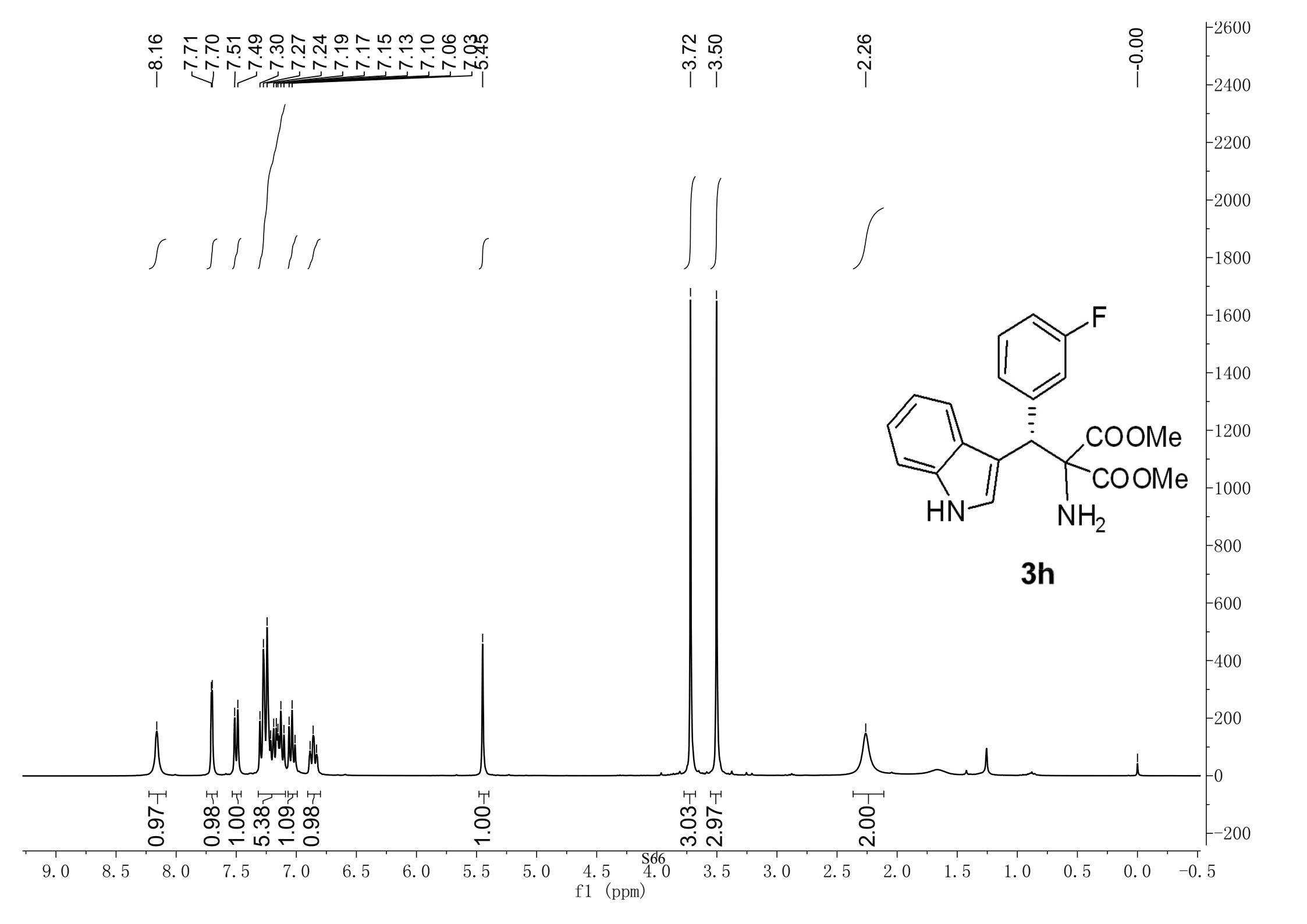

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.585 MF    0.2536 2002.36755  131.62038  50.1002
   2  12.481 BB    0.3757 1994.36133   82.35471  49.8998

Totals :                  3996.72888  213.97509


==========================================================================
                        *** End of Report ***
```

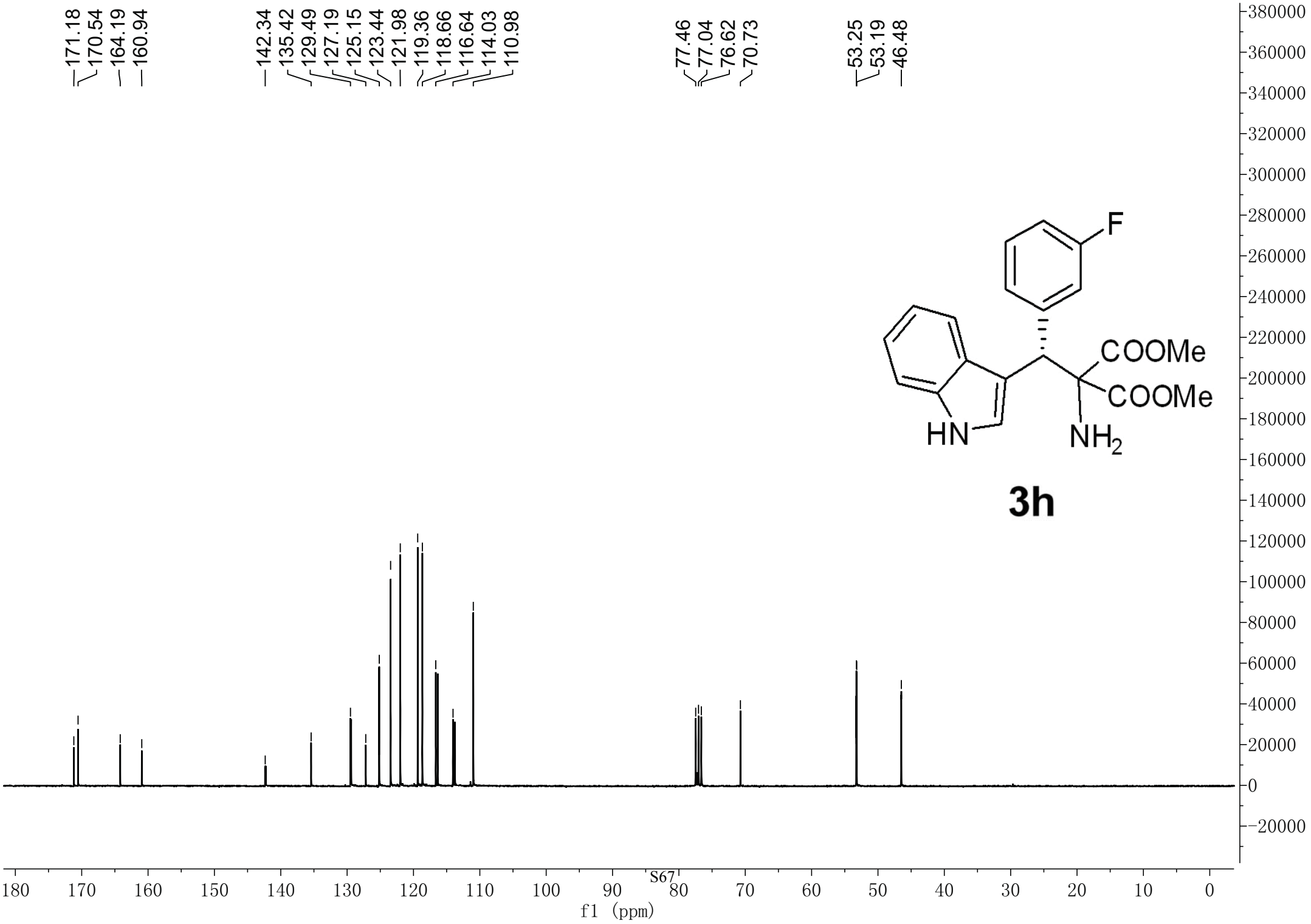
Data File C:\CHEM32\1\DATA\XUB2013\SLL001079.D
Sample Name: SLL2013-7-17-1

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/18/2013 10:36:03 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/18/2013 10:35:14 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:22:42 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



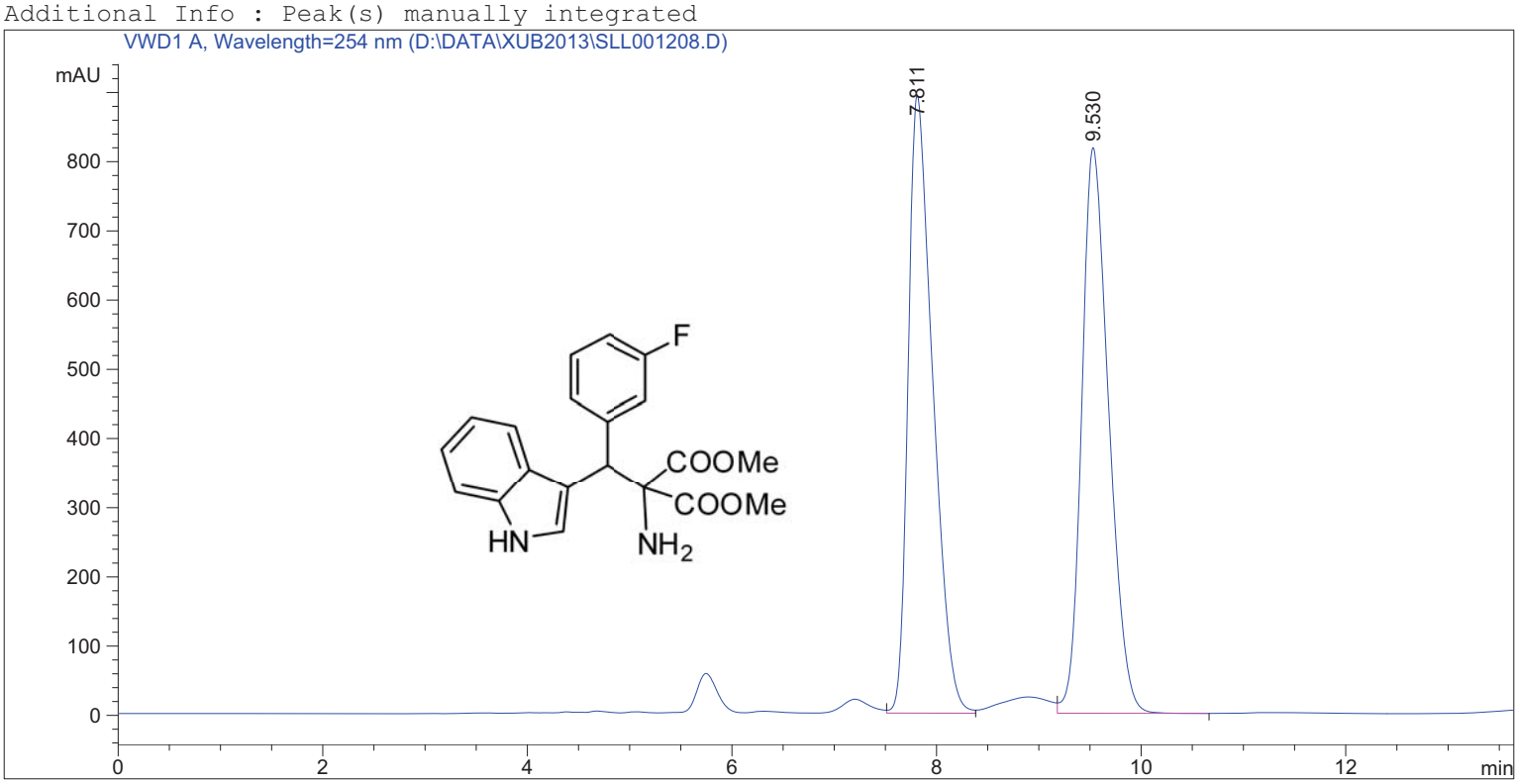

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.627 BB    0.2269 4846.68555  323.99557  90.7786
   2  12.624 BB    0.3641  492.33041   20.97863   9.2214

Totals :                  5339.01596  344.97421

==========================================================================
                        *** End of Report ***
```


8.16
7.71
7.70
7.51
7.49
7.30
7.27
7.24
7.19
7.17
7.15
7.13
7.10
7.06
7.03
5.45
3.72
3.50
2.26
0.00
0.97
0.98
1.00
5.38
1.09
0.98
1.00
3.03
2.97
2.00
F
COOMe
COOMe
HN
NH2
3h
f1 (ppm)


171.18
170.54
164.19
160.94
142.34
135.42
129.49
127.19
125.15
123.44
121.98
119.36
118.66
116.64
114.03
110.98
77.46
77.04
76.62
70.73
53.25
53.19
46.48
F
COOMe
COOMe
HN
NH2
3h
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001208.D
Sample Name: SLL2013-8-25-1-0

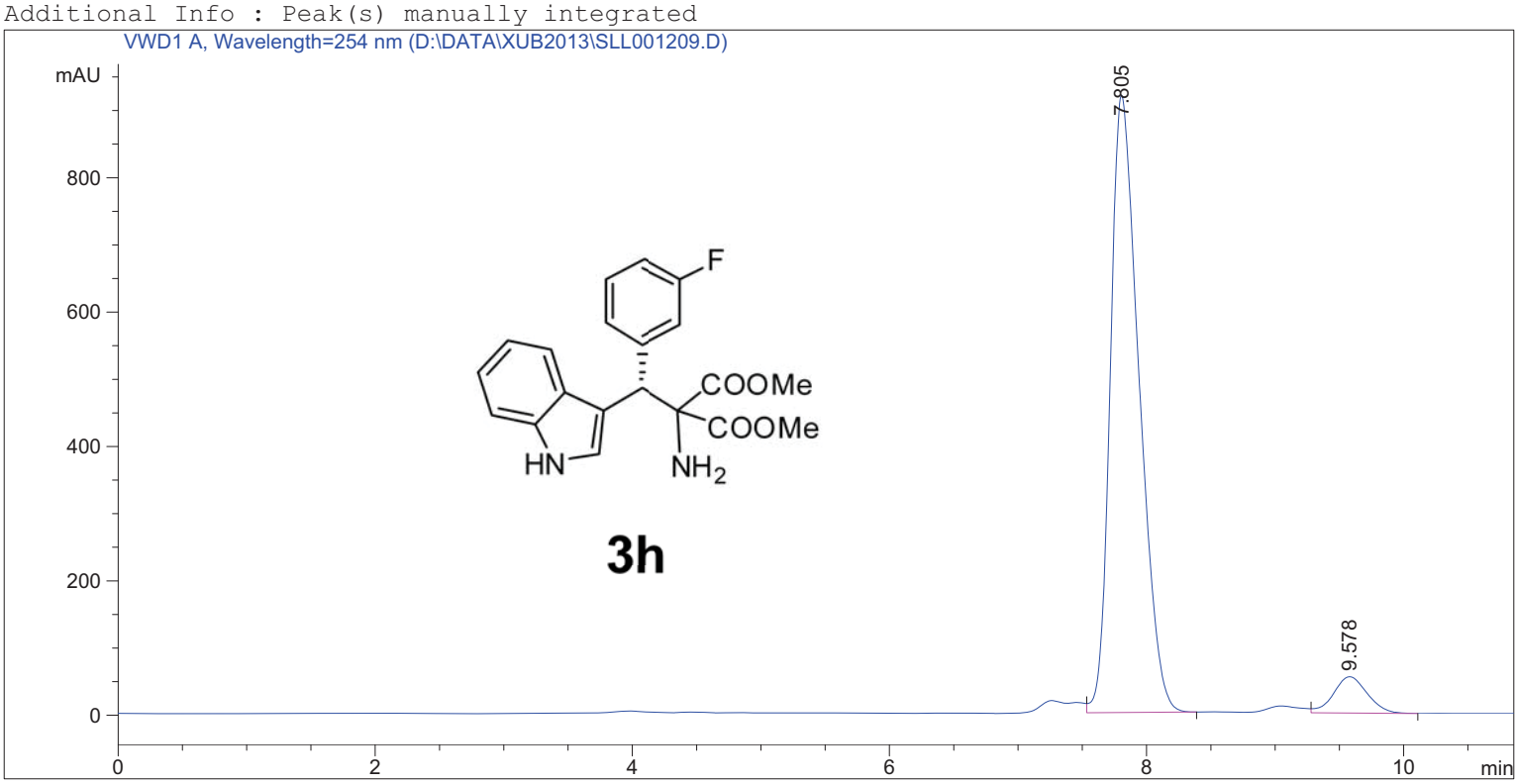
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 9/11/2013 8:01:55 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 9/11/2013 8:01:06 PM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:11:47 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



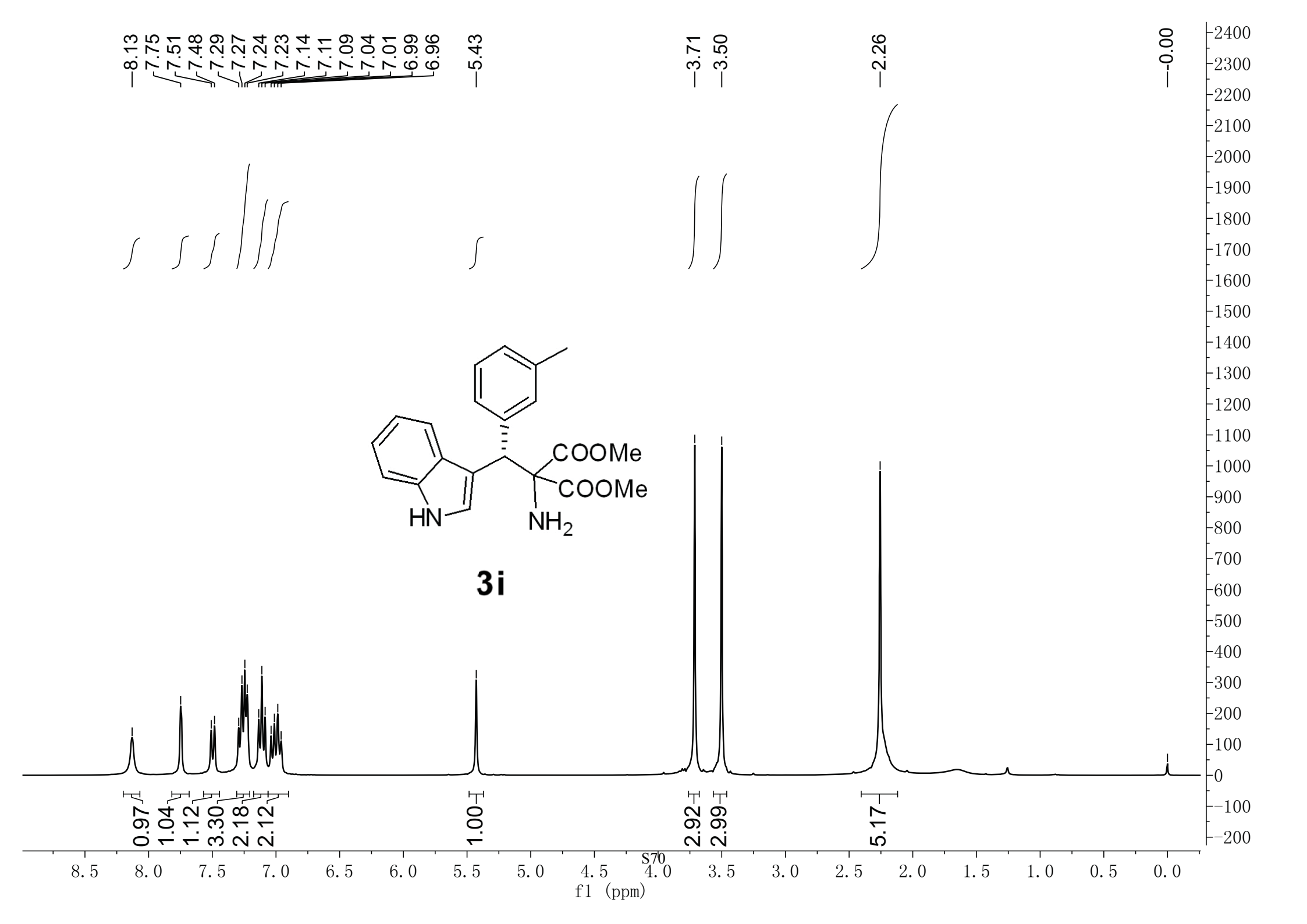

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.811 VV    0.2495 1.49731e4   893.05853  49.9346
   2   9.530 VB    0.2783 1.50123e4   817.39374  50.0654

Totals :                  2.99854e4  1710.45227


==========================================================================
                        *** End of Report ***
```

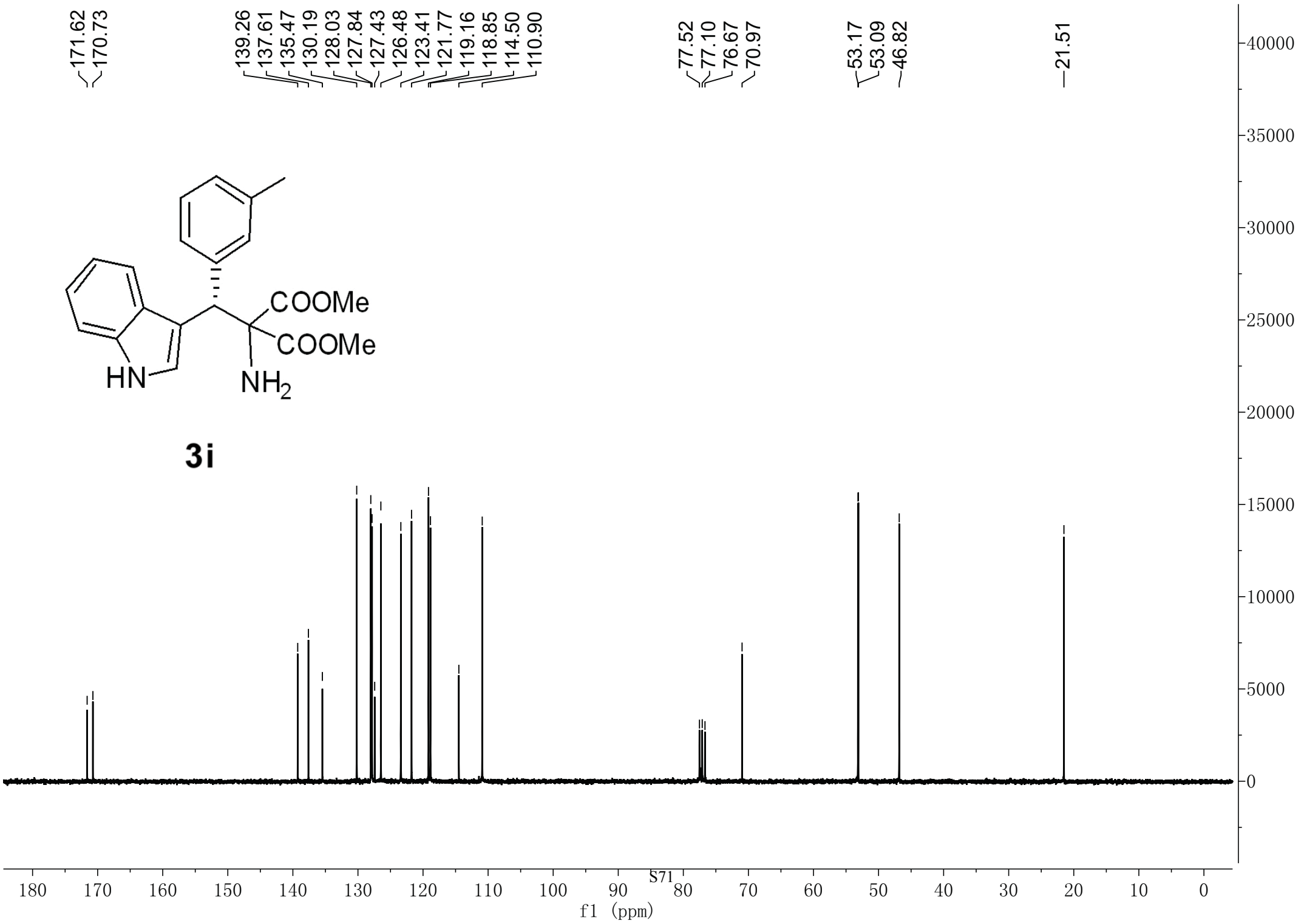
Data File D:\DATA\XUB2013\SLL001209.D
Sample Name: SLL2013-8-27-1

```
================================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 9/11/2013 8:18:24 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 9/11/2013 8:15:41 PM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:11:47 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



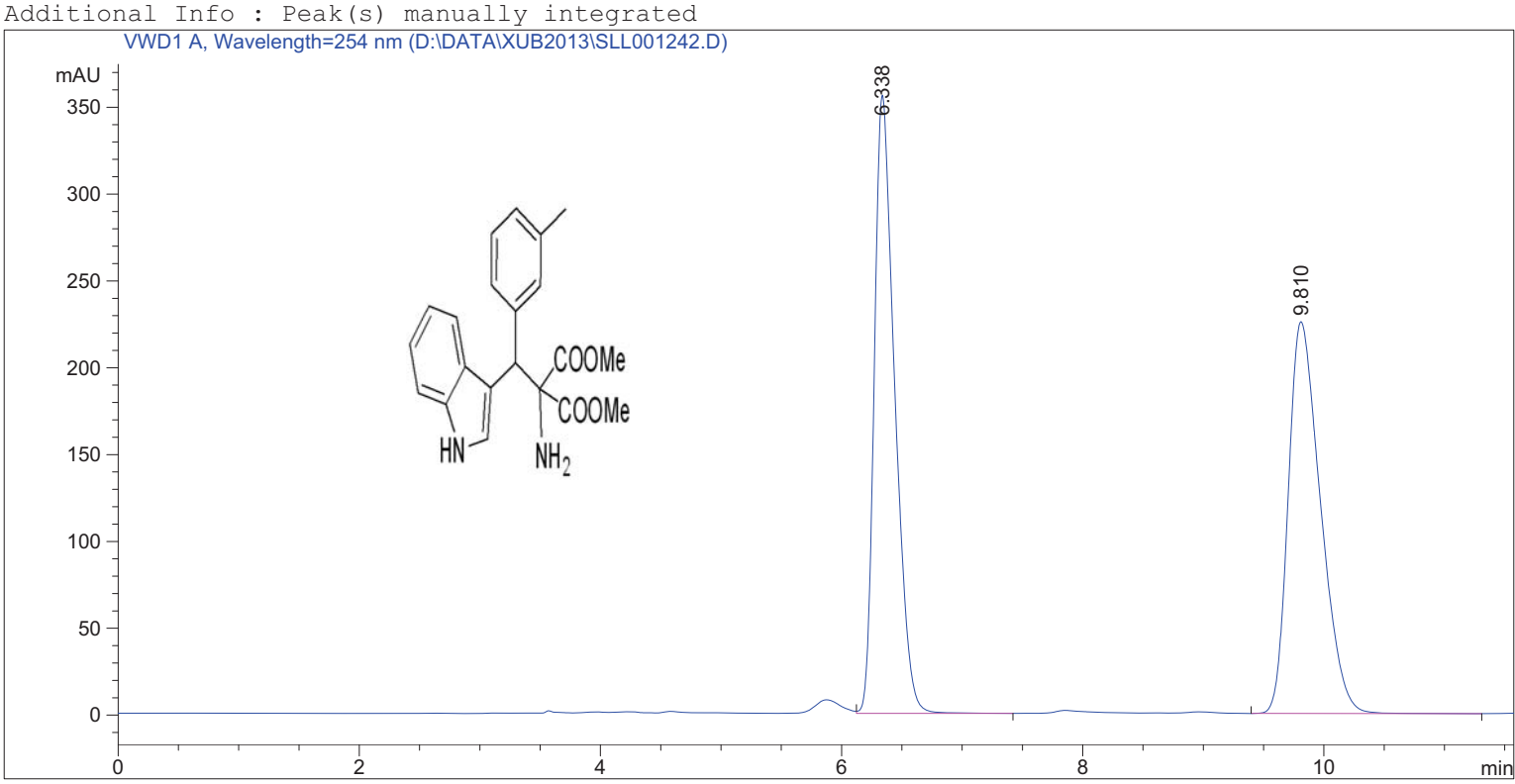

```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.805 VB    0.2354 1.42980e4   918.91626  93.5436
   2   9.578 VB    0.2782  986.85138   54.13845   6.4564

Totals :                  1.52849e4   973.05471

================================================================================
                          *** End of Report ***
```


8.13
7.75
7.51
7.48
7.29
7.27
7.24
7.23
7.14
7.11
7.09
7.04
7.01
6.99
6.96
5.43
3.71
3.50
2.26
0.00
COOMe
COOMe
HN
NH2
3i
0.97
1.04
1.12
3.30
2.18
2.12
1.00
2.92
2.99
5.17
f1 (ppm)


171.62
170.73
139.26
137.61
135.47
130.19
128.03
127.84
127.43
126.48
123.41
121.77
119.16
118.85
114.50
110.90
77.52
77.10
76.67
70.97
53.17
53.09
46.82
21.51
COOMe
COOMe
HN
NH2
3i
40000
35000
30000
25000
20000
15000
10000
5000
0
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001242.D
Sample Name: SLL2013-9-16-1-0

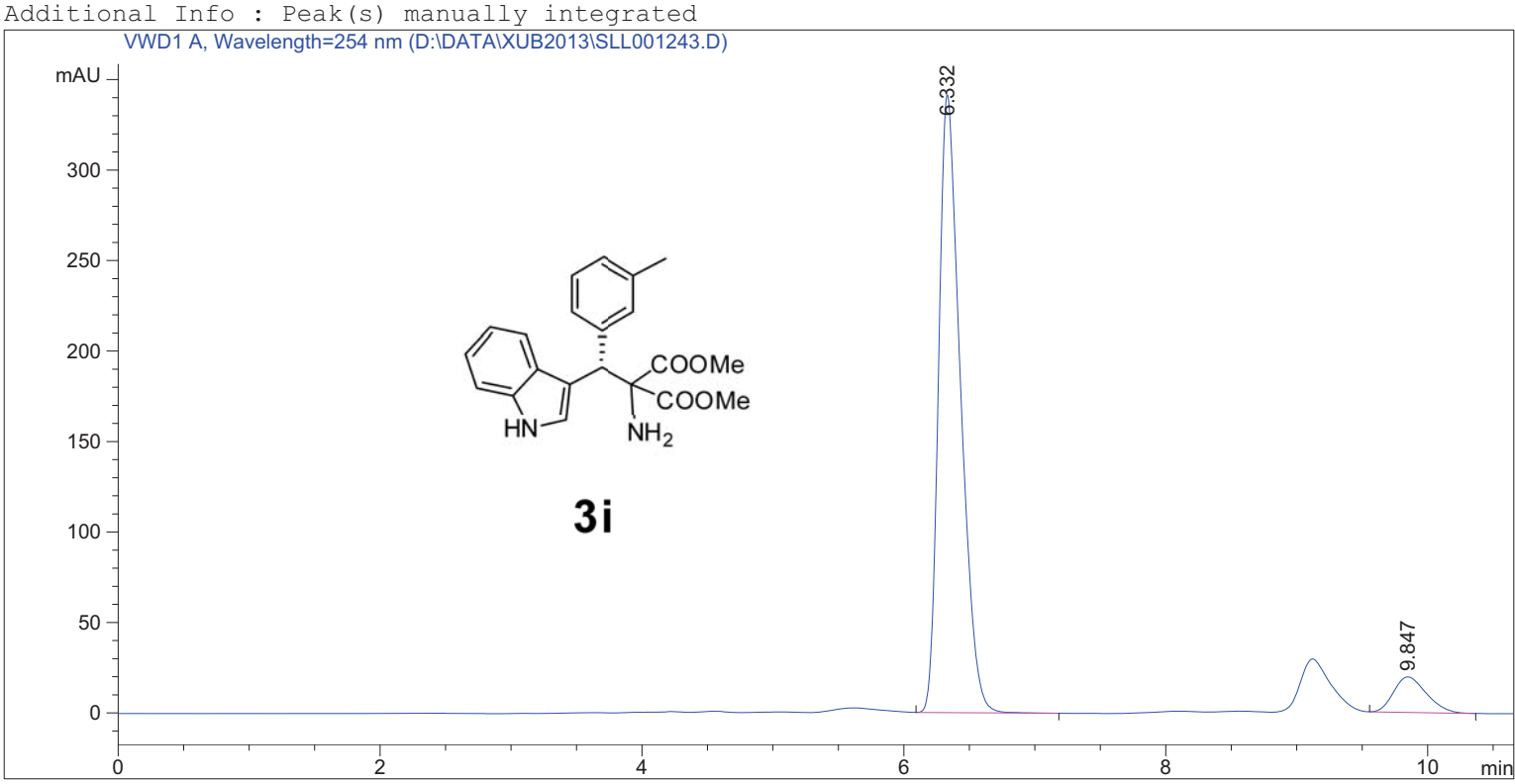
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 9/22/2013 4:55:54 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 9/22/2013 4:55:36 PM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:13:55 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



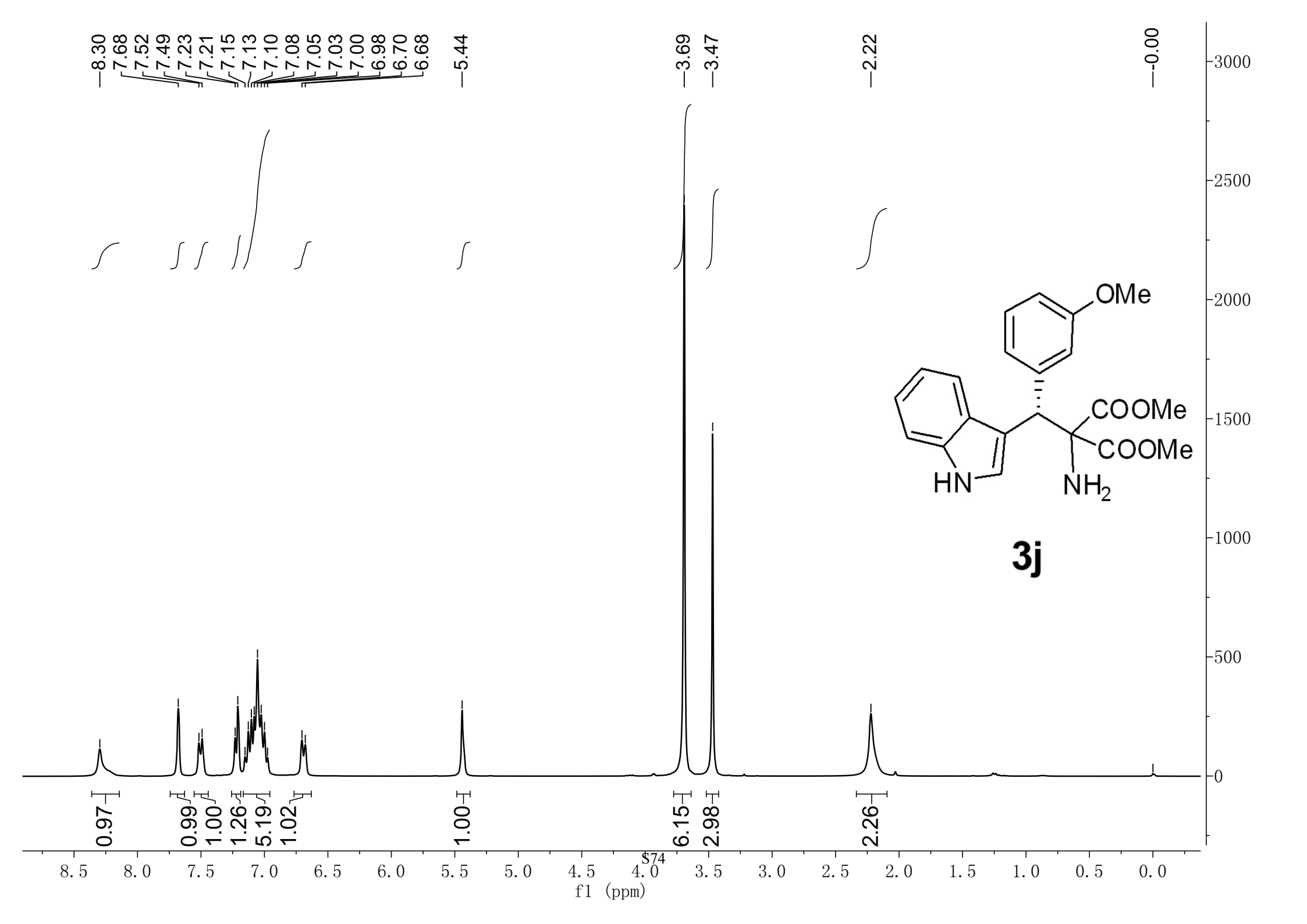

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.338 VB    0.1750 4185.50293  355.93018  50.0446
   2   9.810 BB    0.2816 4178.04834  225.59811  49.9554

Totals :                  8363.55127  581.52829

==========================================================================
                        *** End of Report ***
```

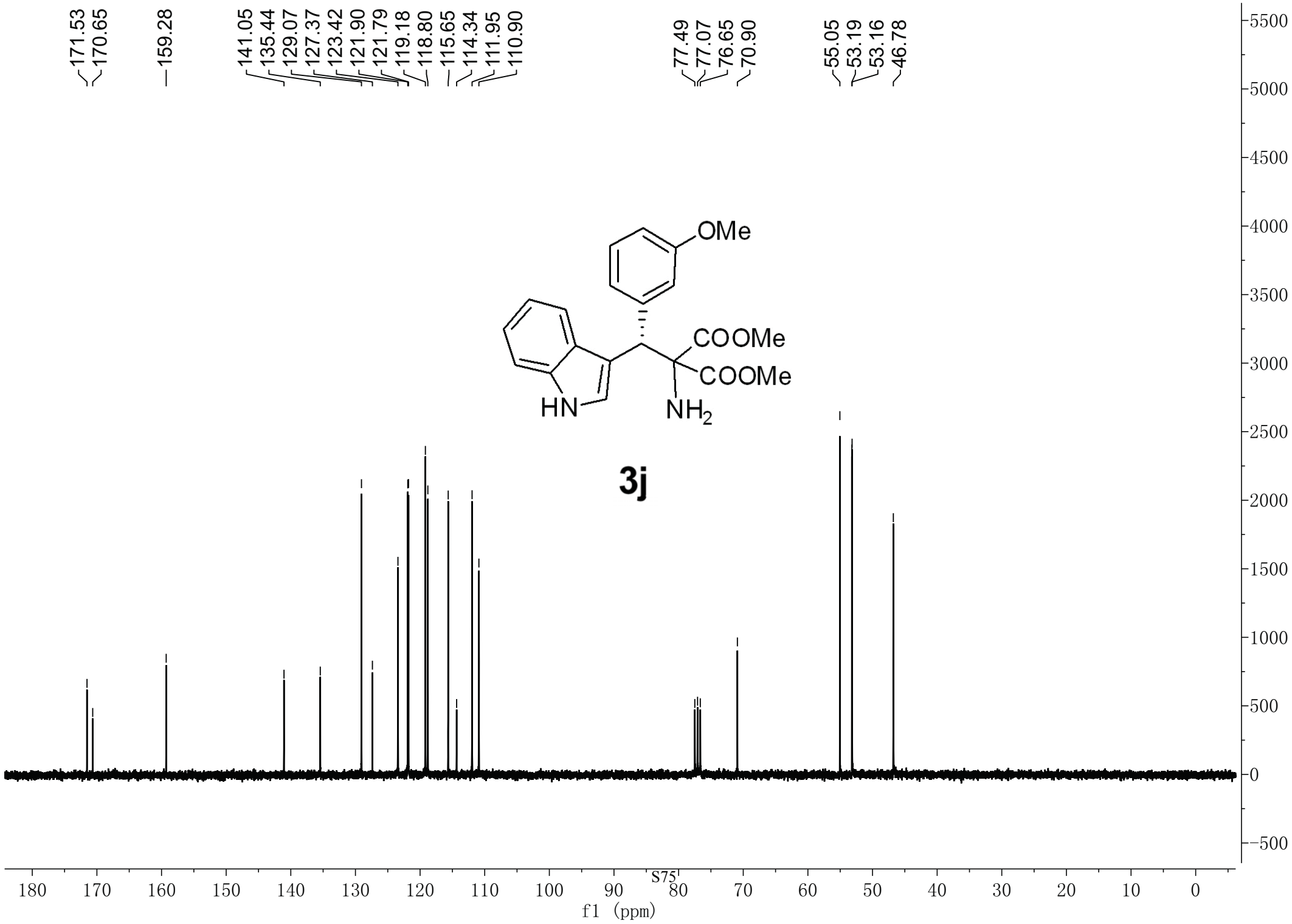
Data File D:\DATA\XUB2013\SLL001243.D
Sample Name: SLL2013-9-16-1

```
==========================================================================
Acq. Operator    : LNF
Acq. Instrument  : Instrument 1                    Location : Vial 1
Injection Date   : 9/22/2013 5:08:04 PM
                                                   Inj Volume : No inj
Acq. Method      : D:\METHOD\AD_30_1.M
Last changed     : 9/22/2013 5:07:34 PM by LNF
                   (modified after loading)
Analysis Method  : D:\METHOD\AD_30_1.M
Last changed     : 9/29/2013 11:15:04 AM by LNF
                   (modified after loading)
Additional Info  : Peak(s) manually integrated
```



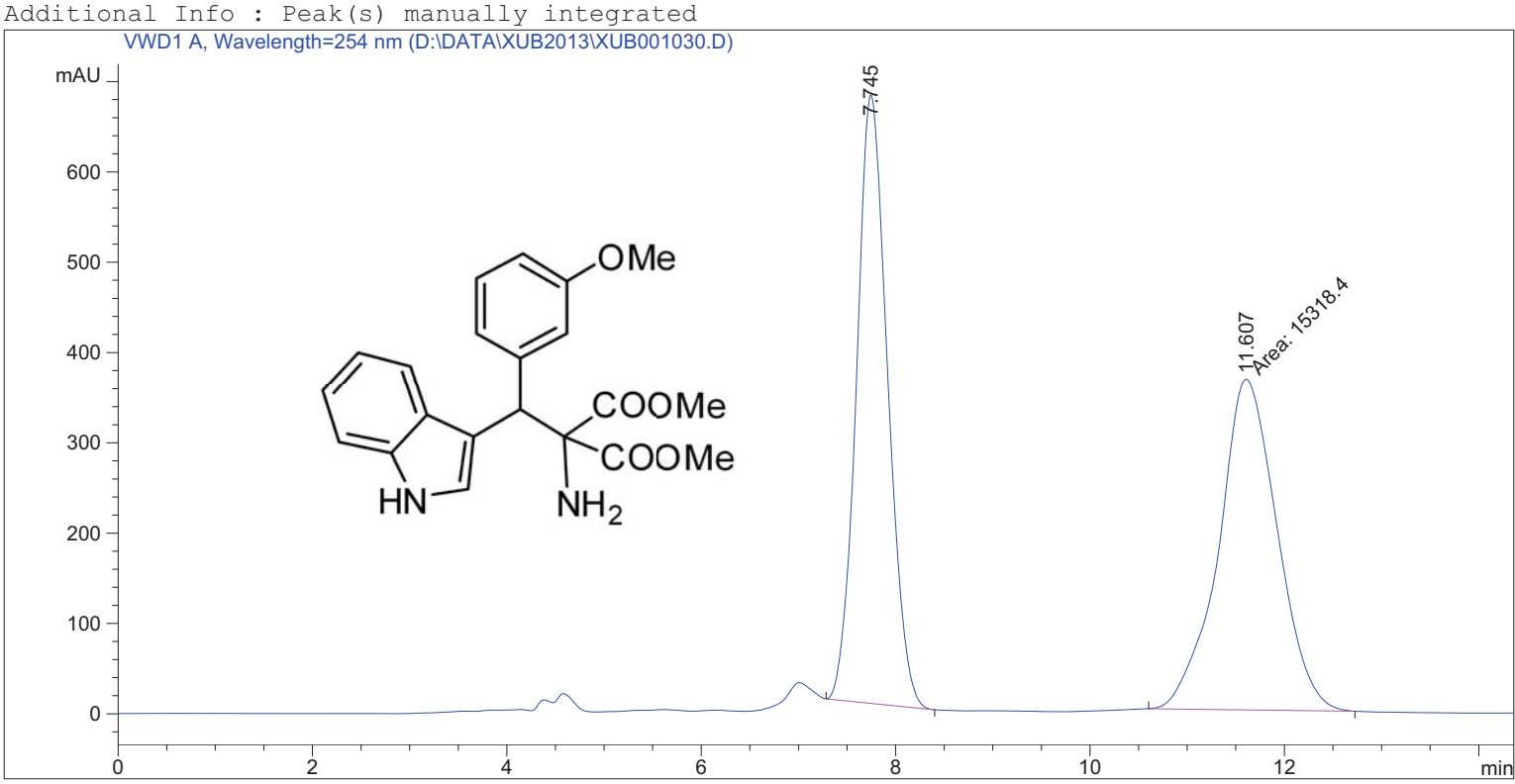

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.332 BB    0.1734 3965.93164  341.31339  92.0630
   2   9.847 BB    0.2667  341.91437   19.68316   7.9370

Totals :                  4307.84601  360.99654


==========================================================================
                          *** End of Report ***
```


8.30
7.68
7.52
7.49
7.23
7.21
7.15
7.13
7.10
7.08
7.05
7.03
7.00
6.98
6.70
6.68
5.44
3.69
3.47
2.22
0.00
OMe
COOMe
COOMe
HN
NH2
3j
0.97
0.99
1.00
1.26
5.19
1.02
1.00
6.15
2.98
2.26
f1 (ppm)


171.53
170.65
159.28
141.05
135.44
129.07
127.37
123.42
121.90
121.79
119.18
118.80
115.65
114.34
111.95
110.90
77.49
77.07
76.65
70.90
55.05
53.19
53.16
46.78
OMe
COOMe
COOMe
HN
NH2
3j
f1 (ppm)

Data File D:\DATA\XUB2013\XUB001030.D
Sample Name: SLL2013-6-26-1-0

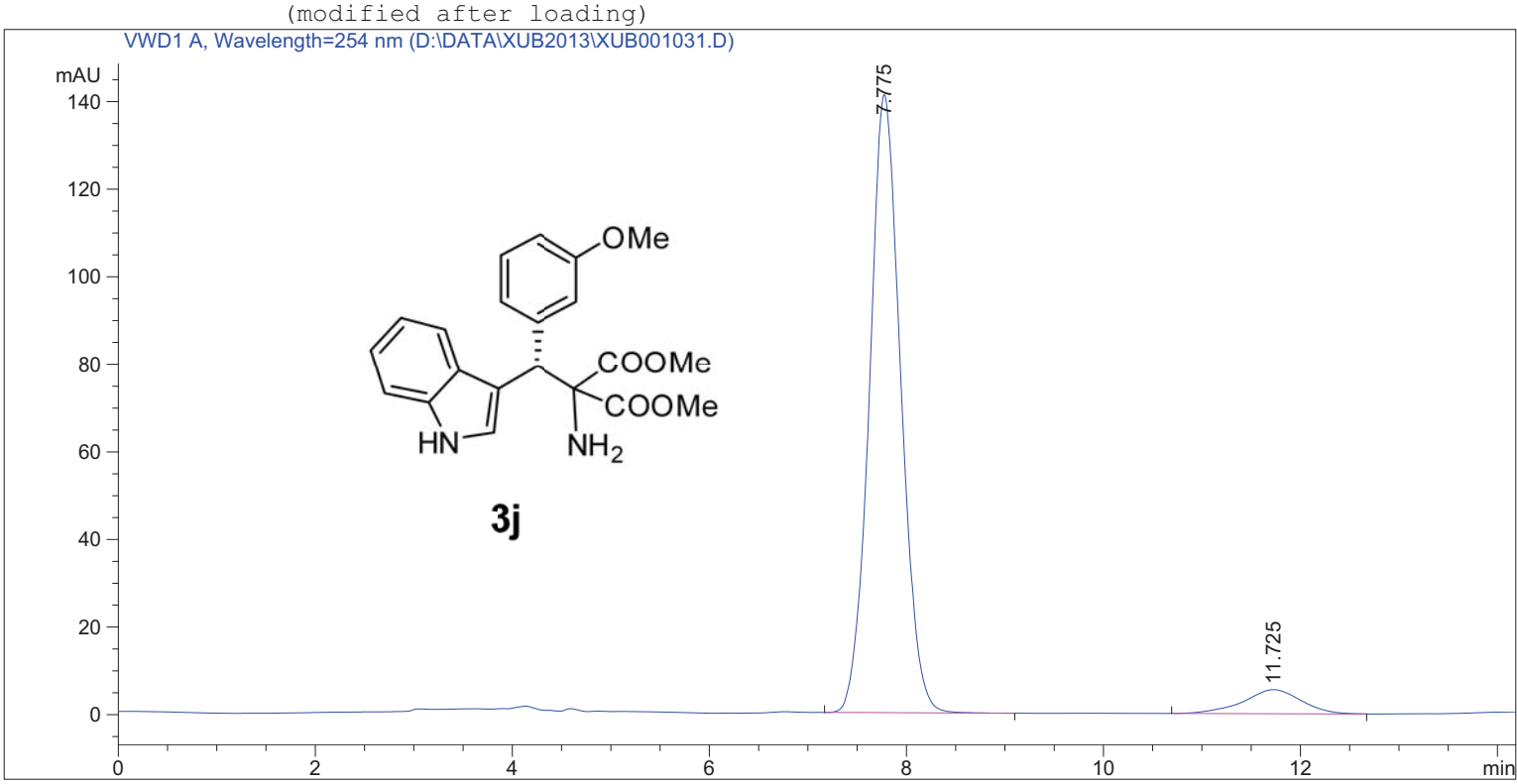
```
================================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/30/2013 10:14:35 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 6/30/2013 10:14:19 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:18:38 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



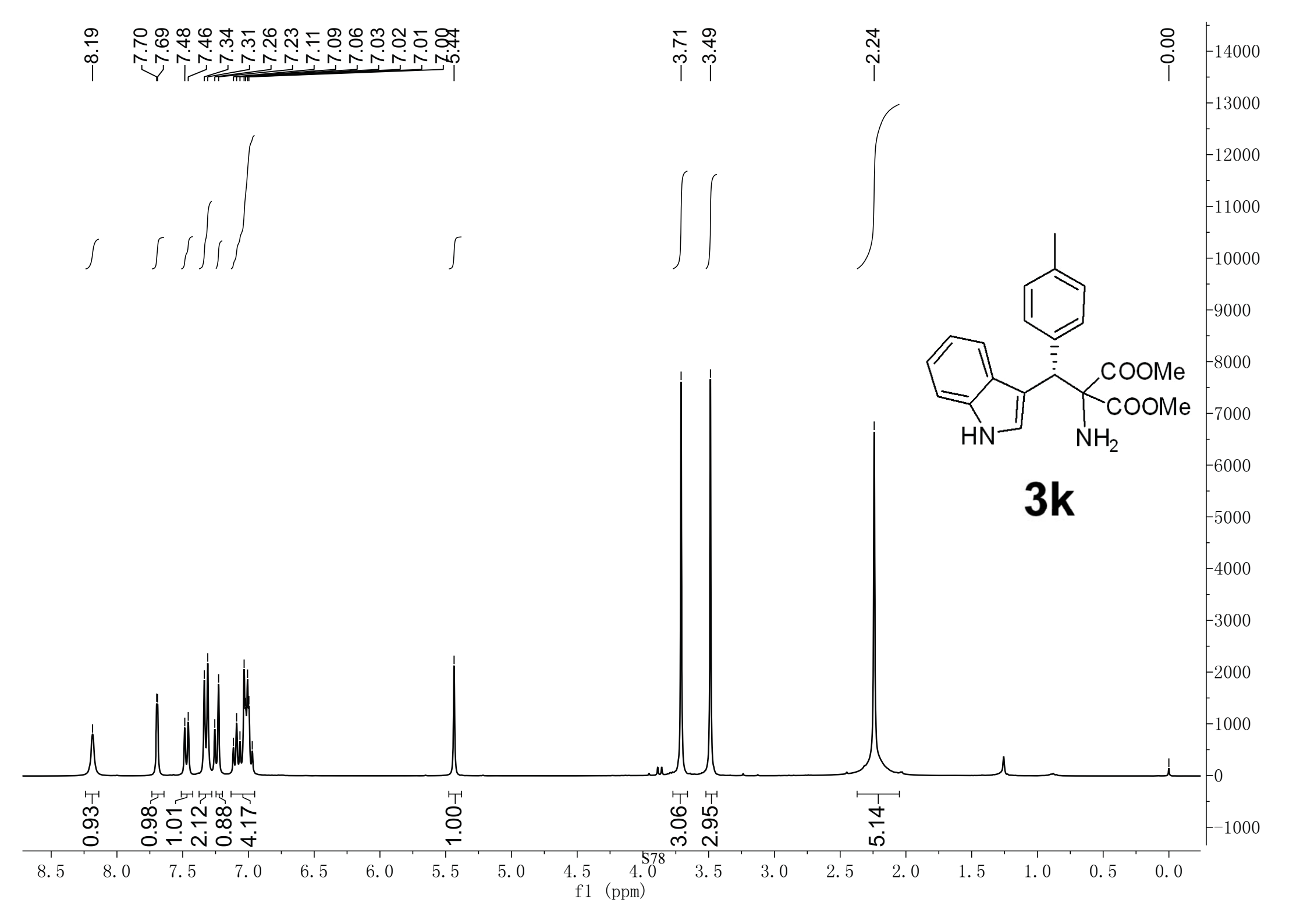

```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.745 BB    0.3378 1.49631e4   673.45099  49.4134
   2  11.607 MM    0.6978 1.53184e4   365.86505  50.5866

Totals :                  3.02815e4  1039.31604

================================================================================
                          *** End of Report ***
```

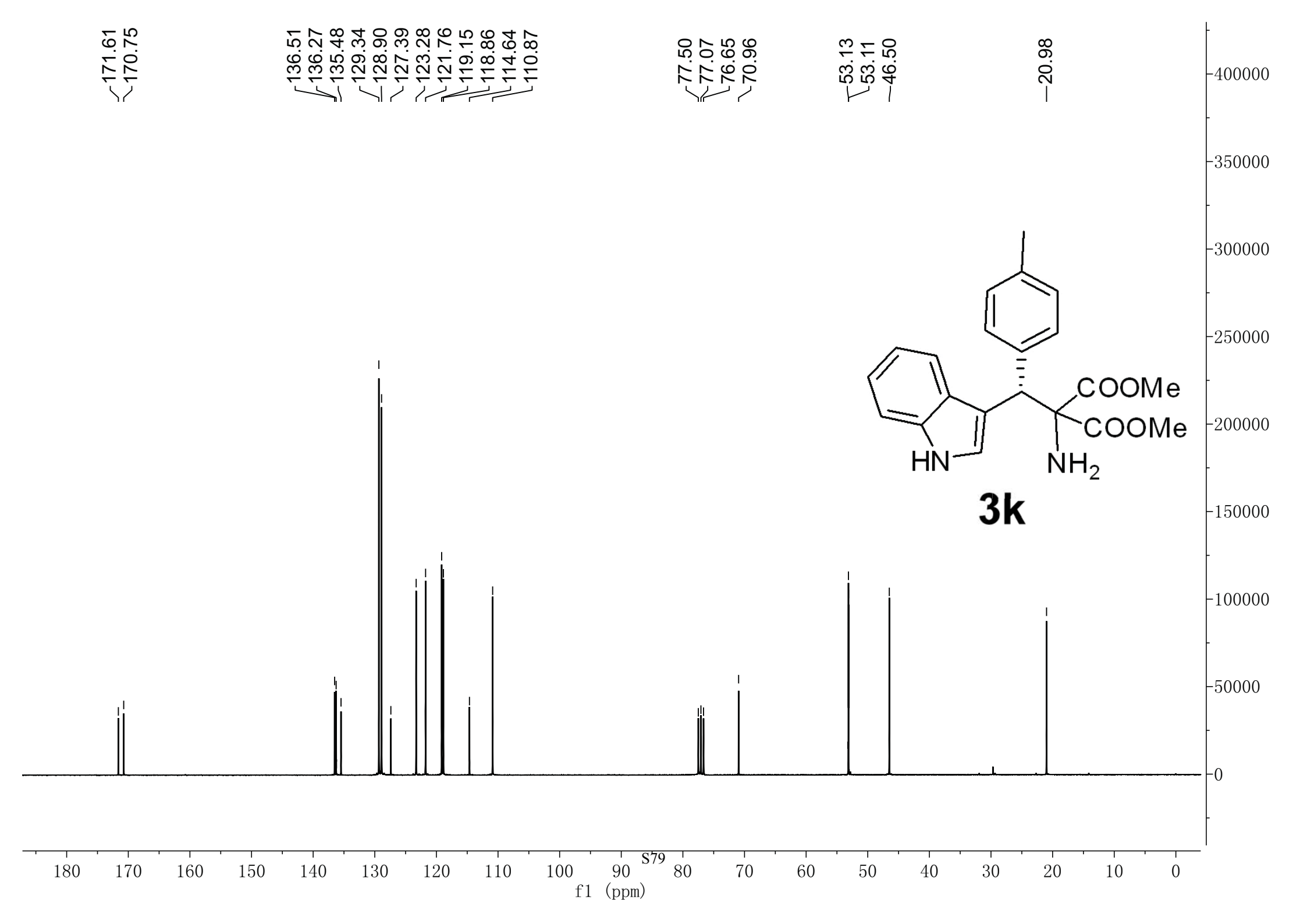
Data File D:\DATA\XUB2013\XUB001031.D
Sample Name: SLL2013-6-26-1

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/30/2013 10:29:13 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 6/30/2013 10:29:01 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:15:49 AM by LNF
                  (modified after loading)
```



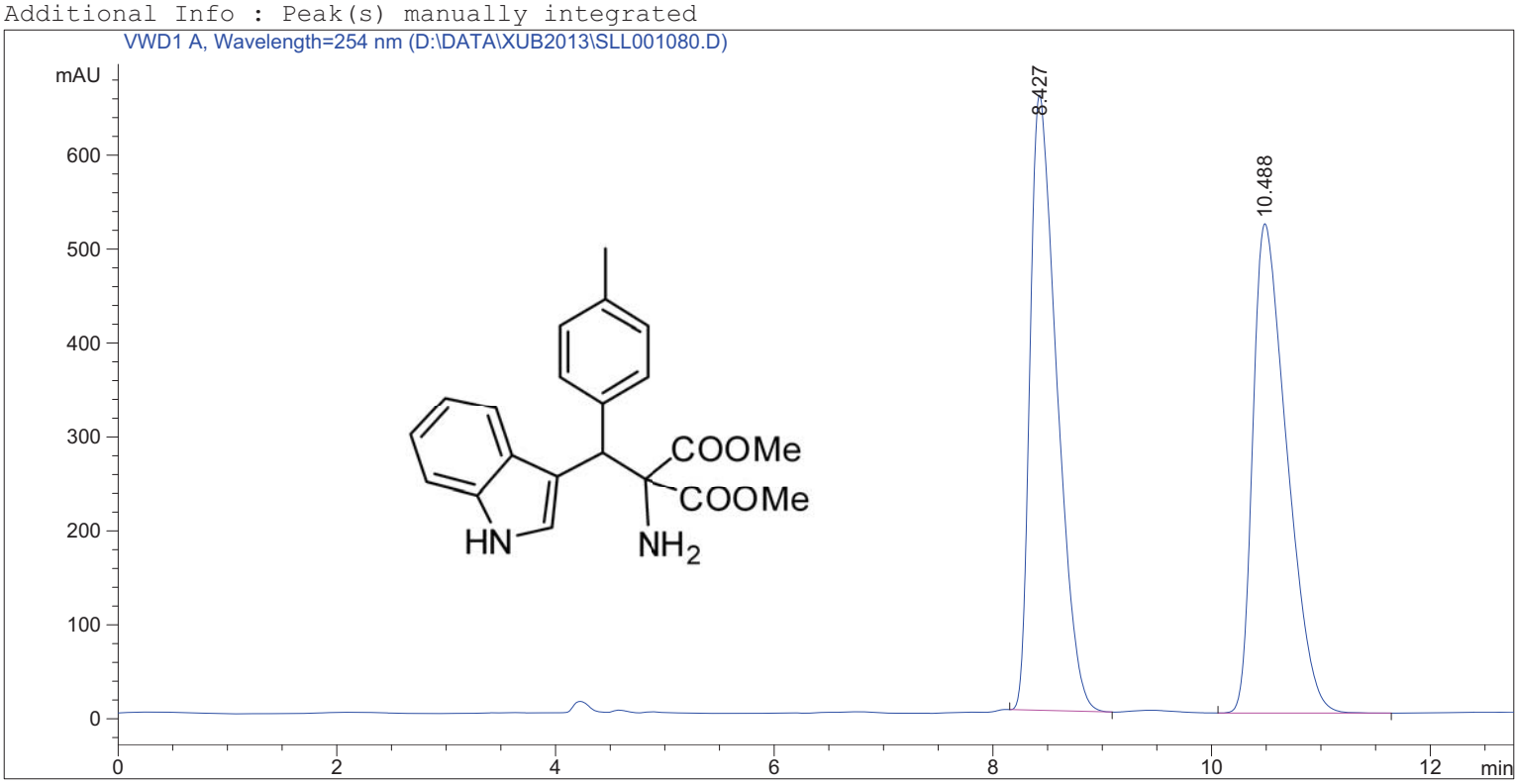

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.775 BB    0.3345 3112.39038  141.13080  93.2533
   2  11.725 BB    0.5958  225.17696    5.49788   6.7467

Totals :                  3337.56734  146.62868

==========================================================================
                          *** End of Report ***
```


8.19
7.70
7.69
7.48
7.46
7.34
7.31
7.26
7.23
7.11
7.09
7.06
7.03
7.02
7.01
7.00
5.44
3.71
3.49
2.24
0.00
0.93
0.98
1.01
2.12
0.88
4.17
1.00
3.06
2.95
5.14
COOMe
COOMe
HN
NH2
3k
f1 (ppm)


171.61
170.75
136.51
136.27
135.48
129.34
128.90
127.39
123.28
121.76
119.15
118.86
114.64
110.87
77.50
77.07
76.65
70.96
53.13
53.11
46.50
20.98
COOMe
COOMe
HN
NH2
3k
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001080.D
Sample Name: SLL2013-7-17-2-2

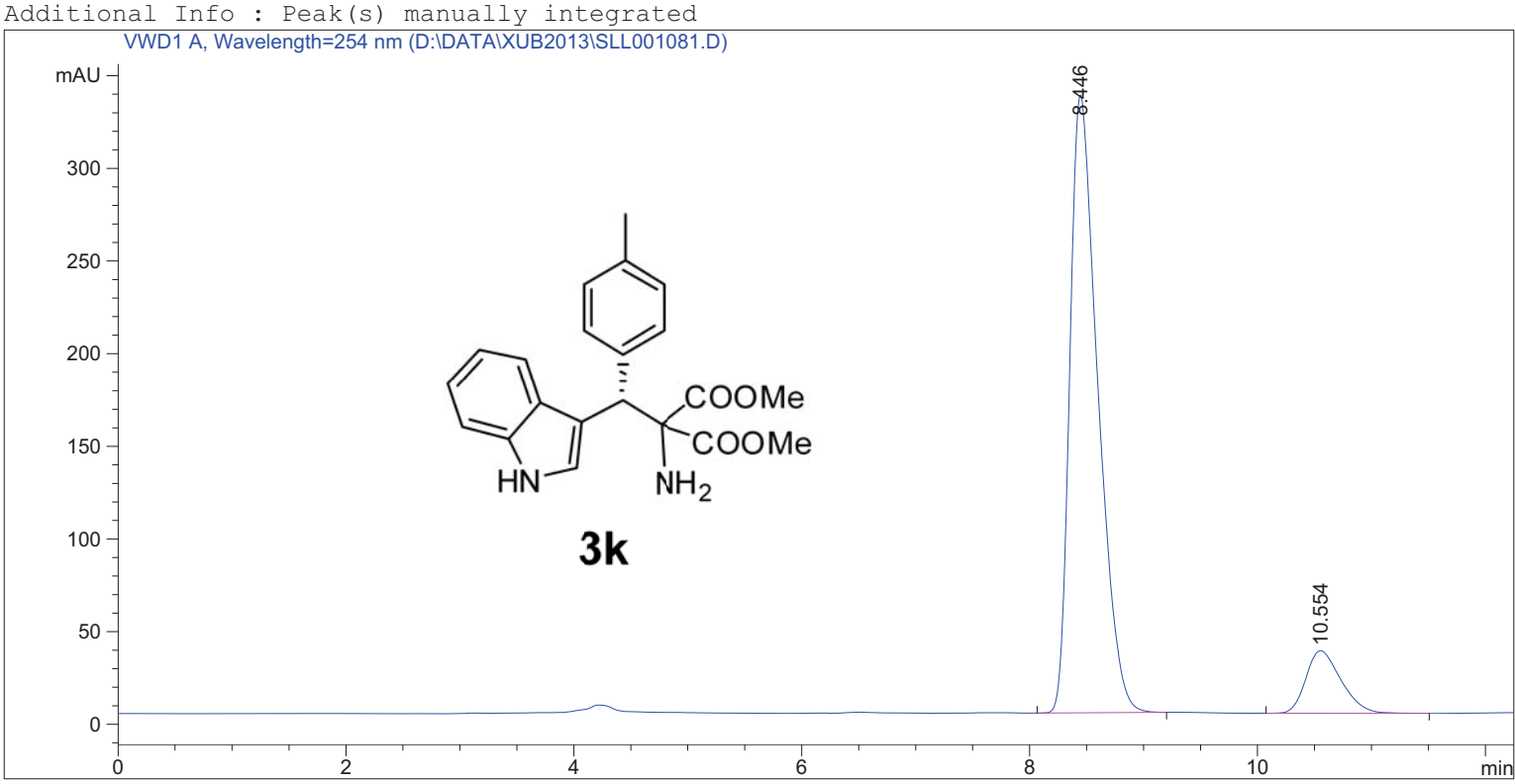
```
================================================================================
Acq. Operator    : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/18/2013 10:51:09 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/18/2013 10:49:43 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:22:42 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



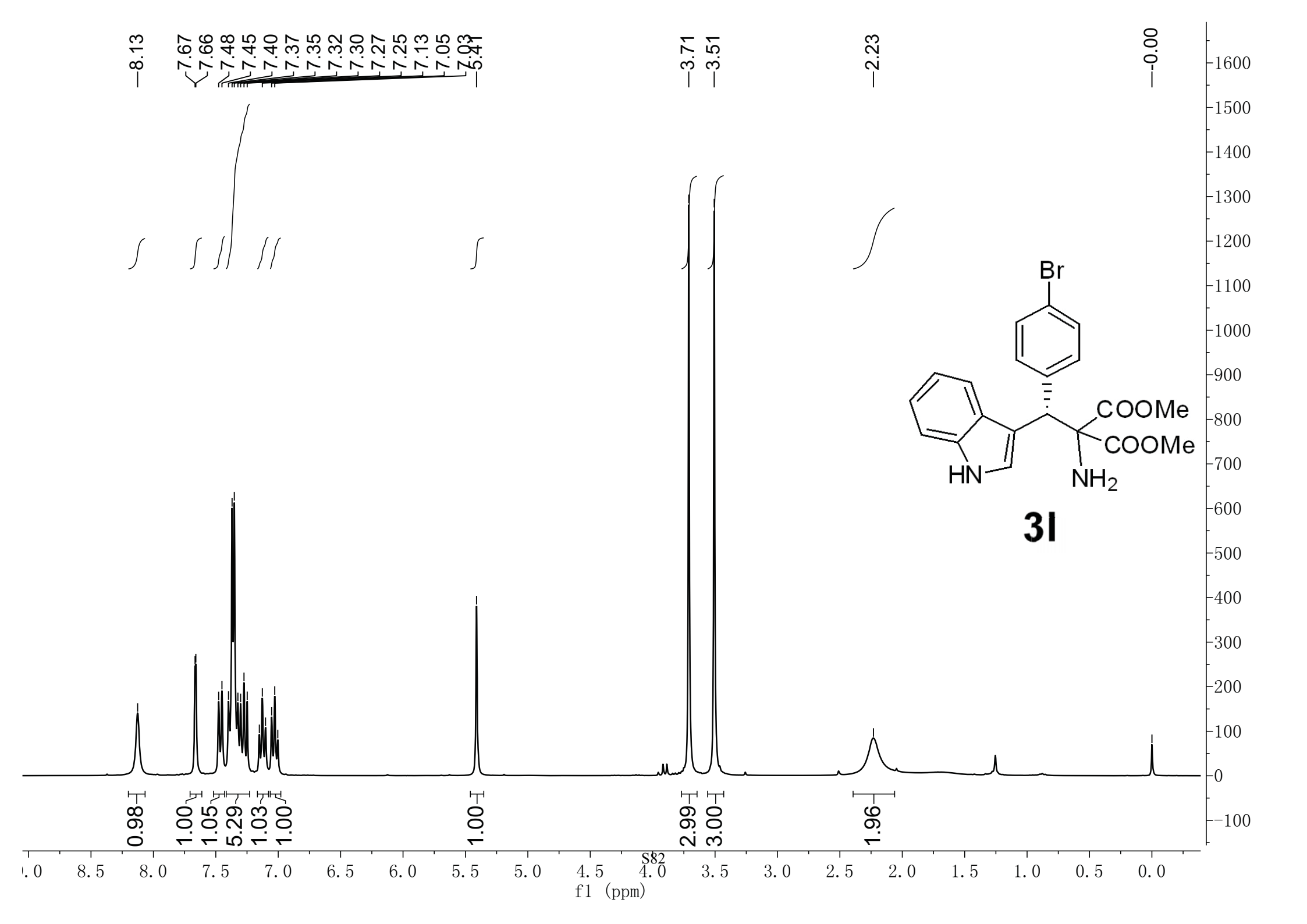

```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.427 BB    0.2574 1.11781e4   654.95245  49.7952
   2  10.488 BB    0.3267 1.12700e4   520.91382  50.2048

Totals :                  2.24481e4  1175.86627


================================================================================
                          *** End of Report ***
```

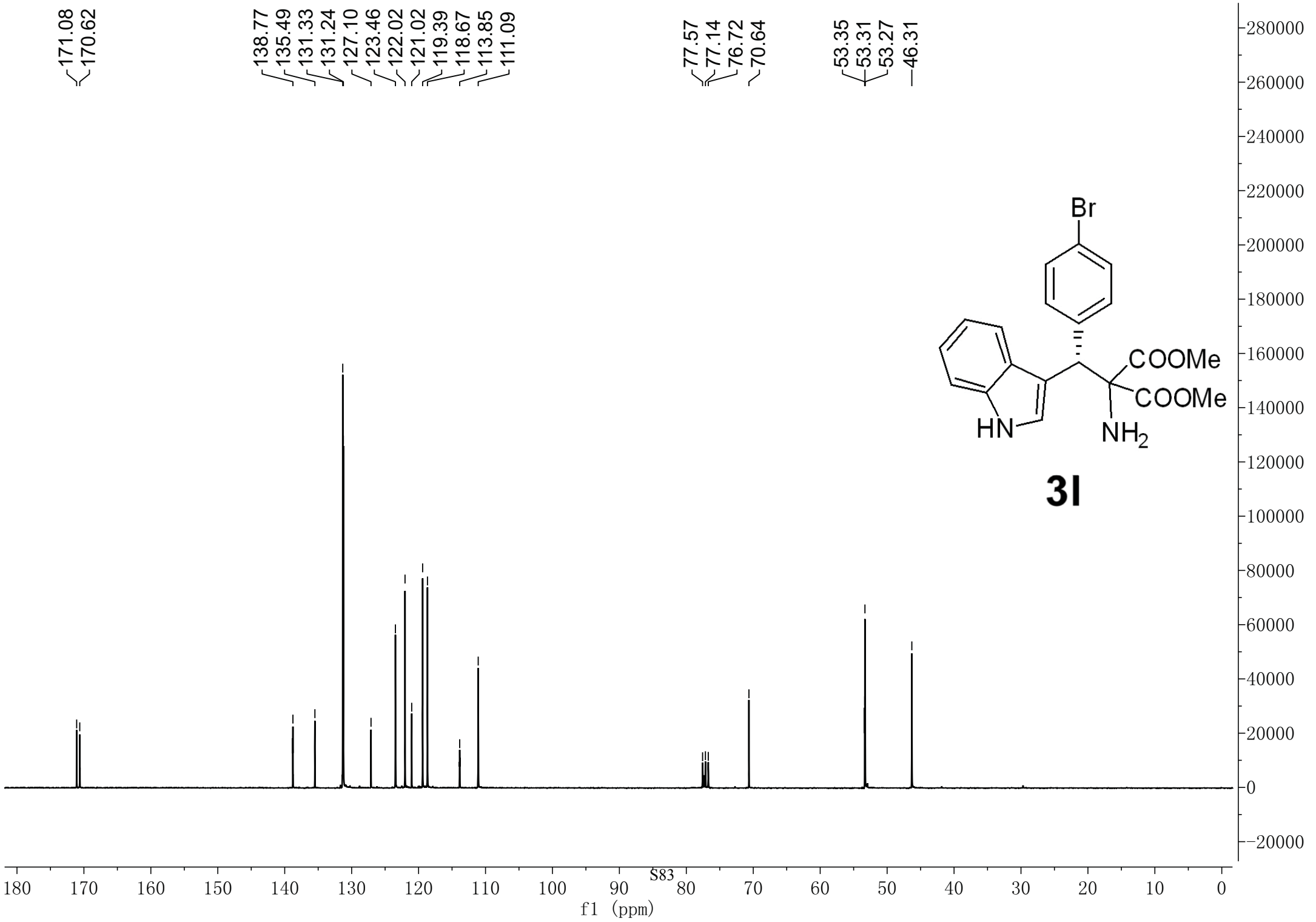
Data File D:\DATA\XUB2013\SLL001081.D
Sample Name: SLL2013-7-17-2

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/18/2013 11:05:24 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/18/2013 11:04:00 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:22:42 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



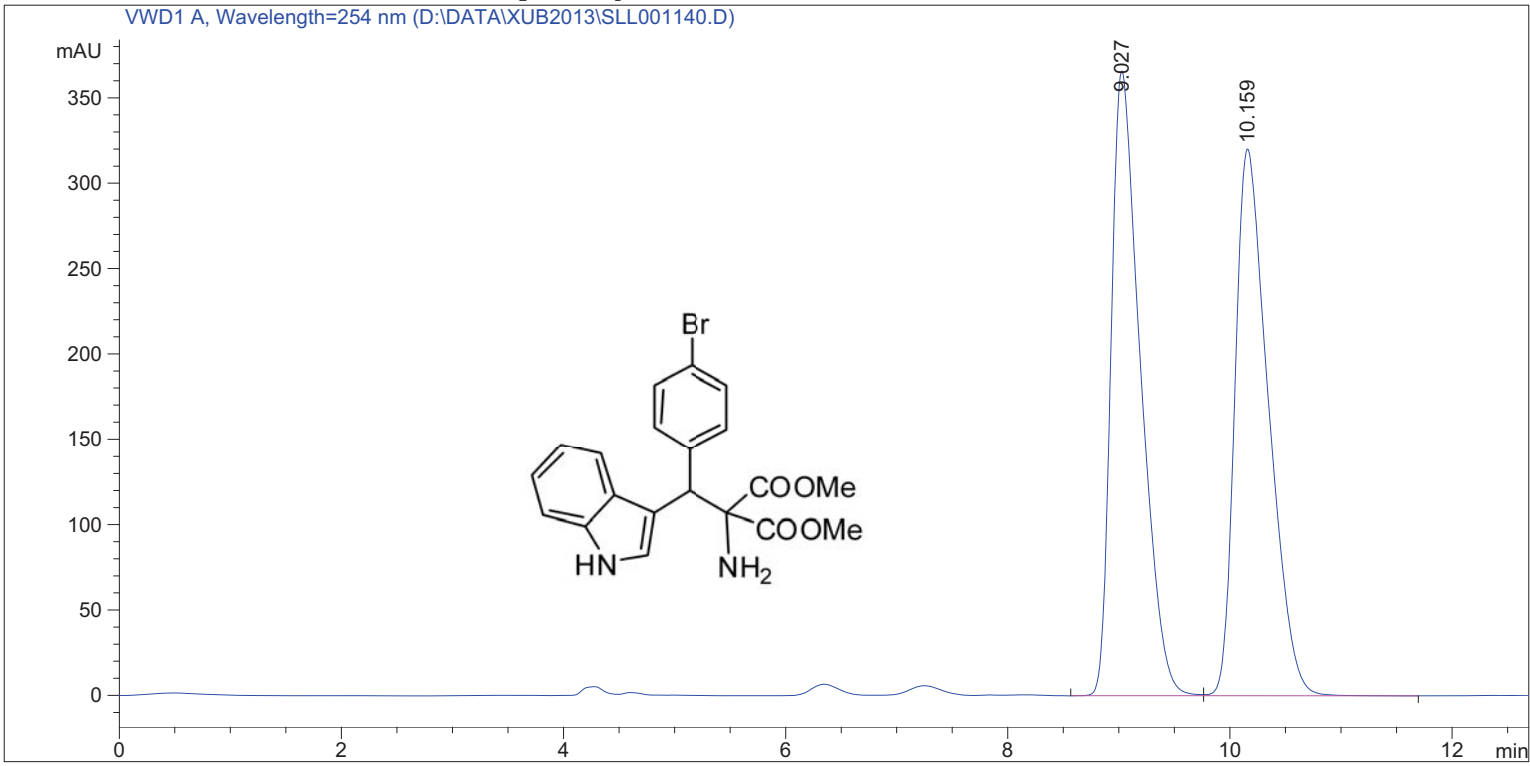

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.446 BB    0.2556 5679.96338  333.28549  88.7369
   2  10.554 BB    0.3260  720.93781   33.80577  11.2631

Totals :                  6400.90118  367.09126


==========================================================================
                          *** End of Report ***
```


8.13
7.67
7.66
7.48
7.45
7.40
7.37
7.35
7.32
7.30
7.27
7.25
7.13
7.05
7.03
5.41
3.71
3.51
2.23
0.00
0.98
1.00
1.05
5.29
1.03
1.00
1.00
2.99
3.00
1.96
Br
COOMe
COOMe
HN
NH2
3l
f1 (ppm)


171.08
170.62
138.77
135.49
131.33
131.24
127.10
123.46
122.02
121.02
119.39
118.67
113.85
111.09
77.57
77.14
76.72
70.64
53.35
53.31
53.27
46.31
Br
COOMe
COOMe
HN
NH2
3l
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001140.D
Sample Name: SLL2013-7-2-1-0

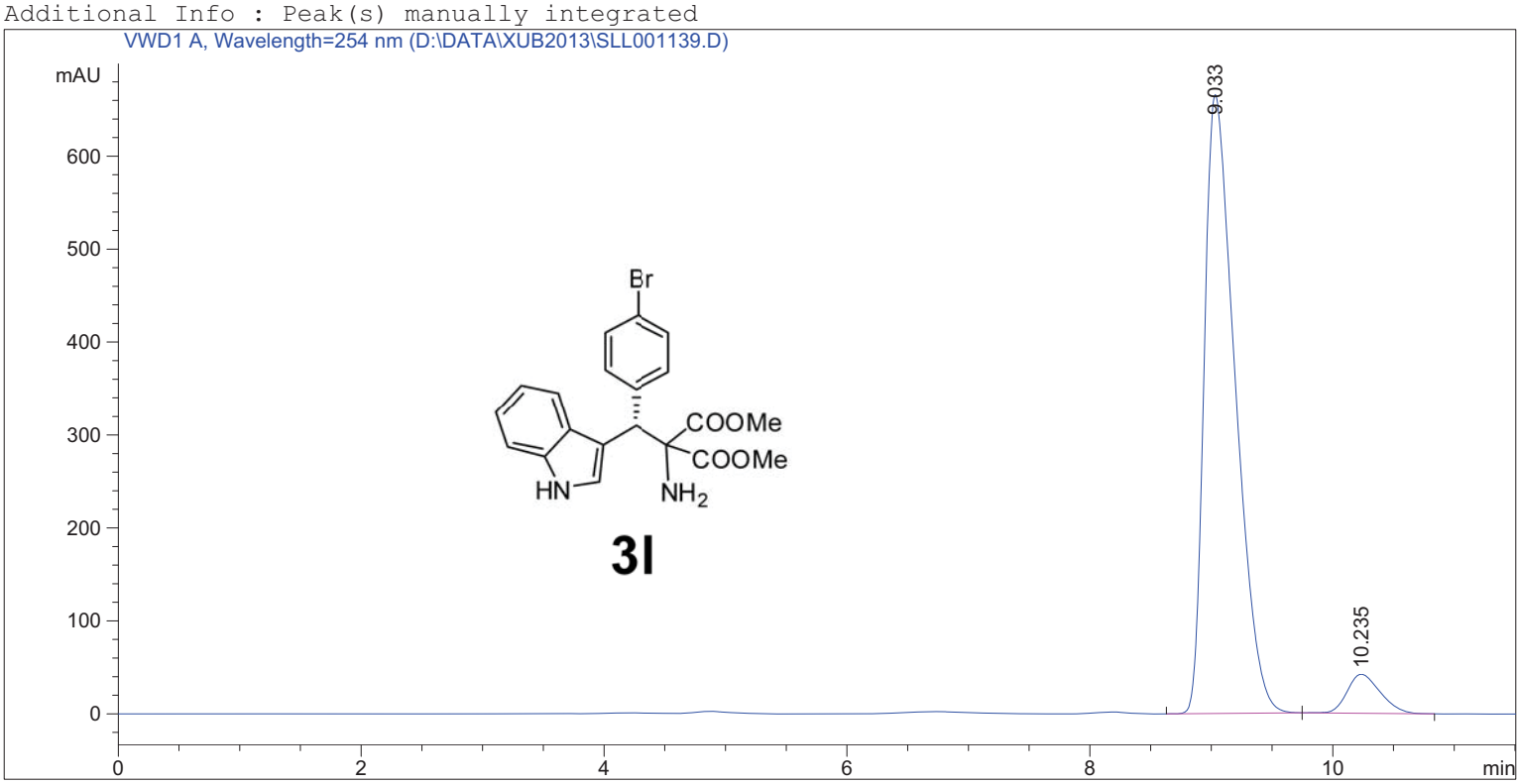
```
==========================================================================
Acq. Operator    : xub
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 8/1/2013 11:51:33 AM
                                                  Inj Volume : No inj
Acq. Method      : D:\METHOD\AD_30_1.M
Last changed     : 8/1/2013 11:51:14 AM by xub
Analysis Method : D:\METHOD\AD_30_1.M
Last changed     : 8/8/2013 3:22:33 PM by xub
Additional Info : Peak(s) manually integrated
```



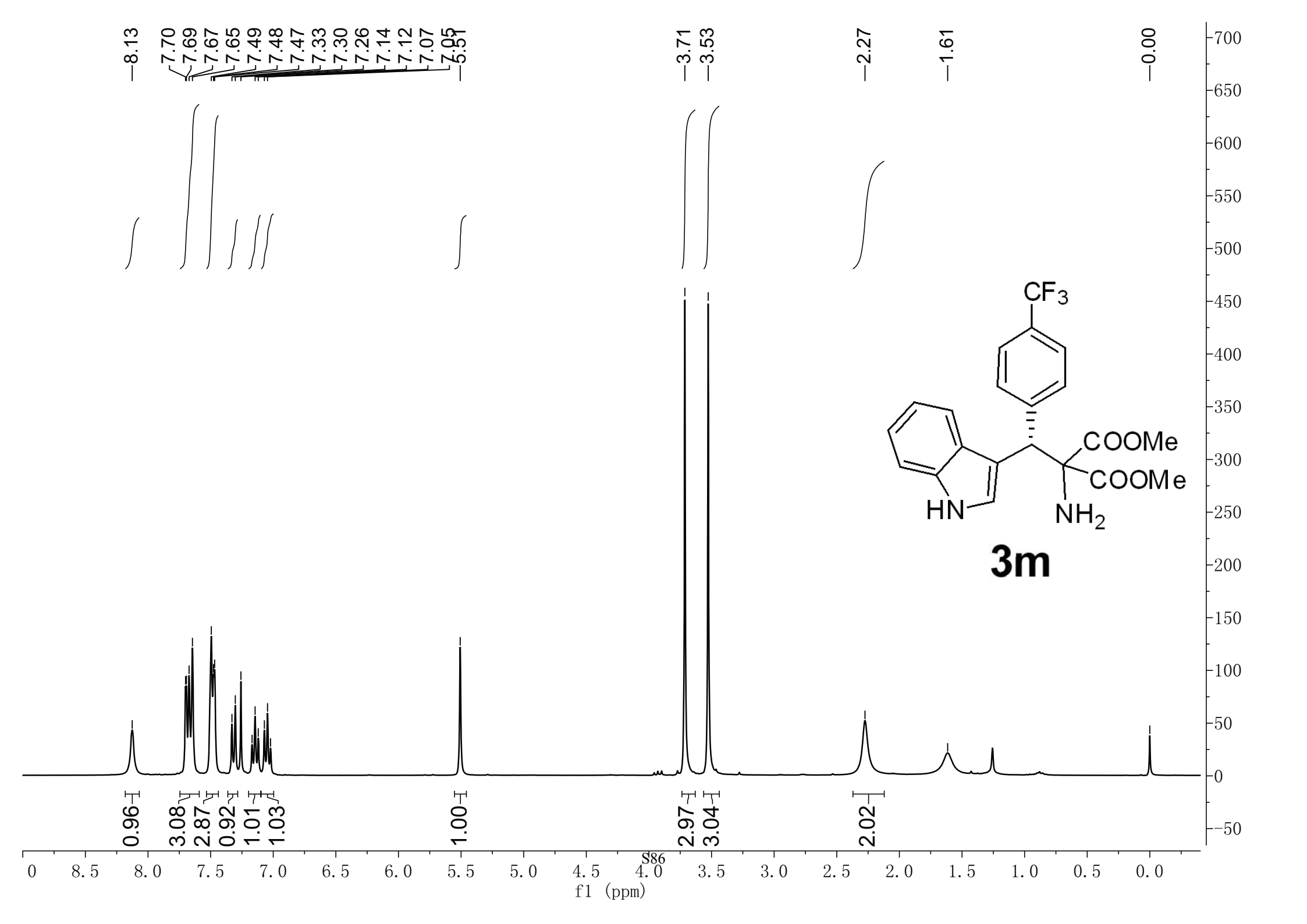

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.027 BV    0.2709 6715.02148  365.94077  50.0516
   2  10.159 VB    0.3125 6701.18262  320.34384  49.9484

Totals :                  1.34162e4   686.28461

==========================================================================
                          *** End of Report ***
```

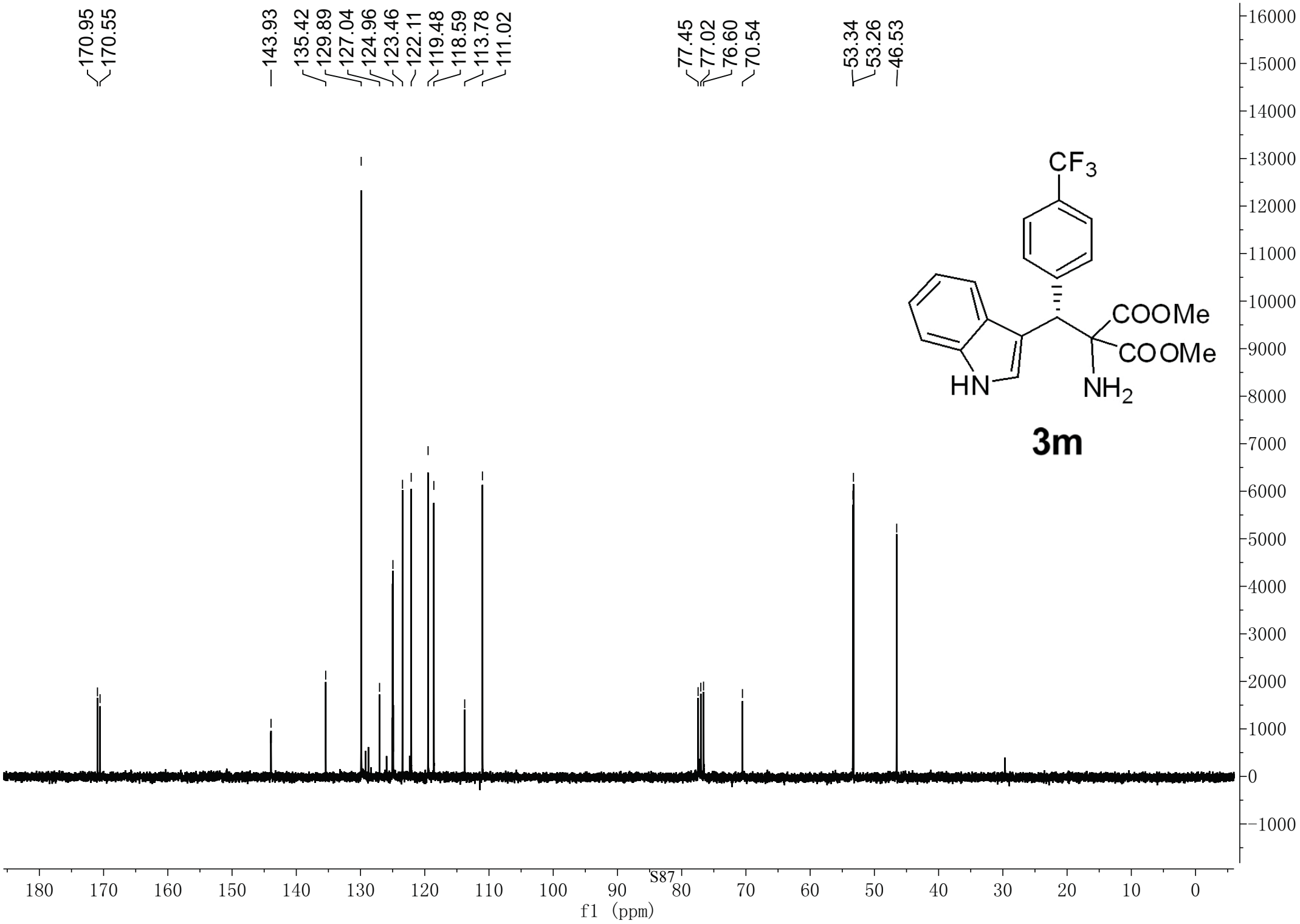
Data File D:\DATA\XUB2013\SLL001139.D
Sample Name: SLL2013-7-30-1

```
==========================================================================
Acq. Operator    : xub
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 8/1/2013 11:39:39 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 8/1/2013 11:37:32 AM by xub
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:35:21 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



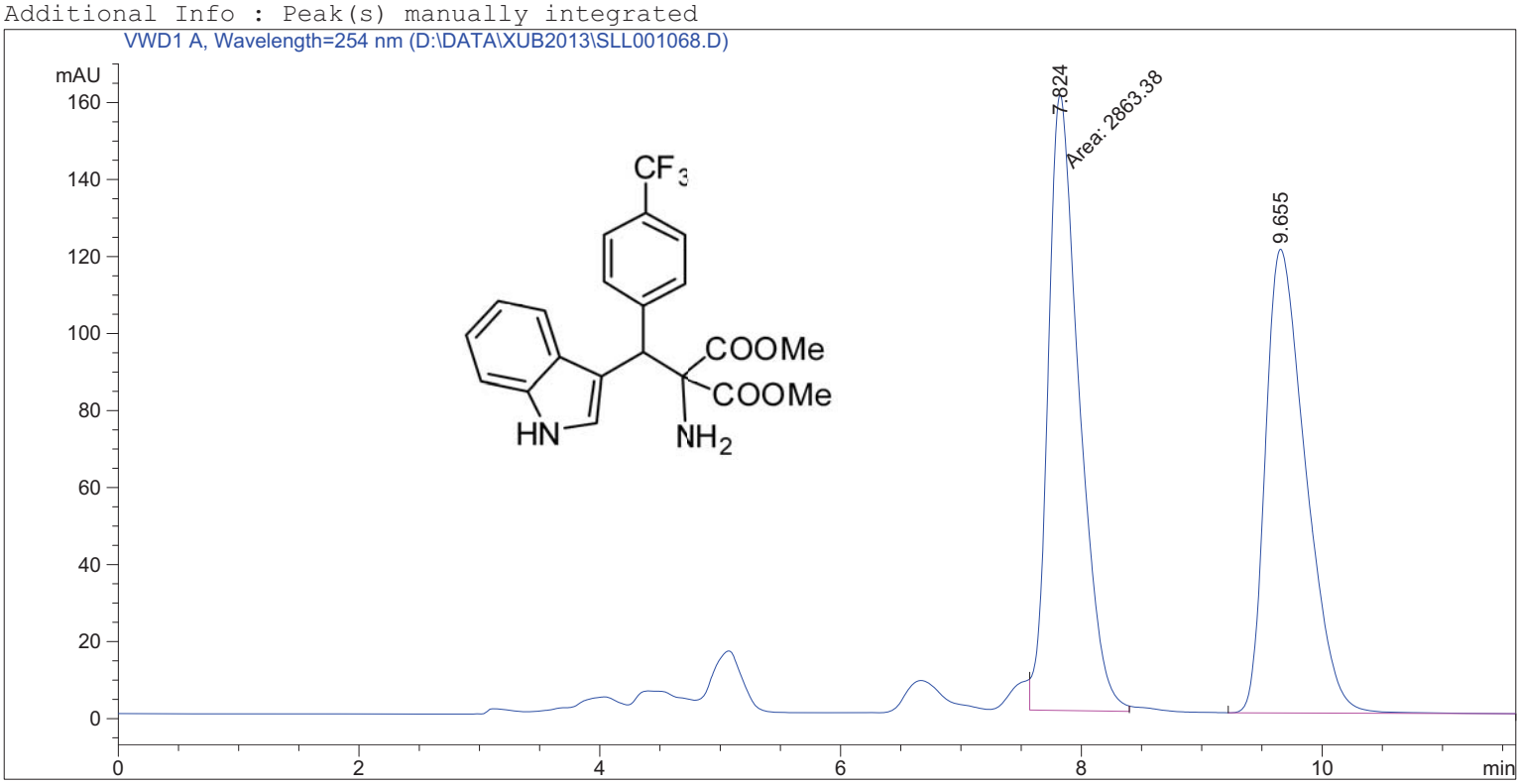

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.033 BB    0.2687 1.17630e4   665.74445  93.6670
   2  10.235 BB    0.2906  795.31909   42.04733   6.3330

Totals :                  1.25583e4   707.79177


==========================================================================
                          *** End of Report ***
```


8.13
7.70
7.69
7.67
7.65
7.49
7.48
7.47
7.33
7.30
7.26
7.14
7.12
7.07
7.05
5.51
3.71
3.53
2.27
1.61
0.00
0.96
3.08
2.87
0.92
1.01
1.03
1.00
2.97
3.04
2.02
f1 (ppm)
CF3
COOMe
COOMe
HN
NH2
3m


170.95
170.55
143.93
135.42
129.89
127.04
124.96
123.46
122.11
119.48
118.59
113.78
111.02
77.45
77.02
76.60
70.54
53.34
53.26
46.53
CF3
COOMe
COOMe
HN
NH2
3m
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001068.D
Sample Name: SLL2013-7-6-2-0

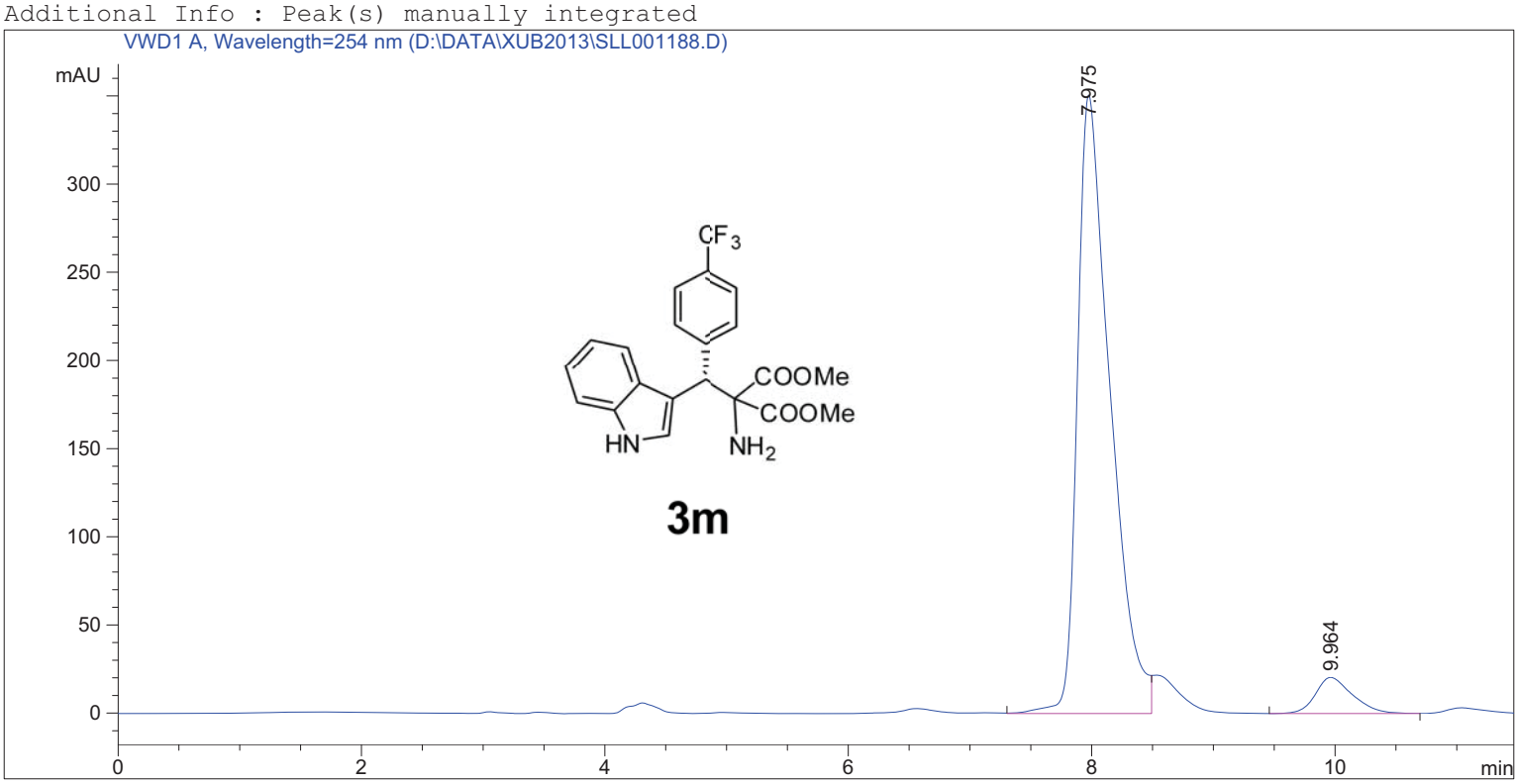
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/9/2013 4:49:13 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\IA_20_1.M
Last changed    : 7/9/2013 4:47:33 PM by LNF
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:10:22 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



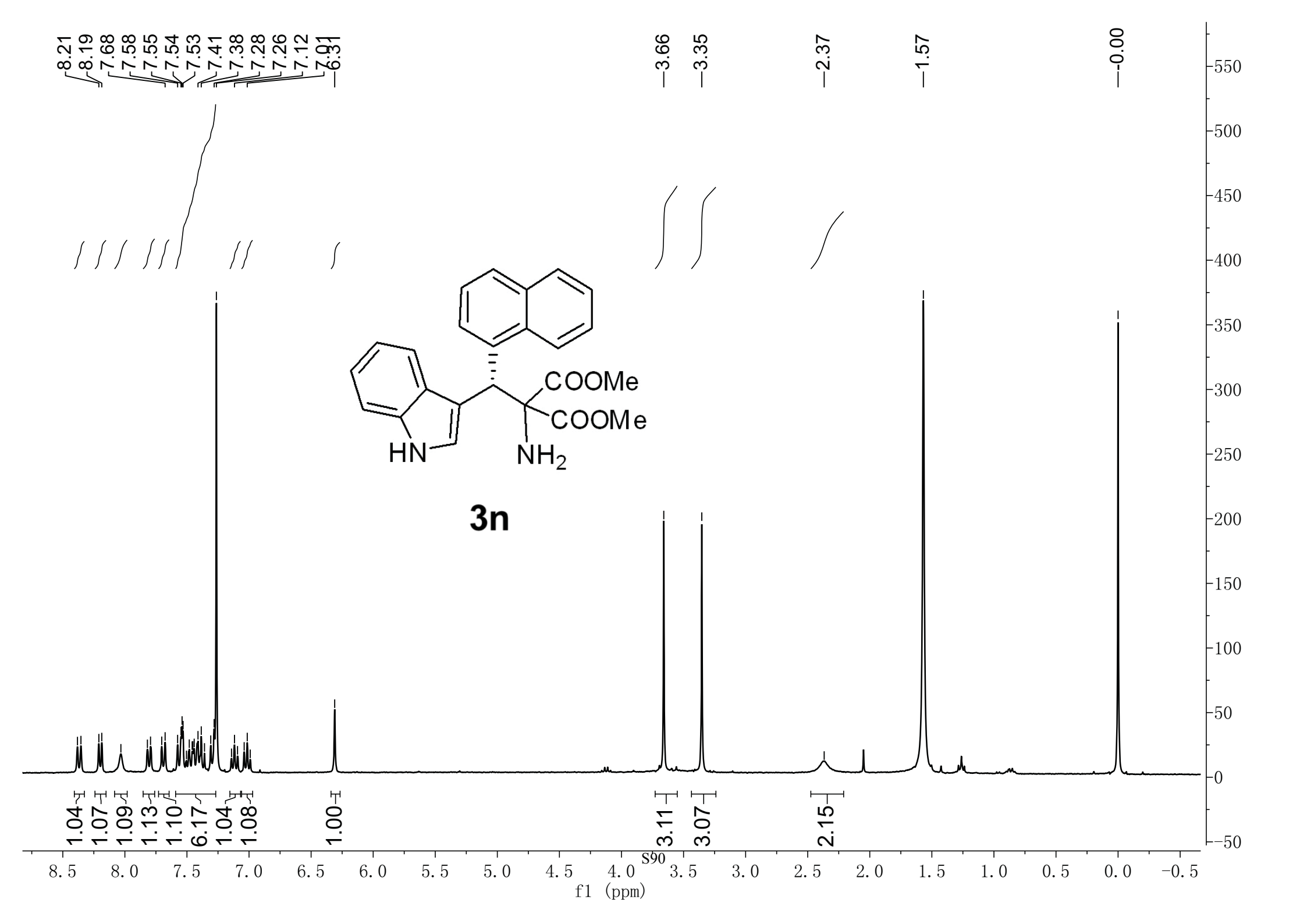

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.824 MF    0.2984 2863.37549  159.92139  50.6426
   2   9.655 BBA   0.3431 2790.70703  120.45130  49.3574

Totals :                  5654.08252  280.37269

==========================================================================
                        *** End of Report ***
```

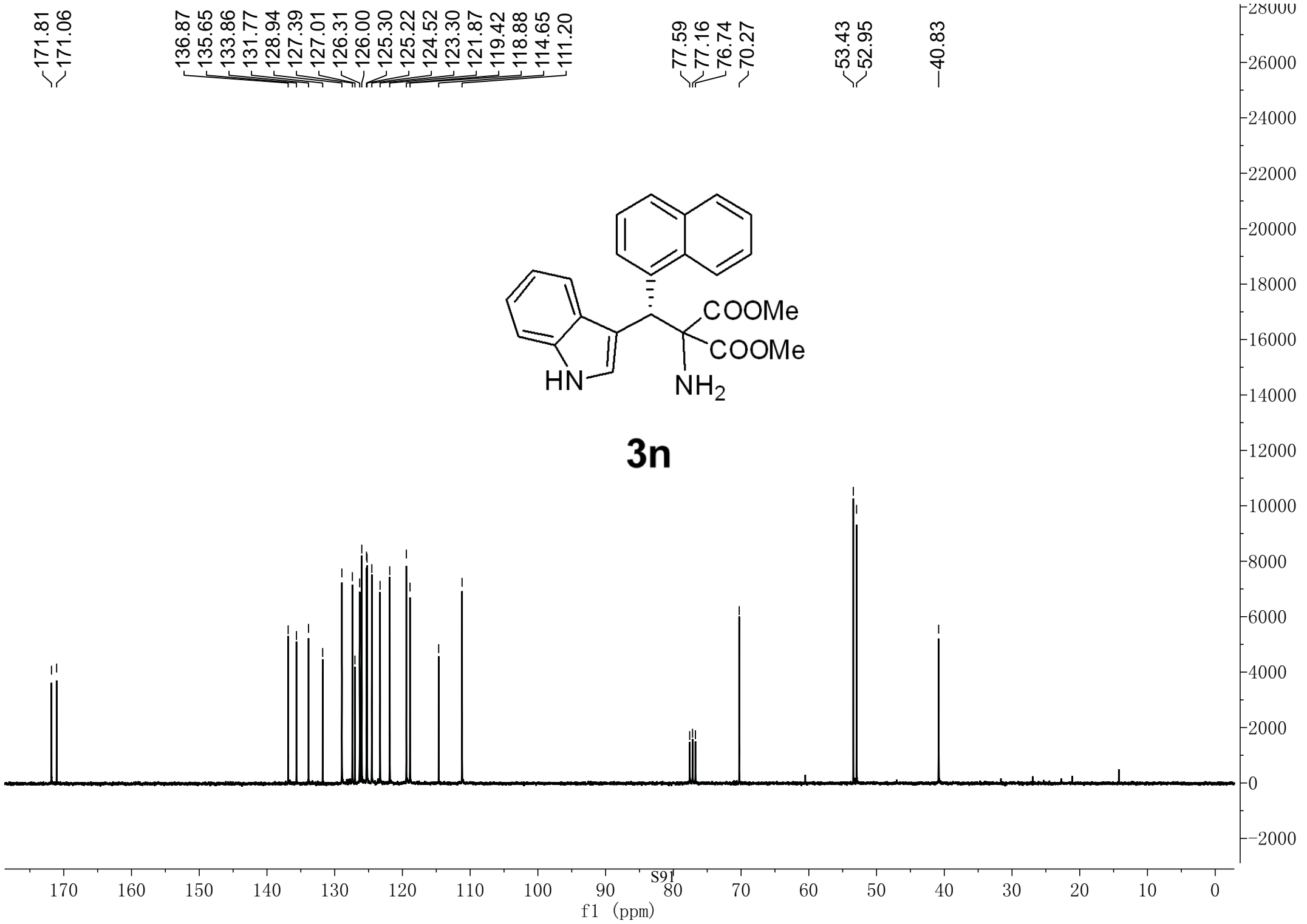
Data File D:\DATA\XUB2013\SLL001188.D
Sample Name: SLL2013-8-23-4

```
==========================================================================
Acq. Operator    : xub
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 9/3/2013 4:45:06 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 9/3/2013 4:56:25 PM by xub
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:11:47 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



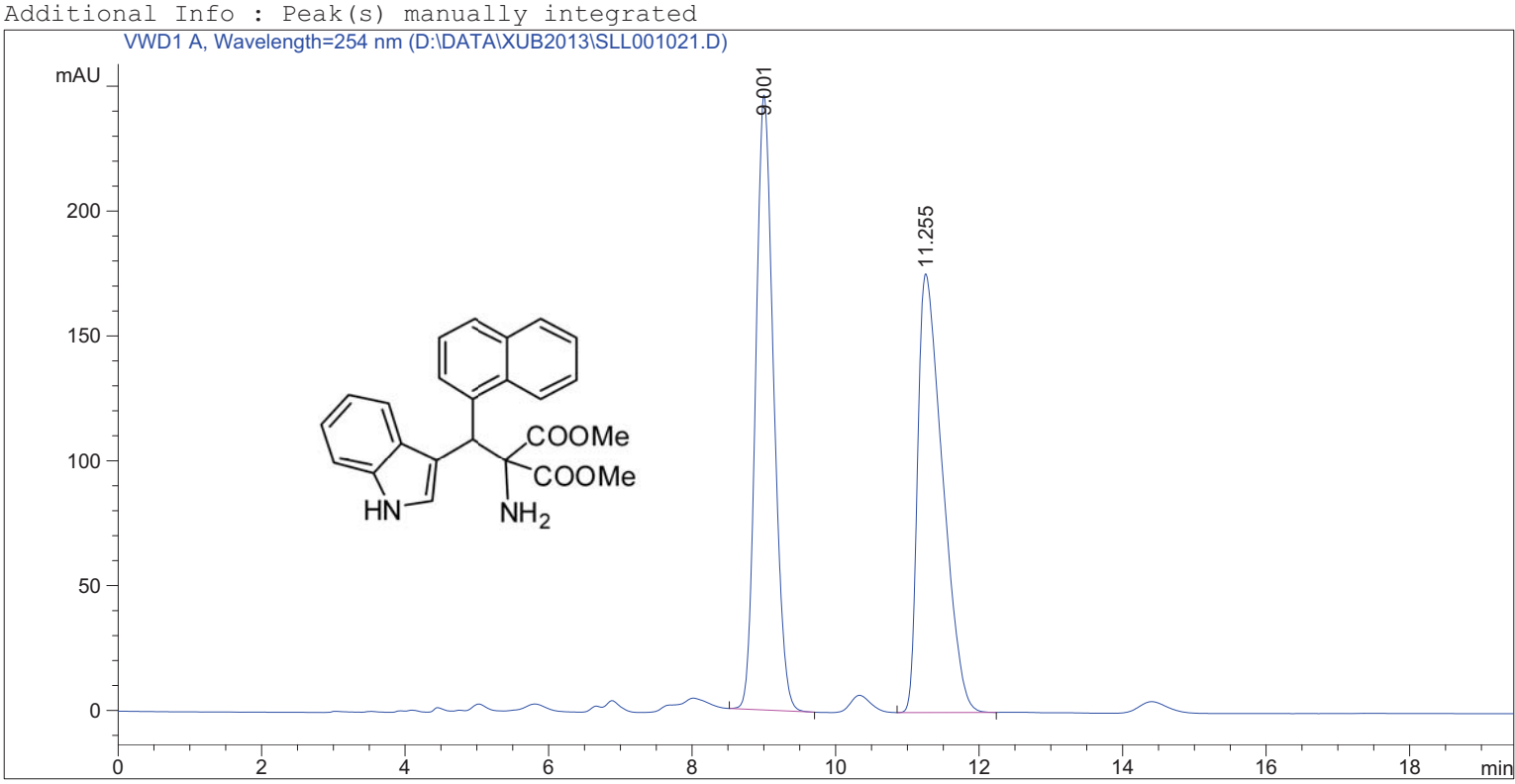

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.975 VV    0.2678 6561.84912  350.70450  93.8104
   2   9.964 BB    0.3118  432.94830   20.50582   6.1896

Totals :                  6994.79742  371.21031


==========================================================================
                          *** End of Report ***
```


8.21
8.19
7.68
7.58
7.55
7.54
7.53
7.41
7.38
7.28
7.26
7.12
7.01
6.31
3.66
3.35
2.37
1.57
0.00
COOMe
COOMe
HN
NH2
3n
1.04
1.07
1.09
1.13
1.10
6.17
1.04
1.08
1.00
3.11
3.07
2.15
f1 (ppm)


171.81
171.06
136.87
135.65
133.86
131.77
128.94
127.39
127.01
126.31
126.00
125.30
125.22
124.52
123.30
121.87
119.42
118.88
114.65
111.20
77.59
77.16
76.74
70.27
53.43
52.95
40.83
COOMe
COOMe
HN
NH2
3n
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001021.D
Sample Name: SLL2013-6-24-1-0

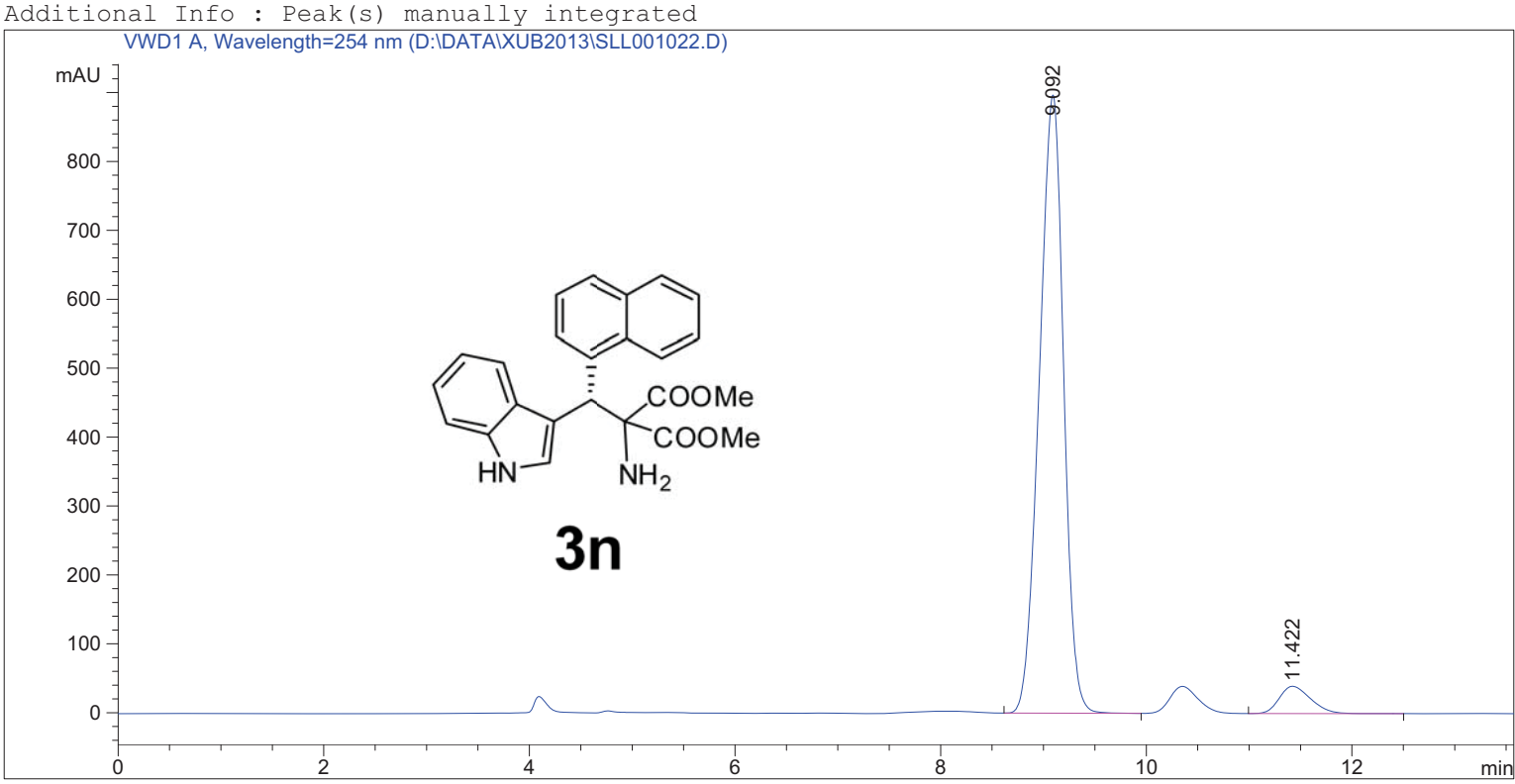
```
================================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/26/2013 11:14:10 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 6/26/2013 11:10:36 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:11:39 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



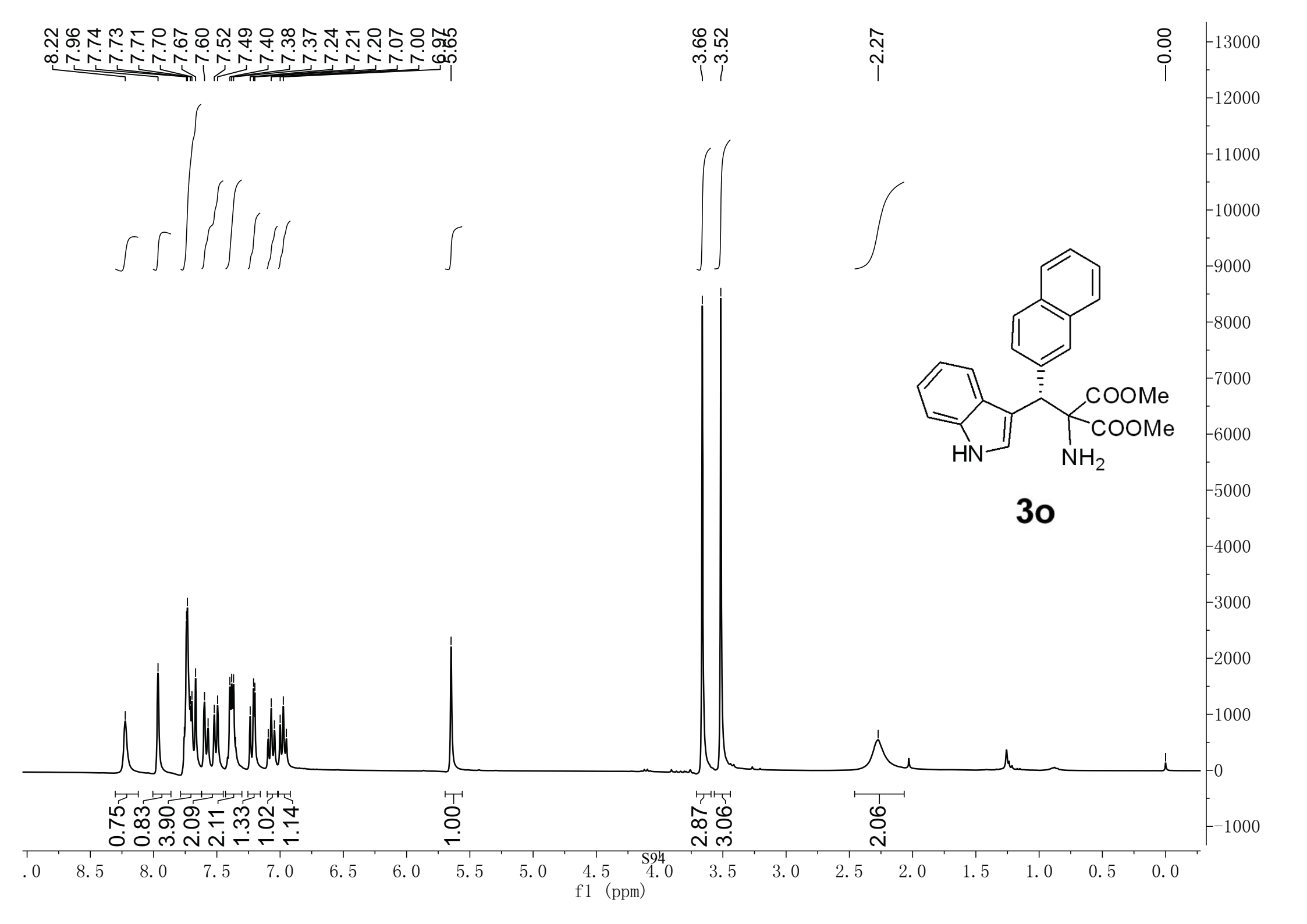

```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.001 BB    0.2646 4263.36328  246.24826  50.2196
   2  11.255 BB    0.3666 4226.06982  175.67380  49.7804

Totals :                  8489.43311  421.92206


================================================================================
                          *** End of Report ***
```

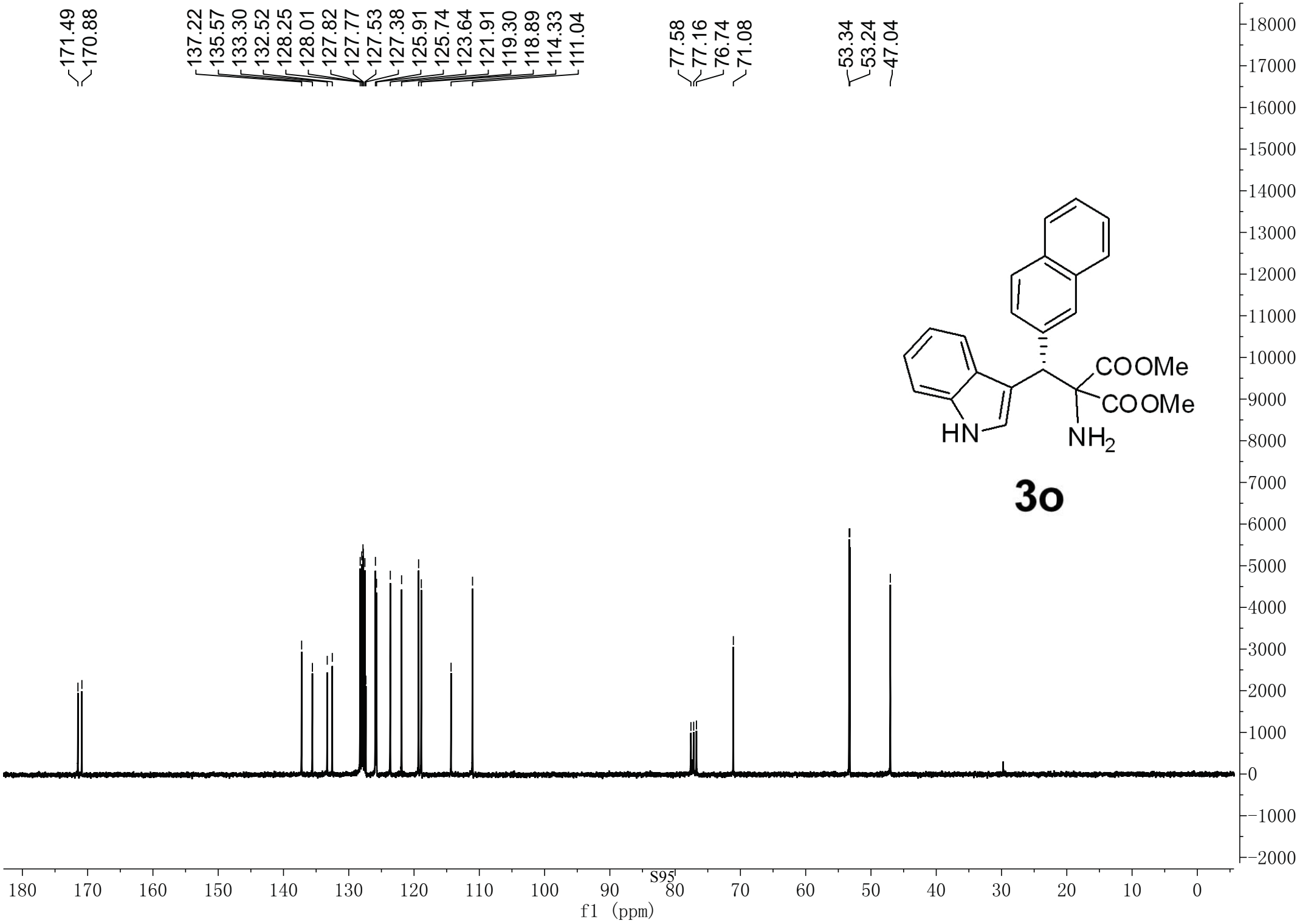
Data File D:\DATA\XUB2013\SLL001022.D
Sample Name: SLL2013-6-24-1

```
==========================================================================
Acq. Operator    : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/26/2013 11:33:56 AM
                                                  Inj Volume : No inj
Acq. Method      : D:\METHOD\AD_30_1.M
Last changed     : 6/26/2013 11:33:40 AM by LNF
                   (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed     : 9/29/2013 9:11:39 AM by LNF
                   (modified after loading)
Additional Info : Peak(s) manually integrated
```



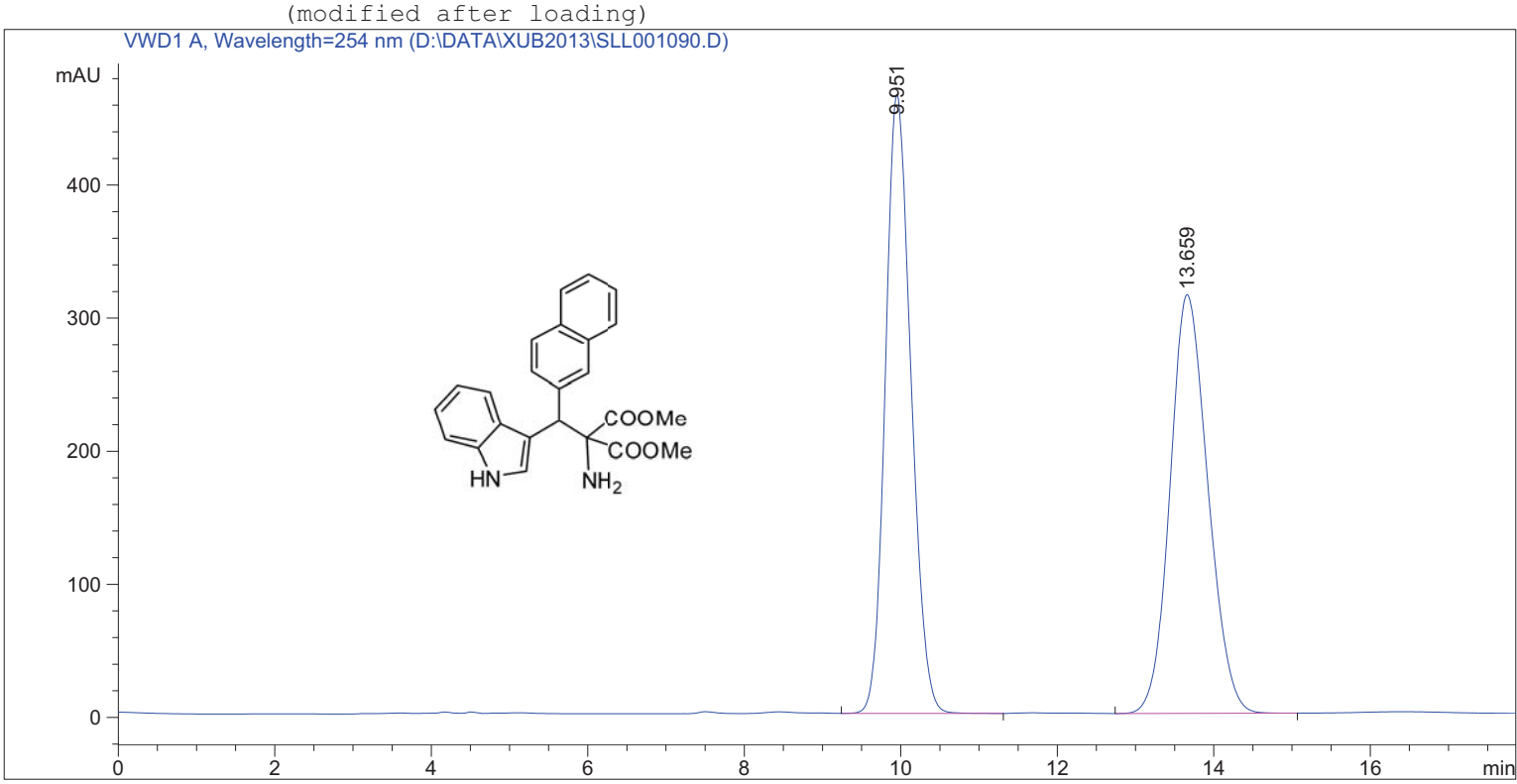

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.092 BB    0.2553 1.47039e4   896.99725  94.6123
   2  11.422 BB    0.3284  837.31812   39.57407   5.3877

Totals :                  1.55412e4   936.57132


==========================================================================
                        *** End of Report ***
```


8.22
7.96
7.74
7.73
7.71
7.70
7.67
7.60
7.52
7.49
7.40
7.38
7.37
7.24
7.21
7.20
7.07
7.00
6.97
6.95
3.66
3.52
2.27
0.00
COOMe
COOMe
HN
NH2
3o
0.75
0.83
3.90
2.09
2.11
1.33
1.02
1.14
1.00
2.87
3.06
2.06
f1 (ppm)


171.49
170.88
137.22
135.57
133.30
132.52
128.25
128.01
127.82
127.77
127.53
127.38
125.91
125.74
123.64
121.91
119.30
118.89
114.33
111.04
77.58
77.16
76.74
71.08
53.34
53.24
47.04
COOMe
COOMe
HN
NH2
3o
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001090.D
Sample Name: SLL2013-7-19-1-0

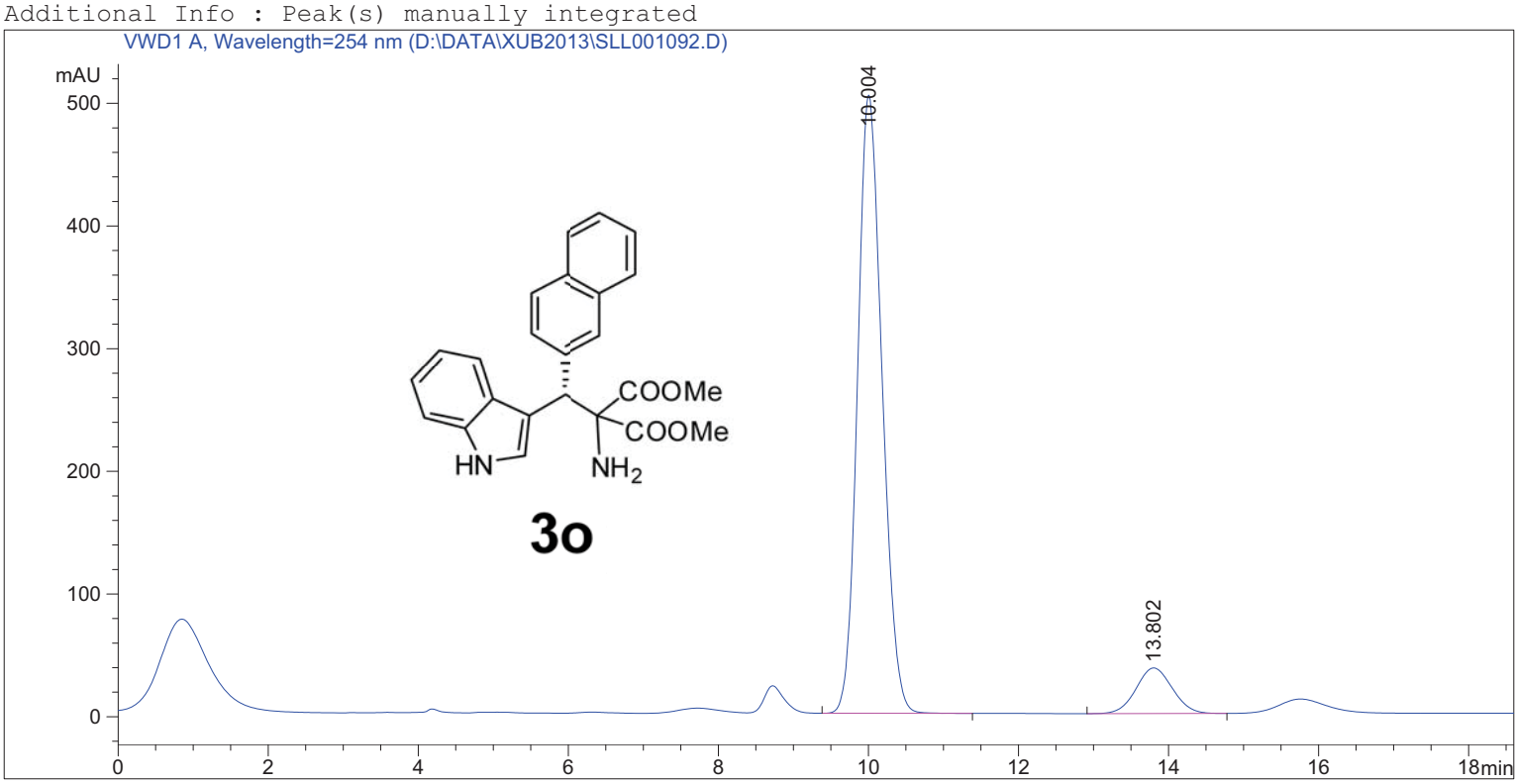
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/25/2013 10:26:39 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/25/2013 10:26:20 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:35:21 AM by LNF
                  (modified after loading)
```



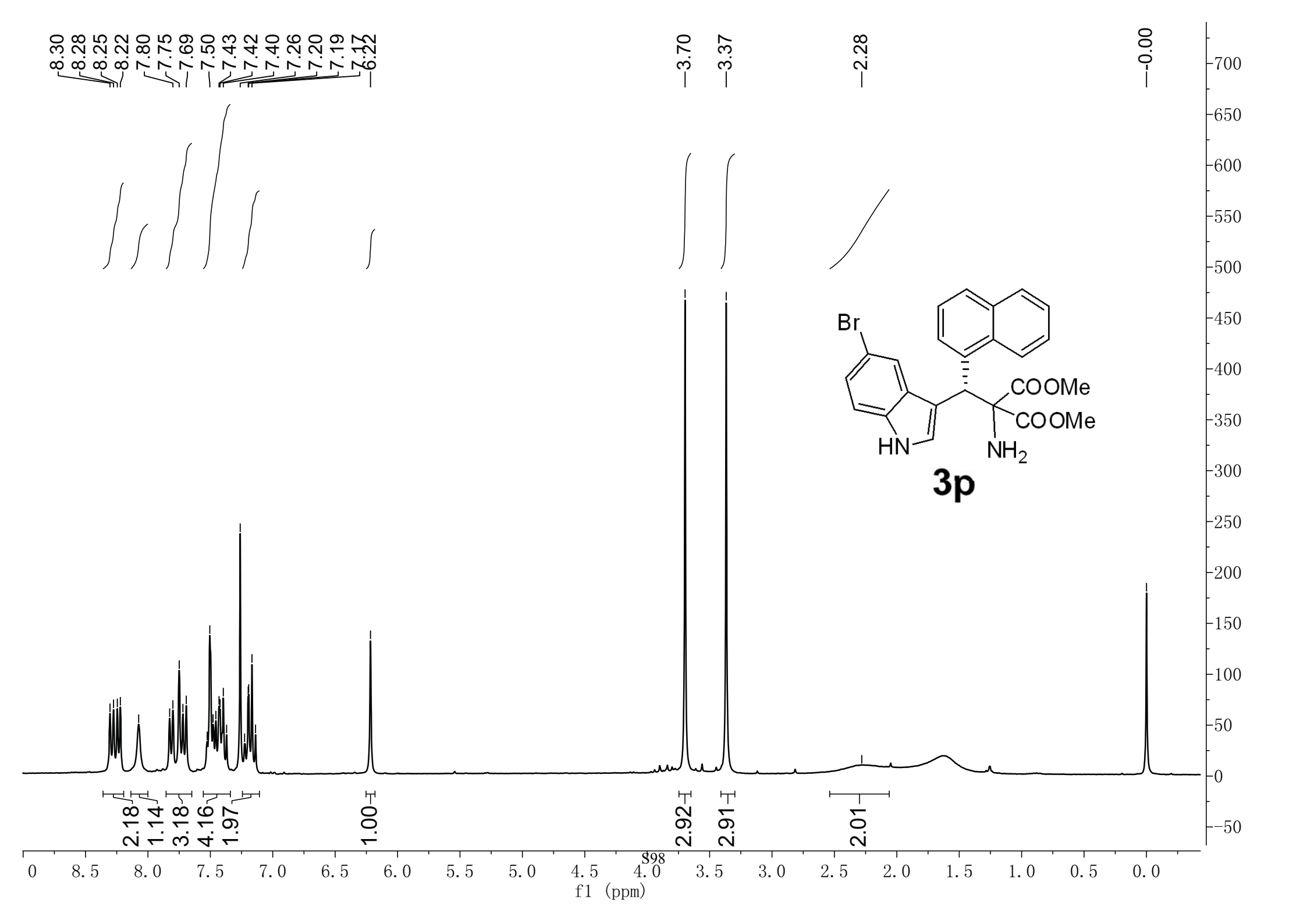

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.951 BB    0.3505 1.06006e4   464.84909  50.0472
   2  13.659 BB    0.5191 1.05806e4   314.58789  49.9528

Totals :                  2.11811e4   779.43698

==========================================================================
                          *** End of Report ***
```

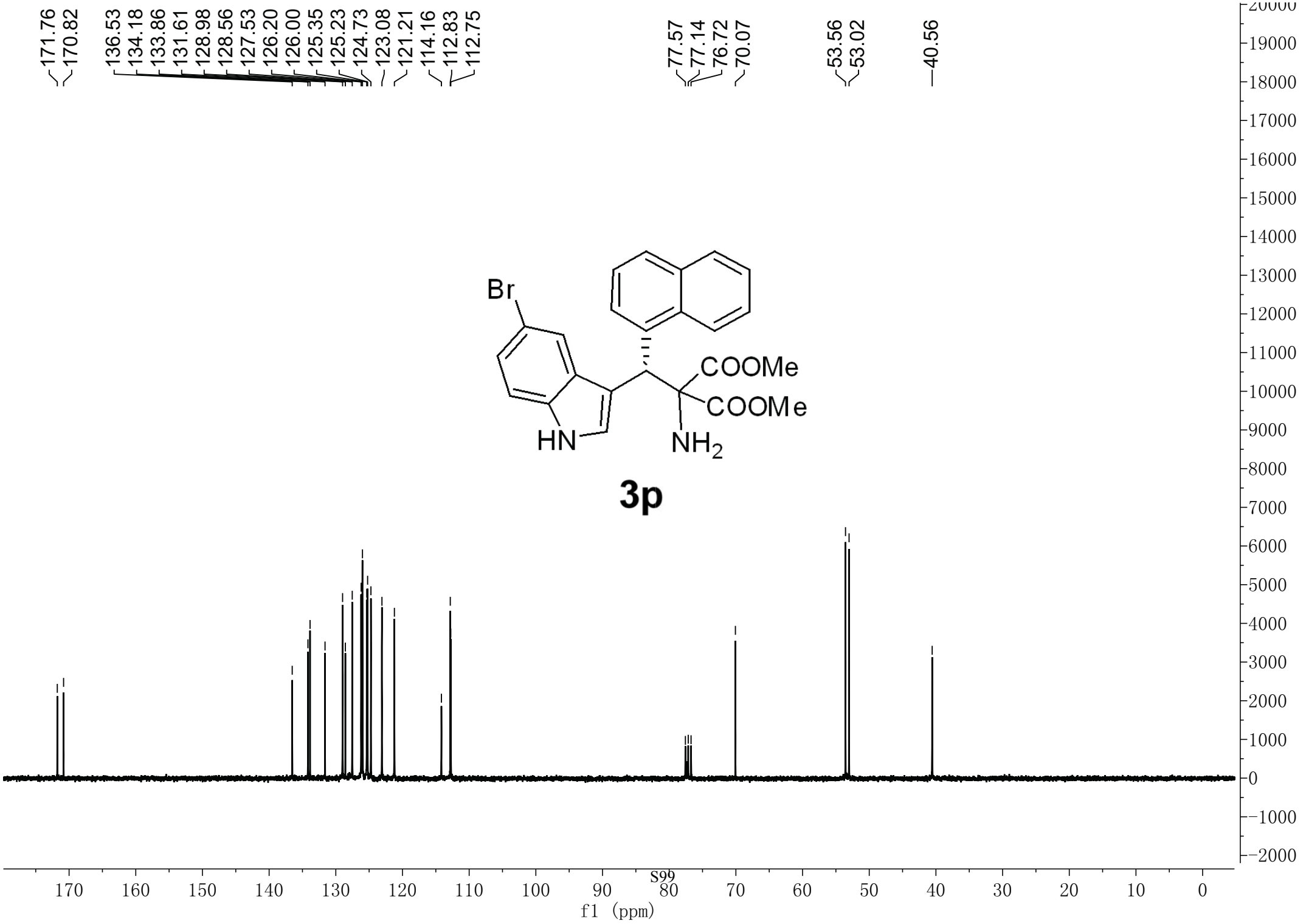
Data File D:\DATA\XUB2013\SLL001092.D
Sample Name: SLL2013-7-19-1

```
    ======================================================================
    Acq. Operator   : LNF
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 7/25/2013 10:59:41 AM
                                                    Inj Volume : No inj
    Acq. Method     : D:\METHOD\AD_30_1.M
    Last changed    : 7/25/2013 10:59:27 AM by LNF
                      (modified after loading)
    Analysis Method : D:\METHOD\AD_30_1.M
    Last changed    : 9/29/2013 11:10:22 AM by LNF
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



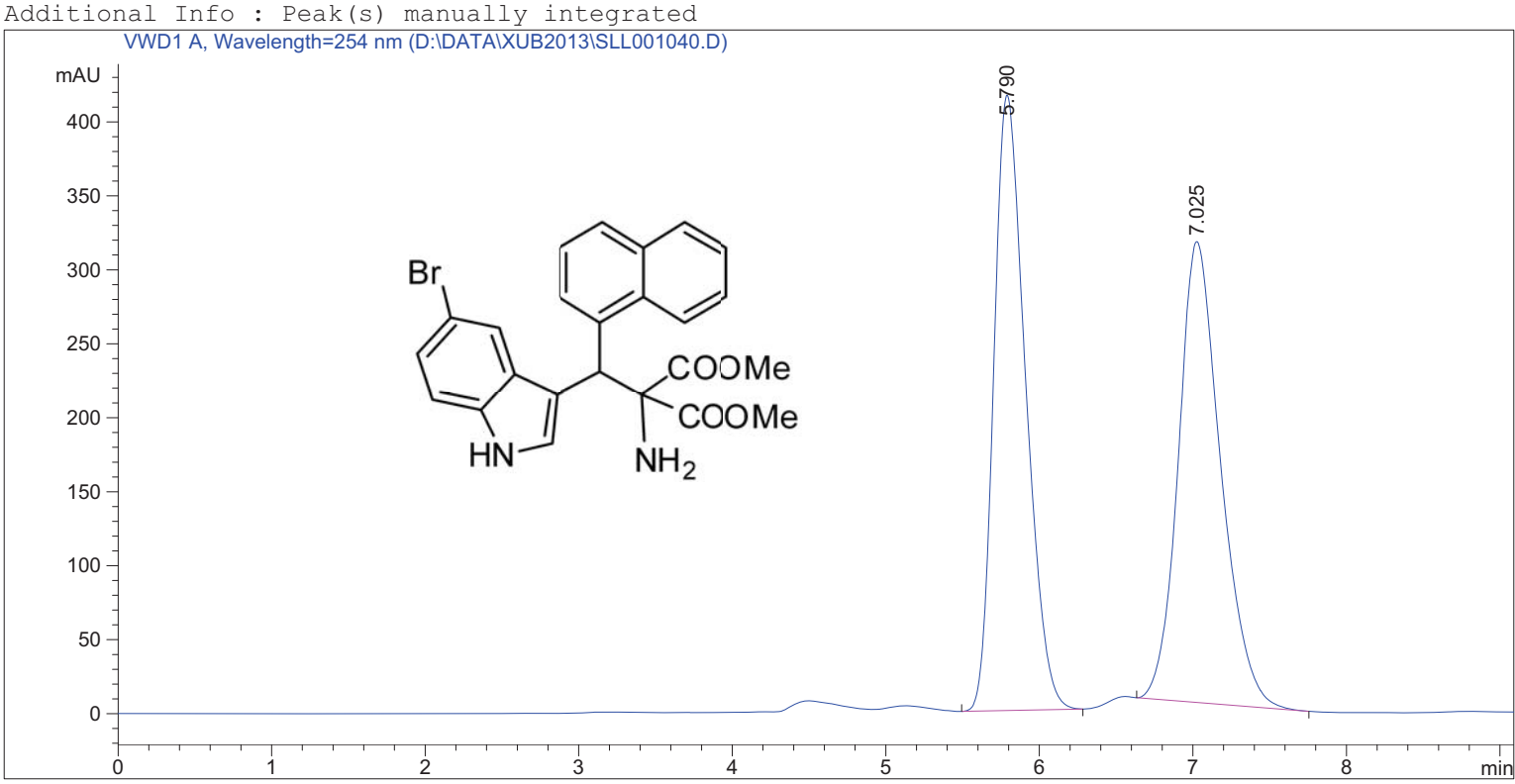

```
    ======================================================================
                               Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  10.004 BB    0.3507 1.14376e4   503.89600  90.1625
       2  13.802 BB    0.5227 1247.93640   37.17181   9.8375

    Totals :                  1.26856e4   541.06781


    ======================================================================
                               *** End of Report ***
```


8.30
8.28
8.25
8.22
7.80
7.75
7.69
7.50
7.43
7.42
7.40
7.26
7.20
7.19
7.17
6.22
3.70
3.37
2.28
0.00
Br
COOMe
COOMe
HN
NH2
3p
2.18
1.14
3.18
4.16
1.97
1.00
2.92
2.91
2.01
f1 (ppm)


171.76
170.82
136.53
134.18
133.86
131.61
128.98
128.56
127.53
126.20
126.00
125.35
125.23
124.73
123.08
121.21
114.16
112.83
112.75
77.57
77.14
76.72
70.07
53.56
53.02
40.56
Br
COOMe
COOMe
HN
NH2
3p
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001040.D
Sample Name: SLL2013-7-1-1

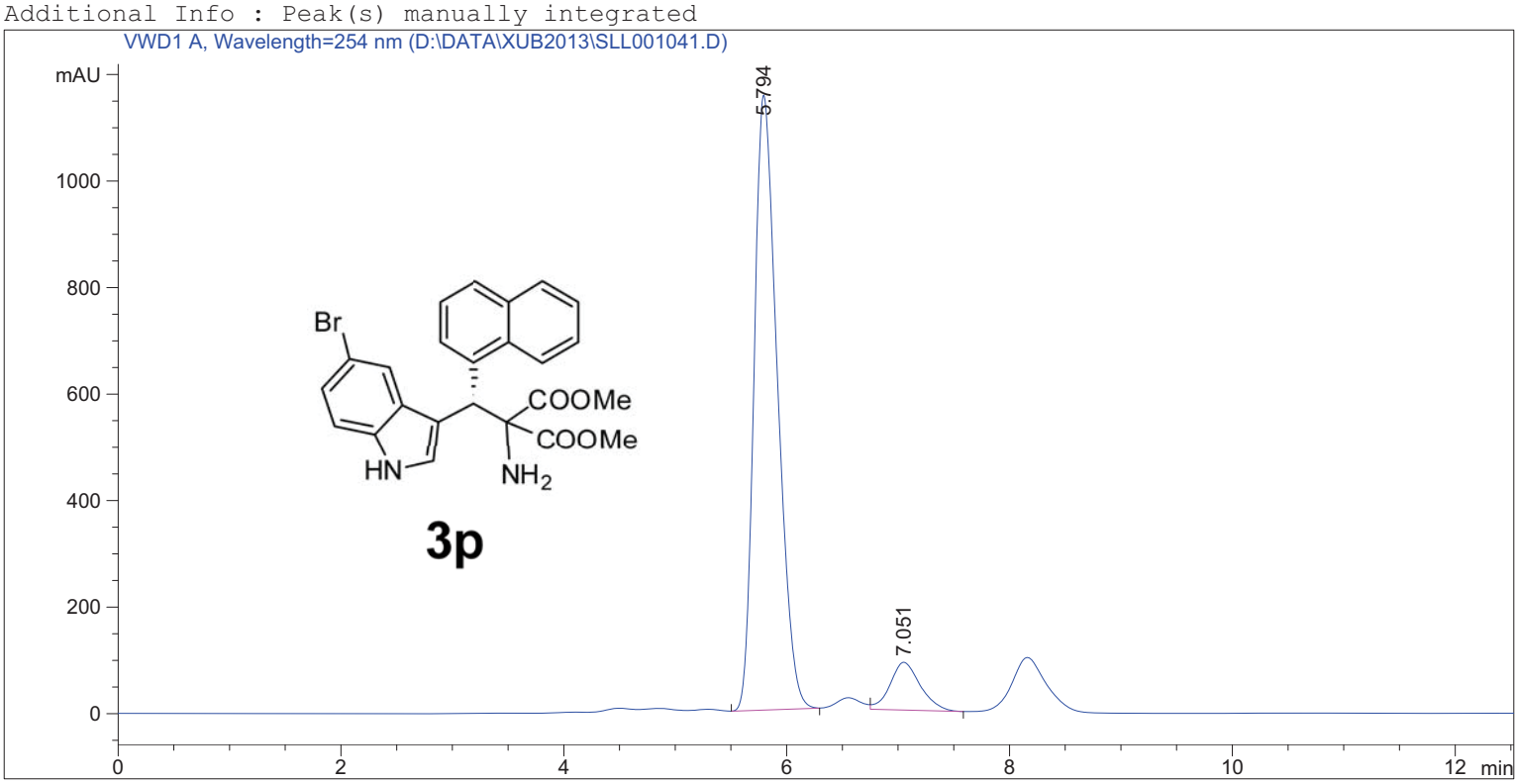
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/4/2013 10:12:08 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/4/2013 10:11:28 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:23:05 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



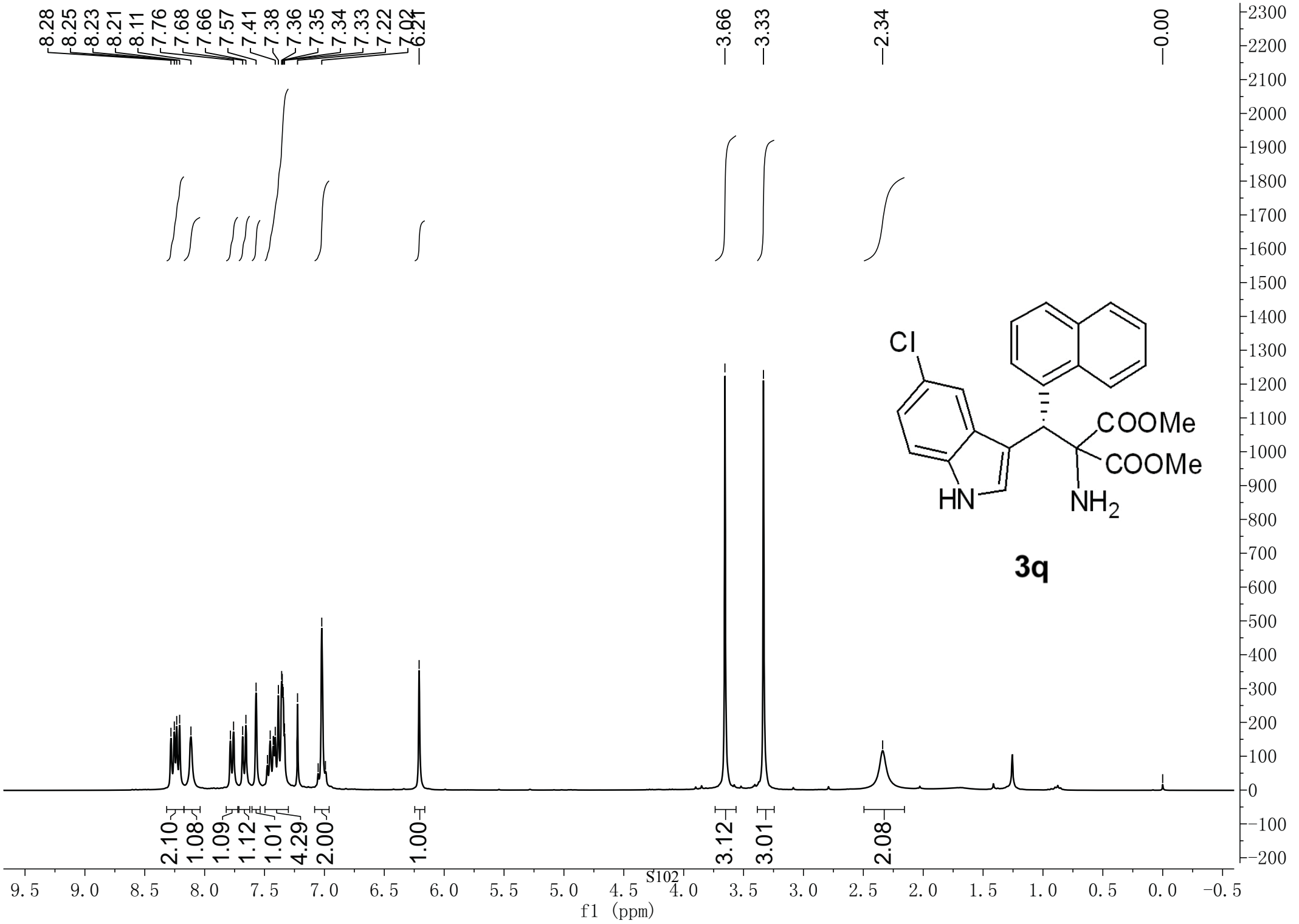

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.790 BB    0.2184 5970.79688  415.92734  50.0872
   2   7.025 BB    0.2854 5950.01611  311.51218  49.9128

Totals :                  1.19208e4   727.43951

==========================================================================
                          *** End of Report ***
```

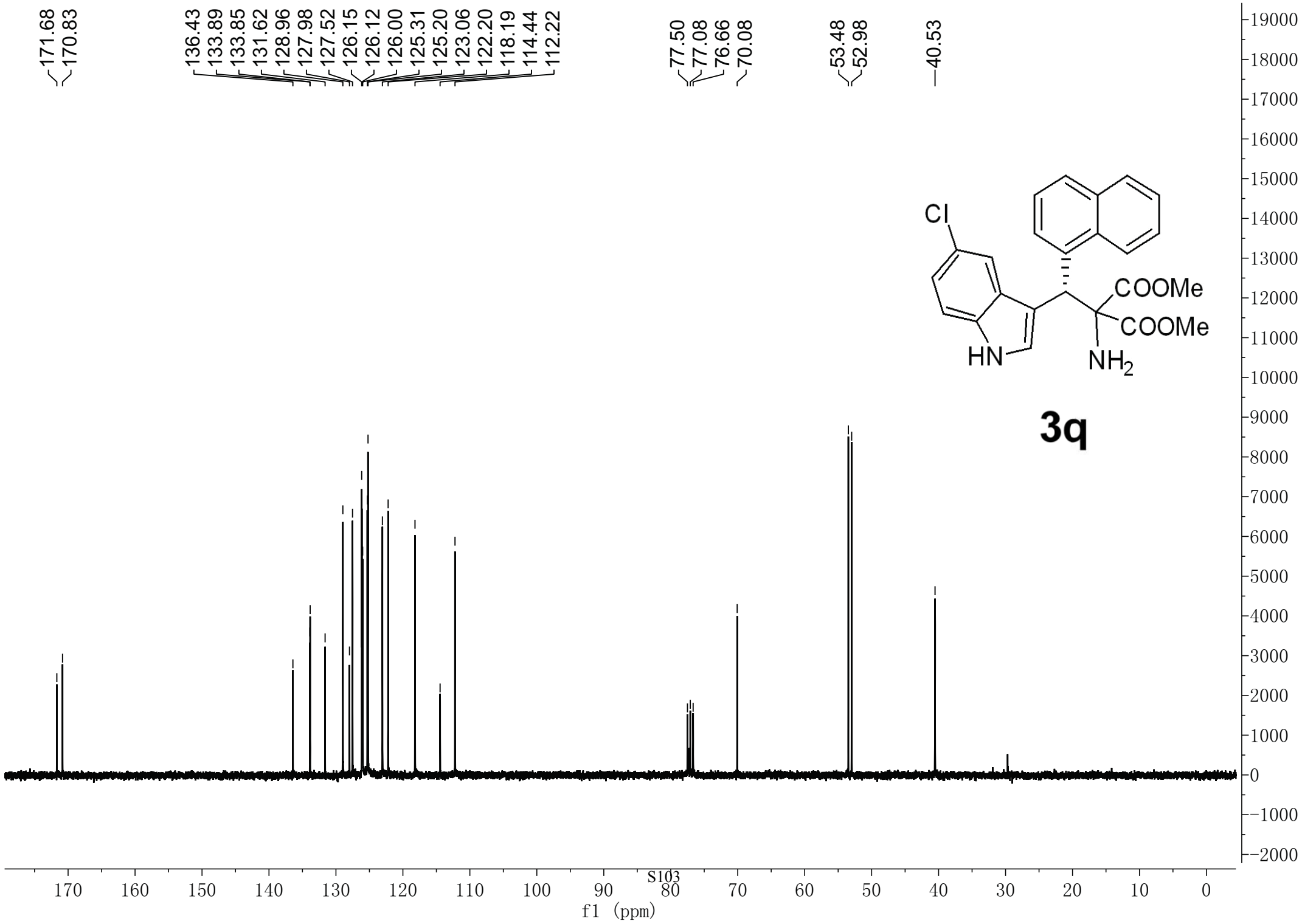
Data File D:\DATA\XUB2013\SLL001041.D
Sample Name: SLL2013-7-1-1

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/4/2013 10:22:41 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/4/2013 10:21:19 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:27:32 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



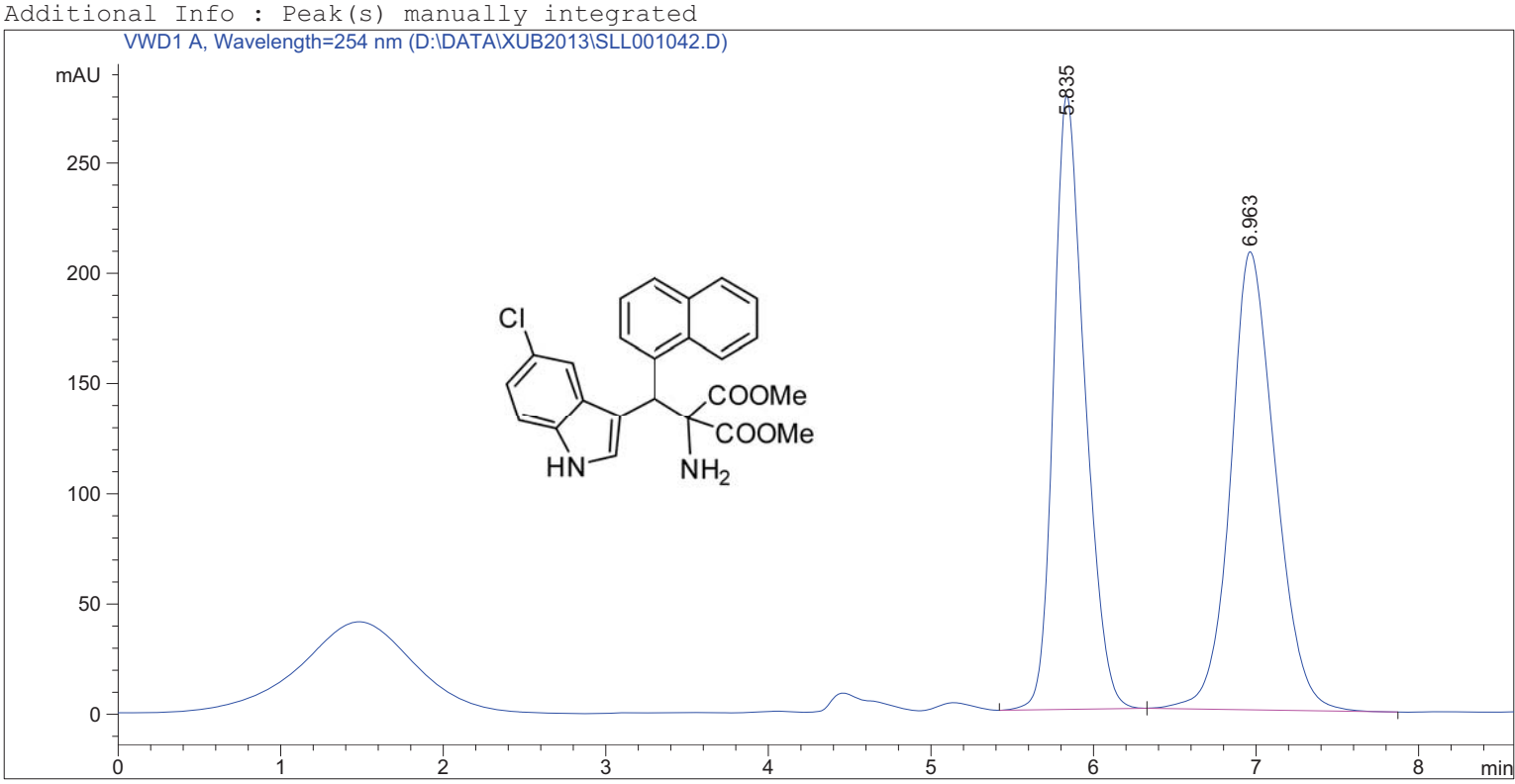

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.794 BB    0.2219 1.67713e4  1154.48596  90.8222
   2   7.051 VB    0.2830 1694.77100   89.70208   9.1778

Totals :                  1.84661e4  1244.18804

==========================================================================
                        *** End of Report ***
```


8.28
8.25
8.23
8.21
8.11
7.76
7.68
7.66
7.57
7.41
7.38
7.36
7.35
7.34
7.33
7.22
7.02
6.21
3.66
3.33
2.34
0.00
Cl
COOMe
COOMe
HN
NH2
3q
2.10
1.08
1.09
1.12
1.01
4.29
2.00
1.00
3.12
3.01
2.08
f1 (ppm)


171.68
170.83
136.43
133.89
133.85
131.62
128.96
127.98
127.52
126.15
126.12
126.00
125.31
125.20
123.06
122.20
118.19
114.44
112.22
77.50
77.08
76.66
70.08
53.48
52.98
40.53
Cl
COOMe
COOMe
HN
NH2
3q
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001042.D
Sample Name: SLL2013-7-1-3-0

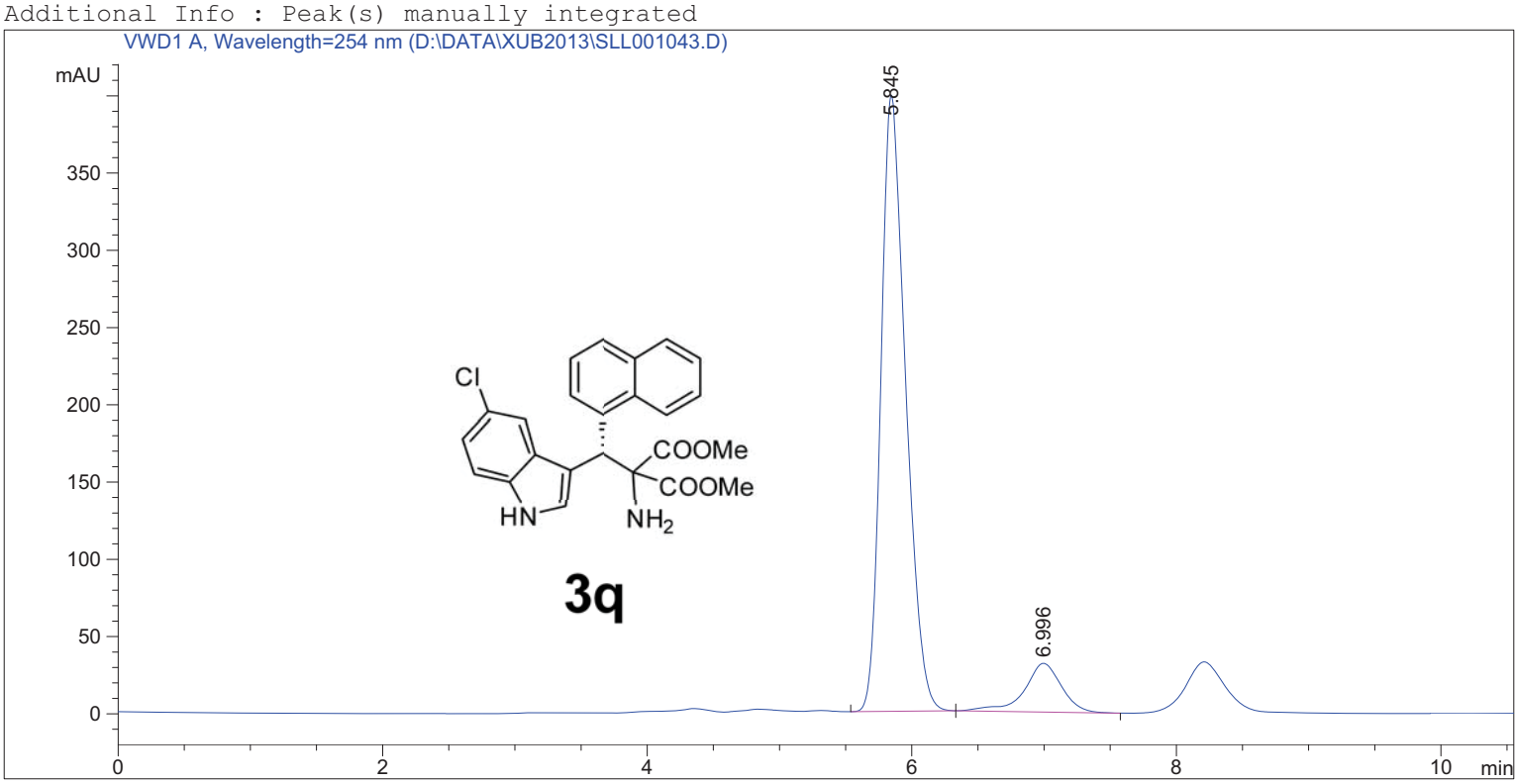
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/4/2013 10:35:34 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/4/2013 10:35:18 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:30:09 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



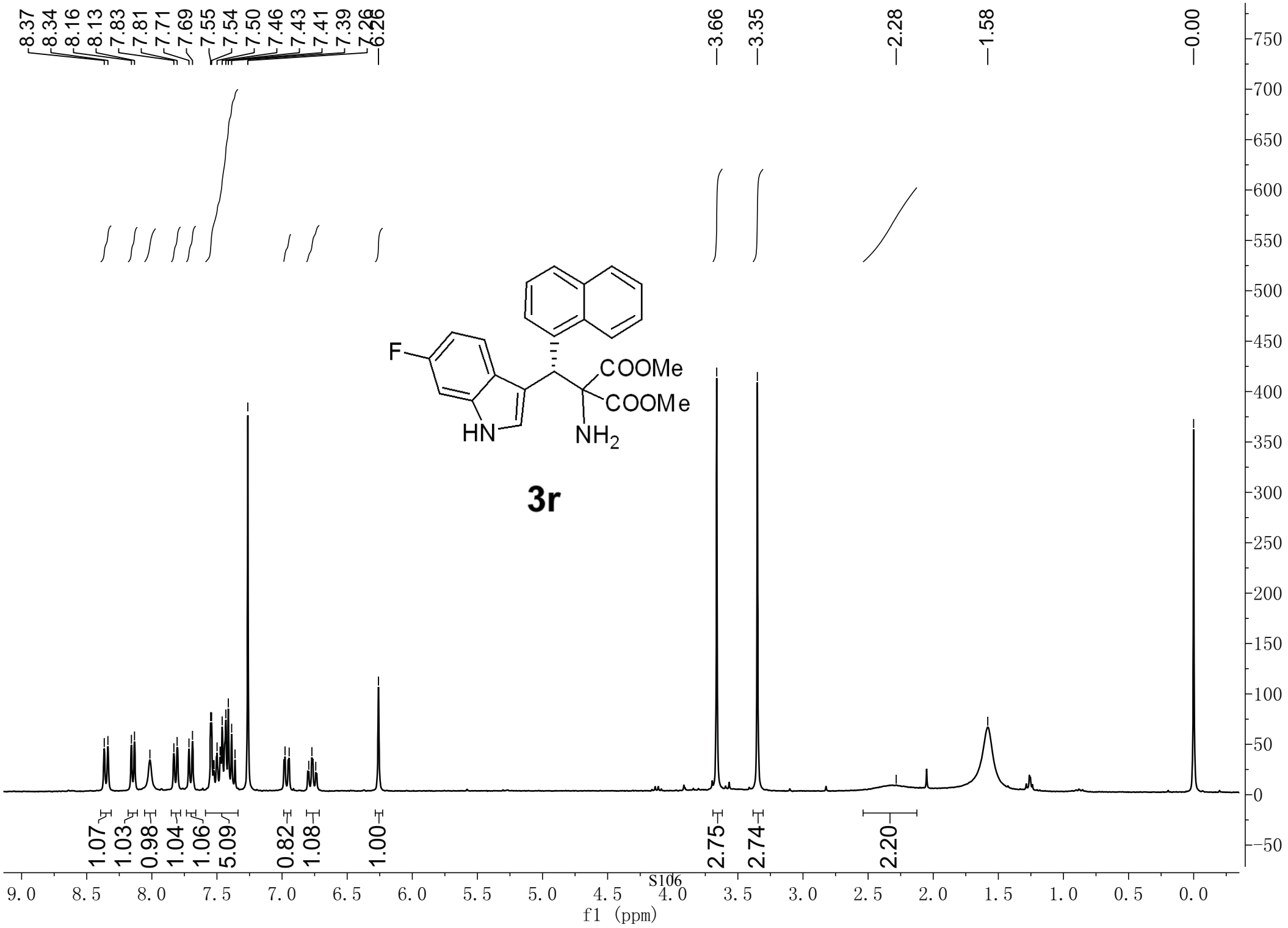

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.835 BB    0.1996 3722.54883  278.55453  48.3372
   2   6.963 BB    0.2875 3978.66675  207.74121  51.6628

Totals :                  7701.21558  486.29575

==========================================================================
                          *** End of Report ***
```

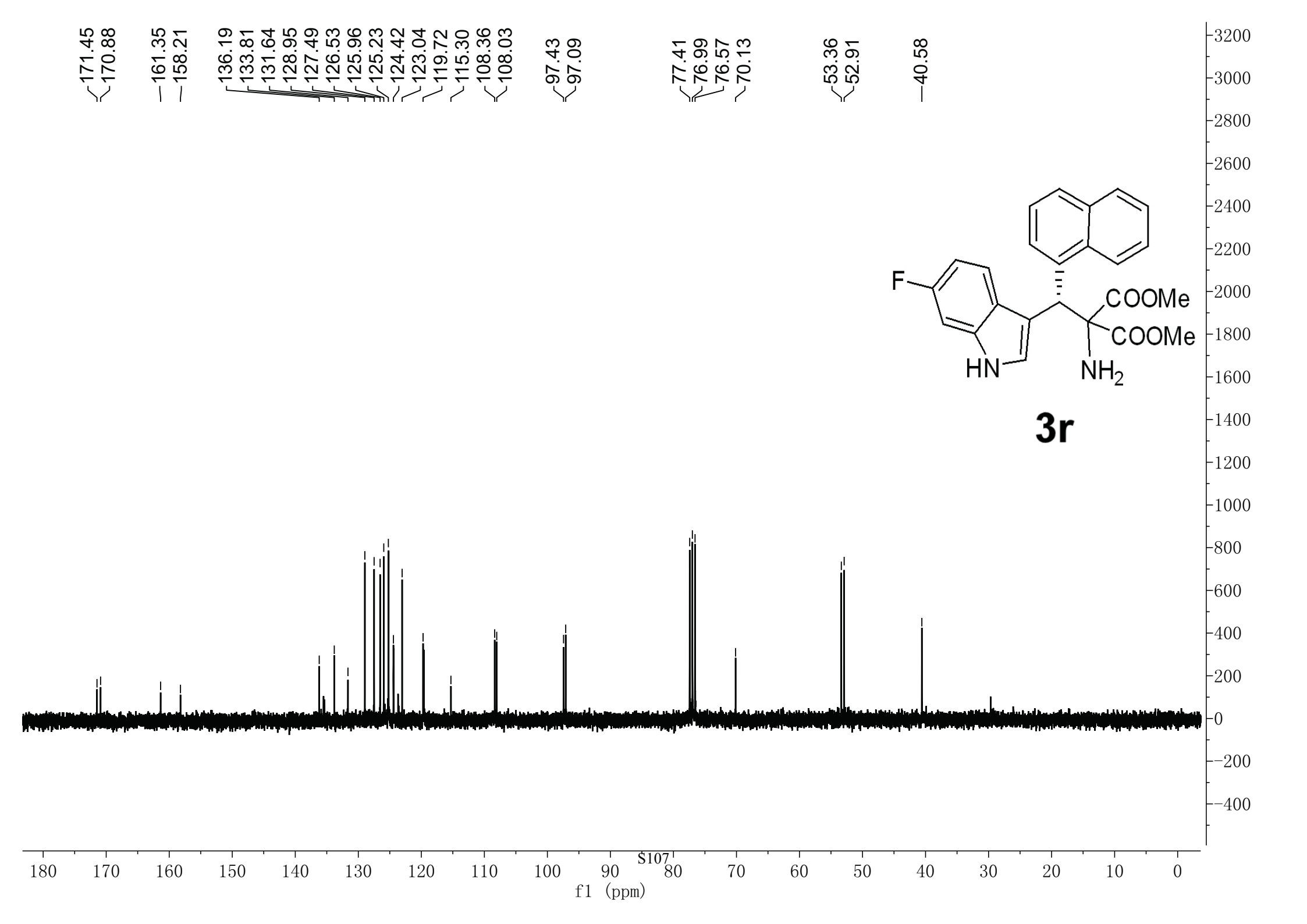
Data File D:\DATA\XUB2013\SLL001043.D
Sample Name: SLL2013-7-1-3

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 7/4/2013 10:44:30 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 7/4/2013 10:44:13 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:30:09 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



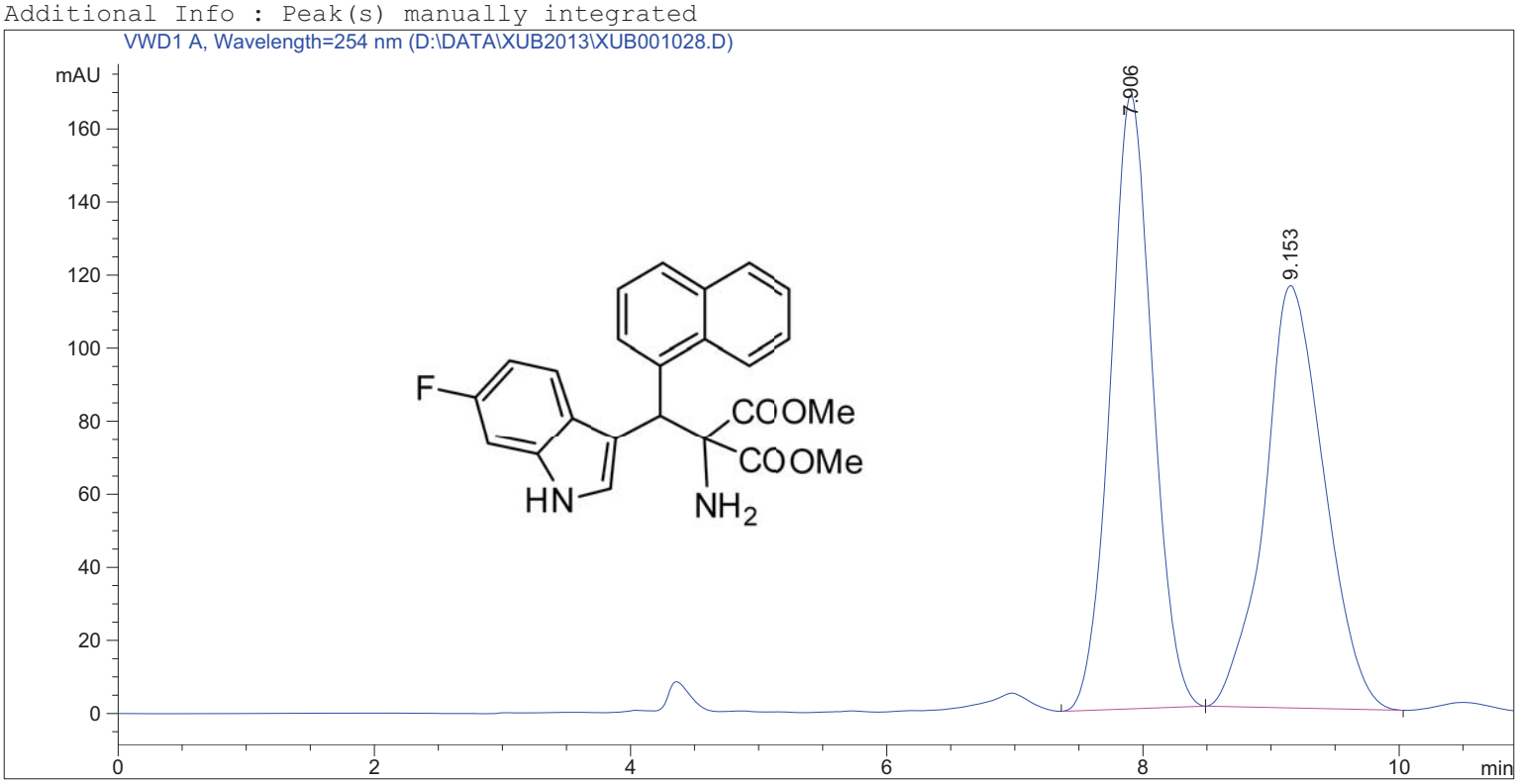

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.845 BB    0.1936 5175.02490  398.87814  89.3453
   2   6.996 BB    0.2894  617.13690   31.54902  10.6547

Totals :                  5792.16180  430.42716

==========================================================================
                        *** End of Report ***
```


8.37
8.34
8.16
8.13
7.83
7.81
7.71
7.69
7.55
7.54
7.50
7.46
7.43
7.41
7.39
7.26
6.26
3.66
3.35
2.28
1.58
0.00
F
COOMe
COOMe
HN
NH2
3r
1.07
1.03
0.98
1.04
1.06
5.09
0.82
1.08
1.00
2.75
2.74
2.20
f1 (ppm)


171.45
170.88
161.35
158.21
136.19
133.81
131.64
128.95
127.49
126.53
125.96
125.23
124.42
123.04
119.72
115.30
108.36
108.03
97.43
97.09
77.41
76.99
76.57
70.13
53.36
52.91
40.58
F
COOMe
COOMe
HN
NH2
3r
f1 (ppm)

Data File D:\DATA\XUB2013\XUB001028.D
Sample Name: SLL2013-6-27-4-0

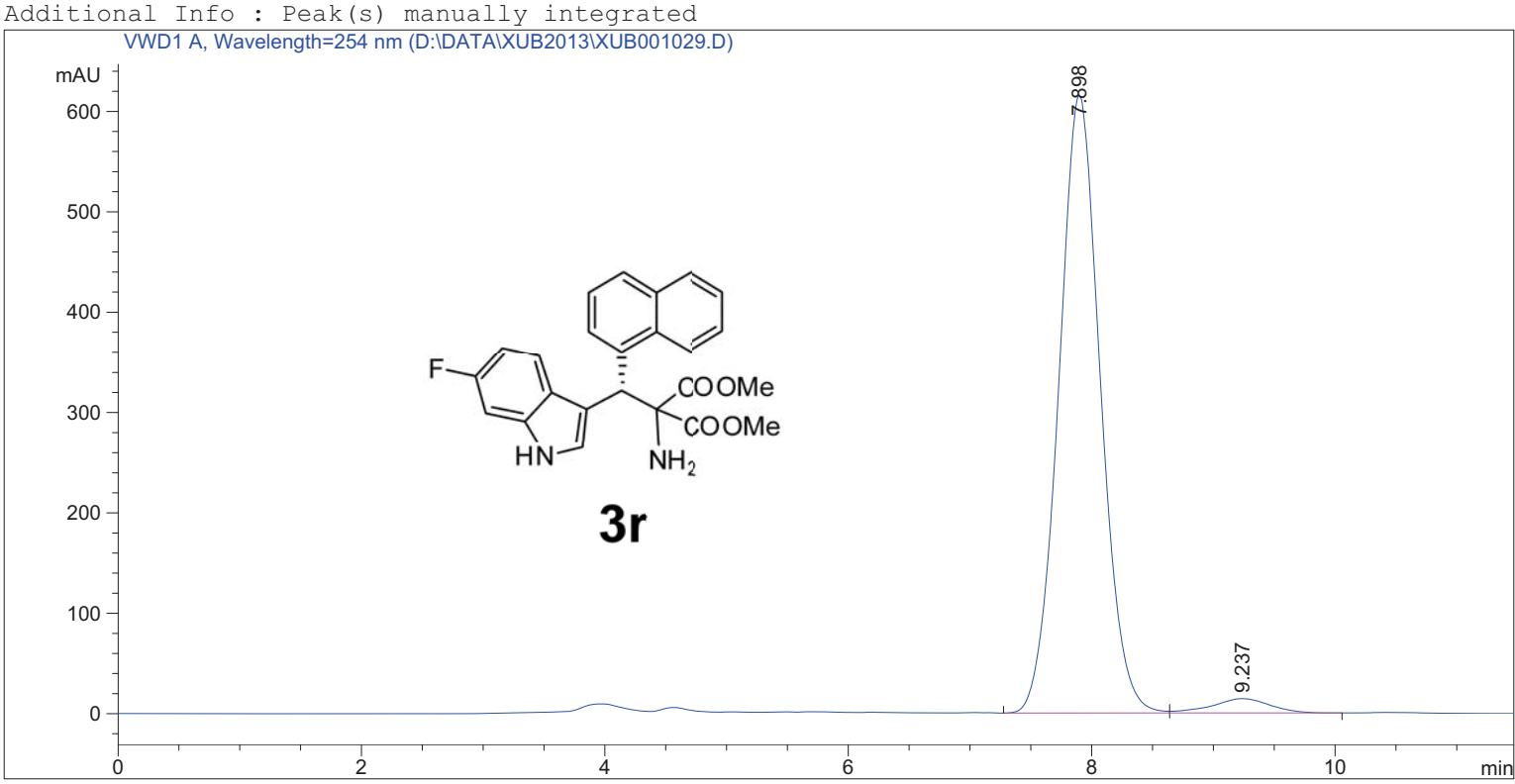
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/30/2013 9:51:30 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 6/30/2013 9:51:02 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:23:05 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



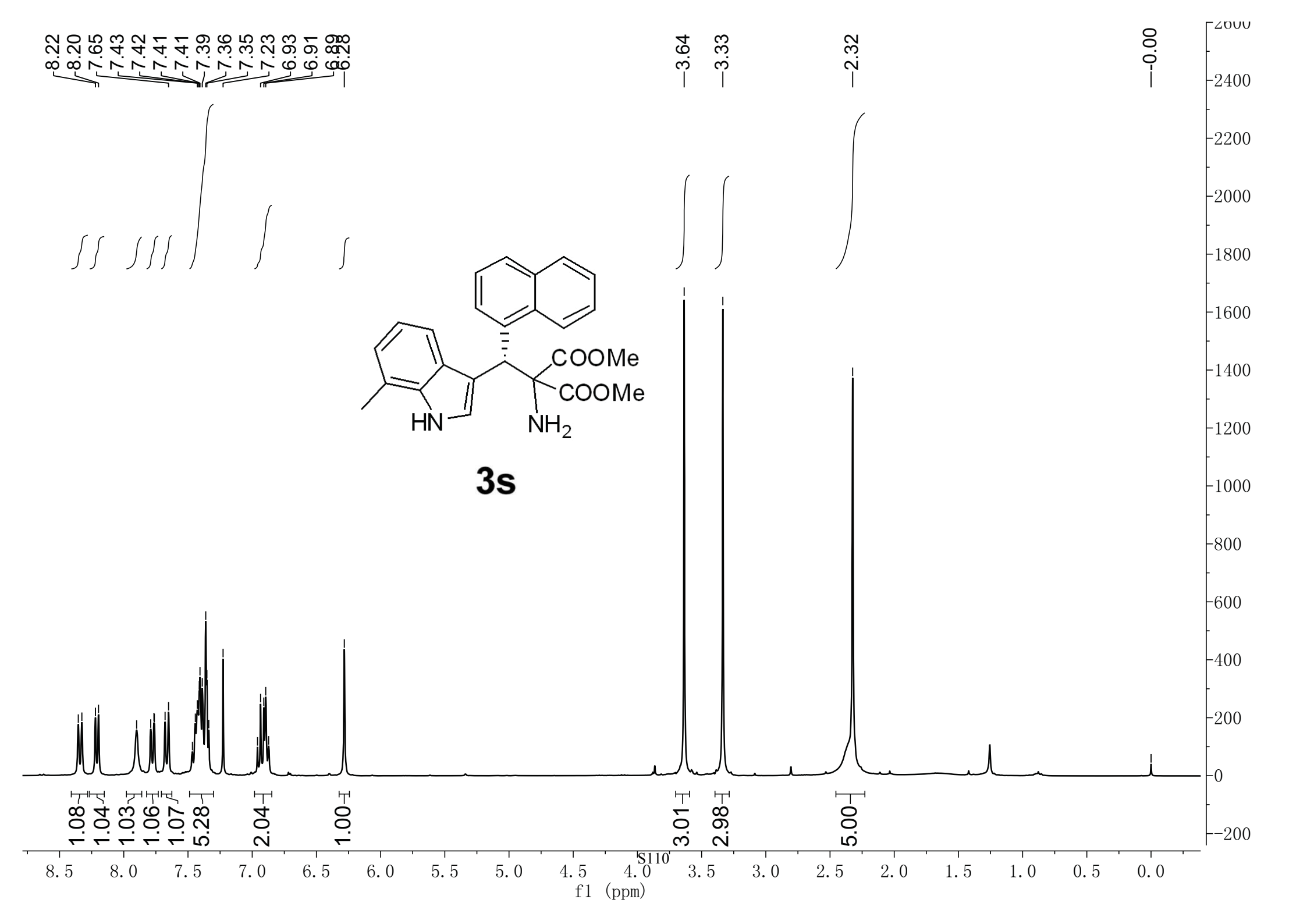

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.906 BB    0.3466 3837.04468  167.98697  50.7017
   2   9.153 BB    0.4885 3730.84058  115.64092  49.2983

Totals :                  7567.88525  283.62789

==========================================================================
                        *** End of Report ***
```

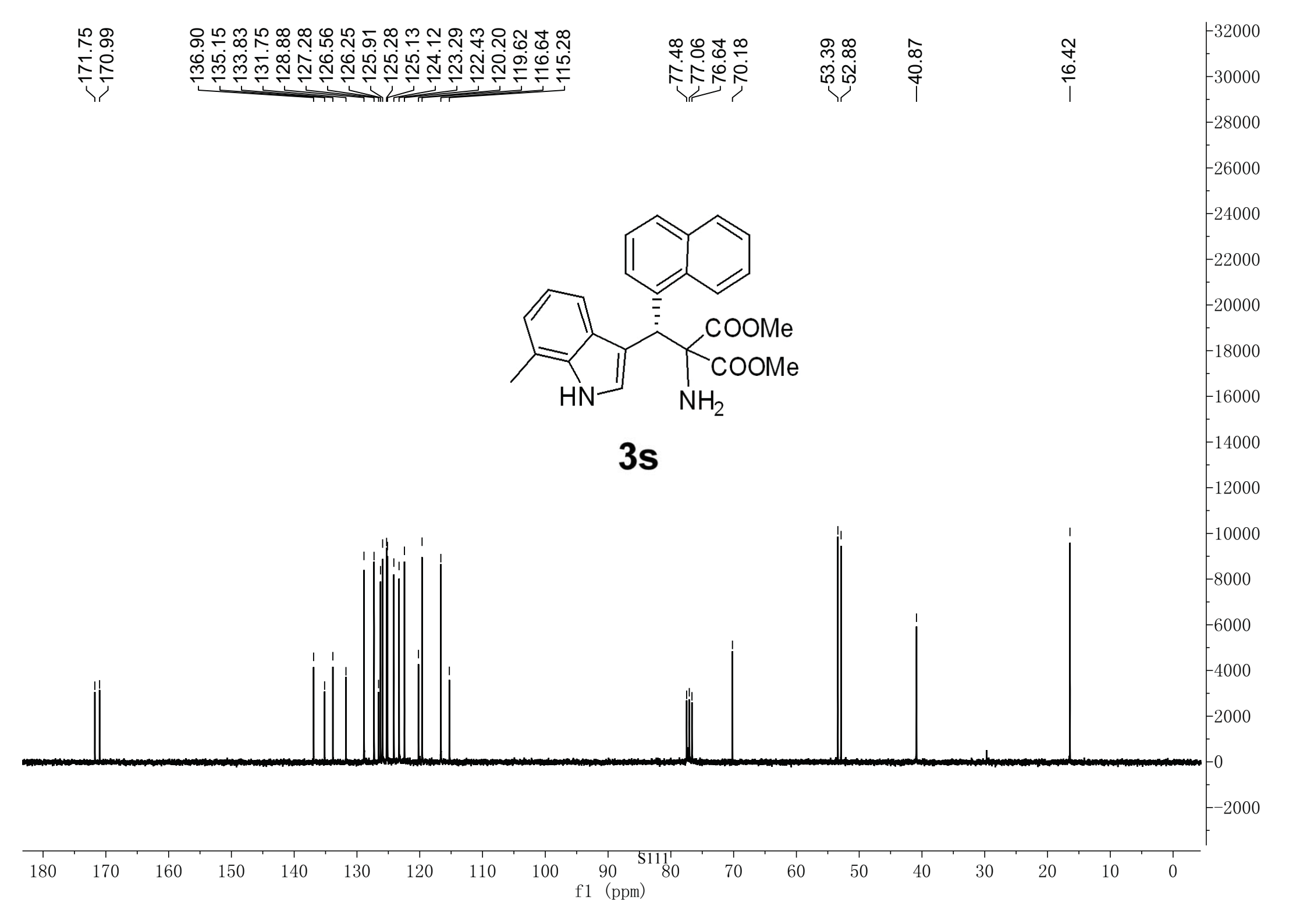
Data File D:\DATA\XUB2013\XUB001029.D
Sample Name: SLL2013-6-27-4

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/30/2013 10:02:47 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 6/30/2013 10:02:30 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:27:32 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



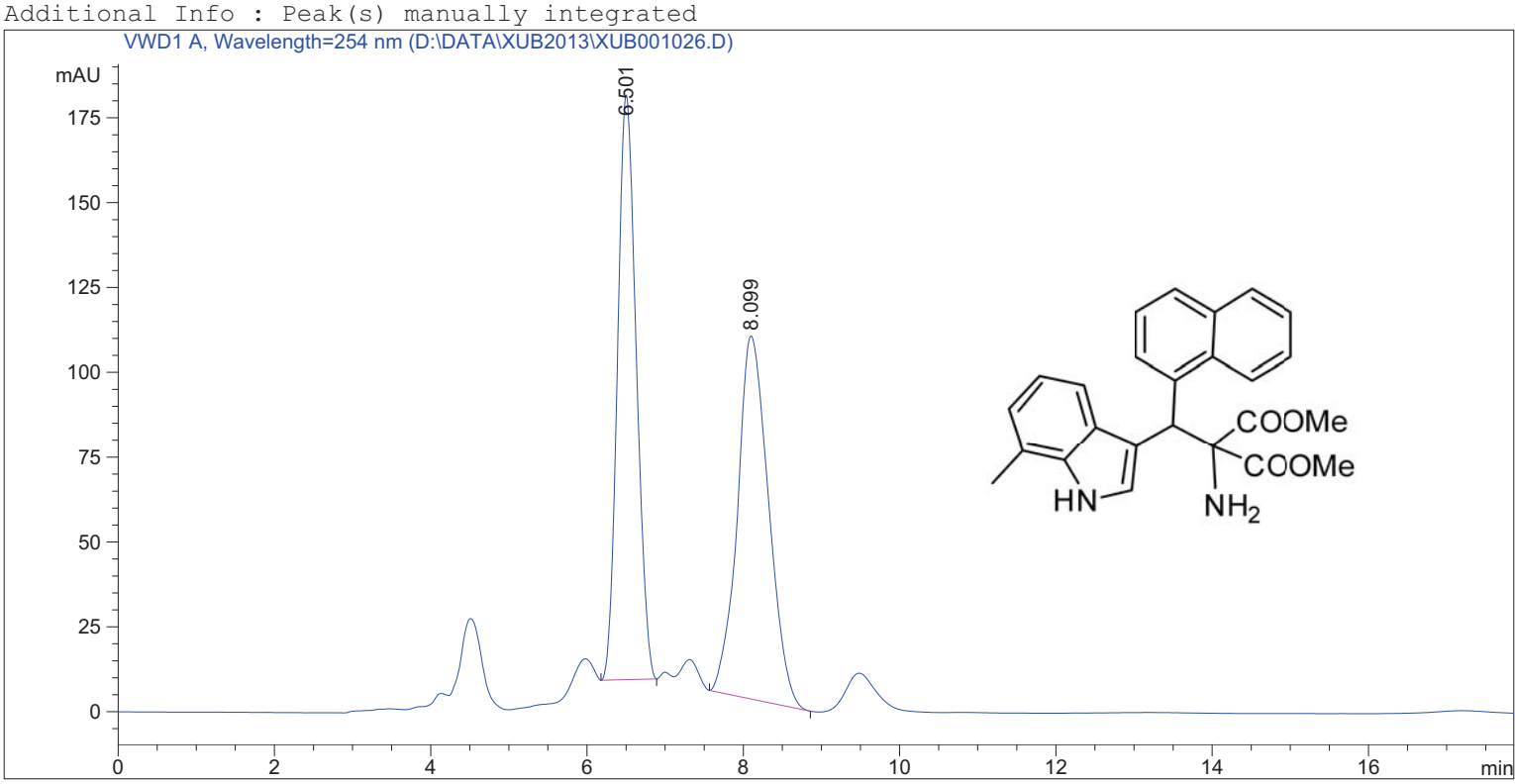

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.898 BV    0.3598 1.46007e4   615.49194  96.8613
   2   9.237 VB    0.4958  473.11789   14.33148   3.1387

Totals :                  1.50738e4   629.82343


==========================================================================
                        *** End of Report ***
```


8.22
8.20
7.65
7.43
7.42
7.41
7.41
7.39
7.36
7.35
7.23
6.93
6.91
6.89
6.88
6.28
3.64
3.33
2.32
0.00
COOMe
COOMe
HN
NH2
3s
1.08
1.04
1.03
1.06
1.07
5.28
2.04
1.00
3.01
2.98
5.00
f1 (ppm)


171.75
170.99
136.90
135.15
133.83
131.75
128.88
127.28
126.56
126.25
125.91
125.28
125.13
124.12
123.29
122.43
120.20
119.62
116.64
115.28
77.48
77.06
76.64
70.18
53.39
52.88
40.87
16.42
COOMe
COOMe
HN
NH2
3s
f1 (ppm)

Data File D:\DATA\XUB2013\XUB001026.D
Sample Name: SLL2013-6-27-3-0

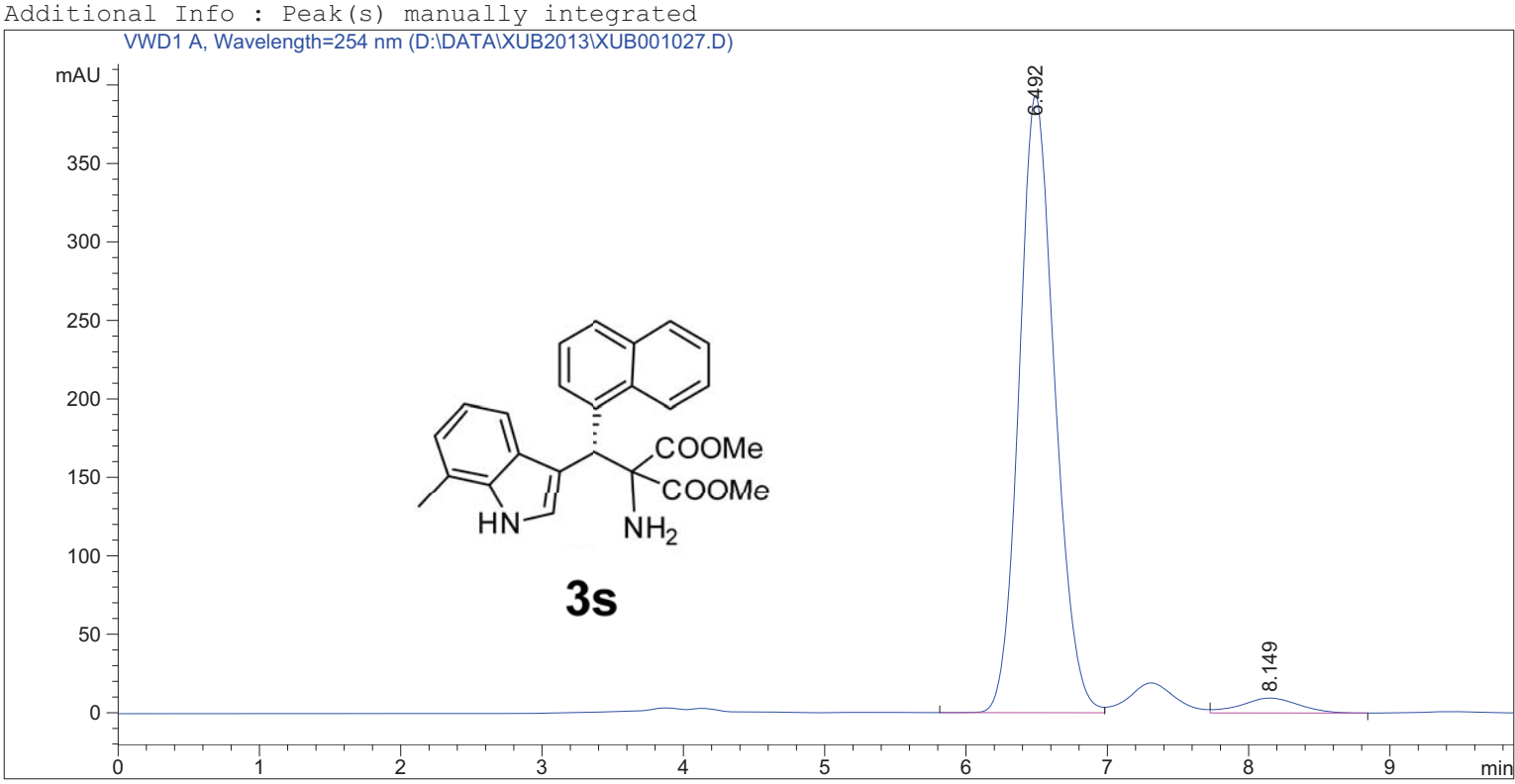
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/30/2013 9:20:33 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 6/30/2013 9:18:53 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:21:28 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



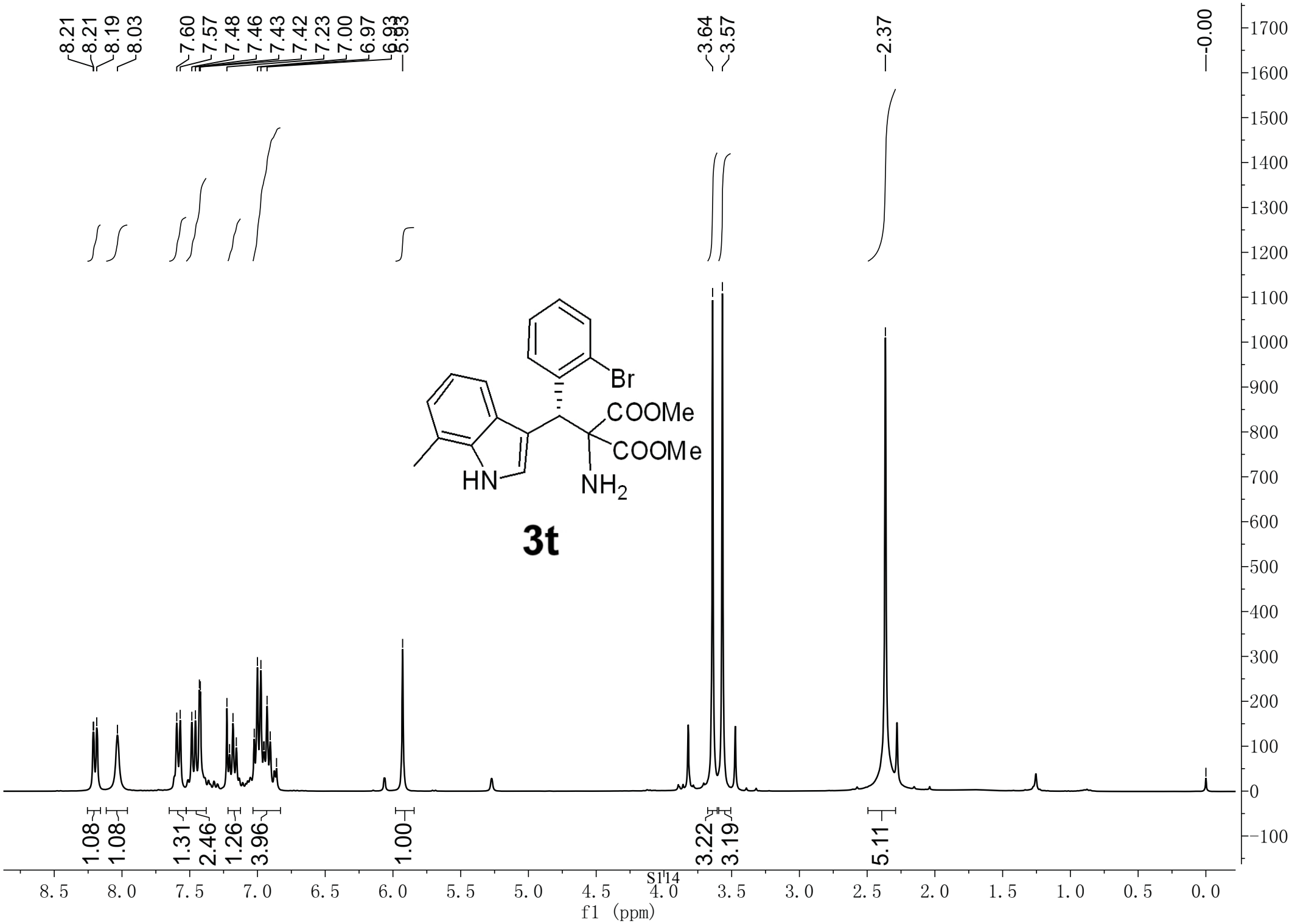

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.501 BB    0.2568 2867.11011  172.28059  49.1922
   2   8.099 BB    0.4234 2961.27686  106.98239  50.8078

Totals :                  5828.38696  279.26299


==========================================================================
                          *** End of Report ***
```

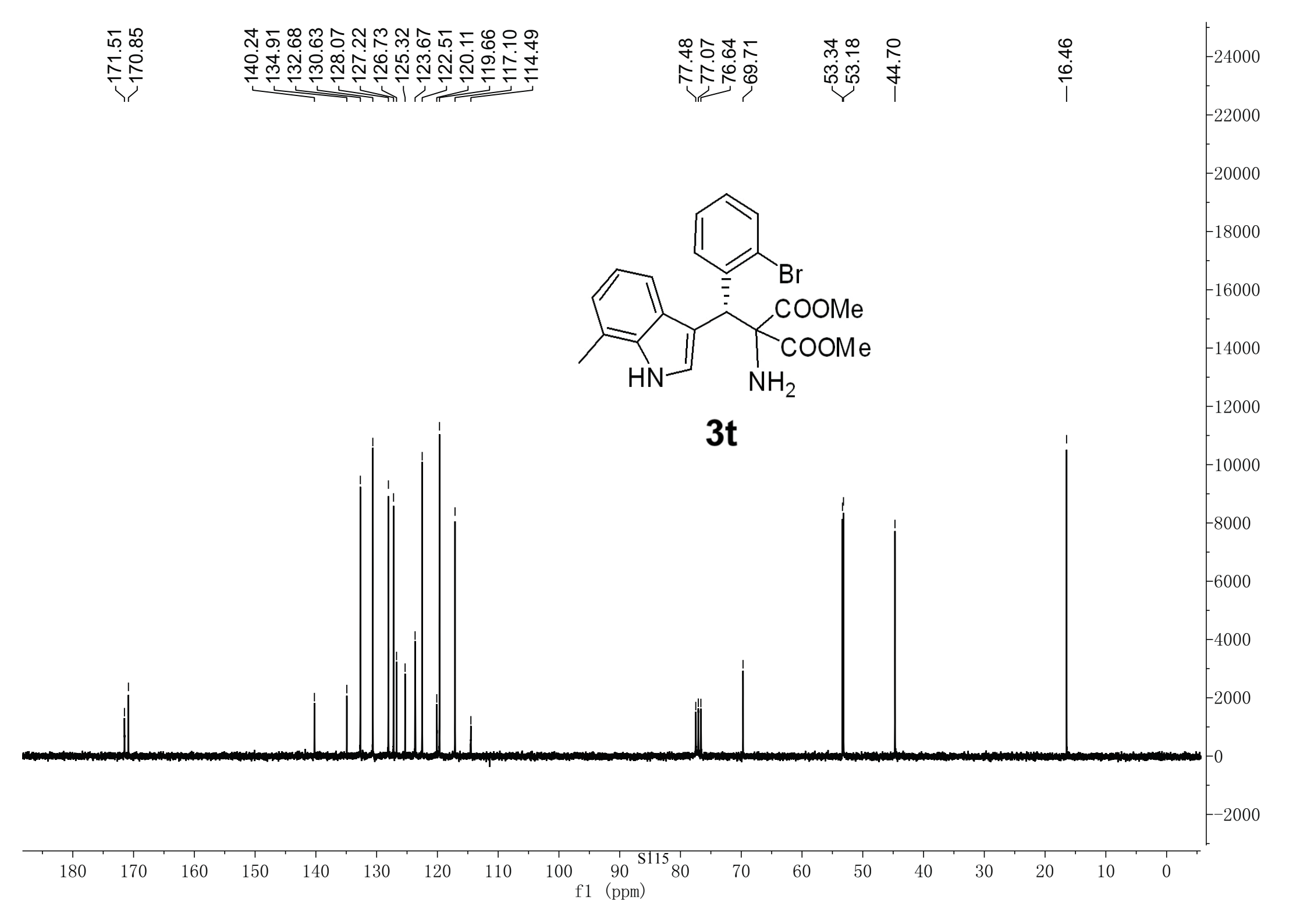
Data File D:\DATA\XUB2013\XUB001027.D
Sample Name: SLL2013-6-27-3

```
================================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 6/30/2013 9:41:05 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 6/30/2013 9:38:28 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 9:27:32 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



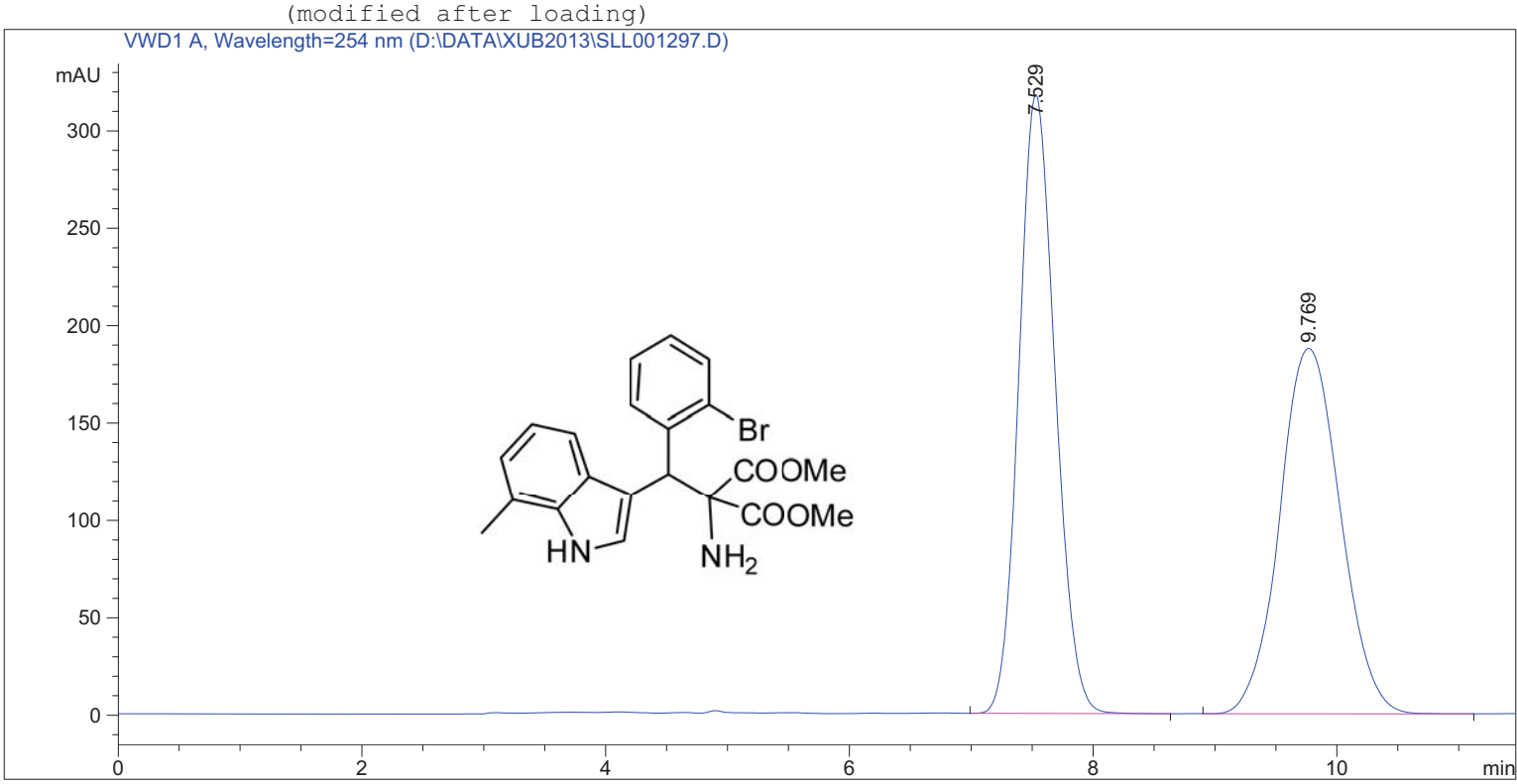

```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.492 BV    0.2657 6901.94873  393.62778  96.2468
   2   8.149 VB    0.4267  269.14395    9.49700   3.7532

Totals :                  7171.09268  403.12478

================================================================================
                          *** End of Report ***
```


8.21
8.21
8.19
8.03
7.60
7.57
7.48
7.46
7.43
7.42
7.23
7.00
6.97
6.93
5.93
3.64
3.57
2.37
0.00
Br
COOMe
COOMe
HN
NH2
3t
1.08
1.08
1.31
2.46
1.26
3.96
1.00
3.22
3.19
5.11
f1 (ppm)


171.51
170.85
140.24
134.91
132.68
130.63
128.07
127.22
126.73
125.32
123.67
122.51
120.11
119.66
117.10
114.49
77.48
77.07
76.64
69.71
53.34
53.18
44.70
16.46
Br
COOMe
COOMe
HN
NH2
3t
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001297.D
Sample Name: SLL2013-7-22-1-0

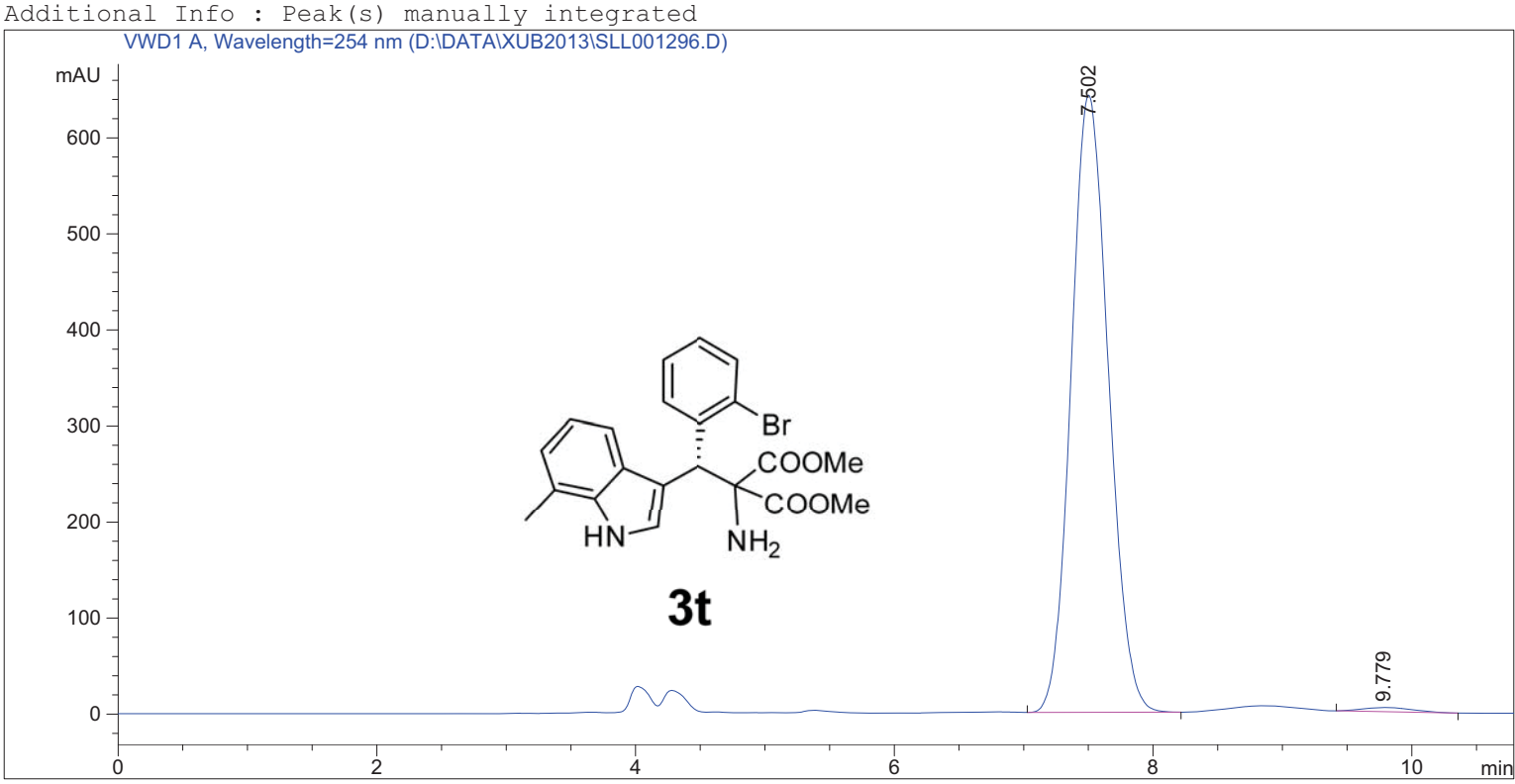
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 10/5/2013 4:36:23 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 10/5/2013 4:32:30 PM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/8/2013 8:55:31 AM by LNF
                  (modified after loading)
```



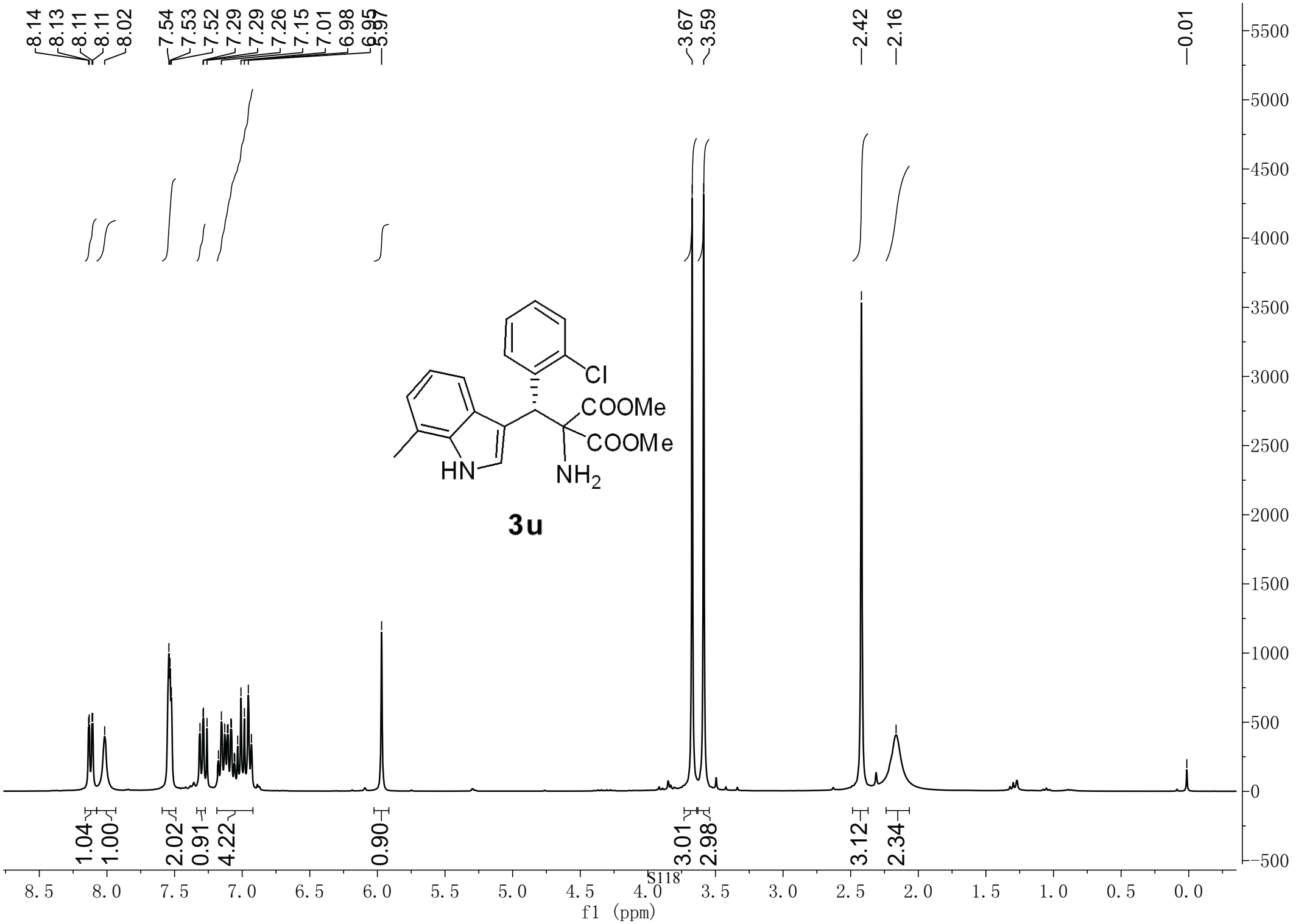

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.529 BB    0.3210 6558.94775  317.65265  50.3293
   2   9.769 BB    0.5396 6473.12451  187.54112  49.6707

Totals :                  1.30321e4   505.19377

==========================================================================
                          *** End of Report ***
```

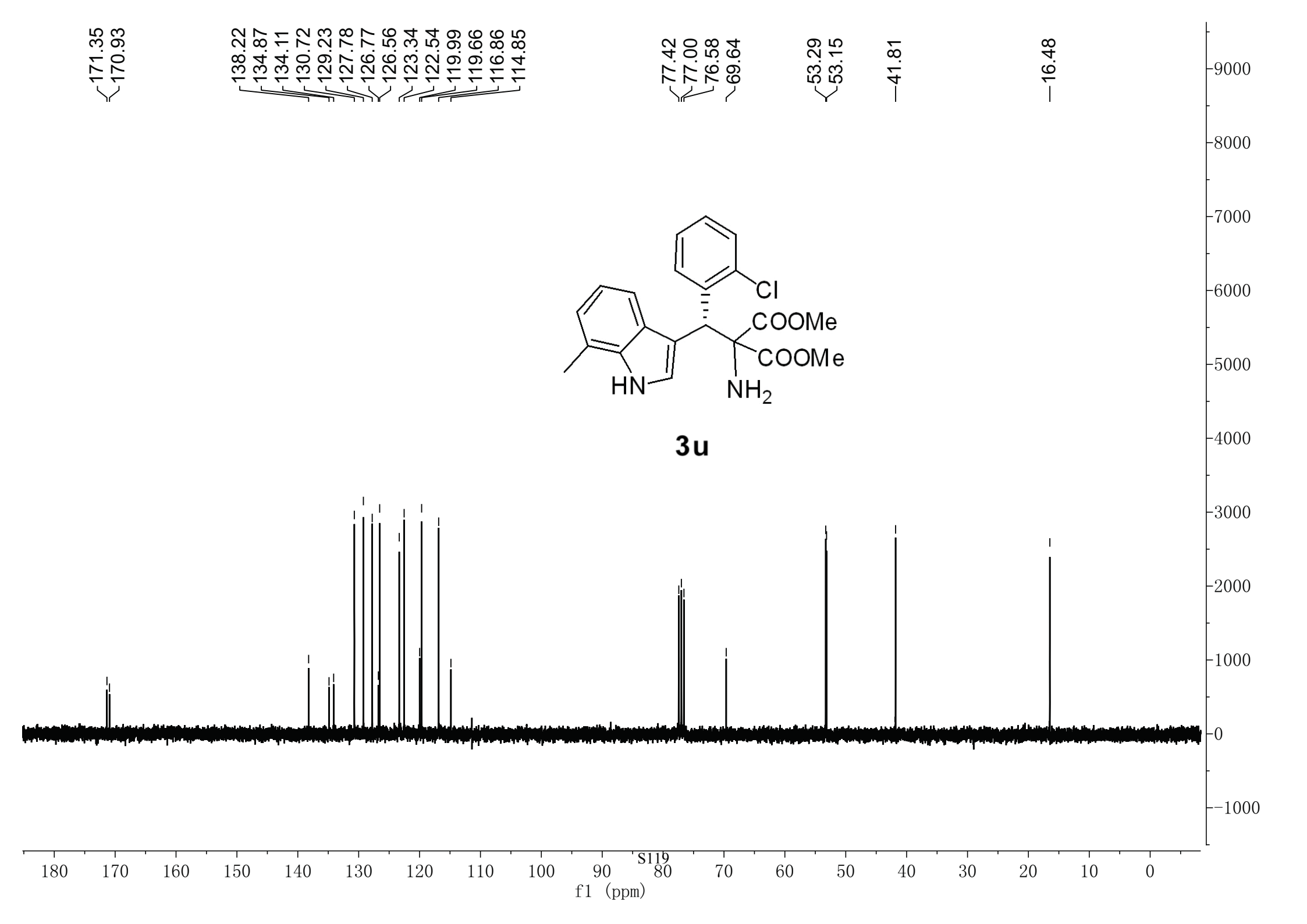
Data File D:\DATA\XUB2013\SLL001296.D
Sample Name: SLL2013-10-1-1

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 10/5/2013 4:21:38 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 10/5/2013 4:18:06 PM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/8/2013 8:55:31 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



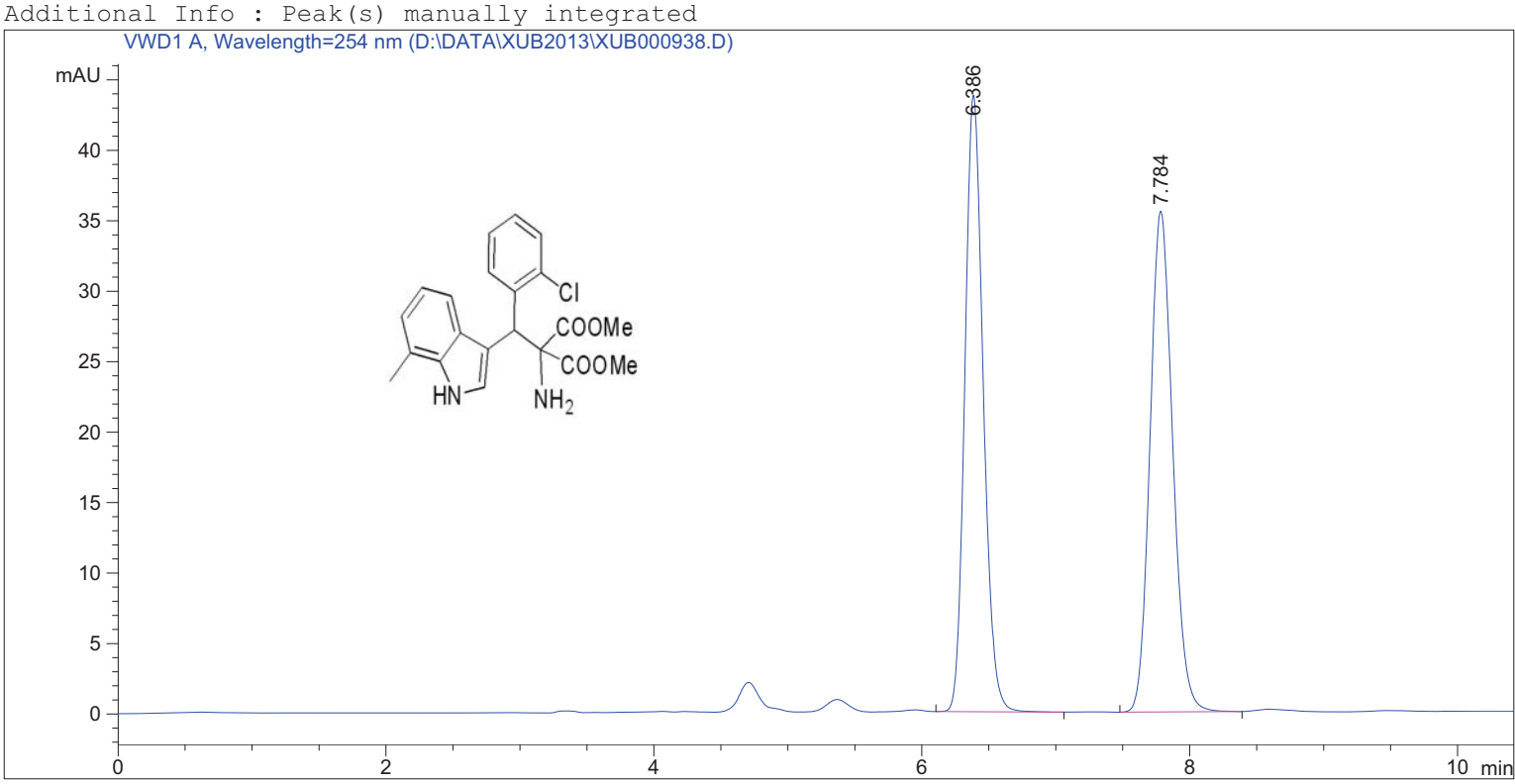

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.502 BB    0.3113 1.28967e4   642.57489  99.1152
   2   9.779 BB    0.4307  115.12862    4.29392   0.8848

Totals :                  1.30119e4   646.86881

==========================================================================
                        *** End of Report ***
```


8.14
8.13
8.11
8.11
8.02
7.54
7.53
7.52
7.29
7.29
7.26
7.15
7.01
6.98
6.95
5.97
3.67
3.59
2.42
2.16
0.01
Cl
COOMe
COOMe
HN
NH2
3u
1.04
1.00
2.02
0.91
4.22
0.90
3.01
2.98
3.12
2.34
f1 (ppm)


171.35
170.93
138.22
134.87
134.11
130.72
129.23
127.78
126.77
126.56
123.34
122.54
119.99
119.66
116.86
114.85
77.42
77.00
76.58
69.64
53.29
53.15
41.81
16.48
Cl
COOMe
COOMe
HN
NH2
3u
f1 (ppm)

Data File D:\DATA\XUB2013\XUB000938.D
Sample Name: XUB2013-5-28-0

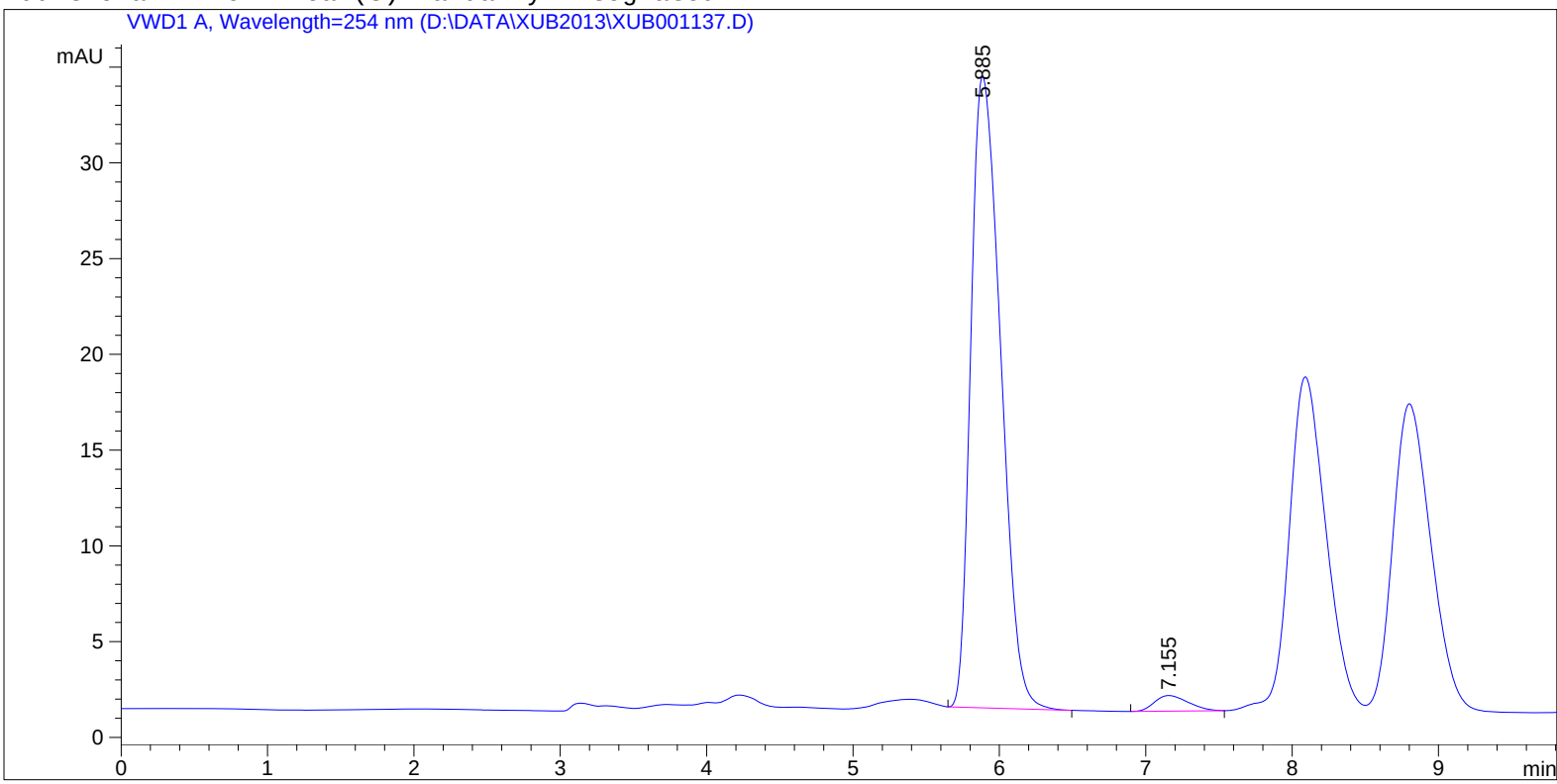
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 5/30/2013 4:32:26 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 5/30/2013 4:23:30 PM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:15:04 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



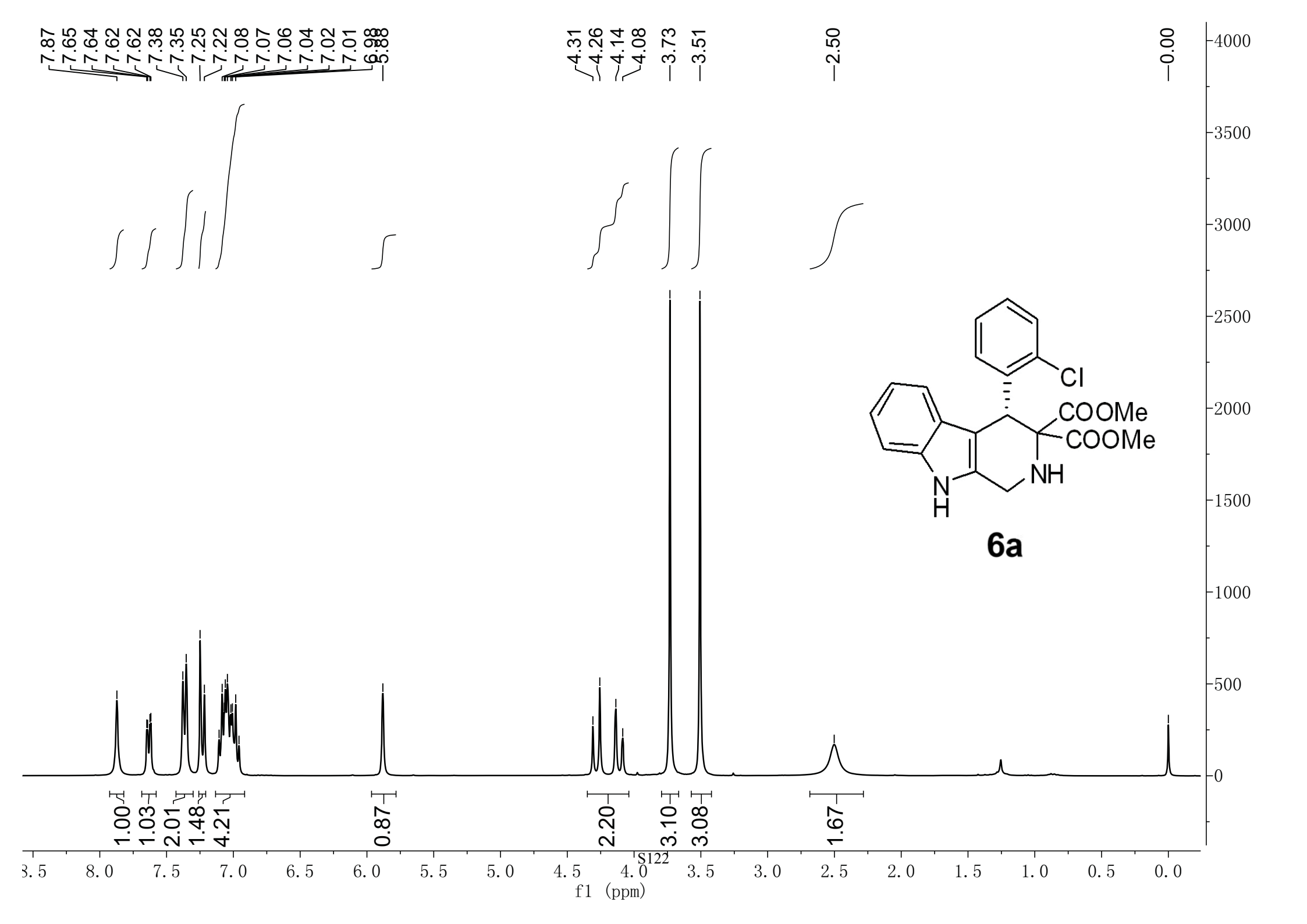

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.386 BB    0.1459  416.28671   43.76836  49.9183
   2   7.784 BB    0.1822  417.64859   35.54234  50.0817

Totals :                   833.93530   79.31070


==========================================================================
                        *** End of Report ***
```

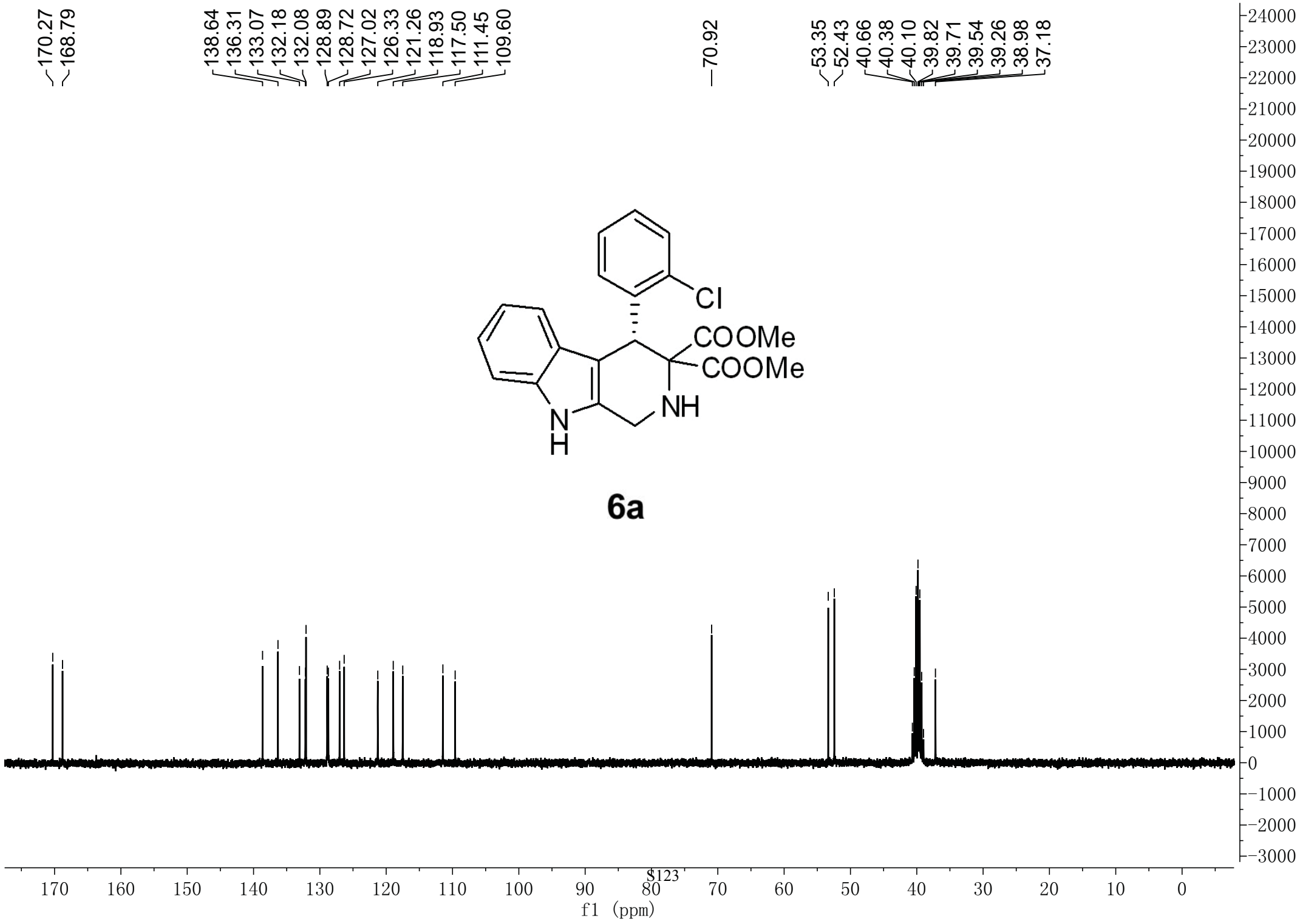
Data File D:\DATA\XUB2013\XUB001137.D
Sample Name: XUB2013-7-31-4

```
==========================================================================
Acq. Operator   : xub
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 8/1/2013 11:06:08 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 4/12/2013 1:11:20 PM by LNF
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 9/29/2013 11:16:31 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



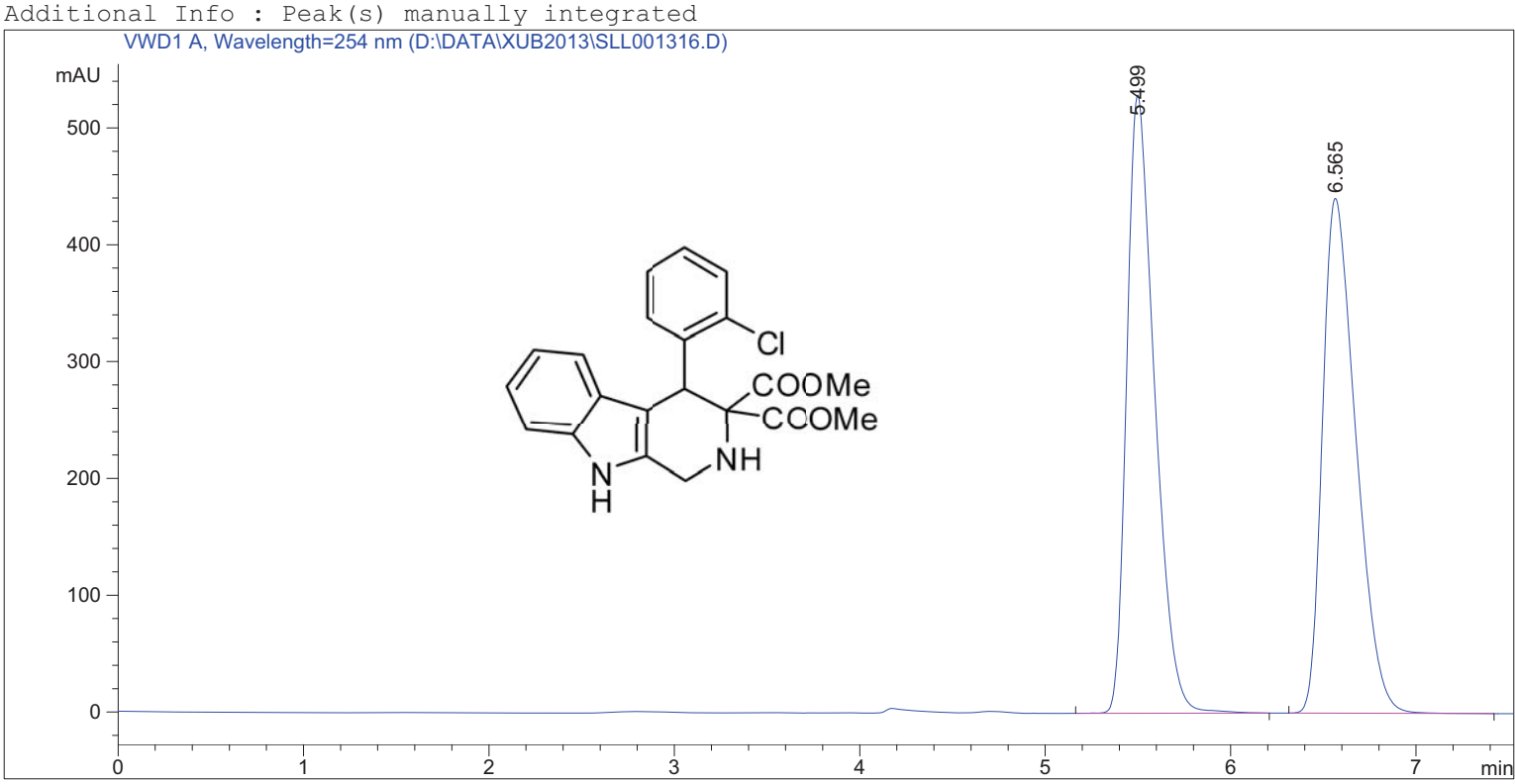

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.885 BB    0.2279  467.01678   32.96791  97.2880
   2   7.155 BB    0.2493   13.01832 8.13524e-1   2.7120

Totals :                   480.03511   33.78144


==========================================================================
                         *** End of Report ***
```


7.87
7.65
7.64
7.62
7.62
7.38
7.35
7.25
7.22
7.08
7.07
7.06
7.04
7.02
7.01
6.98
5.88
4.31
4.26
4.14
4.08
3.73
3.51
2.50
0.00
Cl
COOMe
COOMe
NH
N
H
6a
1.00
1.03
2.01
1.48
4.21
0.87
2.20
3.10
3.08
1.67
f1 (ppm)


170.27
168.79
138.64
136.31
133.07
132.18
132.08
128.89
128.72
127.02
126.33
121.26
118.93
117.50
111.45
109.60
70.92
53.35
52.43
40.66
40.38
40.10
39.82
39.71
39.54
39.26
38.98
37.18
Cl
COOMe
COOMe
NH
N
H
6a
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001316.D
Sample Name: SLL2013-9-25-3-0

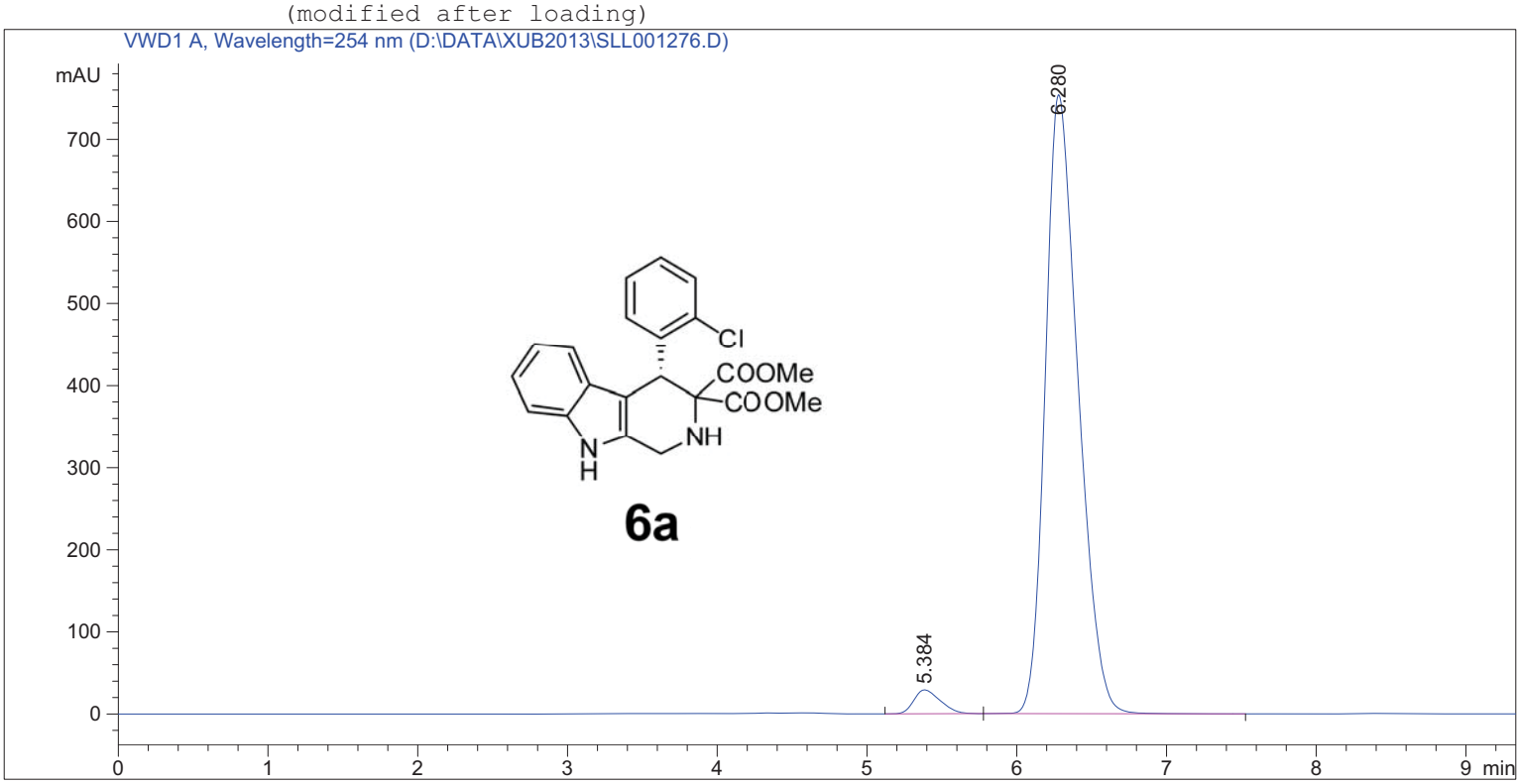
```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 10/8/2013 9:36:16 AM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 10/8/2013 9:35:30 AM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/8/2013 9:24:42 AM by LNF
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



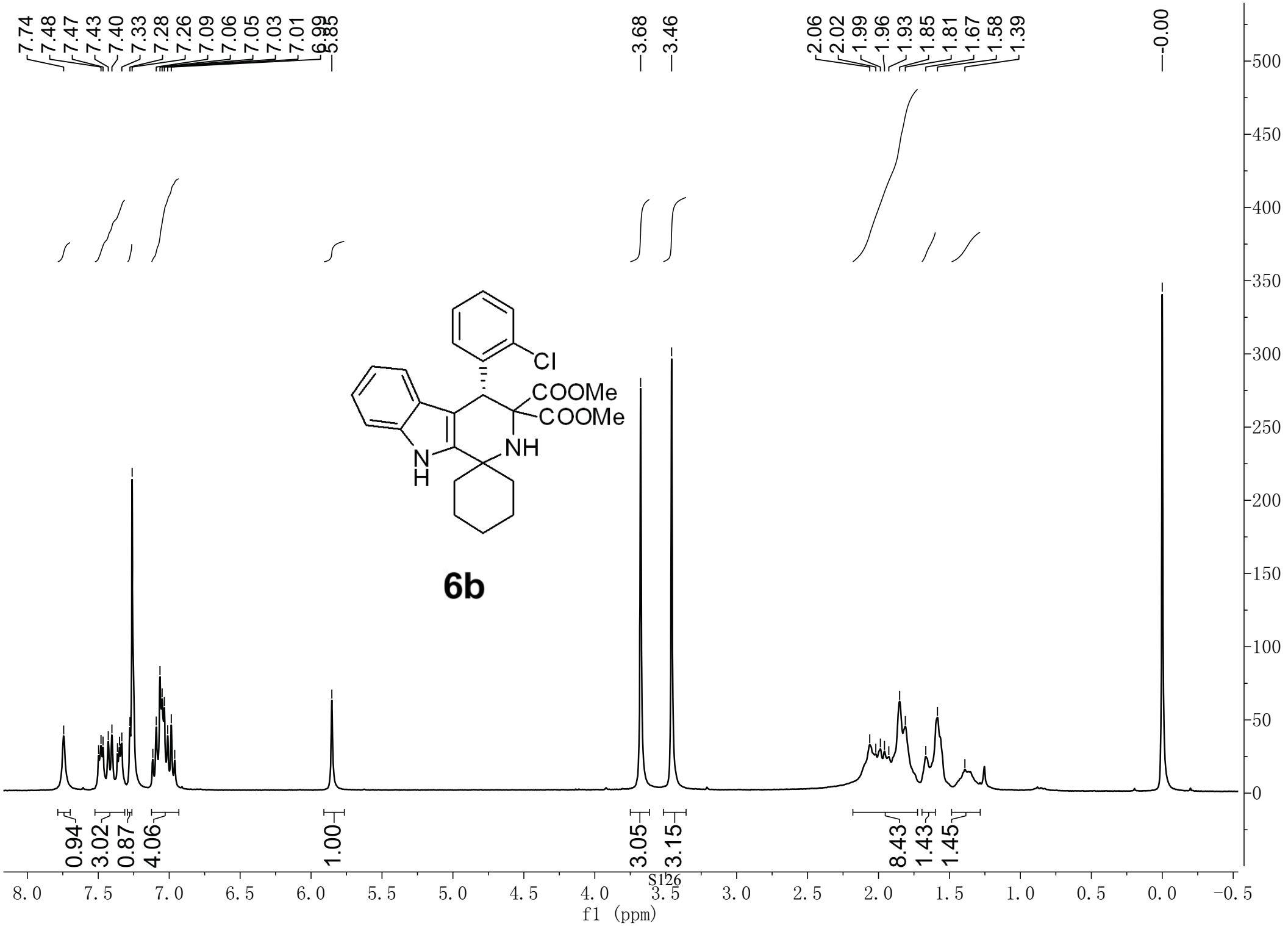

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.499 BB    0.1566 5518.29883  529.05353  50.0694
   2   6.565 BBA   0.1923 5502.99658  440.70435  49.9306

Totals :                  1.10213e4   969.75787


==========================================================================
                        *** End of Report ***
```

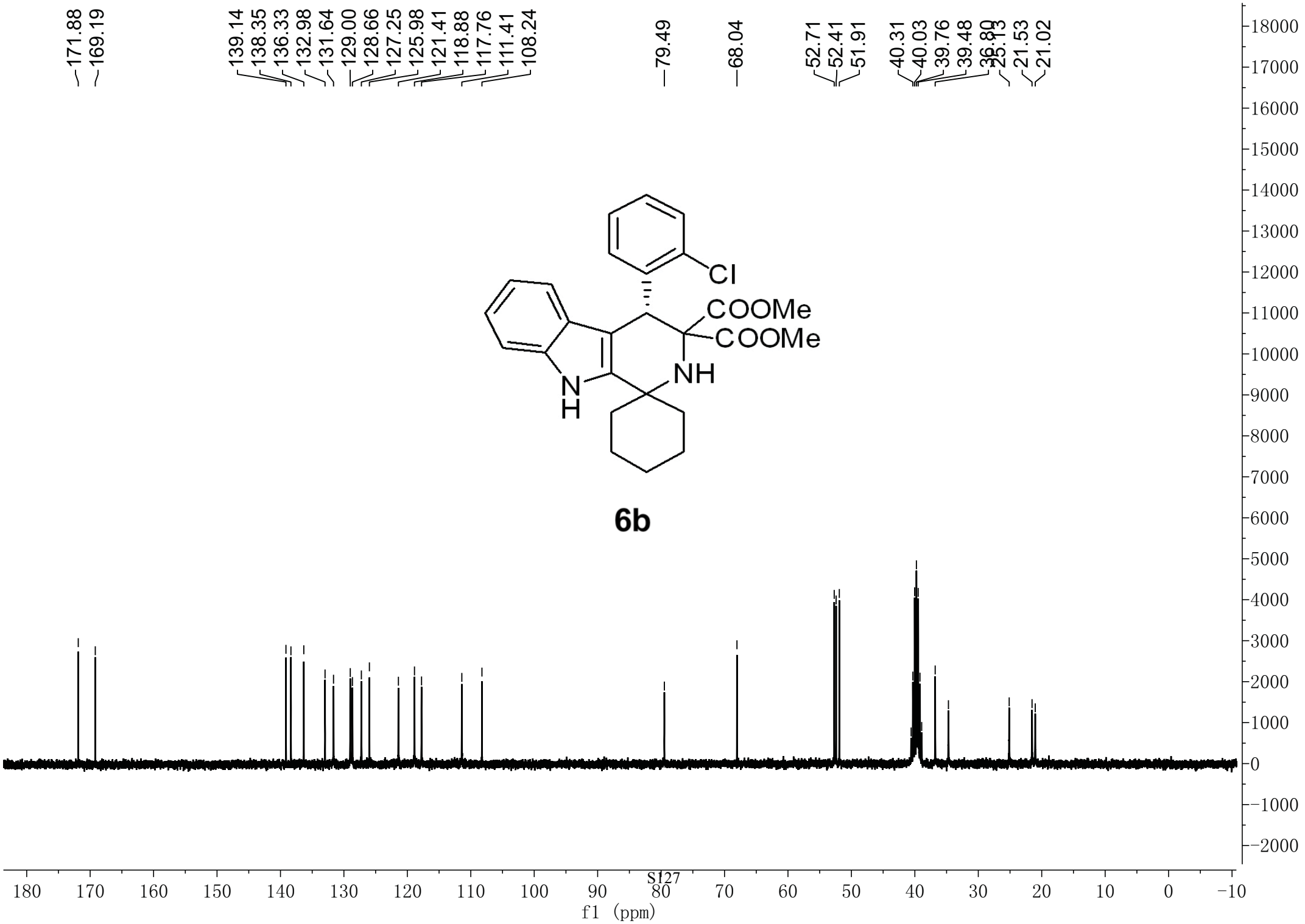
Data File D:\DATA\XUB2013\SLL001276.D
Sample Name: SLL2013-9-27-1

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 9/28/2013 12:03:33 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 9/28/2013 12:11:01 PM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/8/2013 9:06:59 AM by LNF
                  (modified after loading)
```



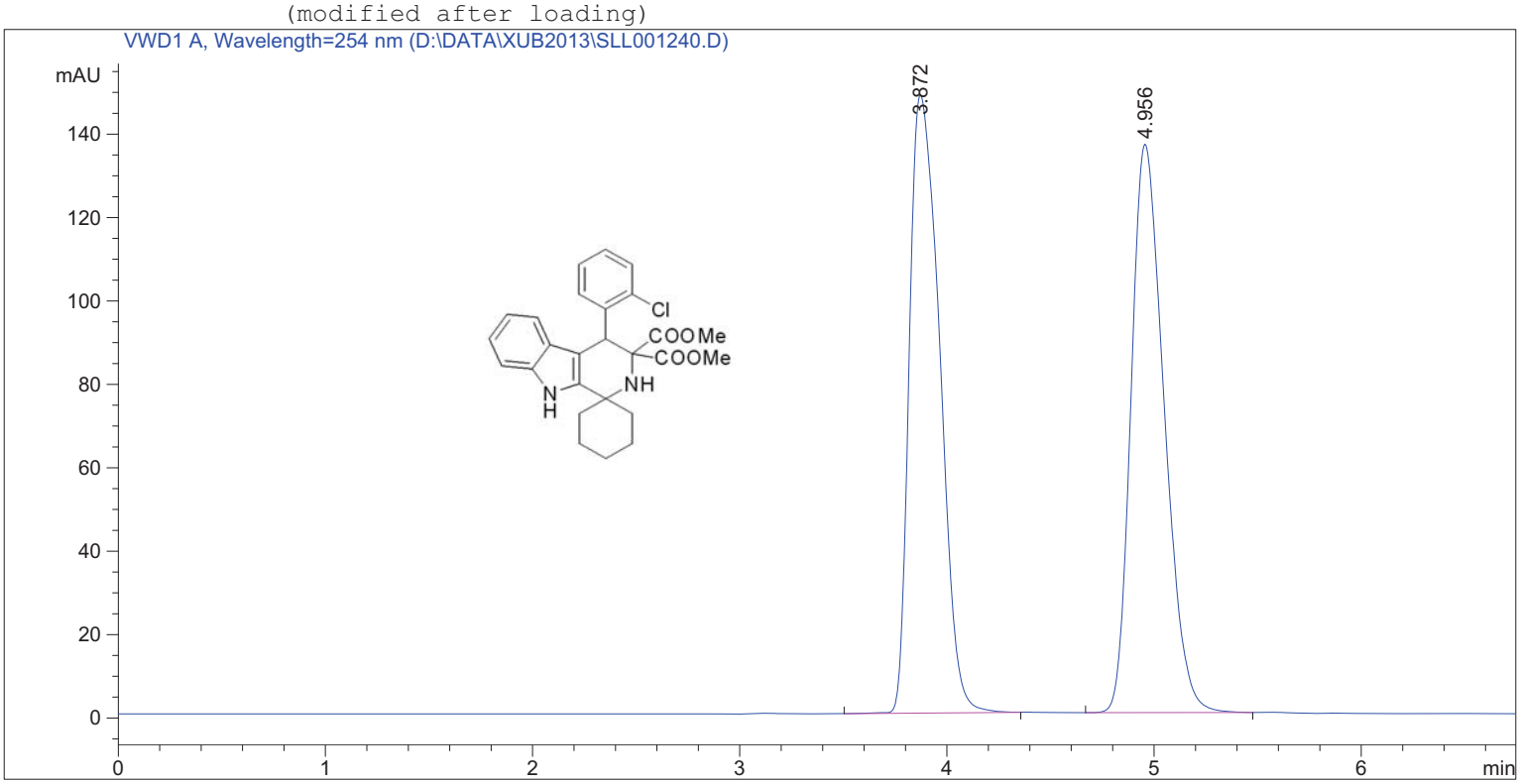

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.384 BB    0.1880  357.64783   29.19826   3.0291
   2   6.280 BB    0.2310 1.14495e4   754.04010  96.9709

Totals :                  1.18071e4   783.23836

==========================================================================
                        *** End of Report ***
```


7.74
7.48
7.47
7.43
7.40
7.33
7.28
7.26
7.09
7.06
7.05
7.03
7.01
6.99
5.85
3.68
3.46
2.06
2.02
1.99
1.96
1.93
1.85
1.81
1.67
1.58
1.39
0.00
Cl
COOMe
COOMe
NH
N
H
6b
0.94
3.02
0.87
4.06
1.00
3.05
3.15
8.43
1.43
1.45
f1 (ppm)


171.88
169.19
139.14
138.35
136.33
132.98
131.64
129.00
128.66
127.25
125.98
121.41
118.88
117.76
111.41
108.24
79.49
68.04
52.71
52.41
51.91
40.31
40.03
39.76
39.48
36.80
25.13
21.53
21.02
Cl
COOMe
COOMe
NH
N
H
6b
f1 (ppm)

Data File D:\DATA\XUB2013\SLL001240.D
Sample Name: XUB2013-8-20-0

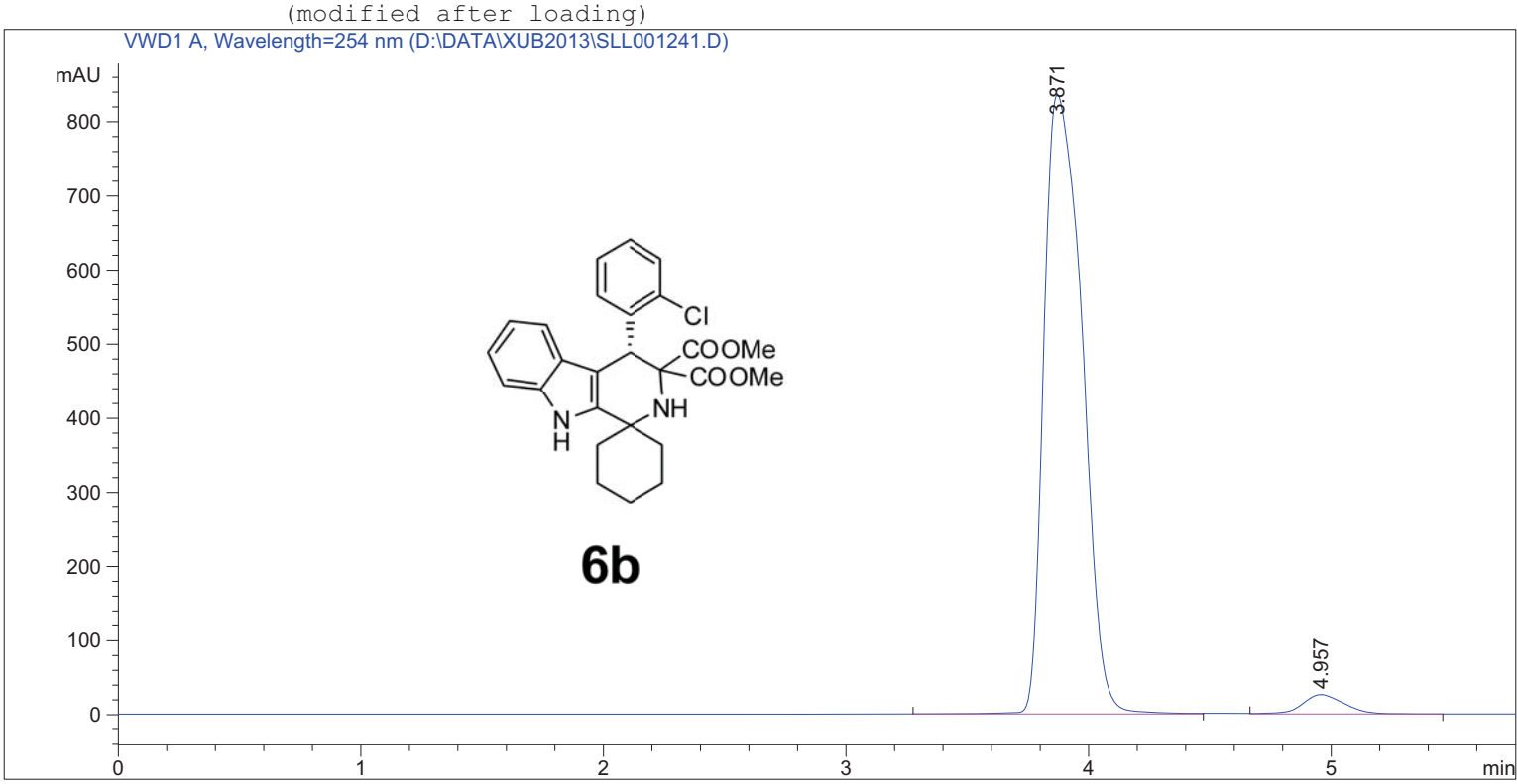
```
================================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 9/22/2013 4:41:07 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 9/22/2013 4:40:29 PM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/8/2013 8:57:56 AM by LNF
                  (modified after loading)
```




```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.872 BB    0.1696 1508.27576  148.28589  49.9706
   2   4.956 BB    0.1715 1510.05188  136.30138  50.0294

Totals :                  3018.32764  284.58727

================================================================================
                          *** End of Report ***
```

Data File D:\DATA\XUB2013\SLL001241.D
Sample Name: SLL2013-9-16-2

```
==========================================================================
Acq. Operator   : LNF
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 9/22/2013 4:49:45 PM
                                                  Inj Volume : No inj
Acq. Method     : D:\METHOD\AD_30_1.M
Last changed    : 9/22/2013 4:47:56 PM by LNF
                  (modified after loading)
Analysis Method : D:\METHOD\AD_30_1.M
Last changed    : 10/8/2013 9:05:14 AM by LNF
                  (modified after loading)
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.871 BV    0.1814 9138.14063  835.73248  96.7052
   2   4.957 VB    0.1845  311.34055   26.07045   3.2948

Totals :                  9449.48117  861.80293

==========================================================================
                        *** End of Report ***
```