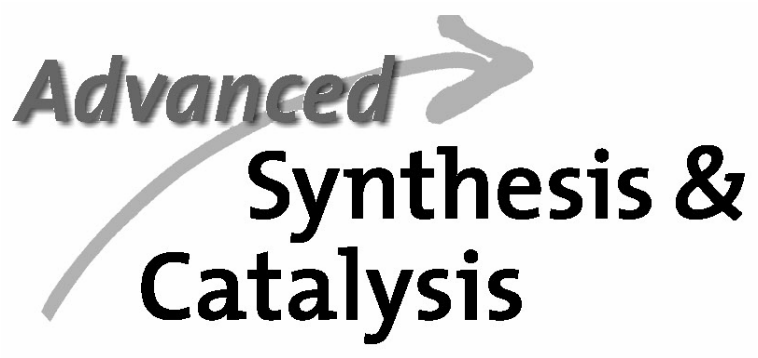

Supporting Information

Palladium(II)-Catalyzed One-Pot Enantioselective Synthesis of Arylglycine Derivatives from Ethyl Glyoxylate, *p*-Toluenesulfonyl Isocyanate and Arylboronic Acids

Huixiong Dai, Miao Yang and Xiyan Lu*

State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Lu, Shanghai 200032, China

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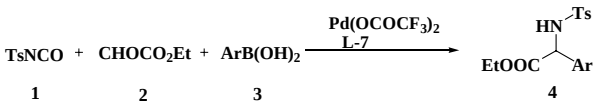
I. General.

Palladium acetate and Palladium trifluroacetate was purchased from Aldrich. Ethyl glyoxylate (50% solution in toluene) was purchased from Lancaster. Phenylboronic acid was purchased from BeiJing Wisdom Chemicals Co., Ltd. All other arylboronic acids were purchased from HeBei DeLongTai Chemicals Co., Ltd. All solvents were dried and distilled before use according to the standard methods. The chiral nitrogen-containing ligands were prepared according to the published procedures.[1] NMR spectra were recorded on a Varian Mercury V_300 spectrometer. Infrared spectra were obtained on a Bio-Rad FTS-185 machine. Mass spectra were provided on Agilent 5973 or Agilent 1100 machines. All melting points were uncorrected. The optical rotation was measured on a Perkin-Elmer 341 polarimeter. The enantiomeric excess values were determined after separation of the enantiomers by HPLC on a Perkin-Elmer (785A, 200 IC Pump) or Waters (515 Pump, 2487. Dual Absorbance Detector) instruments.

II. Typical Procedure for Palladium(II)-Catalyzed One-pot Synthesis of Chiral Arylglycine Derivatives from Ethyl Glyoxylate, *p*-Toluenesulfonyl Isocyanate and Arylboronic Acids

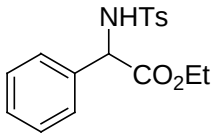


To a Schlenk tube were added *p*-toluenesulphonylisocyanate (**1**) (0.355 mmol),

ethylglyoxylate (**2**) (0.355 mmol), $Pd(CF_3CO_2)_2$ (5 mol %), L-**7** (6 mol %) in refluxing toluene (2 mL) for 3 h, then phenylboronic acid (**3a**) (0.71 mmol) was added for another 24 h. The reaction mixture was allowed to cool to room temperature and diluted with EtOAc. The organic layer was washed with brine and the aqueous layer was extracted with EtOAc. The combined organic layer was dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was subjected to silica gel chromatography (eluent: EtOAc/ petroleum ether) to isolate the product.

III. Characterization Data of Compounds **4**.

***N*-tosyl-3-phenylglycine ethyl ester.**[2]

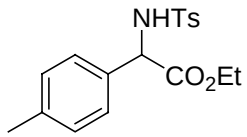


4a

White solid, mp: 90-91 °C. (lit[2]: 85-86 °C (racemic form)); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.63 (d, *J* =8.4 Hz, 2H), 7.18-7.28 (m, 7H), 5.66 (d, *J* = 6.9 Hz, 1H), 5.03 (d, *J* = 7.5 Hz, 1H), 3.96-4.07 (m, 2H), 2.38 (s, 3H), 1.10 (t, *J* = 6.9 Hz, 3H); IR (KBr): ν 3272, 2993, 1716, 1459, 1336, 1164, 1090, 818 cm^{-1}; MS (70 eV, EI) *m/z*: 260 (M^+ - CO_2Et, 100), 155, 91, 77, 65.

The ee value was determined by chiral HPLC using a Chiralcel OJ-H column with hexane : isopropanol = 80 : 20, flow = 0.7 mL/min; ee: 75%; $[\alpha]_D^{20}$ = +48.6 (*c* 1.05, $CHCl_3$).

***N*-tosyl-4-tolylglycine ethyl ester.**

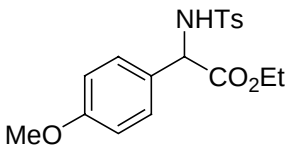


4b

White solid, mp: 87-89 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.64 (d, *J* = 7.8 Hz, 2H), 7.18 (d, *J* = 7.8 Hz, 2H), 7.11 (d, *J* = 8.1 Hz, 2H), 7.04 (d, *J* = 8.1 Hz, 2H), 5.90 (d, *J* = 9.0 Hz, 1H), 4.99 (d, *J* = 8.4 Hz, 1H), 3.93-4.01 (m, 2H), 2.37 (s, 3H), 2.28 (s, 3H), 1.07 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.1, 143.3, 138.2, 136.8, 132.3, 129.3, 129.2, 127.1, 126.8, 61.9, 59.0, 21.4, 20.9; IR (KBr): ν 3274, 2980, 1734, 1339, 1167, 1076 cm^{-1}; MS (70 eV, EI) *m/z*: 274 (M^+ - CO_2Et, 100), 201, 155, 118, 91, 77. 65; HRMS Calcd for $C_{15}H_{16}NO_2S$ (M^+ - CO_2Et): 274.0902. Found: 274.0906.

The ee value was determined by chiral HPLC using a Chiralcel OJ-H column with hexane : isopropanol = 80 : 20, flow = 0.7 mL/min; ee: 70%; $[\alpha]_D^{20}$ = +71.1 (*c* 1.01, $CHCl_3$).

***N*-tosyl-4-methoxyphenylglycine ethyl ester.**



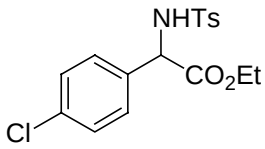
4c

White solid, mp: 87-89^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.63 (d, *J* = 8.1 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 2H), 7.13 (d, *J* = 8.7 Hz, 2H), 6.76 (d, *J* = 8.4 Hz, 2H), 5.80 (d, *J* = 8.1 Hz, 1H), 4.98 (d, *J* = 8.1 Hz, 1H), 3.97-4.00 (m, 2H), 3.76 (s, 3H), 2.38 (s, 3H),

1.09 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.2, 159.6, 143.3, 136.9, 129.4, 128.3, 127.4, 127.1, 114.0, 62.0, 58.8, 55.2, 21.4, 13.8; IR (KBr): ν 3282, 2972, 1741, 1612, 1514, 1338, 1160, 1087, 1025, 815, 667 cm^{-1}; MS (70 eV, EI) m/z (%): 363 (M^+), 290(M^+ - CO_2Et), 173, 155, 134, 105, 91(100), 77, 65, 43; HRMS Calcd for $C_{15}H_{16}NO_3S$ (M^+ - CO_2Et): 290.0851. Found: 290.0847.

The ee value was determined by chiral HPLC using a Chiralcel OJ-H column with hexane : isopropanol = 80 : 20, flow = 0.7 mL/min; ee: 60%; $[\alpha]_D^{20}$ = +58.1 (*c* 0.51, $CHCl_3$).

***N*-tosyl-4-chlorophenylglycine ethyl ester.**



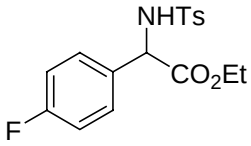
4d

White solid, mp: 86-87°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.60 (d, *J* = 8.1 Hz, 2H), 7.14-7.27 (m, 6H), 5.76 (d, *J* = 7.5 Hz, 1H), 5.01 (d, *J* = 7.5 Hz, 1H), 3.96-4.09 (m, 2H), 2.39 (s, 3H), 1.10 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.6, 143.7, 136.7, 134.5, 133.9, 129.5, 128.8, 128.5, 127.1, 62.5, 58.7, 21.5, 13.8; IR (KBr): ν 3270, 2989, 1717, 1597, 1493, 1343, 1262, 1168, 1092, 920, 698 cm^{-1}; MS (70 eV, EI) *m/z*: 296, 294 (M^+ - CO_2Et), 274, 155, 138, 91 (100), 77, 65, 41; Anal. Cald for $C_{17}H_{18}ClNO_4S$: C, 55.51; H, 4.93; N, 3.81. Found: C, 55.66; H, 4.98; N, 3.60.

The ee value was determined by chiral HPLC using a Chiralcel OJ-H column with hexane : isopropanol = 90 : 10, flow = 0.7 mL/min; ee: 81%; $[\alpha]_D^{20}$ = +87.9 (*c* 1.03,

$CHCl_3$).

***N*-tosyl-4-fluorophenylglycine ethyl ester.**

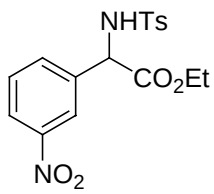


4e

White solid, mp: 80-81°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.63 (d, *J* = 8.1 Hz, 2H), 7.18-7.24 (m, 4H), 6.92 (t, *J* = 8.7 Hz, 2H), 6.01 (d, *J* = 7.8 Hz, 1H), 5.03 (d, *J* = 8.1 Hz, 1H), 3.97-4.04 (m, 2H), 2.38 (s, 3H), 1.09 (t, *J* = 7.2 Hz, 3H); ^{19}F NMR (282 MHz, $CDCl_3$): δ -113.6; ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.7, 164.2, 160.9, 143.5, 136.7, 131.3, 131.2, 129.4, 128.9, 128.8, 127.1, 115.7, 115.4, 62.3, 58.6, 21.4, 13.7; IR (KBr): ν 3267, 1747, 1604, 1508, 1336, 1224, 1162, 1077, 1012, 815 cm^{-1}; MS (70 eV, EI) *m/z*: 278 (M^+ - CO_2Et), 155, 122, 91 (100), 65, 41; HRMS Calcd for $C_{14}H_{13}NO_2FS$ (M^+ - CO_2Et): 278.0651. Found: 278.0647.

The ee value was determined by chiral HPLC using a Chiralcel OJ-H column with hexane : isopropanol = 90 : 10, flow = 0.7 mL/min; ee: 64%; $[\alpha]_D^{20}$ = +66.8 (*c* 0.51, $CHCl_3$).

***N*-tosyl-3-nitrophenylglycine ethyl ester.**



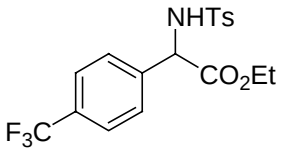
4f

White solid, mp: 132-134°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.10 (d, *J* = 8.1 Hz, 1H),

8.03 (s, 1H), 7.59-7.68 (m, 3H), 7.47 (t, *J* = 8.1 Hz, 1H), 7.19 (d, *J* = 8.1 Hz, 2H), 5.99 (d, *J* = 6.9 Hz, 1H), 5.14 (d, *J* = 6.9 Hz, 1H), 4.05-4.12 (m, 2H), 2.37 (s, 3H), 1.13 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.8, 143.9, 137.5, 136.5, 133.4, 129.7, 129.6, 127.1, 123.3, 122.2, 62.9, 58.6, 21.4, 13.8; IR (KBr): ν 3256, 2987, 1728, 1533, 1456, 1351, 1164, 1089, 1016, 725 cm^{-1}; MS (70 eV, EI) *m/z*: 305 (M^+ - CO_2Et), 155, 91 (100), 77, 65, 41; Anal. Cald for $C_{17}H_{18}N_2O_6S$: C, 53.96; H, 4.79; N, 7.40. Found: C, 53.96; H, 5.01; N, 7.18.

The ee value was determined by chiral HPLC using a Chiralcel OJ-H column with hexane : isopropanol = 60 : 40, flow = 0.7 mL/min; ee: 68%; $[\alpha]_D^{20}$ = +93.5 (*c* 0.51, $CHCl_3$).

***N*-tosyl-4-trifluromethylphenylglycine ethyl ester.**



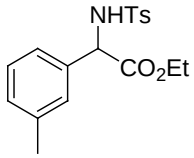
4g

White solid, mp: 108-110^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.58 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.1 Hz, 2H), 7.35 (d, *J* = 7.8 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 6.14 (d, *J* = 7.8 Hz, 1H), 5.12 (d, *J* = 7.8 Hz, 1H), 4.05 (m, 2H), 2.36 (s, 3H), 1.10 (t, *J* = 7.2 Hz, 3H); ^{19}F NMR (282 MHz, $CDCl_3$): δ -63.2; ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.2, 143.7, 139.2, 136.6, 129.4, 127.6, 127.0, 126.8, 125.5 (q, *J* = 3.9 Hz), 62.6, 58.9, 21.3, 13.7; IR (KBr): ν 3250, 1747, 1433, 1327, 1166, 1128, 1068, 1019, 852, 668 cm^{-1}; MS (70 eV, EI) *m/z*: 328 (M^+ - CO_2Et), 246, 172, 155, 145, 91 (100), 77, 65; Anal. Cald for $C_{18}H_{18}F_3NO_4S$: C, 53.86; H, 4.52; N, 3.49. Found: C, 54.04; H, 4.70; N,

3.44.

The ee value was determined by chiral HPLC using a Chiralcel OD-H column with hexane : isopropanol = 97 : 3, flow = 0.7 mL/min; ee: 71%; $[\alpha]_D^{20}$ = +61.3 (*c* 0.85, $CHCl_3$).

***N*-tosyl-3-tolylglycine ethyl ester.**

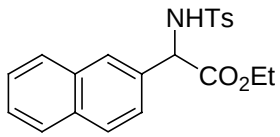


4h

White solid, mp: 86-88°C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.63 (d, *J* = 8.1 Hz, 2H), 7.13-7.20 (m, 3H), 6.99-7.06 (m, 3H), 5.80 (d, *J* = 8.1 Hz, 1H), 5.01 (d, *J* = 8.4 Hz, 1H), 3.92-4.05 (m, 2H), 2.37 (s, 3H), 2.23(s, 3H), 1.09 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.0, 143.3, 138.4, 136.9, 135.0, 129.3, 129.1, 128.5, 127.6, 127.1, 124.1, 62.1, 59.3, 21.4, 21.2, 13.8; IR (KBr): ν 3288, 2987, 1742, 1438, 1338, 1201, 1162, 1088, 1017, 815 cm^{-1}; MS (70 eV, EI) *m/z*: 274 (M^+ - CO_2Et, 100), 155, 118, 91, 77, 65; HRMS Calcd for $C_{15}H_{16}NO_2S$ (M^+ - CO_2Et): 274.0902. Found: 274.0902.

The ee value was determined by chiral HPLC using a Chiralcel OJ-H column with hexane : isopropanol = 80 : 20, flow = 0.7 mL/min; ee: 70%; $[\alpha]_D^{20}$ = +88.5 (*c* 0.50, $CHCl_3$).

***N*-tosyl-β-naphthylglycine ethyl ester.**



4i

White solid, mp: 115-116^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.64-7.77 (m, 4H), 7.60 (d, *J* = 8.1 Hz, 2H), 7.42-7.47 (m, 2H), 7.31 (d, *J* = 8.4 Hz, 1H), 7.03 (d, *J* = 8.1 Hz, 2H), 6.10 (d, *J* = 8.1 Hz, 1H), 5.22 (d, *J* = 8.1 Hz, 1H), 3.93-4.09 (m, 2H), 2.22 (s, 3H), 1.07 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.6, 143.3, 136.8, 132.9, 132.8, 132.3, 129.2, 128.6, 127.9, 127.4, 127.0, 126.6, 126.4, 126.3, 124.3, 62.2, 59.5, 21.2, 13.7; IR (KBr): ν 3285, 2992, 1715, 1435, 1340, 1168, 1078, 905, 818 cm^{-1}; MS (70 eV, EI) *m/z*: 383 (M^+), 310(M^+ - CO_2Et, 100), 155, 127, 91, 77, 65, 41; Anal. Cald for $C_{21}H_{21}NO_4S$: C, 65.78; H, 5.52; N, 3.65. Found: C, 65.86; H, 5.55; N, 3.41.

The ee value was determined by chiral HPLC using a Chiralcel AD-H column with hexane : isopropanol = 65 : 35, flow = 0.7 mL/min; ee: 72%; $[\alpha]_D^{20}$ = +89.2 (*c* 0.50, $CHCl_3$).

IV. Synthesis of *N*-tosyl-(*S*)-phenylglycine ethyl ester from (S)-phenylglycine.

(*S*)-phenylglycine ethyl ester hydrochloride. To a Schlenk bottle were charged (*S*)-phenylglycine (1.51 g, 10 mmol, ee >99%) and EtOH (10 mL, 0.17 mol), the mixture was cooled to 0 ^{o}C and $SOCl_2$ (2 mL, 26.8 mmol) was dropped to it and refluxed overnight. After cooled to rt, Et_2O was added to the solution and white solid was separated. The solid was filtered and dried to give the (*S*)-phenylglycine ethyl ester hydrochloride.[3] Yield: 82%. M.p 182-183 ^{o}C. ^{1}H NMR (300 MHz, D_2O): δ

7.52-7.54 (m, 3H), 7.45-7.48 (m, 2H), 5.26 (s, 1H), 4.27-4.33 (m, 2H), 1.22 (t, *J*= 7.2 Hz, 3H). $[\alpha]_D^{20}$ = +94.7° (*c* 1.005, H_2O). (lit[3b] $[\alpha]_D^{20}$ = +91 °, *c* 1.00, H_2O).

N-tosyl-(*S*)-phenylglycine ethyl ester. To a Schlenk tube were added (*S*)-phenylglycine ethyl ester hydrochloride (620 mg, 2.9 mmol), CH_2Cl_2 (20 mL) and $^iPr_2NEt$ (2.3 g, 18 mmol) and the mixture was cooled to 0 °C. Then TsCl (680 mg, 3.6 mmol) was added slowly to the mixture and stirred at rt overnight under N_2. The reaction mixture was concentrated under reduced pressure and purified by flash column chromatography (ethyl acetate: petroleum ether = 1:6 to 1:2) to obtain the product *N*-tosyl-(*S*)-phenylglycine ethyl ester[4]. Yield: 82%. M.p 102-103 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.63 (d, *J*=8.1 Hz, 2H), 7.19-7.28 (m, 7H), 5.66 (d, *J*=8.1 Hz, 1H), 5.03 (d, *J*=7.5 Hz, 1H), 3.94-4.08 (m, 2H), 2.39 (s, 3H), 1.10 (t, *J*= 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.0, 143.4, 136.8, 135.3, 129.4, 128.6, 128.4, 127.1, 127.0, 62.1, 59.3, 21.4, 13.7; IR (KBr): ν 3281, 2995, 2967, 2904, 1716, 1498, 1459, 1441, 1336, 1266, 1168, 1090, 820, 1022, 697 cm^{-1}; MS (70 eV, EI) *m/z*: 260 (M^+ - CO_2Et, 100), 155, 91, 77, 65.

The ee value was determined by chiral HPLC using a Chiralcel OD-H column (150 mm) with hexane : isopropanol = 90 : 10, flow = 0.7 mL/min; ee: 99%; $[\alpha]_D^{20}$ = +105.1° (*c* 1.09, $CDCl_3$).

IV. References

[1] (a) Evans, D. A.; Woerpel, K. A.; Hinman, M. M.; Faul M. M. *J. Am. Chem. Soc.* **1991**, *113*, 726. (b) Corey, E. J.; Imai, N.; Zhang, H. *J. Am. Chem. Soc.* **1991**,

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Palladium(II)-Catalyzed One-Pot Enantioselective Synthesis of Arylglycine Derivatives from Ethyl Glyoxylate, *p*-Toluenesulfonyl Isocyanate and Arylboronic Acids

Huixiong Dai, Miao Yang and Xiyan Lu*

State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Lu, Shanghai 200032, China

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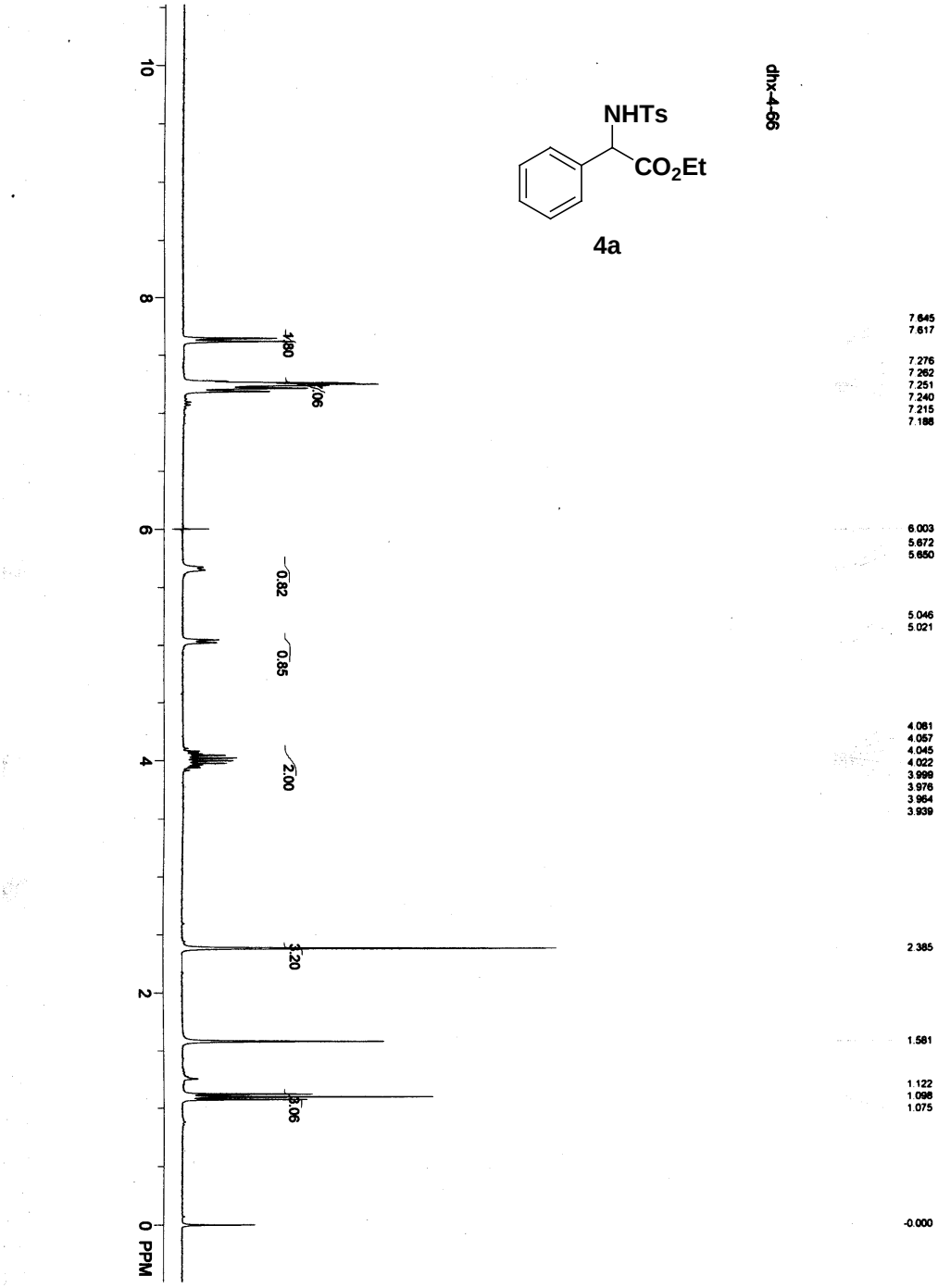
dhx-4-66
NHTs
CO2Et
4a
10
8
6
4
2
0 PPM
1.80
2.06
0.82
0.85
2.00
3.20
3.06
7.645
7.617
7.276
7.262
7.251
7.240
7.215
7.188
6.003
5.672
5.650
5.046
5.021
4.081
4.057
4.045
4.022
3.999
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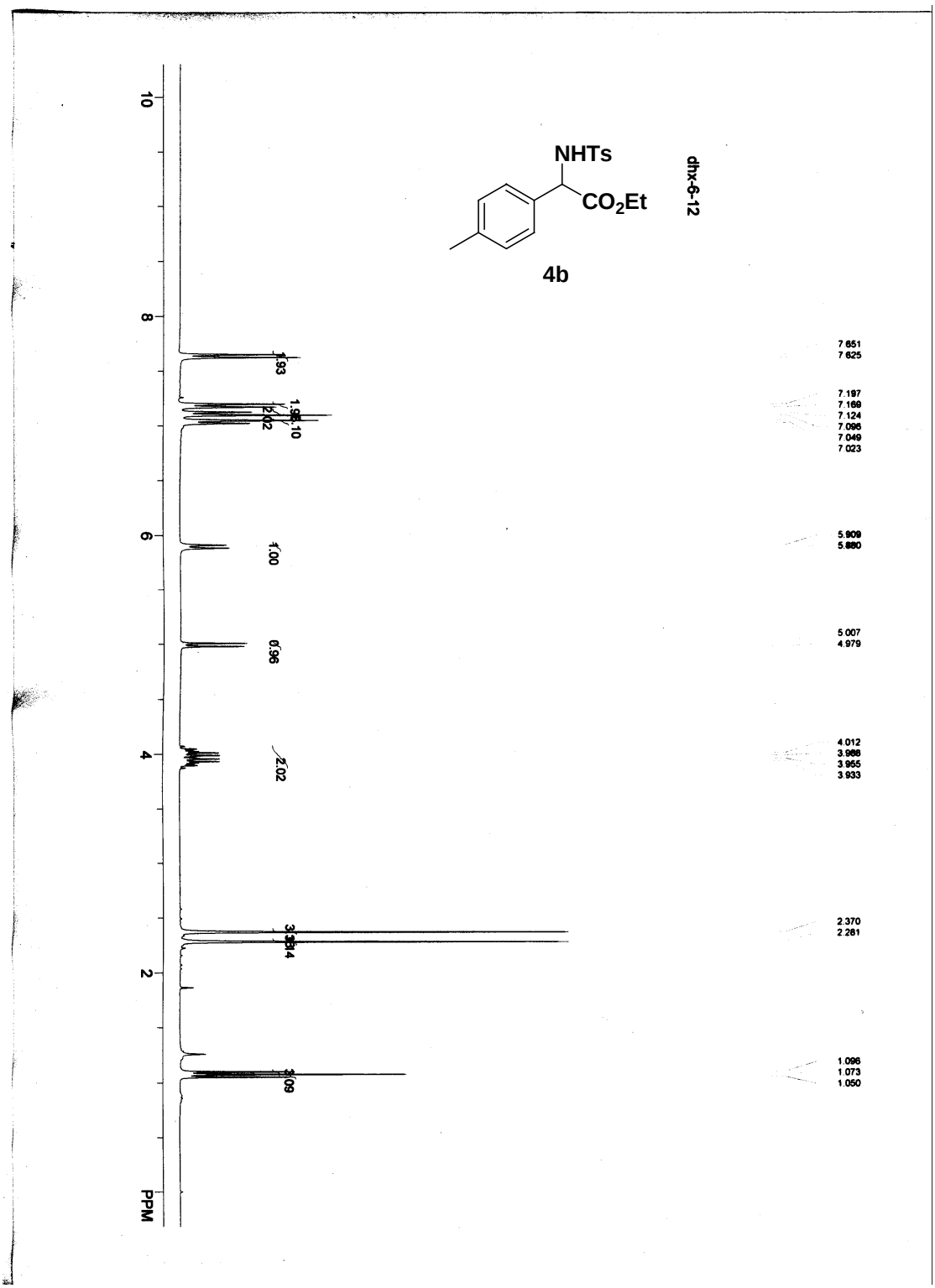
3.964
3.939
2.385
1.581
1.122
1.098
1.075
-0.000


NHTs
CO2Et
4b
dhx-6-12
10
8
6
4
2
PPM
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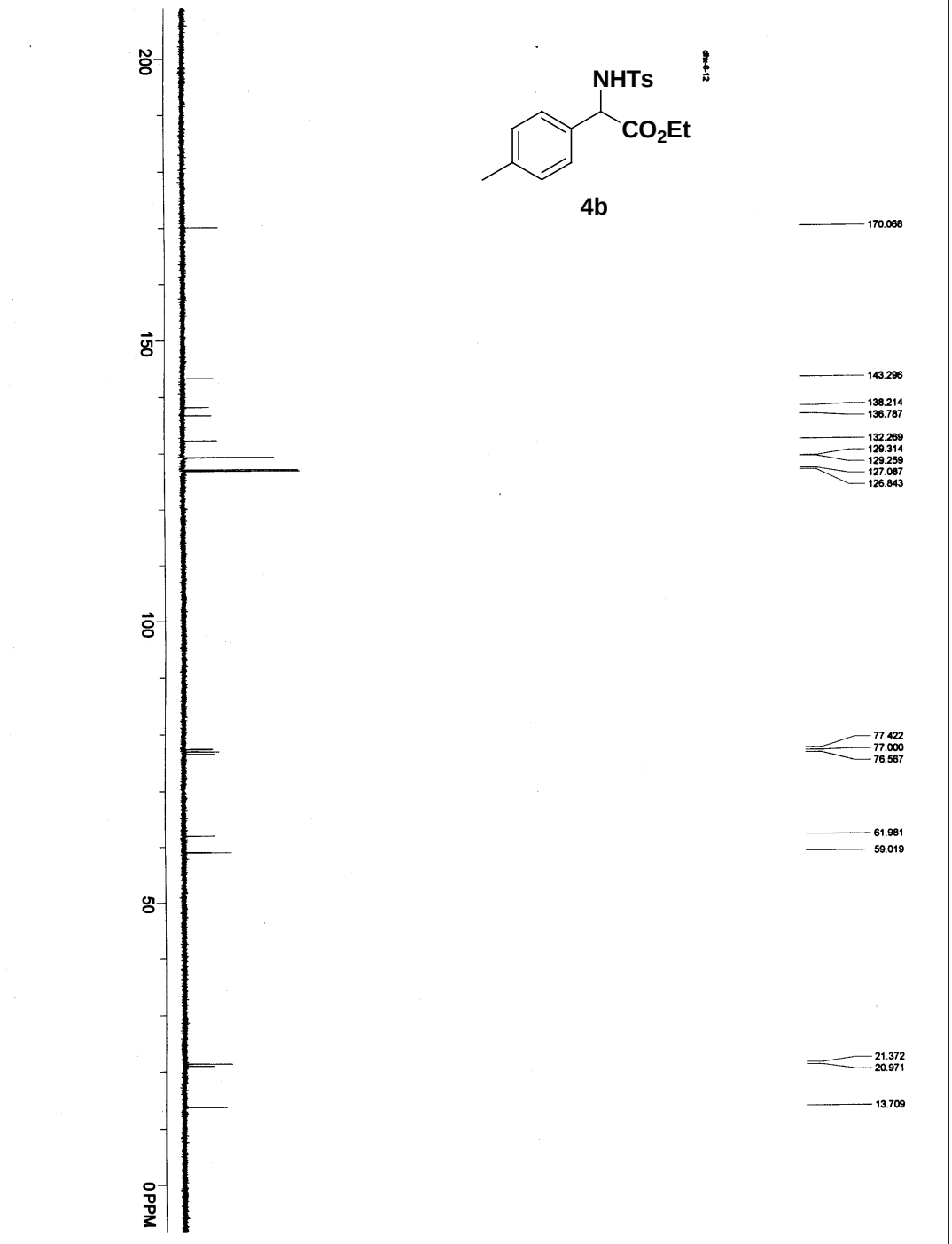
1.95
2.02
3.10
1.00
0.96
2.02
3.04
1.09
7.651
7.625
7.197
7.169
7.124
7.096
7.049
7.023
5.909
5.880
5.007
4.979
4.012
3.988
3.955
3.933
2.370
2.261
1.096
1.073
1.050


NHTs
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4b
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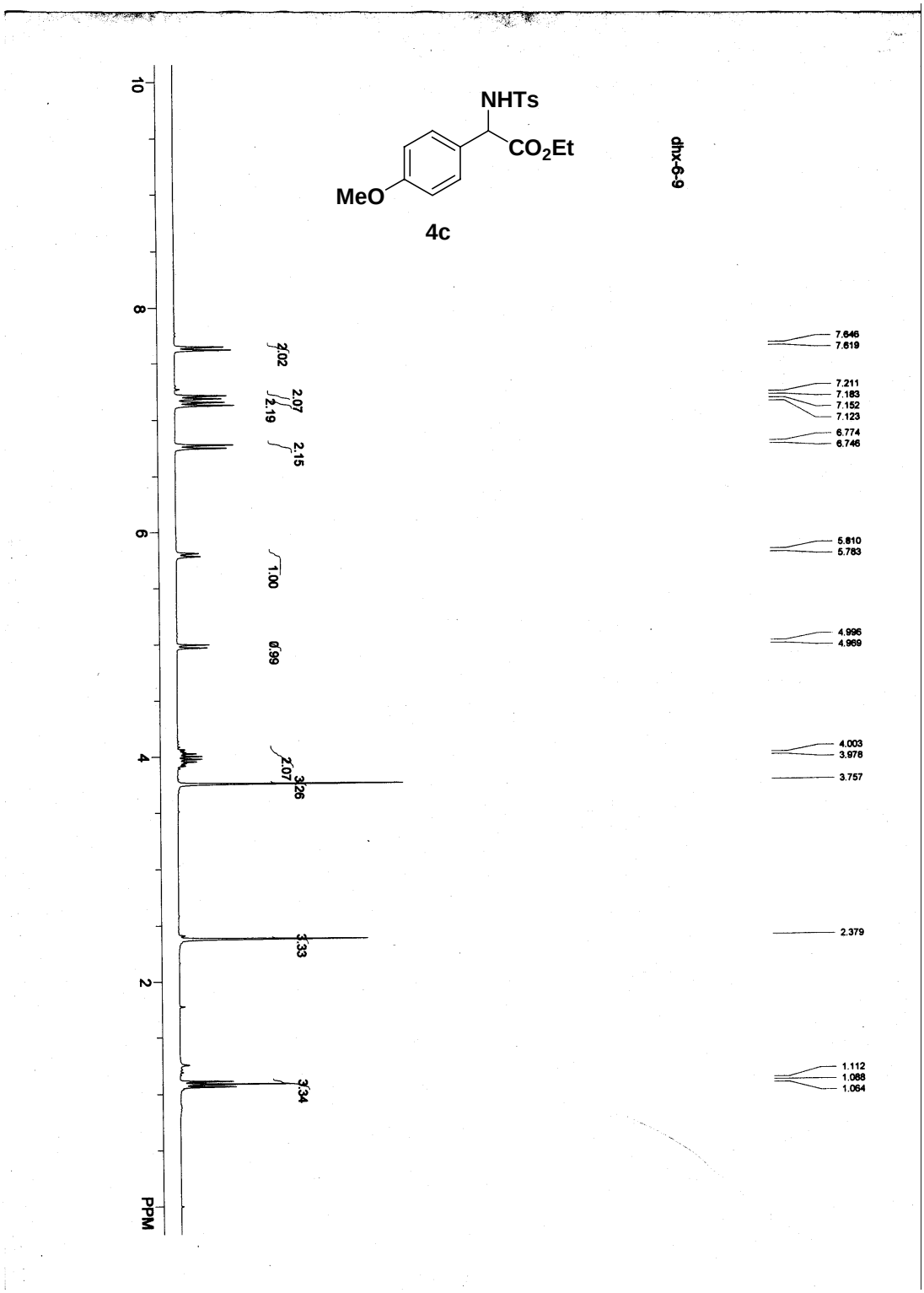
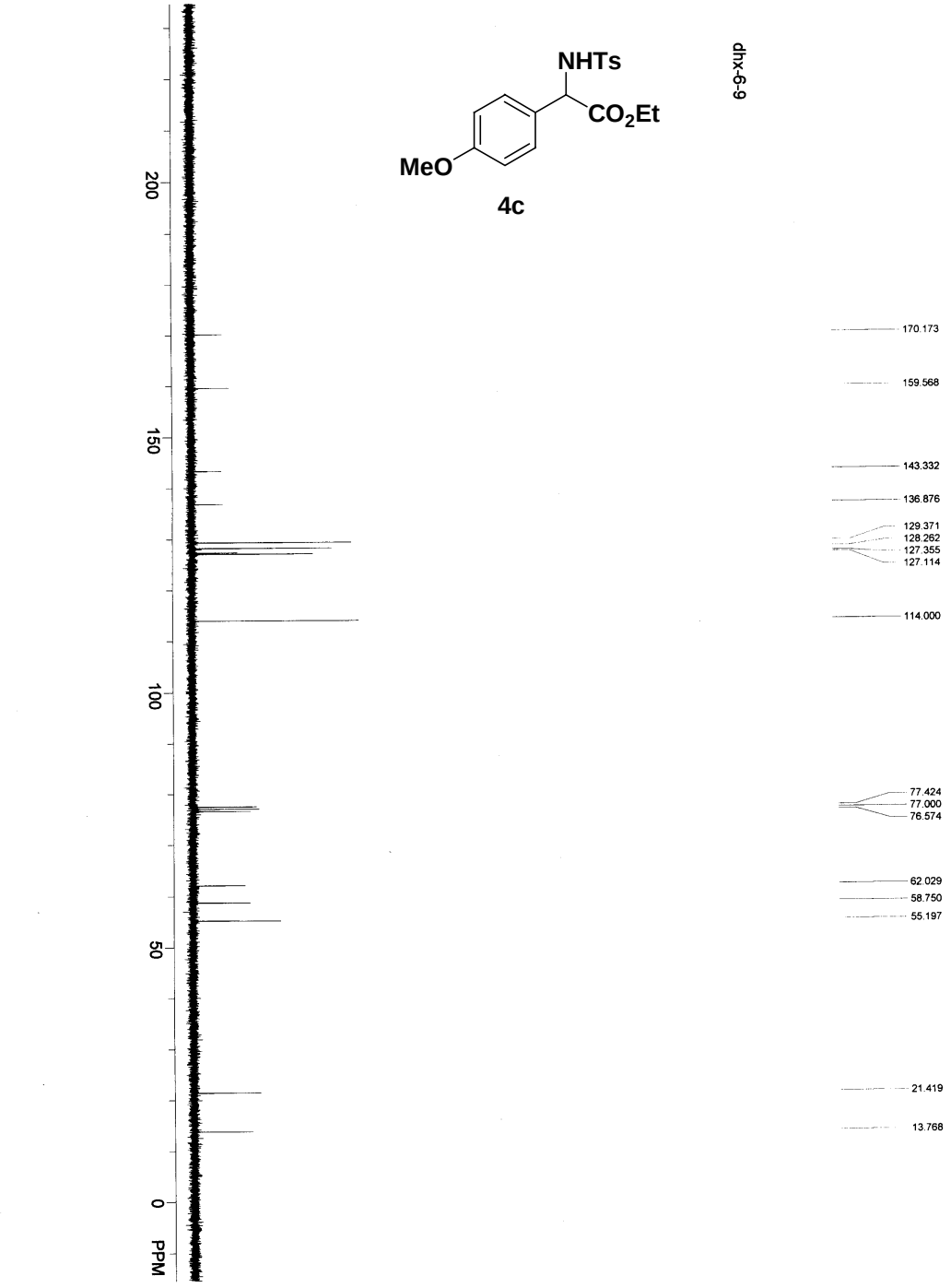
138.214
136.787
132.269
129.314
129.259
127.067
126.843
77.422
77.000
76.567
61.981
59.019
21.372
20.971
13.709
200
150
100
50
0 PPM


NHTs
CO2Et
MeO
4c
dhx-6-9
10
8
6
4
2
PPM
2.02
2.07
2.19
2.15
1.00
0.99
2.07
3.26
3.33
3.34
7.646
7.619
7.211
7.183
7.152
7.123
6.774
6.746
5.810
5.783
4.996
4.969
4.003
3.978
3.757
2.379
1.112
1.088
1.064


NHTs
CO2Et
MeO
4c
dhx-6-9
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143.332
136.876
129.371
128.262
127.355
127.114
114.000
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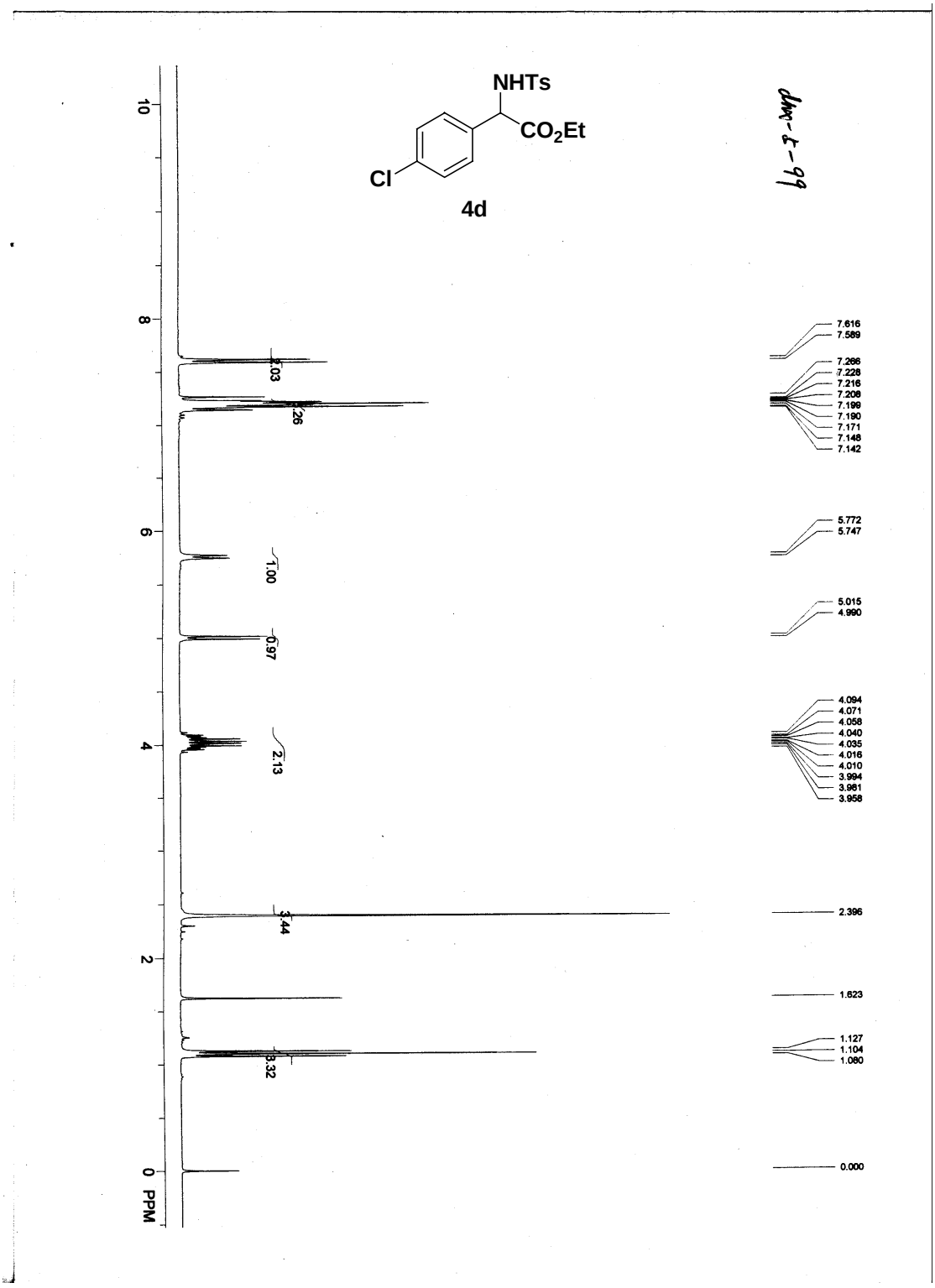
77.000
76.574
62.029
58.750
55.197
21.419
13.768
200
150
100
50
0
PPM


NHTs
CO2Et
Cl
4d
dmf-2-99
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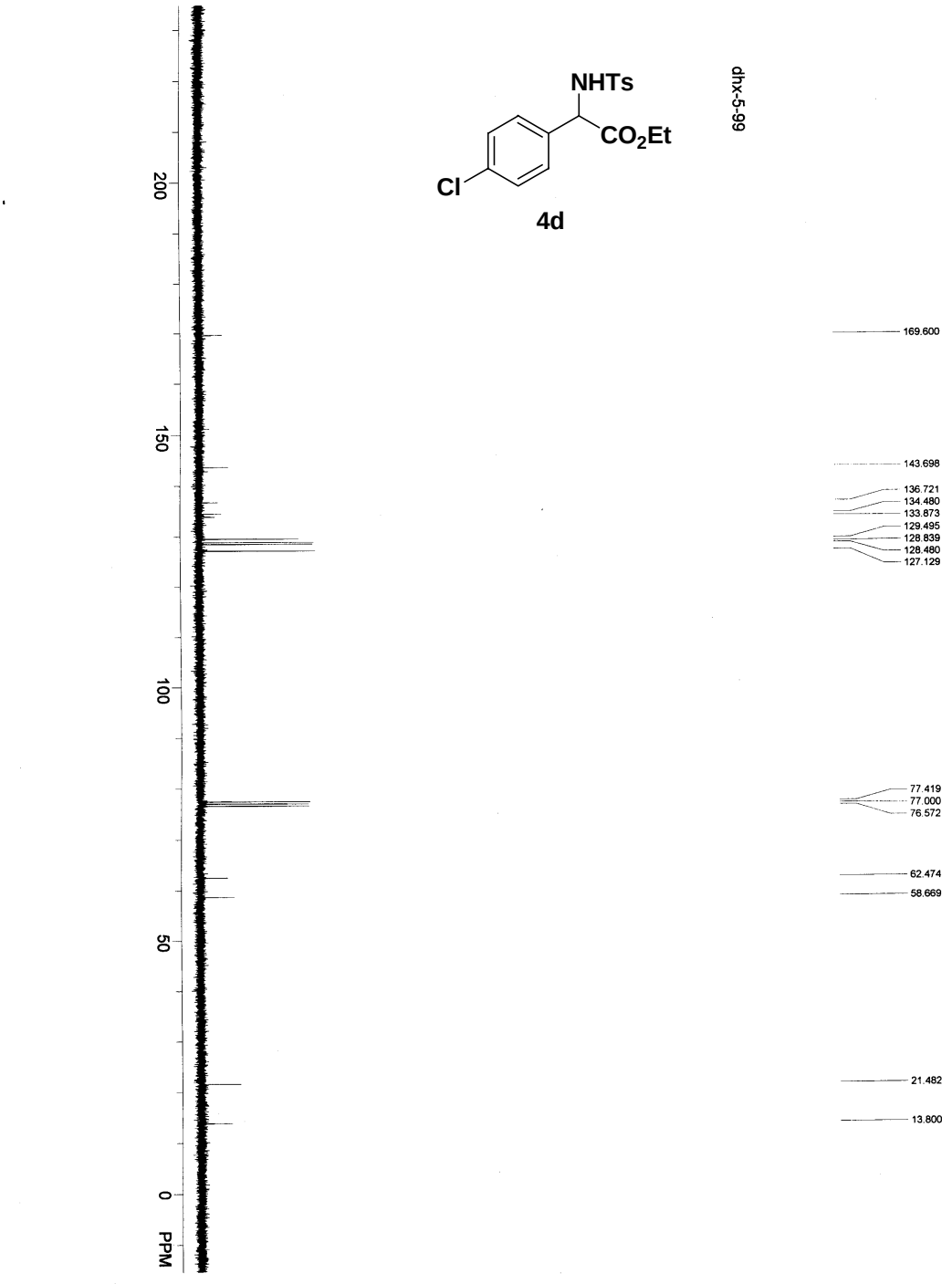
7.589
7.266
7.228
7.216
7.208
7.199
7.190
7.171
7.148
7.142
5.772
5.747
5.015
4.990
4.094
4.071
4.058
4.040
4.035
4.016
4.010
3.994
3.981
3.958
2.396
1.623
1.127
1.104
1.080
0.000
2.03
2.26
1.00
0.97
2.13
3.44
3.32
10
8
6
4
2
0 PPM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CO2Et
Cl
4d
dtx-5-99
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136.721
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133.873
129.495
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128.480
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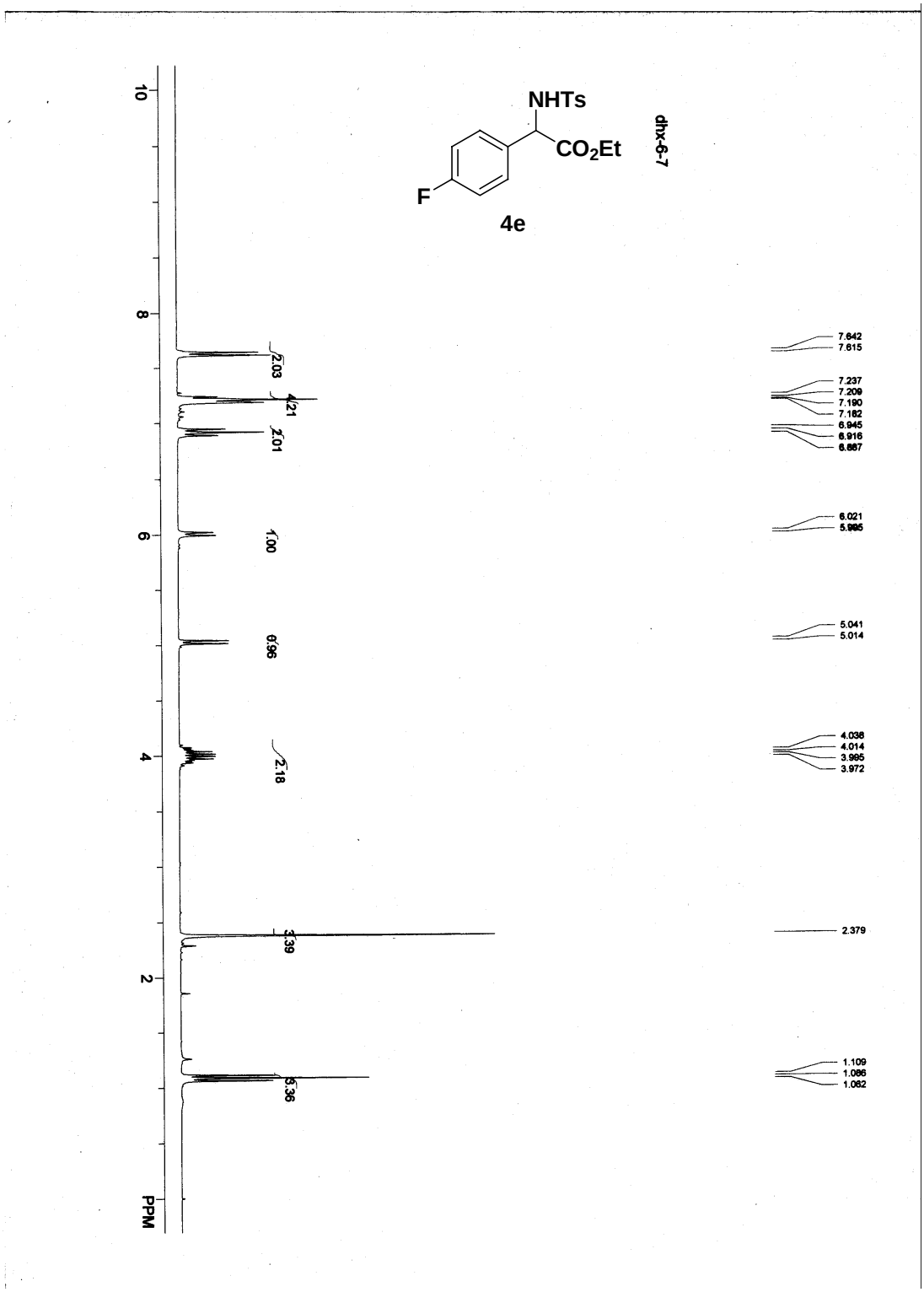
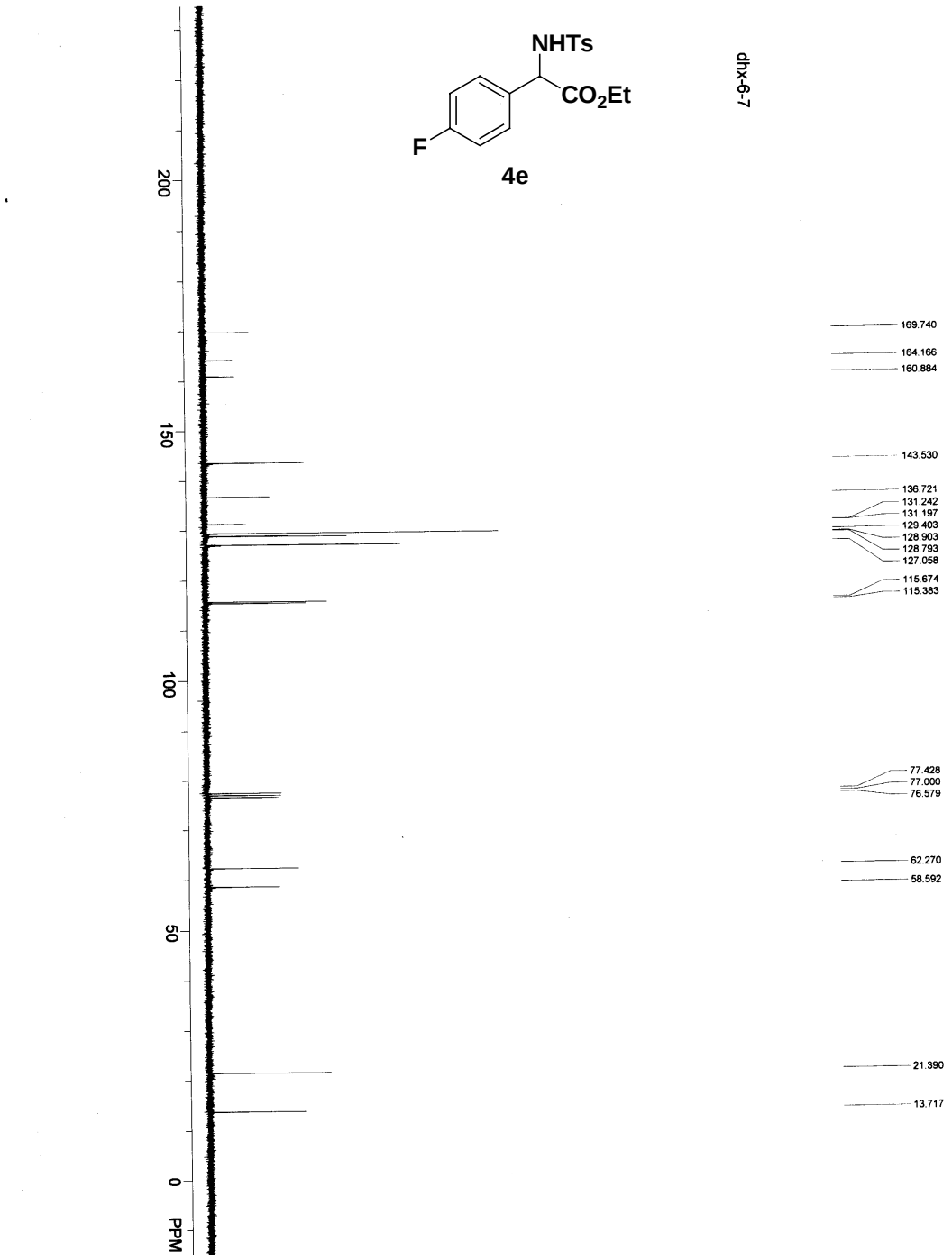
77.419
77.000
76.572
62.474
58.669
21.482
13.800
200
150
100
50
0
PPM


NHTs
CO2Et
F
4e
dhx-6-7
10
8
6
4
2
PPM
2.03
4.21
2.01
1.00
6.96
2.18
3.39
3.36
7.642
7.615
7.237
7.209
7.190
7.182
6.945
6.916
6.887
6.021
5.995
5.041
5.014
4.036
4.014
3.995
3.972
2.379
1.109
1.086
1.062


NHTs
CO2Et
F
4e
dhx-6-7
200
150
100
50
0
PPM
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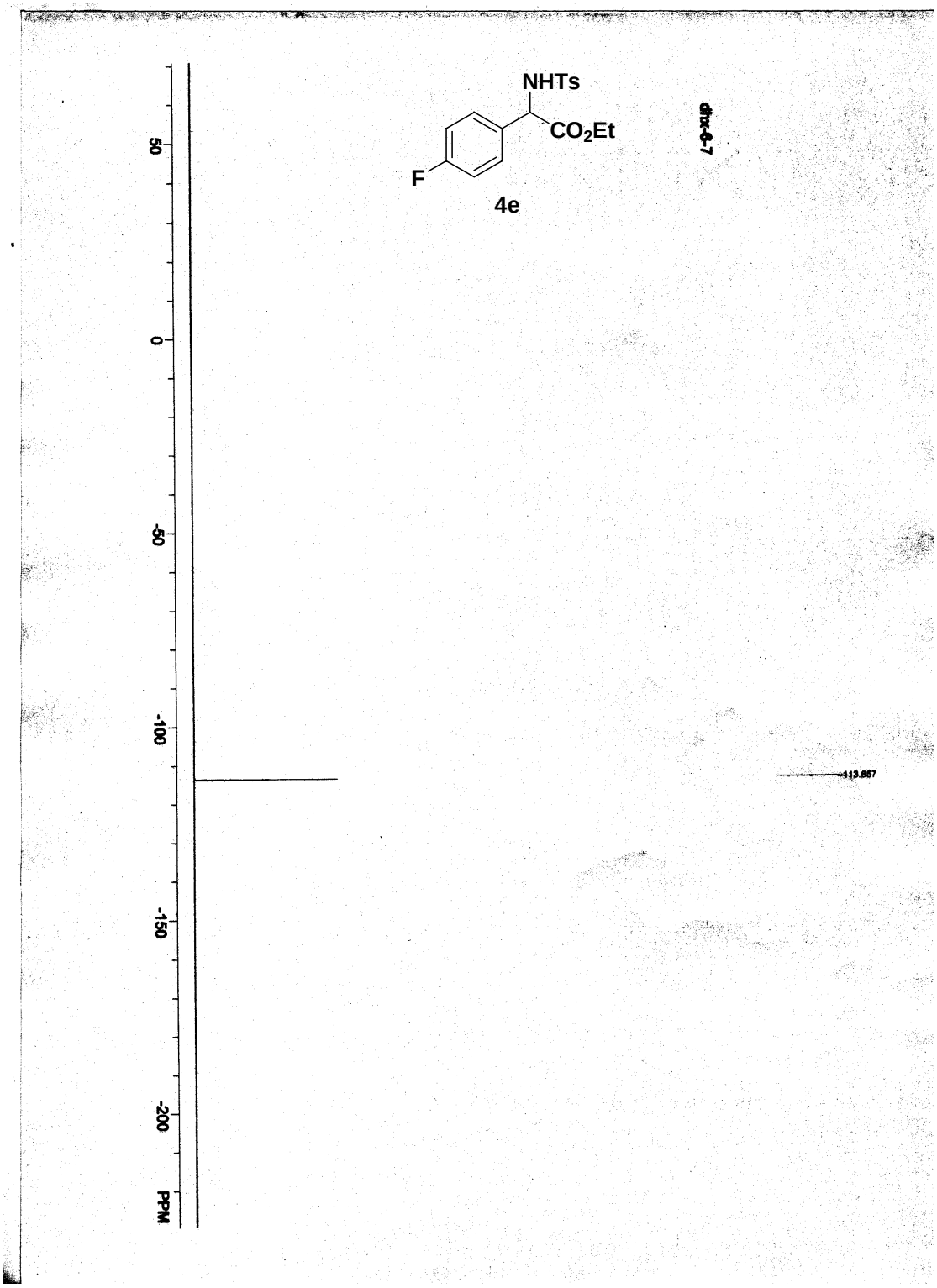
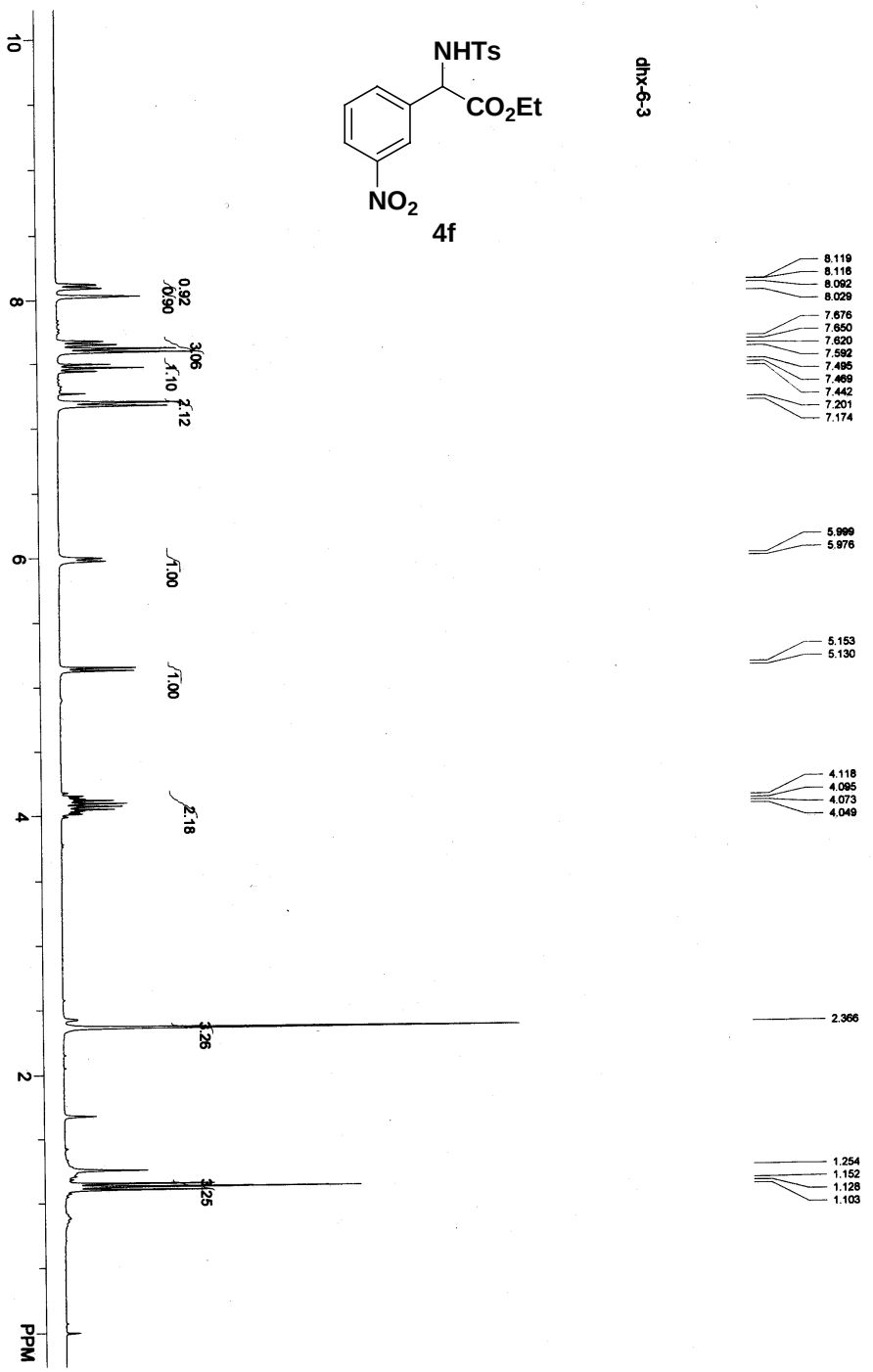
164.166
160.884
143.530
136.721
131.242
131.197
129.403
128.903
128.793
127.056
115.674
115.383
77.428
77.000
76.579
62.270
58.592
21.390
13.717


NHTs
CO2Et
F
4e
dhx-6-7
50
0
-50
-100
-150
-200
PPM
-113.857


NHTs
CO2Et
NO2
4f
dhx-6-3
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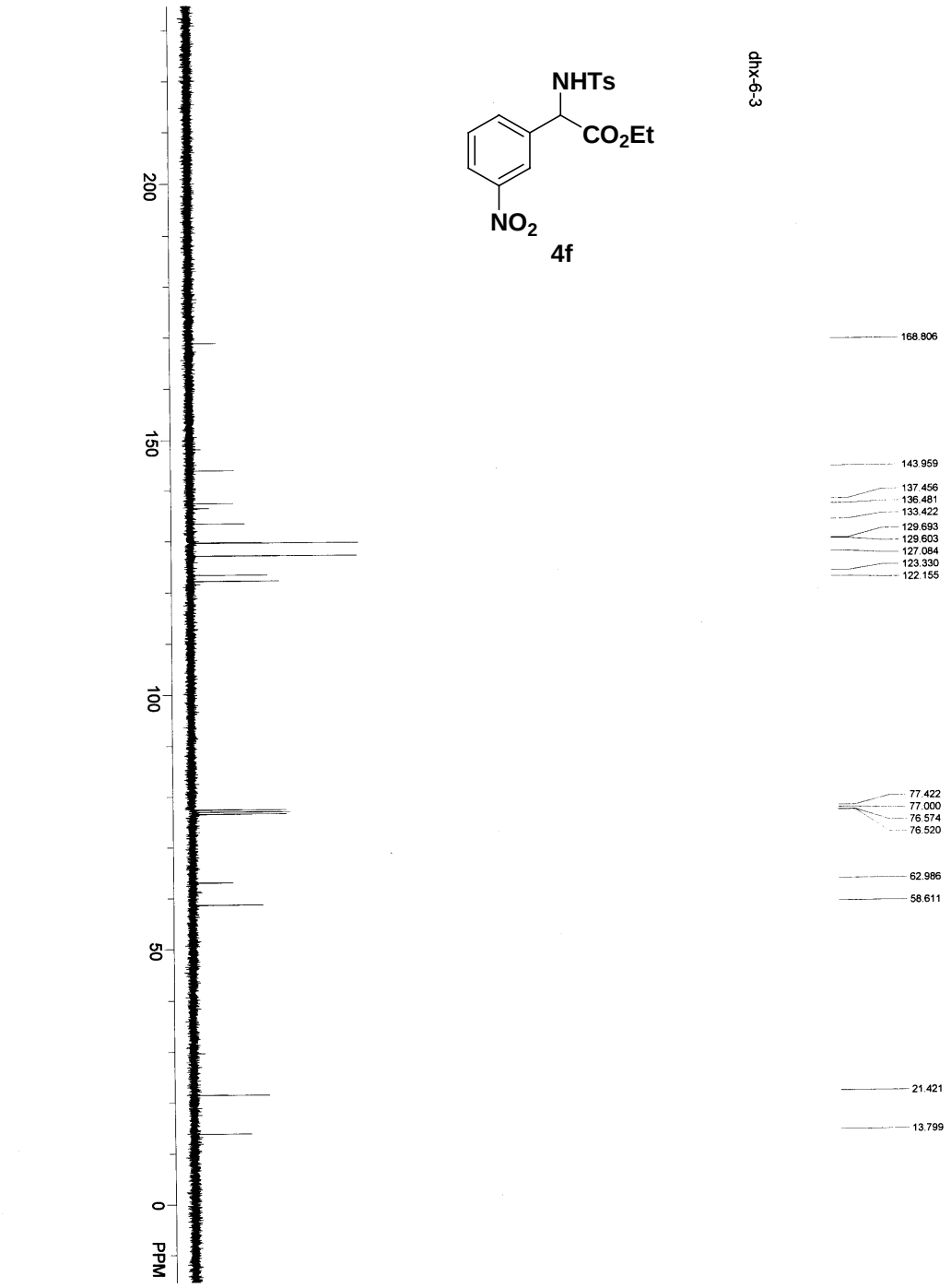
8.116
8.092
8.029
7.676
7.650
7.620
7.592
7.495
7.469
7.442
7.201
7.174
5.999
5.976
5.153
5.130
4.118
4.095
4.073
4.049
2.366
1.254
1.152
1.128
1.103
0.92
0.90
3.06
1.10
2.12
1.00
1.00
2.18
3.26
4.25
10
8
6
4
2
PPM


NHTs
CO2Et
NO2
4f
dhx-6-3
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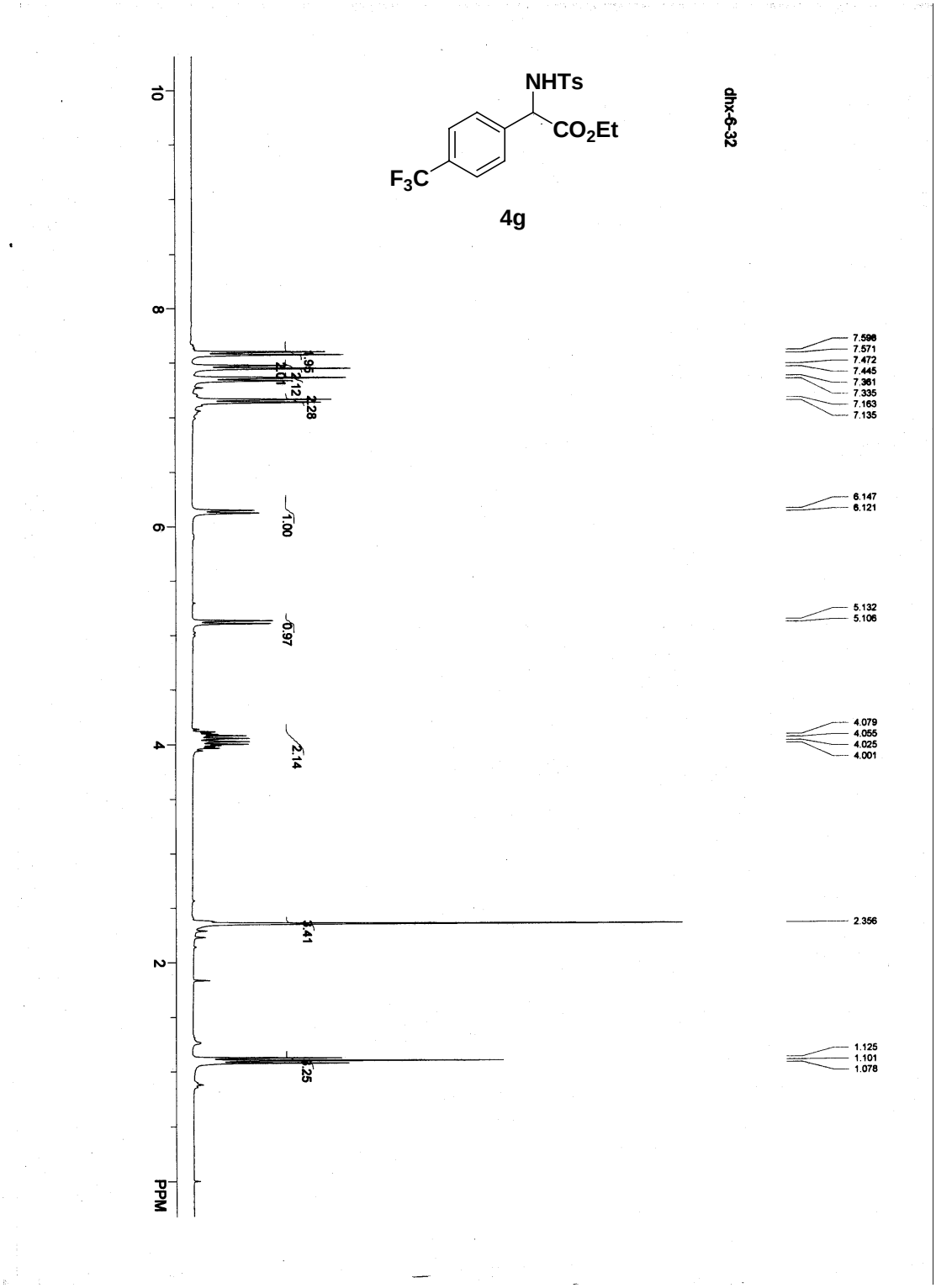
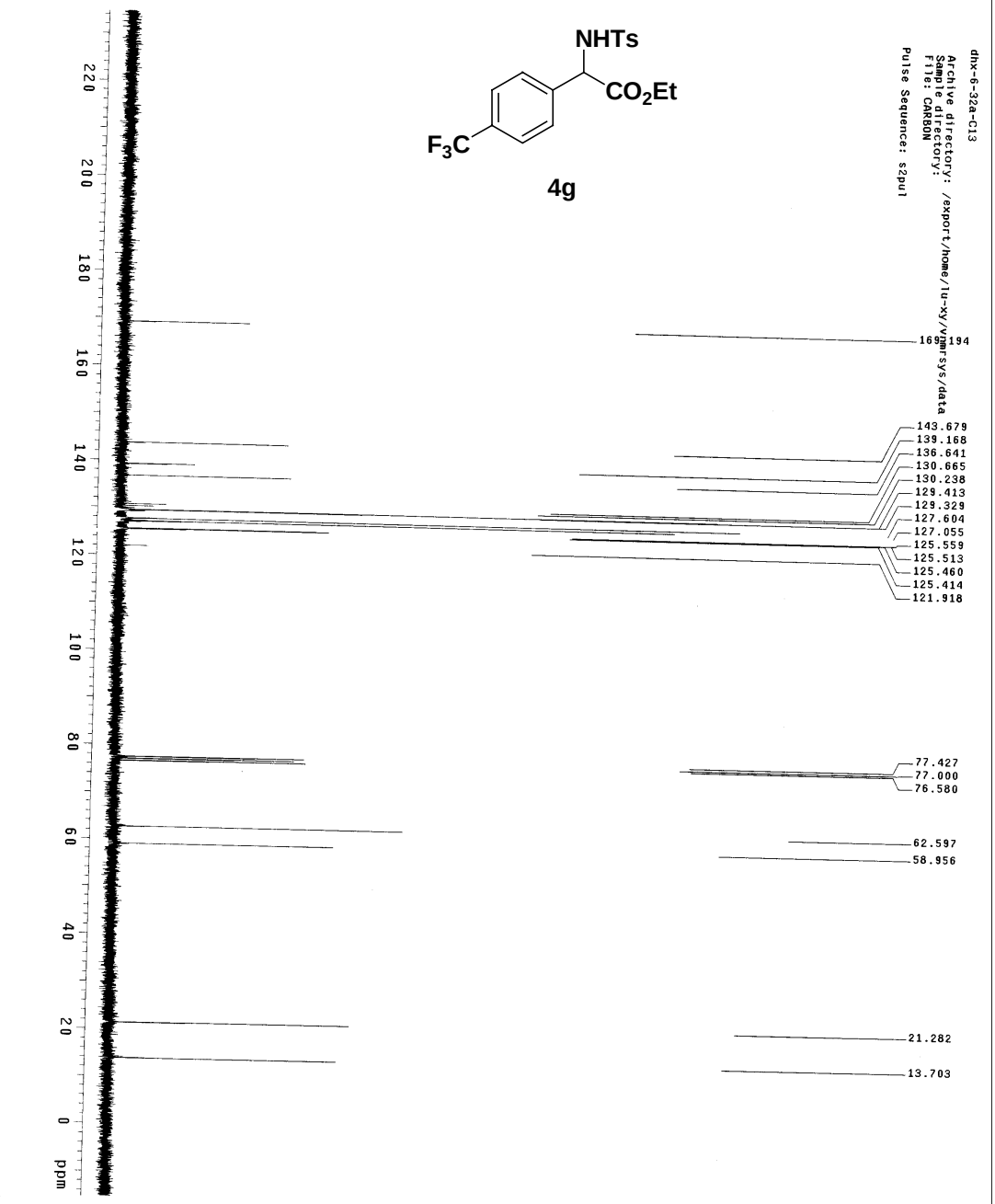
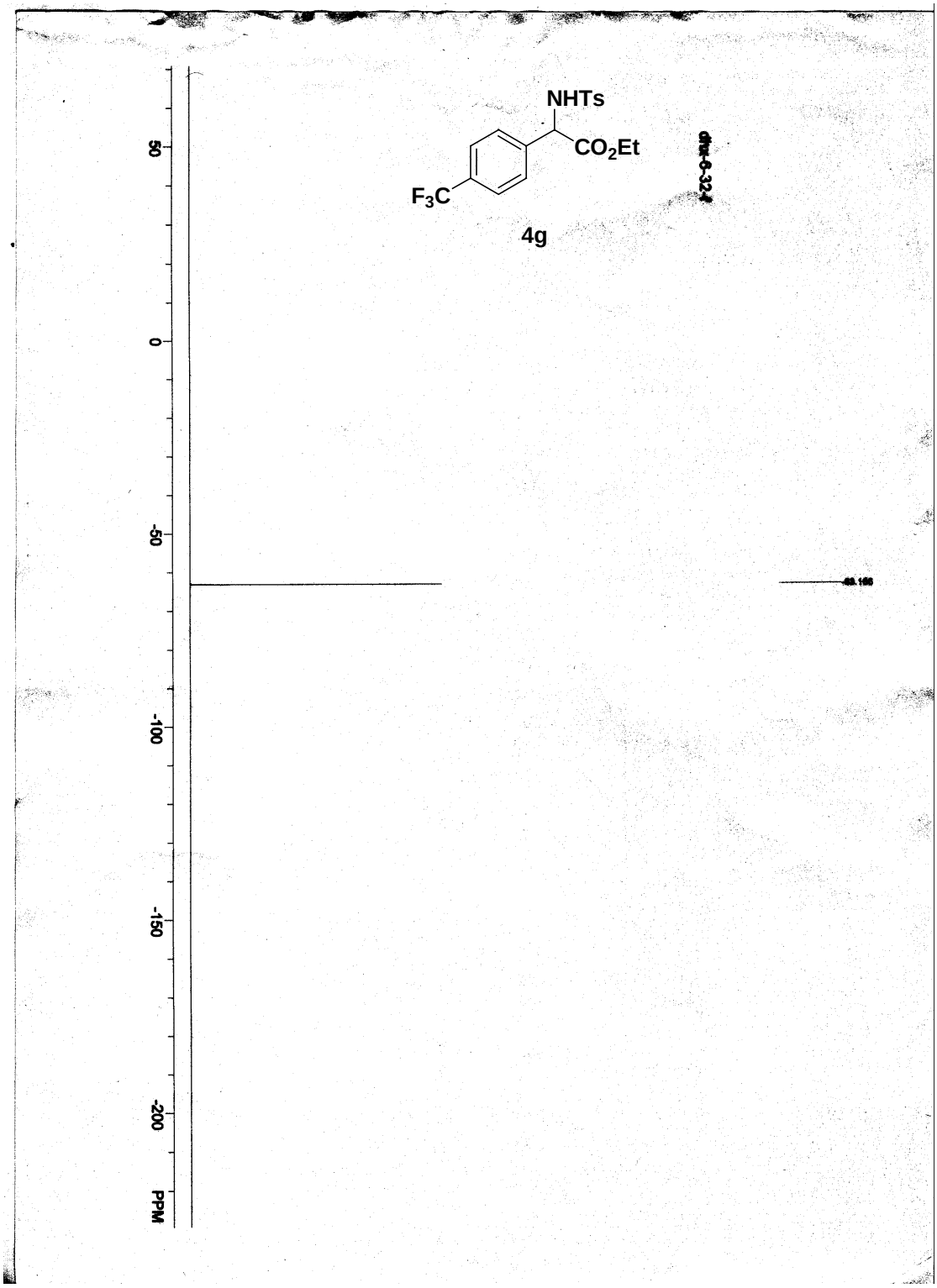
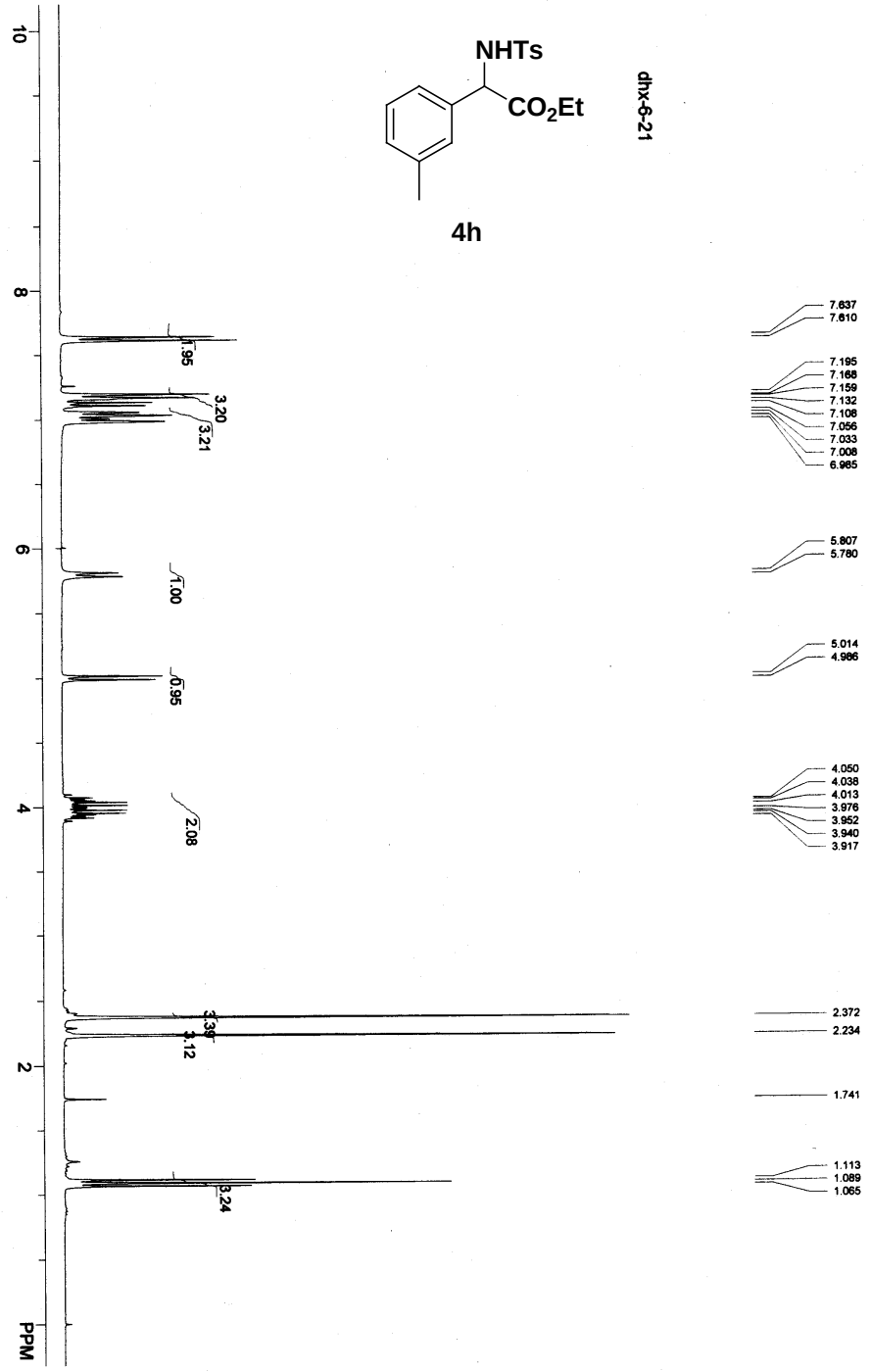
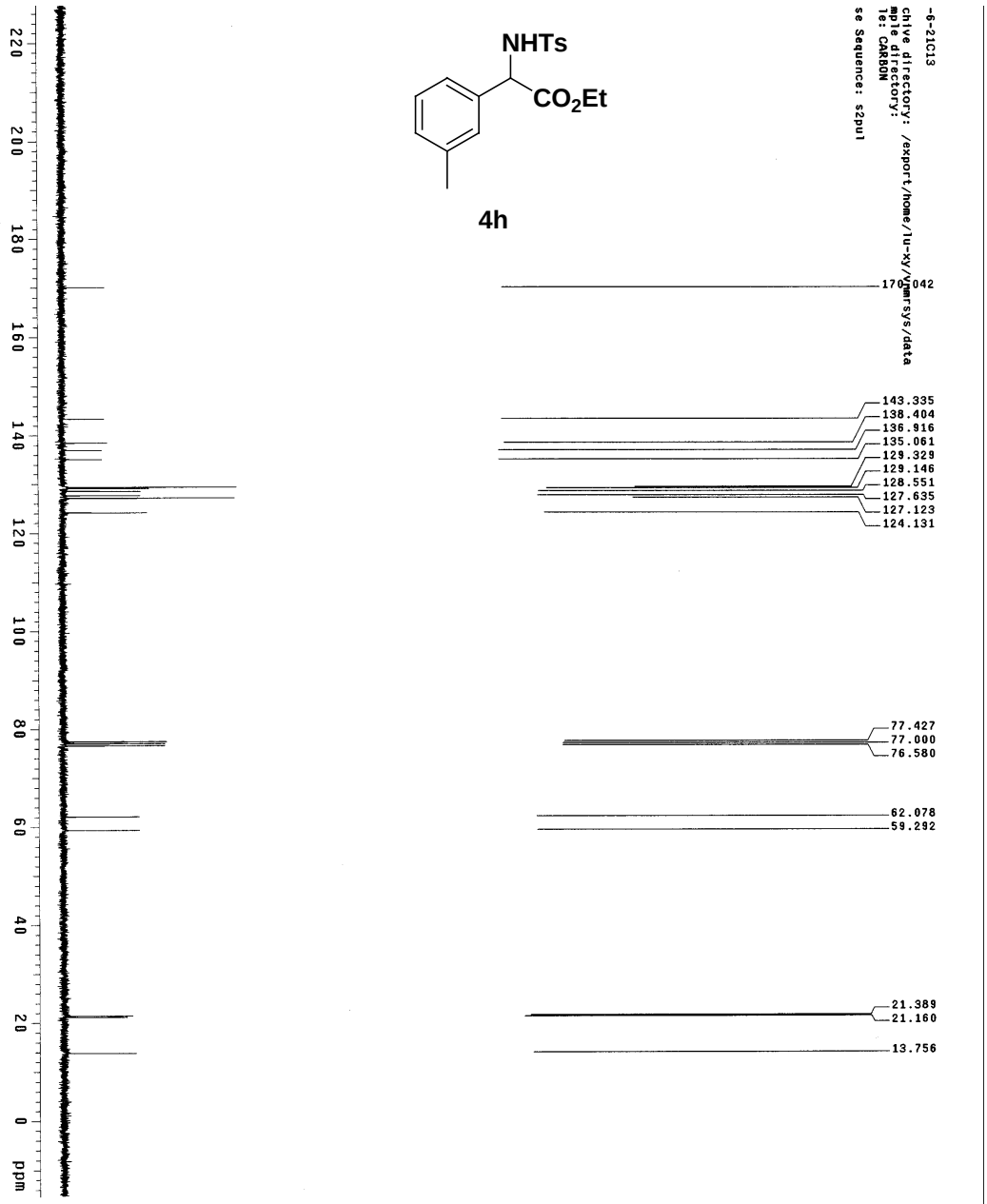
137.456
136.481
133.422
129.693
129.603
127.084
123.330
122.155
77.422
77.000
76.574
76.520
62.986
58.611
21.421
13.799
200
150
100
50
0
PPM

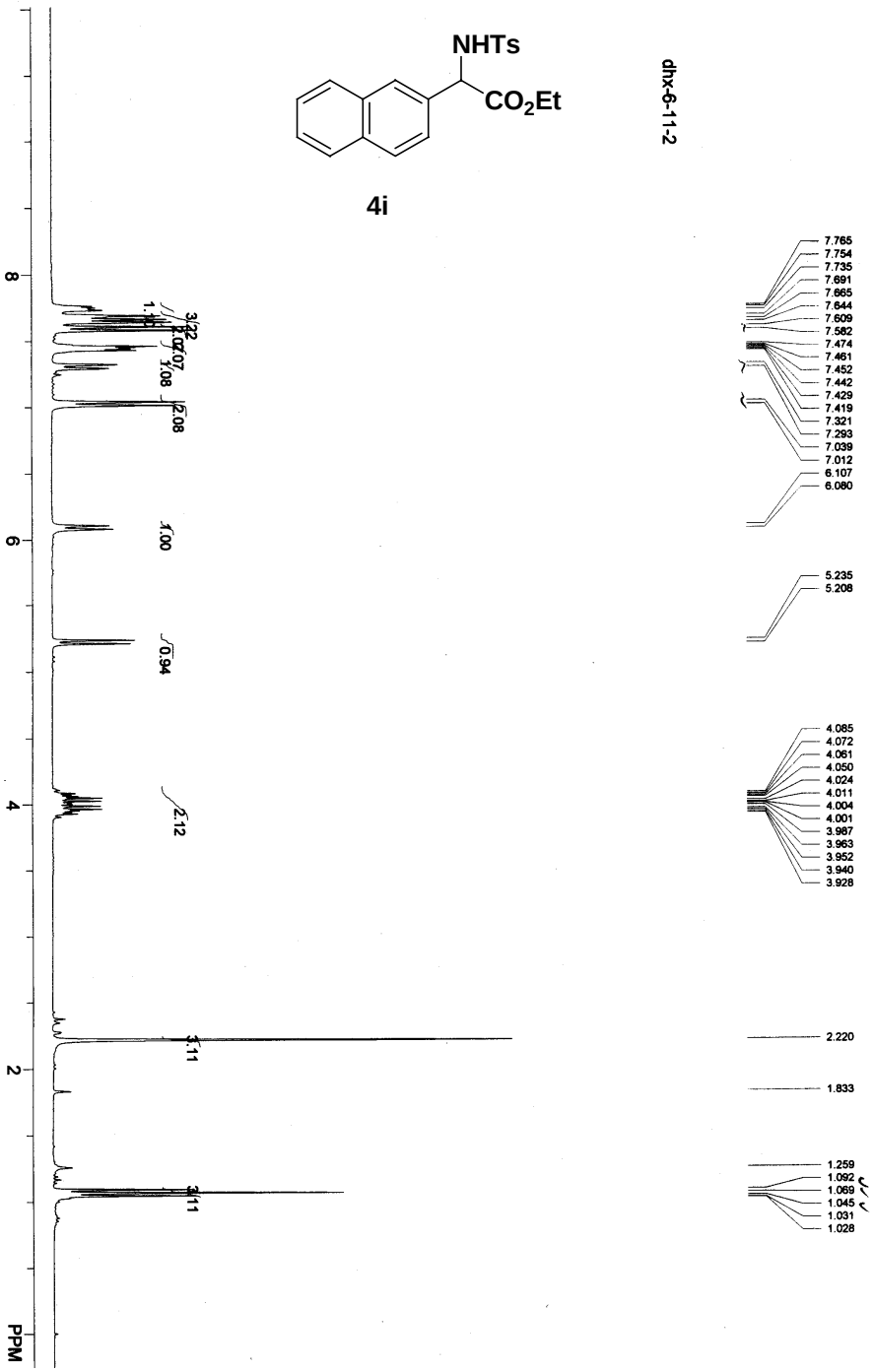

NHTs
CO2Et
F3C
4g
dhx-6-32
7.598
7.571
7.472
7.445
7.361
7.335
7.163
7.135
6.147
6.121
5.132
5.106
4.079
4.055
4.025
4.001
2.356
1.125
1.101
1.078
1.95
2.01
2.12
2.28
1.00
0.97
2.14
3.41
3.25
10
8
6
4
2
PPM


NHTs
CO2Et
F3C
4g
dhx-6-32a-C13
Archive directory: /export/home/lu-xy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
169.194
143.679
139.168
136.641
130.665
130.238
129.413
129.329
127.604
127.055
125.559
125.513
125.460
125.414
121.918
77.427
77.000
76.580
62.597
58.956
21.282
13.703
220
200
180
160
140
120
100
80
60
40
20
0
ppm


NHTs
CO2Et
F3C
4g
50
0
-50
-100
-150
-200
PPM


NHTs
CO2Et
4h
dhx-6-21
10
8
6
4
2
PPM
1.95
3.20
3.21
1.00
0.95
2.08
3.39
3.12
3.24
7.637
7.610
7.195
7.168
7.159
7.132
7.108
7.056
7.033
7.008
6.985
5.807
5.780
5.014
4.986
4.050
4.038
4.013
3.976
3.952
3.940
3.917
2.372
2.234
1.741
1.113
1.089
1.065


NHTs
CO2Et
4h
-6-21C13
chive directory: /export/home/lu-xy/vnmrsys/data
mple directory:
le: CARBON
se Sequence: s2pul
170.042
143.335
138.404
136.916
135.061
129.329
129.146
128.551
127.635
127.123
124.131
77.427
77.000
76.580
62.078
59.292
21.389
21.160
13.756
220
200
180
160
140
120
100
80
60
40
20
0
ppm


NHTs
CO2Et
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dhx-6-11-2
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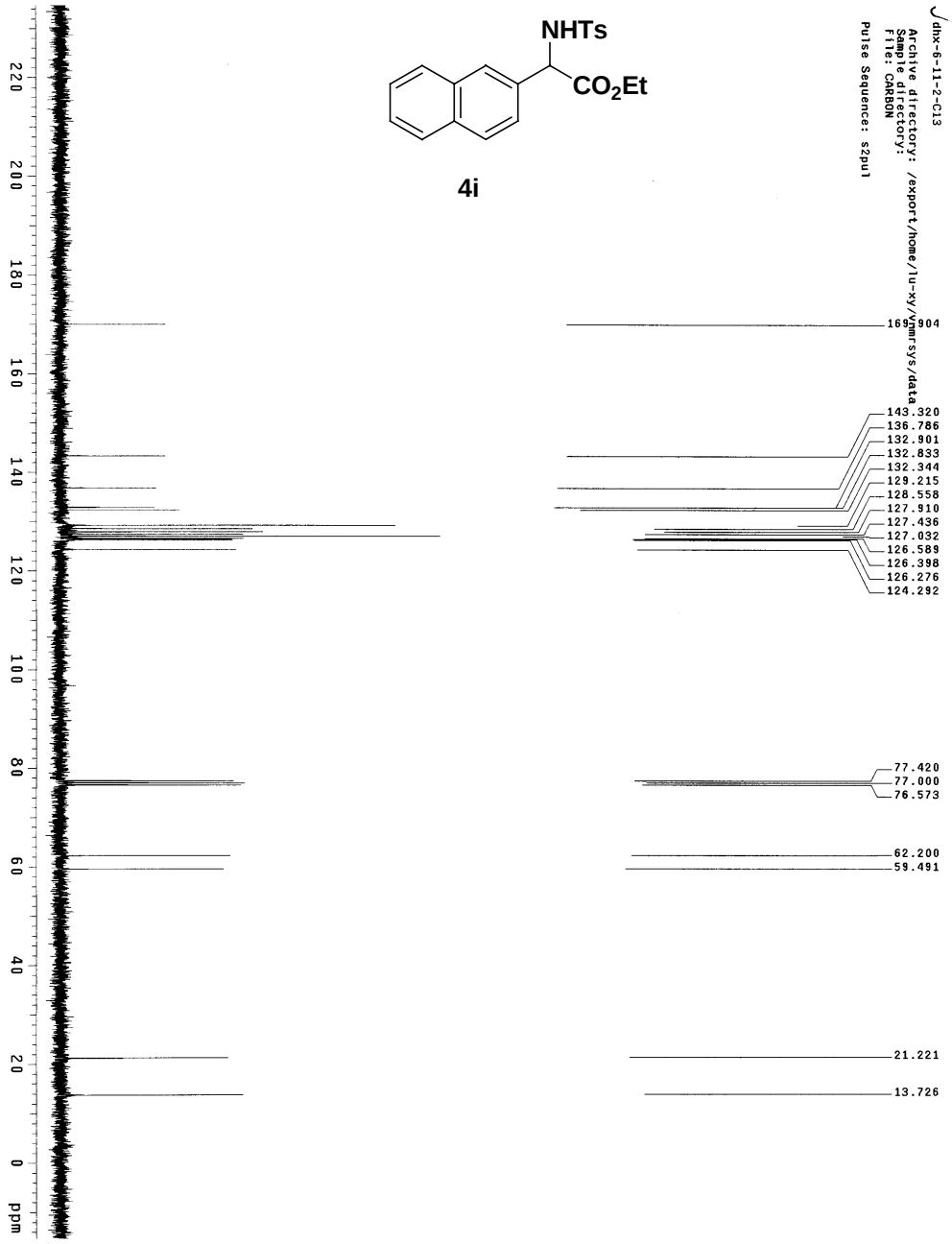
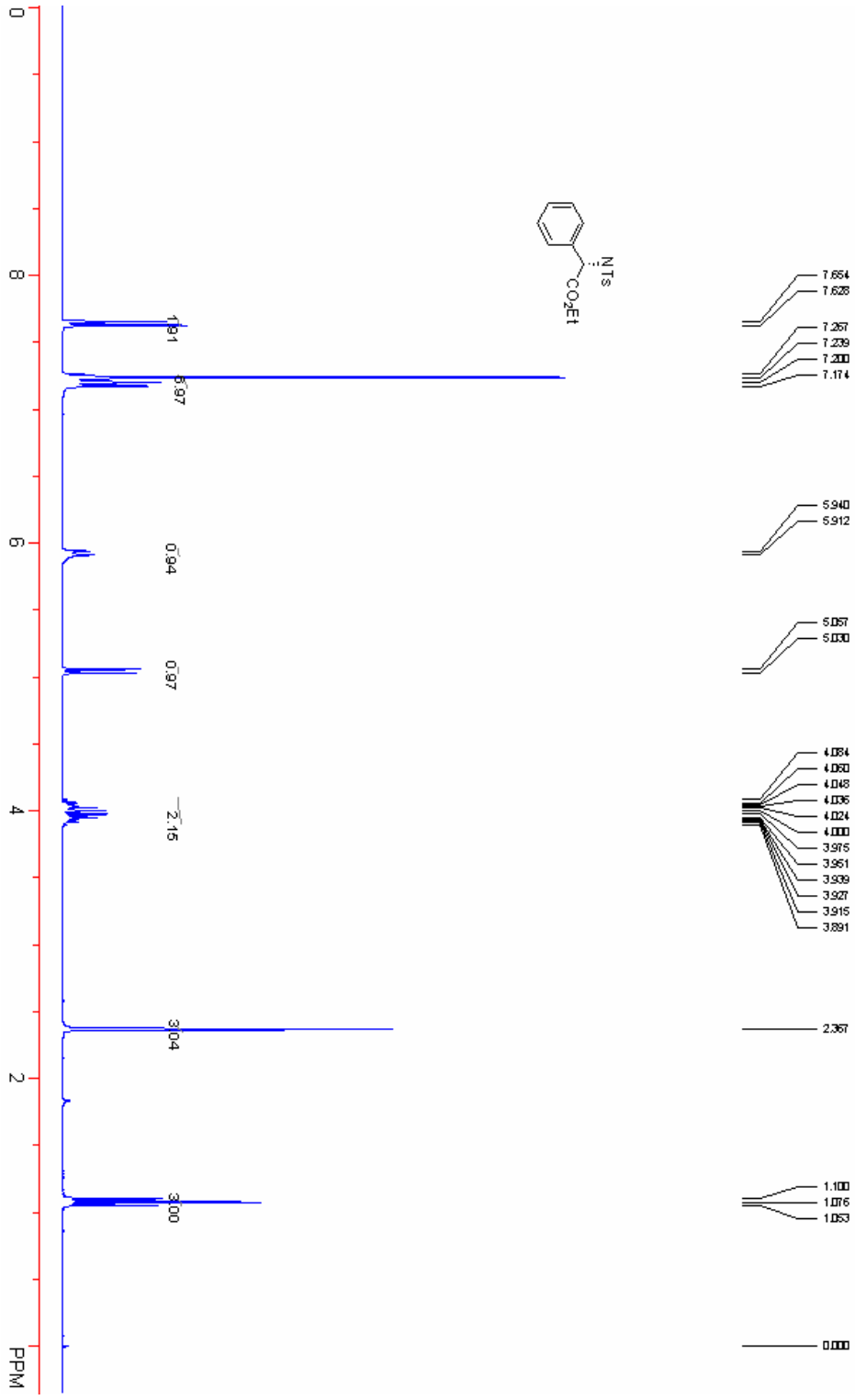
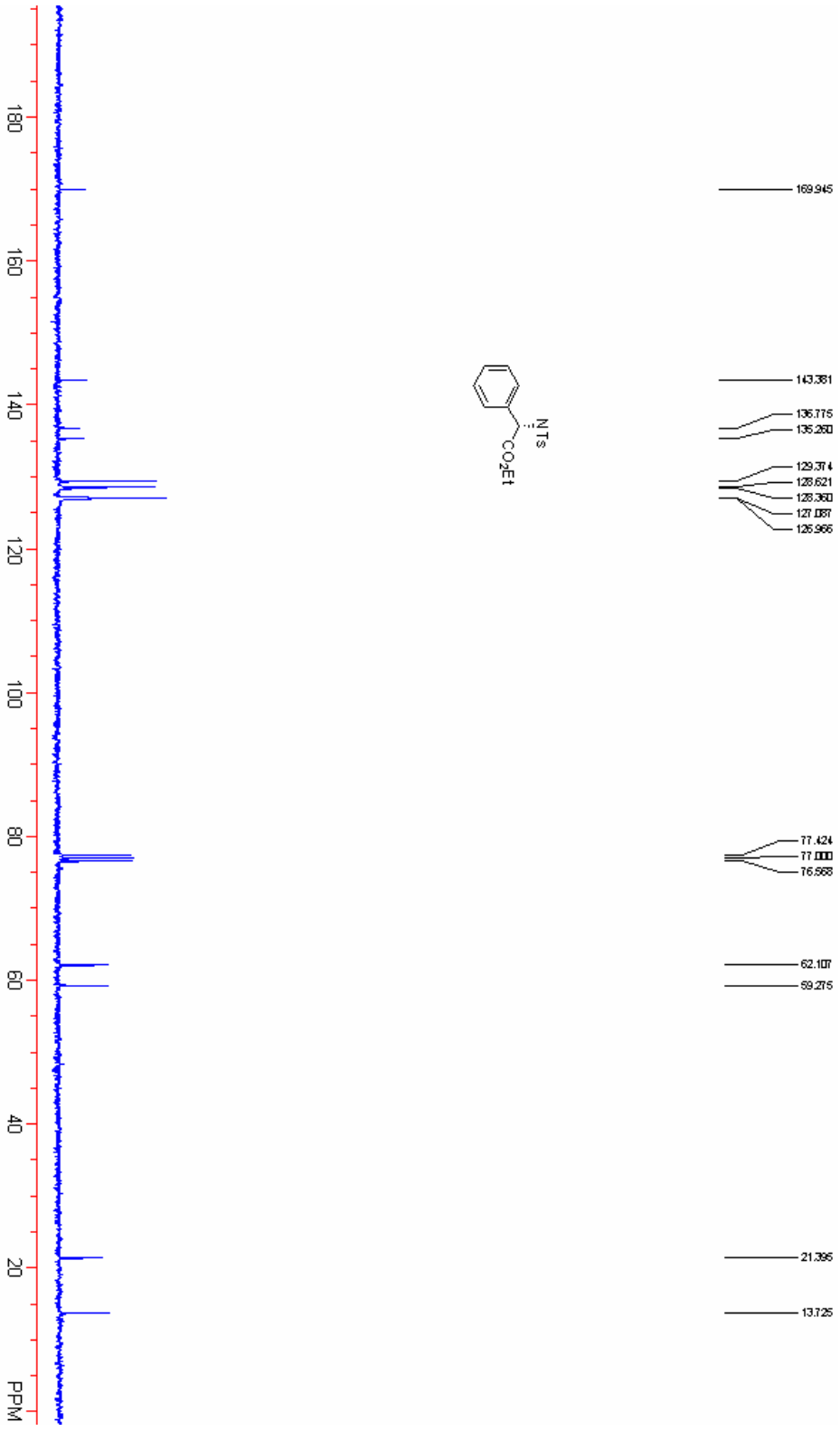
7.735
7.691
7.665
7.644
7.609
7.582
7.474
7.461
7.452
7.442
7.429
7.419
7.321
7.293
7.039
7.012
6.107
6.080
5.235
5.208
4.085
4.072
4.061
4.050
4.024
4.011
4.004
4.001
3.987
3.963
3.952
3.940
3.928
2.220
1.833
1.259
1.092
1.069
1.045
1.031
1.028
3.22
3.07
1.08
2.08
1.00
0.94
2.12
3.11
3.11
8
6
4
2
PPM


NHTs
CO2Et
4i
dhx-6-11-2-C13
Archive directory: /export/home/lu-xy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
169.904
143.320
136.786
132.901
132.833
132.344
129.215
128.558
127.910
127.436
127.032
126.589
126.398
126.276
124.292
77.420
77.000
76.573
62.200
59.491
21.221
13.726
220
200
180
160
140
120
100
80
60
40
20
0
ppm


NTs
CO2Et
7.654
7.628
7.267
7.239
7.200
7.174
5.940
5.912
5.057
5.030
4.084
4.060
4.048
4.036
4.024
4.000
3.975
3.951
3.939
3.927
3.915
3.891
2.367
1.100
1.076
1.053
0.000
1.91
3.97
0.94
0.97
2.15
3.04
3.00
0
2
4
6
8
PPM


180
160
140
120
100
80
60
40
20
PPM
NTs
CO2Et
169.945
143.381
136.775
135.260
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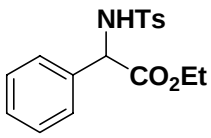
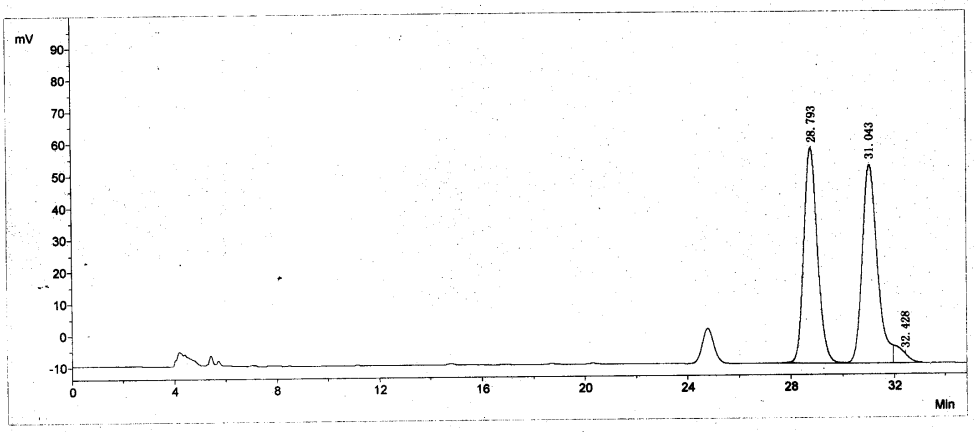
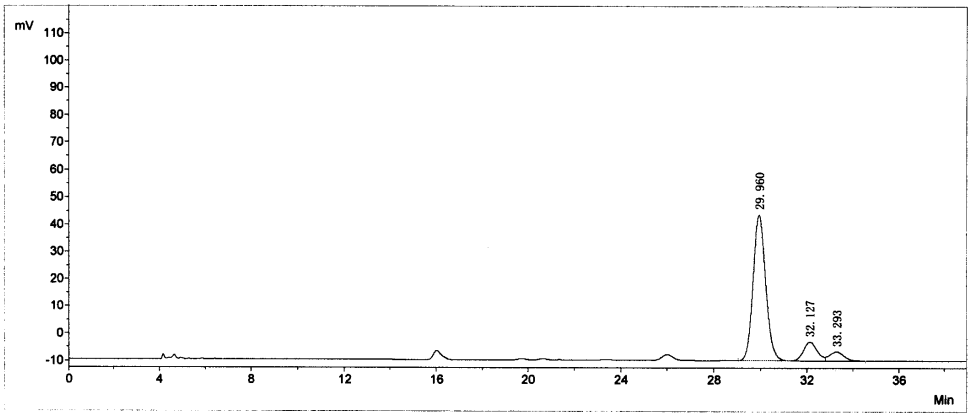
77.000
76.568
62.107
59.275
21.395
13.725



4a

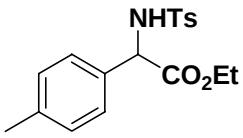


No.	R.Time	PeakHeight	PeakArea	PerCent
1	28.793	67312.0	2467030.6	47.6511
2	31.043	62119.8	2545064.3	49.1584
3	32.428	2994.5	165180.8	3.1905
Total		132426.3	5177275.7	100.0000

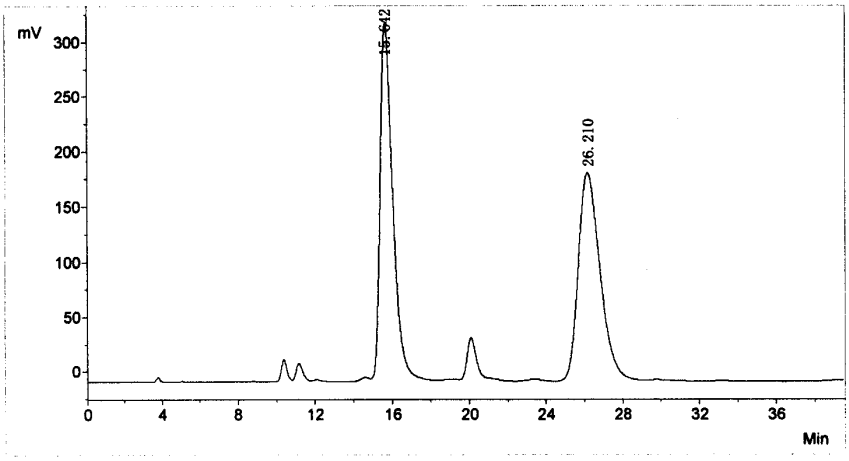


No.	R.Time	PeakHeight	PeakArea	PerCent
1	29.960	53380.3	2002378.7	82.5241
2	32.127	6891.0	279947.5	11.5375
3	33.293	3311.1	144091.5	5.9384
Total		63582.4	2426417.7	100.0000

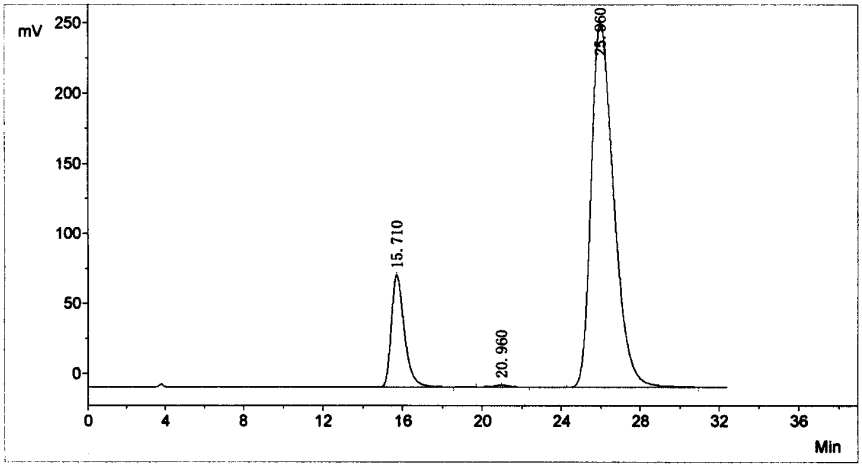
75.5%



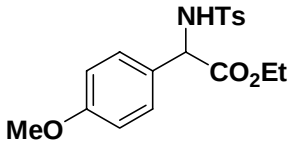
4b



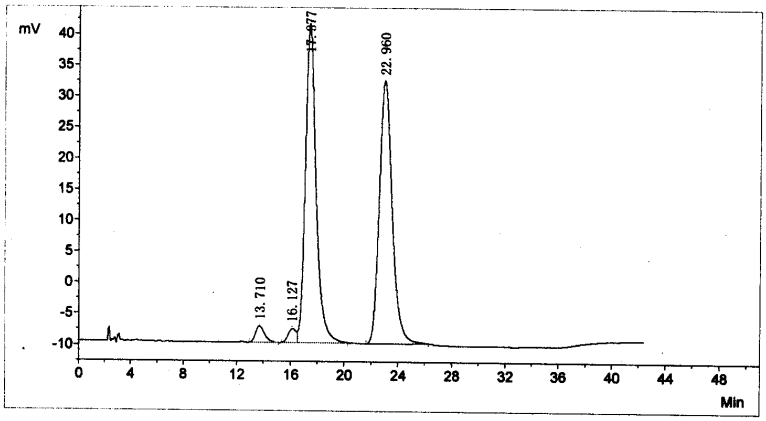
序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1	Unknown	15.642	323083.7	14539617.1	49.3987
2	2	Unknown	26.210	188500.4	14893565.9	50.6013
合计:				511584.2	29433183.0	100.0000



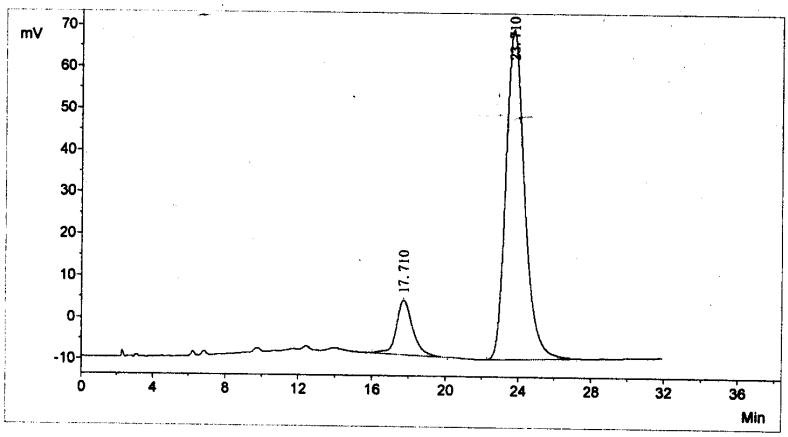
No.	PeakNo	ID.Name	R.Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	15.710	79608.4	3624594.0	14.8547
2	2	Unknown	20.960	1479.1	94118.9	0.3857
3	3	Unknown	25.960	258895.8	20681631.7	84.7596
Total				339983.2	24400344.6	100.0000



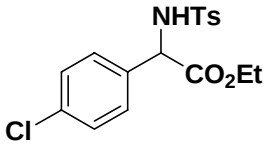
4c



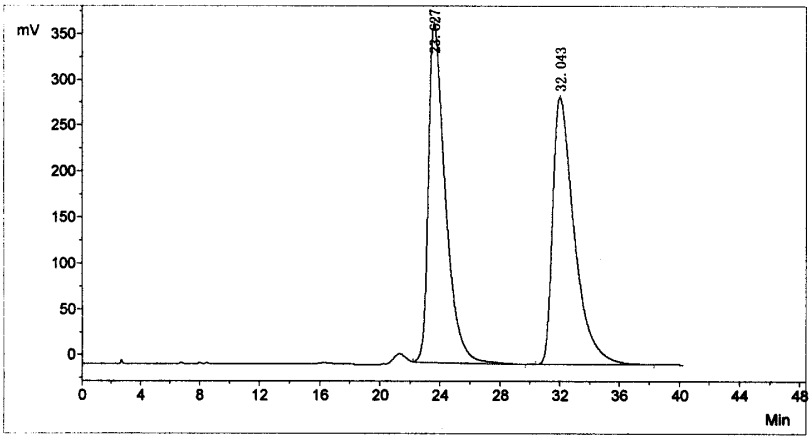
No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	13.710	2637.0	123015.1	2.1000
2	2	Unknown	16.127	2163.4	91405.4	1.5604
3	3	Unknown	17.377	51230.4	2833793.4	48.3759
4	4	Unknown	22.960	42269.1	2809648.6	47.9637
Total				98299.9	5857862.5	100.0000



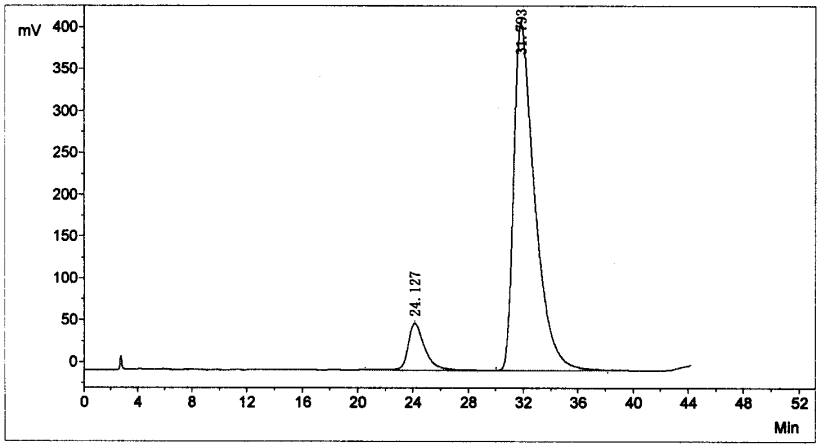
No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	17.710	12983.9	788240.7	12.3636
2	2	Unknown	23.710	78286.8	5587240.2	87.6364
Total				91270.6	6375480.9	100.0000



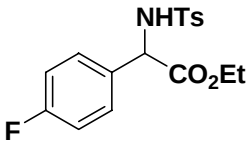
4d



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	23.627	368827.2	28853129.3	49.6827
2	2	Unknown	32.043	290334.4	29221715.0	50.3173
Total				659161.6	58074844.3	100.0000



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	24.127	55335.2	4577706.5	9.4346
2	2	Unknown	31.793	413357.6	43942899.9	90.5654
Total				468692.8	48520606.4	100.0000



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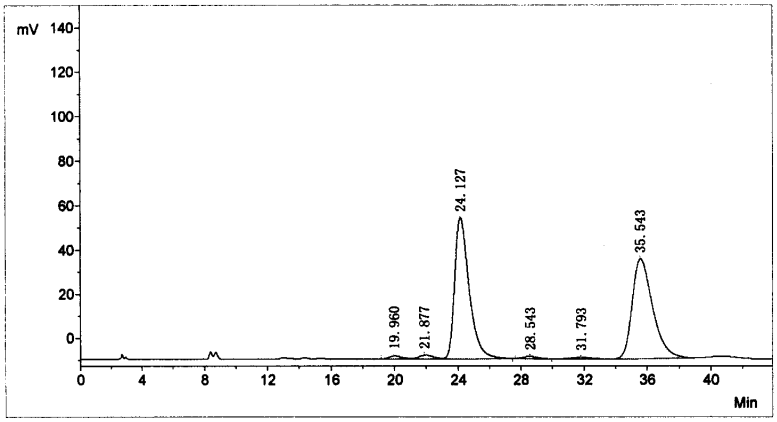
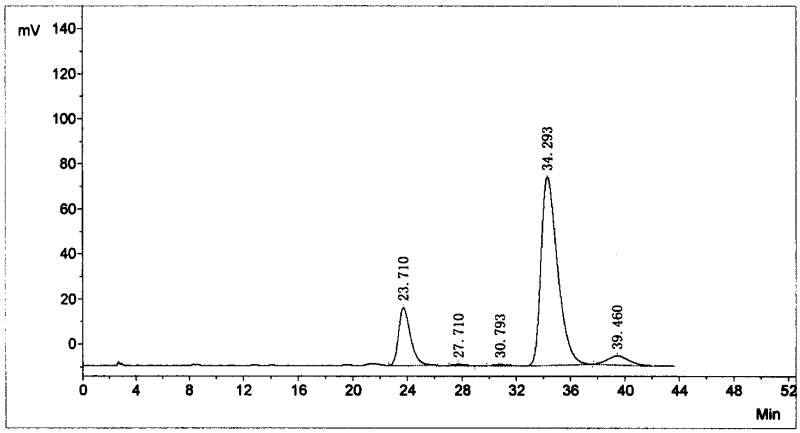
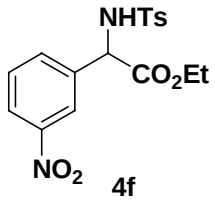


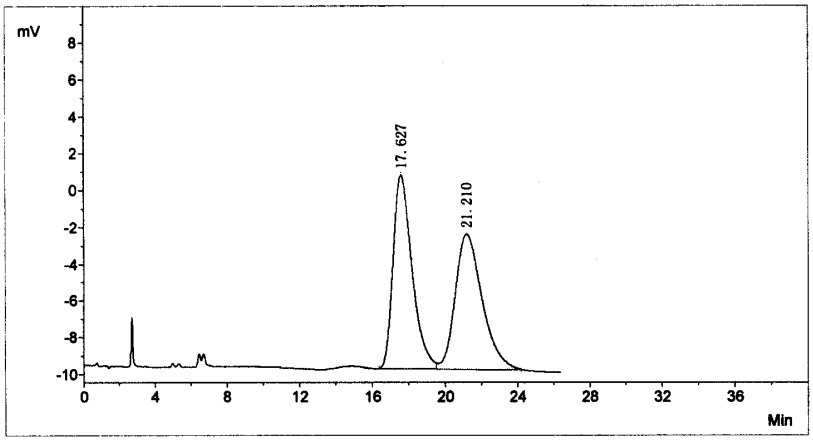
No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	19.960	1303.5	75038.4	0.8979
2	2	Unknown	21.877	1550.0	99430.1	1.1898
3	3	Unknown	24.127	63672.9	4048587.5	48.4458
4	4	Unknown	28.543	922.6	58964.8	0.7056
5	5	Unknown	31.793	681.0	51576.7	0.6172
6	6	Unknown	35.543	45027.6	4023349.6	48.1438
Total				113157.6	8356947.1	100.0000



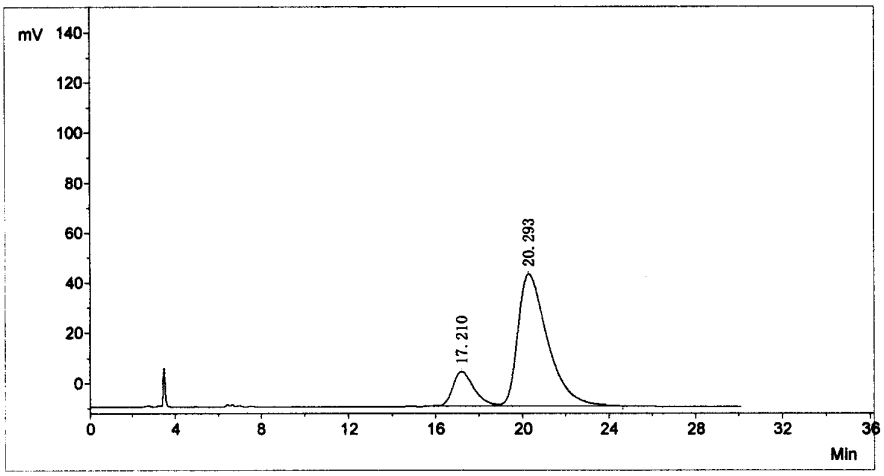
No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	23.710	25440.9	1533424.9	16.9343
2	2	Unknown	27.710	479.1	28314.1	0.3127
3	3	Unknown	30.793	475.6	34963.2	0.3861
4	4	Unknown	34.293	83391.1	7045363.3	77.8051
5	5	Unknown	39.460	3783.8	413082.7	4.5619
Total				113570.6	9055148.2	100.0000



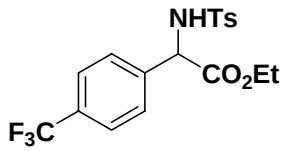
4f



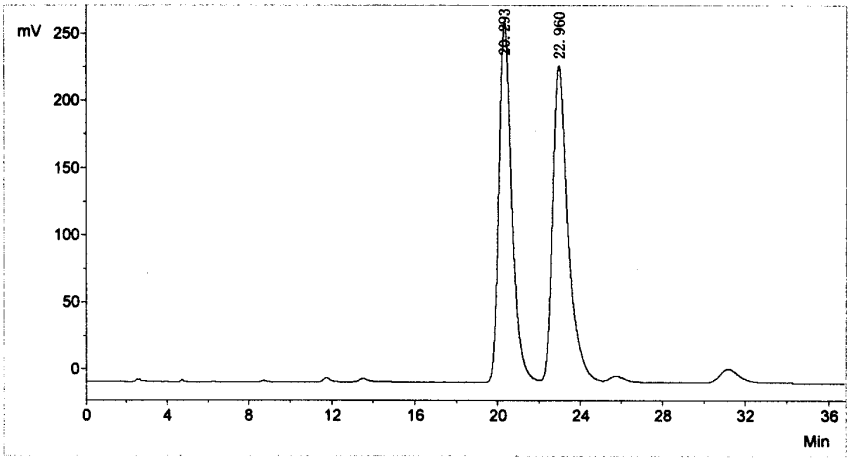
No.	PeakNo	ID.Name	R.Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	17.627	10516.0	761632.6	50.2268
2	2	Unknown	21.210	7348.1	754753.3	49.7732
Total				17864.0	1516385.9	100.0000



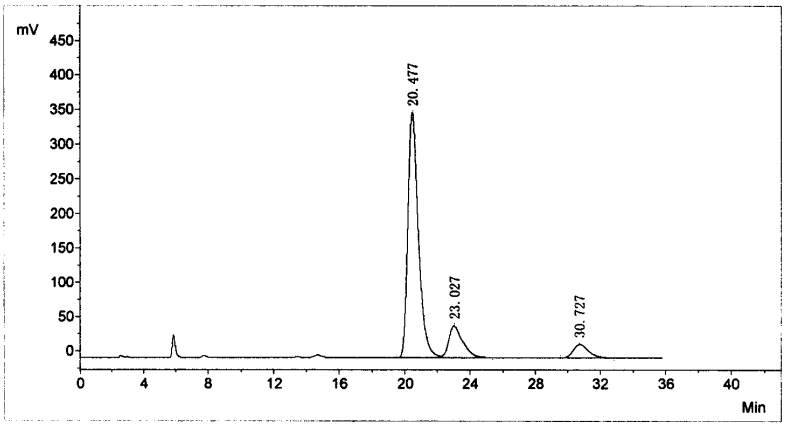
No.	PeakNo	ID.Name	R.Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	17.210	13817.0	948476.5	15.9275
2	2	Unknown	20.293	52559.5	5006493.3	84.0725
Total				66376.5	5954969.8	100.0000



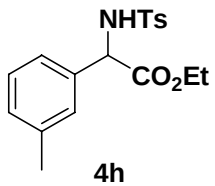
4g



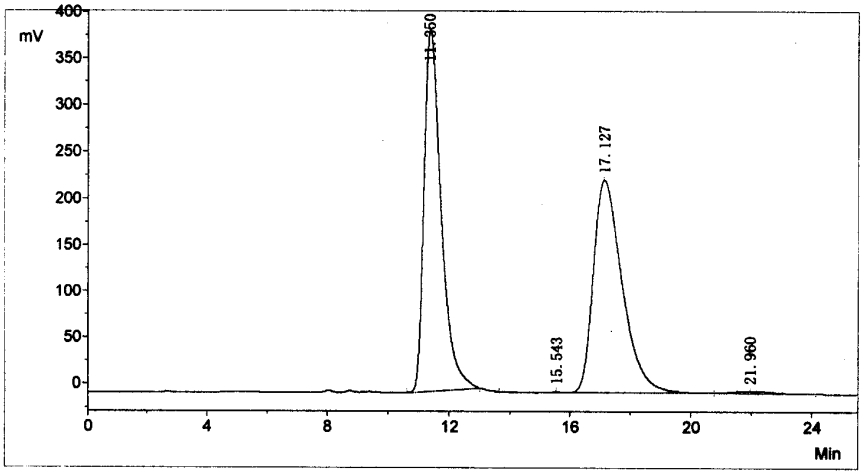
序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1	Unknown	20.293	265960.5	11551735.2	48.9321
2	2	Unknown	22.960	234804.1	12055932.4	51.0679
合计:				500764.6	23607667.6	100.0000



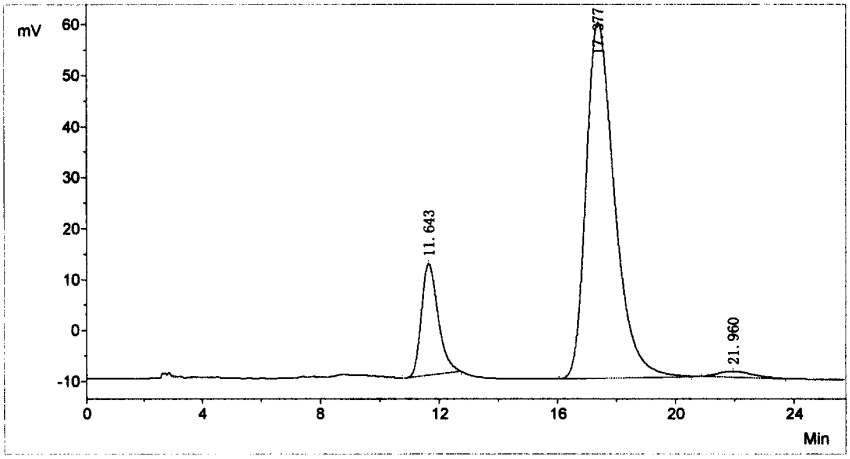
No.	PeakNo	ID.Name	R.Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	20.477	354442.0	15800266.7	79.9847
2	2	Unknown	23.027	46011.7	2697999.8	13.6579
3	3	Unknown	30.727	19565.6	1255852.3	6.3574
Total				420019.3	19754118.8	100.0000



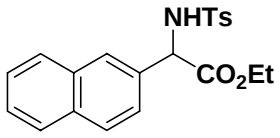
4h



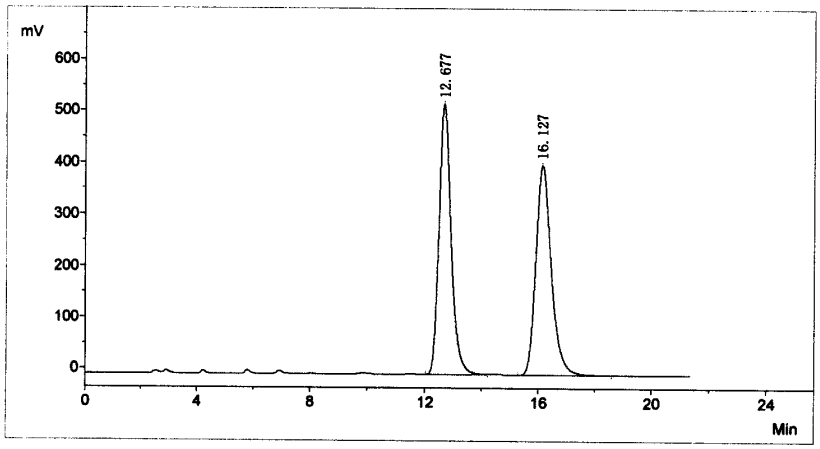
No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	11.350	386433.8	15213020.2	49.6456
2	3	Unknown	17.127	229046.7	15230816.4	49.7036
3	4	Unknown	21.960	2422.4	199426.3	0.6508
Total				617902.8	30643262.9	100.0000



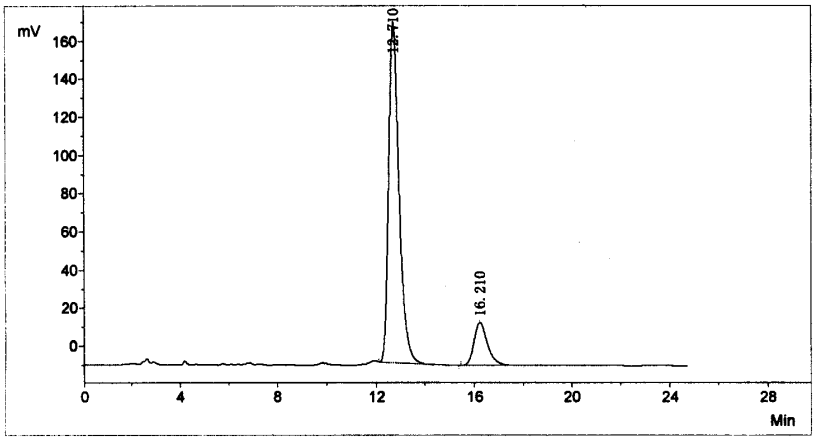
No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	11.643	21807.7	847319.2	15.1104
2	2	Unknown	17.377	69724.1	4674274.0	83.3570
3	3	Unknown	21.960	1091.2	85942.0	1.5326
Total				92623.0	5607535.2	100.0000



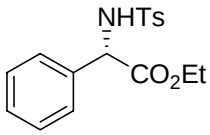
4i



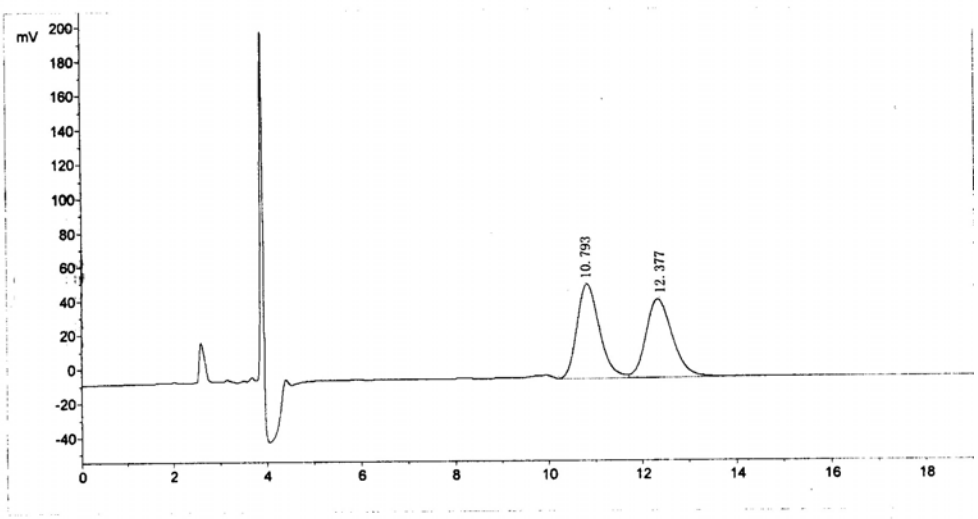
No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	12.677	522397.0	15396693.2	49.8764
2	2	Unknown	16.127	405458.7	15473032.7	50.1236
Total				927855.6	30869725.9	100.0000



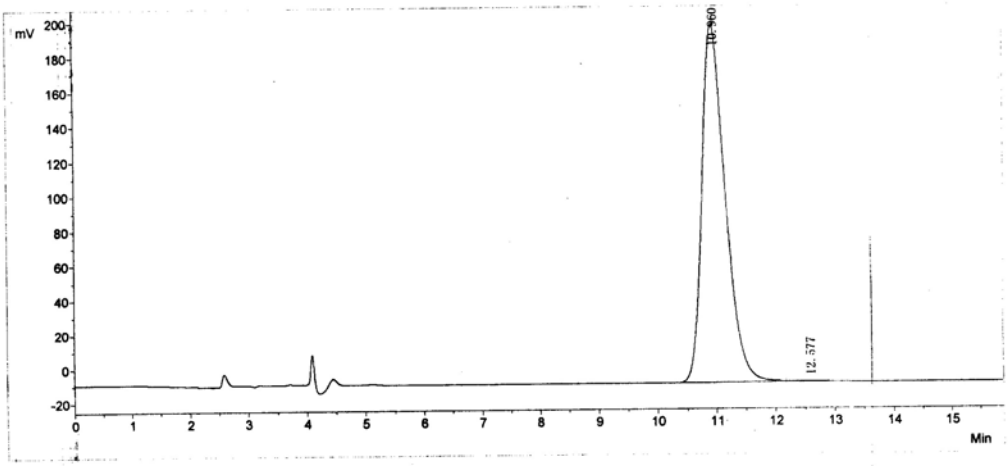
No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	12.710	177838.2	5212192.9	86.0654
2	2	Unknown	16.210	22295.5	843894.4	13.9346
Total				200133.6	6056087.3	100.0000



N-Tosyl-(*S*)-phenylglycine
ethyl ester



No.	PeakNo	R. Time	PeakHeight	PeakArea	PerCent
1	1	10.793	54222.7	1817963.4	50.8072
2	2	12.377	45016.3	1760199.1	49.1928
Total			99239.0	3578162.5	100.0000



No.	PeakNo	R. Time	PeakHeight	PeakArea	PerCent
1	1	10.960	205646.1	5653486.5	99.8639
2	2	12.577	257.5	7703.3	0.1361
Total			205903.6	5661189.8	100.0000