Highly Enantioselective Synthesis of γ-nitro Heteroaromatic Ketones in a Double Sterecontrolled Manner Catalyzed by Bifunctional Thiourea Catalysts Based on Dehydroabietic Amine: A Double Sterecontrolled Approach to Pyrrolidine Carboxylic Acids

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Supporting information

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1.0 General Methods: All reactions were carried out under an argon atmosphere condition unless otherwise noted and solvents were dried according to established procedures. Reactions were monitored by thin layer chromatography (TLC), column chromatography purifications were carried out using silica gel GF254. Proton nuclear magnetic resonance (^{1}HNMR) spectra were recorded on Brucker 300 MHz or 400 MHz spectrometer in $CDCl_3$ unless otherwise noted and carbon nuclear magnetic resonance (^{13}CNMR) spectra were recorded on Brucker 300 MHz or 400 MHz spectrometer in $CDCl_3$ using tetramethylsilane (TMS) as internal standard unless otherwise noted. Data are presented as follows: chemical shift, integration, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, cm = complex multiplet) and coupling constant in Hertz (Hz). Infrared (IR) spectra were recorded on a FT-IR spectrometer. Optical rotations were recorded on a Perkin-Elmer 341 polarimeter. HR-MS was measured with an APEX II 47e mass spectrometer. Melting points were measured on an XT-4 melting point apparatus and were uncorrected. The ee values determination was carried out using chiral high-performance liquid chromatography (HPLC) with Daicel Chiracel AD-H or AS-H column on Waters with a 2996 UV-detector and the dr values determined by 400 Hz ^{1}H NMR.

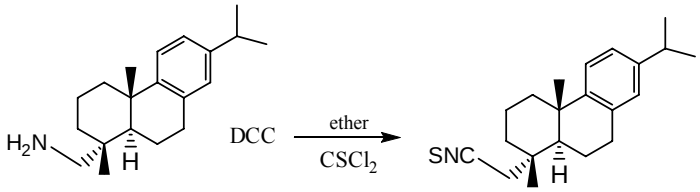
Materials: Dehydroabietic amine, *N,N*'-dicyclohexylcarbodiimide, carbobenzoxy chloride, the ketones, Zinc powder and Lithium diisopropylamide (2 M solution in THF) were commercially available from Acros and Aldrich.

Abbreviations used: EtOAc- ethyl acetate, THF- tetrahydrofuran, MeOH- methanol, AcOH- acetic acid, *i*PrOH- isopropanol, CH_2Cl_2- dichloromethane, LDA- Lithium diisopropylamide and CbzCl- carbobenzoxy chloride.

2.0 Dehydroabietic amine-substituted primary amine-thiourea bifunctional catalysts:

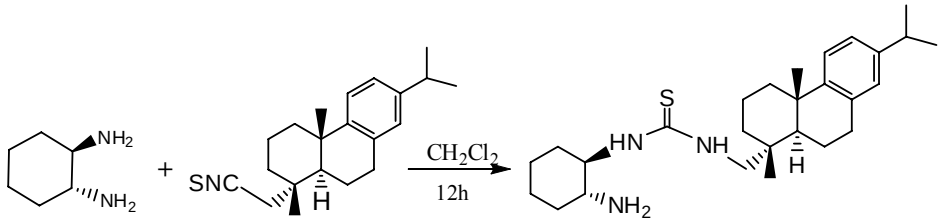
The development of novel chiral catalysts for asymmetric synthesis remains one of the most challenging topics in modern synthetic organic chemistry. A key goal of the synthesis of catalysts is to both maximize the efficiency of using readily available materials and minimize the generation of waste. Thus, the natural organic products and their easily available derivatives as chiral scaffolds for the design and synthesis of catalysts have received much attention so far. Although the natural rosin with excellent structural backbone and well-defined stereocenters is abundant in nature, its easily available derivatives have rarely been developed in synthesis of asymmetric catalysts for asymmetric catalytic reactions to date. In this text, starting from dehydroabietic amine as an easily available and inexpensive natural rosin derivative, a new class of dehydroabietic amine-substituted primary amine-thiourea bifunctional catalysts were designed and synthesized, which have been successfully applied to the highly enantioselective and stereodivergent synthesis of γ-nitro heteroaromatic ketones.

The procedure for synthesis of chiral primary amine-thiourea



285 10mmol 2.85g | 10mmol 2.1g | 327 **1a'** 3.0 g, yield=92%

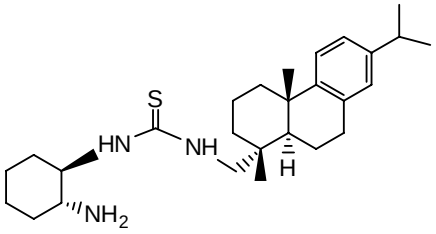
Carbon bisulfide (4.0 mL) and *N,N'*-dicyclohexylcarbodiimide (10 mmol, 2.1 g) were added to a solution of dehydroabietic amine (10 mmol, 2.85 g) in dry ether (35 mL) at 0 °C. The reaction mixture was allowed to warm slowly to room temperature over a period of 3 h and then was stirred for a further 12 h at room temperature. After separation of the precipitated thiourea by filtration, the solvent was removed under reduced pressure. After column chromatography on silica gel eluted with 25% ethyl acetate in hexanes, the isothiocyanate **1a'**as a white solid was isolated in 92% yield (3.0 g).



114 1.14g | **1a** 3.0 g, | (*1R,2R*)-**L** 411 2.6g, yield=64%

The isothiocyanate **1a'**(3.0 g) was added over a period of 1.5 h to a stirred solution of (*R,R*)-1,2-diaminocyclohexane (10 mmol, 1.14 g) in dry dichloromethane (80 mL). The reaction mixture was stirred for a further 12 h at room temperature. The solvent was removed under reduced pressure. After column chromatography on silica gel (eluent, ethyl acetate / MeOH= 6:1), the product (1*R*,2*R*)-**L3** as a white solid was isolated in 64% yield (2.6 g).

(1*R*,2*R*)-L3:1-((1*R*,2*R*)-2-aminocyclohexyl)-3-(((1R,4a*S*,10a*R*)-7-isopropyl-1,4a-dimethyl-1, 2,3,4,4a,9,10,10a-octahydrophenanthren-1-yl)methyl)thiourea

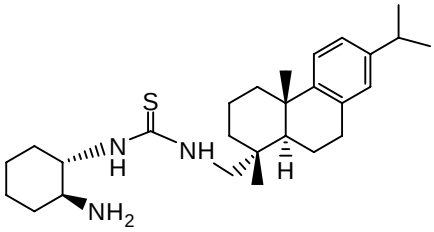


$[\alpha]^{20}_{D}$= +69 (*c*=1.0, $CHCl_3$)； mp 88-89 °C.

^{1}H NMR (300 MHz, [D^6]DMSO): δ 7.38 (br, 1 H), 7.12–7.15 (d, *J* = 8.1 Hz, 1 H), 6.92–6.94 (d, *J* = 8.1 Hz, 1 H), 6.83(s, 1H), 3.74(br, 1 H), 3.38–3.42(m, 2 H), 2.68–2.79(m, 3 H),

2.39–2.48(m, 1 H), 2.23–2.27 (d, *J* = 12.9 Hz, 1 H), 1.97–2.00(m, 1 H), 1.49–1.81(m, 7 H), 1.34–1.38(m, 2 H), 1.24–1.28(m, 2 H), 1.07–1.19(m, 9 H), 0.91–1.04(m, 7 H); ^{13}C NMR (75 MHz, [D^6]DMSO): δ 147.0, 144.9, 134.5, 126.4, 124.1, 123.5, 54.2, 44.5, 37.9, 37.3, 36.99, 35.8, 34.2, 32.9, 31.5, 29.6, 25.1, 24.5, 24.3, 23.9, 18.7, 18.5, 18.2. IR: 3259, 3066, 2929, 2860, 2213, 1549, 1452, 1378, 1234, 1202, 1087, 910, 823, 732, 647 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{27}H_{43}N_3S$ [M +H]$^+$: 442.3250; found: 442.3245, 1.1ppm.

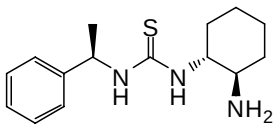
(1*S*,2*S*)-L3:1-((1*S*,2*S*)-2-aminocyclohexyl)-3-(((1R,4a*S*,10a*R*)-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-1-yl)methyl)thiourea



$[\alpha]^{20}_D$= -57 (*c*=1.1, $CHCl_3$)； mp 87-88°C.

^{1}H NMR (300 MHz, [D^6]DMSO): δ 7.40 (br, 1 H), 7.14–7.16 (d, *J* = 8.1 Hz, 1 H), 6.93–6.96 (dd, *J* = 1.5 Hz, 8.1Hz, 1 H), 6.84(s, 1H), 3.75(br, 1 H), 3.35–3.44(m, 2 H), 2.71–2.78(m, 3 H), 2.40–2.46(m, 1 H), 2.24–2.28 (d, *J* = 12.9 Hz, 1 H), 2.00(m, 1 H), 1.47–1.87(m, 7 H), 1.35–1.40(m, 2 H), 1.21–1.25(m, 2 H), 1.13–1.16(m, 9 H), 0.95–0.96(m, 7 H); ^{13}C NMR (75 MHz, [D^6]DMSO): δ 147.0, 144.9, 134.4, 126.4, 124.0, 123.5, 54.2, 44.5, 38.0, 37.4, 37.0, 34.2, 32.8, 31.5, 29.7, 25.1, 24.5, 24.3, 23.9, 23.88, 18.7, 18.5, 18.2. IR: 3355, 3114, 3031, 2919, 2851, 1953, 1683, 1549, 1478, 1380, 1233, 1062, 947, 875, 754, 698, 628, 543 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{27}H_{43}N_3S$ [M +H]$^+$: 442.3250; found: 442.3244, 1.3ppm.

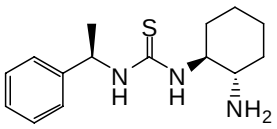
(1*R*,2*R*)-L1:1-((1*R*,2*R*)-2-aminocyclohexyl)-3-((*R*)-1-phenylethyl)thiourea



$[\alpha]^{20}_D$= +36 (*c*=1.1, $CHCl_3$)； mp 59-60 °C.

^{1}H NMR (400 MHz, [D6]DMSO): δ 7.22–7.31(m, 5 H), 5.43(br, 1 H), 3.66(br, 1 H), 2.40–2.50(m, 1 H), 1.94–1.99(m, 1 H), 1.72–1.79(m, 1 H),1.47–1.58(m, 2 H), 1.39(d, *J* = 6.8 Hz, 3 H), 0.84–1.17(m, 4 H); ^{13}C NMR (100 MHz, [D6]DMSO): δ 144.5, 128.3, 126.7, 126.1, 59.6, 54.2, 52.3, 34.4, 31.4, 24.6, 24.5, 22.6. HRMS-ESI (m/z): calcd for $C_{15}H_{23}N_3S$ [M +H]$^+$: 278.1685; found: 278.1684, 0.4ppm.

(1*S*,2*S*)-L1: 1-((1*S*,2*S*)-2-aminocyclohexyl)-3-((*R*)-1-phenylethyl)thiourea



$[\alpha]^{20}_D$= -89 (*c*=1.4, $CHCl_3$)； mp 62-63 °C.

^{1}H NMR (400 MHz, [D^{6}]DMSO): δ 7.11–7.30(m, 5 H), 5.40(br, 1 H), 3.65(br, 1 H), 2.36–2.48(m, 1 H), 1.91–1.96(m, 1 H), 1.74–1.77(m, 1 H),1.55–1.67(m, 2 H), 1.38(d, J = 6.8 Hz, 3 H), 0.99–1.29(m, 4 H); ^{13}C NMR (100 MHz, [D^{6}]DMSO): δ 181.4, 144.4, 128.1, 126.5, 126.0, 59.7, 54.1, 52.1, 34.4, 31.3, 24.4, 24.3, 22.4. HRMS-ESI (m/z): calcd for $C_{15}H_{23}N_3S$ [M +H]$^{+}$: 278.1685; found: 278.1688, 1.1ppm.

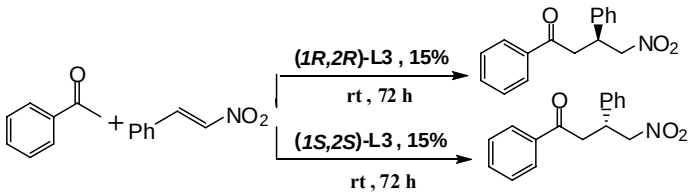
3.0 General procedure for the racemic addition of ketones to nitroalkenes:

Method A: To a stirred solution of ketone (0.4 mmol) in THF(2.0 mL) under argon, LDA (0.4 mmol, 0.2 mL 2 M solution in THF) was added at –78 °C. After the solution was stirred for 0.5 h at -78 °C. Then, nitroalkene (0.2 mmol) was added and stirred for 3 h. Ammonium chloride (saturated aq.) was added to quench the reaction and the mixture was extracted with dichloromethane and dried with sodium sulfate. The solvent was removed at reduced pressure and the residue was purified through column chromatography on silica gel (eluent, ethyl acetate / hexane 1:8) to give the racemic product.

4.0 General procedure for the asymmetric addition of ketones to nitroalkenes:

Method B: To a stirred solution of (1*R*,2*R*)**-L** or (1*S*,2*S*)**-L** (0.03 mmol, 15 mol %), nitroalkene (0.2 mmol) and benzoic acid (0.03 mmol) in dry dichloromethane (1.0 mL) under Ar, ketone (0.6 mmol) was added. The solution was stirred at 0 °C for 1 h, then was stirred at 20 °C. After the reaction was completed (monitored by TLC), the resulting mixture was concentrated under reduced pressure and the residue was purified through column chromatography on silica gel (eluent, ethyl acetate / hexane 1:8) to give the optical pure product. The enantiomeric purity of the product was determined by using HPLC.

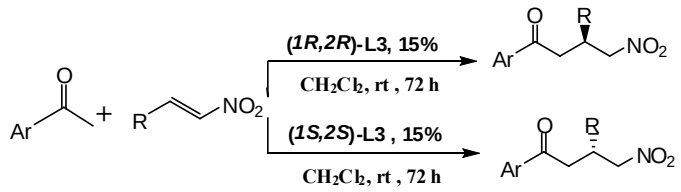
5.0 Table 1. The efficacy of (1*R*,2*R*)-L3 and (1*S*,2*S*)-L3 was initially evaluated in different solvents. [a]



Entry	catalyst	solvent	Yield(%) [b]	ee (%) [c]
1	**(1*R*,2*R*)-L3**	THF	18	82 (***S***)
2	**(1*R*,2*R*)-L3**	ether	23	86 (***S***)
3	**(1*R*,2*R*)-L3**	$CHCl_3$	70	90 (***S***)
4	**(1*R*,2*R*)-L3**	toluene	70	92 (***S***)
5	**(1*S*,2*S*)-L3**	toluene	68	91 (***R***)
6	**(1*R*,2*R*)-L3**	CH_2Cl_2	75	98 (***S***)
7	**(1*S*,2*S*)-L3**	CH_2Cl_2	71	98 (***R***)
8 [d]	**(1*R*,2*R*)-L3**	CH_2Cl_2	96	>99 (***S***)
9 [d]	**(1*S*,2*S*)-L3**	CH_2Cl_2	93	98 (***R***)

[a] The reaction was conducted with *trans*- *β*-nitrostyrene (0.2 mmol), acetophenone (0.6 mmol), and several solvents. [b] Isolated yield. [c] The ee values were determined by HPLC, and the configuration was assigned by comparison of retention time and specific rotation with that of the literature data. [d] The reaction was added PhCOOH (0.15 equiv).

Table 2. The efficacy of (1*R*,2*R*)-L3 and (1*S*,2*S*)-L3 was initially evaluated for enantioselective and stereodivergent synthesis of *γ*-nitro heteroaromatic ketones. [a]



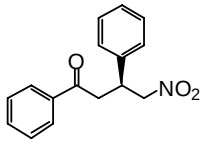
Entry	adduct	Ar	R	Yield(%) [b] (*S*/ *R*)	ee (%) [c] (*S*/ *R*)
1	**3a '**	4-ClPh	Ph	92\ 90	99\ 99
2	**3b '**	4-MeOPh	Ph	81\ 81	>99\ 98
3	**3c '**	3-BrPh	Ph	91\ 87	98\ >99
4	**3d '**	3-MePh	Ph	82\ 83	>99\ 98
5	**3e '**	Ph	4-ClPh	88\ 85	>99\ >99
6	**3f '**	Ph	4-MeOPh	81\ 82	>99\ >99
7	**3g '**	Ph	4-MePh	95\ 92	>99\ 98
8	**3h '**	Ph	3-ClPh	99\ 98	>99\ 99
9	**3i '**	Ph	2-ClPh	99\ 92	>99\ >99
10	**3j '**	Ph	2-MeOPh	81\ 80	99\ 98
11	**3k '**	Ph	2-naphthyl	80\ 78	>99\ >99
12	**2l '**	4-MeOPh	2-furyl	81\ 80	>99\ 98

[a] The reaction was conducted with nitroalkenes (0.2 mmol), aromatic ketones (0.6 mmol), and PhCOOH (0.15 equiv). [b] Isolated yield. [c] The ee values were determined by HPLC, and the configuration was assigned by comparison of retention time and specific rotation with that of the literature data.

6.0 Characterization data

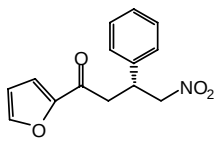
Compounds 3a,[2, 3, 4] **3a'**,[1] **3b'**,[2,3] **3e'**,[3] **3f'**,[3,4] **3g'**,[3] **3i'**,[3,5] **3k'**,[1] **3q**[1] **and 3v**[4] **were known.**

3a: (*S*)-4-nitro-1,3-diphenylbutan-1-one



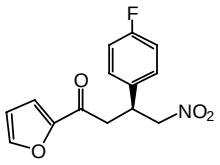
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92–7.94 (d, *J* = 8 Hz, 2 H), 7.57–7.59(m, 1 H), 7.45–7.48(m, 2 H), 7.26–7.34(m, 5 H)，4.82–4.87(dd, *J* = 6.4 Hz, 12.4 Hz, 1 H)，4.67–4.72(dd, *J* = 8.4 Hz, 12.8 Hz, 1H)，4.24(m, 1 H), 3.45–3.48(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.8, 139.1, 133.5, 129.1, 128.7, 128.0, 127.9, 127.5, 79.6, 41.5, 39.3. IR: 3058, 3029, 2920, 1687, 1544, 1440, 1367, 1268, 1224, 1084, 988, 764, 703, 623, 559 cm^{-1}. ESI-MS**:** *m/z* 270 [M^+].

3b: (*R*)-1-(furan-2-yl)-4-nitro-3-phenylbutan-1-one



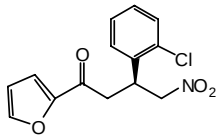
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.56–7.57(dd, *J* = 0.6 Hz, 1.8 Hz 1 H), 7.17–7.36(m, 6 H), 6.52–6.54(dd, *J* = 1.5 Hz, 3.6 Hz 1 H), 4.65–4.84(m, 2 H), 4.14–4.23(m, 1 H), 3.22-3.40(m 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 186.0, 152.3, 146.7, 138.8, 129.0, 127.9, 127.4, 117.5, 112.5, 79.5, 41.2, 39.1. IR: 3352, 2921, 2852, 1655, 1544, 1464, 1381, 1284, 1162, 1091, 1033, 920, 763, 702, 548 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{14}H_{13}NO_4+NH_4^+$: 277.1183; found: 277.1186, 1.1ppm.

3c: (*S*)-3-(4-fluorophenyl)-1-(furan-2-yl)-4-nitrobutan-1-one



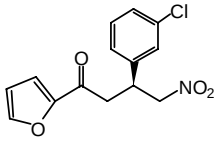
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.57(s,1 H), 7.18–7.27(m, 3 H), 6.98–7.04(m, 2 H), 6.53–6.55(dd, *J* = 1.5 Hz, 3.6 Hz, 1 H), 4.76–4.82(dd, *J* = 6.6 Hz, 12.6 Hz, 1 H), 4.62–4.69(dd, *J* =8.1 Hz, 12.3 Hz, 1 H), 4.14–4.23(m, 1 H), 3.21–3.37(m, 2 H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 185.7, 163.9, 160.6, 152.3, 146.7, 134.5, 129.2, 117.6, 116.1, 115.8, 112.6, 79.5, 41.2, 38.4. IR: 3136, 2923, 2854, 1674, 1552, 1511, 1467, 1380, 1228, 1161, 1029, 838, 767, 554 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{14}H_{12}FNO_4+NH_4^+$: 295.1089; found: 295.1096, 2.4ppm.

3d: (*S*)-3-(2-chlorophenyl)-1-(furan-2-yl)-4-nitrobutan-1-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.58–7.59(dd, *J* = 0.6 Hz, 1.5 Hz, 1 H), 7.39–7.42(m, 1 H), 7.19–7.30(m, 4 H), 6.54–6.55(dd, *J* = 1.8 Hz, 3.6 Hz, 1 H)，4.85–4.88(d, *J* = 6.9 Hz, 2 H)，4.60–4.69(m, 1 H), 3.34–3.48(m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 185.8, 152.3, 146.7, 135.9 133.8, 130.4, 129.0, 128.4, 127.4, 117.6, 112.5, 77.2, 39.6, 35.9. IR: 3135, 2922, 2853, 1673, 1551, 1467, 1378, 1279, 1159, 1035, 881, 759, 679, 593 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{14}H_{12}ClNO_4+NH_4^+$: 311.0793; found: 311.0789, 1.3ppm.

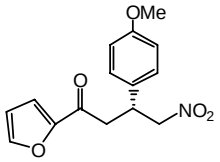
3e: (*S*)-3-(3-chlorophenyl)-1-(furan-2-yl)-4-nitrobutan-1-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.58–7.59 (d, *J* = 0.9 Hz,1 H), 7.40–7.42(dd, *J* = 2.1 Hz, 3.9 Hz, 1 H)，7.21–7.30(m, 4 H), 6.53–6.55(dd, *J* = 1.5 Hz, 3.6 Hz, 1 H)，4.85–4.87(d, *J* = 6.9 Hz,2 H), 4.60–4.69(m, 1 H), 3.34–3.48(m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 185.8, 152.3, 146.8, 135.9, 133.8, 130.4, 129.1, 128.4, 127.4, 117.6, 112.5, 77.2, 39.6, 35.9. IR: 3136, 2922,

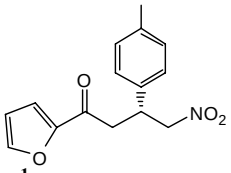
2854, 1674, 1552, 1468, 1379, 1280, 1160, 1036, 882, 760, 679, 593 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{14}H_{12}ClNO_4+NH_4^+$: 311.0793; found: 311.0798, 1.6ppm.

3f: (*R*)-1-(furan-2-yl)-3-(4-methoxyphenyl)-4-nitrobutan-1-one



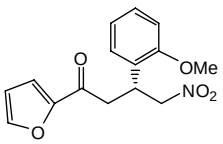
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.57(s 1 H), 7.17–7.20(t, *J* = 4.2 Hz, 3 H), 6.83–6.86 (d, *J* = 8.7Hz,2 H), 6.52–6.54(dd, *J* = 1.5 Hz, 3.3 Hz, 1 H), 4.74–4.80(dd, *J* = 6.6 Hz, 12.3 Hz, 1 H), 4.61–4.67 (dd, *J* = 8.1 Hz, 12.3 Hz, 1 H), 4.09–4.18(m, 1 H), 3.77(s, 3 H), 3.19–3.36(m, 2 H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 186.1, 159.1, 152.4, 146.6, 130.6, 128.5, 117.5, 114.4, 112.5, 79.8, 55.2, 41.4, 38.5. IR: 3134, 2924, 2848, 1673, 1551, 1514, 1466, 1382, 1252, 1182, 1032, 909, 834, 768, 621, 559 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{15}H_{15}NO_5+NH_4^+$: 307.1288; found: 307.1281, 2.3ppm.

3g: (*R*)-1-(furan-2-yl)-4-nitro-3-*p*-tolylbutan-1-one



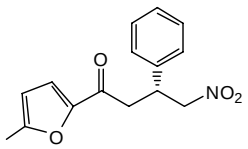
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.56–7.57(dd, *J* = 0.9 Hz, 1.5 Hz ,1 H), 7.11–7.19(m, 5 H)，6.52–6.54(dd, *J* = 1.8 Hz, 3.6 Hz ,1 H), 4.75–4.81(dd, *J* = 6.6 Hz, 12.3 Hz, 1 H)，4.62–4.69(dd, *J* = 8.1 Hz,12.3 Hz 1 H), 4.12–4.17(m, 1 H), 3.20–3.38(m, 2 H), 2.31(s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 186.0, 152.4, 146.6, 137.6, 135.7, 129.7, 127.3, 117.5, 112.5, 79.6, 77.2, 41.3, 38.8, 21.0. IR: 3134, 2921, 2853, 1673, 1551, 1467, 1380, 1280, 1160, 1025, 881, 766, 594, 553 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{15}H_{15}NO_4+NH_4^+$: 291.1339; found: 291.1335, 0.6ppm.

3h: (*R*)-1-(furan-2-yl)-3-(2-methoxyphenyl)-4-nitrobutan-1-one



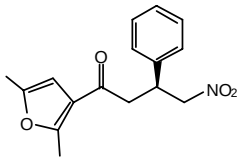
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.56–7.57(t, *J* = 0.6 Hz, 1 H), 7.17–7.27(m, 3 H), 6.87–6.92(t, *J* = 7.5 Hz, 2 H), 6.51–6.53(dd, *J* = 1.5 Hz, 3.6 Hz, 1 H), 4.83–4.85(d, *J* = 7.2 Hz,2 H), 4.33–4.42(m, 1 H), 3.86(s 3 H), 3.31–3.45(m, 2 H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 186.7, 157.2, 152.5, 146.5, 129.4, 129.0, 126.4, 120.9, 117.3, 112.4, 111.0, 77.8, 55.3, 39.5, 35.7. IR: 3134, 2922, 2843, 1674, 1551, 1466, 1379, 1247, 1121, 1025, 882, 758, 595, 515 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{15}H_{15}NO_5+NH_4^+$: 307.1288; found: 307.1292, 1.3ppm.

3i: (*R*)-1-(5-methylfuran-2-yl)-4-nitro-3-phenylbutan-1-one



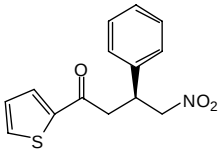
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.23–7.35(m, 5 H),7.09–7.10(d, *J* = 3.6 Hz, 1 H), 6.14–6.15(d, *J* = 3.6 Hz, 1 H), 4.79–4.85(dd, *J* = 6.3 Hz, 12.3 Hz, 1 H), 4.65–4.72(dd, *J* = 8.1 Hz, 12.3 Hz, 1 H), 4.12–4.22(m, 1 H),3.15–3.32(m, 2 H), 2.38(s, 3 H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 185.1, 158.2, 151.1, 138.9, 129.0, 127.9, 127.4, 119.6, 109.3, 79.5, 40.9, 39.3, 14.1. IR: 3123, 2923, 2854, 1665, 1551, 1514, 1434, 1378, 1287, 1207, 1027, 925, 800, 701, 554 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{15}H_{15}NO_4$ $[M+H]^+$: 274.1074; found: 274.1081, 2.6ppm.

3j: (*S*)-1-(2,5-dimethylfuran-3-yl)-4-nitro-3-phenylbutan-1-one



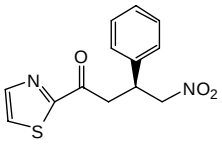
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.24–7.36(m, 5 H),6.15-6.16(d, *J* = 0.9 Hz, 1 H),4.78–4.84(dd, *J* = 6.6 Hz, 12.6 Hz, 1 H), 4.61–4.68(dd, *J* = 8.1 Hz, 12.3 Hz, 1 H), 4.11–4.16(m, 1 H), 3.10–3.13(m, 2 H), 2.51(s, 3 H), 2.24(s, 3 H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 193.0, 157.7, 150.3, 139.3, 129.0, 127.8, 127.4, 121.2, 105.2, 79.6, 43.8, 39.1, 14.4, 13.2. IR: 3063, 2922, 2854, 1673, 1553, 1496, 1378, 1231, 1176, 1008, 960, 765, 702, 622, 554 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{16}H_{17}NO_4$ $[M+H]^+$: 288.1230; found: 288.1235, 1.7ppm.

3k: (*S*)-4-nitro-3-phenyl-1-(thiophen-2-yl)butan-1-one



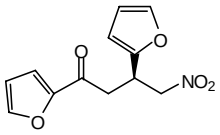
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67–7.68(dd, *J* = 1.2 Hz, 4.0 Hz, 1 H), 7.63–7.64(dd, *J* = 1.2 Hz, 4.0 Hz, 1 H), 7.23–7.34 (m, 5 H), 7.09–7.12(dd, *J* = 4 Hz, 5.2 Hz, 1 H), 4.79–4.85(dd, *J* = 6.4 Hz, 12.4 Hz, 1 H)，4.66–4.71(dd, *J* = 8.0 Hz, 12.4 Hz, 1 H)，4.16–4.20(m, 1 H), 3.30–3.42(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.7, 143.6, 138.8, 134.3, 132.2, 129.1, 128.2, 127.96, 127.4, 79.4, 42.1, 39.5. IR: 3390, 3089, 2921, 2852, 2509, 1954, 1658, 1549, 1413, 1377, 1270, 1235, 1057, 942, 855, 726, 699 cm^{-1}. HRMS-ESI(m/z): calcd for $C_{14}H_{13}NO_3S+NH_4^+$: 293.0954; found: 293.0961, 2.4ppm.

3l: (*S*)-4-nitro-3-phenyl-1-(thiazol-2-yl)butan-1-one



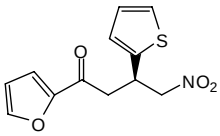
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97–7.98(d, *J* = 3.2 Hz, 1 H), 7.66–7.67(d, *J* = 2.8 Hz, 1 H), 7.22–7.33 (m, 5 H), 4.74–4.79(dd, *J* = 7.2 Hz, 12.4 Hz, 1 H)，4.65–4.70(dd, *J* = 8.0 Hz, 12.4 Hz, 1 H)，4.22–4.25(m, 1 H), 3.72–3.78(dd, *J* = 7.2 Hz, 17.6 Hz, 1 H)，3.53–3.59(dd, *J* = 7.6 Hz, 18.0 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.6, 166.2, 144.8, 138.5, 129.0, 127.9, 127.5, 126.8, 79.6, 41.4, 39.1. IR: 3418, 2928, 2254, 2128, 1652, 1550, 1449, 1379, 1026, 1001, 826, 765, 629 cm^{-1}. HRMS-ES(m/z): calcd for $C_{13}H_{12}N_2O_3S[M+H]^+$: 277.0641; found: 277.0643, 0.7ppm.

3m: (*S*)-1,3-di(furan-2-yl)-4-nitrobutan-1-one



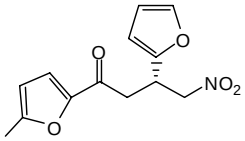
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.60(s, 1 H), 7.23–7.34(m, 2 H)，6.56–6.57(d, *J* = 1.5 Hz 1 H), 6.19–6.30(m 2 H)，4.71–4.83(m, 2 H), 4.25–4.34(m, 1 H), 3.24–3.44(m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 185.5, 152.2, 151.6, 146.8, 142.3, 117.7, 112.5, 110.5, 107.3, 77.2, 38.7, 33.0. IR: 3133, 2922, 2853, 1674, 1553, 1467, 1379, 1282, 1159, 1015, 914, 741, 596 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{12}H_{11}NO_5+NH_4^+$: 267.0975; found: 267.0982, 2.6ppm.

3n: (*S*)-1-(furan-2-yl)-4-nitro-3-(thiophen-2-yl)butan-1-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.59–7.60(d, J=0.9Hz,1 H), 7.19–7.23(m, 2 H)，6.92–6.96(m, 2 H), 6.54–6.56(dd, *J* = 1.8 Hz, 3.6 Hz, 1 H)，4.66–4.85(m, 2 H), 4.47–4.56(m, 1 H), 3.28–3.46(m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 185.5, 152.2, 146.8, 141.5, 127.1, 125.7, 124.8, 117.7, 112.6, 79.8, 41.9, 34.6. IR: 3133, 2921, 2853, 1673, 1553, 1467, 1379, 1250, 1160, 1034, 909, 846, 768, 706, 596, 529 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{12}H_{11}NO_4S+NH_4^+$: 283.0747; found: 283.0742, 1.8ppm.

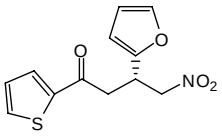
3o: (*R*)-3-(furan-2-yl)-1-(5-methylfuran-2-yl)-4-nitrobutan-1-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.33–7.34(dd, *J* = 0.9 Hz, 1.8 Hz, 1 H), 7.14–7.15(d, *J* = 3.6 Hz, 1 H), 6.27–6.29(dd, *J* = 1.8 Hz, 3.3 Hz, 1 H), 6.17–6.18(m, 2 H), 4.70–4.82(m, 2 H),4.23–4.32(m, 1 H), 3.17–3.36(m, 2 H), 2.40(s, 3 H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 184.7, 158.4, 151.7, 151.0, 142.3, 119.8, 110.5, 109.3, 107.2, 77.2, 38.4, 33.2, 14.1. IR: 3122, 2924,

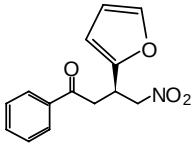
2855, 1664, 1553, 1514, 1377, 1288, 1209, 1146, 1021, 917, 802, 741, 599, 529 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{13}H_{13}NO_5+NH_4^+$: 281.1132; found: 281.1137, 1.8ppm.

3p: (*R*)-3-(furan-2-yl)-4-nitro-1-(thiophen-2-yl)butan-1-one



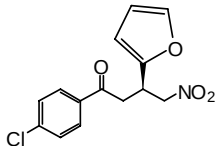
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.72–7.73 (d, *J* = 1.2 Hz, 1 H), 7.66–7.68(dd, *J* = 1.2 Hz, 1 H), 7.33–7.34(dd, *J* = 0.6 Hz, 2.4 Hz, 1 H), 7.13-7.16(dd, *J*=3.9 Hz, 5.1 Hz, 1 H),6.28-6.29 (dd, *J* = 1.8 Hz, 3.3 Hz, 1 H)，6.18–6.19(dd, *J* = 0.6 Hz, 2.7 Hz, 1 H)，4.71–4.84(m, 2 H), 4.26–4.35(m, 1 H), 3.32–3.50(m, 2 H); ^{13}C NMR (75MHz, $CDCl_3$): δ 189.4, 151.6, 143.4, 142.4, 134.5, 132.4, 128.3, 110.5, 107.3, 77.1, 39.6, 33.3. IR: 3111, 2922, 2854, 1661, 1553, 1415, 1377, 1238, 1146, 1014, 856, 732, 598, 494 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{12}H_{11}NO_4S+NH_4^+$: 283.0747; found: 283.0744, 1.1ppm.

3q: (*S*)-3-(furan-2-yl)-4-nitro-1-phenylbutan-1-one



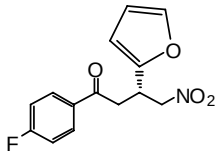
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95–7.97 (d, *J* = 7.6 Hz, 2 H), 7.50–7.62(m, 1 H), 7.46–7.48(m, 2 H), 7.35(m, 1 H), 6.29–6.30(d, *J* = 3.2 Hz, 1 H), 6.19–6.20(d, *J* = 3.2 Hz, 1 H), 4.73–4.84(m, 2 H)，4.33–4.36 (m, 1 H), 3.41–3.56(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.5, 151.9, 142.3, 136.3, 133.6, 128.7, 128.0, 110.5, 107.2, 77.2, 38.96, 33.2. IR: 3121, 3062, 2918, 1685, 1596, 1553, 1505, 1448, 1377, 1213, 1183, 1012, 917, 749, 691, 599 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{14}H_{13}NO_4+NH_4^+$: 277.1183; found: 277.1187, 1.4ppm.

3r: (*S*)-1-(4-chlorophenyl)-3-(furan-2-yl)-4-nitrobutan-1-one



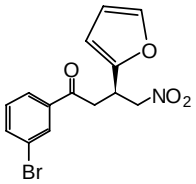
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.88–7.90 (d, *J* = 8.4 Hz,2 H), 7.44–7.47(d, *J* = 9.0 Hz,2 H)，7.33–7.34(d, *J* = 1.2 Hz,1 H), 6.29–6.31(dd, *J* = 2.1 Hz, 3.3 Hz, 1 H)，6.18–6.19(d, *J* = 3.3 Hz, 1 H)，4.71–4.84(m, 2 H), 4.30–4.36(m, 1 H), 3.36–3.54(m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 195.4, 151.7, 142.3, 140.2, 134.5, 129.5, 129.1, 110.5, 107.3, 77.2, 38.9, 33.1. IR: 3355, 3122, 2921, 2853, 1687, 1590, 1553, 1505, 1402, 1377, 1212, 1092, 1012, 918, 818, 740, 598, 530. HRMS-ESI (*m/z*): calcd for $C_{14}H_{12}ClNO_4+NH_4^+$: 311.0793; found: 311.0788, 1.6ppm.

3s: (*R*)-1-(4-fluorophenyl)-3-(furan-2-yl)-4-nitrobutan-1-one



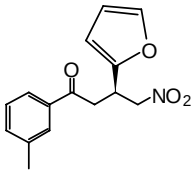
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.96–7.99(m, 2 H), 7.34–7.35(d, *J* = 1.8 Hz, 1 H)，7.12–7.18(m, 2 H), 6.20–6.31(dd, *J* = 1.8 Hz, 3.3 Hz, 1 H)，6.18–6.20(d, *J* = 3.3 Hz, 1 H)，4.71–4.84(m, 2 H), 4.30–4.34(m, 1 H), 3.36–3.54(m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 194.9, 167.8, 164.4, 151.8, 142.3, 130.8, 130.7, 116.1, 115.8, 110.5, 107.2, 77.2, 55.5, 38.8, 33.2. IR: 3350, 3030, 2915, 1671, 1592, 1553, 1446, 1376, 1224, 1085, 1012, 884, 760, 612, 599. HRMS-ESI (*m/z*): calcd for $C_{14}H_{12}FNO_4$+NH_4^+: 295.1093; found: 295.1090, 0.8ppm.

3t: (*S*)-1-(3-bromophenyl)-3-(furan-2-yl)-4-nitrobutan-1-one



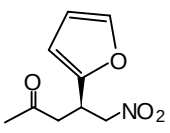
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94(s, 1 H), 7.86–7.88(d, *J* = 6.0 Hz, 1 H)，7.71–7.73(d, *J* = 7.8 Hz, 1 H), 7.34–7.39(m, 2 H)，6.29–6.31(t, *J* = 3.0 Hz, 1 H)，6.19–6.20(d, *J* = 3.3 Hz, 1 H)，4.71–4.83(m, 2 H), 4.28–4.37(m, 1 H), 3.36–3.55(m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 195.2, 151.6, 142.4, 137.9, 136.5, 131.1, 130.4, 126.6, 123.1, 110.5, 107.3, 77.1, 39.1, 33.0. IR: 3362, 3066, 2120, 2852, 1689, 1553, 1505, 1421, 1377, 1285, 1208, 1146, 1071, 1014, 915, 787, 740, 682, 598. HRMS-ESI (*m/z*): calcd for $C_{14}H_{12}BrNO_4$+NH_4^+: 355.0288; found: 355.0285, 0.8ppm.

3u: (*S*)-3-(furan-2-yl)-4-nitro-1-m-tolylbutan-1-one



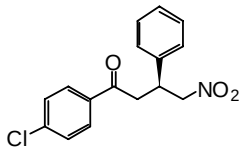
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.73–7.76(d, *J* = 6.6 Hz, 2 H), 7.33–7.42(m, 3 H), 6.28–6.30(dd, *J* = 2.1 Hz, 3.3 Hz, 1 H),6.18–6.19(d, *J* = 3.3 Hz, 1 H), 4.71–4.84(m, 2 H), 4.31–4.38(m, 1 H), 3.37–3.55(m, 2 H), 2.41(s,3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 196.7, 152.0, 142.3, 138.6, 136.3, 134.4, 128.6, 128.5, 125.3, 110.5, 107.2, 77.3, 39.0, 33.2,21.3. IR: 3122, 3030, 2921, 2858, 1684, 1554, 1506, 1430, 1378, 1246, 1160, 1077, 1014, 917, 883, 786, 740, 690, 599. HRMS-ESI (*m/z*): calcd for $C_{15}H_{15}NO_4$+Na^+: 296.0893; found: 296.0888, 1.7ppm.

3v: (S)-4-(furan-2-yl)-5-nitropentan-2-one



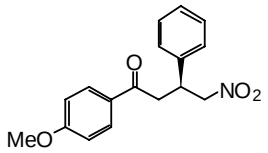
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.33–7.34(dd, *J* = 0.9 Hz, 1.8 Hz, 1 H), 6.29–6.31(dd, *J* = 2.1 Hz, 3.3 Hz, 1 H), 6.14–6.15(d, *J* = 3.3 Hz, 1 H),4.62–4.73(m, 2 H), 4.06–4.15(m, 1 H), 2.85–3.03(m, 2 H) ,2.18(s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 205.0, 151.7, 142.3, 110.5, 107.1, 77.2, 43.5, 32.9, 30.2. IR: 3413, 3122, 2922, 2854, 2519, 1934, 1714, 1551, 1506, 1426, 1372, 1148, 1080, 1012, 915, 809, 738, 648, 597. HRMS-ESI (*m/z*): calcd for $C_9H_{11}NO_4$+NH_4^+: 215.1026; found: 215.1030, 1.9ppm.

3a': (*S*)-1-(4-chlorophenyl)-4-nitro-3-phenylbutan-1-one



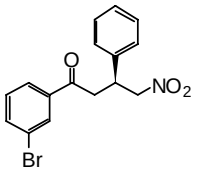
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85–7.87 (d, *J* = 8 Hz, 2 H), 7.26–7.44(m, 7 H), 4.80–4.85(dd, *J* = 7.2 Hz, 12.4 Hz, 1H)，4.67–4.71(dd, *J* = 8.4 Hz, 12.8 Hz, 1H)，4.18–4.25 (m, 1H), 3.37–3.47(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.6, 140.1, 138.9, 134.7, 129.4, 129.1, 129.0, 127.9, 127.4, 79.5, 41.5, 39.3. IR: 3064, 2921, 1685, 1589, 1551, 1492, 1378, 1205, 1092, 993, 822, 764, 701, 529 cm^{-1}. ESI-MS**:** *m/z* 304 [M^+].

3b': (*S*)-1-(4-methoxyphenyl)-4-nitro-3-phenylbutan-1-one



^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89–7.91 (d, *J* = 8.4 Hz, 2 H), 7.25–7.35(m, 5 H), 6.91–6.93 (d, *J* = 8.8 Hz, 2 H), 4.82–4.87(dd, *J* = 6.4 Hz, 12.4 Hz, 1 H)，4.66–4.71(dd, *J* = 8.4 Hz, 12.4 Hz, 1 H)，4.20–4.23 (m, 1 H), 3.87(s, 3 H), 3.33–3.45(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.3, 163.9, 139.3, 130.3, 129.5, 129.0, 127.8, 127.4, 79.6, 55.5, 41.2, 39.5. IR: 3062, 2923, 2845, 1675, 1600, 1551, 1511, 1420, 1377, 1262, 1172, 1027, 833, 701, 557 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{17}H_{17}NO_4$ [M +H]$^+$: 300.1230; found: 300.1233, 1.0ppm.

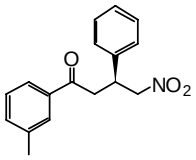
3c': (*S*)-1-(3-bromophenyl)-4-nitro-3-phenylbutan-1-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 8.03(s, 1 H), 7.83–7.85(d, *J* = 6.0 Hz, 1 H), 7.69–7.71(m, 1 H), 7.24–7.36(m, 6 H)，4.79–4.84(dd, *J* = 6.9 Hz, 12.3 Hz, 1 H)，4.66–4.72(dd, *J* = 7.5 Hz, 12.3 Hz, 1 H)，4.17–4.26(m, 1 H),3.41–3.44(m, 2 H); ^{13}C NMR (75MHz, $CDCl_3$): δ 195.5, 138.8, 138.0, 136.4, 131.1, 130.3, 129.1, 128.0, 127.4, 126.5, 79.4, 41.6, 39.1. IR: 3064, 3031, 2922, 2854, 1689, 1551, 1420, 1377, 1201, 1070, 995, 765, 700, 559 cm^{-1}. HRMS-ESI (*m/z*): calcd for

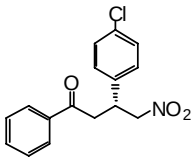
$C_{16}H_{14}BrNO_3$+NH_4^+: 365.0495; found: 365.0491, 1.1ppm.

3d': (*S*)-4-nitro-3-phenyl-1-m-tolylbutan-1-one



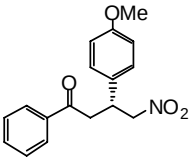
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71–7.72 (m, 2 H), 7.25–7.40(m, 7 H), 4.81–4.86(dd, *J* = 6.8 Hz, 12.8 Hz, 1 H)，4.67–4.71(dd, *J* = 8.4 Hz, 12.8 Hz, 1 H)，4.21–4.24 (m, 1H), 3.38–3.50(m, 2 H) ， 2.40 (s, 3 H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.99, 139.2, 138.6, 136.5, 134.3, 129.0, 128.6, 128.5, 127.8, 127.4, 125.2, 79.6, 41.6, 39.3, 21.3. IR: 3031, 2920, 1683, 1551, 1431, 1378, 1273, 1160, 980, 767, 698, 562 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{17}H_{17}NO_3$+NH_4^+: 301.1547; found: 301.1547, 0.0ppm.

3e': (*R*)-3-(4-chlorophenyl)-4-nitro-1-phenylbutan-1-one



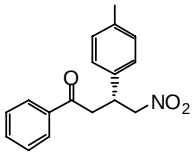
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88–7.91 (m, 2 H), 7.55–7.59 (m, 1 H), 7.43–7.50(m, 2 H), 7.28–7.31 (m, 2 H), 7.19–7.25 (m, 2 H), 4.78–4.82(dd, *J* = 6.4 Hz, 12.4 Hz, 1 H)，4.62–4.67(dd, *J* = 8.4 Hz, 12.8 Hz, 1 H)，4.16–4.24(m, 1 H), 3.36–3.47(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.5, 137.5, 136.2, 133.7, 129.2, 128.9, 128.8, 128.0, 79.3, 41.3, 38.7. IR: 3063, 2922, 2853, 2255, 1684, 1551, 1492, 1445, 1376, 1228, 1093, 1008, 909, 828, 733, 688, 546 cm^{-1}. ESI-MS**:** *m*/*z* 304 [M^+].

3f': (*R*)-3-(4-methoxyphenyl)-4-nitro-1-phenylbutan-1-one



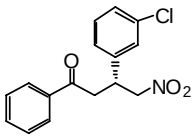
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89–7.91 (d, *J* = 7.6 Hz, 2 H), 7.54–7.58(m, 1 H), 7.42–7.46(m, 2 H), 7.17–7.19(d, *J* = 7.6 Hz, 2 H), 6.83–6.85(d, *J* = 6.8 Hz, 2 H), 4.76–4.80(dd, *J* = 6.4 Hz, 12.0 Hz, 1 H)，4.60–4.65(m, 1 H)，4.14–4.18 (m, 1 H), 3.75(s, 3 H), 3.39–3.47(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.9, 159.0, 136.4, 133.5, 130.9, 128.7, 128.5, 127.99, 114.4, 79.8, 55.2, 41.6, 38.6. IR: 3033, 2919, 2839, 1684, 1610, 1551, 1514, 1446, 1377, 1251, 1181, 1032, 832, 754, 692, 555 cm^{-1}. ESI-MS**:** *m*/*z* 300 [M^+].

3g': (*R*)-4-nitro-1-phenyl-3-p-tolylbutan-1-one



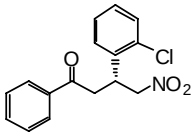
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91–7.93 (d, *J* = 8.4Hz, 2 H), 7.55–7.59(m, 1 H), 7.47–7.43(m, 2 H), 7.12–7.25(m, 4 H)，4.78–4.83(dd, *J* = 6.4 Hz, 12.4 Hz, 1 H)，4.63–4.68(dd, *J* = 8.0 Hz, 12.8 Hz, 1 H)，4.17–4.22(dd, *J* = 7.2 Hz, 14 Hz, 1 H),3.37–3.49(m, 2 H),2.307(s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.9, 137.5, 136.4, 136.0, 133.5, 129.7, 128.7, 128.0, 127.3, 79.7, 41.6, 38.9, 21.0. IR: 3058, 2922, 2862, 1685, 1551, 1516, 1446, 1377, 1270, 1225, 998, 817, 755, 691, 551 cm^{-1}. ESI-MS**:** *m*/*z* 284 [M^+].

3h': (*R*)-3-(3-chlorophenyl)-4-nitro-1-phenylbutan-1-one



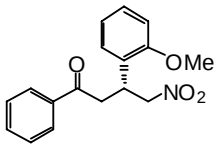
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94–7.96 (d, *J* = 7.2 Hz, 2 H), 7.57–7.61(m, 1 H), 7.41–7.49(m, 3H), 7.20–7.31(m, 3 H)，4.84–4.90(m, 2 H)，4.66–4.73(m, 1 H) 3.51–3.62(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.7, 136.2, 133.8, 133.6, 130.5, 129.0, 128.7, 128.5, 128.0, 127.4, 77.5, 39.9, 36.1. IR: 3063, 2918, 2852, 1684, 1551, 1477, 1444, 1377, 1228, 1038, 998, 755, 689, 560 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{16}H_{14}ClNO_3$+NH_4^+: 321.1000; found: 321.0992, 2.5ppm.

3i': (*R*)-3-(2-chlorophenyl)-4-nitro-1-phenylbutan-1-one



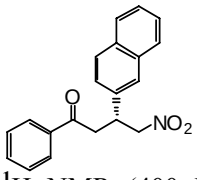
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92–7.94 (m, 2 H), 7.55–7.59(m, 1 H), 7.39–7.47(m, 3 H), 7.19–7.29(m, 3 H)，4.85–4.87(m, 2 H)，4.65–4.71(m, 1 H) 3.50–3.60(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.7, 136.2, 133.8, 133.6, 130.4, 129.0, 128.7, 128.4, 128.0, 127.4, 77.5, 39.8, 36.1. IR: 3063, 2918, 1684, 1596, 1551, 1477, 1442, 1376, 1228, 1039, 998, 755, 689, 560 cm^{-1}. ESI-MS**:** *m*/*z* 304 [M^+].

3j': (*R*)-3-(2-methoxyphenyl)-4-nitro-1-phenylbutan-1-one



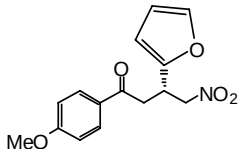
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91–7.93 (m, 2 H), 7.53–7.57 (m, 1 H), 7.42–7.46(m, 2 H), 7.18–7.25 (m, 2 H), 6.86–6.91 (m, 2 H), 4.83–4.86(m, 2 H)，4.38–4.42(m, 1 H)，3.85(s, 3 H), 3.51–3.53(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.6, 157.1, 136.6, 133.3, 129.5, 128.9, 128.6, 128.0, 126.6, 120.9, 110.98, 77.8, 55.3, 39.7, 35.9. IR: 3063, 2923, 2852, 1684, 1598, 1550, 1494, 1445, 1377, 1246, 1120, 1025, 754, 690 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{17}H_{17}NO_4$ +NH_4^+: 317.1496; found: 317.1498, 0.6ppm.

3k': (*R*)-3-(naphthalen-2-yl)-4-nitro-1-phenylbutan-1-one



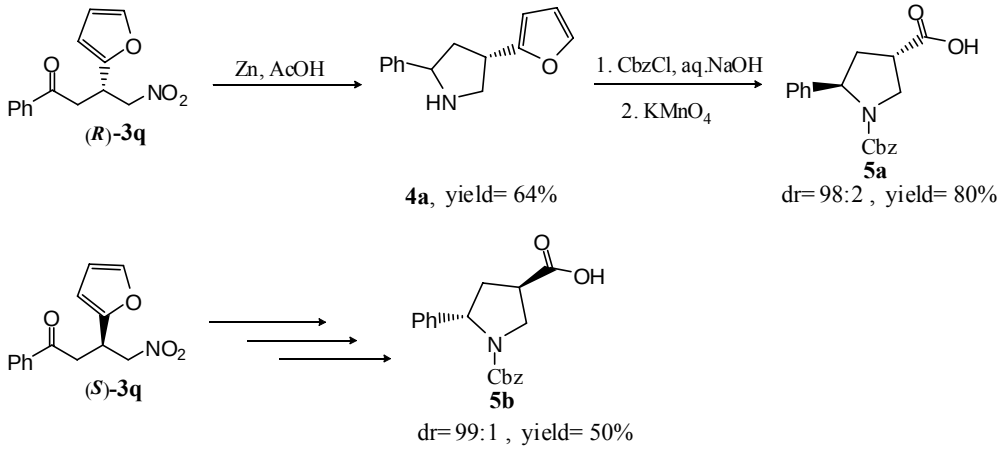
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90–7.93 (m, 2 H), 7.77–7.83(m, 3 H), 7.72(s, 1 H), 7.54–7.58(m, 1 H)，7.24–7.49(m, 5 H), 4.88–4.93(dd, *J* = 6.8 Hz, 12.4 Hz, 1 H)，4.75–4.80(dd, *J* = 8.0 Hz, 12.4 Hz, 1 H)，4.36–4.43(m, 1 H),3.47–3.60(m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.8, 136.5, 133.6, 133.4, 132.8, 128.97, 128.7, 128.0, 127.8, 127.7, 126.5, 126.4, 126.2, 125.1, 79.4, 41.6, 39.4. IR: 3353, 3056, 2921, 2853, 1684, 1550, 1445, 1445, 1377, 1273, 1222, 997, 819, 751, 689, 479 cm^{-1}. ESI-MS**:** *m/z* 320 [M^+].

3l': (*R*)-3-(furan-2-yl)-1-(4-methoxyphenyl)-4-nitrobutan-1-one



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.92–7.95(dd, *J* = 1.8 Hz, 6.9 Hz, 2 H), 7.34(s, 1 H), 6.93–6.96 (m, 2 H), 6.29–6.30(dd, *J* = 2.1 Hz, 3.3 Hz, 1 H)，6.18–6.19(d, *J* = 3.3 Hz, 1 H)，4.71–4.85(m, 2 H), 4.28–4.37(m, 1 H), 3.88(s, 3 H), 3.33–4.51(m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 187.1, 152.1, 142.2, 130.4, 129.3, 113.9, 110.5, 107.1, 77.2, 55.5, 38.6, 33.3. IR: 3342, 2922, 2852, 2519, 1931, 1673, 1560, 1553, 1510, 1421, 1375, 1260, 1172, 1021, 916, 833, 741, 597. HRMS-ESI (*m/z*): calcd for $C_{15}H_{15}NO_5$ [M +H]$^+$: 290.1023; found: 290.1025, 0.7ppm.

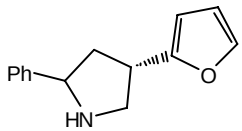
7.0 Procedure for the synthesis of compounds 4a, 5a, 5b, 6a and 7a.



To a solution of **(*R*)-3q** (600 mg, 2.3 mmol) in 20 mL of AcOH was added Zinc powder (30 equiv 5.0 g) in portions at 55 ºC. The resultant mixture was stirred for 2.0 h at 65 ºC (monitored by TLC). After Zinc powder was filtered off, the filtrate was cooled to 0 ºC. The filtrate was diluted with ethyl acetate and neutralized by the addition of sodium hydrogen carbonate (70 % saturated aq). The mixture was extracted with dichloromethane (30 mL ×

4), washed with brine and dried with sodium sulfate. Concentration and flash chromatography (eluent, ethyl acetate / hexane 1:4) afforded **4a** (315 mg, 64%) as a colorless oil.

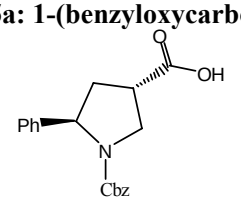
4a: (4*R*)-4-(furan-2-yl)-2-phenylpyrrolidine



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.23–4.40(m, 6 H), 6.27–6.30(m, 1 H), 6.03–6.07(dd, *J* = 3.3 Hz, 9.6 Hz, 1 H),4.33–4.38(*anti*: m, 1 H), 4.24–4.29(*syn*: m, 1 H), 3.48–3.55 (*anti*: m, 2 H), 3.25–3.40, (*syn*: m, 3 H), 3.12–3.17 (*anti*: m, 1 H), 2.56–2.57(*syn:* m, 1 H), 2.34–2.43 (*anti*: m, 1 H), 2.25(br, 1 H), 2.07–2.14 (*anti*: m, 1 H), 1.89–1.94(*syn*: m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 157.6, 157.5, 144.4, 143.8, 141.3, 141.2, 128.5, 128.4, 127.1, 127.0, 126.6, 126.5, 110.1, 104.3, 104.2, 63.2, 62.0, 52.6, 52.4, 40.5, 40.0, 39.3, 38.3. IR: 3337, 3027, 2930, 2870, 1952, 1596, 1500, 1452, 1401, 1343, 1184, 1074, 1010, 940, 802, 734, 701, 598. HRMS-ESI (*m/z*): calcd for $C_{14}H_{15}NO$ $[M+H]^+$: 214.1226; found: 214.1227, 0.5ppm.

4a (315 mg, 1.47 mmol) was dissolved in diethyl ether (5 mL), 3 M sodium hydrate solution (20 mL) was then added and the reaction was cooled to –3 °C. To this mixture was added CbzCl (0.24 mL, 1.62 mmol), and then was allowed to stir at room temperature for 3 h (monitored by TLC). The mixture was extracted with dichloromethane (30 mL × 4), washed with brine and dried with sodium sulfate. The solvent was removed under reduced pressure to give the unpurified intermediate product as a colorless oil. To a solution of the unpurified intermediate product in 10 mL of acetone was added 20% potassium hydroxide solution (0.5 mL), and a solution of $KMnO_4$ (1.9 g, 11.8 mmol, 20 mL) was then added in portions at 20 ºC. The reaction mixture was stirred for 1 h at room temperature (monitored by TLC). After filteration and the precipitation was washed with hot water (10 mL × 5), the filtrate was concentrated under reduced pressure to 15 mL and acidified by adding 6 N HCl (8 mL). The mixture was extracted with dichloromethane (30 mL × 4), washed with brine and dried with sodium sulfate. Concentration and flash chromatography (eluent, ethyl acetate / hexane 1:6 to MeOH/ ethyl acetate 1:4) afforded **5a** (370 mg, 80%) as a white solid. In the same manner, starting from (***S***)-**3q**, **5b** was attained with an overall yield of 50%. The dr values determined by 400 Hz ^{1}H NMR. The configurations were confirmed by comparison of the configuration of **3q**, 400Hz ^{1}H NMR, and CD spectrum and NOE measurements.

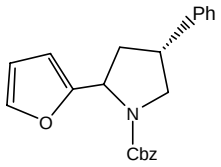
5a: 1-(benzyloxycarbonyl)-5-phenylpyrrolidine-3-carboxylic acid



$[\alpha]^{20}_{D}$= -20 (*c*=1.0, $CHCl_3$)； mp 65-66 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 10.46-10.48(br, 1 H),

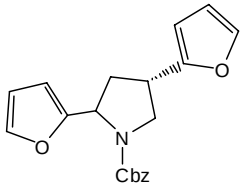
7.16–7.36(m, 9 H), 6.88(m, 1 H), 4.93–5.15(m, 3 H), 3.89–3.95(m, 2 H), 3.15–3.20m, 1 H), 2.60 (m, 1 H), 2.11–2.19(m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 178.1, 154.9, 142.9, 128.6, 128.5, 128.2, 127.9, 127.6, 127.2, 125.3, 67.0, 60.7, 49.4 (48.8), (41.2) 40.5, 38.6 (37.6). IR: 3063, 3032, 2956, 2897, 2636, 2250, 1954, 1706, 1495, 1417, 1354, 1180, 1124, 1058, 1029, 912, 735, 698, 613, 552. HRMS-ESI (*m/z*): calcd for $C_{19}H_{19}NO_4$ [M +H]$^+$: 326.1387; found: 326.1389, 0.6ppm.

6a: (4*R*)-benzyl 2-(furan-2-yl)-4-phenylpyrrolidine-1-carboxylate



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.20–7.34(m, 11 H), 6.25–6.31(m, 1 H), 6.10(s, 1 H), 5.07–5.26(m, 3H), 3.98–4.07(m, 1 H), 3.43-3.73(m 2 H), 2.26-2.45(m 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 155.4, 154.7, 141.6, 140.6, 136.7, 128.7, 128.5, 128.3, 127.9, 127.7, 127.1, 126.9, 110.3, 106.4, 106.0, 66.9, 55.3, 55.1, 52.8, 42.5, 41.4, 39.4, 38.1. IR: 3650, 3337, 2931, 2870, 2652, 1952, 1597, 1501, 1452, 1344, 1238, 1148, 1074, 1010, 940, 802, 734, 701, 598, 543 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{22}H_{21}NO_3$ [M +H]$^+$: 348.1594; found: 348.1589, 1.4ppm.

7a: (4*R*)-benzyl 2,4-di(furan-2-yl)pyrrolidine-1-carboxylate



^{1}H NMR (300 MHz, $CDCl_3$): δ 7.21–7.33(m, 7 H), 6.24–6.29(m, 3 H), 6.07(s, 1 H), 5.05–5.27(m, 3 H), 3.54–3.92(m, 2 H), 2.35-2.40(m 2 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 155.0, 154.6, 154.3, 141.6, 136.7, 128.4, 128.3, 127.9, 127.7, 110.2, 106.5, 106.1, 105.2, 66.9, 54.8, 50.5, 37.3, 36.5, 36.0, 35.5. IR: 3393, 3117, 2952, 2889, 1800, 1706, 1597, 1503, 1412, 1354, 1116, 1010, 736, 699, 601, 569, 464 cm^{-1}. HRMS-ESI (*m/z*): calcd for $C_{20}H_{19}NO_4$ [M +H]$^+$: 338.1387; found: 338.1387, 0ppm.

8.0 References

[1] Liu, K.; Cui, H-F.; Nie, J.; Dong, K-Y.; Li, X-J.; Ma, J-A. *Org. Lett*. **2007**, *9*, 923.

[2] Vakylya, B.; Varga, S.; Csapmpai, A.; Soós, T. *Org. Lett.* **2005**, *7*, 1967.

[3] Dixon, D. J.; Richardson, R. D.; *Synlett.* **2006**, *1*, 81.

[4] Huang, H.; Jacobsen, E. N. *J. Am. Chem. Soc.* **2006**, *128*, 7170.

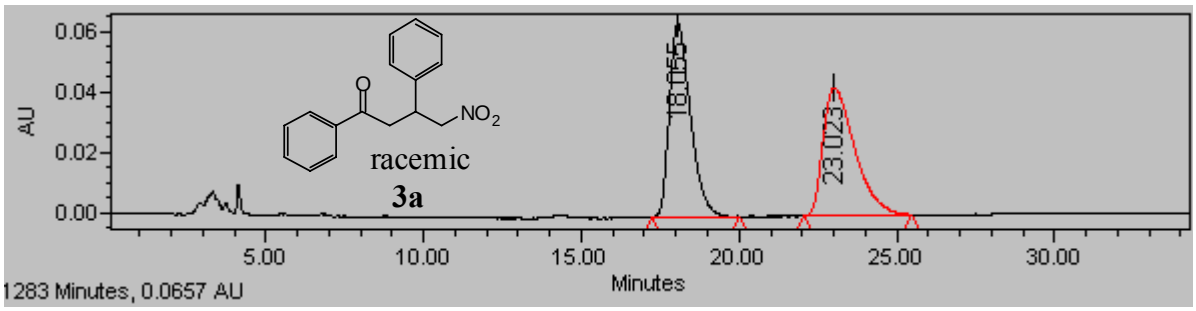
[5] Corey, E. J.; Zhang, F.-Y. *Org. Lett.* **2000**, *2*, 4257.

9.0 Optical rotation and chiral-phase HPLC data

products	$[\alpha]^{20}_{D}$	*i*PrOH/Hexane	HPLC data (Daicel Chiralpak)
(*S*)-3a	-22(*c*=1.0,$CHCl_3$)	15/85(AS-H)	254 nm, 0.9 mL/min, t_R = 18.4 min (major)
(*R*)-3a	+12(*c*=1.6,$CHCl_3$)	15/85(AS-H)	254 nm, 0.9 mL/min, t_R = 17.9 min, 22.9 min (major)
(*S*)-3b	-14(*c*=1.1,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 13.3 min (major), 16.5min
(*R*)-3b	+4(*c*=1.0,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 13.2 min, 17.0 min (major)
(*S*)-3c	-25(*c*=1.5,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 12.5 min (major), 16.1 min
(*R*)-3c	+10(*c*=1.2,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 12.5 min, 16.0 min (major)
(*S*)-3d	-20(*c*=1.2,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 11.9 min (major), 13.1 min
(*R*)-3d	+22(*c*=3.0,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 16.5 min, 21.7 min (major)
(*S*)-3e	-15(*c*=1.0,$CHCl_3$)	15/85(AD-H)	268 nm, 0.6 mL/min, t_R = 18.2 min (major), 20.0min
(*R*)-3e	+18(*c*=1.5,$CHCl_3$)	15/85(AD-H)	268nm, 0.6 mL/min, t_R = 18.2 min, 20.1 min (major)
(*S*)-3f	-10(*c*=1.6,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 16.5 min (major)
(*R*)-3f	+13(*c*=1.0,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 16.5min, 21.3 min (major)
(*S*)-3g	-20(*c*=0.7,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 11.8 min (major), 15.3min
(*R*)-3g	+12(*c*=0.8,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 11.7 min, 15.1 min (major)
(*S*)-3h	-9(*c*=1.3,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 10.6 min (major), 13.8 min
(*R*)-3h	+2(*c*=1.7,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 10.6 min, 13.9 min (major)
(*S*)-3i	-3(*c*=1.5,$CHCl_3$)	15/85(AD-H)	281 nm, 1.0 mL/min, t_R = 10.6 min (major), 13.2 min
(*R*)-3i	+2(*c*=1.5,$CHCl_3$)	15/85(AD-H)	281 nm, 1.0 mL/min, t_R = 10.5 min (major), 13.1min
(*S*)-3j	-3(*c*=1.0,$CHCl_3$)	15/85(AD-H)	275 nm, 1.0 mL/min, t_R = 7.5 min (major)
(*R*)-3j	+1(*c*=1.0,$CHCl_3$)	15/85(AD-H)	275 nm, 1.0 mL/min, t_R = 7.7 min, 8.9 min (major)
(*S*)-3k	-62(*c*=1.0,$CHCl_3$)	15/85(AD-H)	261 nm, 1.0 mL/min, t_R = 13.1 min (major), 17.7min
(*R*)-3k	+6(*c*=2.2,$CHCl_3$)	15/85(AD-H)	261 nm, 1.0 mL/min, t_R = 17.7 min (major)
(*S*)-3l	-18(*c*=3.0,$CHCl_3$)	15/85(AD-H)	287 nm, 0.6 mL/min, t_R = 24.7 min (major)
(*R*)-3l	+4(*c*=1.6,$CHCl_3$)	15/85(AD-H)	287 nm, 0.6 mL/min, t_R = 25.5 min (major)
(*S*)-3m	-9(*c*=1.0,$CHCl_3$)	15/85(AD-H)	268 nm, 0.6 mL/min, t_R = 21.3 min (major), 22.6 min
(*R*)-3m	+9(*c*=1.2,$CHCl_3$)	15/85(AD-H)	268 nm, 0.6 mL/min, t_R = 20.9 min, 22.1 min (major)
(*S*)-3n	-38(*c*=1.1,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 13.6 min (major), 15.9 min
(*R*)-3n	+6(*c*=2.0,$CHCl_3$)	15/85(AD-H)	268 nm, 1.0 mL/min, t_R = 13.6 min, 15.8 min (major)
(*S*)-3o	-13(*c*=1.6,$CHCl_3$)	15/85(AD-H)	281 nm, 1.0 mL/min, t_R = 9.0 min (major), 10.6 min
(*R*)-3o	+3(*c*=1.5,$CHCl_3$)	15/85(AD-H)	281 nm, 1.0 mL/min, t_R = 9.0 min, 10.6 min (major)
(*S*)-3p	-2(*c*=0.7,$CHCl_3$)	15/85(AD-H)	259 nm, 1.0 mL/min, t_R = 11.7 min (major)
(*R*)-3p	+6(*c*=1.7,$CHCl_3$)	15/85(AD-H)	259 nm, 1.0 mL/min, t_R = 13.2 min (major)
(*S*)-3q	-16(*c*=0.6,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 11.1 min (major), 13.4 min
(*R*)-3q	+3(*c*=1.2,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 10.8 min, 13.0 min (major)
(*S*)-3r	-20(*c*=2.4,$CHCl_3$)	15/85(AD-H)	253 nm, 1.0 mL/min, t_R = 13.3 min (major), 15.9 min
(*R*)-3r	+16(*c*=0.8,$CHCl_3$)	15/85(AD-H)	253 nm, 1.0 mL/min, t_R = 13.3 min, 15.9 min (major)
(*S*)-3s	-10(*c*=2.6,$CHCl_3$)	15/85(AD-H)	245 nm, 1.0 mL/min, t_R = 11.3 min (major), 13.0 min
(*R*)-3s	+4(*c*=2.5,$CHCl_3$)	15/85(AD-H)	245nm, 1.0 mL/min, t_R = 11.4 min, 13.1 min (major)
(*S*)-3t	-6(*c*=1.5,$CHCl_3$)	15/85(AD-H)	245 nm, 1.0 mL/min, t_R = 9.5 min (major), 10.4 min
(*R*)-3t	+13(*c*=2.5,$CHCl_3$)	15/85(AD-H)	245 nm, 1.0 mL/min, t_R = 9.9 min, 10.8 min (major)
(*S*)-3u	-4(*c*=1.5,$CHCl_3$)	15/85(AD-H)	245 nm, 1.0 mL/min, t_R = 8.3 min (major)

(*R*)-3u	+11(*c*=1.7,$CHCl_3$)	15/85(AD-H)	245 nm, 1.0 mL/min, t_R = 8.3 min, 9.5 min (major)
(*S*)-3v	-7(*c*=1.7,$CHCl_3$)	15/85(AD-H)	213 nm, 1.0 mL/min, t_R = 7.4 min (major), 8.1 min
(*R*)-3v	+5(*c*=1.2,$CHCl_3$)	15/85(AD-H)	213 nm, 1.0 mL/min, t_R = 7.4 min, 8.1 min (major)
(*S*)- 3a'	-12(*c*=0.6,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 16.8 min (major), 21.3 min
(*R*)- 3a'	+17(*c*=1.0,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 16.5 min, 21.7 min (major)
(*S*)- 3b'	-25(*c*=0.7,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 12.8 min (major)
(*R*)- 3b'	+8(*c*=1.0,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 12.0 min, 18.3 min (major)
(*S*)- 3c'	-13(*c*=1.7,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 11.7 min (major), 13.4 min
(*R*)- 3c'	+13(*c*=1.8,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 11.8min, 13.6 min (major)
(*S*)- 3d'	-10(*c*=0.6,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 9.8 min (major)
(*R*)- 3d'	+6(*c*=1.3,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 9.9 min, 12.3 min (major)
(*S*)- 3e'	-28(*c*=1.2,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 14.5 min (major)
(*R*)- 3e'	+16(*c*=1.1,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 14.7 min, 20.6 min (major)
(*S*)- 3f'	-20(*c*=1.0,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 15.7 min (major)
(*R*)- 3f'	+12(*c*=1.0,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 21.8 min (major)
(*S*)- 3g'	-20(*c*=1.8,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 11.1 min (major)
(*R*)- 3g'	+16(*c*=0.8,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 10.8 min, 14.3 min (major)
(*S*)- 3h'	-20(*c*=1.4,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 10.1 min (major)
(*R*)- 3h'	+14(*c*=1.1,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 10.1 min, 12.2 min (major)
(*S*)- 3i'	-18(*c*=1.4,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 11.8 min (major)
(*R*)- 3i'	+17(*c*=1.6,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 11.7 min,13.8 min (major)
(*S*)- 3j'	-8(*c*=1.2,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 11.0 min (major), 14.8 min
(*R*)- 3j'	+5(*c*=1.4,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 12.2 min, 16.6 min (major)
(*S*)- 3k'	-14(*c*=1.3,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 15.7 min (major), 19.3 min
(*R*)- 3k'	+16(*c*=1.6,$CHCl_3$)	15/85(AD-H)	254 nm, 1.0 mL/min, t_R = 15.7 min, 19.7 min (major)
(*S*)- 3l'	-14(*c*=1.0,$CHCl_3$)	15/85(AD-H)	215 nm, 1.0 mL/min, t_R =20.1 min (major), 24.7 min
(*R*)- 3l'	+7(*c*=0.7,$CHCl_3$)	15/85(AD-H)	215 nm, 1.0 mL/min, t_R = 19.8 min, 24.3 min (major)

10.0 Copies of HPLC spectra of racemic /chiral Michael products



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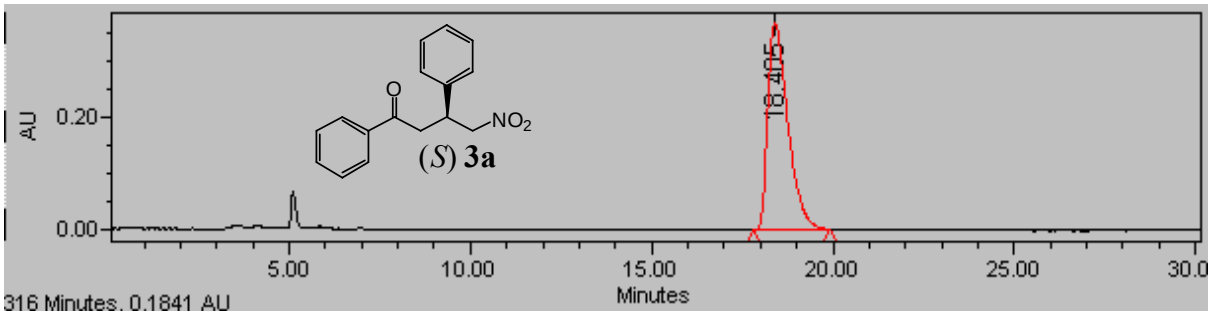

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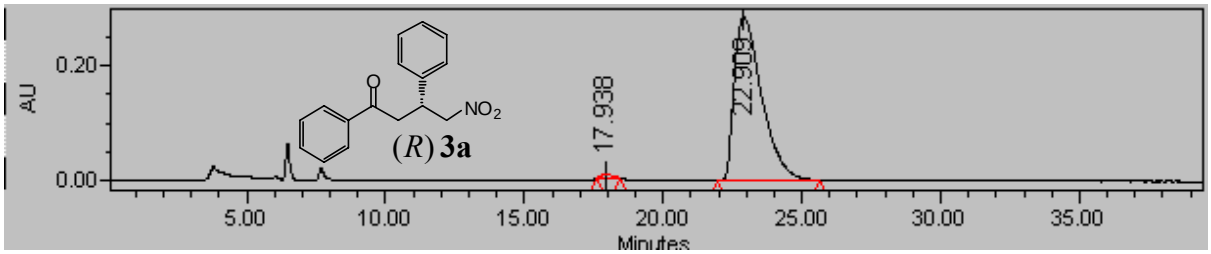

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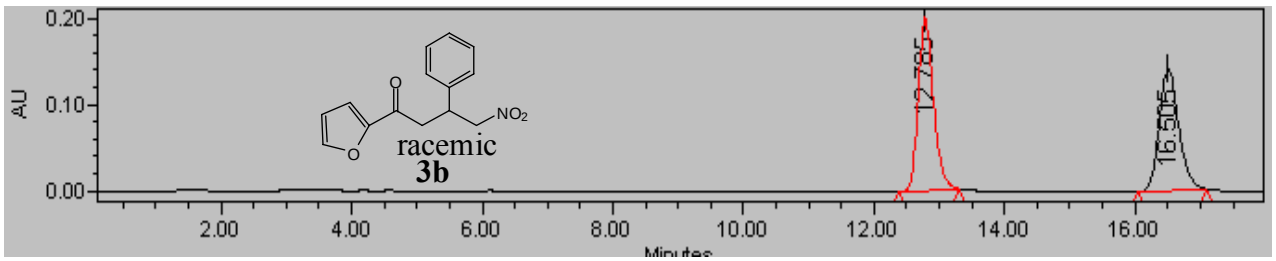

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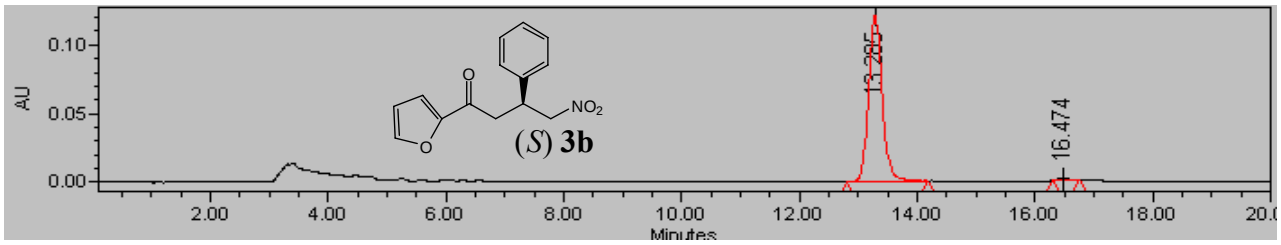

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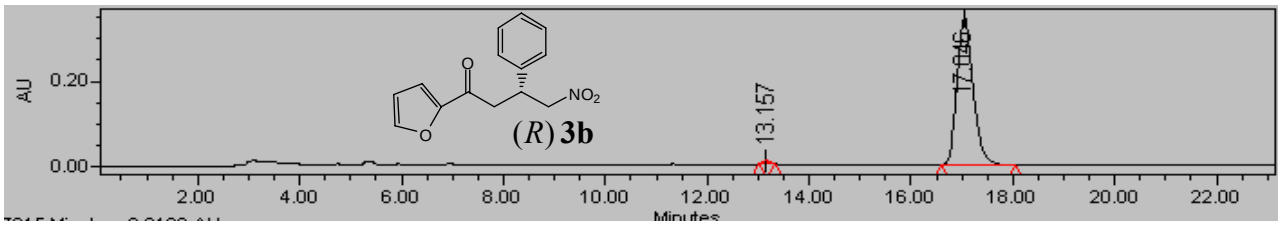

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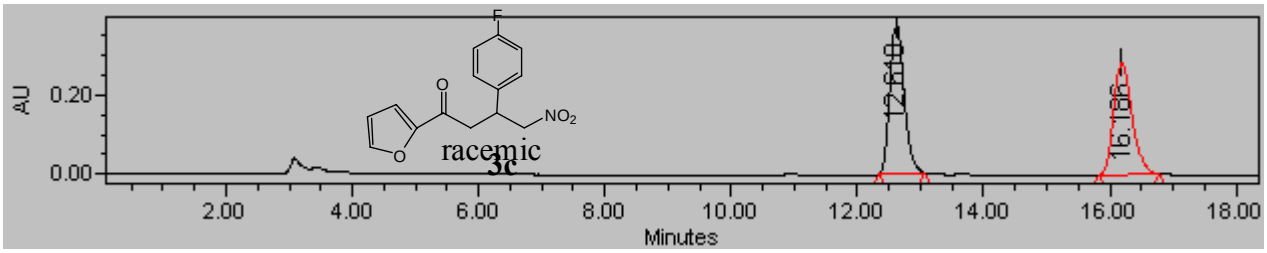

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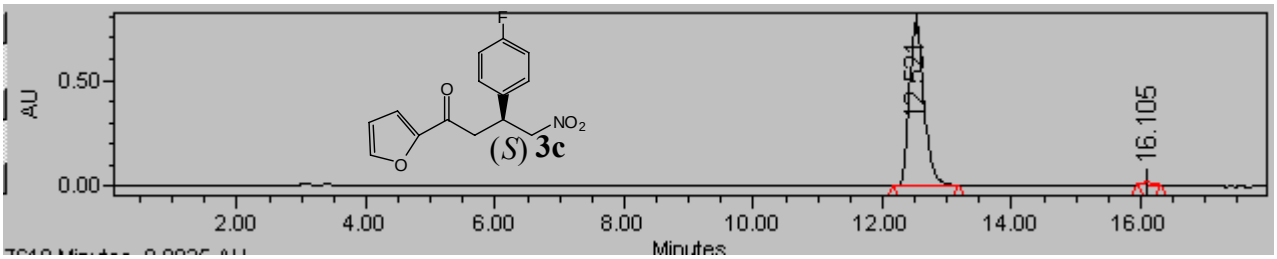

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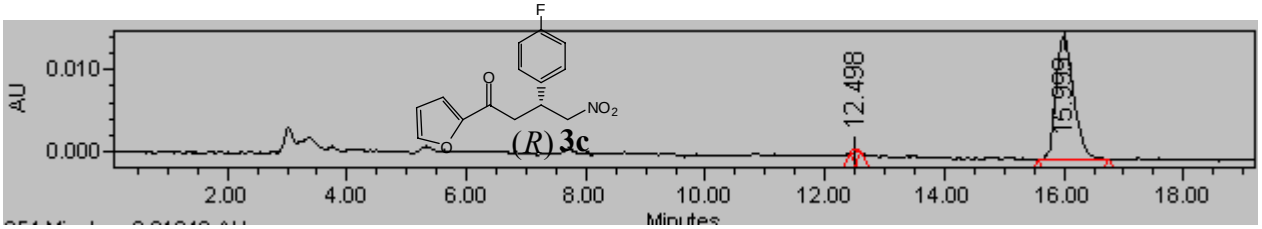

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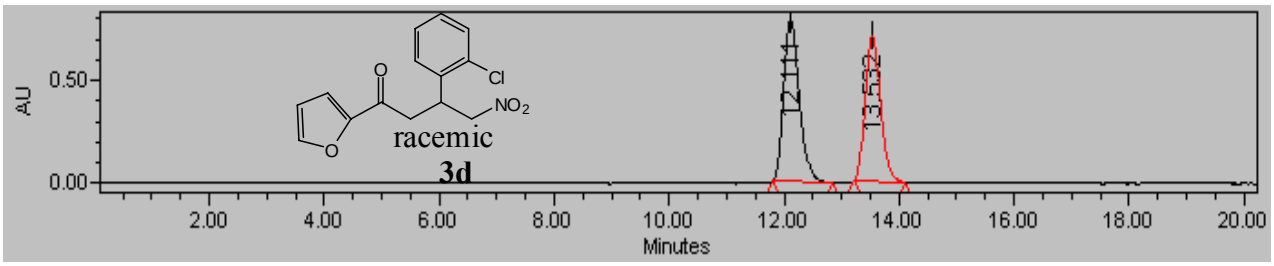

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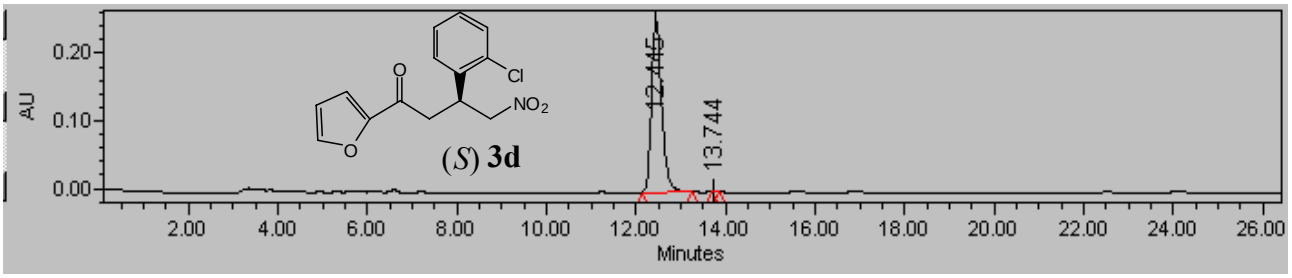

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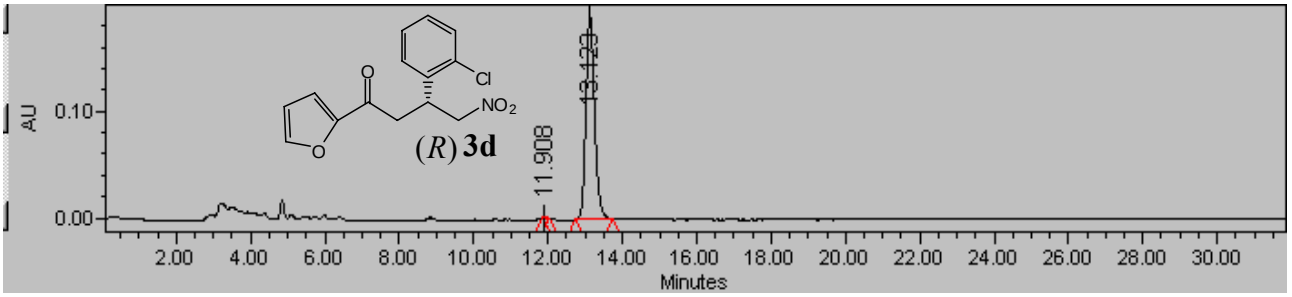

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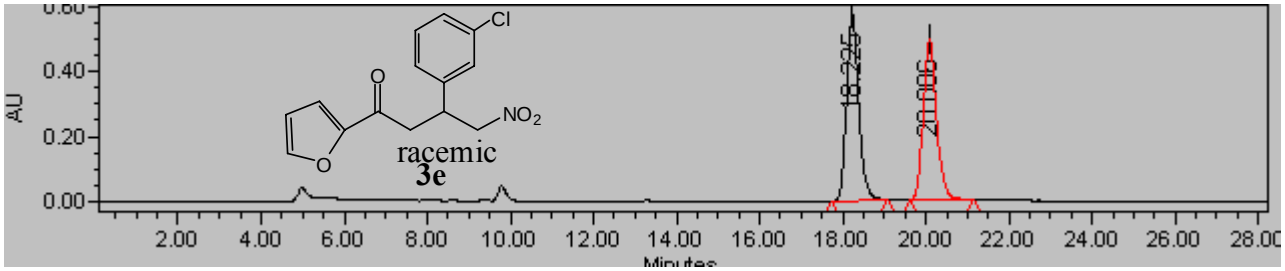
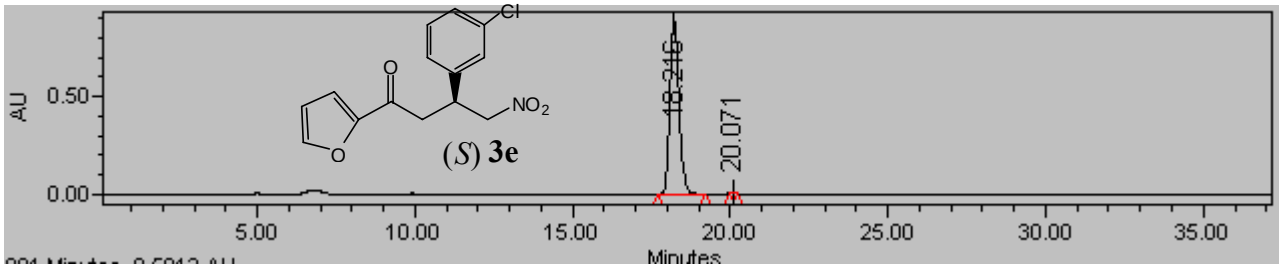
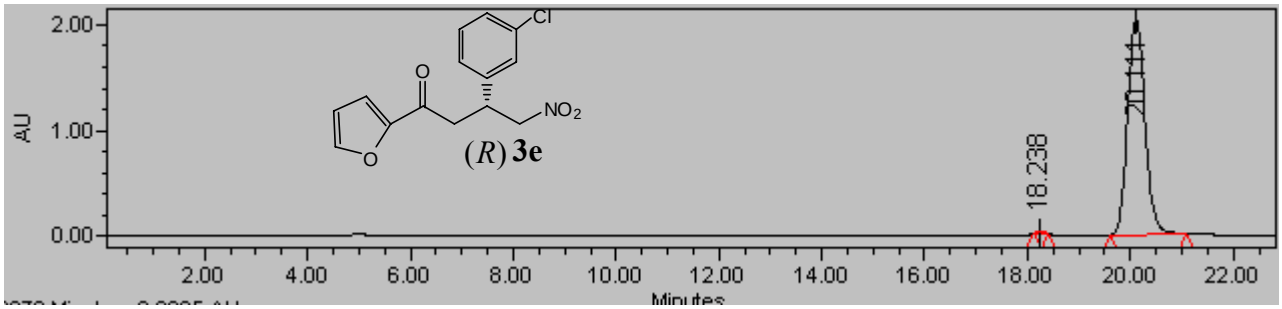
1		11.908	8095	0.26	1146	bb			Unknown
2		13.123	3144513	99.74	191078	bb			Unknown



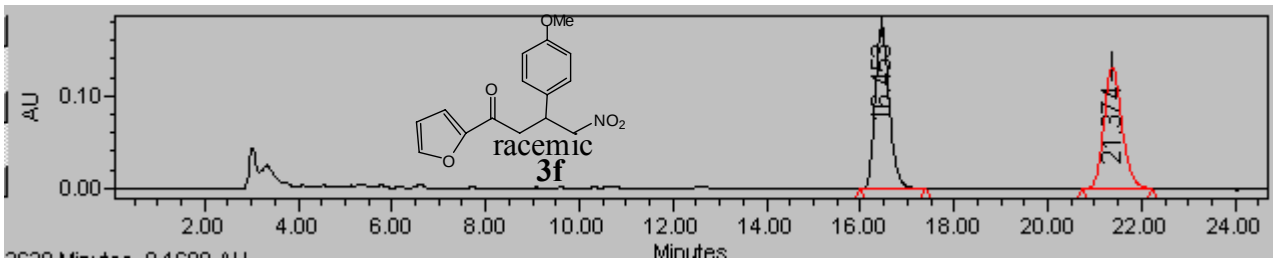
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		18.225	11678120	51.65	573954	bb			Unknown
2		20.086	10933964	48.35	493107	bb			Unknown



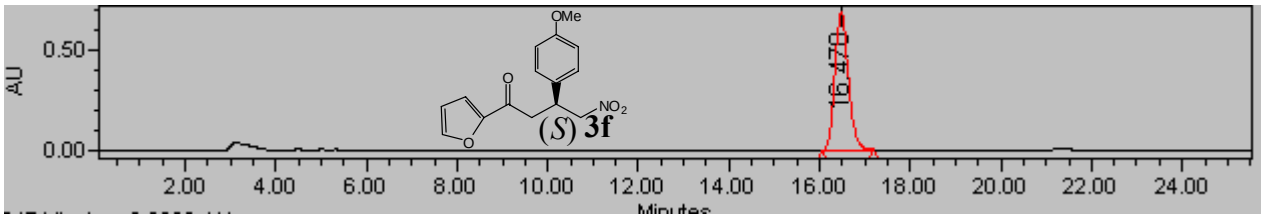
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		18.216	18148099	99.80	893783	bb			Unknown
2		20.071	36496	0.20	3911	bb			Unknown



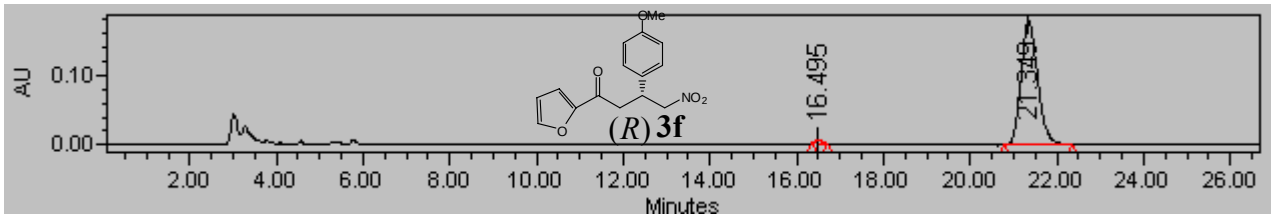
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		18.238	205516	0.43	18281	bb			Unknown
2		20.111	47970953	99.57	2038291	bb			Unknown



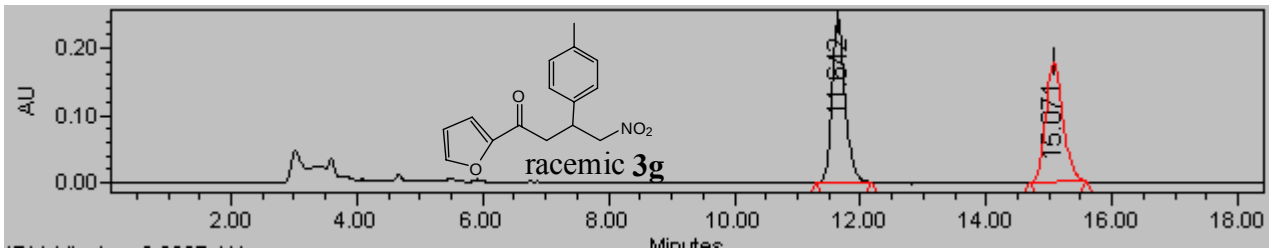
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		16.453	3694035	51.16	177645	bb			Unknown
2		21.374	3526633	48.84	131234	bb			Unknown



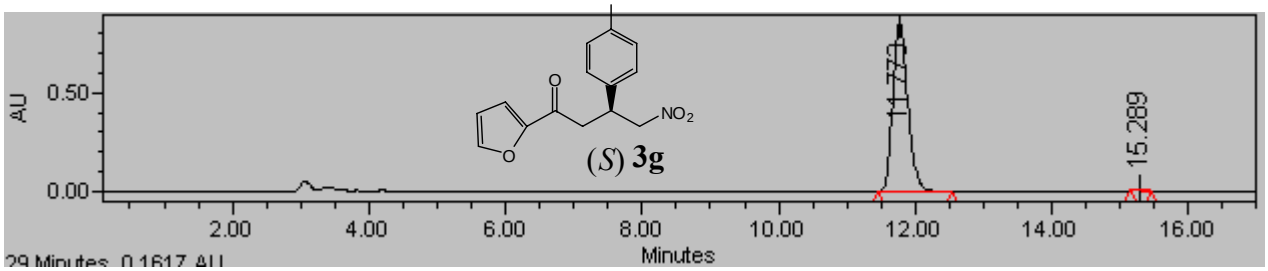
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		16.478	14036787	100.00	678961	bb			Unknown



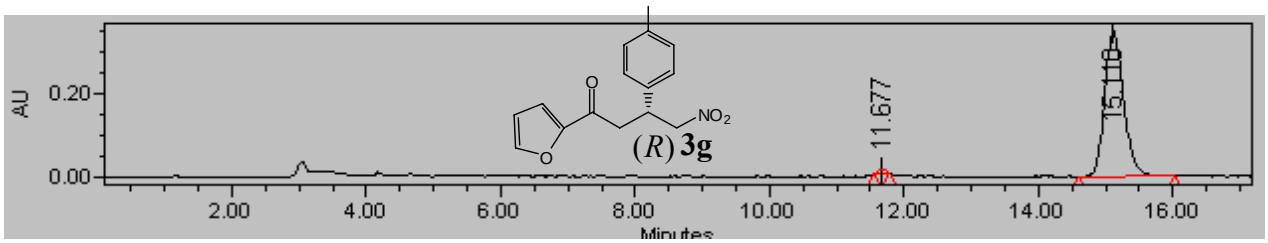
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		16.495	47951	1.00	3561	bb			Unknown
2		21.349	4725671	99.00	177616	bb			Unknown



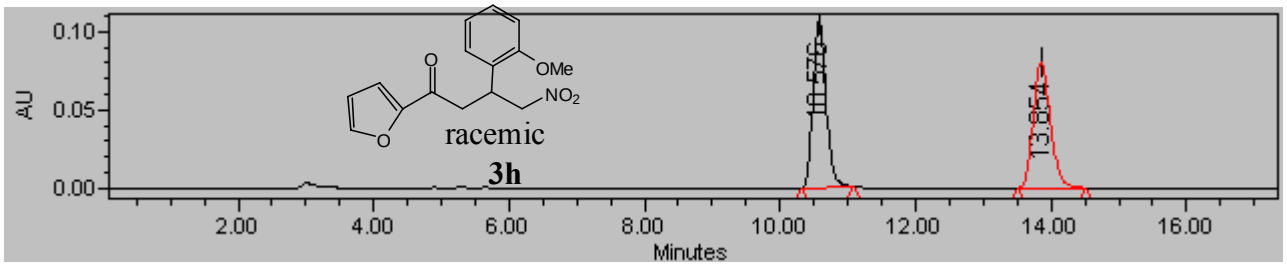
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.642	3615555	51.90	244154	bb			Unknown
2		15.071	3351303	48.10	177495	bb			Unknown



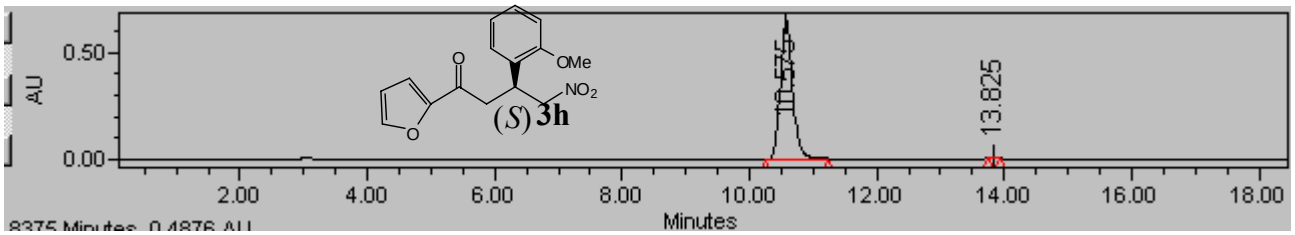
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.773	12965857	99.31	849455	bb			Unknown
2		15.289	89535	0.69	8194	bb			Unknown



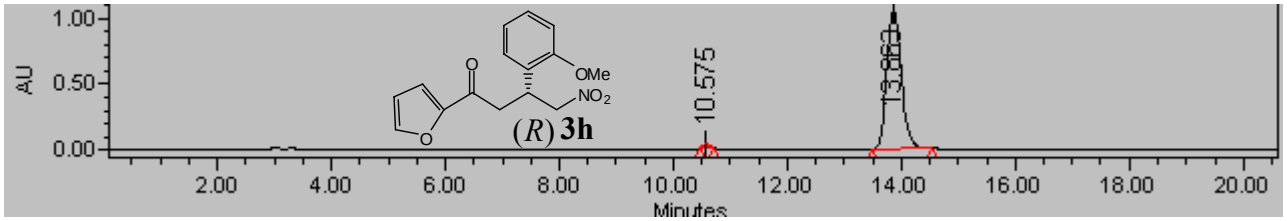
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.677	76437	1.11	8674	bb			Unknown
2		15.110	6827188	98.89	349923	bb			Unknown



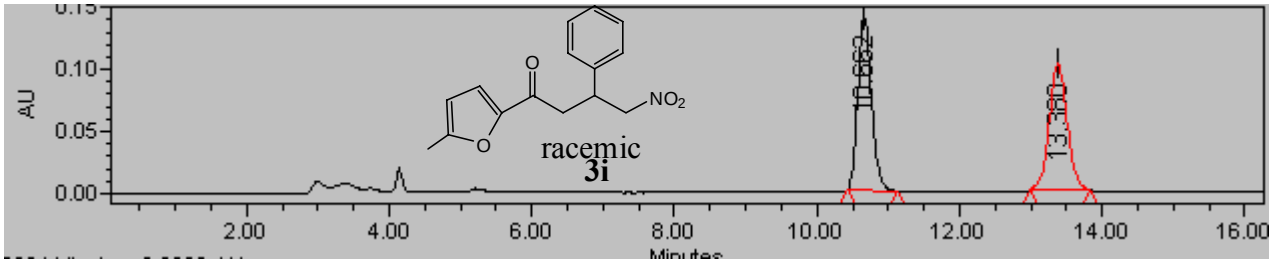
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.576	1391299	49.98	105630	bb			Unknown
2		13.854	1392303	50.02	80002	bb			Unknown



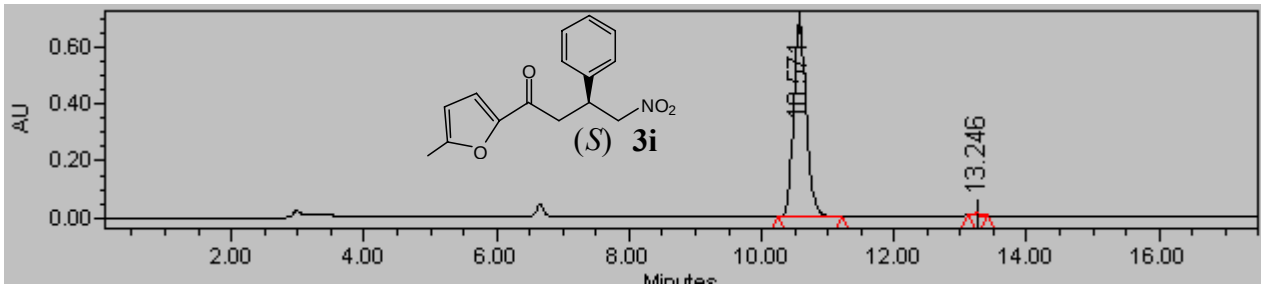
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.575	8674088	99.78	649680	bb			Unknown
2		13.825	18792	0.22	2915	bb			Unknown



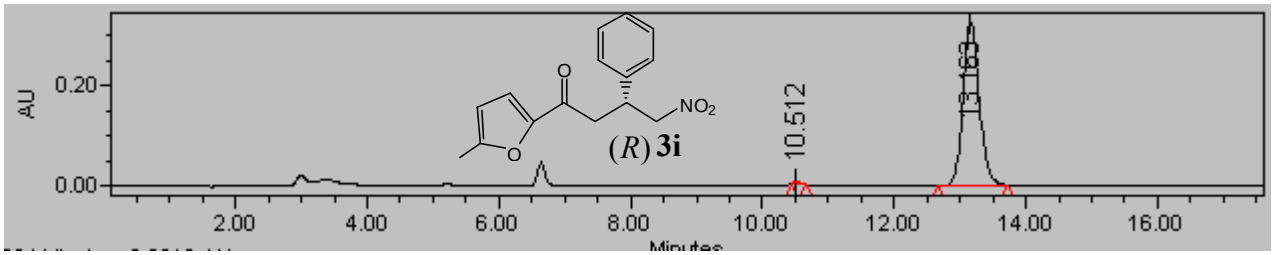
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.575	204890	1.09	24761	bb			Unknown
2		13.863	18672125	98.91	1061459	bb			Unknown



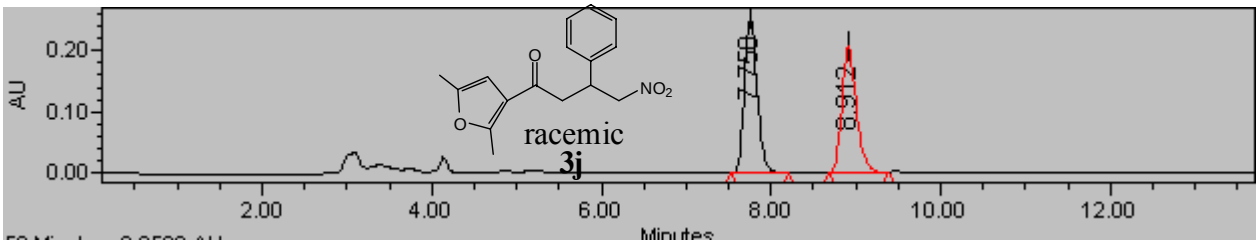
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.662	1852189	51.05	139874	bb			Unknown
2		13.380	1776130	48.95	101364	bb			Unknown



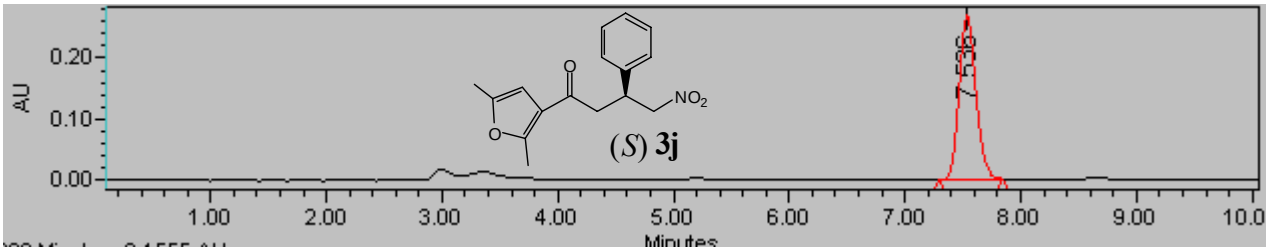
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.571	9392831	99.24	693714	bb			Unknown
2		13.246	71790	0.76	6622	bb			Unknown



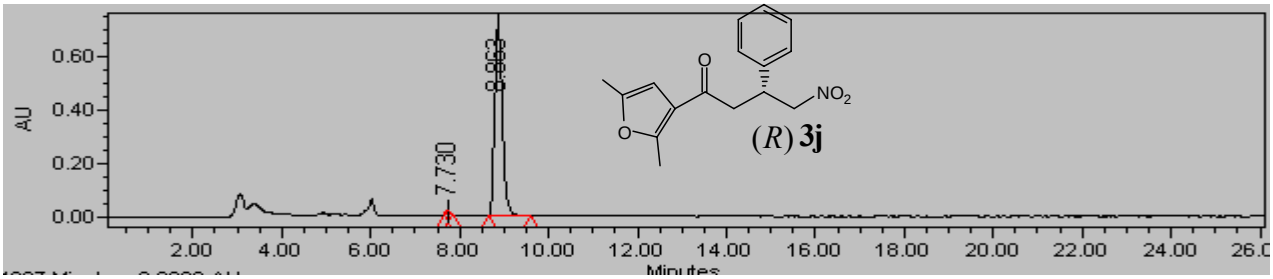
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.512	31694	0.56	4075	bb			Unknown
2		13.163	5578646	99.44	326838	bb			Unknown



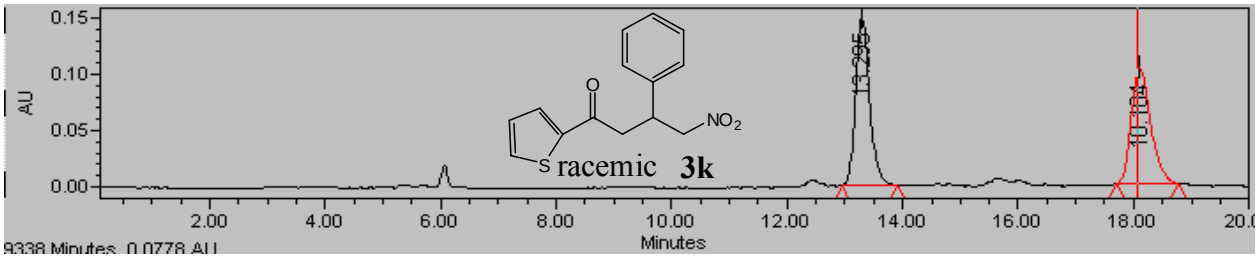
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		7.758	2628098	52.07	257922	bb			Unknown
2		8.912	2418715	47.93	204982	bb			Unknown



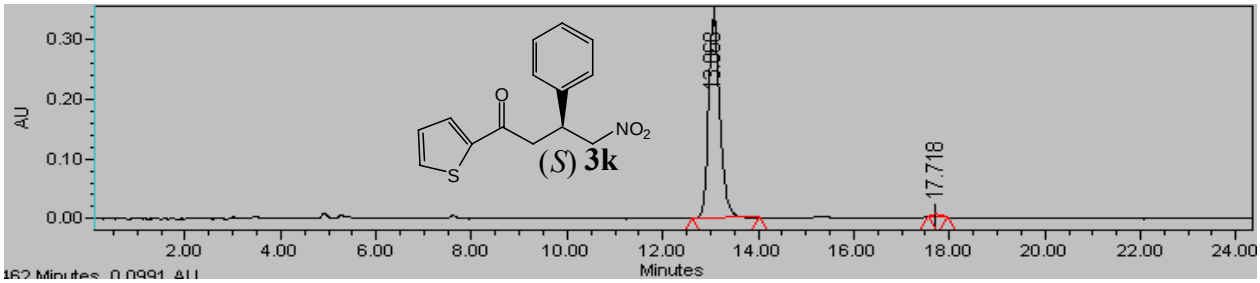
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		7.536	2600074	100.00	268368	bb			Unknown



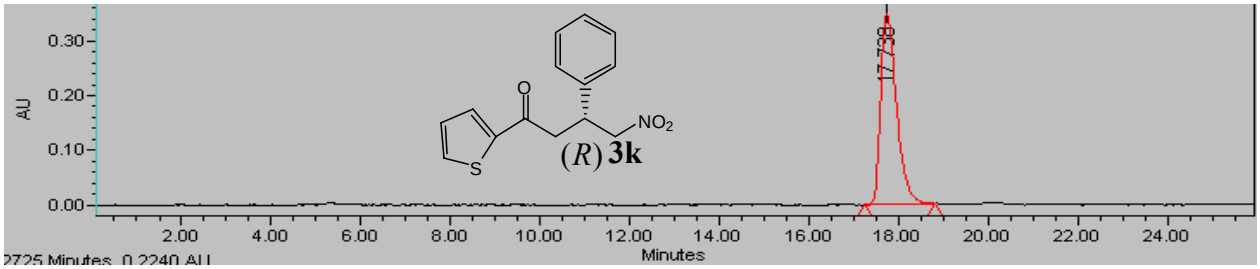
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		7.730	93866	1.09	13418	bb			Unknown
2		8.863	8541781	98.91	724227	bb			Unknown



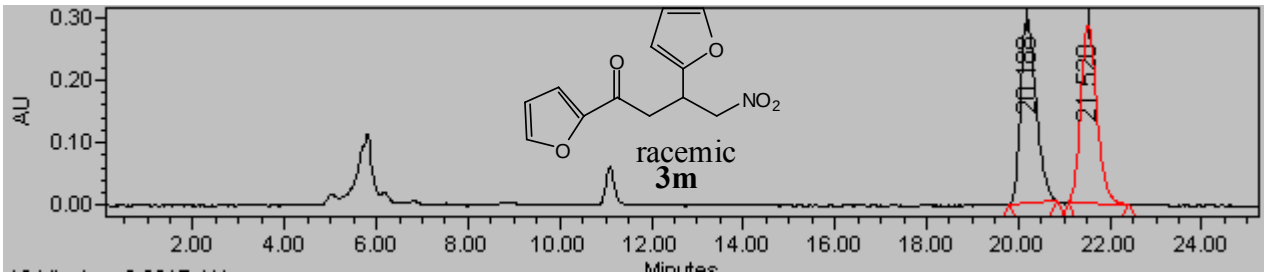
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		13.295	2510481	51.24	149773	bb			Unknown
2		18.104	2388546	48.76	102732	bb			Unknown



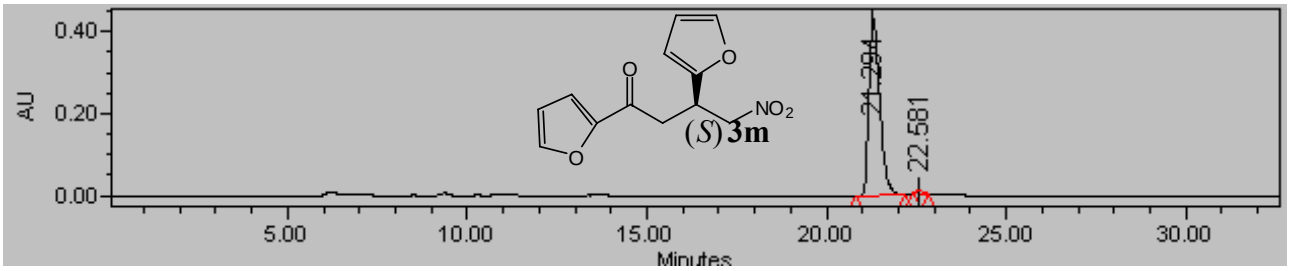
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		13.068	5659805	99.10	338856	bb			Unknown
2		17.718	51439	0.90	3988	bb			Unknown



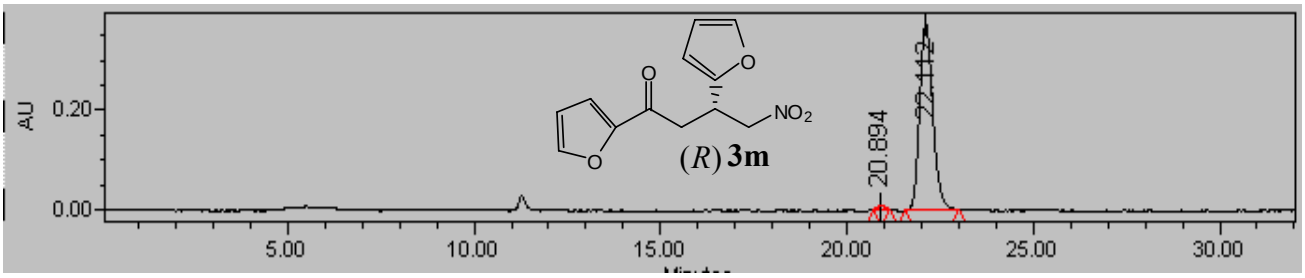
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		17.738	8498802	100.00	350180	bb			Unknown



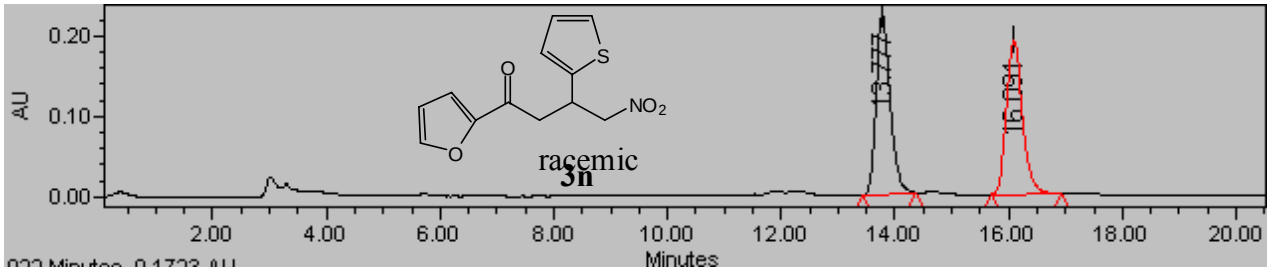
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		20.188	6999644	50.18	297620	bb			Unknown
2		21.520	6948851	49.82	284722	bb			Unknown



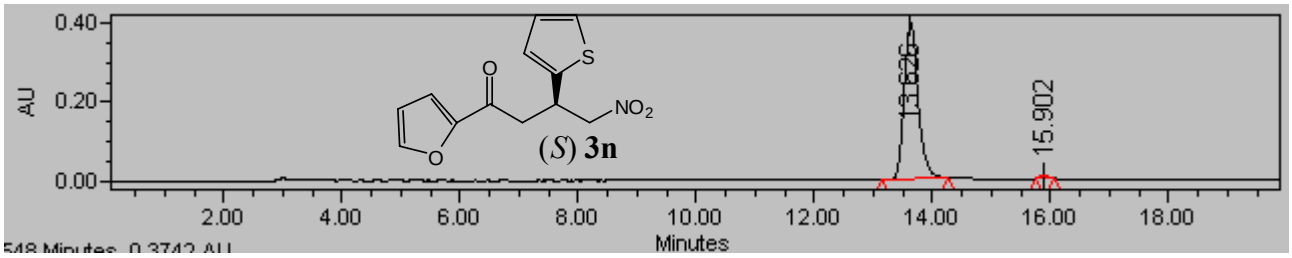
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		21.294	9064293	99.02	432595	bb			Unknown
2		22.581	89941	0.98	6109	bb			Unknown



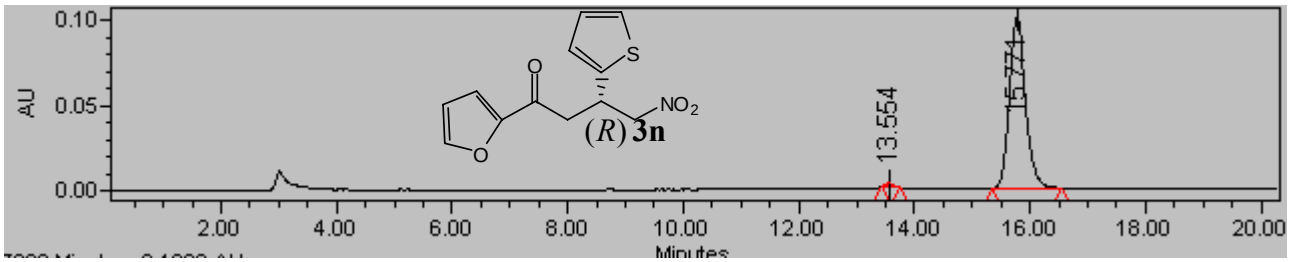
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		20.894	89825	0.97	5935	bb			Unknown
2		22.112	9151473	99.03	376437	bb			Unknown



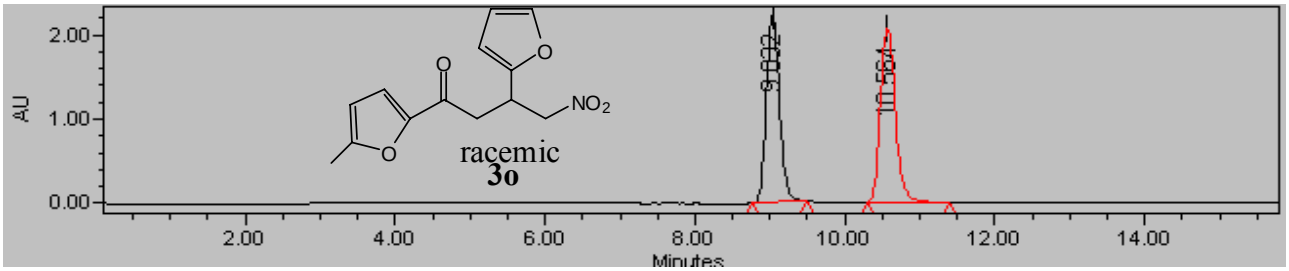
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		13.777	3818730	50.49	224004	bb			Unknown
2		16.091	3744999	49.51	190754	bb			Unknown



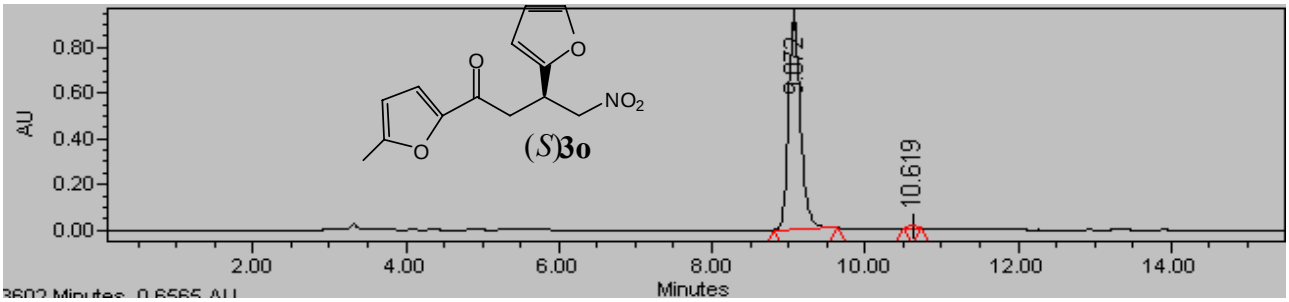
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		13.626	6655413	99.04	396708	bb			Unknown
2		15.902	64693	0.96	5568	bb			Unknown



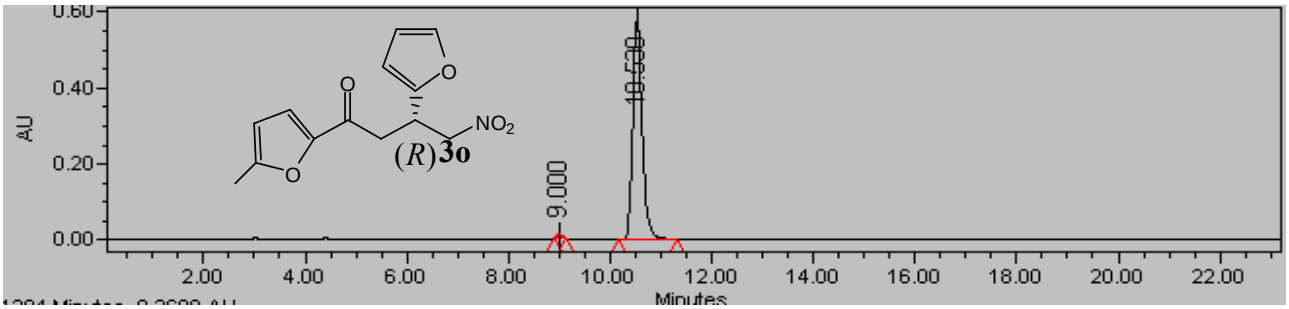
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		13.554	19670	1.00	1994	bb			Unknown
2		15.771	1943729	99.00	100744	bb			Unknown



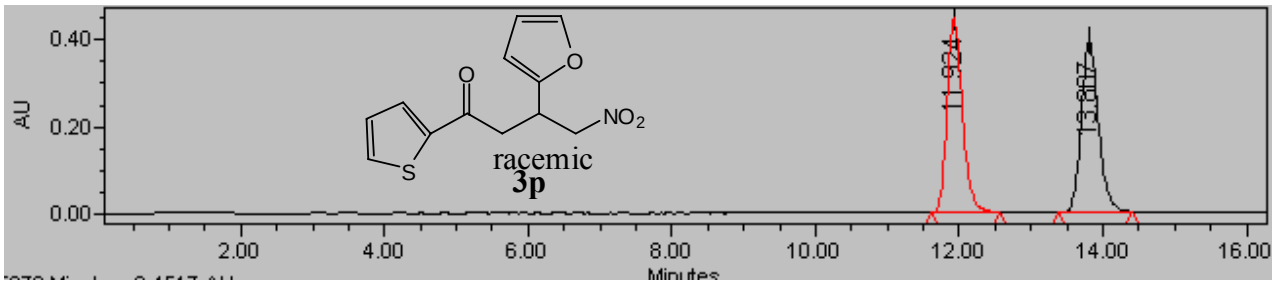
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		9.032	26946189	48.40	2213979	bb			Unknown
2		10.564	28728171	51.60	2064134	bb			Unknown



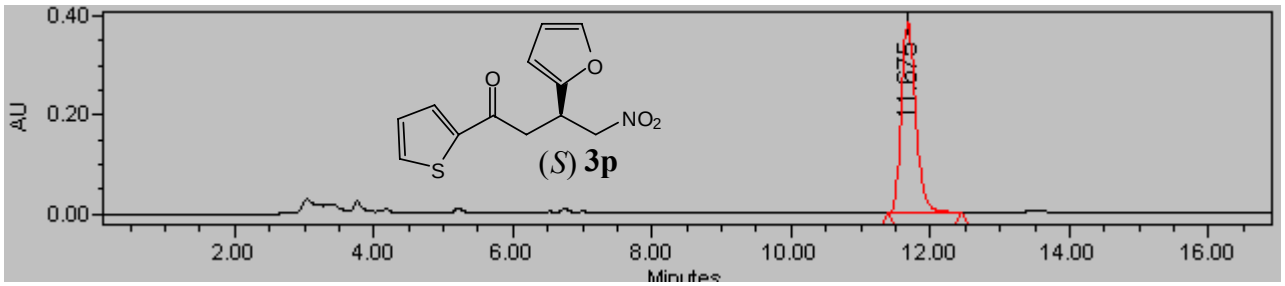
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		9.072	10220648	99.13	919909	bb			Unknown
2		10.619	90037	0.87	10716	bb			Unknown



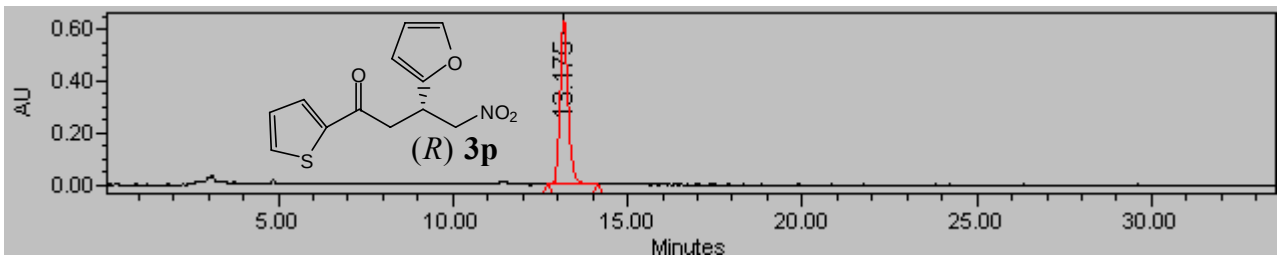
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		9.000	70279	0.92	9313	bb			Unknown
2		10.530	7531960	99.08	580835	bb			Unknown



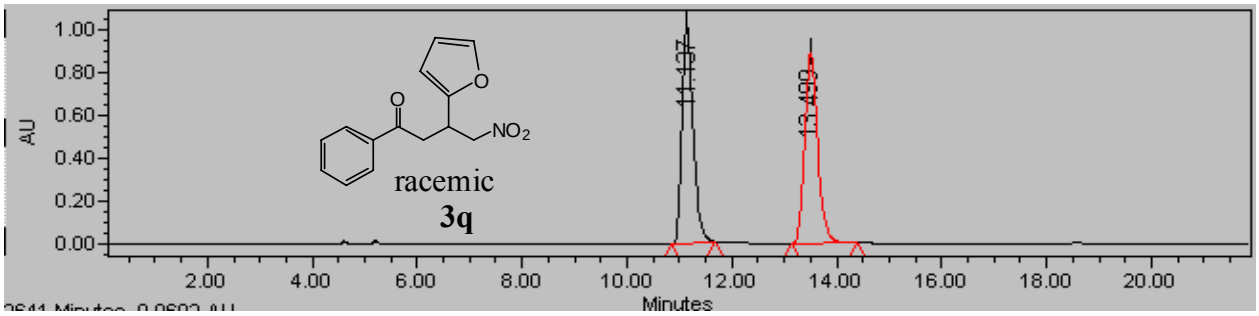
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.924	6612964	50.11	450141	bb			Unknown
2		13.807	6584564	49.89	395531	bb			Unknown



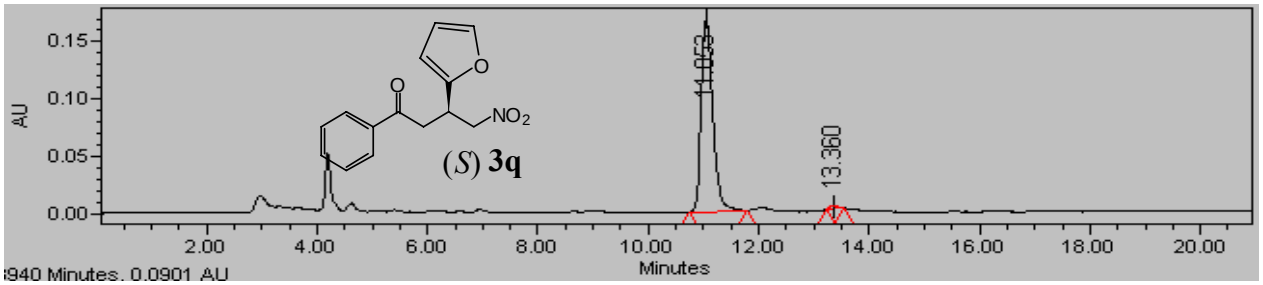
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.675	5511278	100.00	385344	bb			Unknown



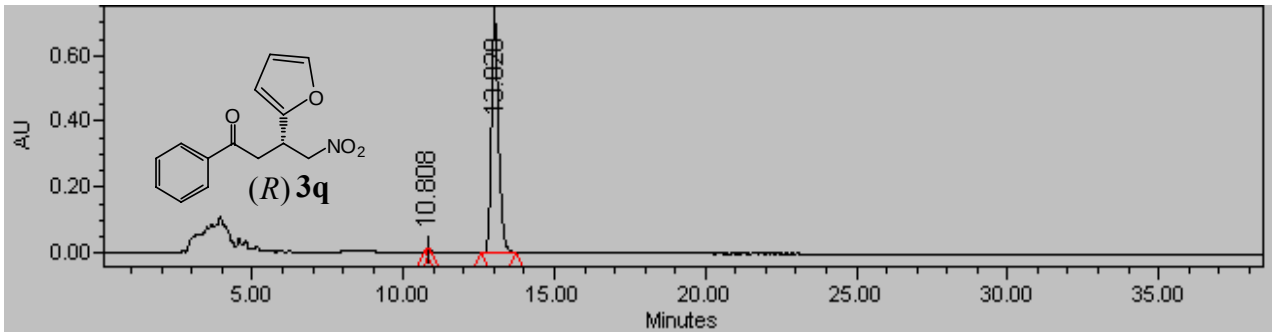
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		13.175	10239913	100.00	631697	bb			Unknown



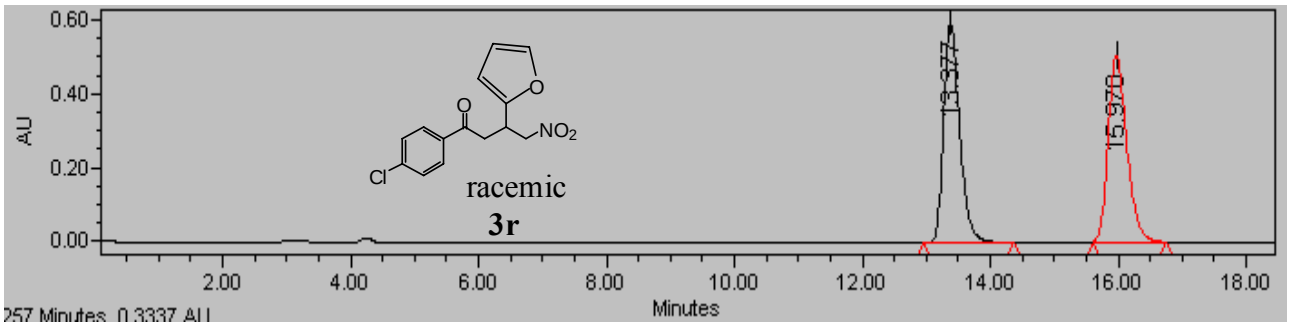
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.137	14977382	49.57	1032224	bb			Unknown
2		13.499	15235673	50.43	887852	bb			Unknown



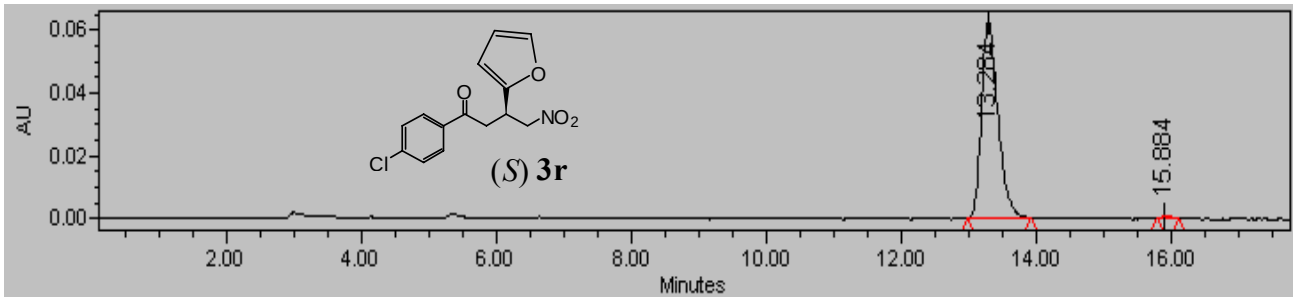
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.053	2394733	99.08	168848	bb			Unknown
2		13.360	22346	0.92	2222	bb			Unknown



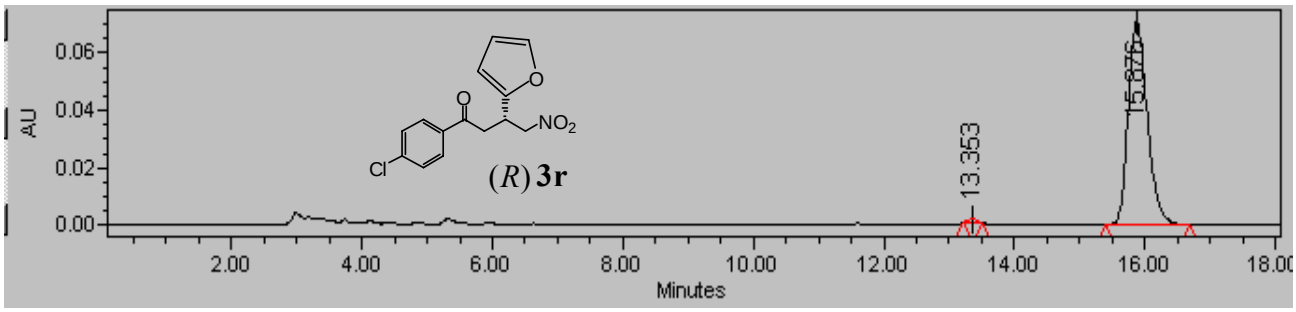
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.808	64627	0.53	7620	bb			Unknown
2		13.020	12030885	99.47	715911	bb			Unknown



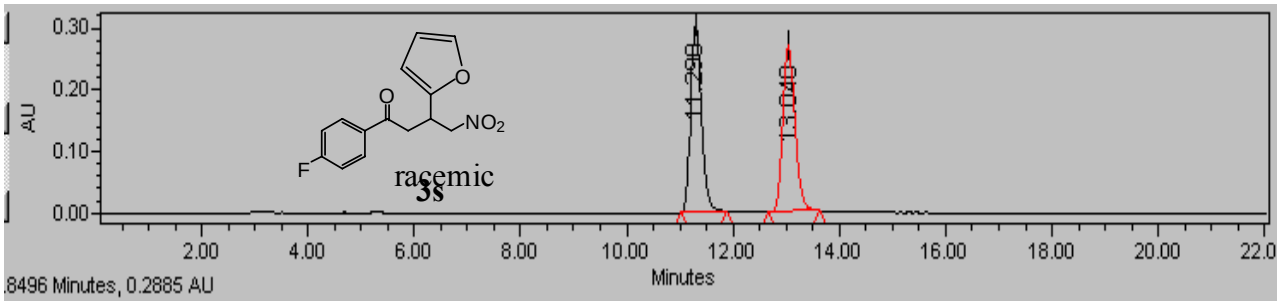
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		13.377	10343132	50.35	601500	bb			Unknown
2		15.970	10199754	49.65	504941	bb			Unknown



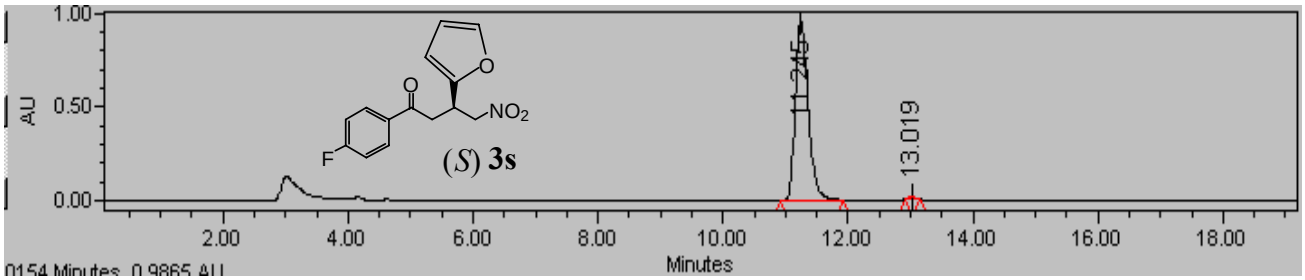
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		13.284	1066640	99.60	62752	bb			Unknown
2		15.884	4282	0.40	364	bb			Unknown



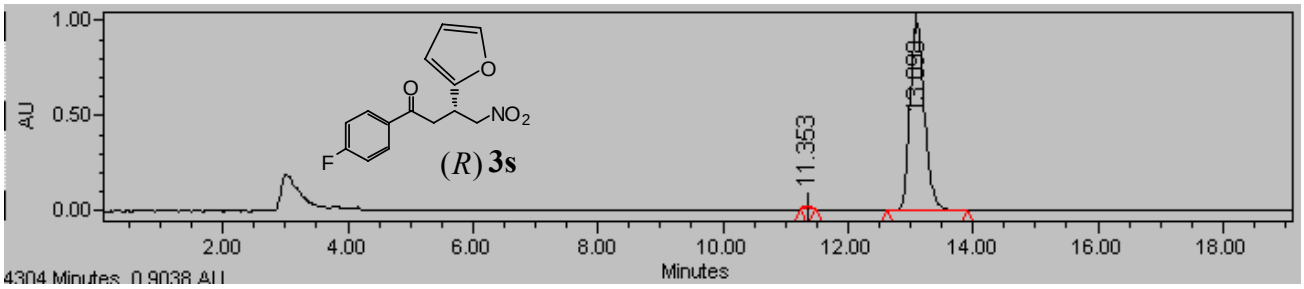
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		13.353	11206	0.78	1162	bb			Unknown
2		15.876	1432048	99.22	71218	bb			Unknown



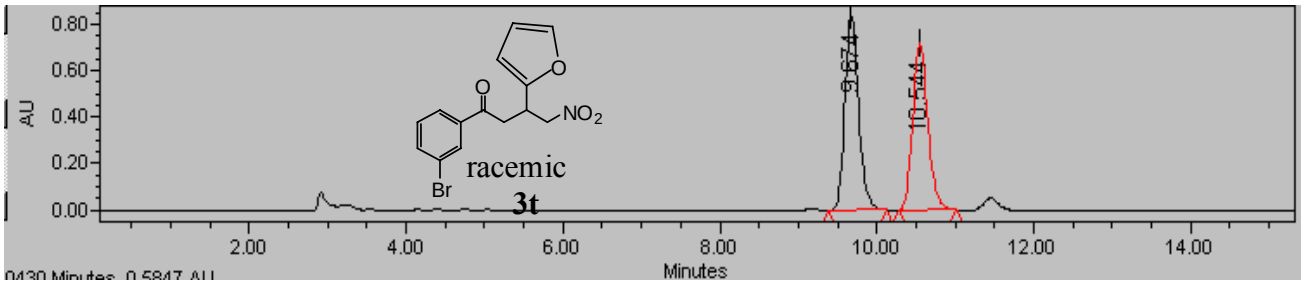
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.290	4236015	50.01	306995	bb			Unknown
2		13.040	4234056	49.99	268467	bb			Unknown



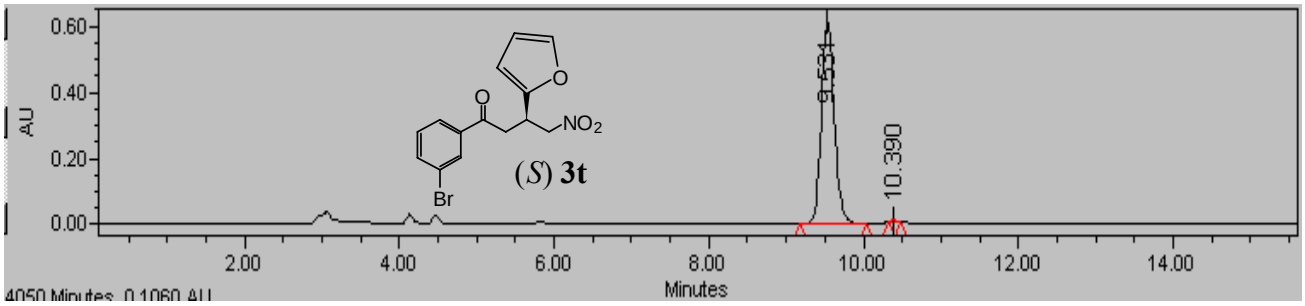
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.245	13570714	99.45	961490	bb			Unknown
2		13.019	74800	0.55	8674	bb			Unknown



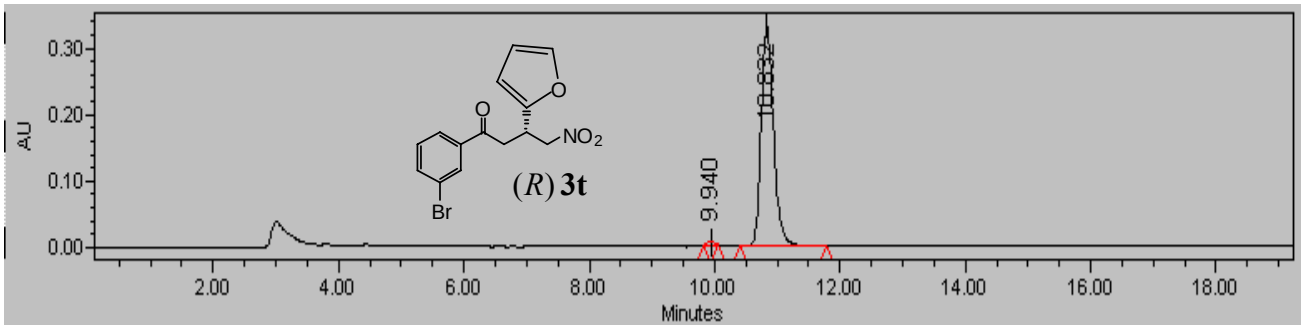
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.353	149736	0.92	16825	bb			Unknown
2		13.098	16046371	99.08	989534	bb			Unknown



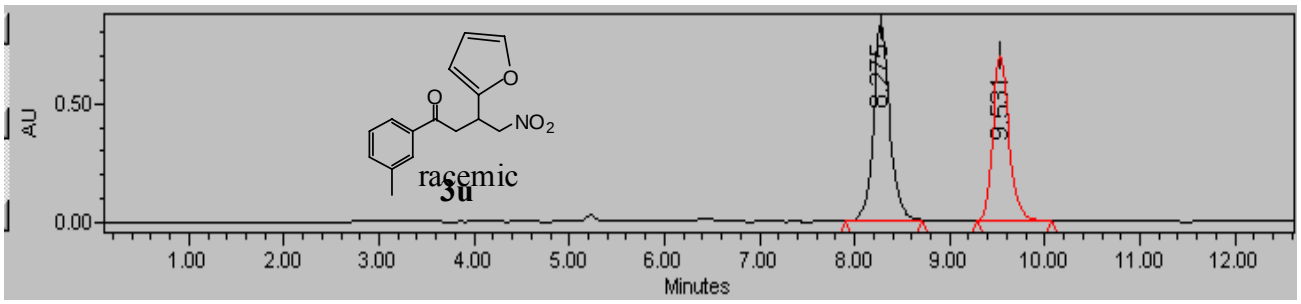
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		9.674	10220084	51.80	840258	bb			Unknown
2		10.544	9508964	48.20	717603	bb			Unknown



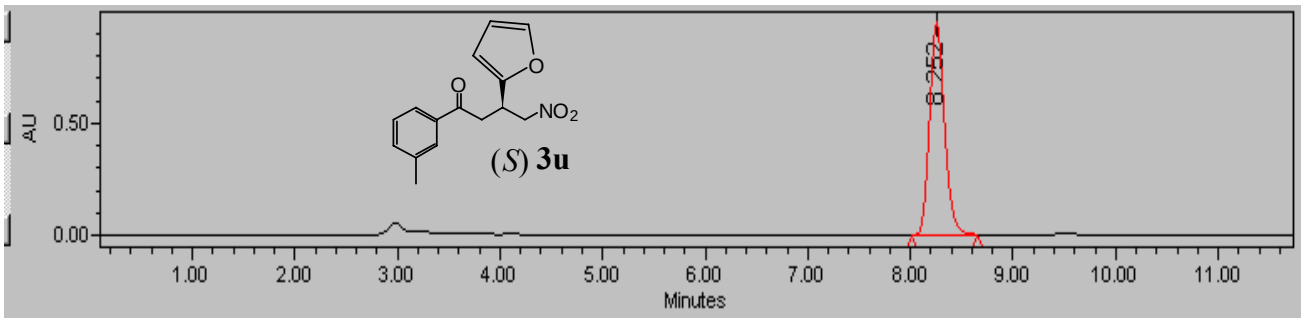
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		9.531	7149186	99.73	620323	bb			Unknown
2		10.390	19651	0.27	3123	bb			Unknown



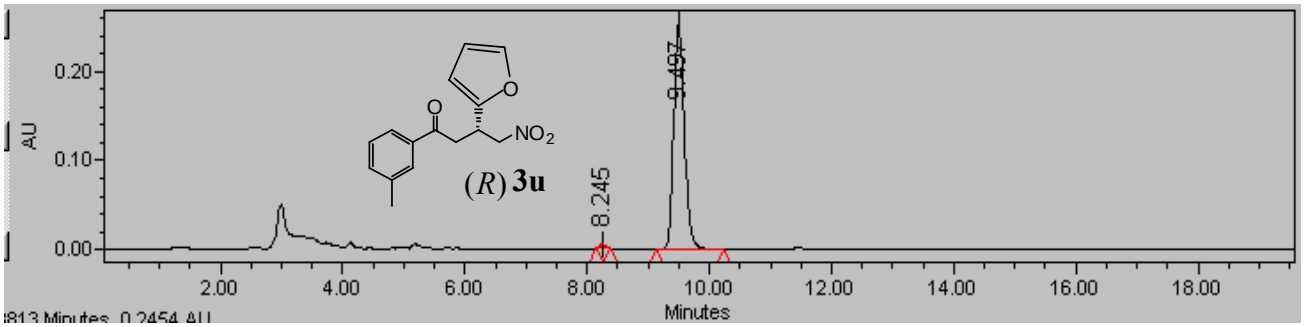
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		9.940	41334	0.89	5028	bb			Unknown
2		10.832	4589035	99.11	334702	bb			Unknown



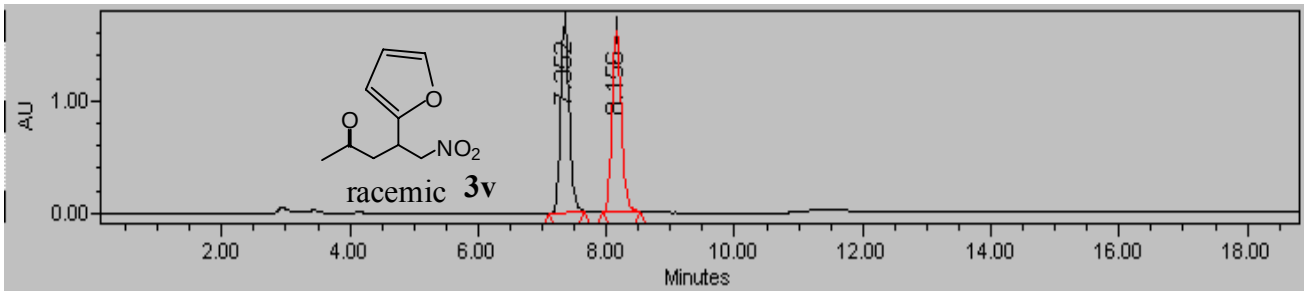
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		8.275	9854980	54.29	829962	bb			Unknown
2		9.531	8297940	45.71	696762	bb			Unknown



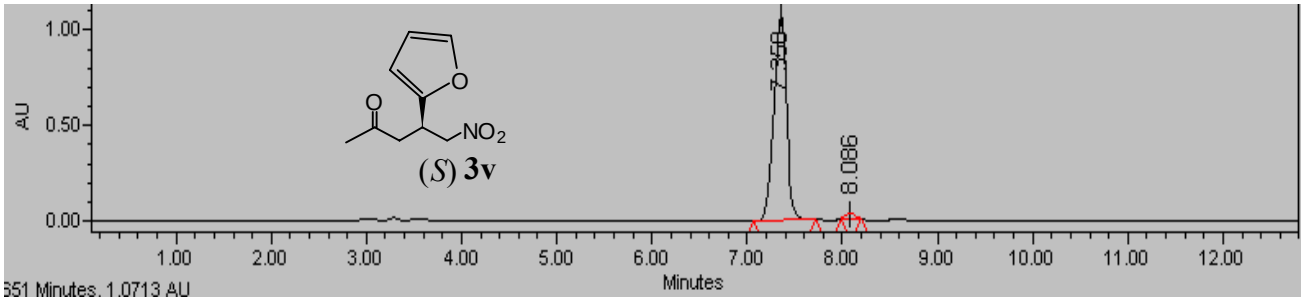
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		8.252	9775916	100.00	946388	bb			Unknown



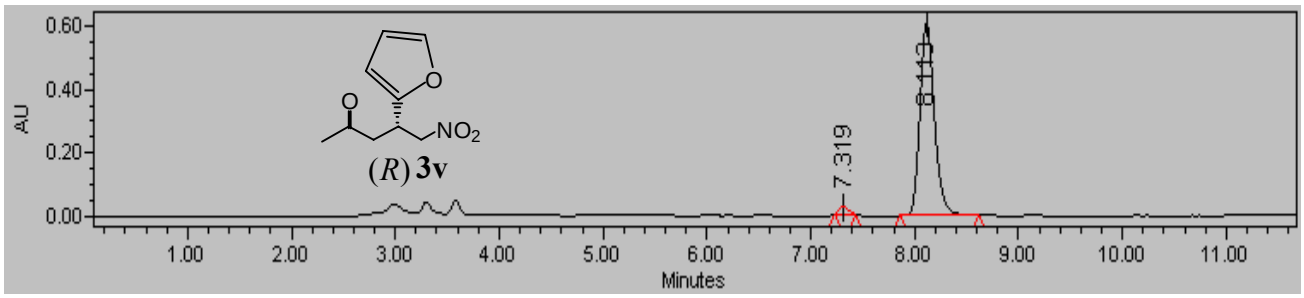
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		8.245	31180	1.01	4246	bb			Unknown
2		9.497	3054735	98.99	255332	bb			Unknown



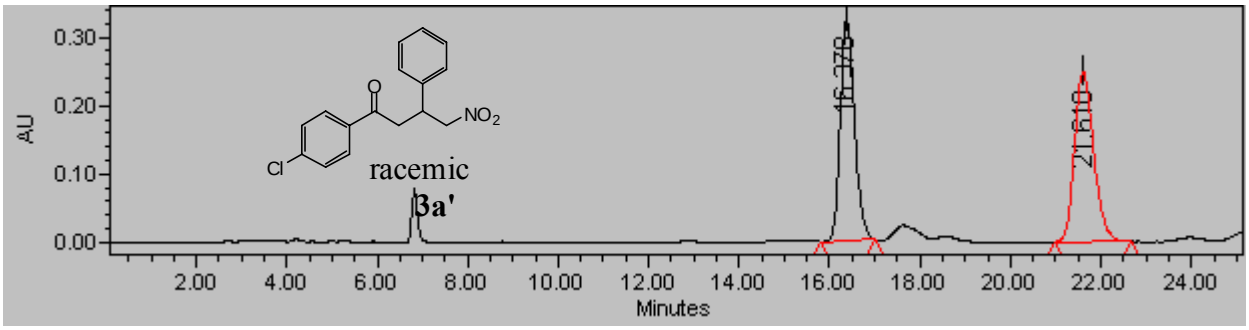
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		7.352	15890629	49.23	1701371	bb			Unknown
2		8.156	16386414	50.77	1618038	bb			Unknown



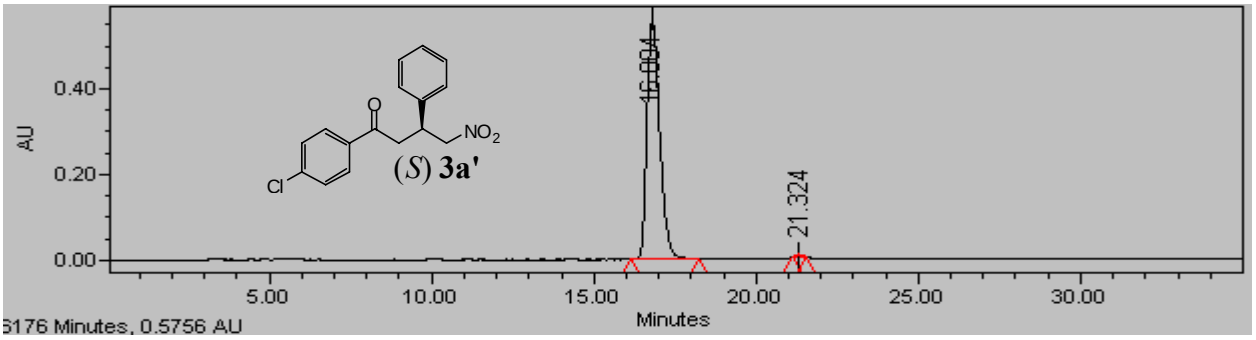
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		7.350	9987785	98.04	1081080	bb			Unknown
2		8.086	200094	1.96	27606	bb			Unknown



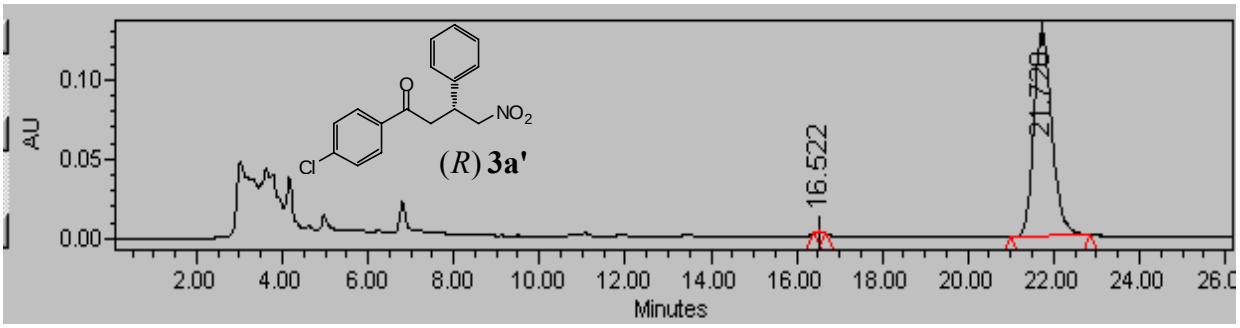
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		7.319	143369	2.35	21731	bb			Unknown
2		8.113	5956920	97.65	612058	bb			Unknown



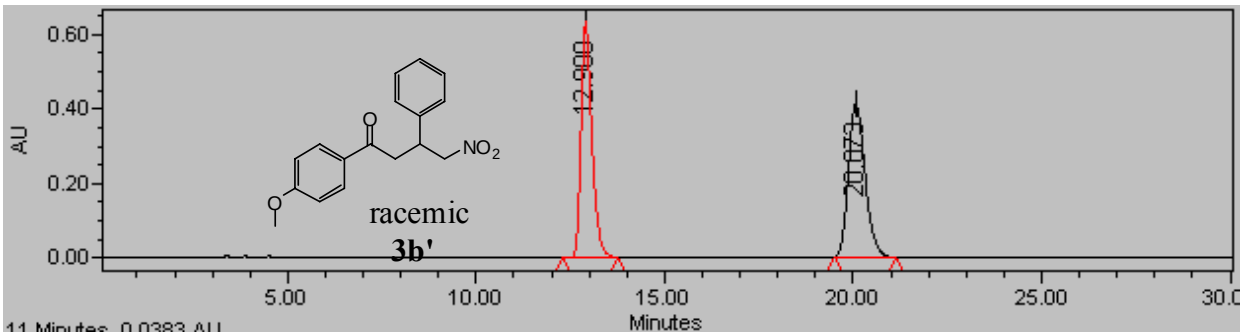
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		16.378	7080841	49.33	326405	bb			Unknown
2		21.610	7272969	50.67	248767	bb			Unknown



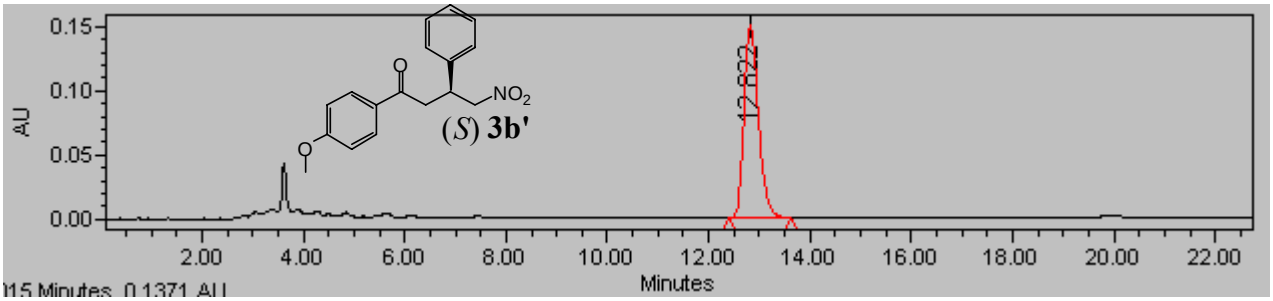
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		16.804	15196342	99.54	561874	bb			Unknown
2		21.324	70686	0.46	4809	bb			Unknown



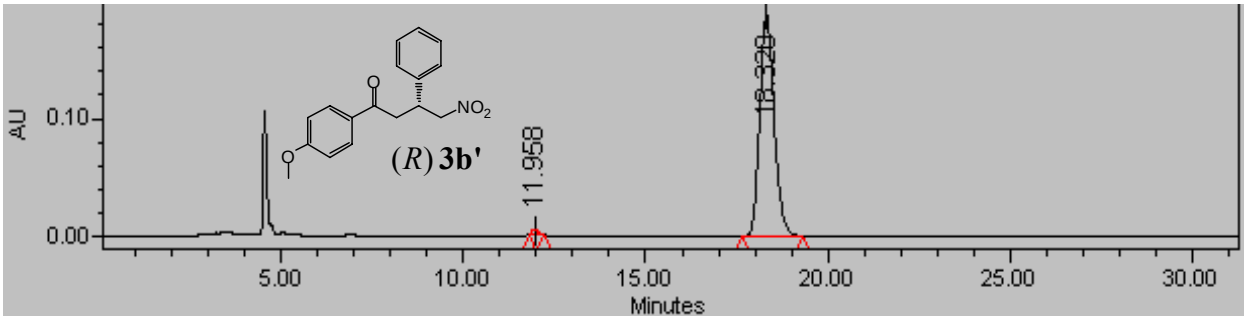
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		16.522	14136	0.37	1374	bb			Unknown
2		21.728	3768331	99.63	129000	bb			Unknown



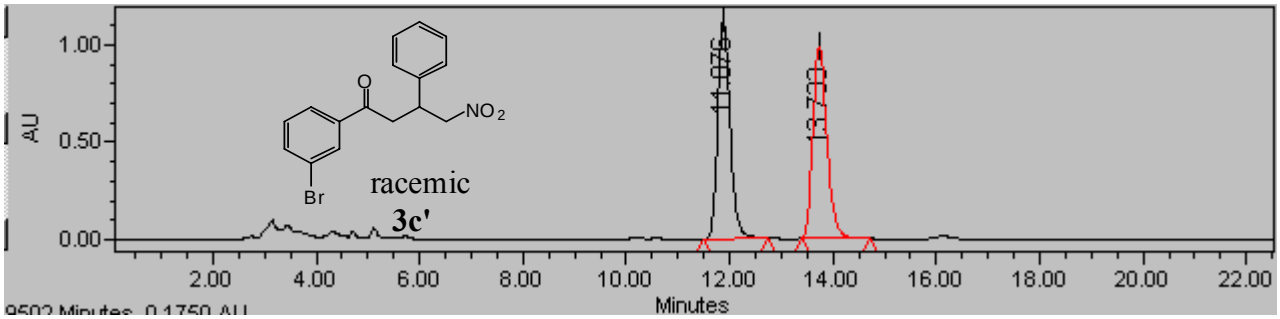
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		12.900	12717655	50.07	632446	bb			Unknown
2		20.073	12681705	49.93	410557	bb			Unknown



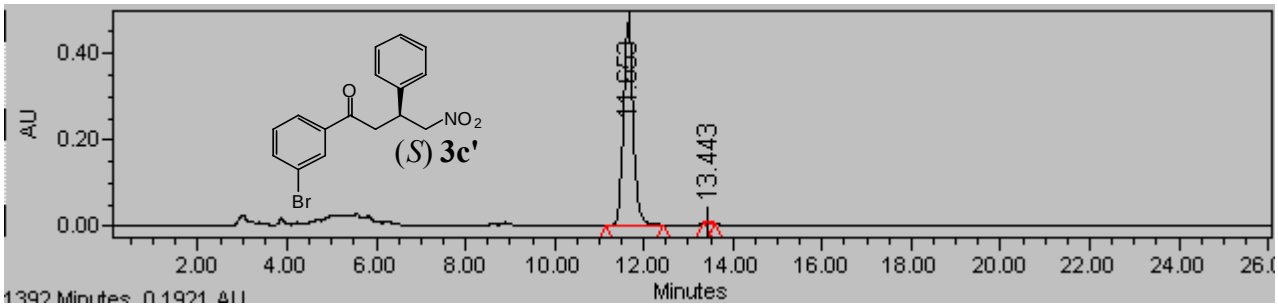
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		12.822	2964598	100.00	151074	bb			Unknown



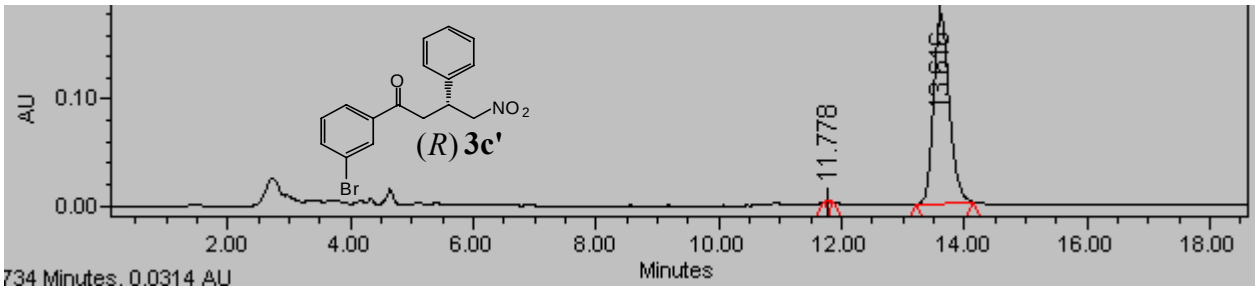
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.958	56745	1.08	4146	bb			Unknown
2		18.320	5188091	98.92	189303	bb			Unknown



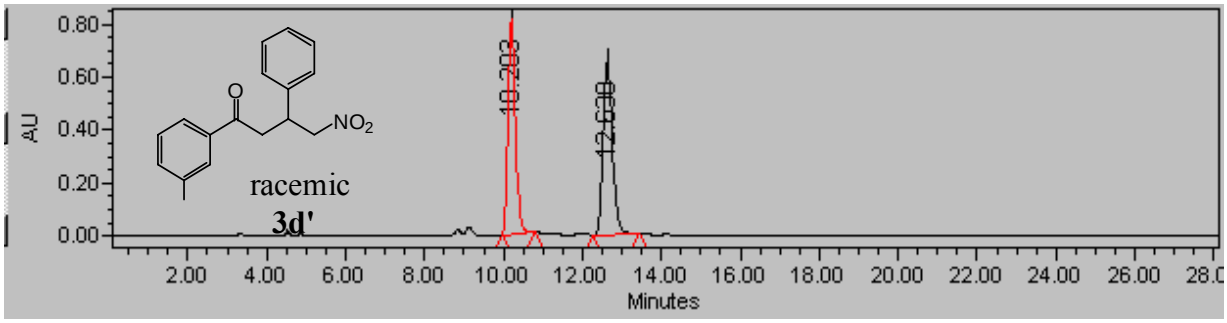
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.876	18678067	50.42	1135199	bb			Unknown
2		13.733	18366054	49.58	983054	bb			Unknown



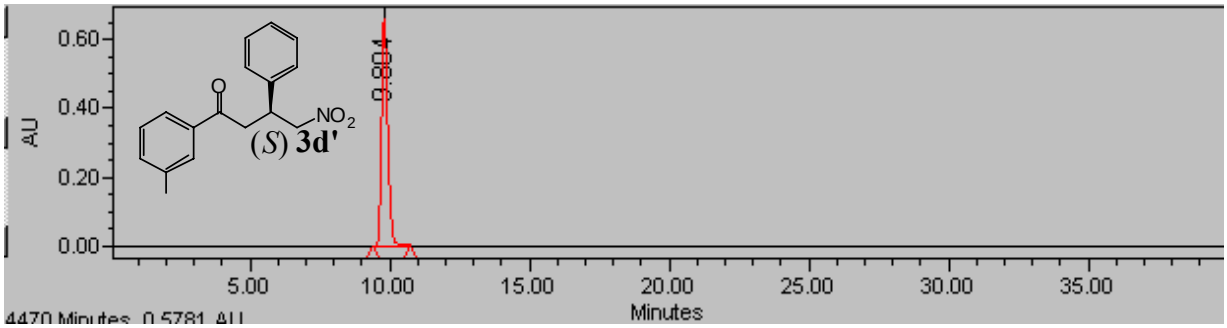
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.653	7279565	99.37	472396	bb			Unknown
2		13.443	45999	0.63	4971	bb			Unknown



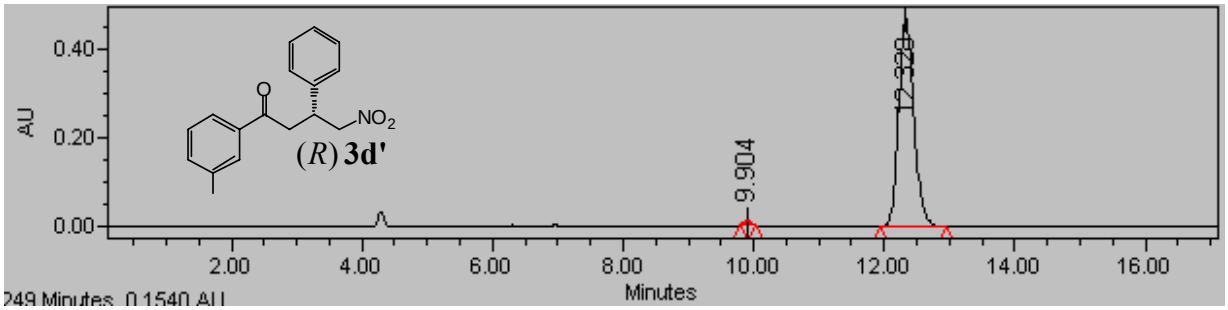
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.778	6249	0.19	977	bb			Unknown
2		13.616	3199256	99.81	177660	bb			Unknown



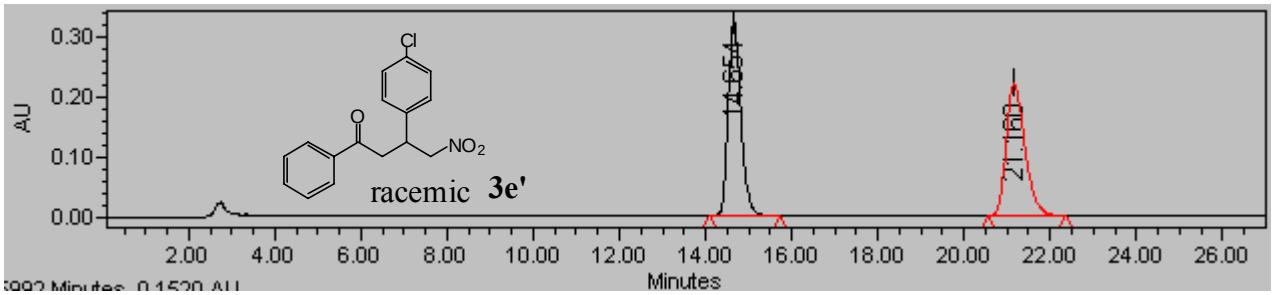
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.203	10412057	49.61	808914	bb			Unknown
2		12.630	10574298	50.39	651455	bb			Unknown



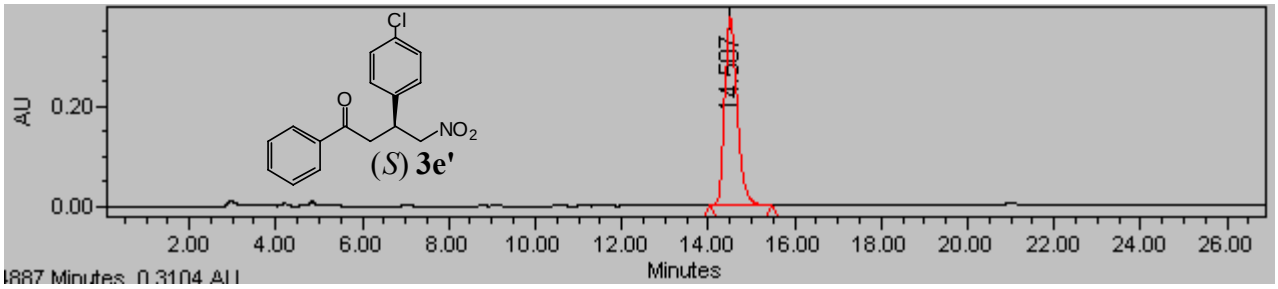
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		9.804	10165676	100.00	661670	bb			Unknown



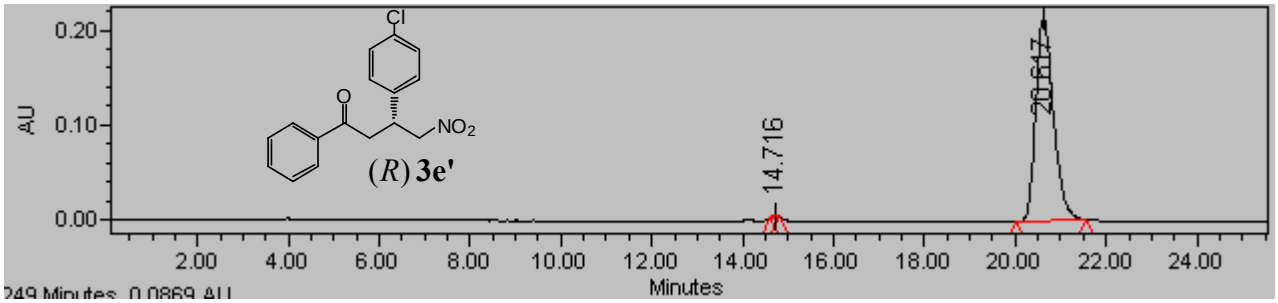
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		9.904	69219	0.90	8447	bb			Unknown
2		12.328	7593140	99.10	468855	bb			Unknown



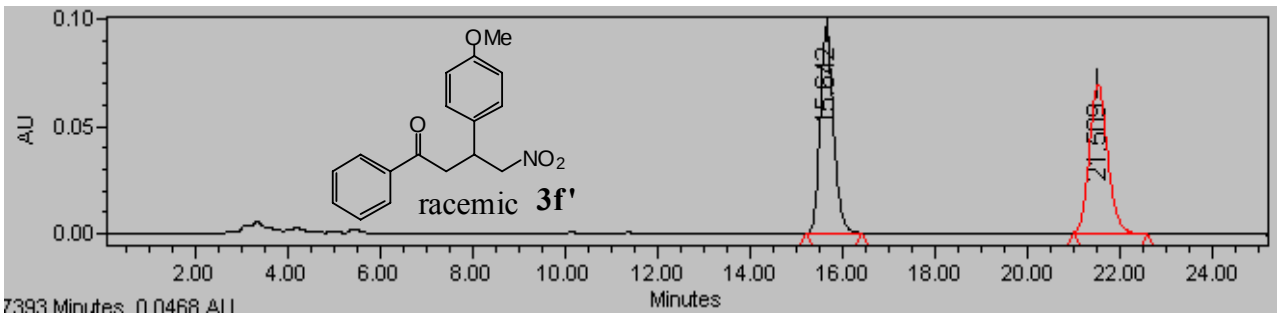
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		14.654	6517351	49.86	323649	bb			Unknown
2		21.160	6553144	50.14	219754	bb			Unknown



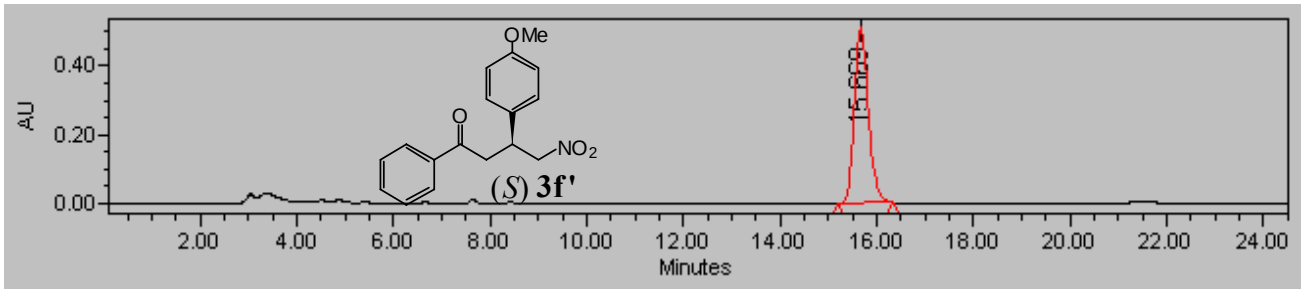
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		14.507	7560526	100.00	376530	bb			Unknown



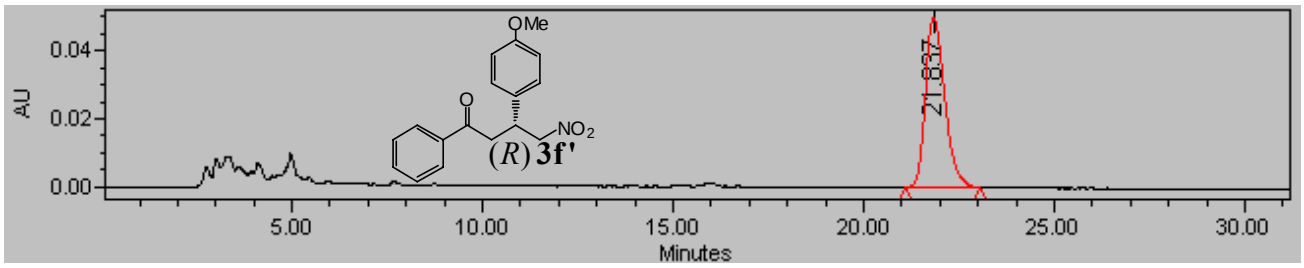
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		14.716	15479	0.27	2041	bb			Unknown
2		20.617	5797970	99.73	214835	bb			Unknown



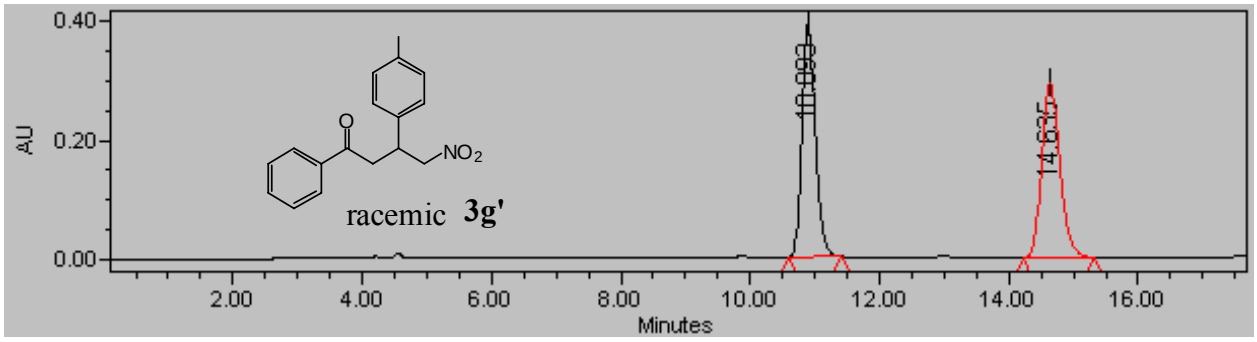
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		15.642	1944192	50.34	96351	bb			Unknown
2		21.509	1918096	49.66	69726	bb			Unknown



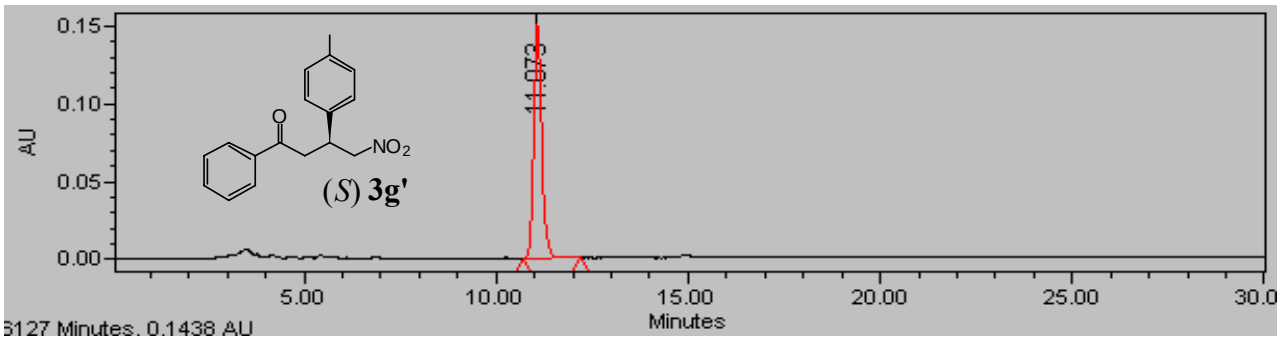
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		15.669	10350131	100.00	510974	bb			Unknown



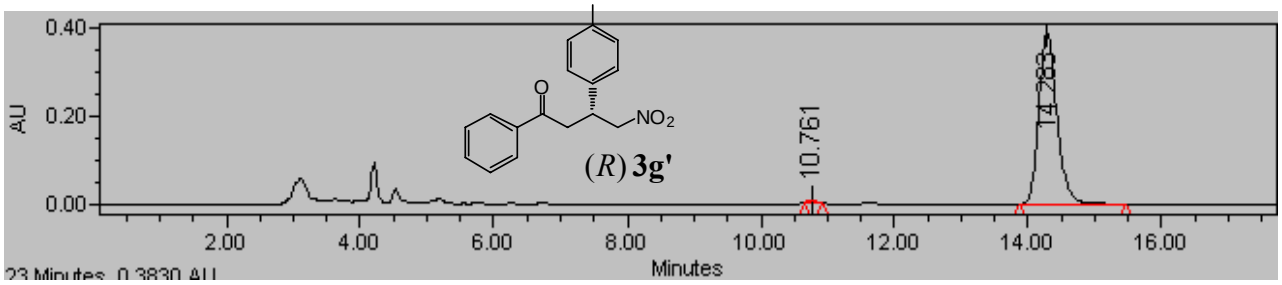
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		21.837	1742774	100.00	49476	bb			Unknown



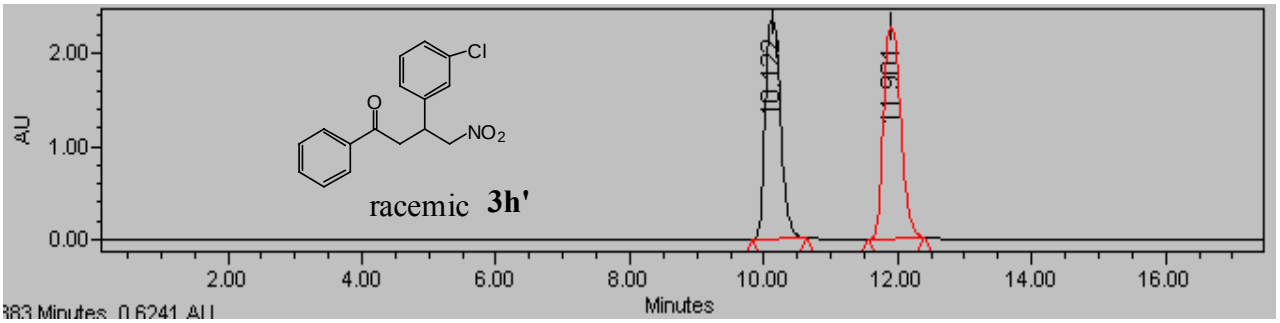
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.893	5646690	49.56	396026	bb			Unknown
2		14.635	5747983	50.44	295825	bb			Unknown



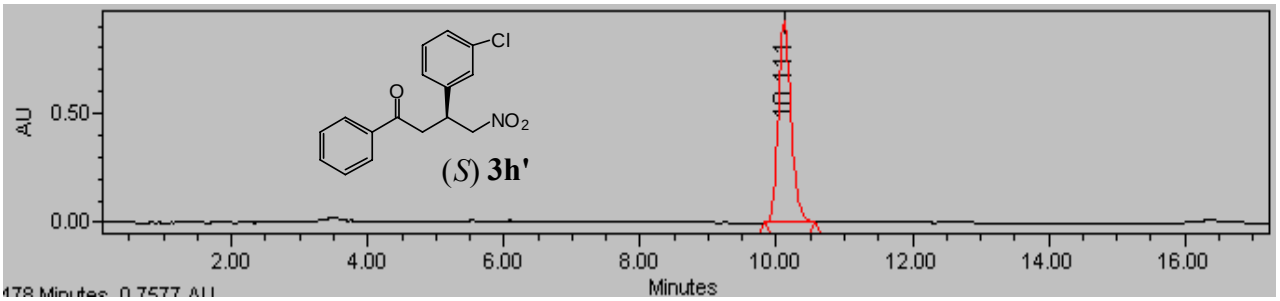
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.073	2187901	100.00	149932	bb			Unknown



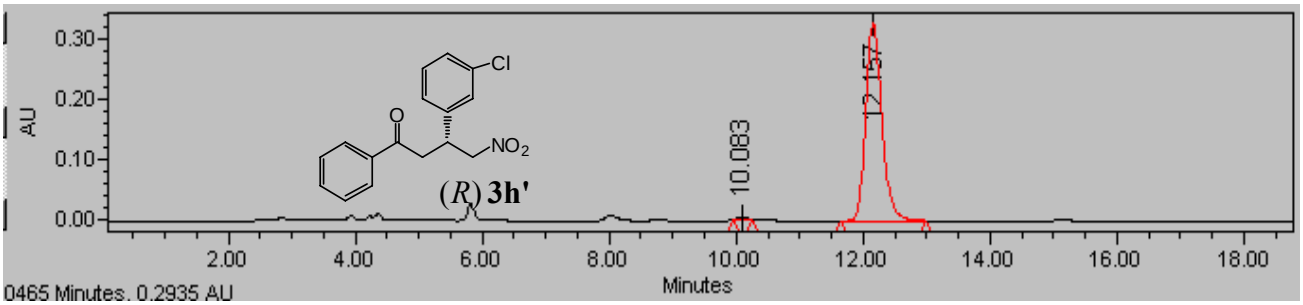
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.761	61923	0.84	6667	bb			Unknown
2		14.283	7277761	99.16	388407	bb			Unknown



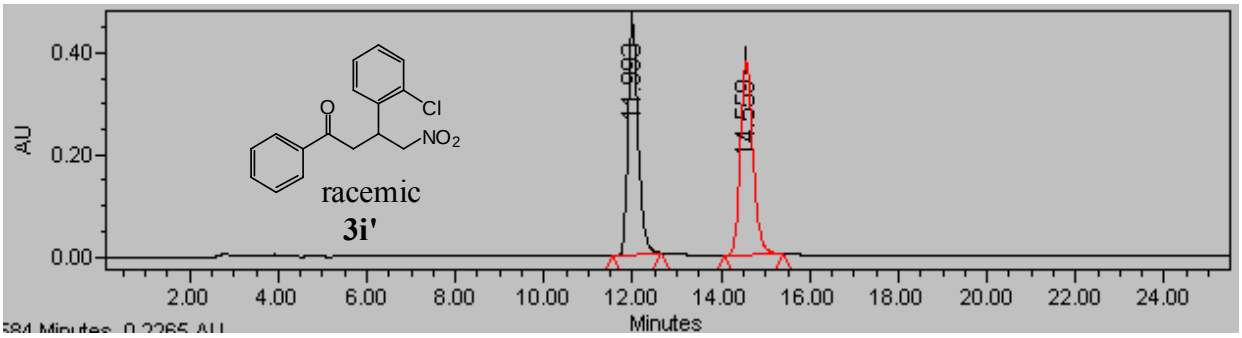
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.122	37641005	48.37	2350498	bb			Unknown
2		11.901	40172062	51.63	2267148	bb			Unknown



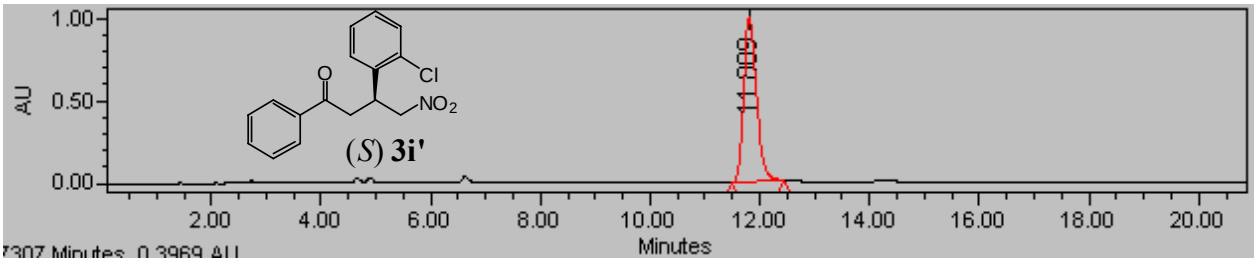
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.111	12010507	100.00	920138	bb			Unknown



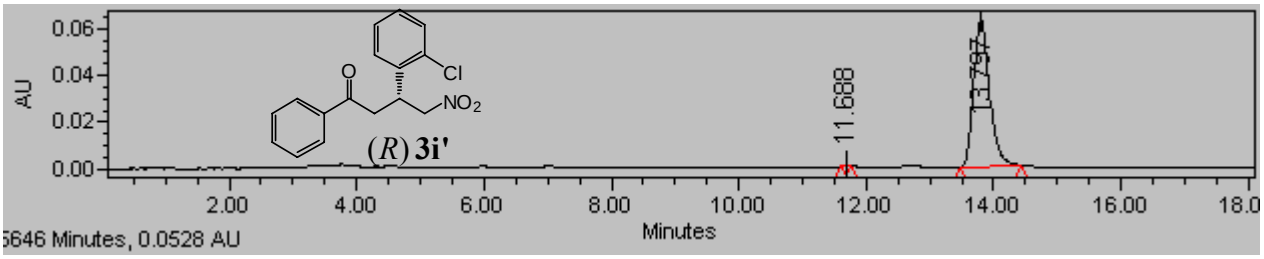
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.083	37600	0.65	3764	bb			Unknown
2		12.157	5713314	99.35	327311	bb			Unknown



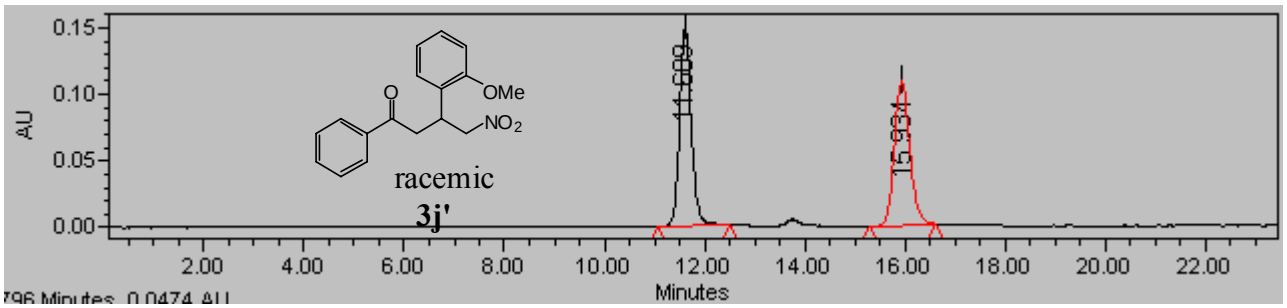
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.993	7444701	49.81	455017	bb			Unknown
2		14.559	7501408	50.19	380617	bb			Unknown



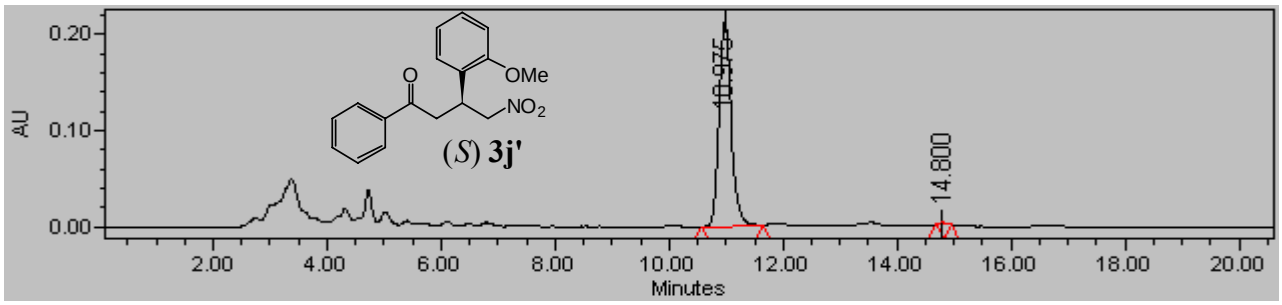
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.809	15889389	100.00	998982	bb			Unknown



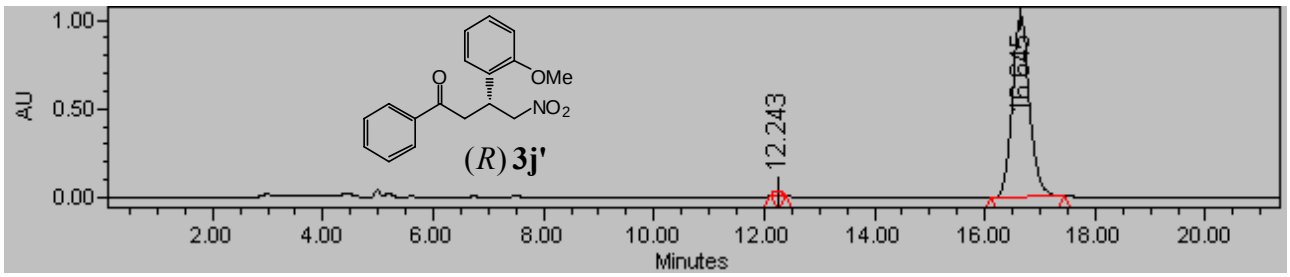
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.688	2970	0.28	492	bb			Unknown
2		13.797	1076404	99.72	63157	bb			Unknown



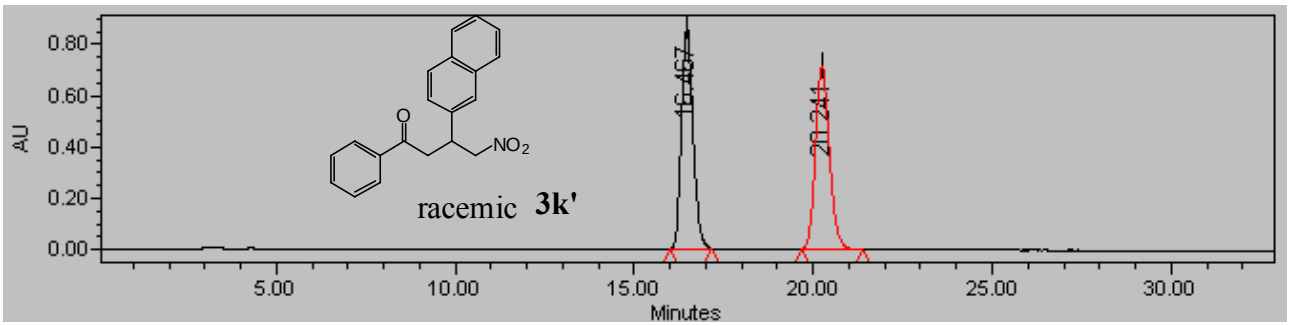
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		11.609	2432952	51.37	151763	bb			Unknown
2		15.934	2302725	48.63	108572	bb			Unknown



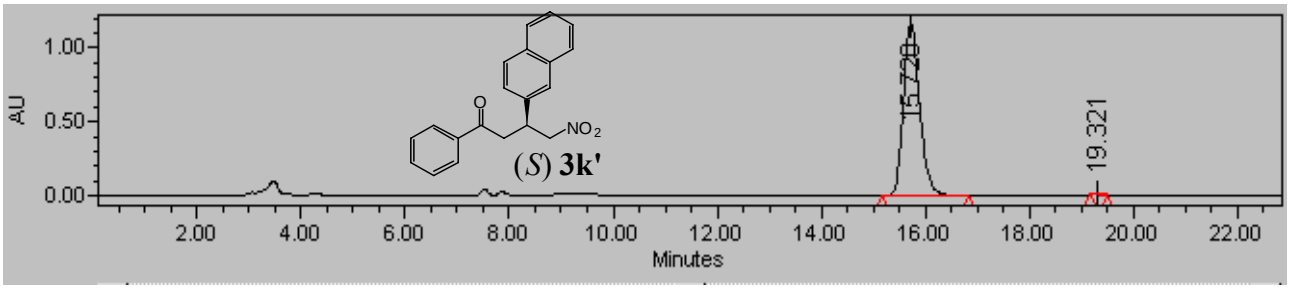
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		10.975	3103394	99.44	214152	bb			Unknown
2		14.800	17587	0.56	1752	bb			Unknown



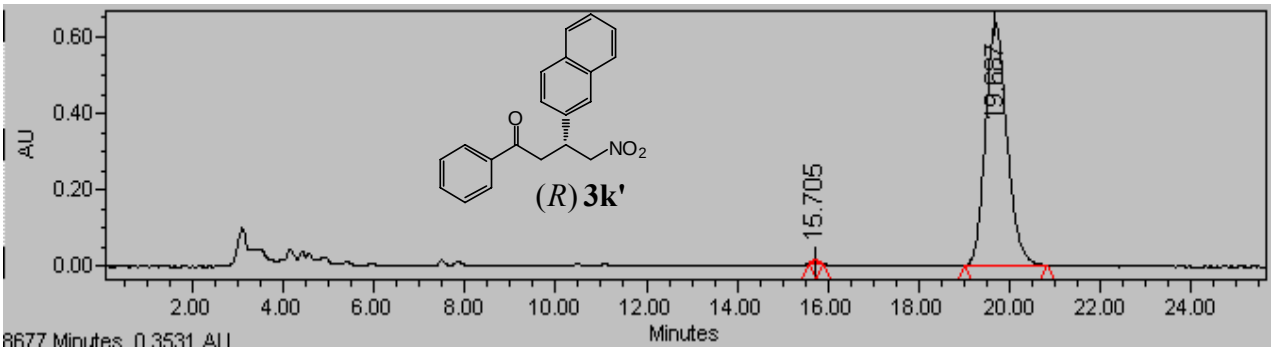
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		12.243	214993	0.96	22303	bb			Unknown
2		16.645	22242080	99.04	1019453	bb			Unknown



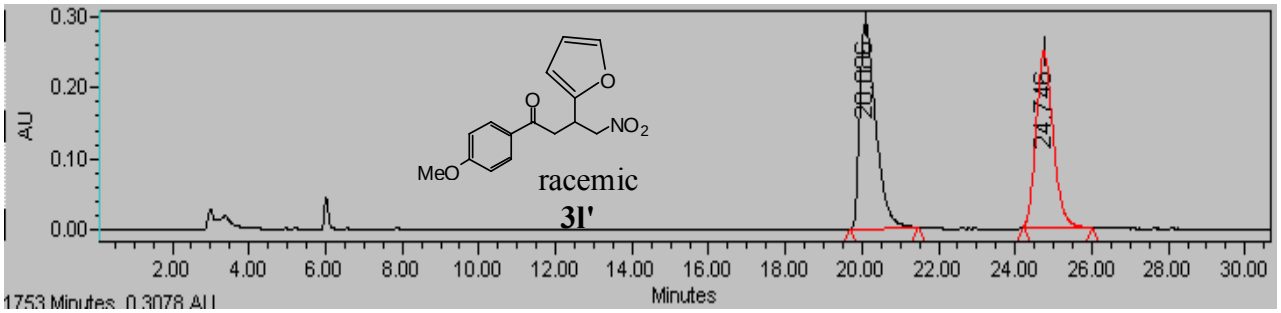
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		16.467	19310894	49.76	867391	bb			Unknown
2		20.241	19497351	50.24	715471	bb			Unknown



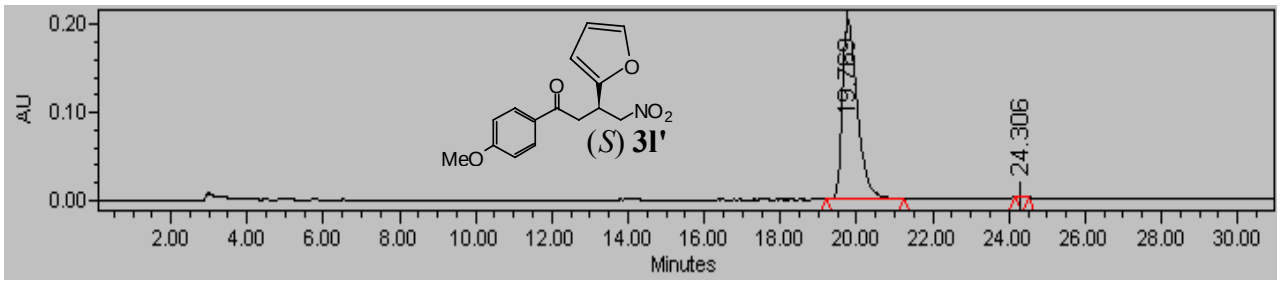
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		15.720	25250286	99.70	1167990	bb			Unknown
2		19.321	76606	0.30	6202	bb			Unknown



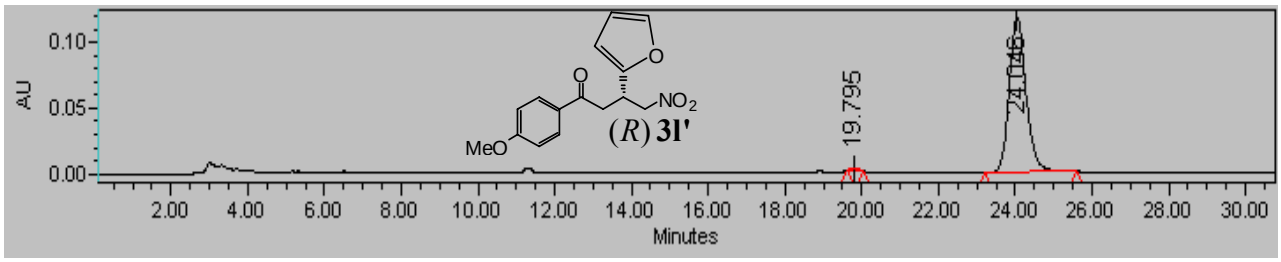
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		15.705	76041	0.36	6402	bb			Unknown
2		19.687	20878619	99.64	632875	bb			Unknown



	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		20.086	8353818	52.44	291028	bb			Unknown
2		24.746	7577557	47.56	245980	bb			Unknown



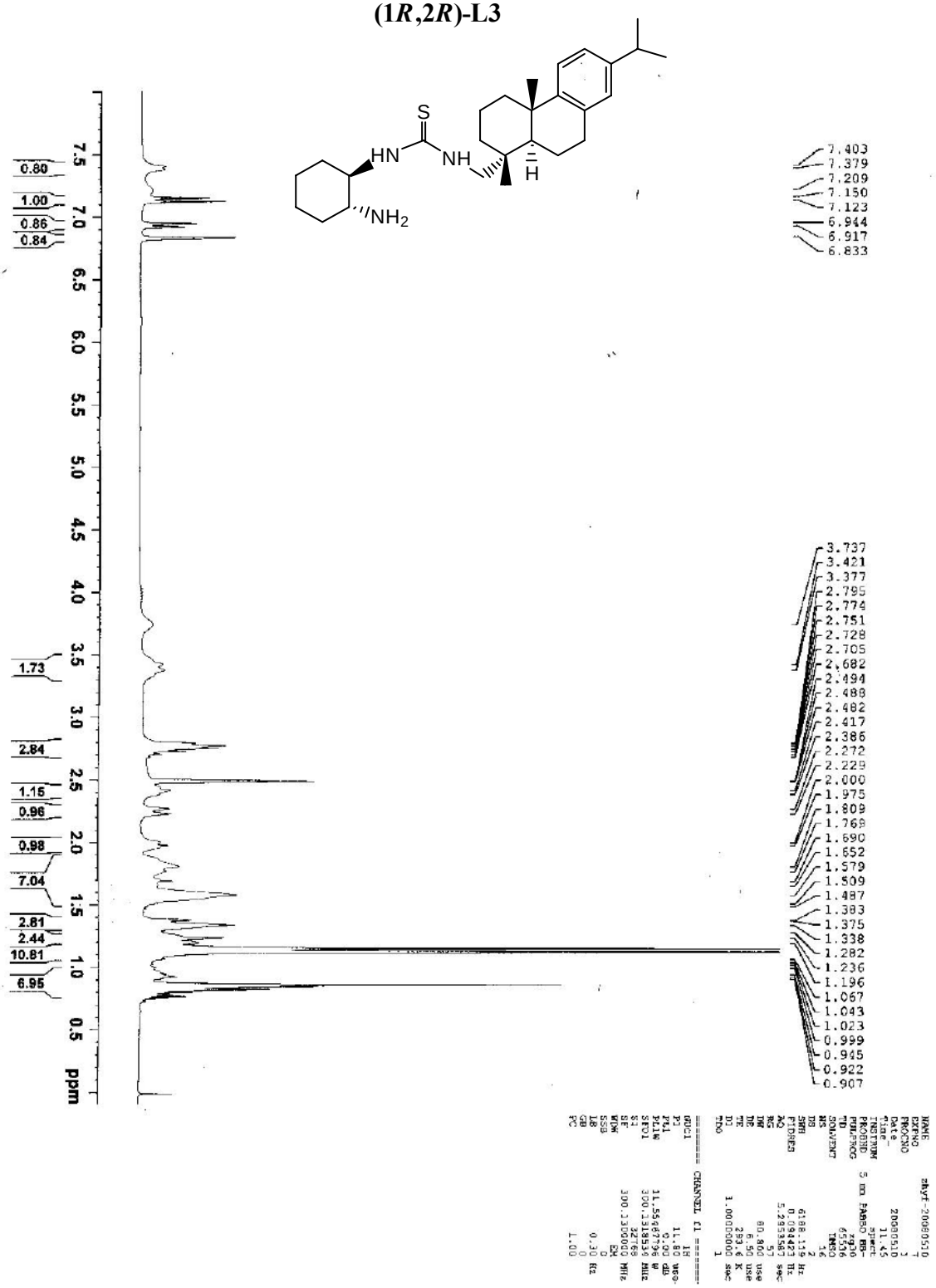
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		19.769	5640447	99.62	205166	bb			Unknown
2		24.306	21408	0.38	1507	bb			Unknown



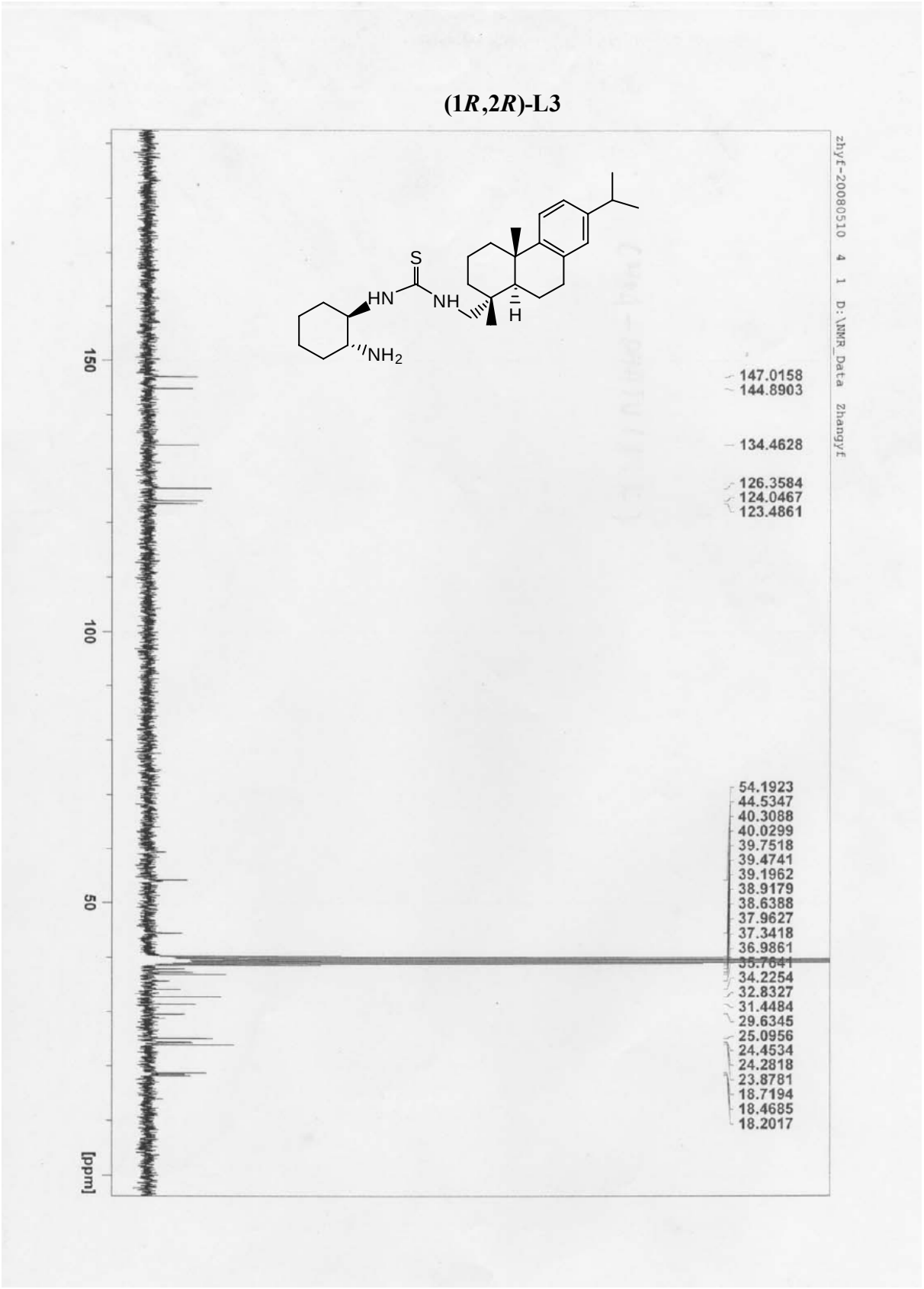
	Name	Retention Time	Area	% Area	Height	Int Type	Amount	Units	Peak Type
1		19.795	28506	0.79	1870	bb			Unknown
2		24.046	3589486	99.21	117371	bb			Unknown

11.0 Copies of NMR spectra of chiral Michael products

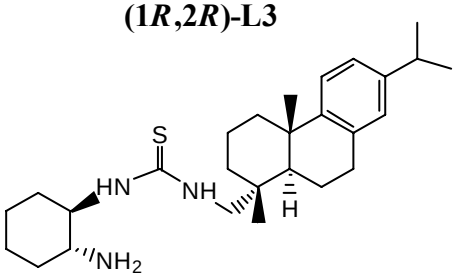
(1*R*,2*R*)-L3

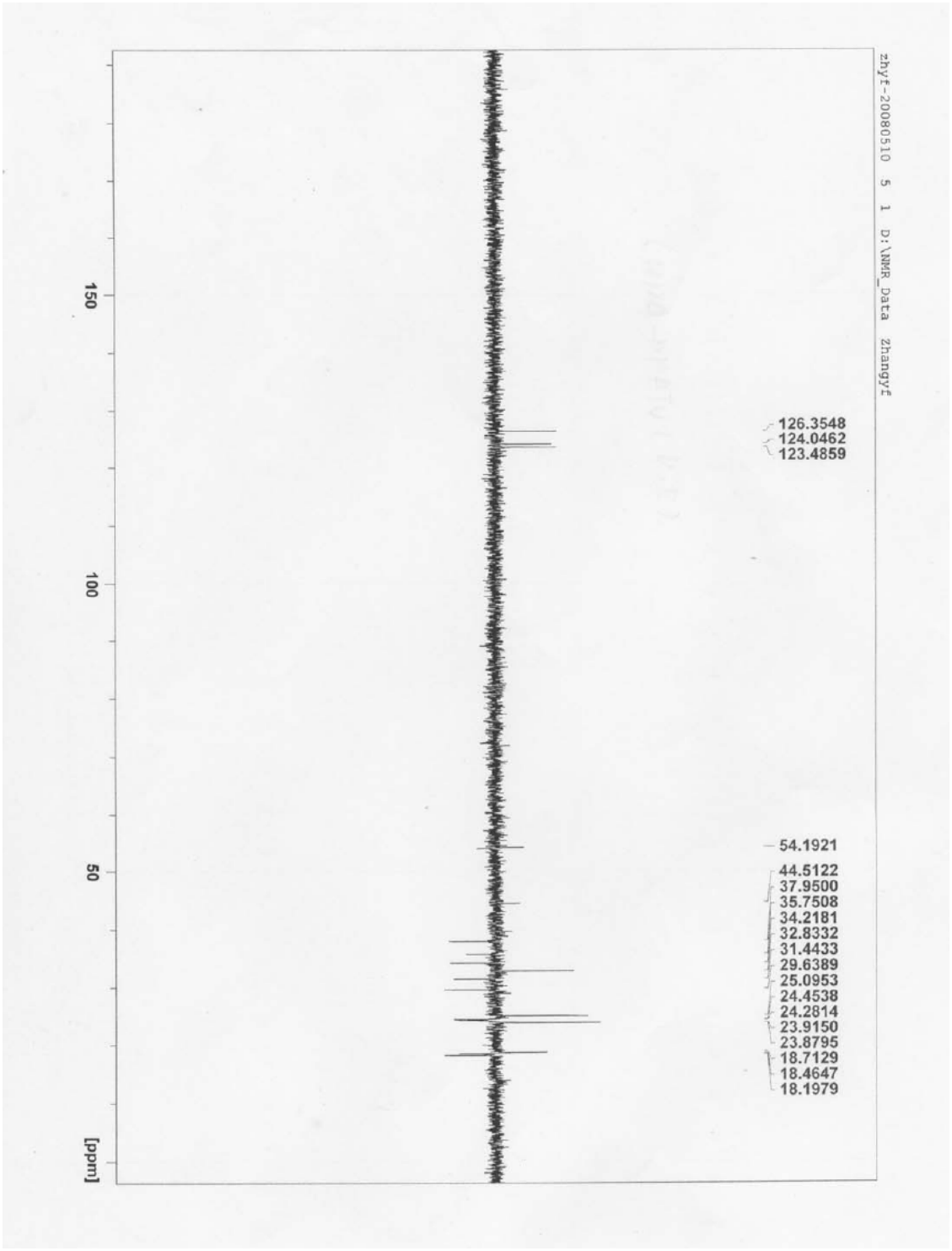


(1*R*,2*R*)-L3

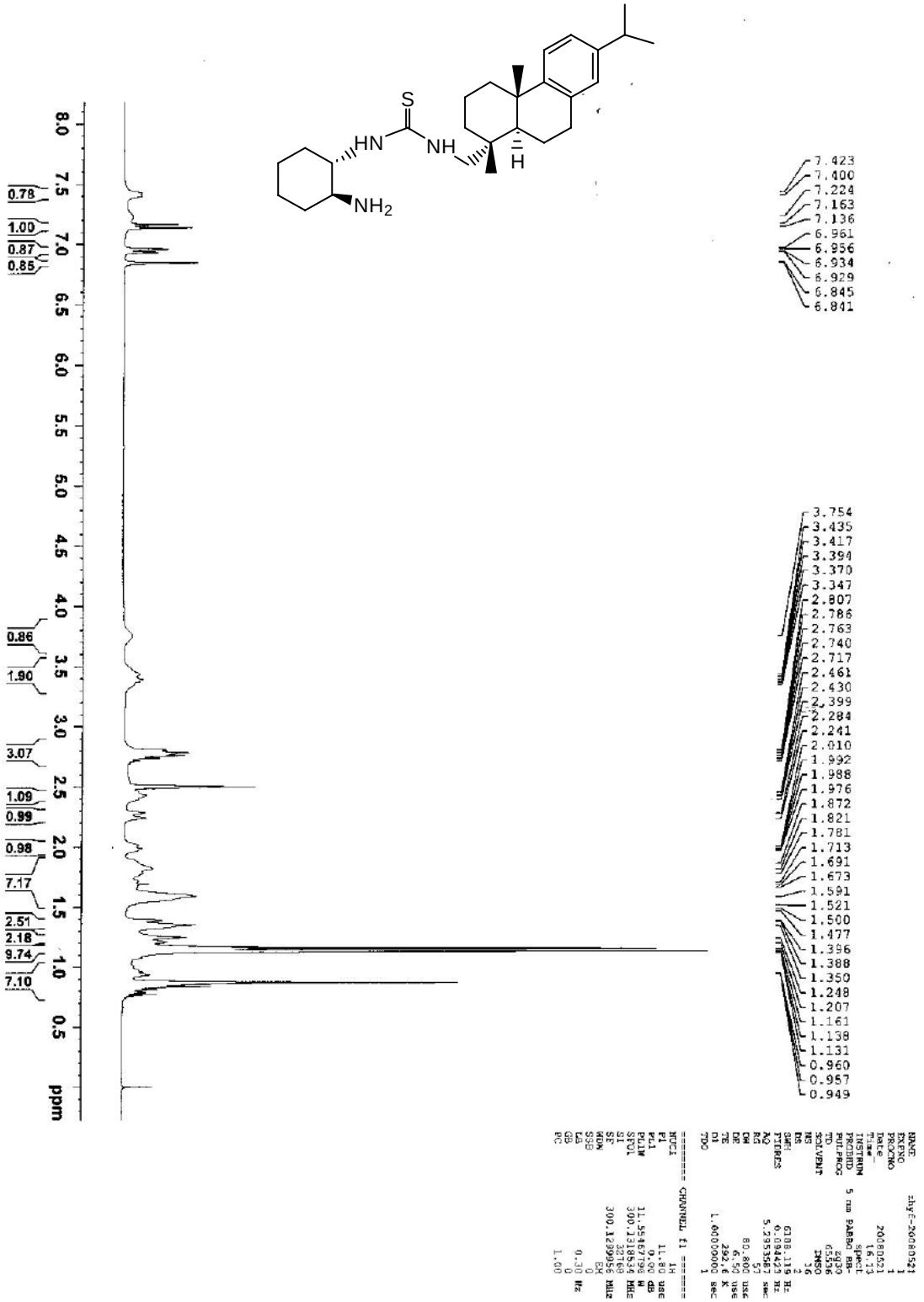

zhyf-20080510 4 1 D:\NMR_Data Zhangyf
147.0158
144.8903
134.4628
126.3584
124.0467
123.4861
54.1923
44.5347
40.3088
40.0299
39.7518
39.4741
39.1962
38.9179
38.6388
37.9627
37.3418
36.9861
35.7641
34.2254
32.8327
31.4484
29.6345
25.0956
24.4534
24.2818
23.8781
18.7194
18.4685
18.2017
150
100
50
[ppm]
S
HN
NH
H
NH2

(1*R*,2*R*)-L3

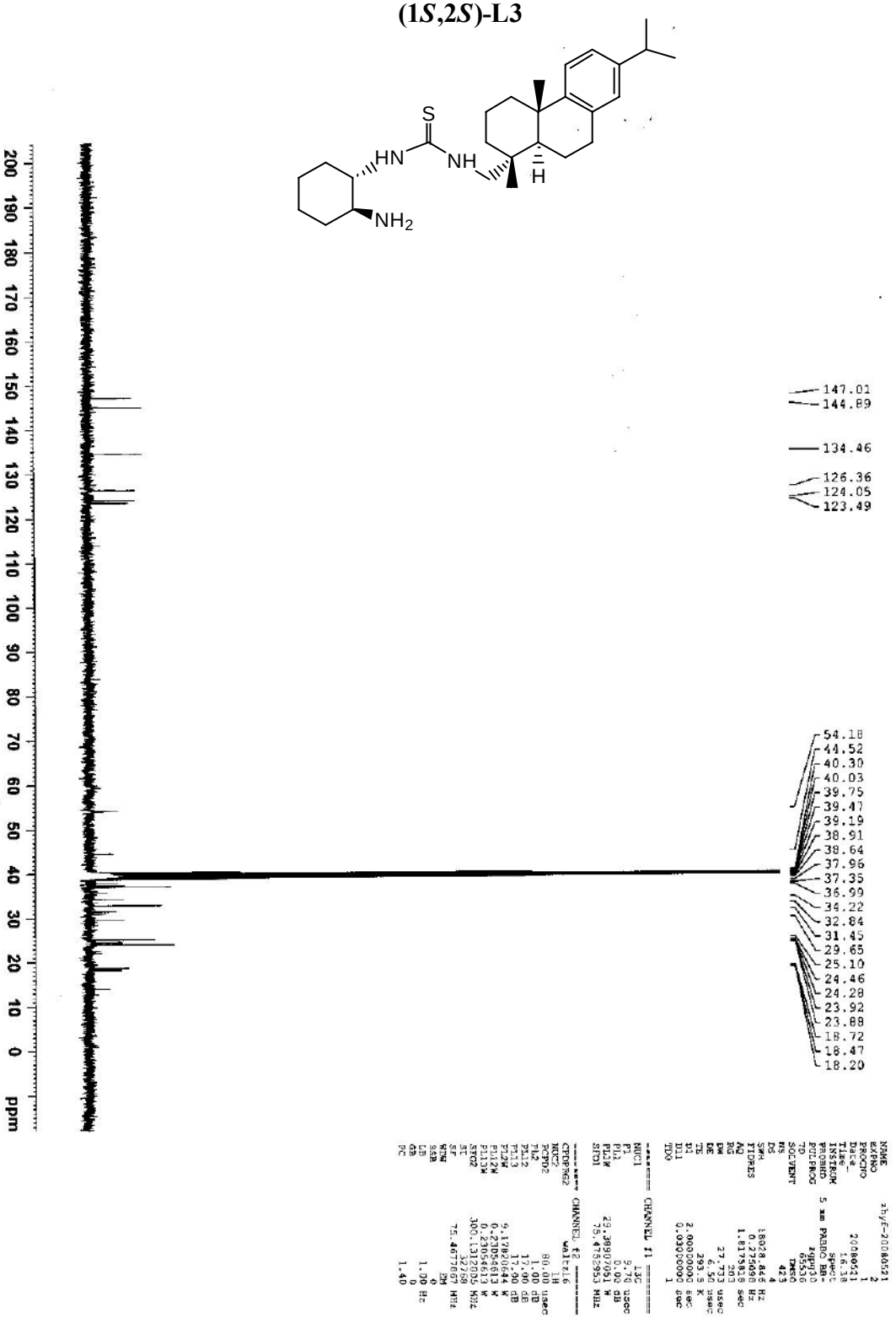

S
HN
NH
H
NH2


zhyf-20080510 5 1 D:\NMR_Data zhangyf
126.3548
124.0462
123.4859
54.1921
44.5122
37.9500
35.7508
34.2181
32.8332
31.4433
29.6389
25.0953
24.4538
24.2814
23.9150
23.8795
18.7129
18.4647
18.1979
150
100
50
[ppm]

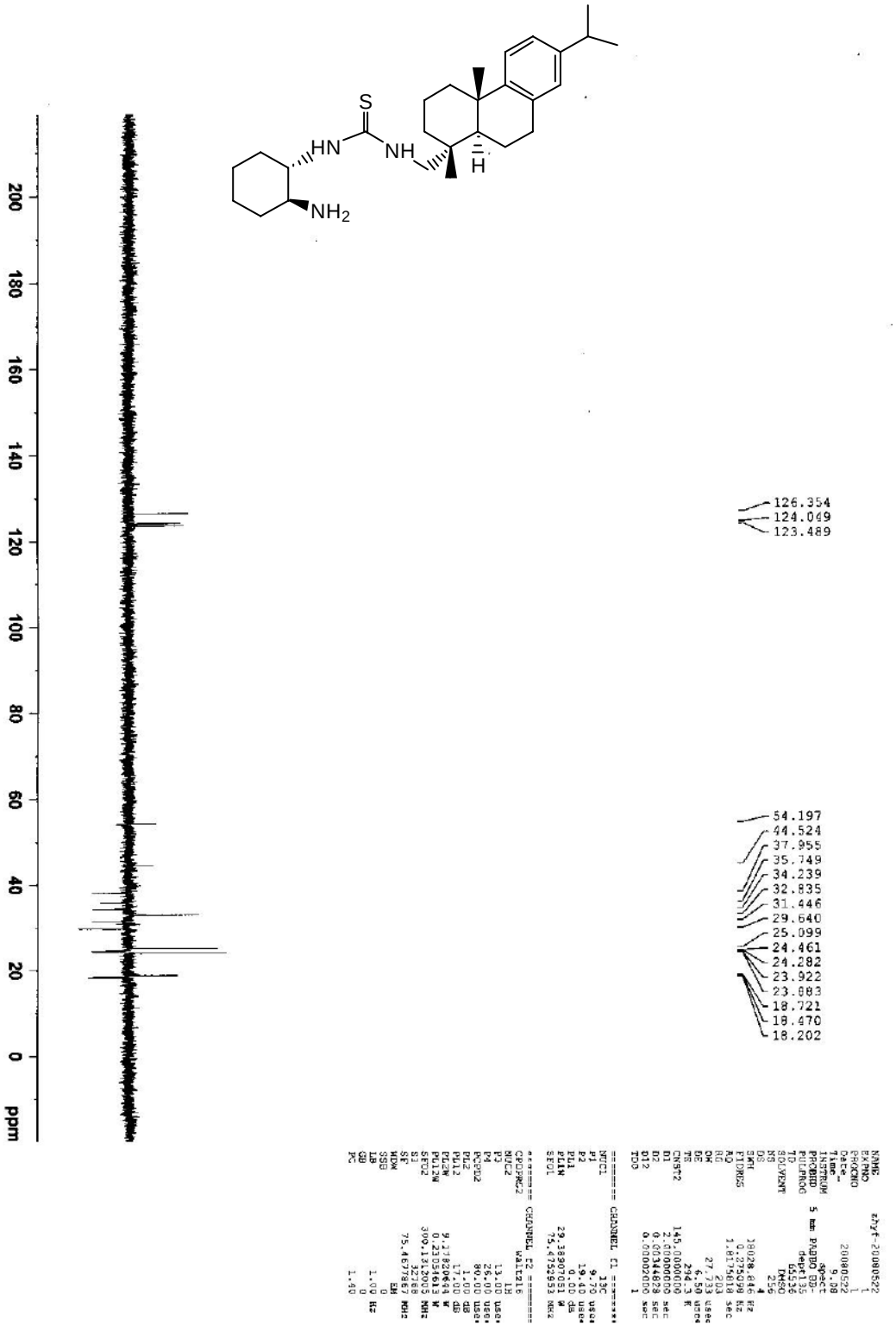
(1*S*,2*S*)-L3


S
HN
NH
H
NH2
7.423
7.400
7.224
7.163
7.136
6.961
6.956
6.934
6.929
6.845
6.841
3.754
3.435
3.417
3.394
3.370
3.347
2.807
2.786
2.763
2.740
2.717
2.461
2.430
2.399
2.284
2.241
2.010
1.992
1.988
1.976
1.872
1.821
1.781
1.713
1.691
1.673
1.591
1.521
1.500
1.477
1.396
1.388
1.350
1.248
1.207
1.161
1.138
1.131
0.960
0.957
0.949
0.78
1.00
0.87
0.85
0.86
1.90
3.07
1.09
0.99
0.98
7.17
2.51
2.18
9.74
7.10
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
ppm

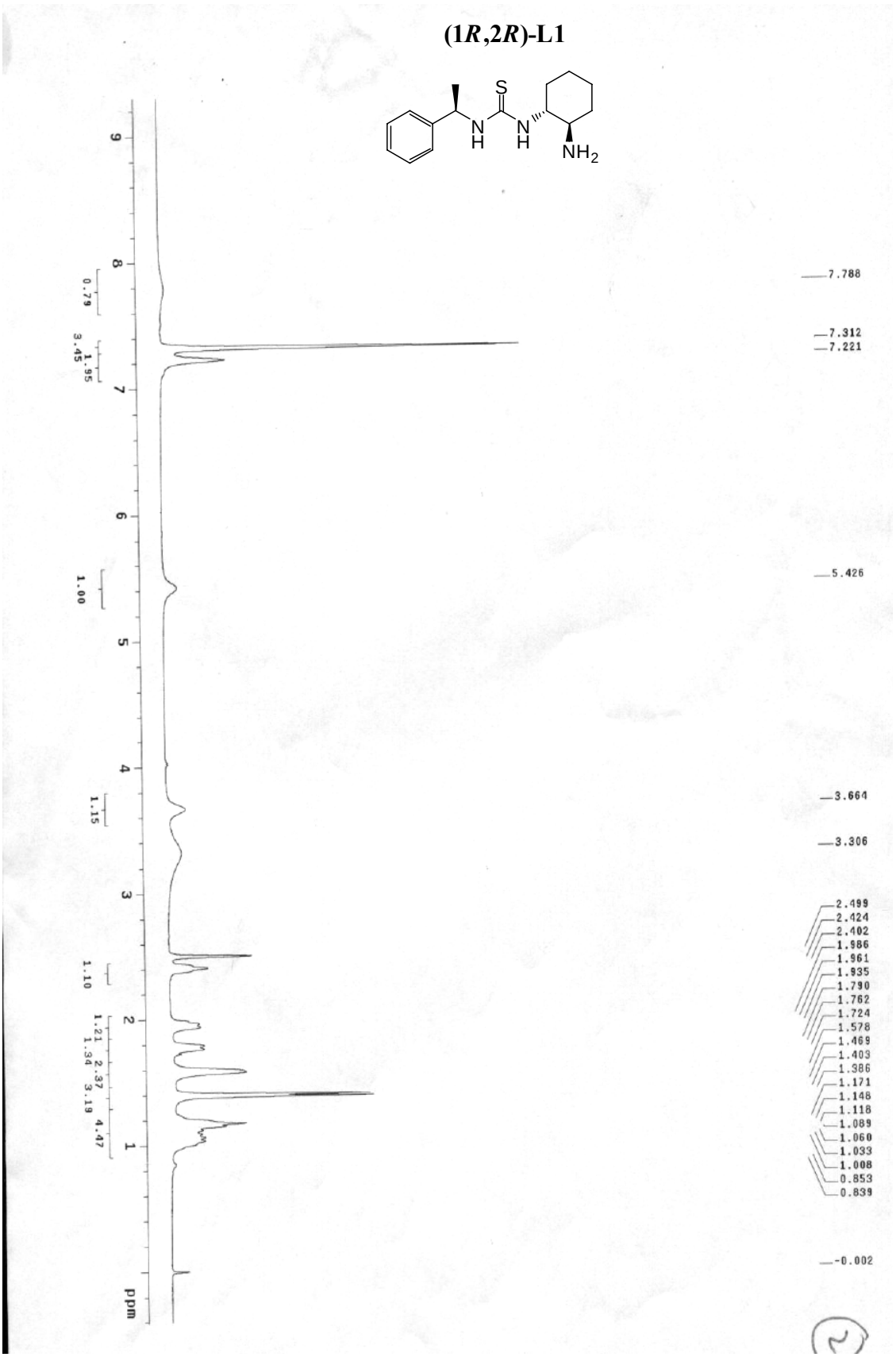
(1*S*,2*S*)-L3


S
HN
NH
H
NH2
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
147.01
144.89
134.46
126.36
124.05
123.49
54.18
44.52
40.30
40.03
39.75
39.47
39.19
38.91
38.64
37.96
37.35
36.99
34.22
32.84
31.45
29.65
25.10
24.46
24.28
23.92
23.88
18.72
18.47
18.20

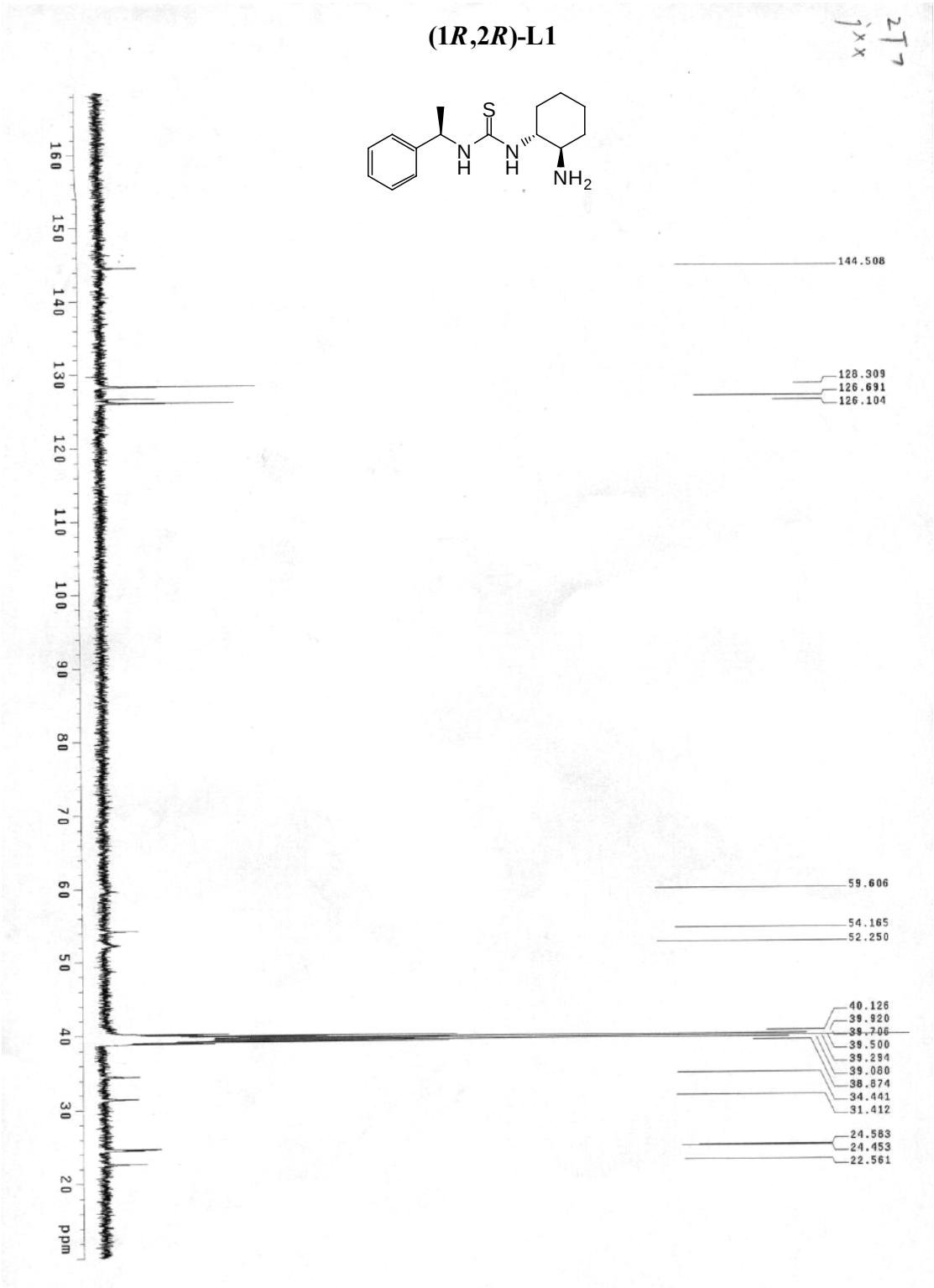
(1*S*,2*S*)-L3


S
HN
NH
H
NH2
200
180
160
140
120
100
80
60
40
20
0
ppm
126.354
124.049
123.489
54.197
44.524
37.955
35.749
34.239
32.835
31.446
29.640
25.099
24.461
24.282
23.922
23.083
18.721
18.470
18.202

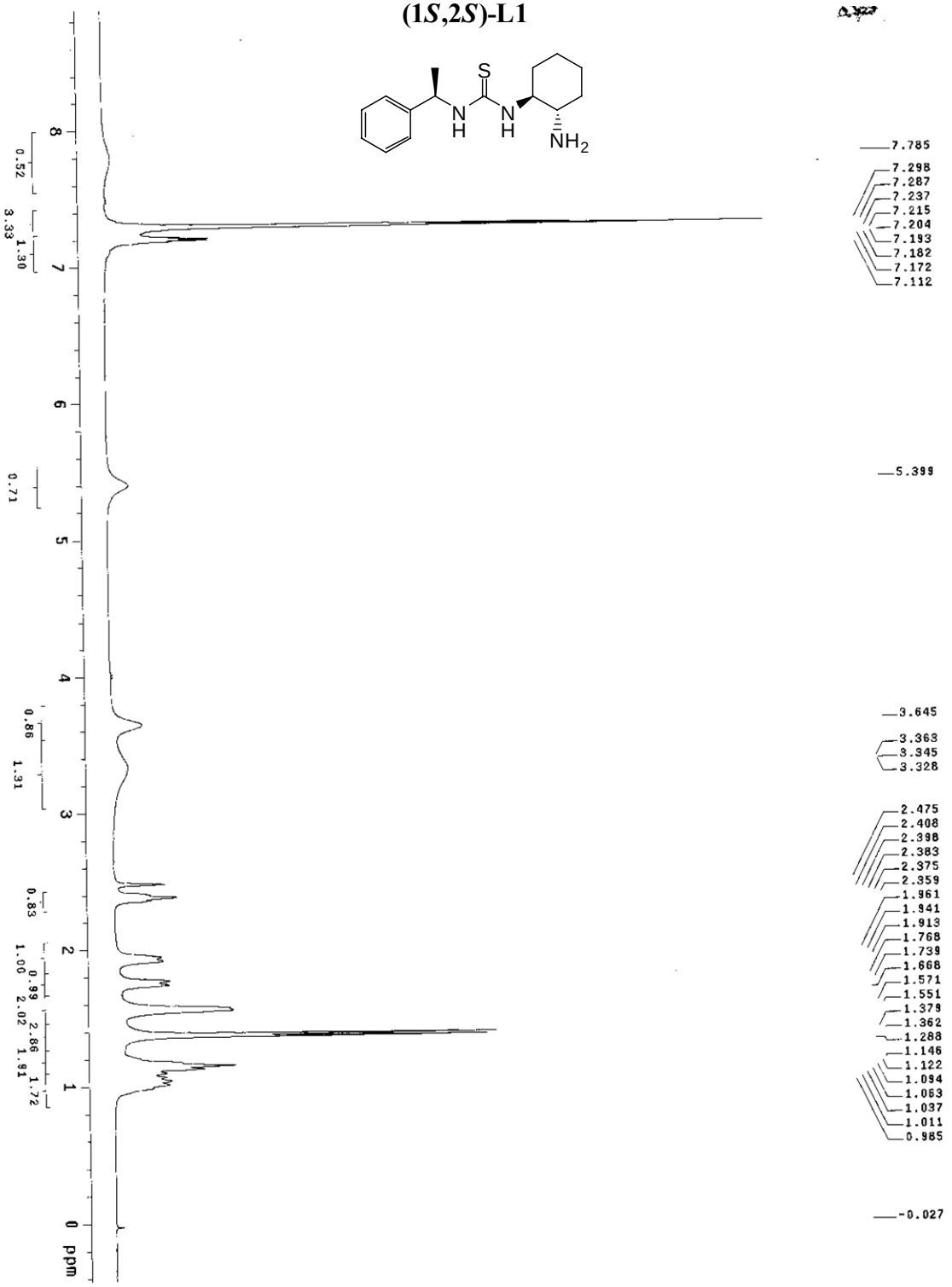
(1*R*,2*R*)-L1


7.788
7.312
7.221
5.426
3.664
3.306
2.499
2.424
2.402
1.986
1.961
1.935
1.790
1.762
1.724
1.578
1.469
1.403
1.386
1.171
1.148
1.118
1.089
1.060
1.033
1.008
0.853
0.839
-0.002
0.79
3.43
1.95
1.00
1.15
1.10
1.21
1.34
2.37
3.19
4.47
ppm

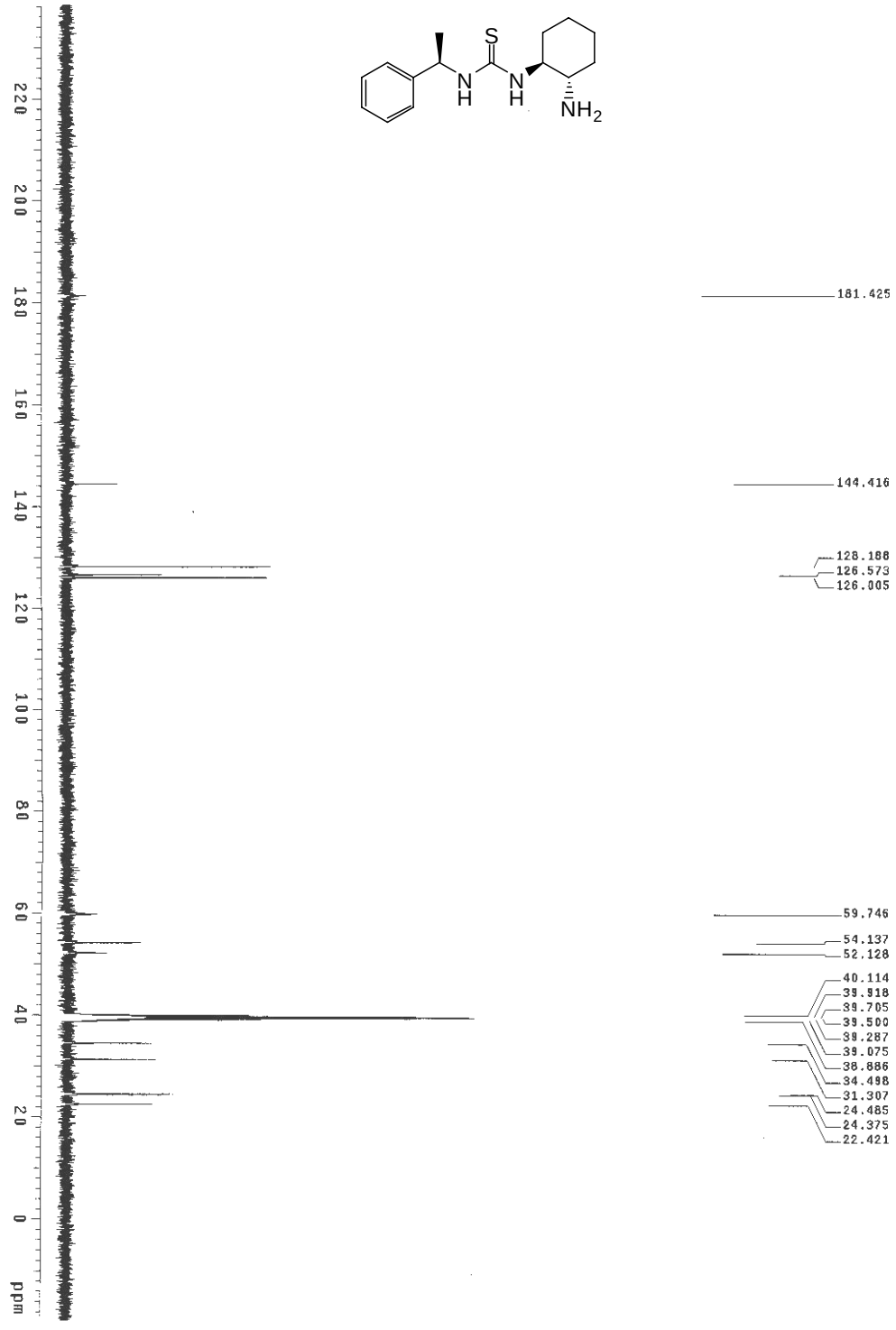
(1*R*,2*R*)-L1


144.508
128.309
126.691
126.104
59.606
54.165
52.250
40.126
39.920
39.706
39.500
39.294
39.080
38.874
34.441
31.412
24.583
24.453
22.561
ppm

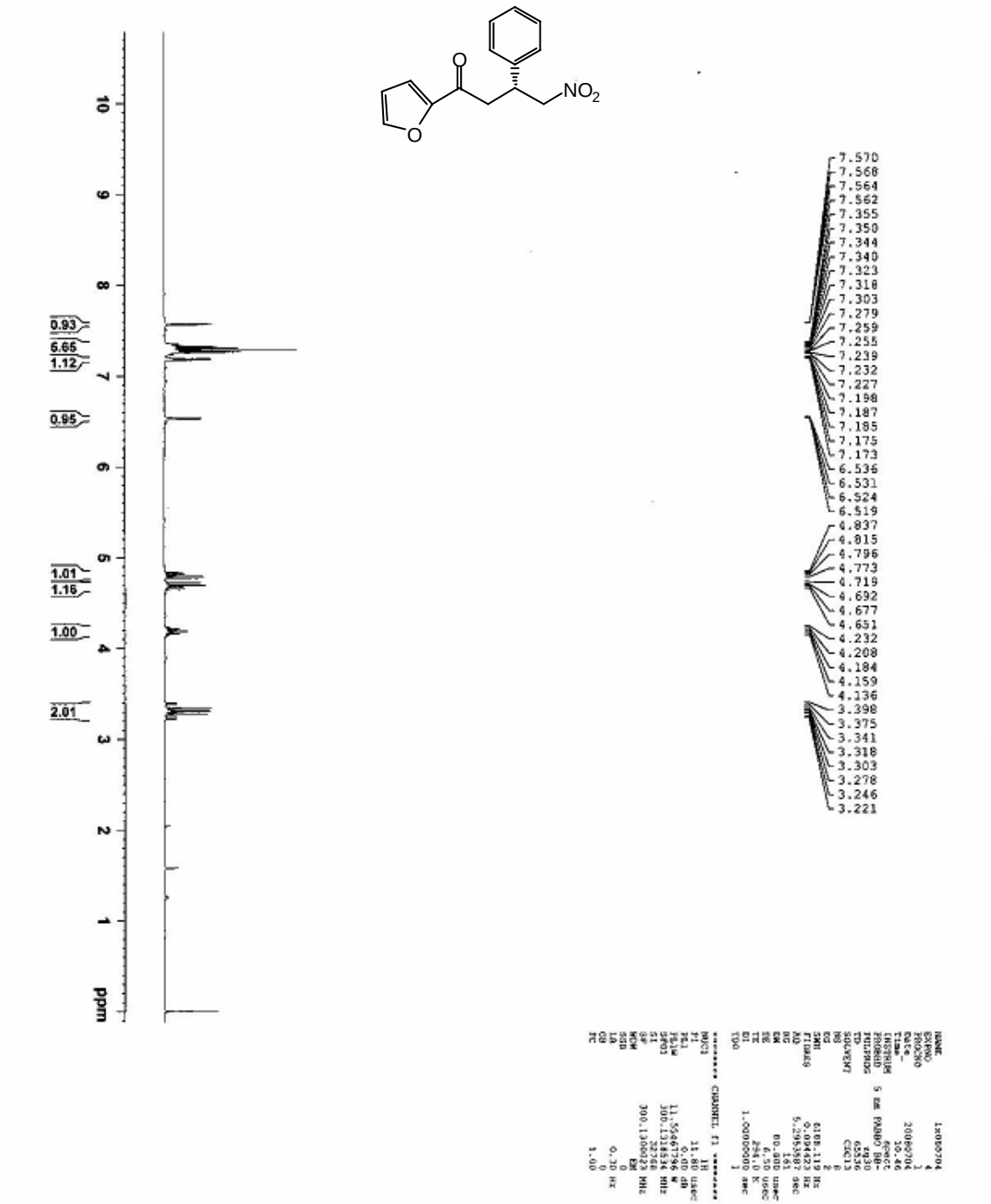
(1*S*,2*S*)-L1


S
N
H
N
H
NH2
7.785
7.298
7.287
7.237
7.215
7.204
7.193
7.182
7.172
7.112
5.399
3.645
3.363
3.345
3.328
2.475
2.408
2.398
2.383
2.375
2.359
1.961
1.941
1.913
1.768
1.739
1.668
1.571
1.551
1.379
1.362
1.288
1.146
1.122
1.094
1.063
1.037
1.011
0.985
-0.027
0.52
3.33
1.30
0.71
0.86
1.31
0.83
1.00
0.99
2.02
2.66
1.91
1.72
ppm

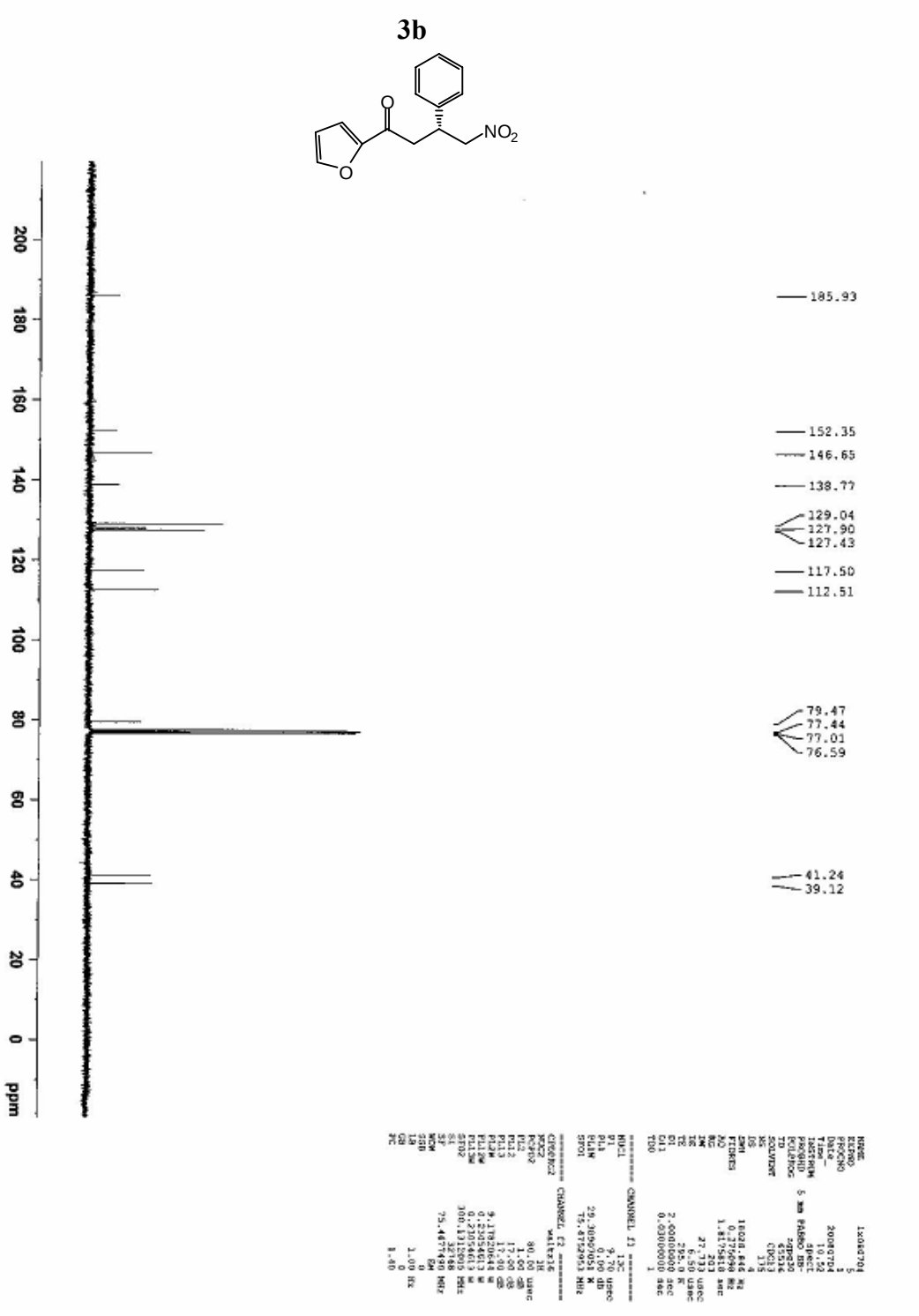
(1*S*,2*S*)-L1


S
N
H
N
H
NH2
181.425
144.416
128.188
126.573
126.005
59.746
54.137
52.128
40.114
39.918
39.705
39.500
39.287
39.075
38.886
34.498
31.307
24.485
24.375
22.421
220
200
180
160
140
120
100
80
60
40
20
0
ppm

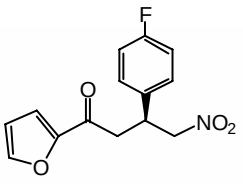
3b

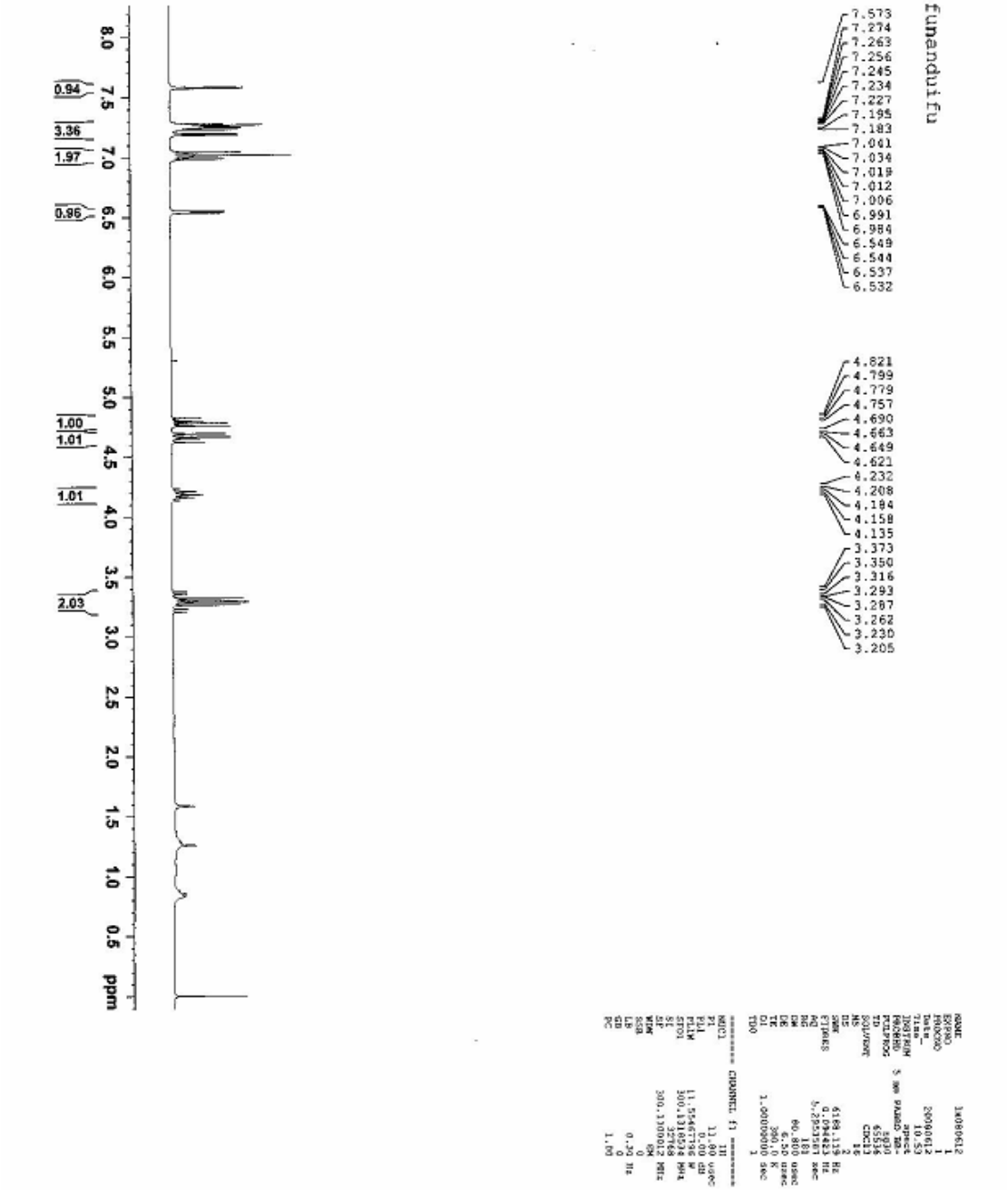

O
NO2
O
0.93
6.65
1.12
0.95
1.01
1.16
1.00
2.01
ppm
7.570
7.568
7.564
7.562
7.355
7.350
7.344
7.340
7.323
7.318
7.303
7.279
7.259
7.255
7.239
7.232
7.227
7.198
7.187
7.185
7.175
7.173
6.536
6.531
6.524
6.519
4.837
4.815
4.796
4.773
4.719
4.692
4.677
4.651
4.232
4.208
4.184
4.159
4.136
3.398
3.375
3.341
3.318
3.303
3.278
3.246
3.221

3b

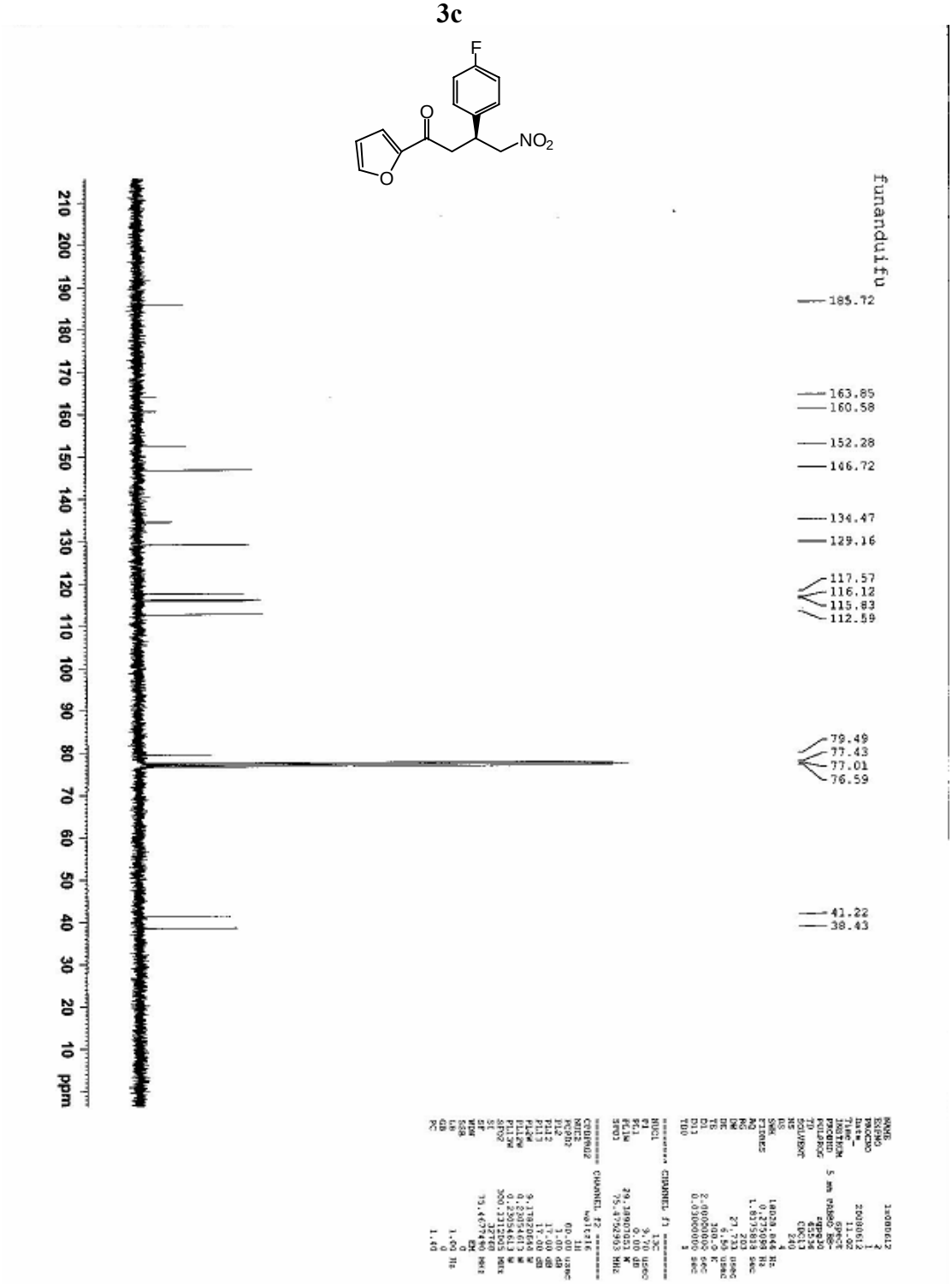

O
NO2
O
200
180
160
140
120
100
80
60
40
20
0
ppm
185.93
152.35
146.65
138.77
129.04
127.90
127.43
117.50
112.51
79.47
77.44
77.01
76.59
41.24
39.12

3c

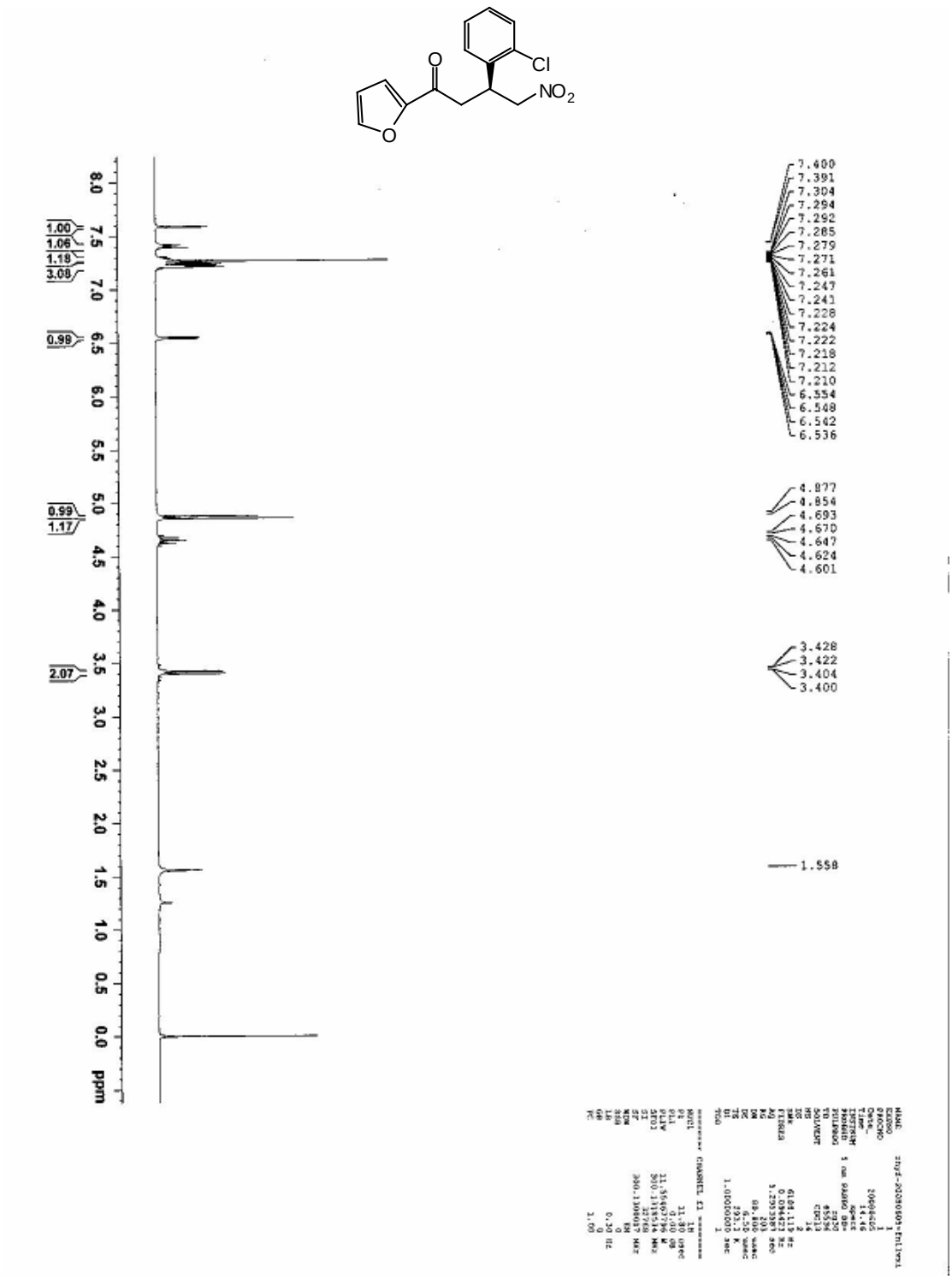

F
O
NO2
O


funandufu
7.573
7.274
7.263
7.256
7.245
7.234
7.227
7.195
7.183
7.041
7.034
7.018
7.012
7.006
6.991
6.984
6.549
6.544
6.537
6.532
4.821
4.799
4.779
4.757
4.690
4.663
4.649
4.621
4.232
4.208
4.184
4.158
4.135
3.373
3.350
3.316
3.293
3.287
3.262
3.230
3.205
0.94
3.36
1.97
0.96
1.00
1.01
1.01
2.03
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
ppm

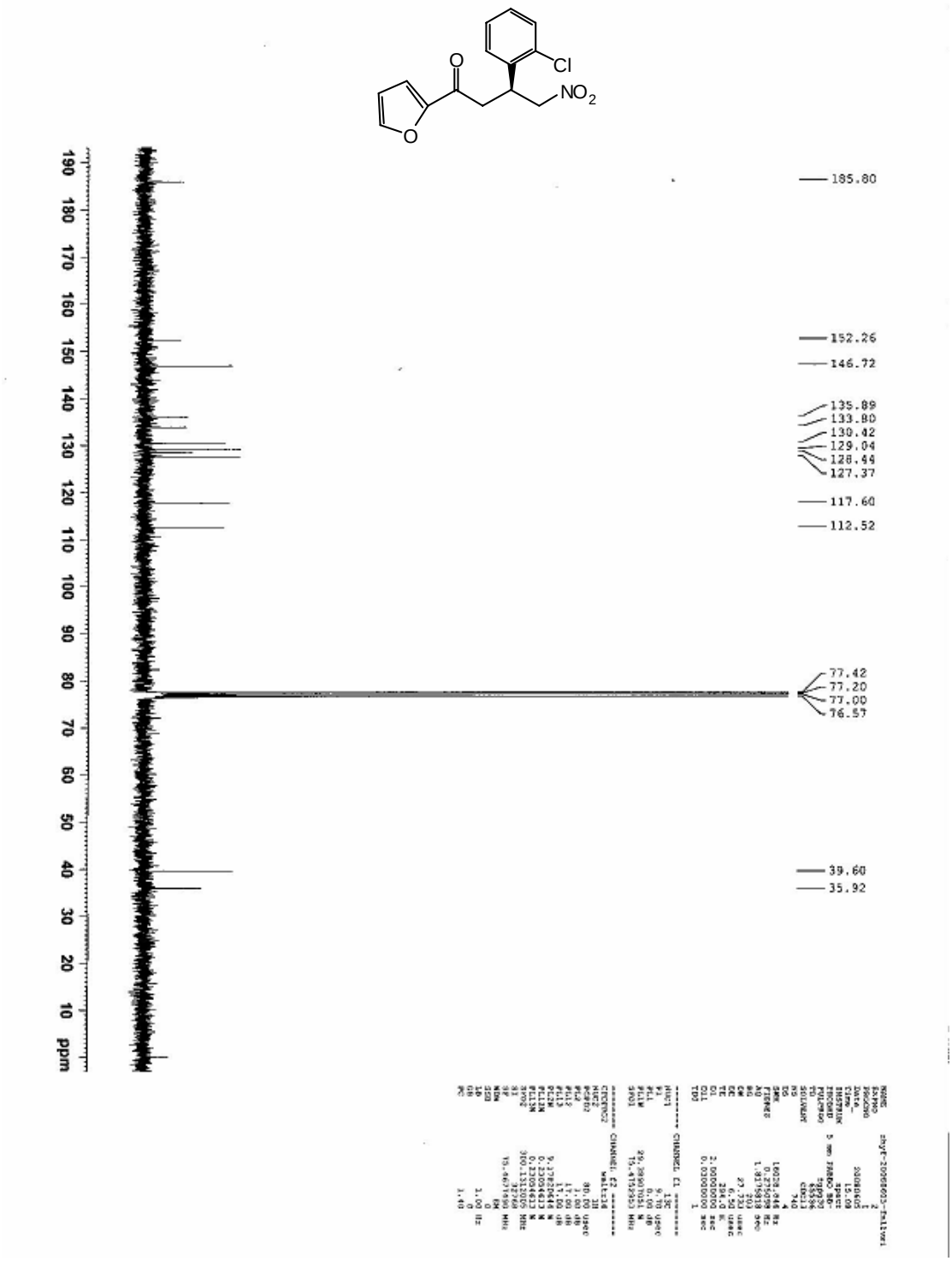
3c


F
O
O
NO2
funarduifu
185.72
163.85
160.58
152.28
146.72
134.47
129.16
117.57
116.12
115.83
112.59
79.49
77.43
77.01
76.59
41.22
38.43
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

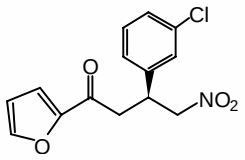
3d

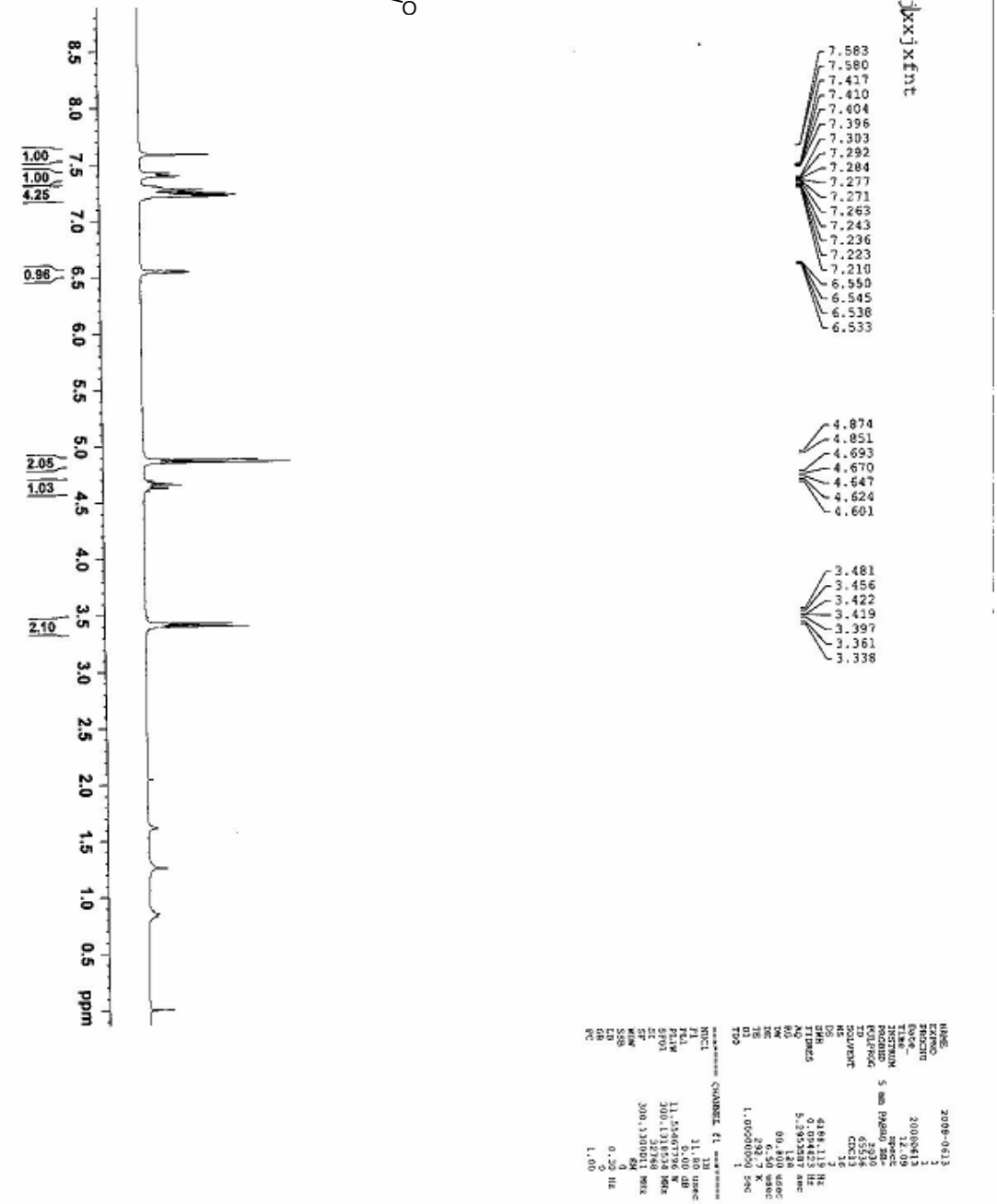

O
Cl
NO2
O
1.00
1.06
1.18
3.08
0.99
0.99
1.17
2.07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
7.400
7.391
7.304
7.294
7.292
7.285
7.279
7.271
7.261
7.247
7.241
7.228
7.224
7.222
7.218
7.212
7.210
6.554
6.548
6.542
6.536
4.877
4.854
4.693
4.670
4.647
4.624
4.601
3.428
3.422
3.404
3.400
1.558

3d

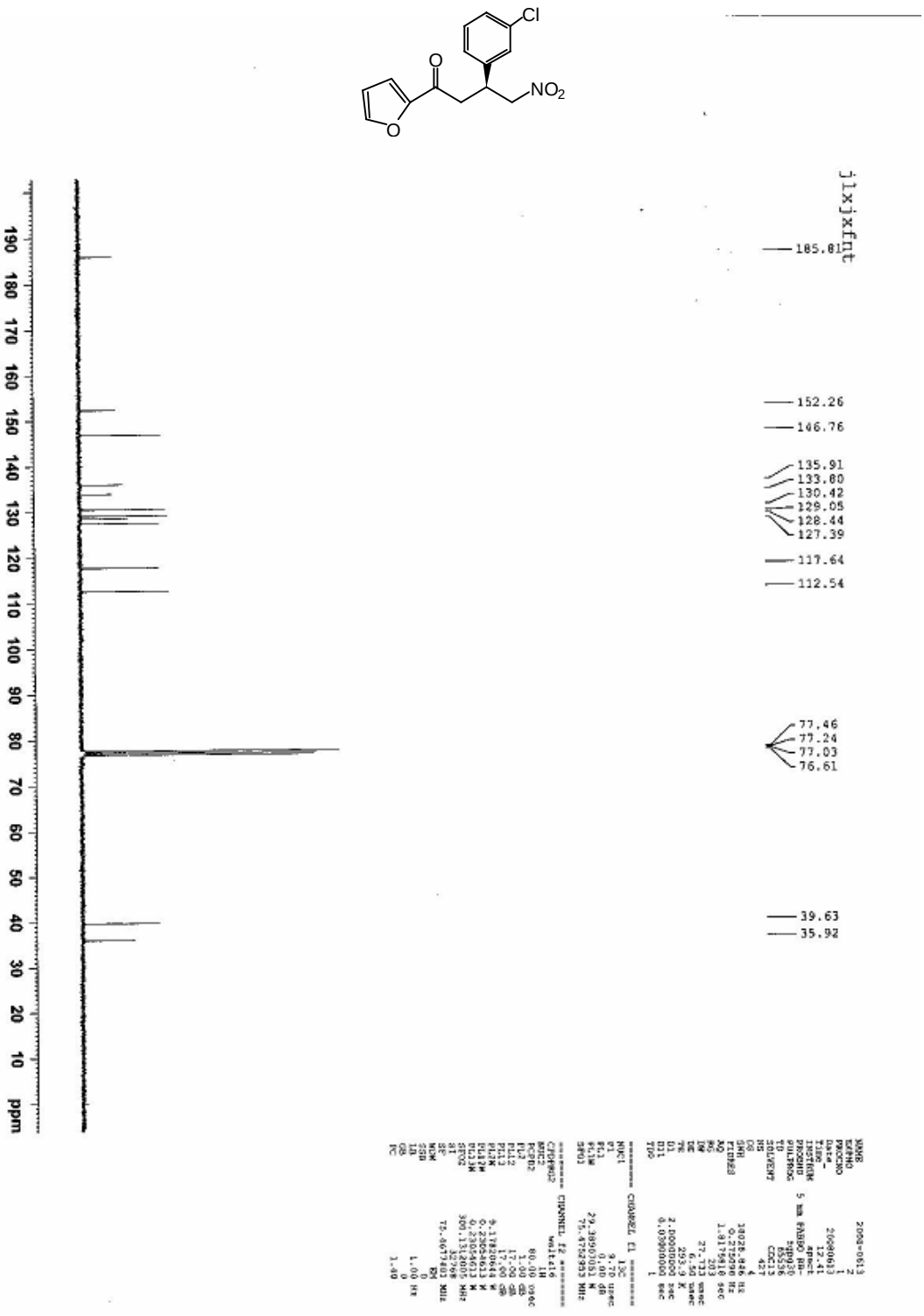

O
Cl
NO2
O
185.80
152.26
146.72
135.89
133.80
130.42
129.04
128.44
127.37
117.60
112.52
77.42
77.20
77.00
76.57
39.60
35.92
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3e

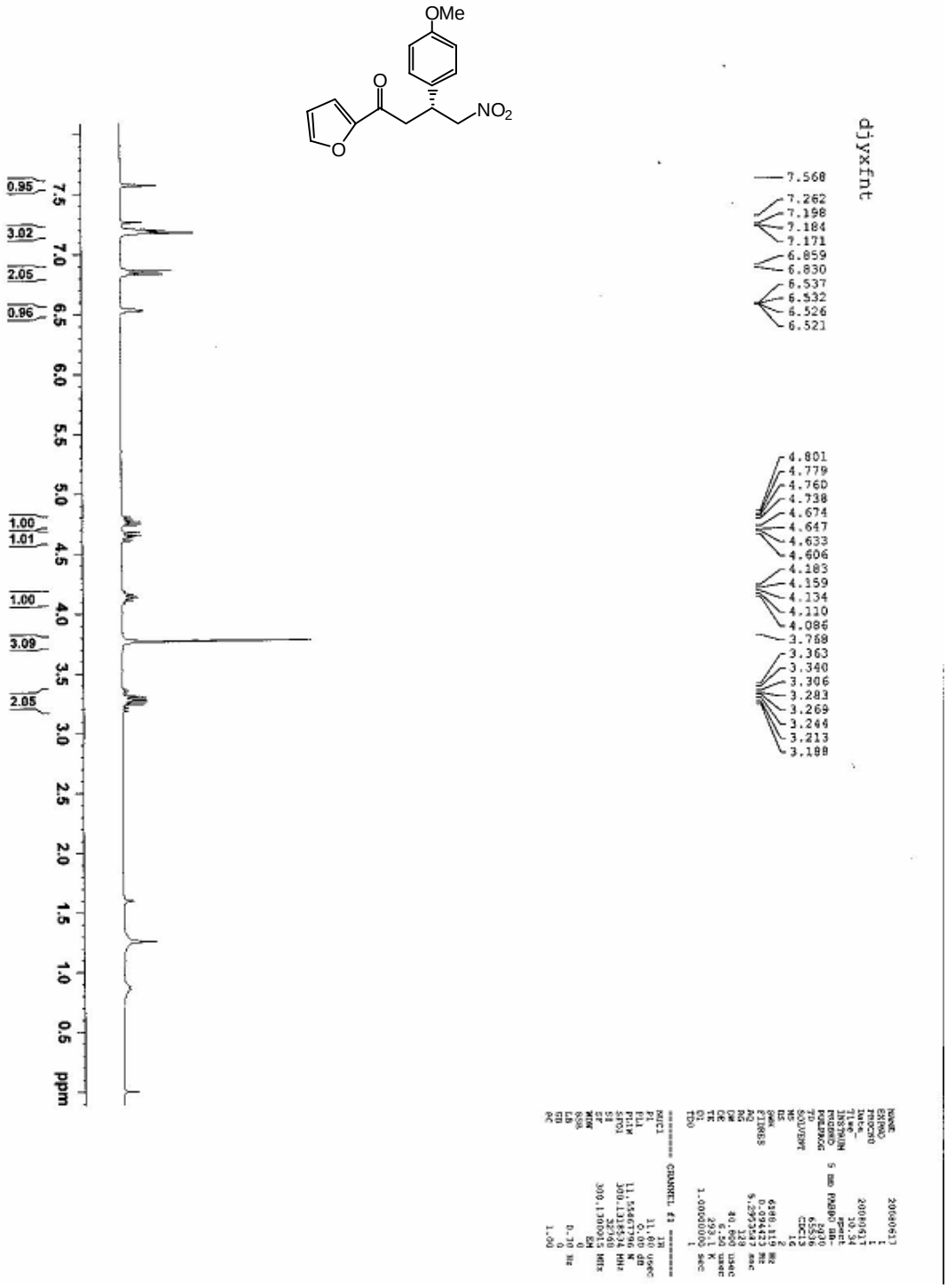

Cl
O
O
NO2


jkxjxfnt
7.583
7.580
7.417
7.410
7.404
7.396
7.303
7.292
7.284
7.277
7.271
7.263
7.243
7.236
7.223
7.210
6.550
6.545
6.538
6.533
4.874
4.851
4.693
4.670
4.647
4.624
4.601
3.481
3.456
3.422
3.419
3.397
3.361
3.338
1.00
1.00
4.25
0.96
2.05
1.03
2.10
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
ppm

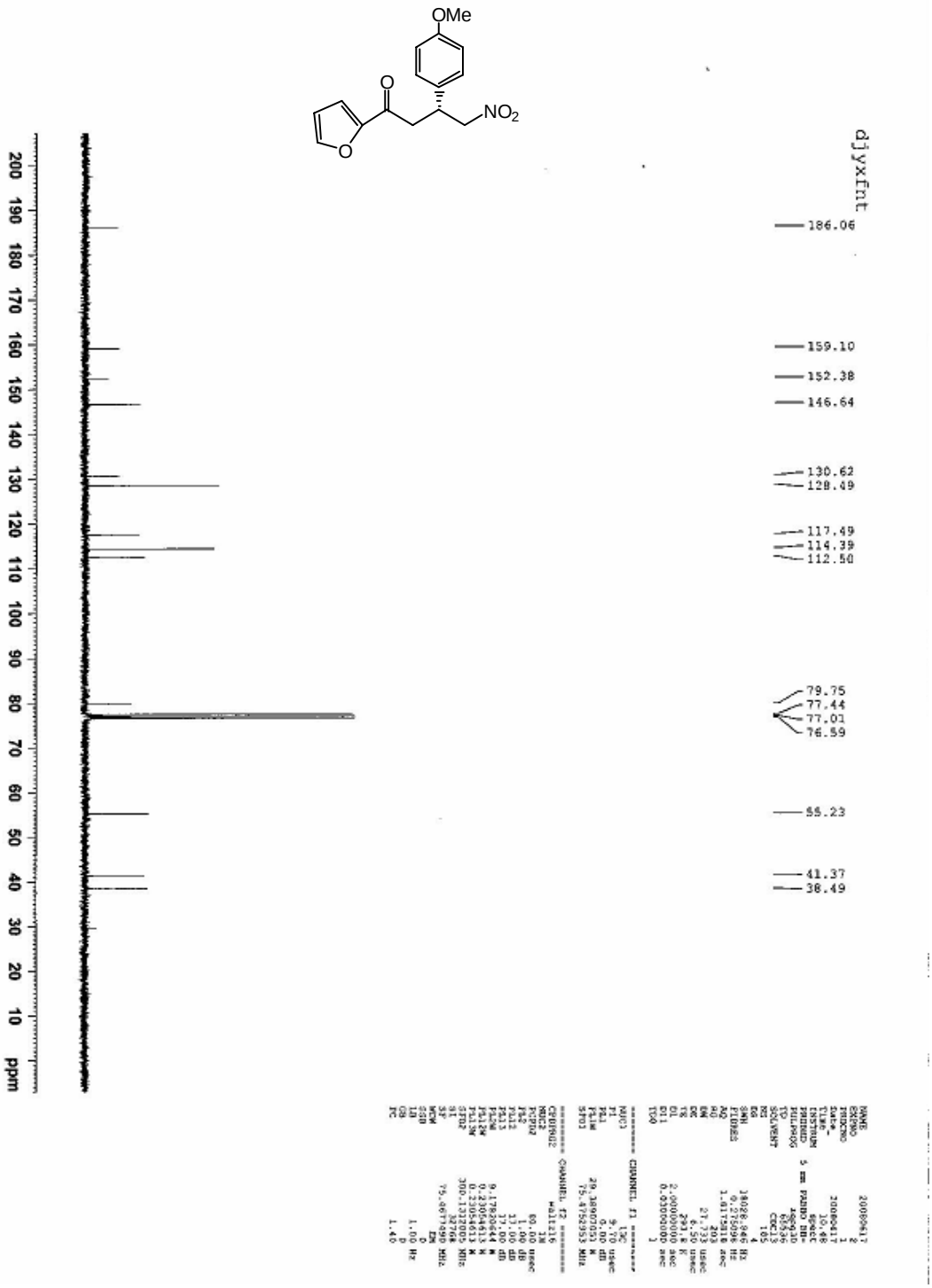
3e


Cl
O
NO2
O
jlxjxfnt
185.81
152.26
146.76
135.91
133.80
130.42
129.05
128.44
127.39
117.64
112.54
77.46
77.24
77.03
76.61
39.63
35.92
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

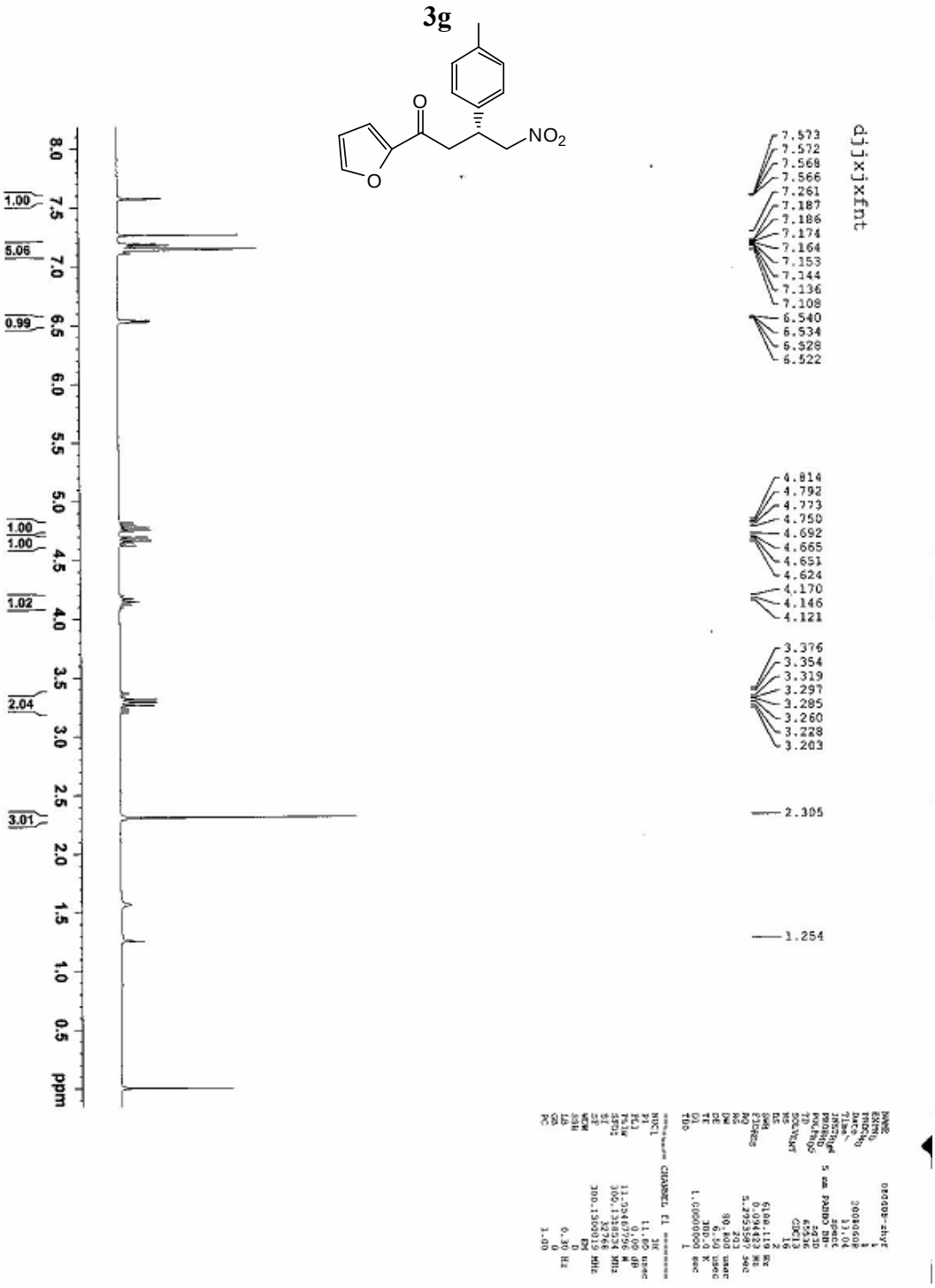
3f


OMe
O
O
NO2
djyxfnt
7.560
7.262
7.198
7.184
7.171
6.859
6.830
6.537
6.532
6.526
6.521
4.801
4.779
4.760
4.738
4.674
4.647
4.633
4.606
4.183
4.159
4.134
4.110
4.086
3.768
3.363
3.340
3.306
3.283
3.269
3.244
3.213
3.188
0.95
3.02
2.05
0.96
1.00
1.01
1.00
3.09
2.05
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
ppm

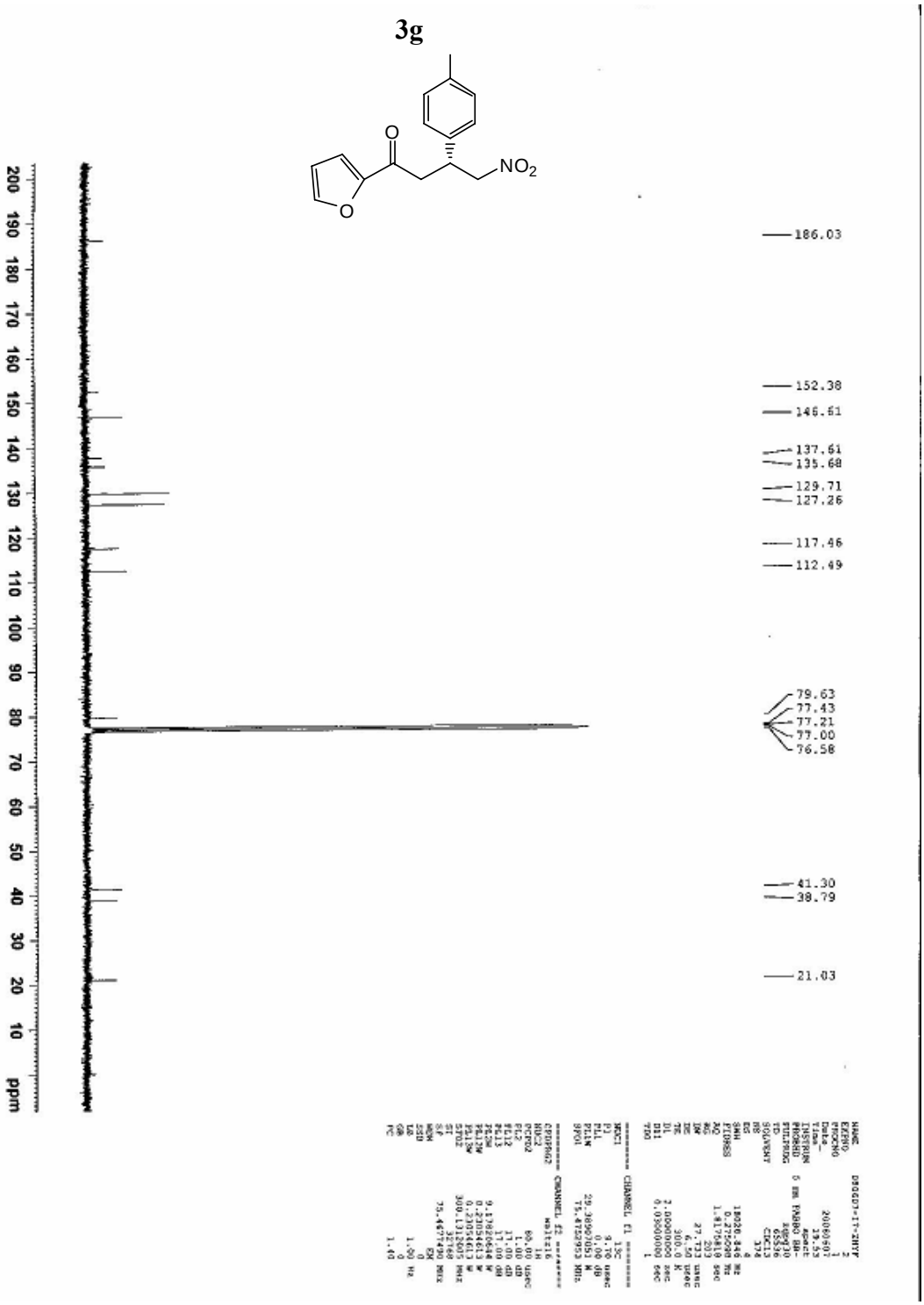
3f


OMe
O
NO2
O
djyxfnt
186.06
159.10
152.38
146.64
130.62
128.49
117.49
114.39
112.50
79.75
77.44
77.01
76.59
55.23
41.37
38.49
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

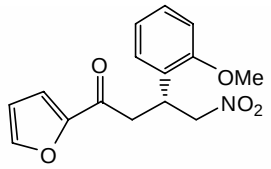
3g

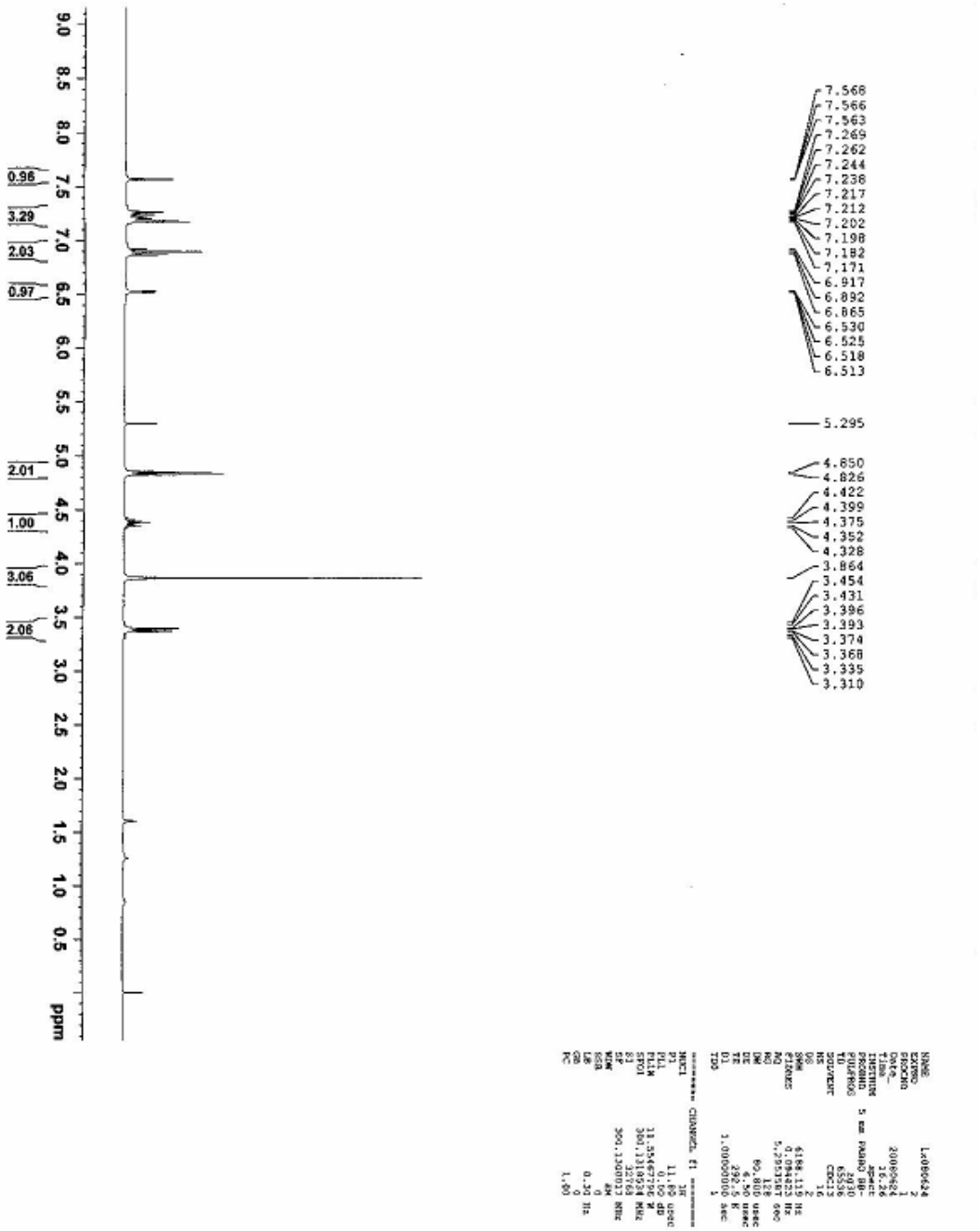

O
O
NO2
djjxjxfnt
7.573
7.572
7.568
7.566
7.261
7.187
7.186
7.174
7.164
7.153
7.144
7.136
7.108
6.540
6.534
6.528
6.522
4.814
4.792
4.773
4.750
4.692
4.665
4.651
4.624
4.170
4.146
4.121
3.376
3.354
3.319
3.297
3.285
3.260
3.228
3.203
2.305
1.254
1.00
5.06
0.99
1.00
1.00
1.02
2.04
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

3g

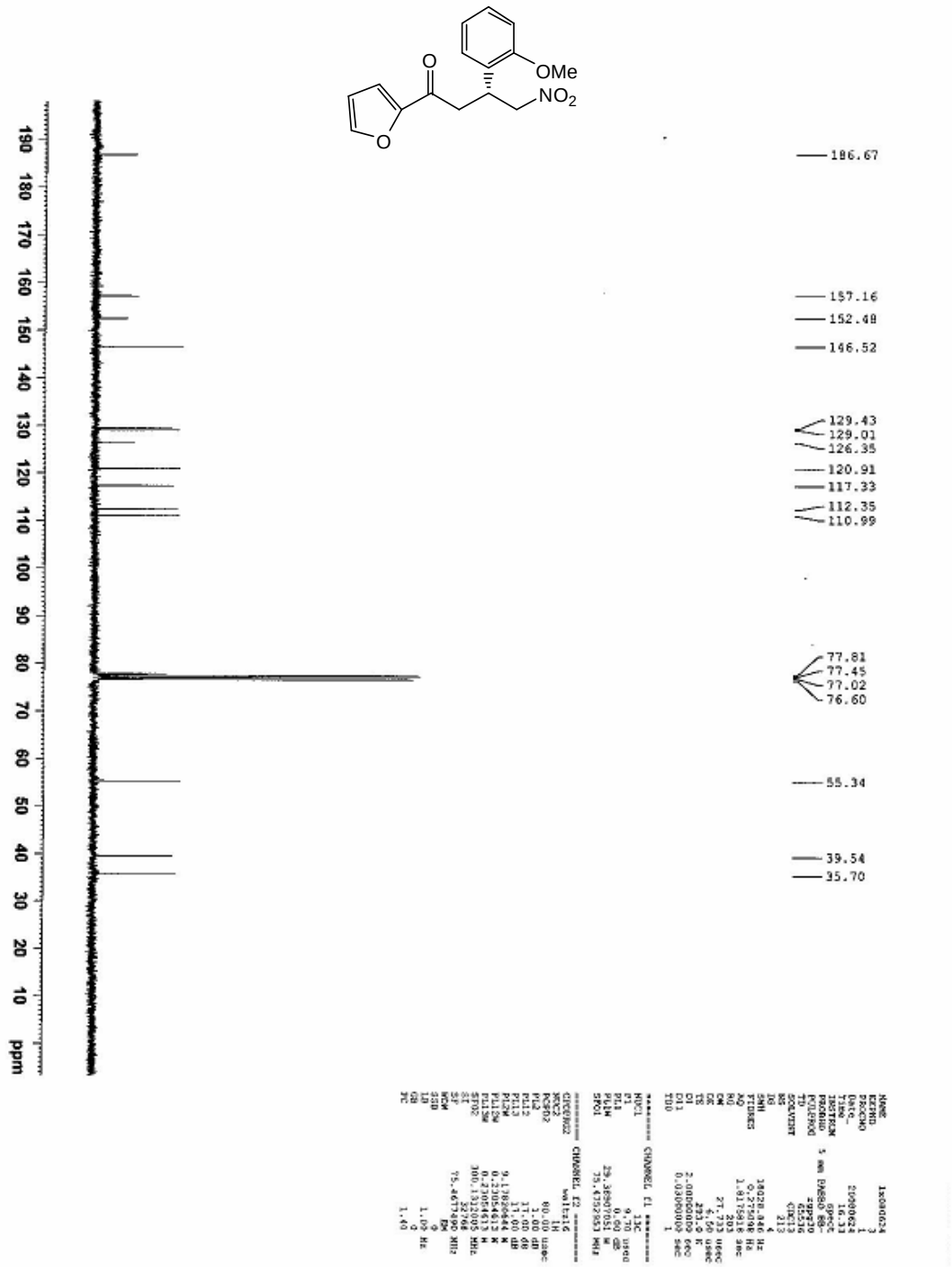

O
O
NO2
186.03
152.38
146.61
137.61
135.68
129.71
127.26
117.46
112.49
79.63
77.43
77.21
77.00
76.58
41.30
38.79
21.03
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3h

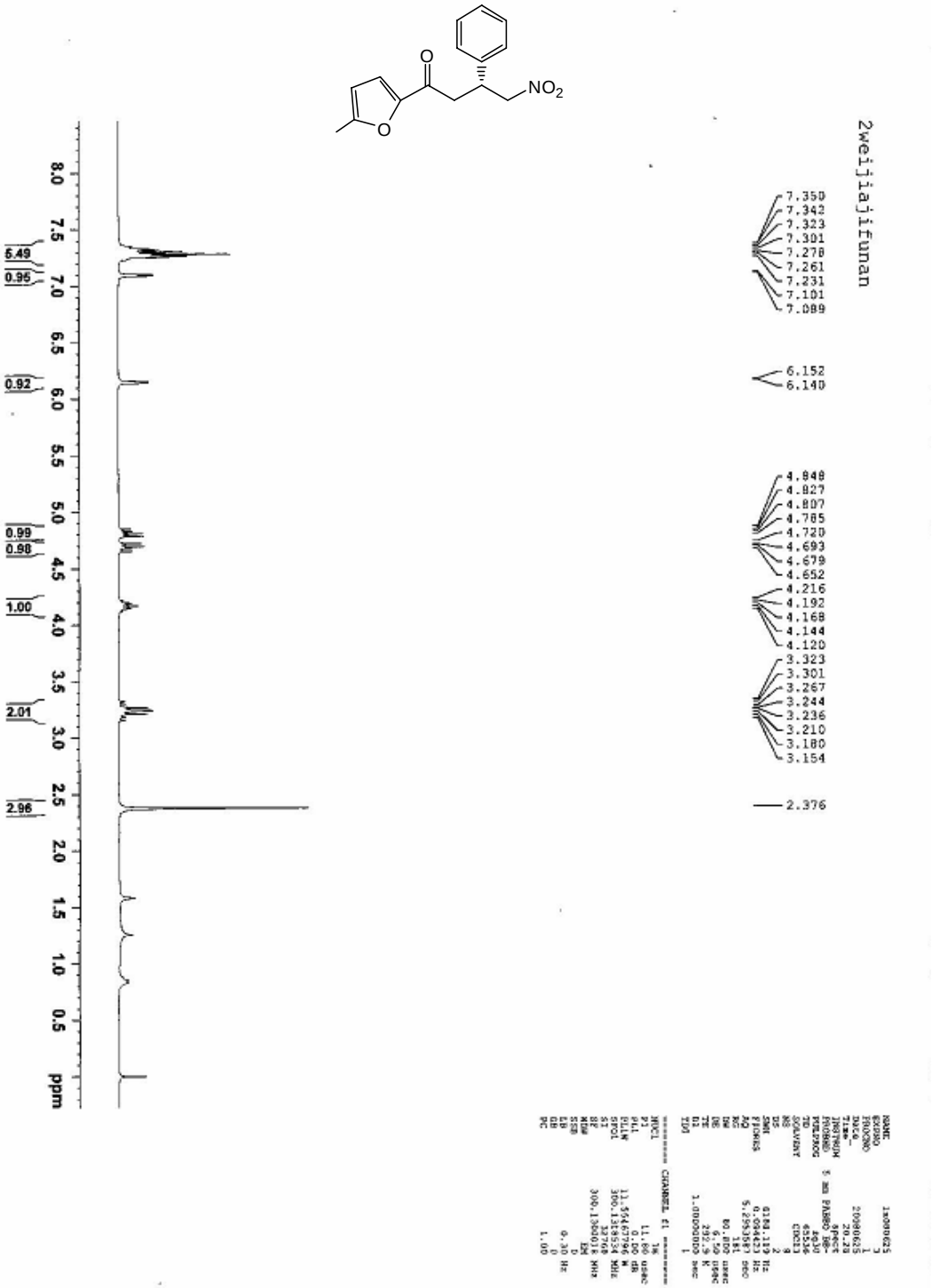

O
OMe
NO2
O


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 ppm
0.96
3.29
2.03
0.97
2.01
1.00
3.06
2.06
7.568
7.566
7.563
7.269
7.262
7.244
7.238
7.217
7.212
7.202
7.198
7.182
7.171
6.917
6.892
6.865
6.530
6.525
6.518
6.513
5.295
4.850
4.826
4.422
4.399
4.375
4.352
4.328
3.864
3.454
3.431
3.396
3.393
3.374
3.368
3.335
3.310

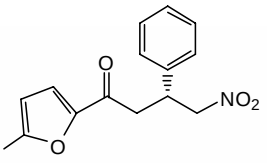
3h

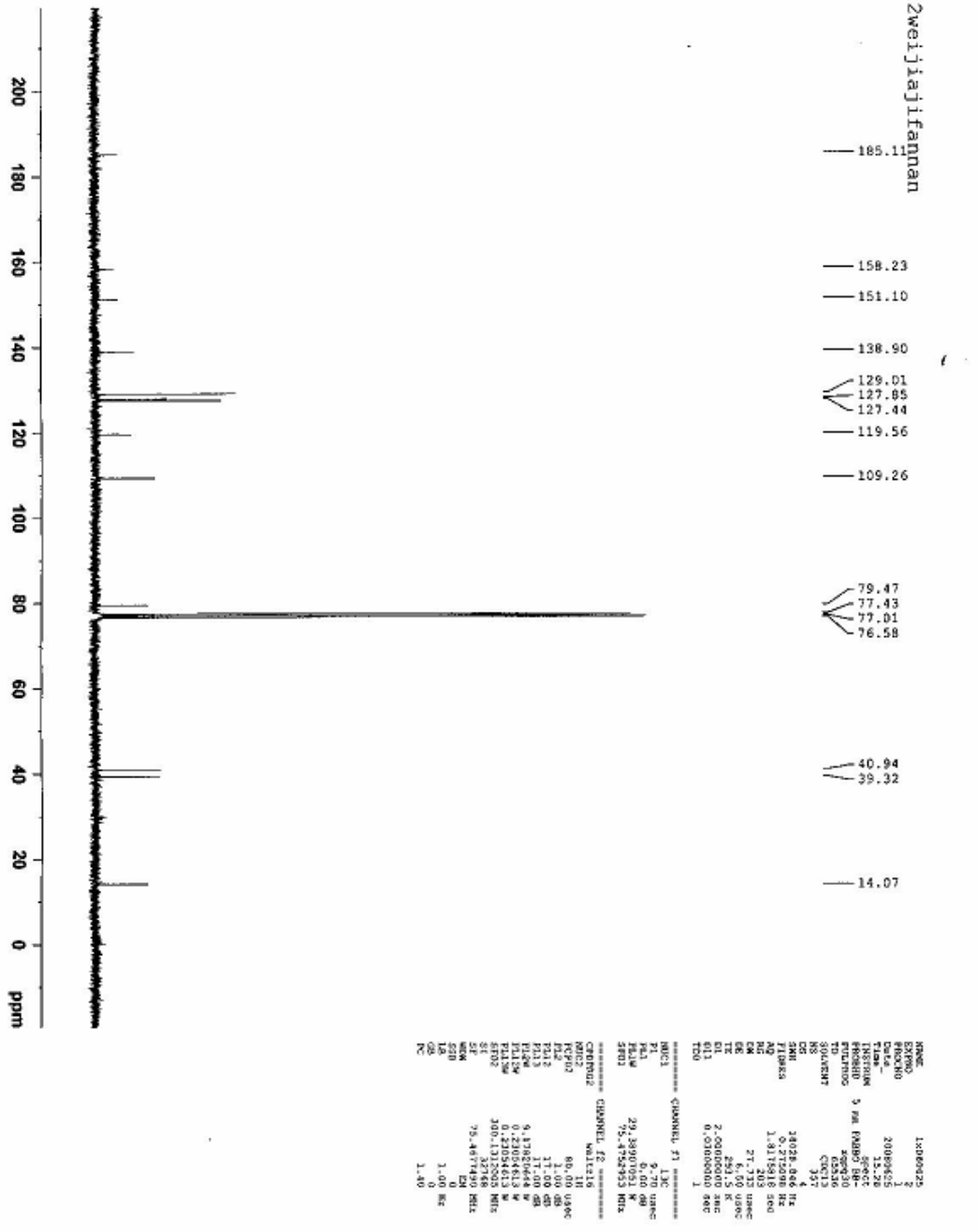

O
OMe
NO2
O
186.67
157.16
152.48
146.52
129.43
129.01
126.35
120.91
117.33
112.35
110.99
77.81
77.45
77.02
76.60
55.34
39.54
35.70
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3i

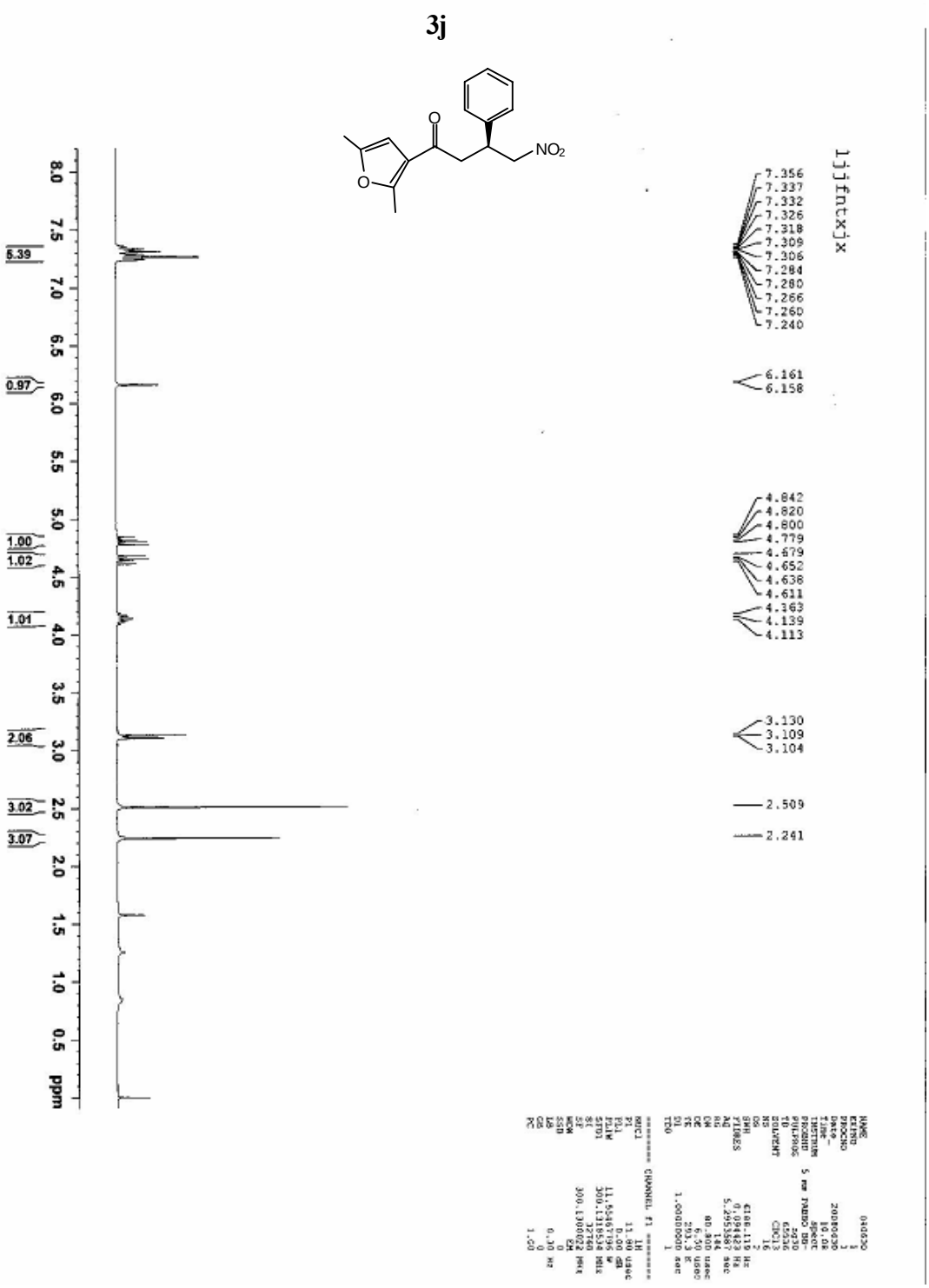

O
NO2
O
2weijiajifunan
7.350
7.342
7.323
7.301
7.278
7.261
7.231
7.101
7.089
6.152
6.140
4.848
4.827
4.807
4.785
4.720
4.693
4.679
4.652
4.216
4.192
4.168
4.144
4.120
3.323
3.301
3.267
3.244
3.236
3.210
3.180
3.154
2.376
6.49
0.95
0.92
0.99
0.98
1.00
2.01
2.96
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

3i

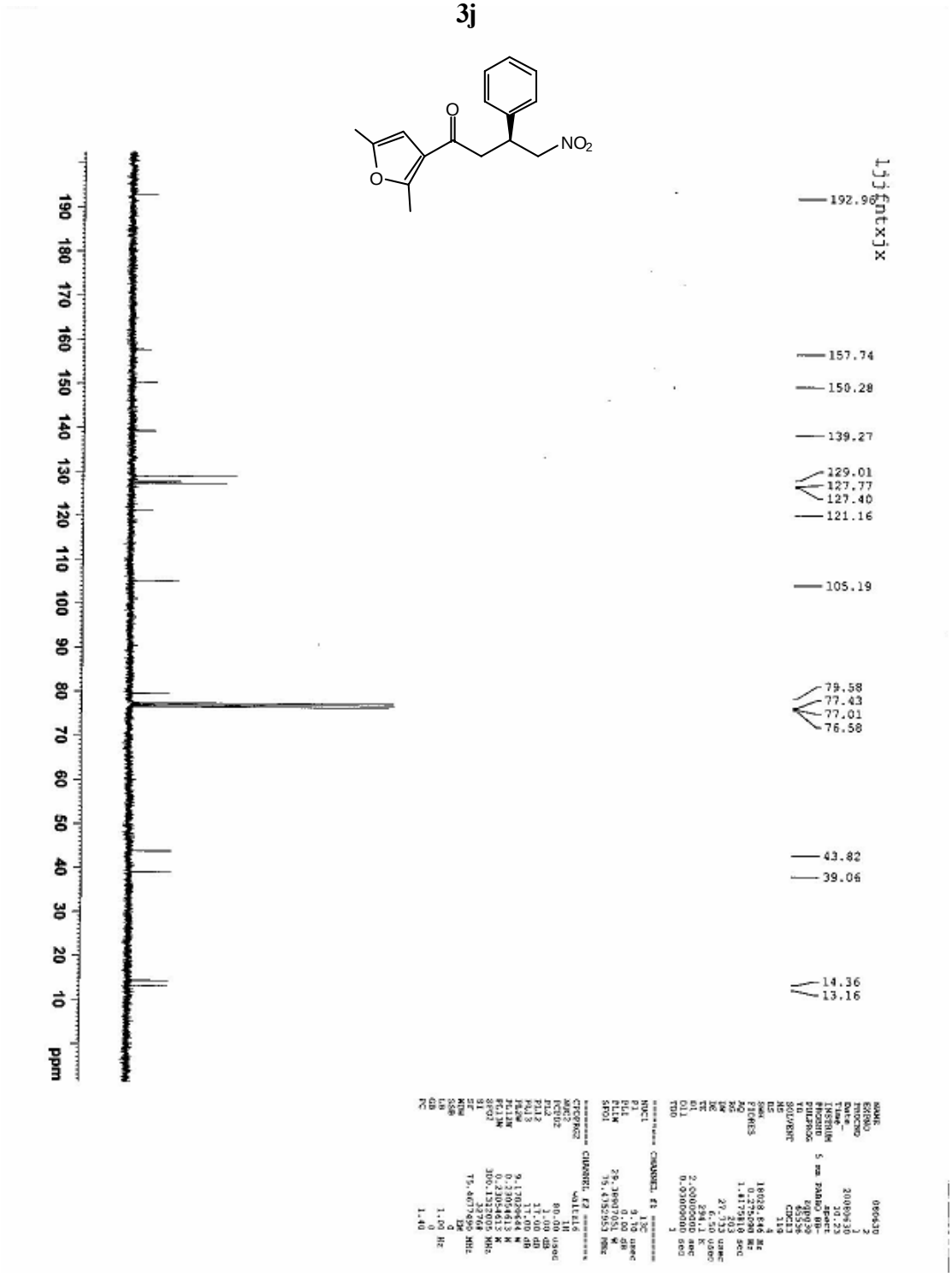

O
NO2
O


2weijiajifanman
185.11
158.23
151.10
138.90
129.01
127.85
127.44
119.56
109.26
79.47
77.43
77.01
76.58
40.94
39.32
14.07
200
180
160
140
120
100
80
60
40
20
0
ppm

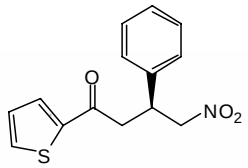
3j

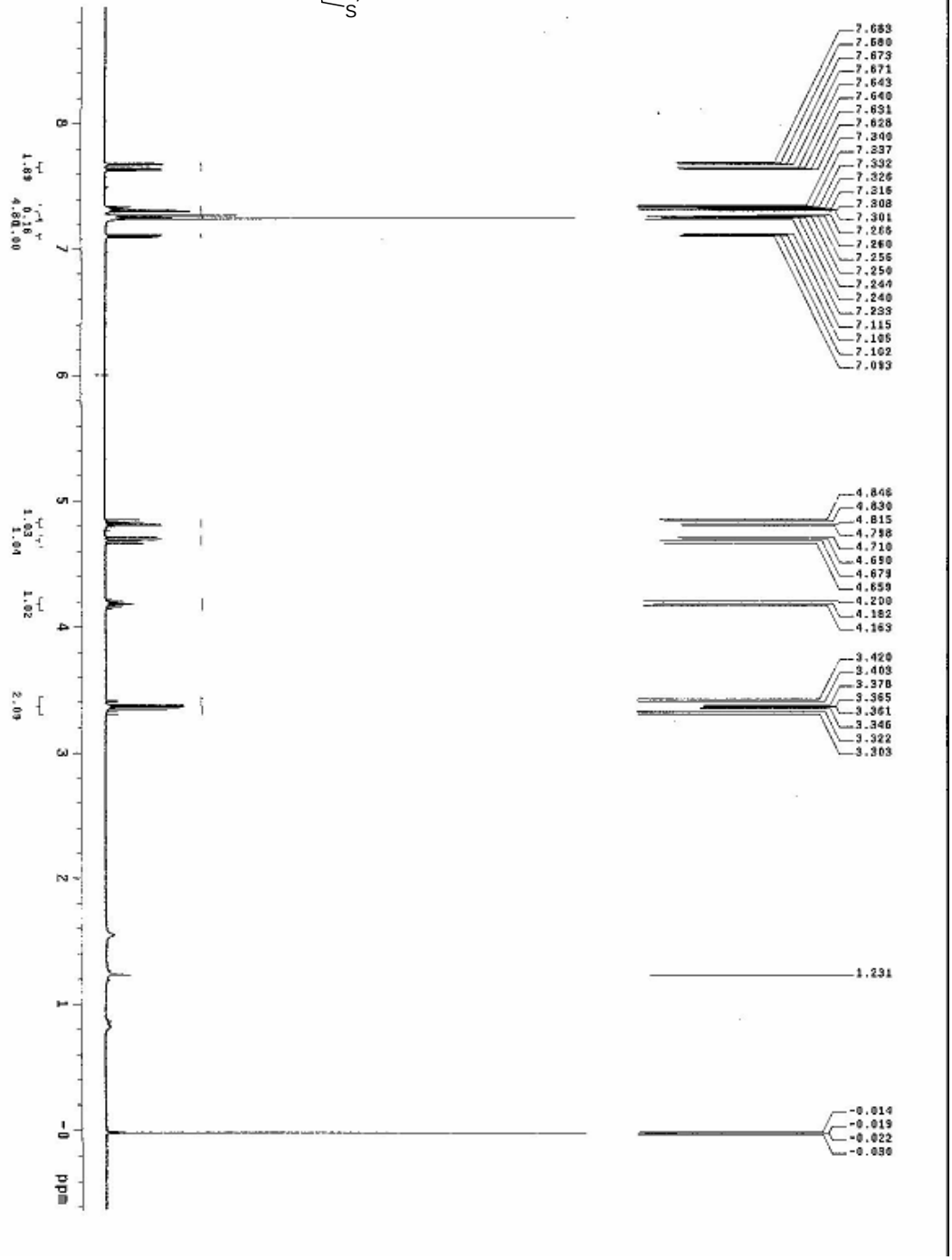

O
O
NO2
ljjfntxjx
7.356
7.337
7.332
7.326
7.318
7.309
7.306
7.284
7.280
7.266
7.260
7.240
6.161
6.158
4.842
4.820
4.800
4.779
4.679
4.652
4.638
4.611
4.363
4.139
4.113
3.130
3.109
3.104
2.509
2.241
5.39
0.97
1.00
1.02
1.01
2.06
3.02
3.07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

3j

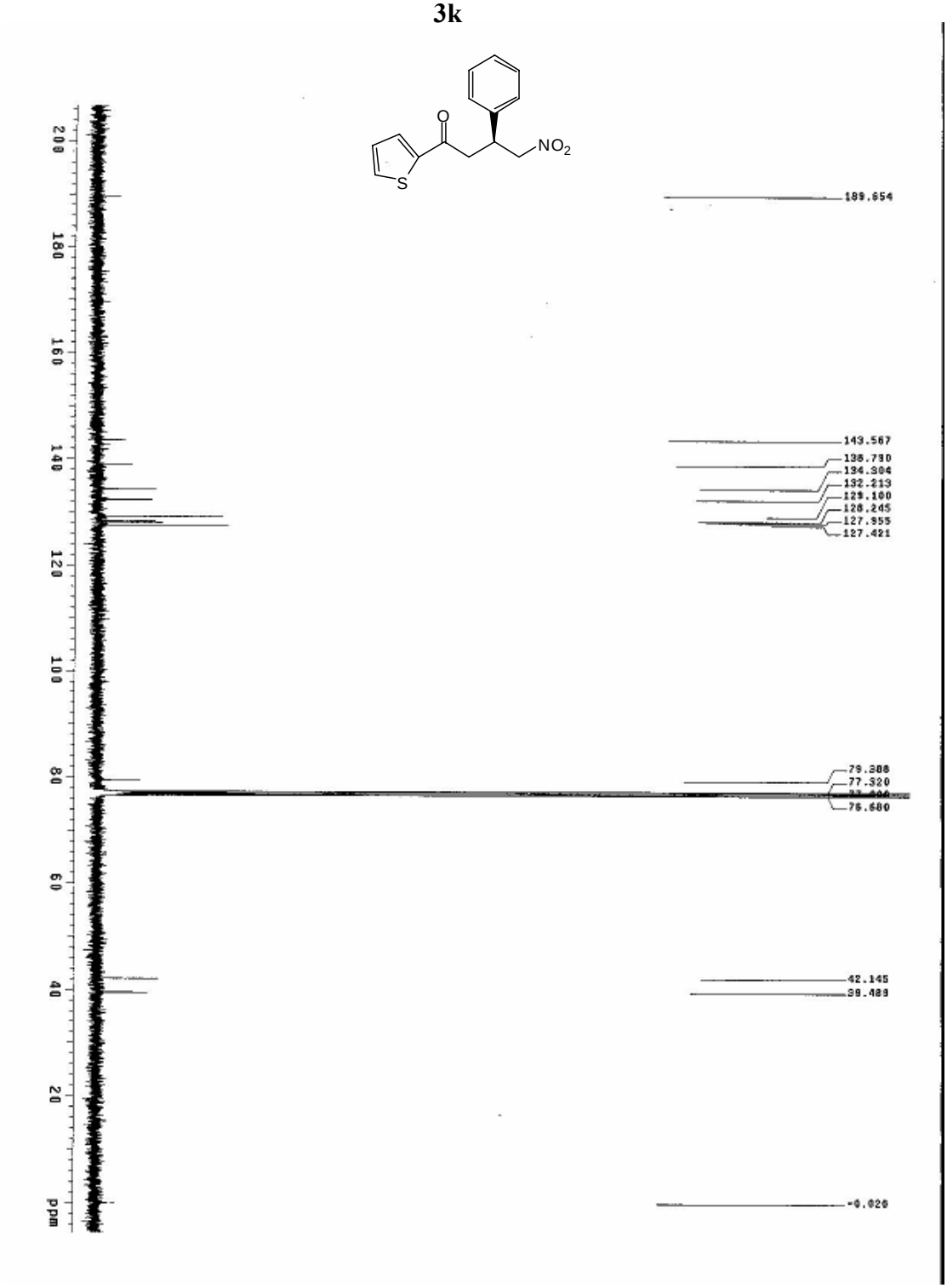

O
O
NO2
ljjfntxjx
192.96
157.74
150.28
139.27
129.01
127.77
127.40
121.16
105.19
79.58
77.43
77.01
76.58
43.82
39.06
14.36
13.16
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3k

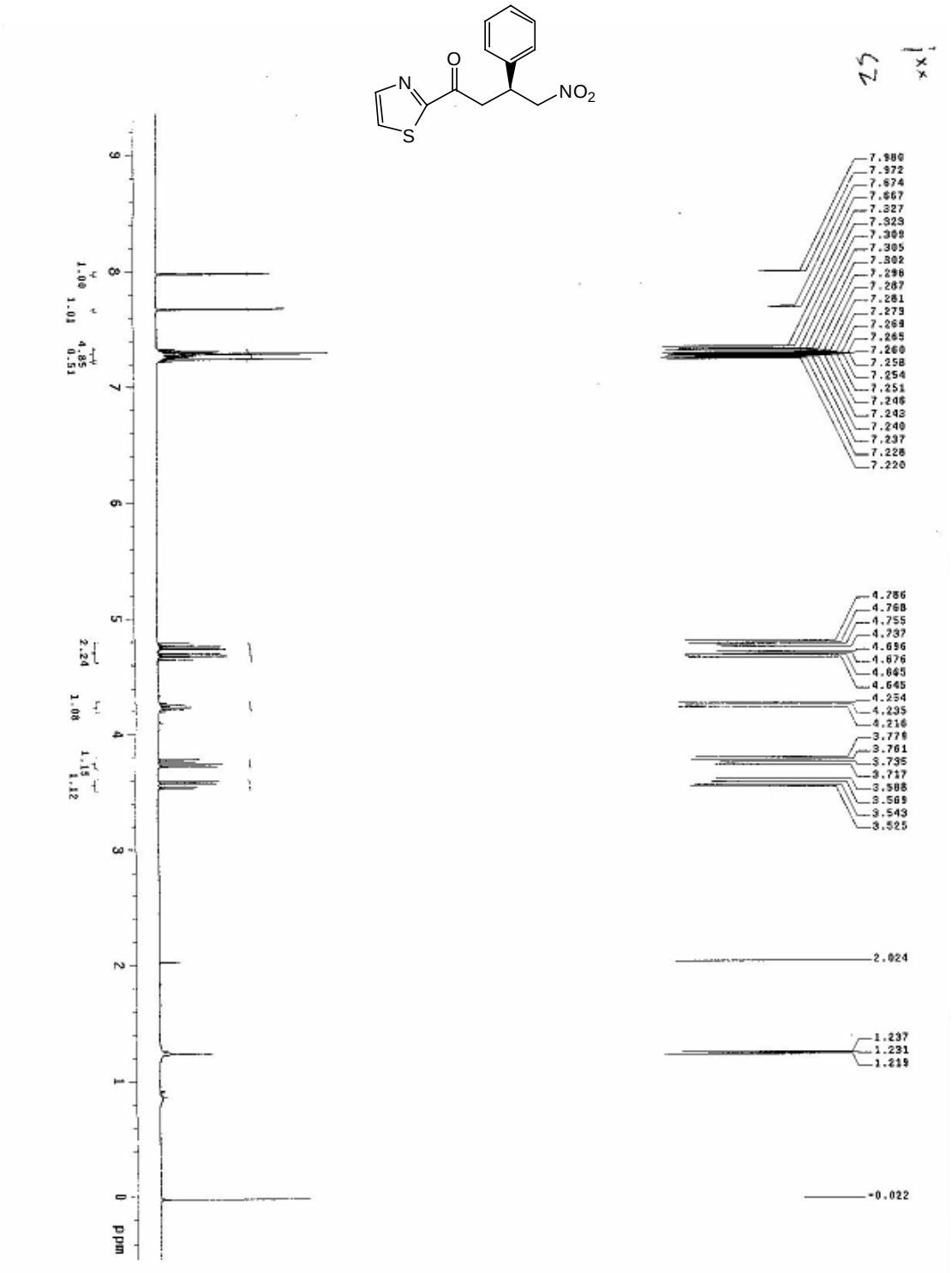

O
NO2
S


7.683
7.680
7.673
7.671
7.643
7.640
7.631
7.628
7.340
7.337
7.332
7.326
7.316
7.308
7.301
7.266
7.260
7.256
7.250
7.244
7.240
7.233
7.115
7.105
7.102
7.093
4.846
4.830
4.815
4.798
4.710
4.690
4.679
4.659
4.200
4.182
4.163
3.420
3.403
3.378
3.365
3.361
3.346
3.322
3.303
1.231
-0.014
-0.019
-0.022
-0.030
ppm

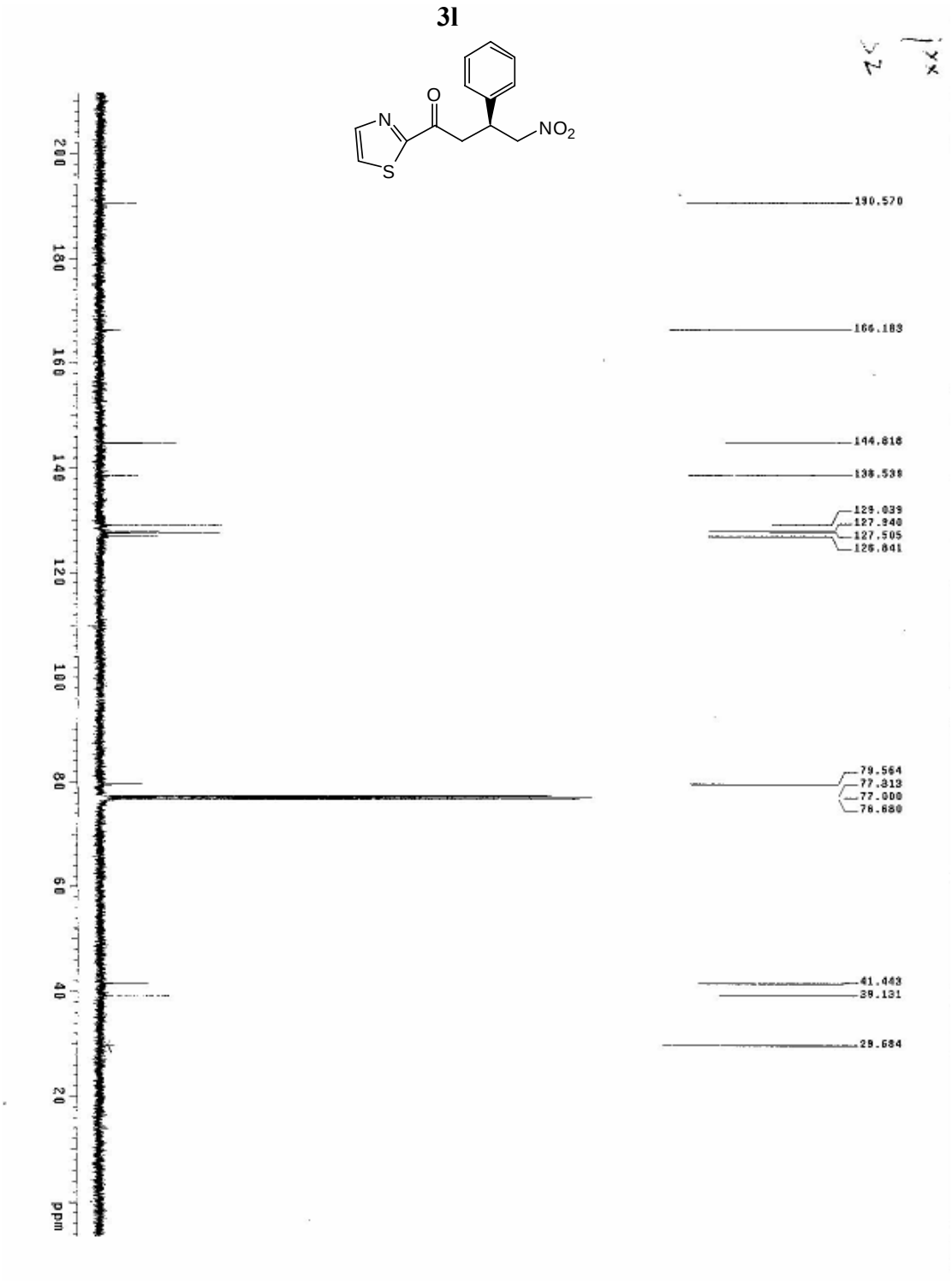
3k


O
NO2
S
189.654
143.567
138.730
134.304
132.213
129.100
128.245
127.955
127.421
79.388
77.320
76.680
42.145
38.488
-0.026
200
180
160
140
120
100
80
60
40
20
ppm

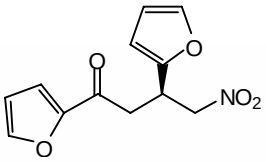
3l

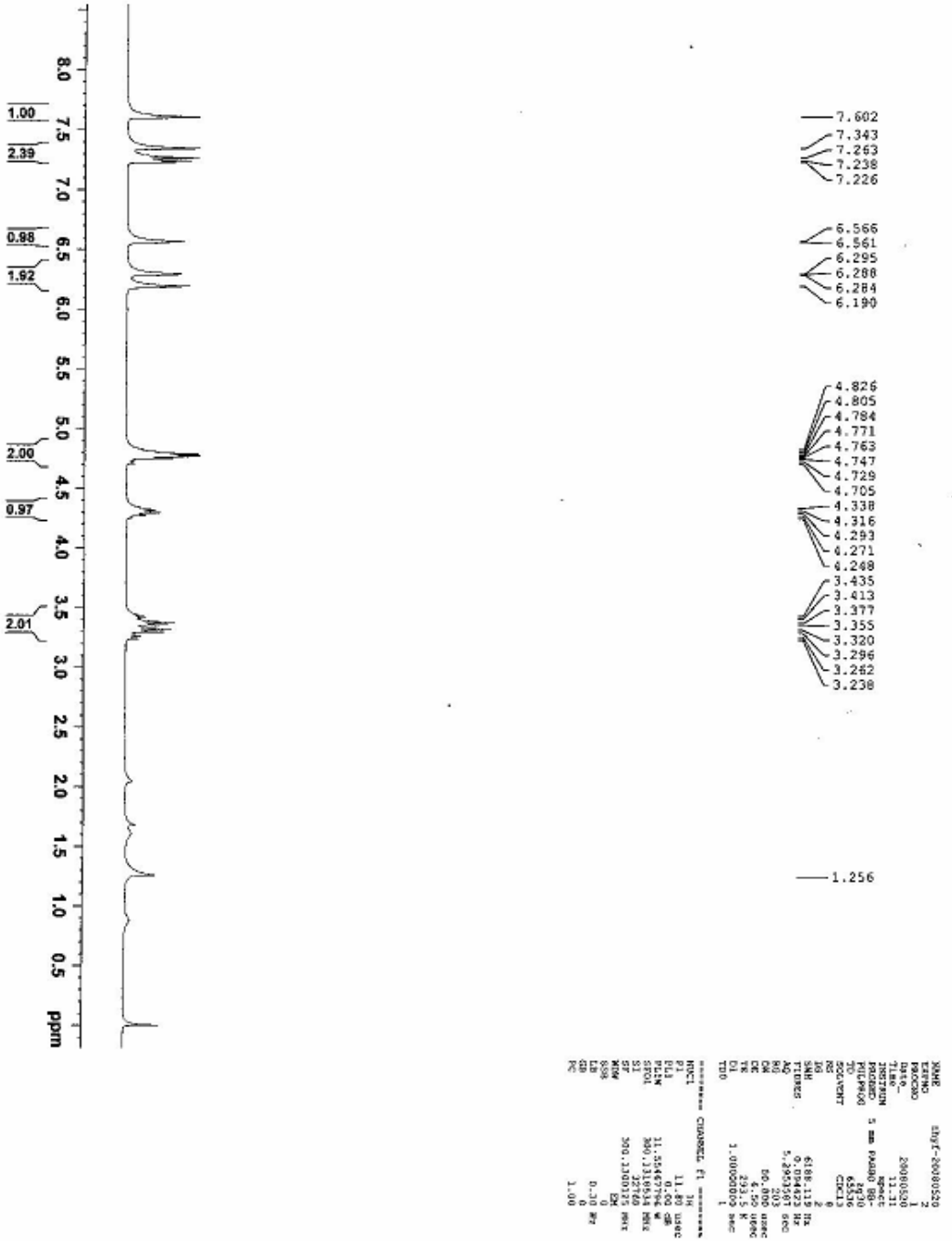

O
N
S
NO2
7.980
7.972
7.674
7.667
7.327
7.323
7.309
7.305
7.302
7.298
7.287
7.281
7.279
7.269
7.265
7.260
7.258
7.254
7.251
7.246
7.243
7.240
7.237
7.228
7.220
4.786
4.768
4.755
4.737
4.686
4.676
4.665
4.645
4.254
4.235
4.216
3.779
3.761
3.735
3.717
3.588
3.569
3.543
3.525
2.024
1.237
1.231
1.219
0.022
ppm

3l

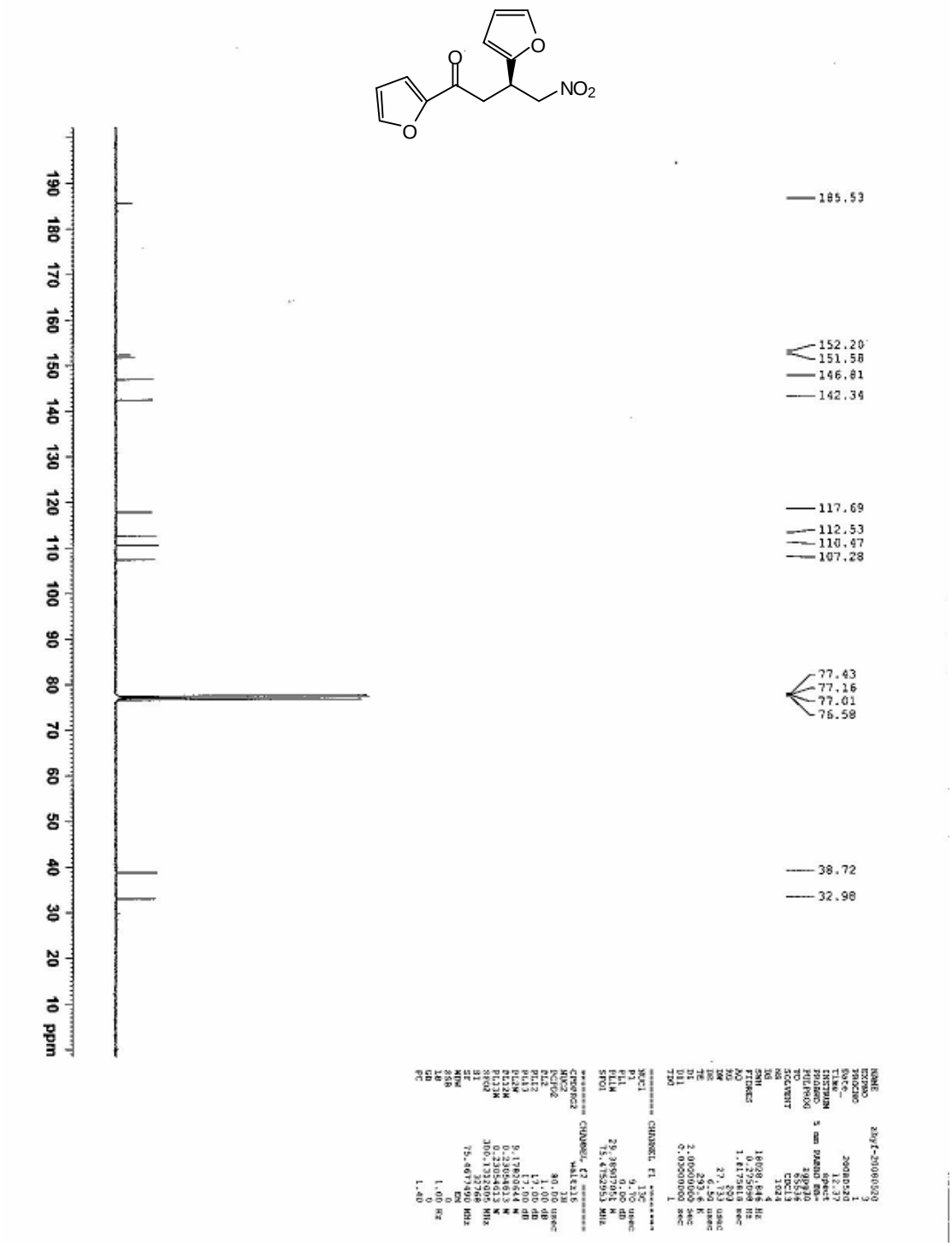

O
N
S
NO2
190.570
166.183
144.816
138.538
129.039
127.940
127.505
126.841
79.564
77.313
77.000
76.680
41.443
39.131
29.684
200
180
160
140
120
100
80
60
40
20
ppm

3m

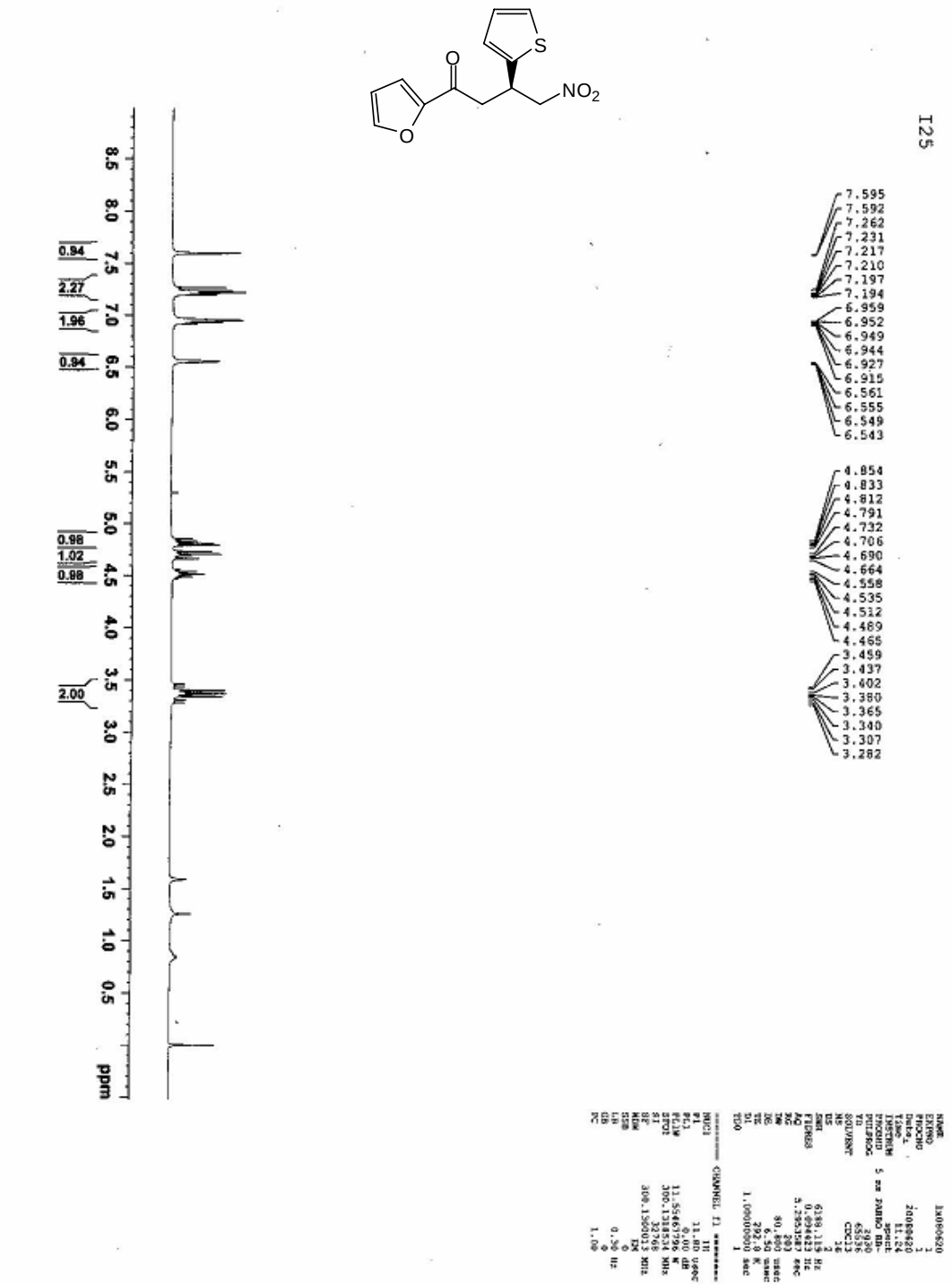

O
O
O
NO2


1.00
2.39
0.98
1.92
2.00
0.97
2.01
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
ppm
7.602
7.343
7.263
7.238
7.226
6.566
6.561
6.295
6.288
6.284
6.190
4.826
4.805
4.784
4.771
4.763
4.747
4.729
4.705
4.338
4.316
4.293
4.271
4.248
3.435
3.413
3.377
3.355
3.320
3.296
3.262
3.238
1.256

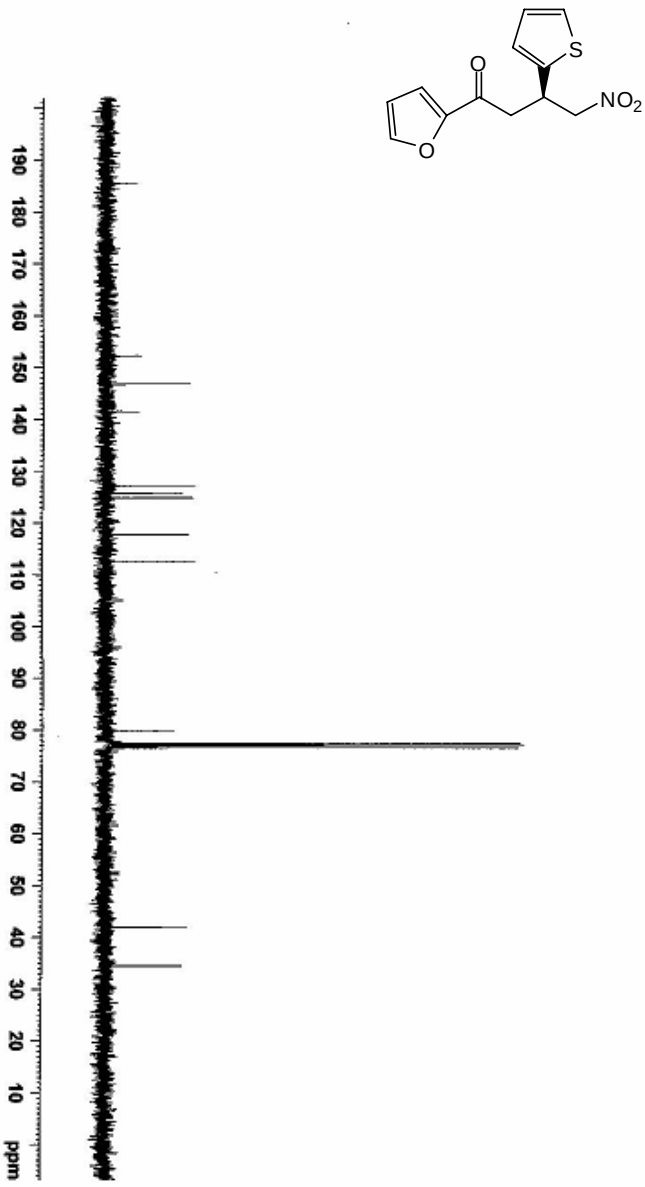
3m


O
O
O
NO2
185.53
152.20
151.58
146.81
142.34
117.69
112.53
110.47
107.28
77.43
77.16
77.01
76.58
38.72
32.98
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

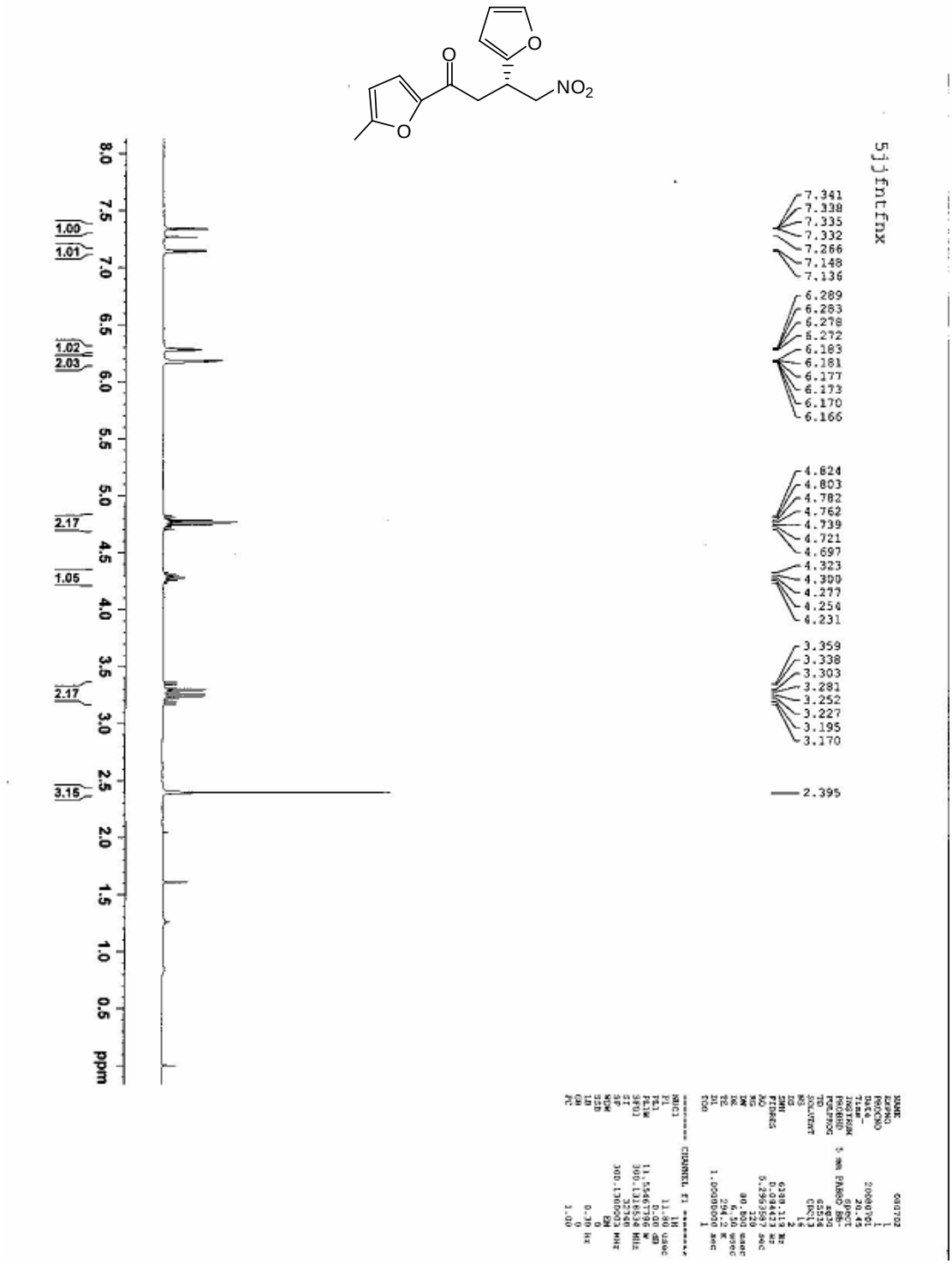
3n


O
S
NO2
O
I25
7.595
7.592
7.262
7.231
7.217
7.210
7.197
7.194
6.959
6.952
6.949
6.944
6.927
6.915
6.561
6.555
6.549
6.543
4.854
4.833
4.812
4.791
4.732
4.706
4.690
4.664
4.558
4.535
4.512
4.489
4.465
3.459
3.437
3.402
3.380
3.365
3.340
3.307
3.282
0.94
2.27
1.96
0.94
0.98
1.02
0.98
2.00
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
ppm

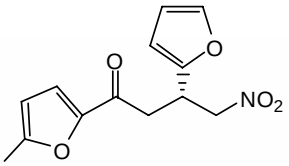
3n

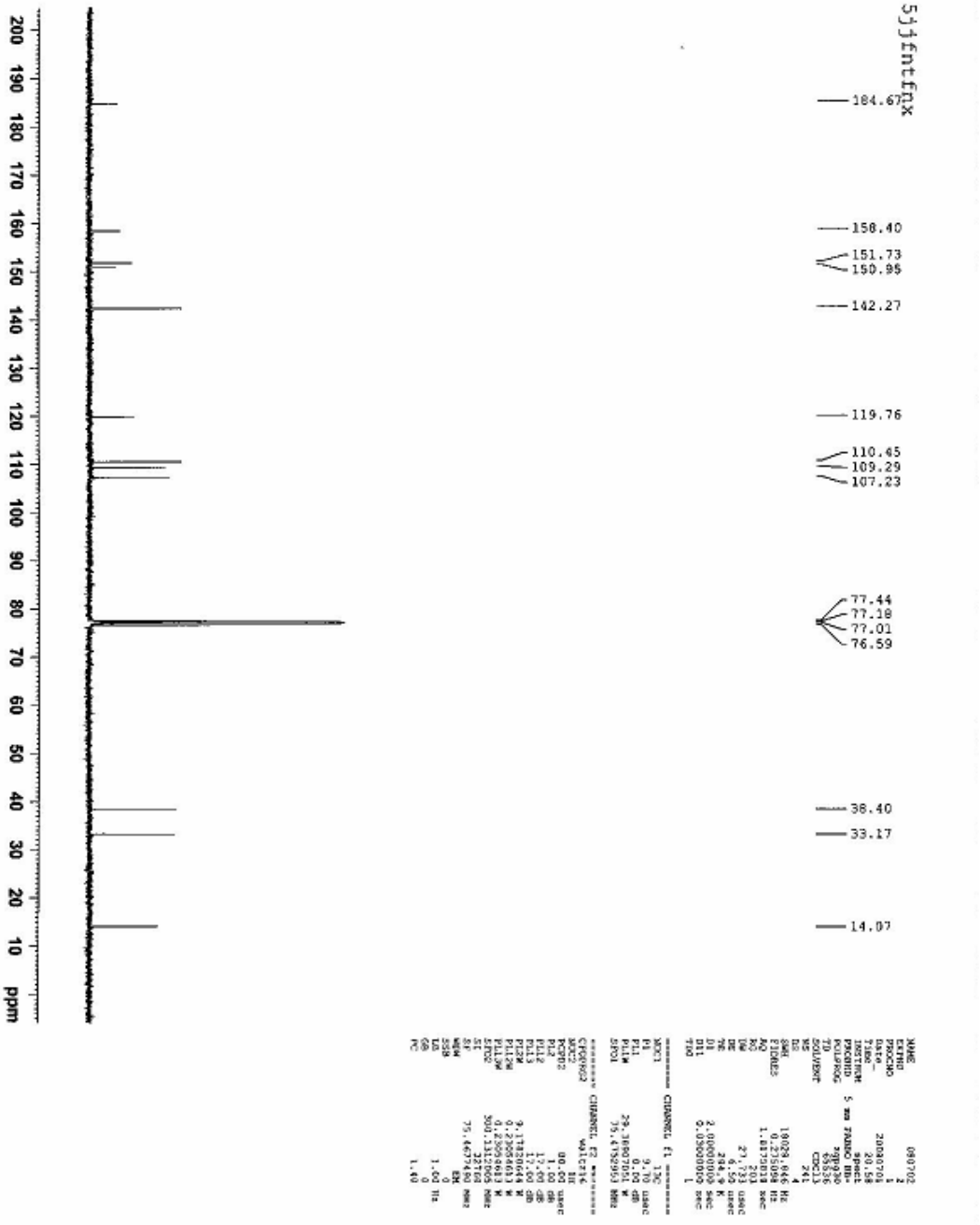

O
S
NO2
O
I25
185.45
152.23
146.82
141.47
127.13
125.67
124.78
117.73
112.57
79.79
77.44
77.02
76.59
41.94
34.56
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3o

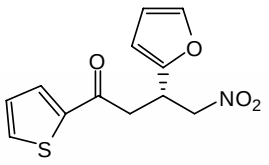

O
O
O
NO2
5jjfntfnx
7.341
7.338
7.335
7.332
7.266
7.148
7.136
6.289
6.283
6.278
6.272
6.183
6.181
6.177
6.173
6.170
6.166
4.824
4.803
4.782
4.762
4.739
4.721
4.697
4.323
4.300
4.277
4.254
4.231
3.359
3.338
3.303
3.281
3.252
3.227
3.195
3.170
2.395
1.00
1.01
1.02
2.03
2.17
1.05
2.17
3.15
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

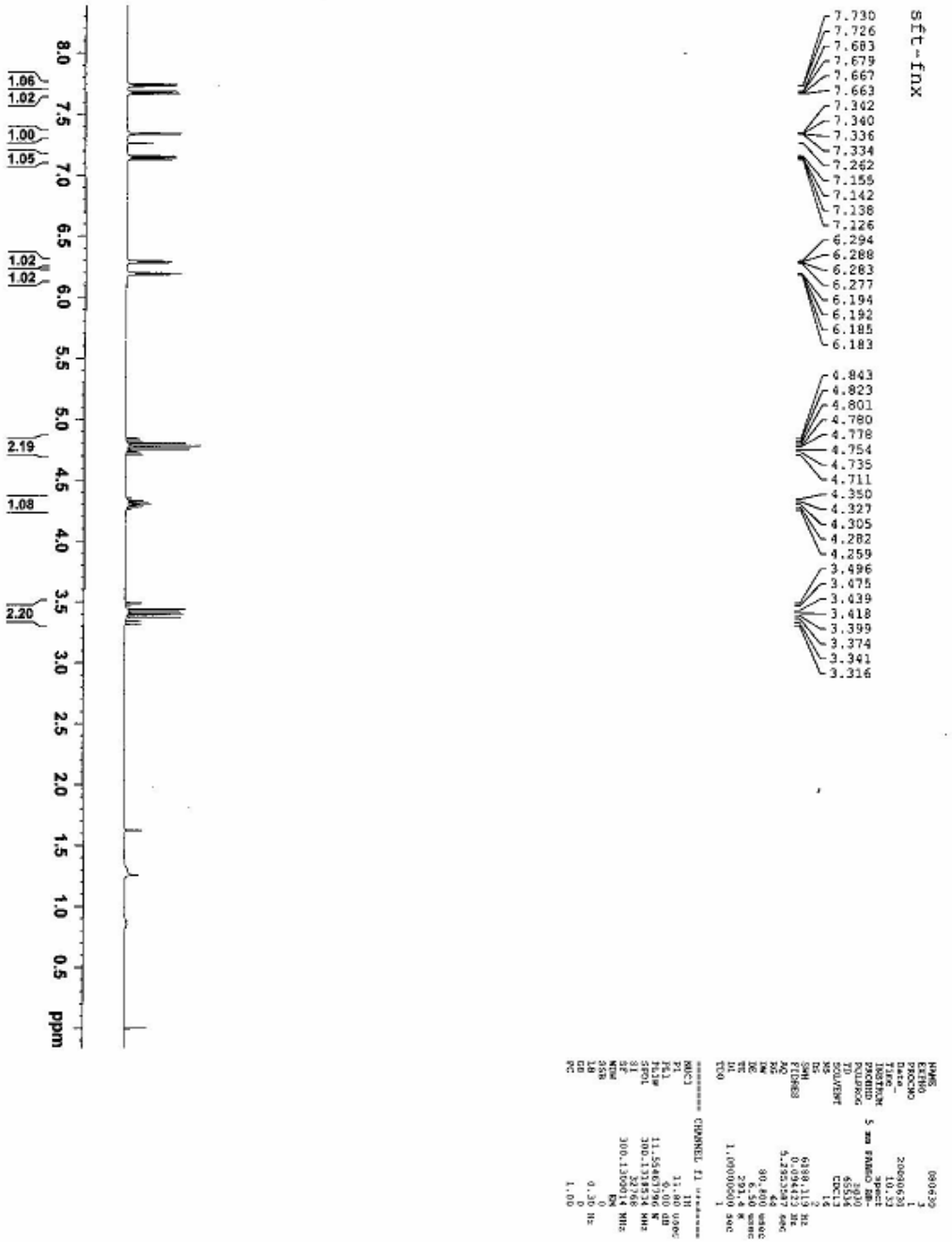
3o


O
O
O
NO2

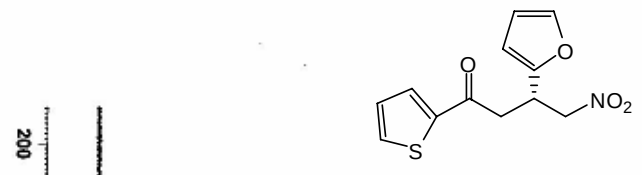

5jjfntfqx
184.67
158.40
151.73
150.95
142.27
119.76
110.45
109.29
107.23
77.44
77.18
77.01
76.59
38.40
33.17
14.07
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

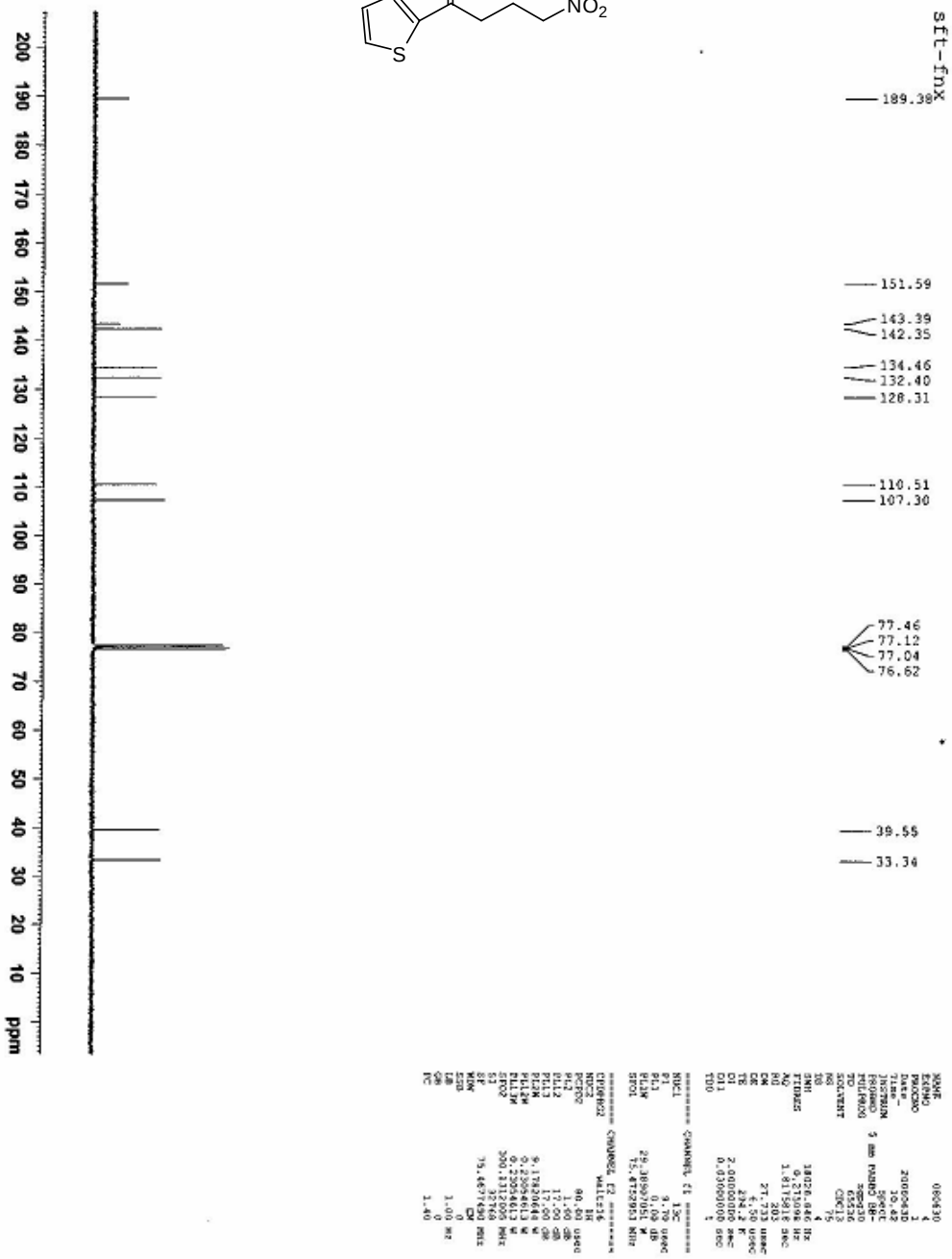
3p


O
O
NO2
S

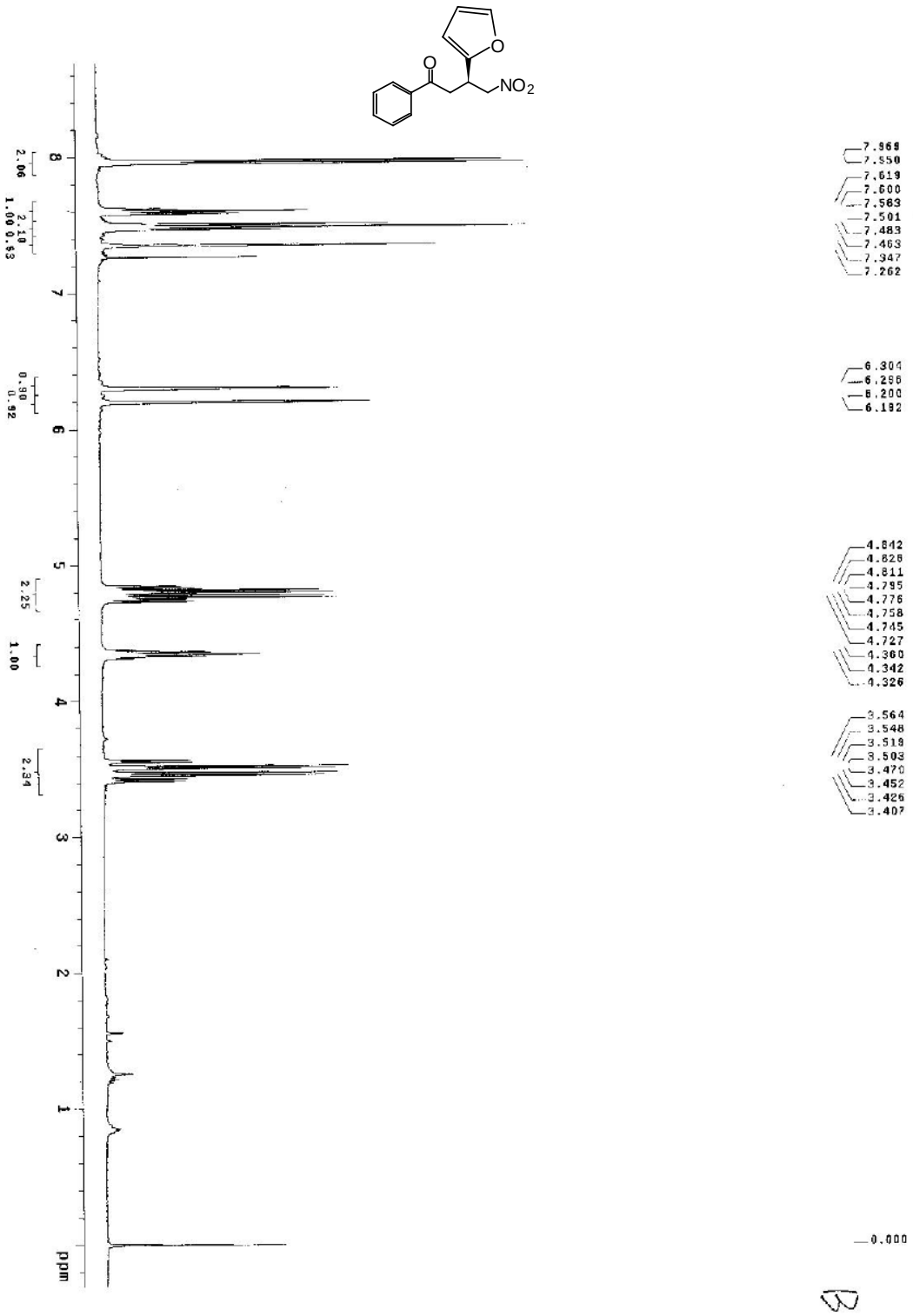

sft-fnX
7.730
7.726
7.683
7.679
7.667
7.663
7.342
7.340
7.336
7.334
7.262
7.155
7.142
7.138
7.126
6.294
6.288
6.283
6.277
6.194
6.192
6.185
6.183
4.843
4.823
4.801
4.780
4.778
4.754
4.735
4.711
4.350
4.327
4.305
4.282
4.259
3.496
3.475
3.439
3.418
3.399
3.374
3.341
3.316
1.06
1.02
1.00
1.05
1.02
1.02
2.19
1.08
2.20
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
ppm

3p

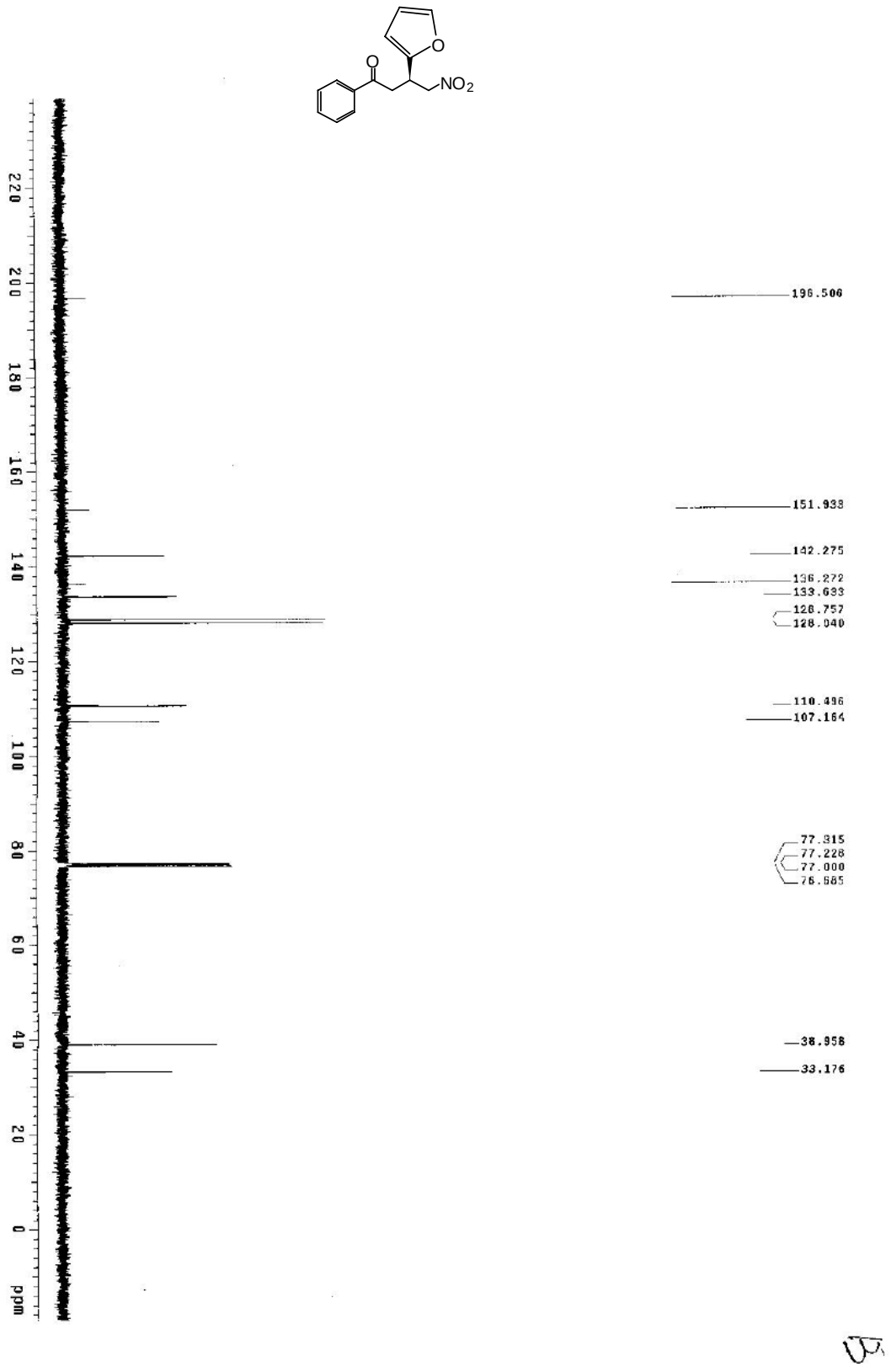

O
O
S
NO2


sft-fnX
189.38
151.59
143.39
142.35
134.46
132.40
128.31
110.51
107.30
77.46
77.12
77.04
76.62
39.55
33.34
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

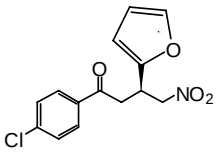
3q

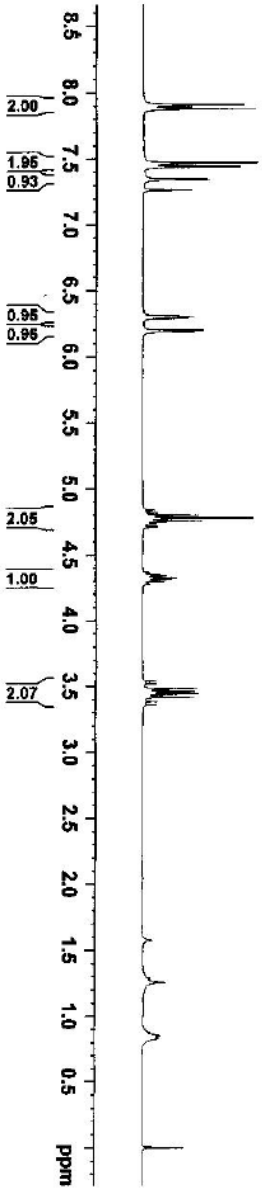

O
O
NO2
7.969
7.550
7.619
7.600
7.563
7.501
7.483
7.463
7.347
7.262
6.304
6.286
6.200
6.182
4.842
4.828
4.811
4.795
4.776
4.758
4.745
4.727
4.360
4.342
4.326
3.564
3.548
3.519
3.503
3.470
3.452
3.426
3.407
0.000
2.06
2.10
1.00
0.93
0.90
0.92
2.25
1.00
2.34
ppm

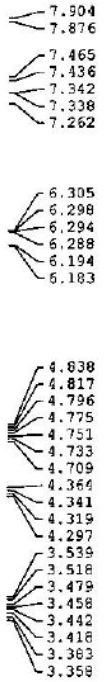
3q

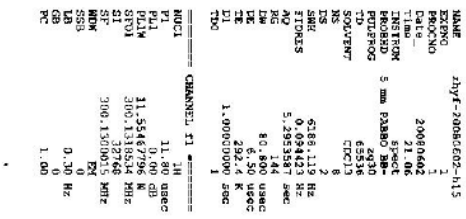

O
O
NO2
220
200
180
160
140
120
100
80
60
40
20
0
ppm
196.506
151.933
142.275
136.272
133.693
128.757
128.040
110.496
107.164
77.315
77.228
77.000
76.685
38.958
33.176

3r

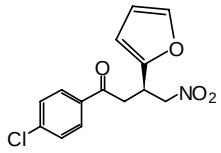

O
O
NO2
Cl

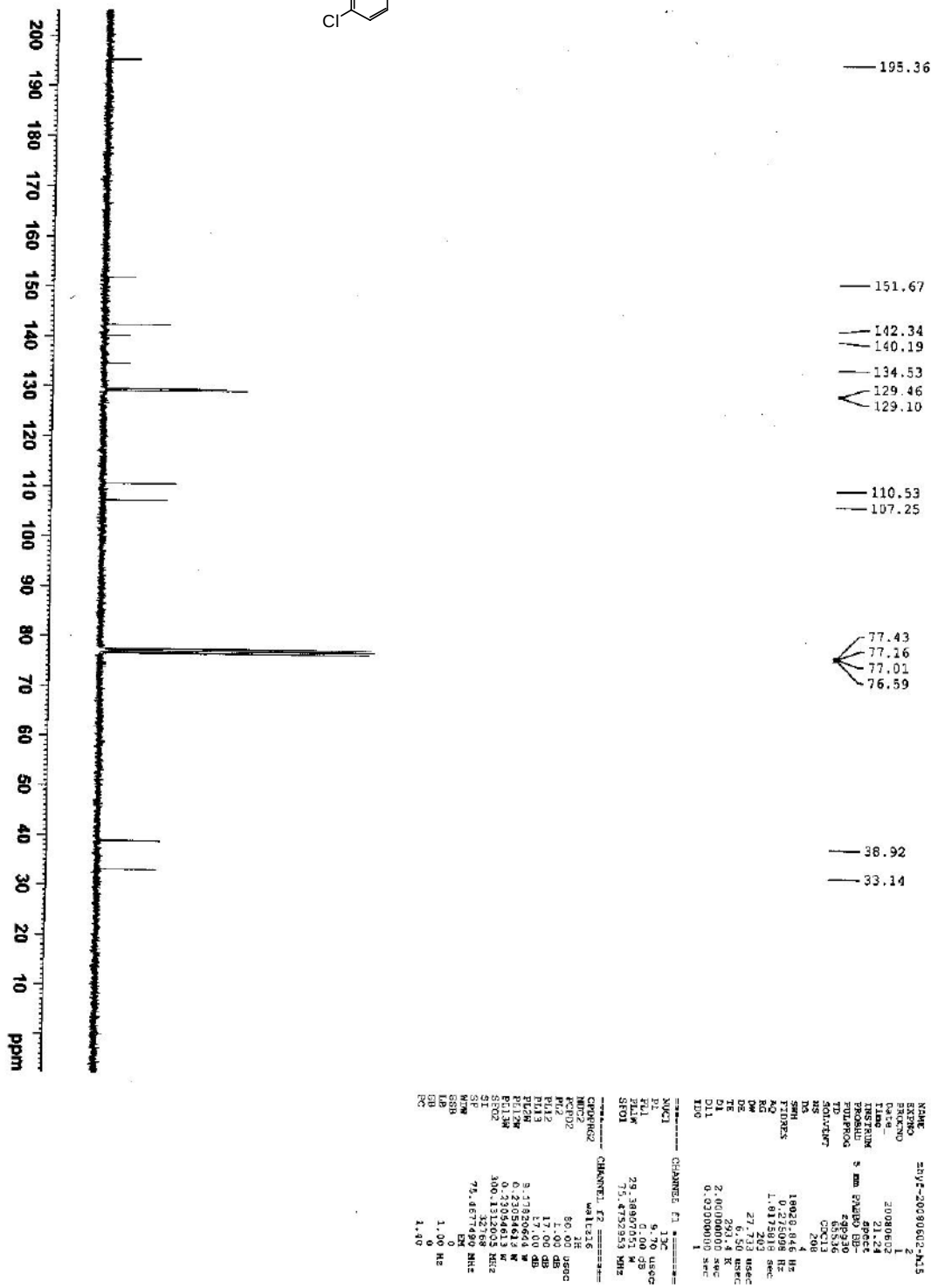

8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.00
1.95
0.93
0.95
0.96
2.05
1.00
2.07


7.904
7.876
7.465
7.436
7.342
7.338
7.262
6.305
6.298
6.294
6.288
6.194
6.183
4.838
4.817
4.796
4.775
4.751
4.733
4.709
4.364
4.341
4.319
4.297
3.539
3.518
3.479
3.458
3.442
3.418
3.383
3.359


NAME zhyf-2006602-h15
EXPNO 1
PROCNO 1
Date_ 20060602
Time 21.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6288.113 Hz
FIDRES 0.095972 Hz
AQ 5.2953587 sec
RG 144
DW 80.800 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PLW1 31.55467796 W
SFO1 300.1318524 MHz
SI 32768
SF 300.1300015 MHz
WDW EM
SSB 2
LB 0.30 Hz
GB 0
PC 1.00

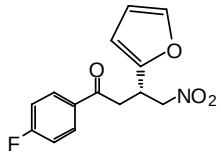
3r

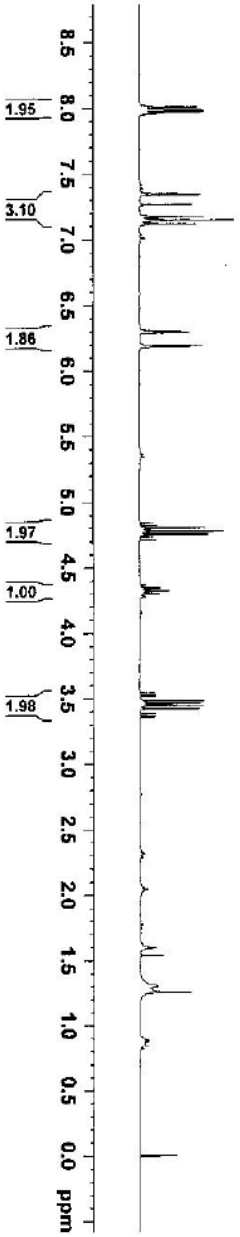


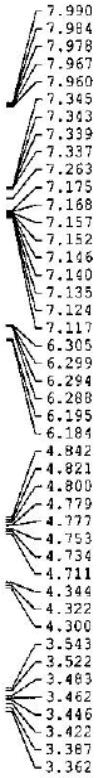


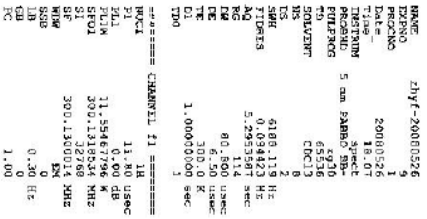
NAME sbyf-20080602-N15
EXPNO 2
PROCNO 1
Date_ 20080603
Time 21.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 208
DS 2
SWH 19038.046 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1 13C
NUC1
P1 9.70 usec
PL1 0.00 dB
PL1W 29.38905144 W
SFO1 75.4752953 MHz
CHANNEL f2 1H
CPDPRG2 waltz16
NUC2
P2 80.00 usec
PCPD2 12.00 dB
PL12 17.00 dB
PL13 17.00 dB
PL2W 8.17820644 W
PL12W 0.23054613 W
PL13W 0.23054613 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3s

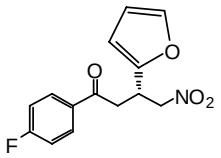

O
O
NO2
F

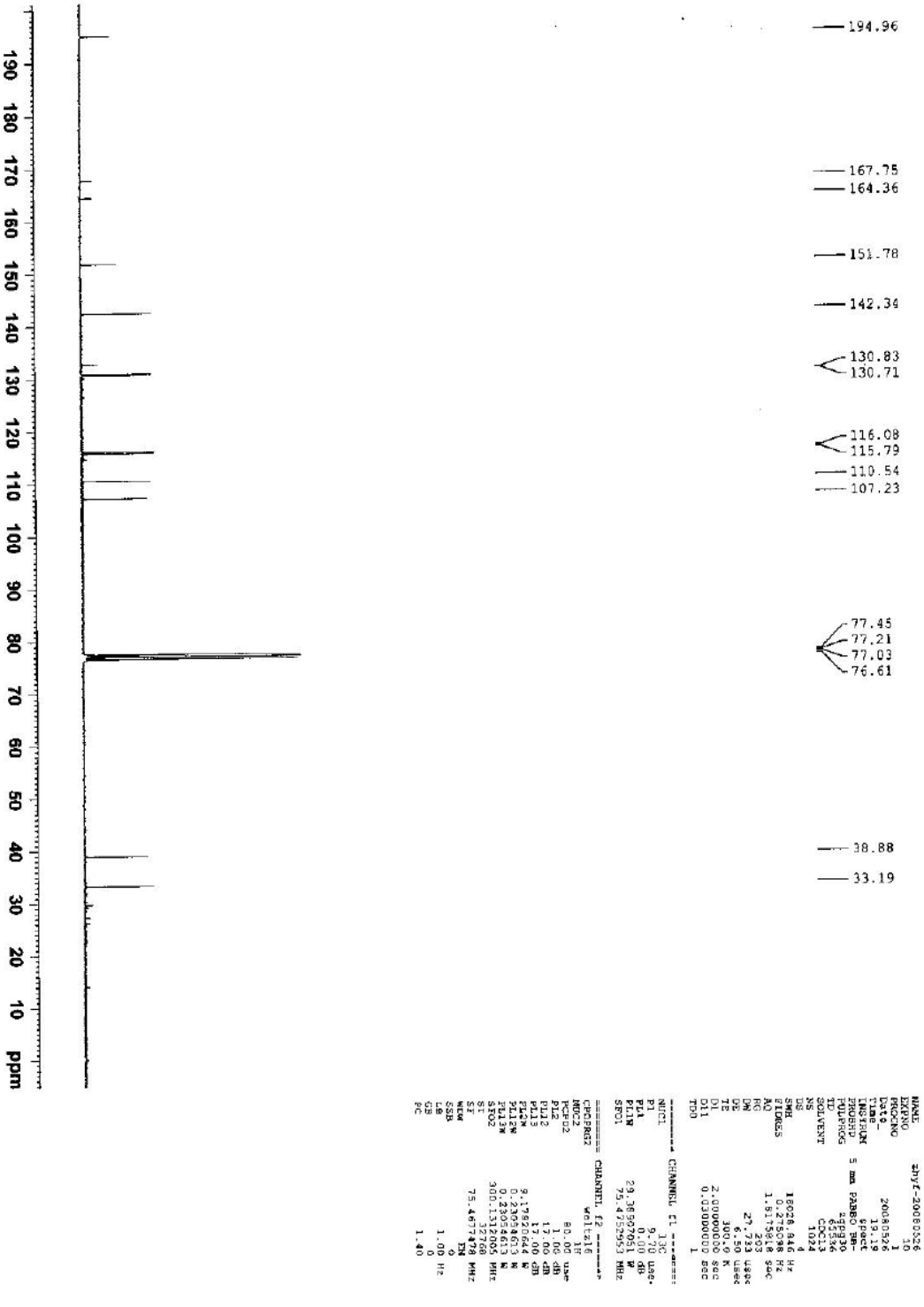

1.95
3.10
1.86
1.97
1.00
1.98
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm


7.990
7.984
7.978
7.967
7.960
7.345
7.343
7.339
7.337
7.263
7.175
7.168
7.157
7.152
7.146
7.140
7.135
7.124
7.117
6.305
6.299
6.294
6.288
6.195
6.184
4.842
4.821
4.800
4.779
4.777
4.753
4.734
4.711
4.344
4.322
4.300
3.543
3.522
3.483
3.462
3.446
3.422
3.387
3.362
1.255

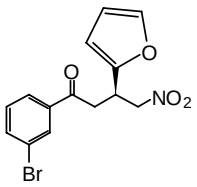

NAME zhyf-20080526
EXPNO 9
PROCNO 1
Date_ 20080526
Time 18.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953587 sec
RG 114
DW 80.750 usec
DE 6.500 usec
TE 300.0 K
D1 1.0000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300014 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

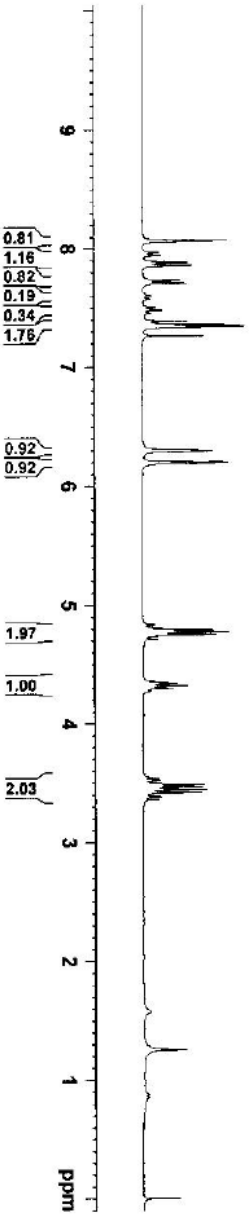
3s

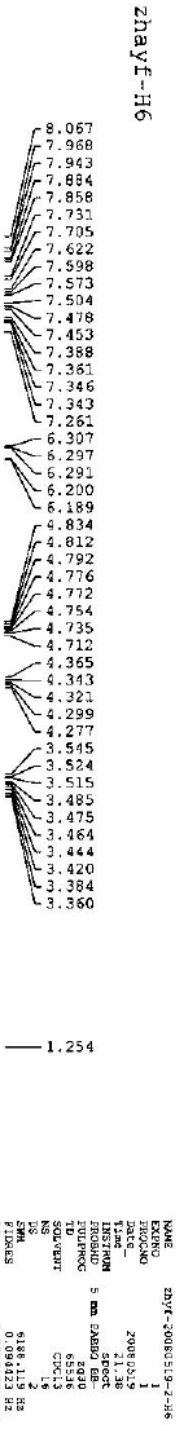

O
O
NO2
F


194.96
167.75
164.36
151.78
142.34
130.83
130.71
116.08
115.79
110.54
107.23
77.45
77.21
77.03
76.61
38.88
33.19
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3t

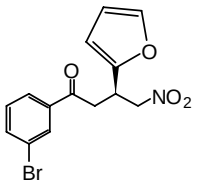


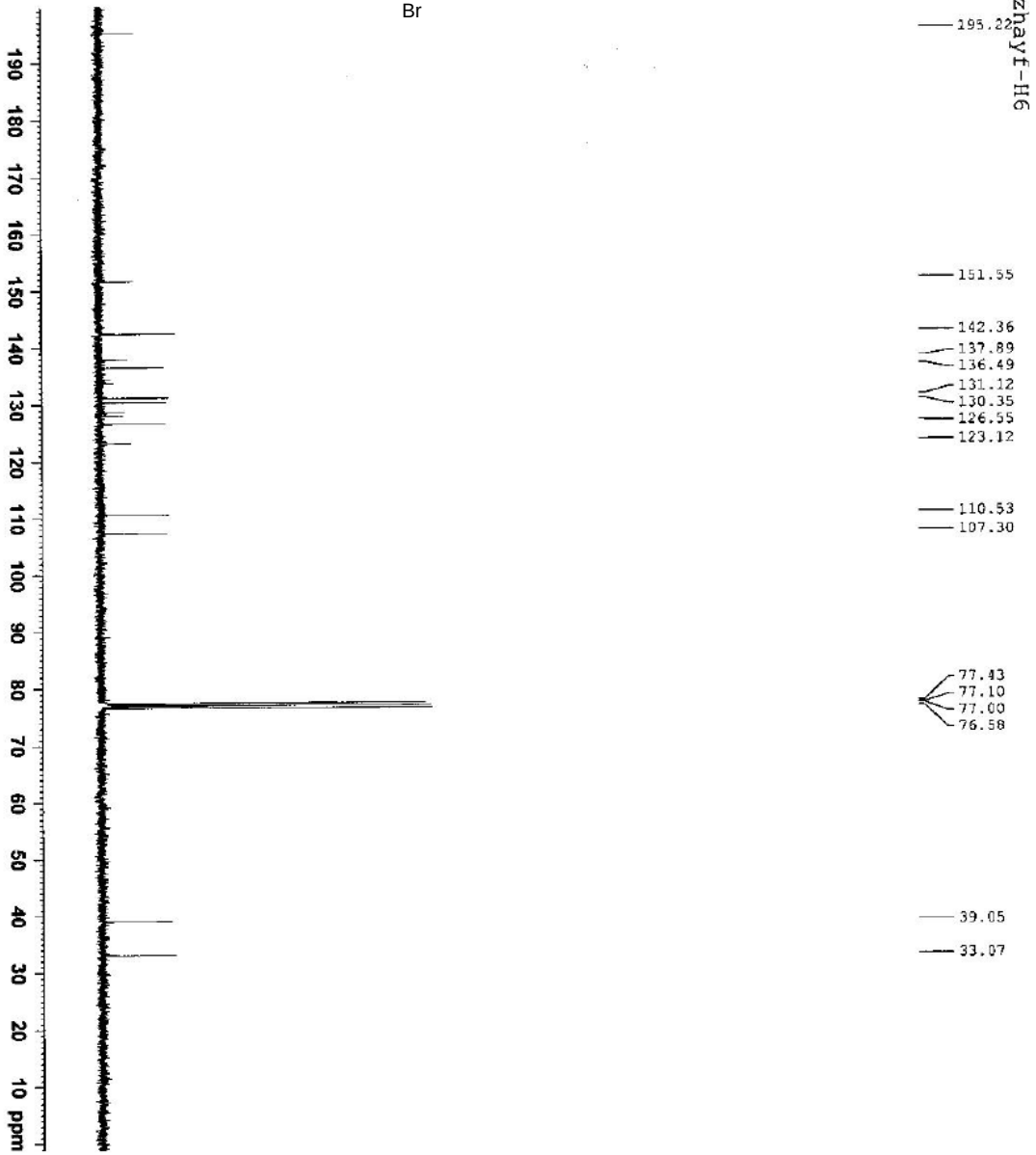




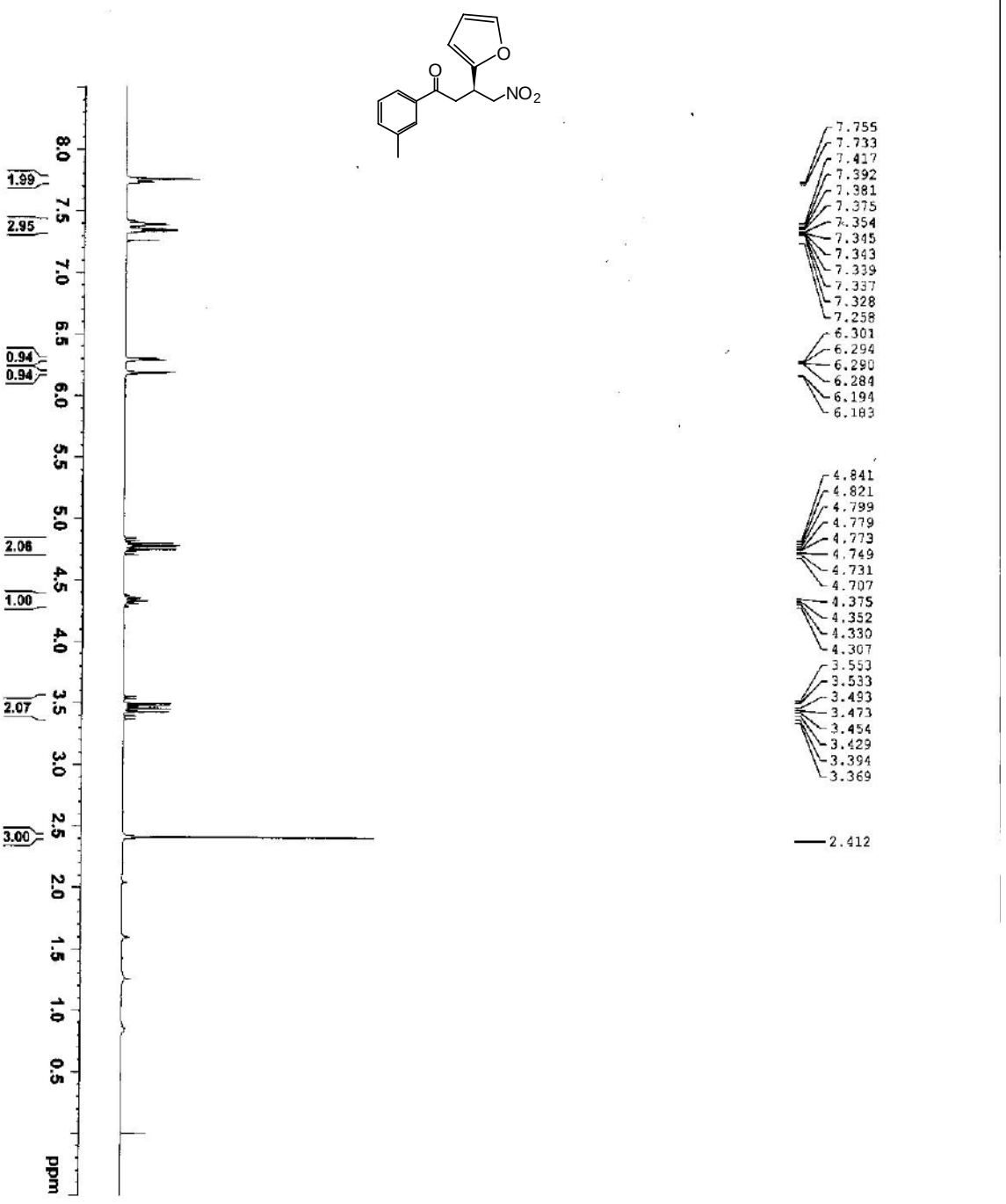
NAME zhayf-20080619zf-H6
EXPNO 1
PROCNO 1
Date_ 20080619
Time 21.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.094417 Hz
AQ 5.295581 sec
RG 181
DW 80.900 usec
DE 6.50 usec
TE 292.8 K
D1 1.0000000 sec
D0 1
------ CHANNEL f1 ------
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55467796 W
SFO1 300.1330554 MHz
SI 32768
SF 300.1300017 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

3t

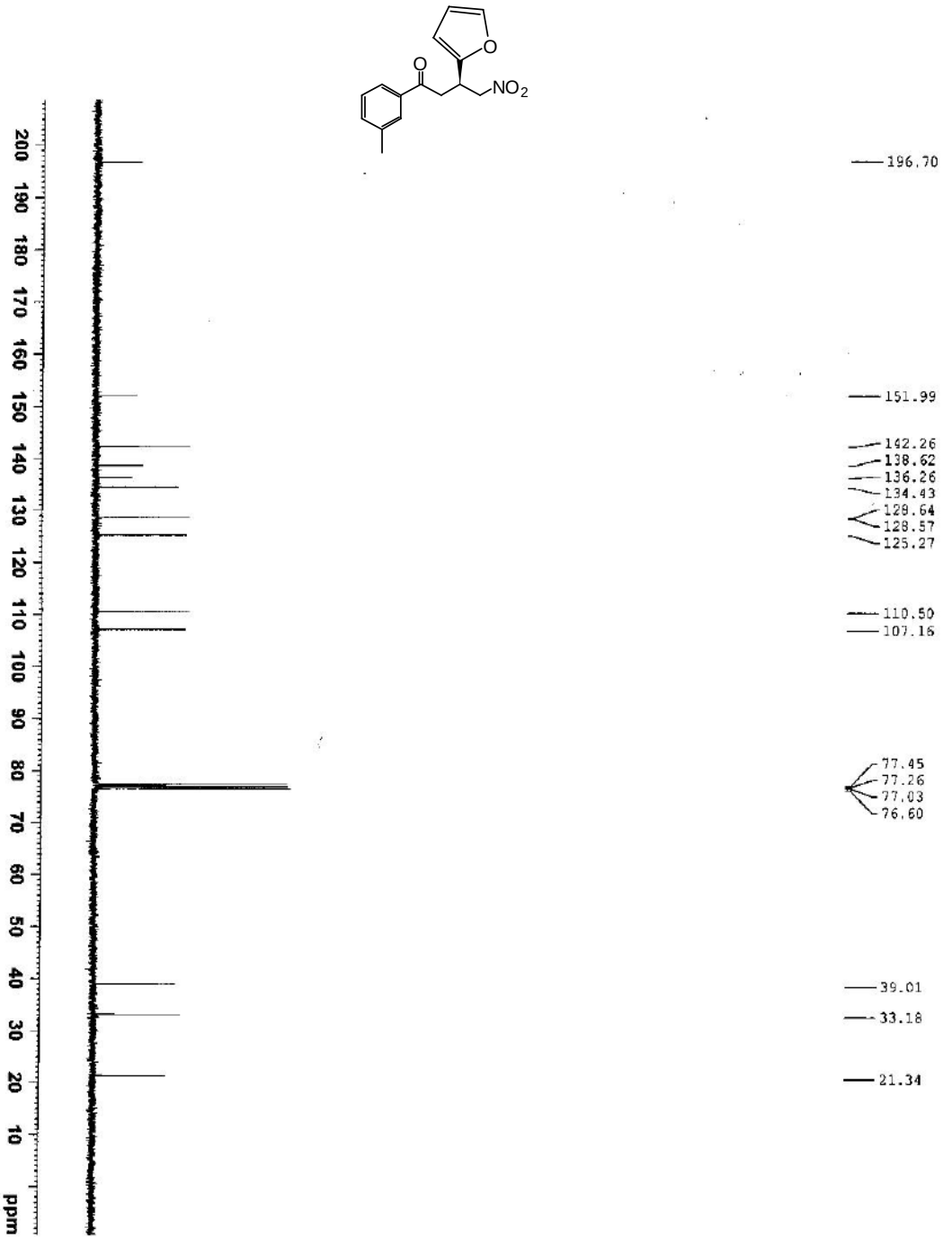

O
O
NO2
Br


zhayf-H6
195.22
151.55
142.36
137.89
136.49
131.12
130.35
126.55
123.12
110.53
107.30
77.43
77.10
77.00
76.58
39.05
33.07
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

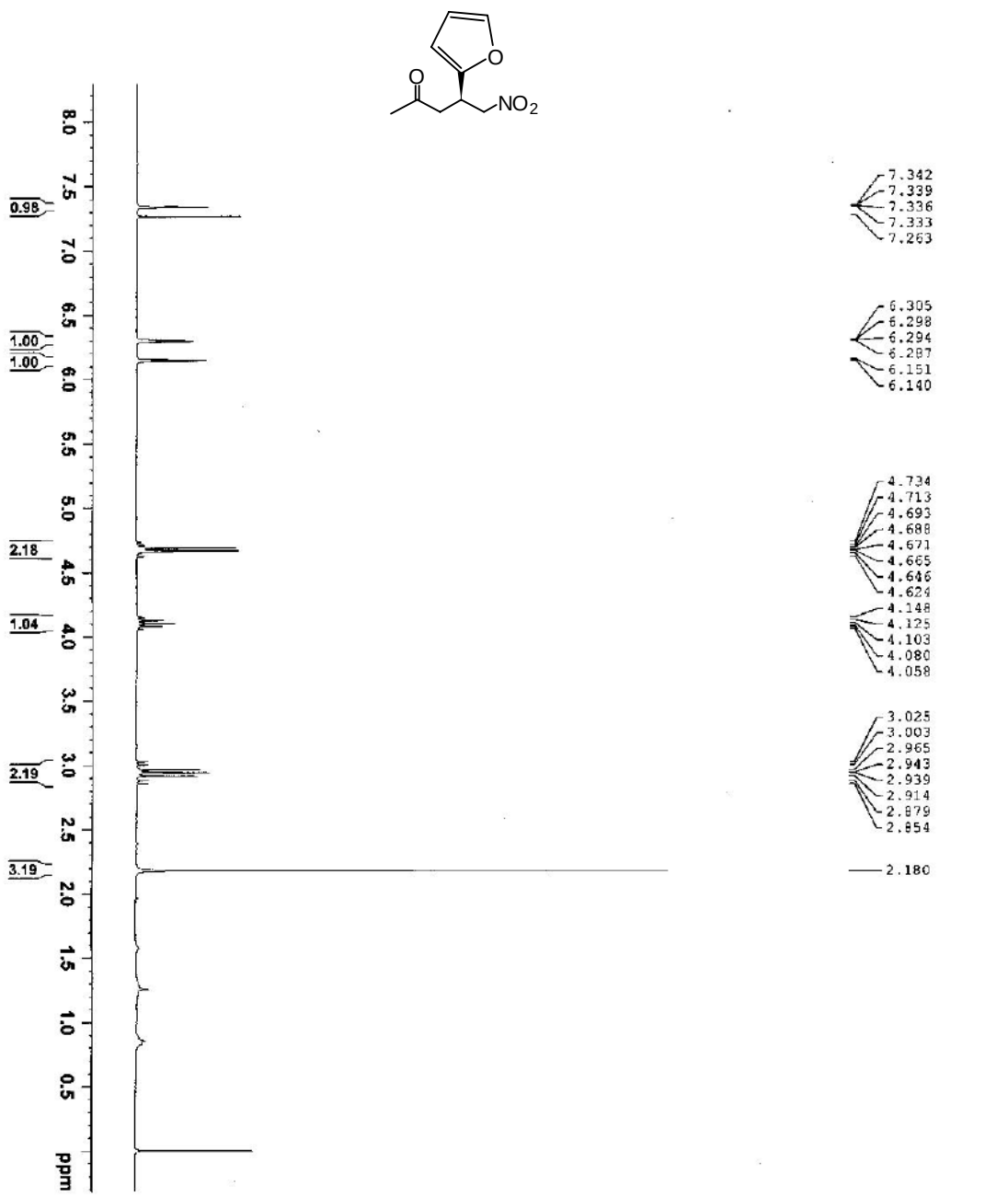
3u


O
O
NO2
7.755
7.733
7.417
7.392
7.381
7.375
7.354
7.345
7.343
7.339
7.337
7.328
7.258
6.301
6.294
6.290
6.284
6.194
6.183
4.841
4.821
4.799
4.779
4.773
4.749
4.731
4.707
4.375
4.352
4.330
4.307
3.553
3.533
3.493
3.473
3.454
3.429
3.394
3.369
2.412
1.99
2.95
0.94
0.94
2.06
1.00
2.07
3.00
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
ppm

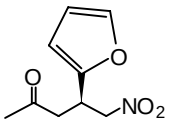
3u

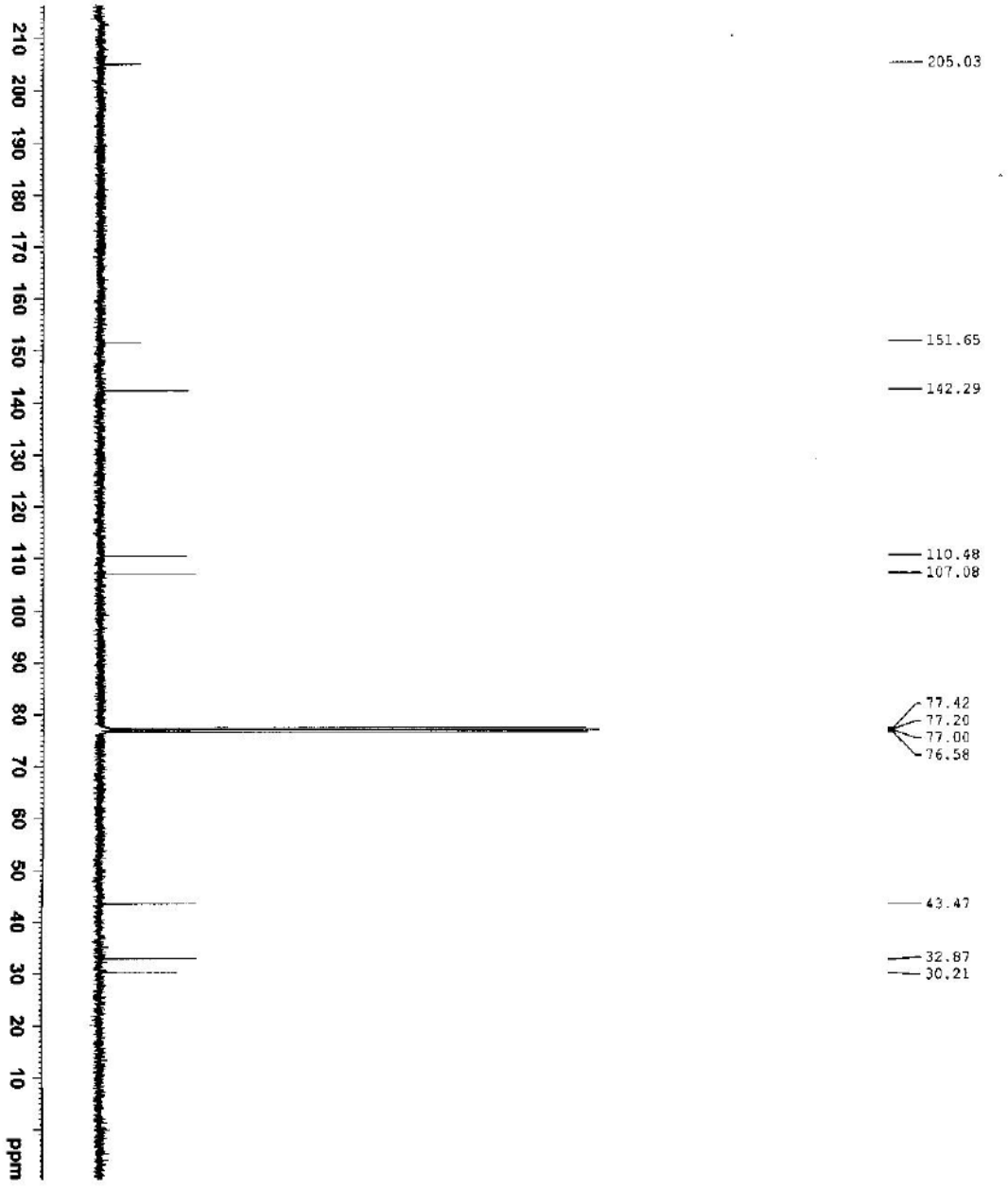

O
O
NO2
196.70
151.99
142.26
138.62
136.26
134.43
129.64
128.57
125.27
110.50
107.16
77.45
77.26
77.03
76.60
39.01
33.18
21.34
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3v

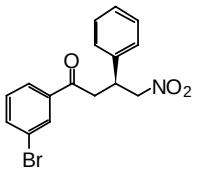

O
O
NO2
7.342
7.339
7.336
7.333
7.263
6.305
6.298
6.294
6.287
6.151
6.140
4.734
4.713
4.693
4.688
4.671
4.665
4.646
4.624
4.148
4.125
4.103
4.080
4.058
3.025
3.003
2.965
2.943
2.939
2.914
2.879
2.854
2.180
0.98
1.00
1.00
2.18
1.04
2.19
3.19
ppm

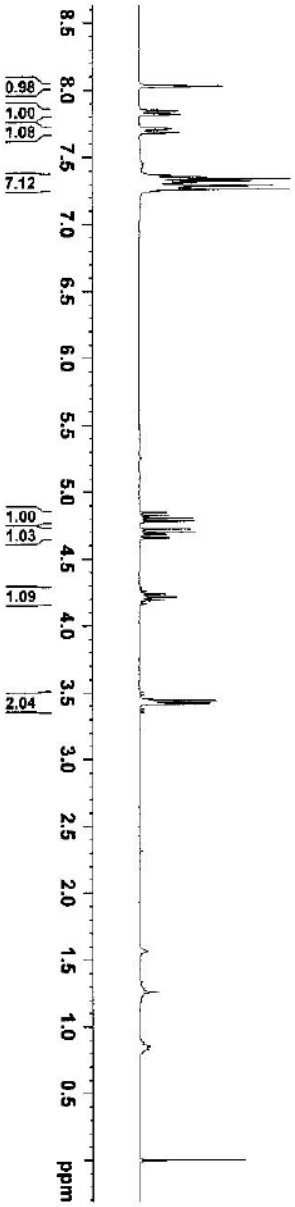
3v

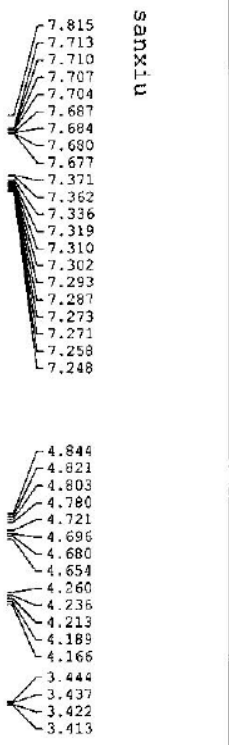

O
O
NO2


205.03
151.65
142.29
110.48
107.08
77.42
77.20
77.00
76.58
43.47
32.87
30.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

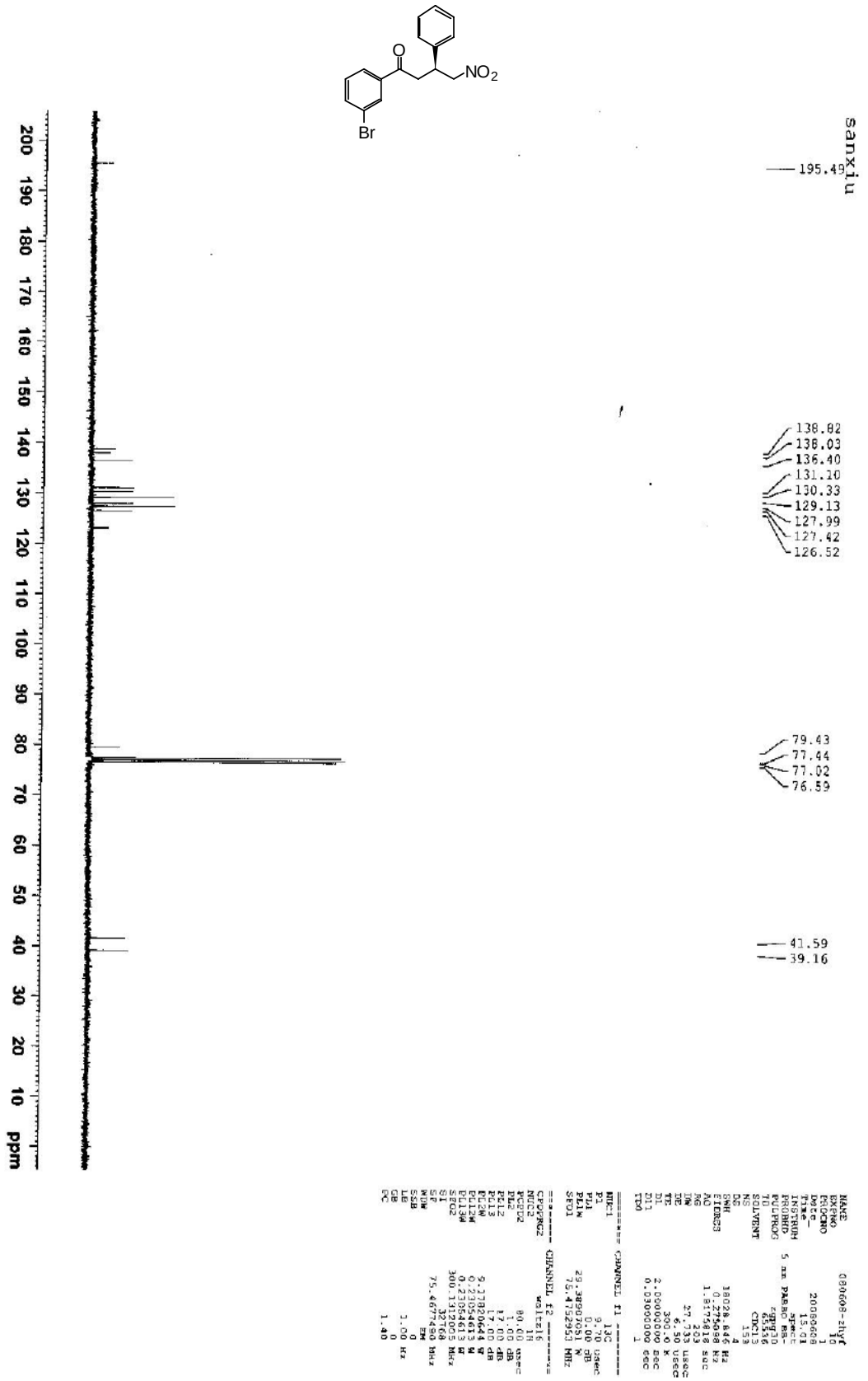
3c'


O
NO2
Br

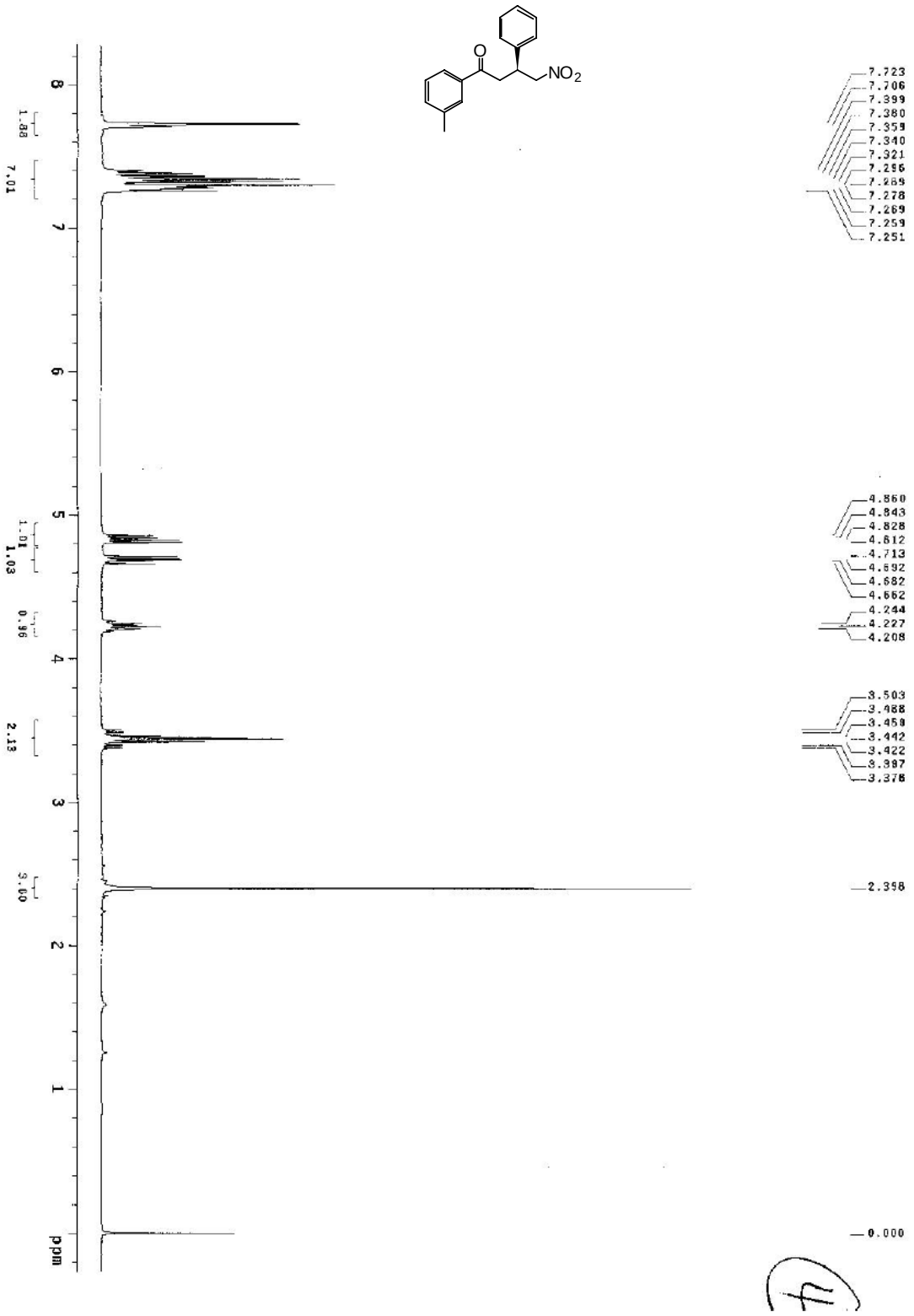

0.98
1.00
1.08
7.12
1.00
1.03
1.09
2.04
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


sanxiu
7.815
7.713
7.710
7.707
7.704
7.687
7.684
7.680
7.677
7.371
7.362
7.336
7.319
7.310
7.302
7.293
7.287
7.273
7.271
7.258
7.248
4.844
4.821
4.803
4.780
4.721
4.696
4.680
4.654
4.260
4.236
4.213
4.189
4.166
3.444
3.437
3.422
3.413

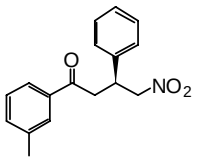
3c'


O
NO2
Br
sanxiu
195.49
138.82
136.03
136.40
131.10
130.33
129.13
127.99
127.42
126.52
79.43
77.44
77.02
76.59
41.59
39.16
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3d'

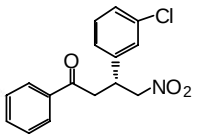

O
NO2
7.723
7.706
7.399
7.380
7.359
7.340
7.321
7.296
7.289
7.278
7.269
7.259
7.251
4.860
4.843
4.828
4.812
4.713
4.692
4.682
4.662
4.244
4.227
4.208
3.503
3.488
3.459
3.442
3.422
3.397
3.378
2.398
0.000
ppm

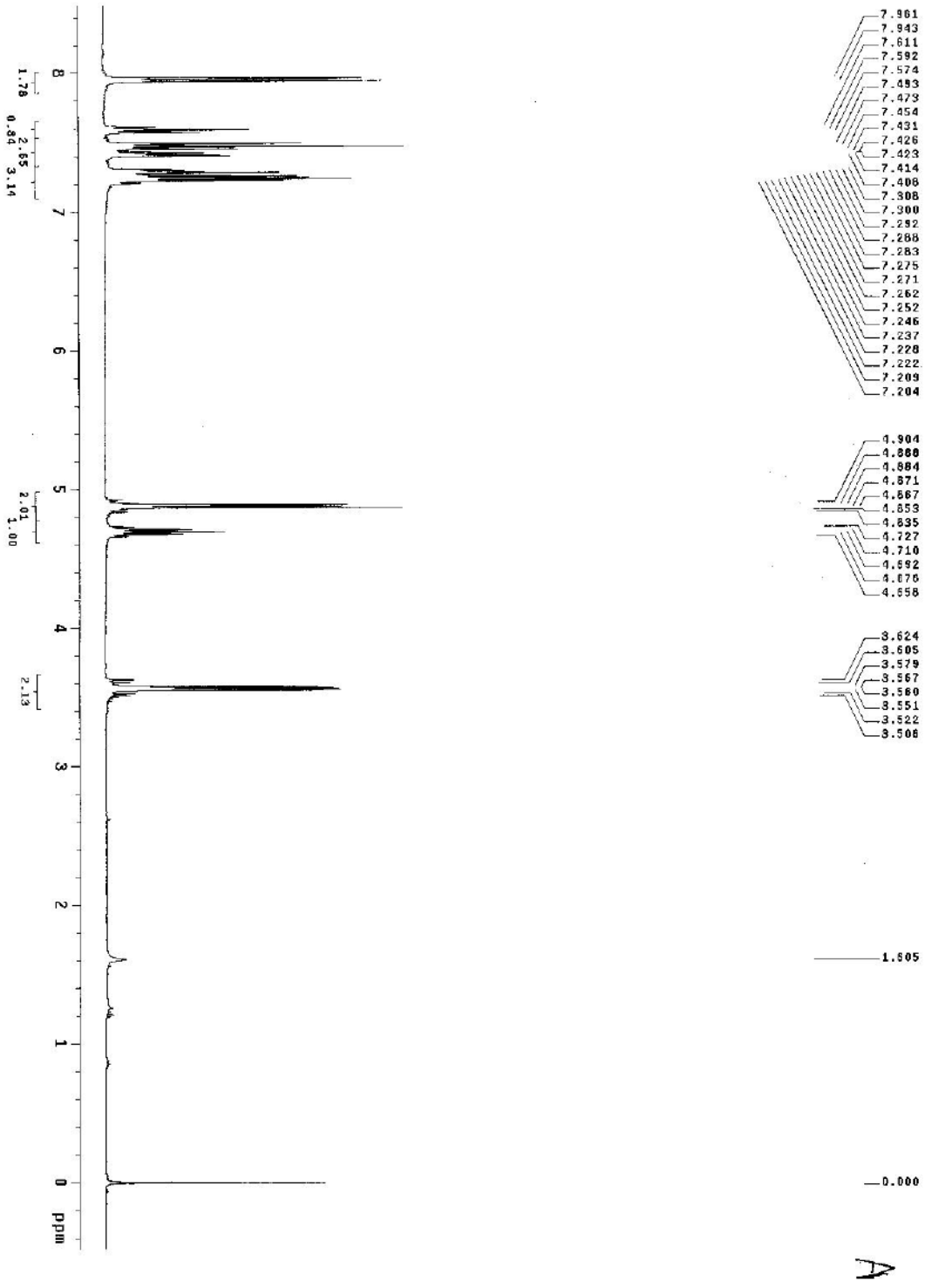
3d'


O
NO2

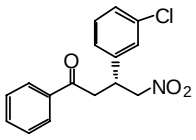
196.987
139.203
138.561
136.454
134.285
129.049
128.592
128.544
127.835
127.449
125.220
78.552
77.323
77.000
76.685
41.581
38.305
21.312
ppm

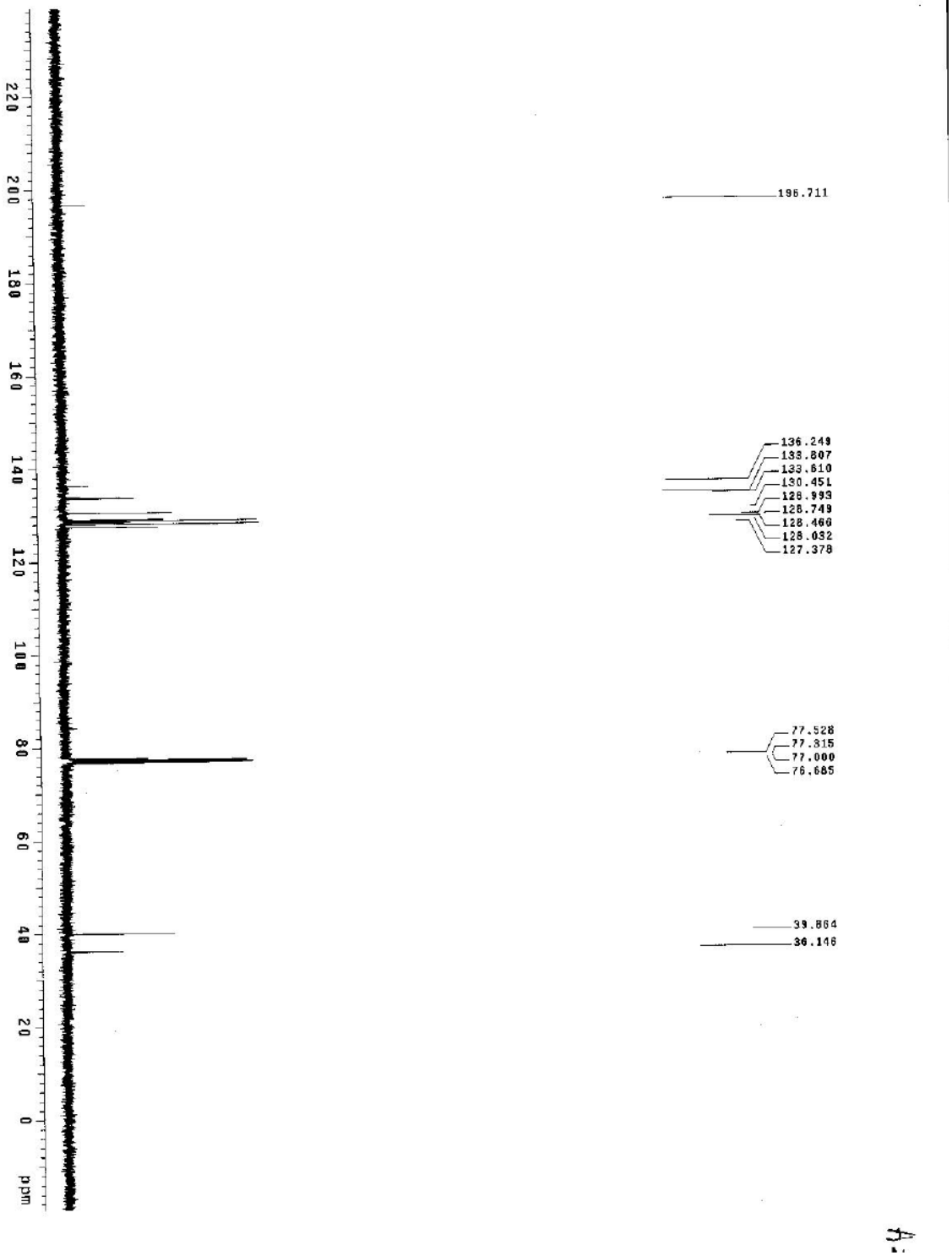
3h'


Cl
O
NO2

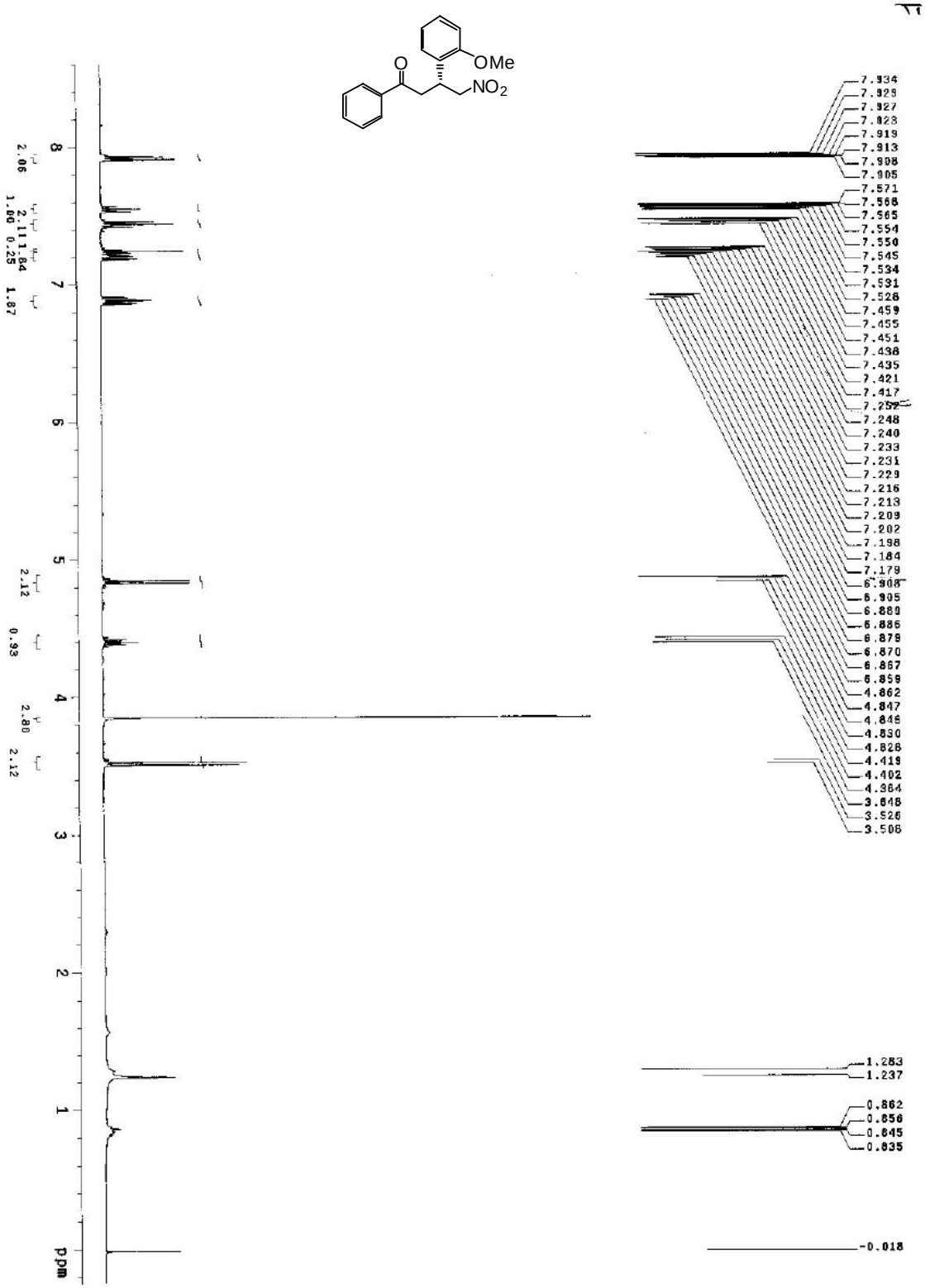

1.78
0.84
2.55
3.14
2.01
1.00
2.13
8
7
6
5
4
3
2
1
0
ppm
7.961
7.943
7.611
7.592
7.574
7.493
7.473
7.454
7.431
7.426
7.423
7.414
7.408
7.308
7.300
7.292
7.288
7.283
7.275
7.271
7.262
7.252
7.246
7.237
7.228
7.222
7.209
7.204
4.904
4.888
4.884
4.871
4.867
4.853
4.835
4.727
4.710
4.692
4.676
4.658
3.624
3.605
3.579
3.567
3.560
3.551
3.522
3.508
1.505
0.000

3h'

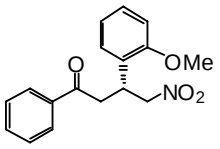

Cl
O
NO2


196.711
136.249
133.807
133.610
130.451
128.993
128.749
128.466
128.032
127.378
77.528
77.315
77.000
76.685
39.864
36.148
220
200
180
160
140
120
100
80
60
40
20
0
ppm

3j'

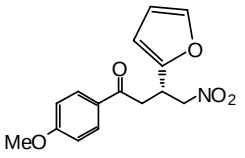

O
OMe
NO2
7.934
7.929
7.927
7.923
7.919
7.913
7.908
7.905
7.571
7.568
7.565
7.554
7.550
7.545
7.534
7.531
7.528
7.459
7.455
7.451
7.438
7.435
7.421
7.417
7.252
7.248
7.240
7.233
7.231
7.229
7.216
7.213
7.209
7.202
7.198
7.184
7.179
6.908
6.905
6.880
6.886
6.879
6.870
6.867
6.859
4.862
4.847
4.846
4.830
4.828
4.419
4.402
4.384
3.548
3.526
3.508
1.283
1.237
0.862
0.856
0.845
0.835
-0.018
ppm

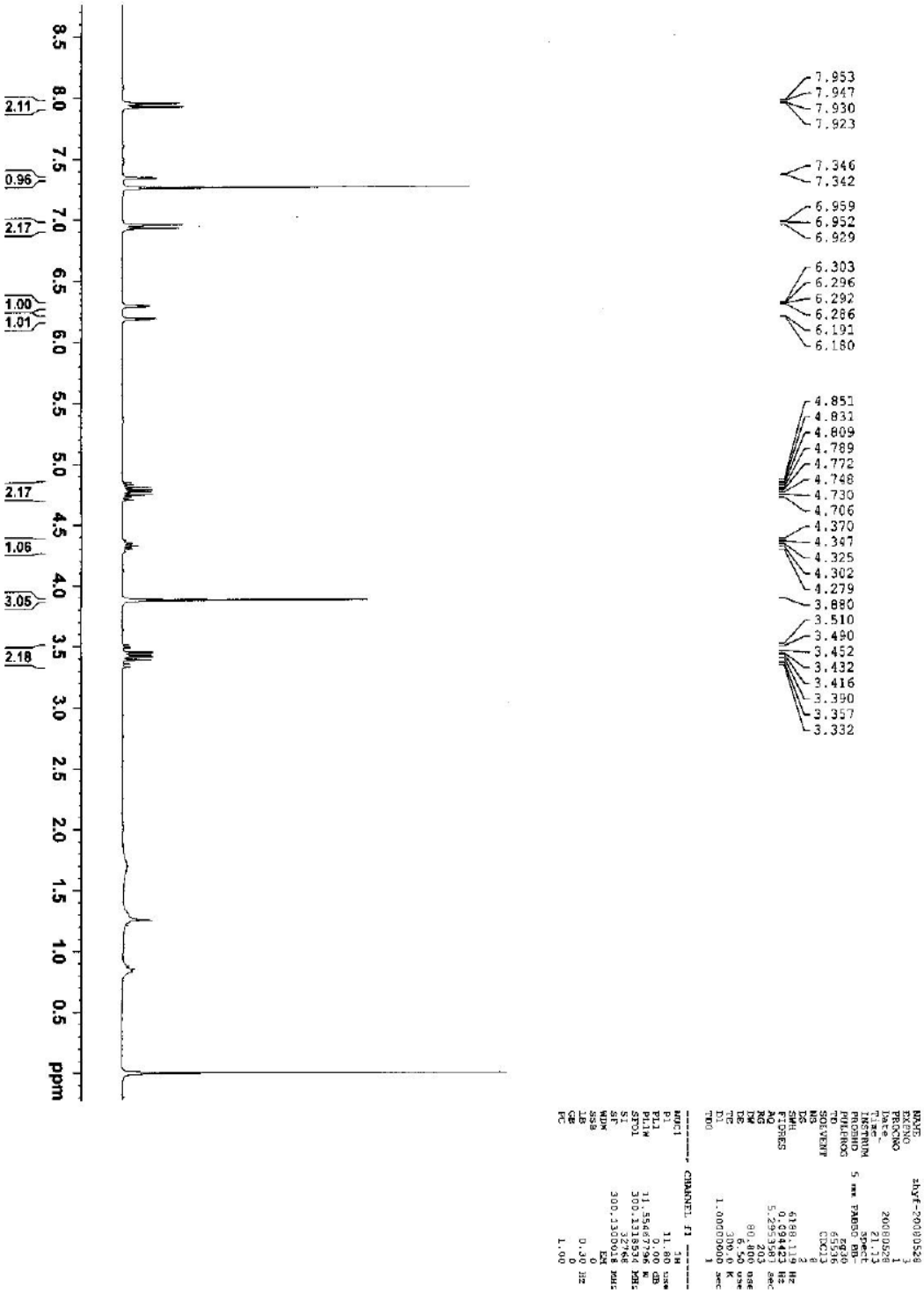
3j'


O
OMe
NO2

197.597
157.141
136.555
133.342
129.481
128.947
128.634
128.009
126.628
120.913
110.978
77.839
77.320
77.000
76.680
55.330
39.726
35.868
29.677
200
180
160
140
120
100
80
60
40
ppm

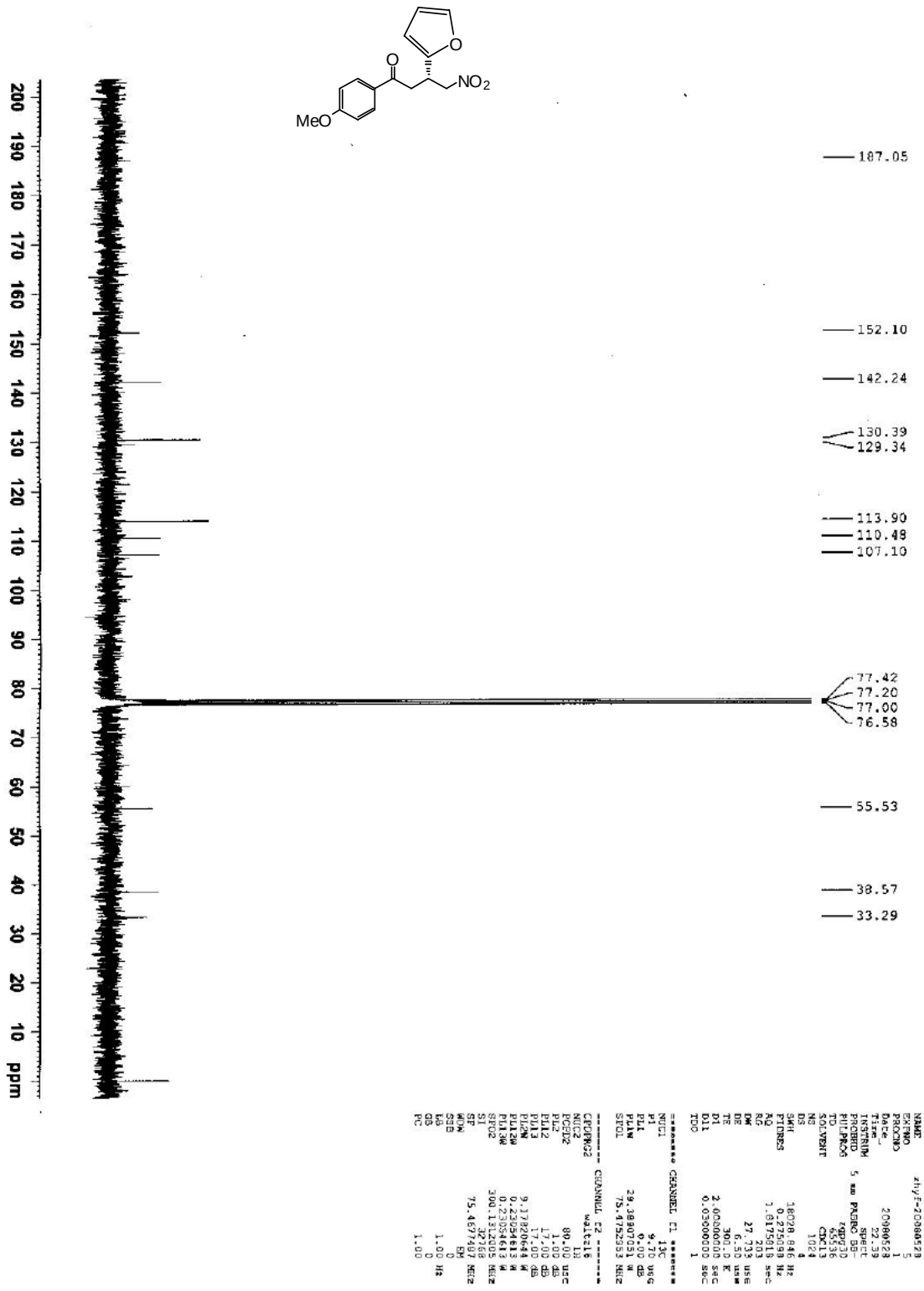
3l'



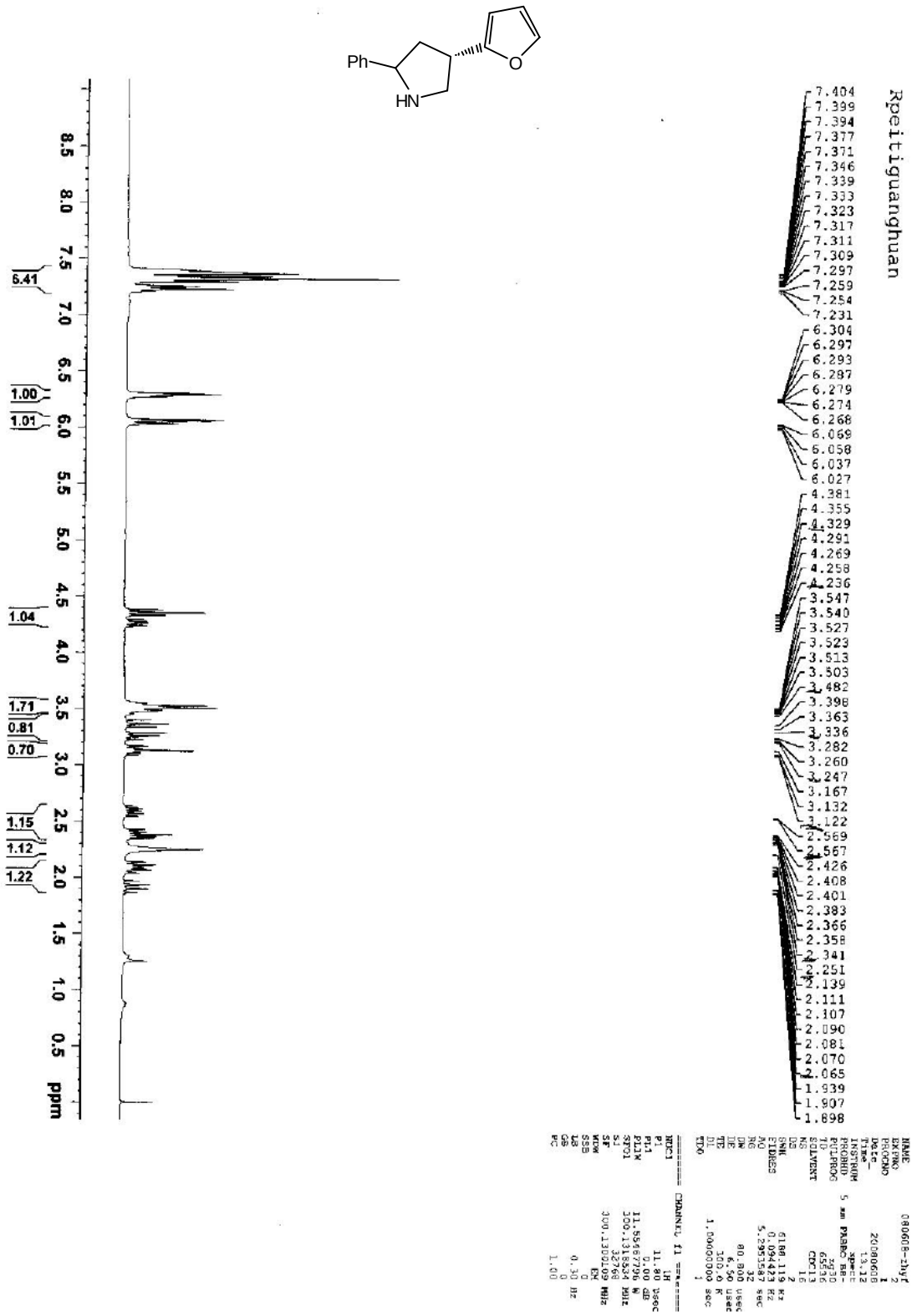


NAME zhyf-20080529
EXPNO 1
PROCNO 1
Date_ 20080528
Time 21.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6188.119 Hz
FIDRES 0.094423 Hz
AQ 5.2953681 sec
RG 228.03
DW 80.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.80 usec
PL1 0.00 dB
PL1W 11.55487796 W
SFO1 300.2318934 MHz
SI 32768
SF 300.1300018 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

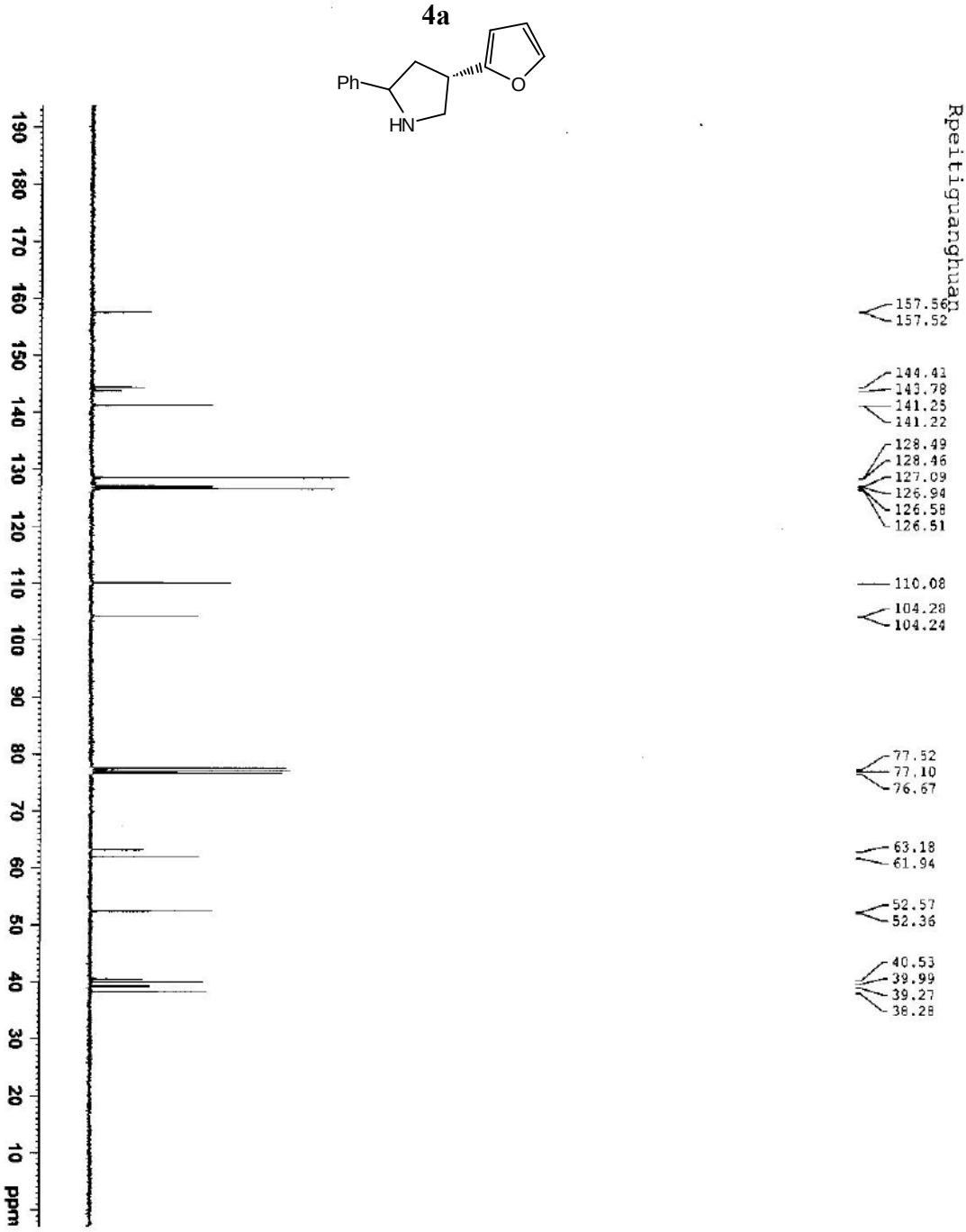
3l'


MeO
O
O
NO2
187.05
152.10
142.24
130.39
129.34
113.90
110.48
107.10
77.42
77.20
77.00
76.58
55.53
38.57
33.29
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

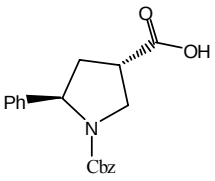
4a

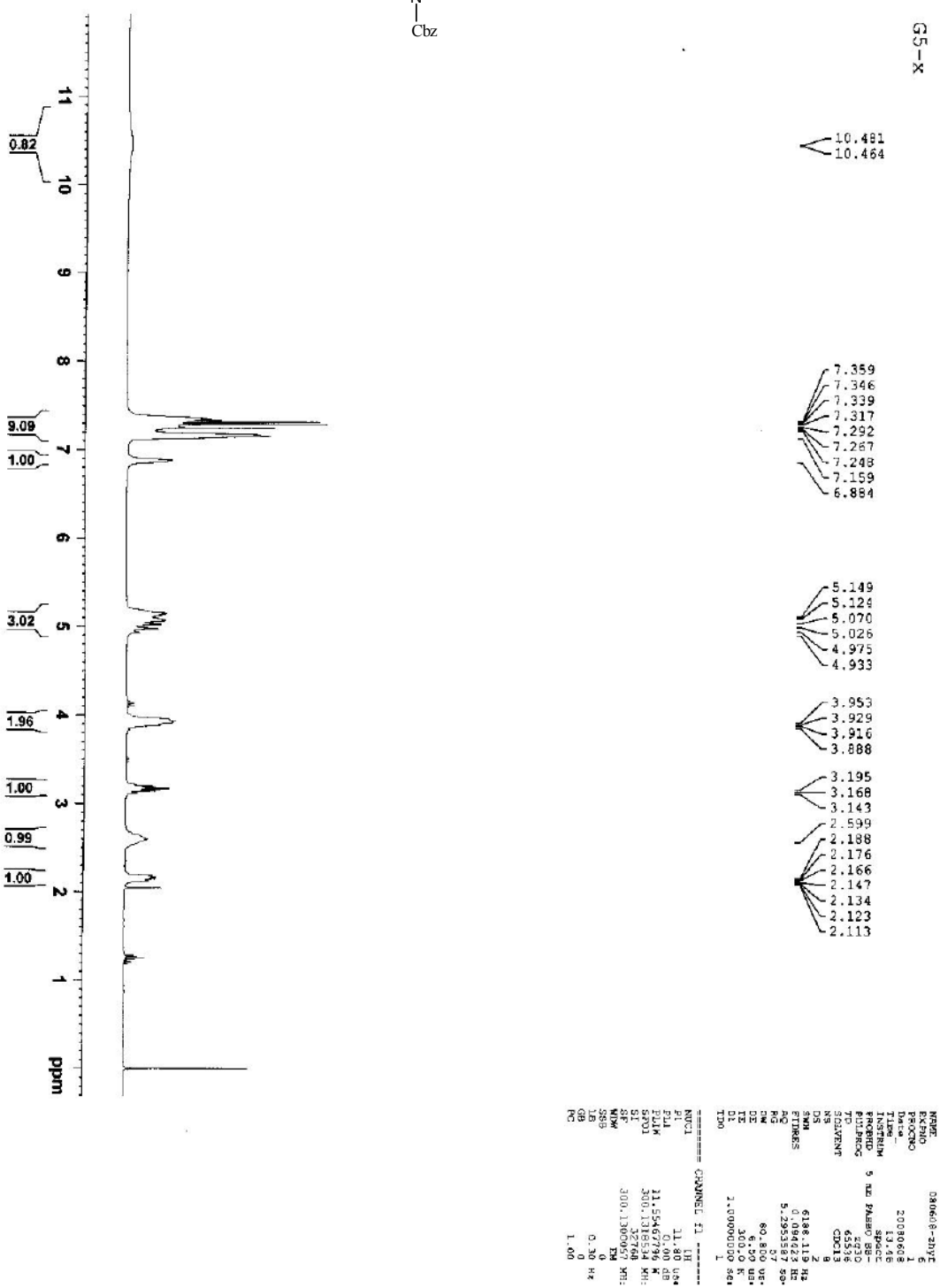

Ph
HN
O
Rpeitiguanghuan
6.41
1.00
1.01
1.04
1.71
0.81
0.70
1.15
1.12
1.22
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

4a

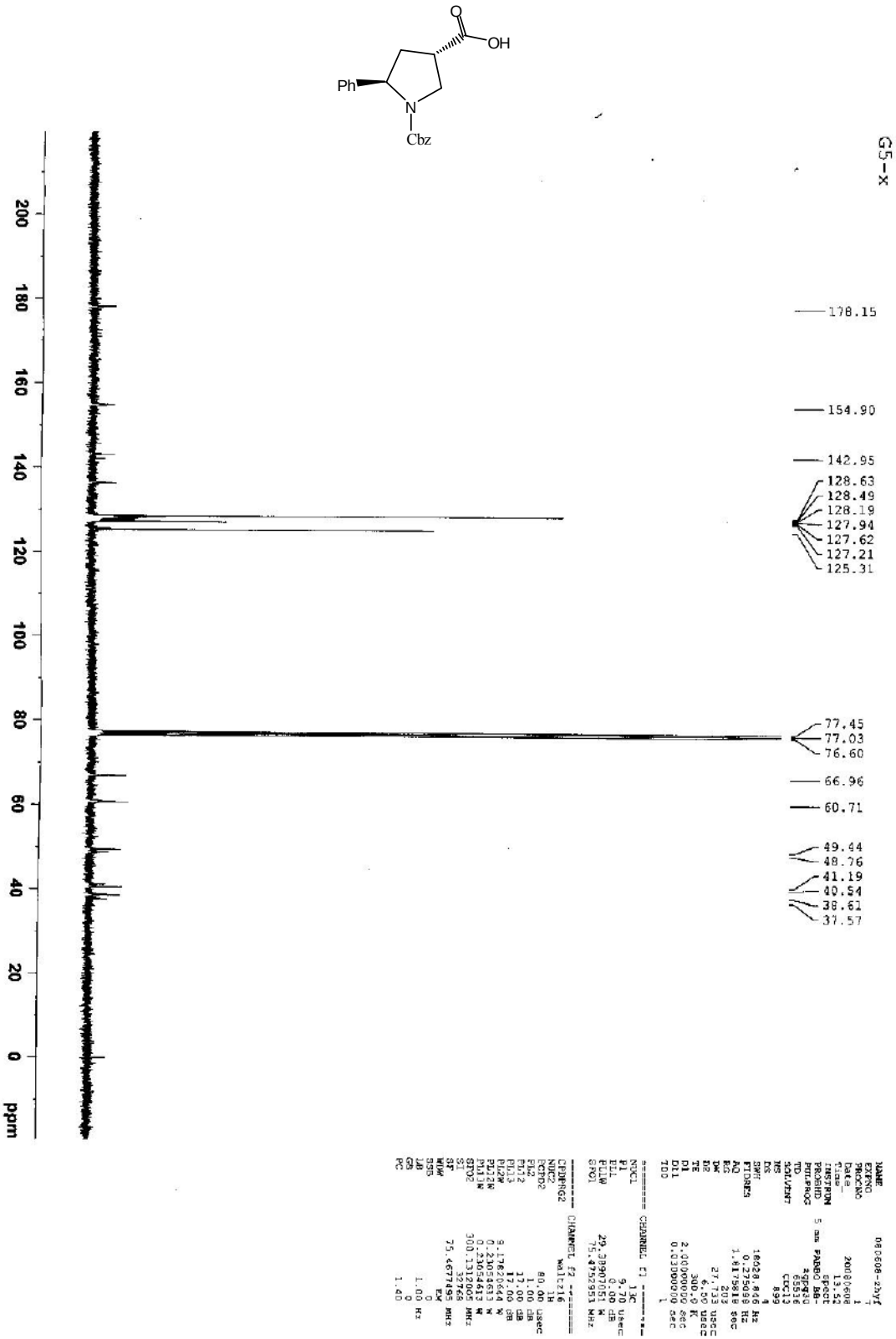

Ph
HN
O
Rpeitiguanghuan
157.56
157.52
144.41
143.78
141.25
141.22
128.49
128.46
127.09
126.94
126.58
126.51
110.08
104.28
104.24
77.52
77.10
76.67
63.18
61.94
52.57
52.36
40.53
39.99
39.27
38.28
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

5a

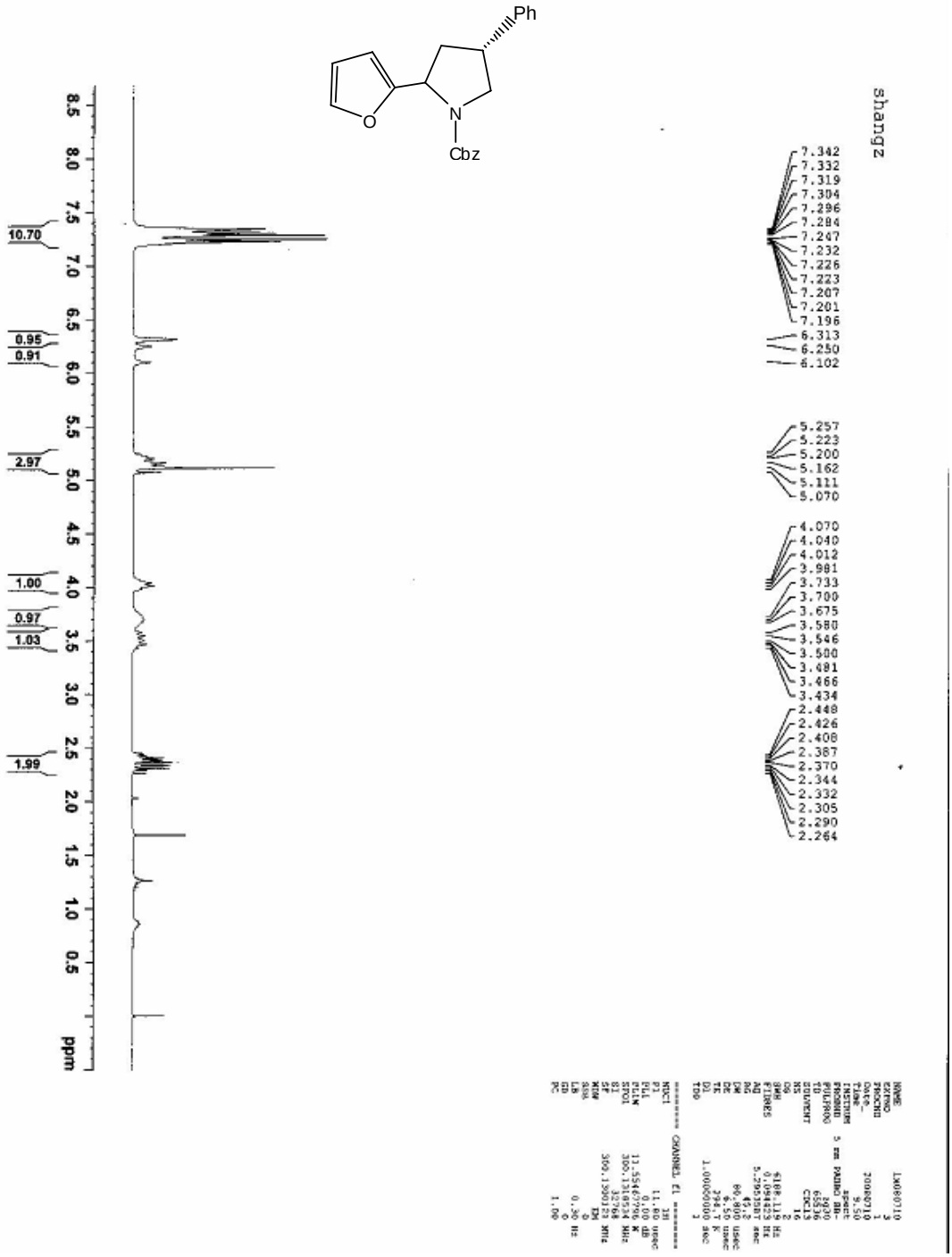

O
OH
Ph
N
Cbz


G5-x
10.481
10.464
7.359
7.346
7.339
7.317
7.292
7.267
7.248
7.159
6.884
5.149
5.129
5.070
5.026
4.975
4.933
3.953
3.929
3.916
3.888
3.195
3.168
3.143
2.599
2.188
2.176
2.166
2.147
2.134
2.123
2.113
0.82
9.09
1.00
3.02
1.96
1.00
0.99
1.00
ppm

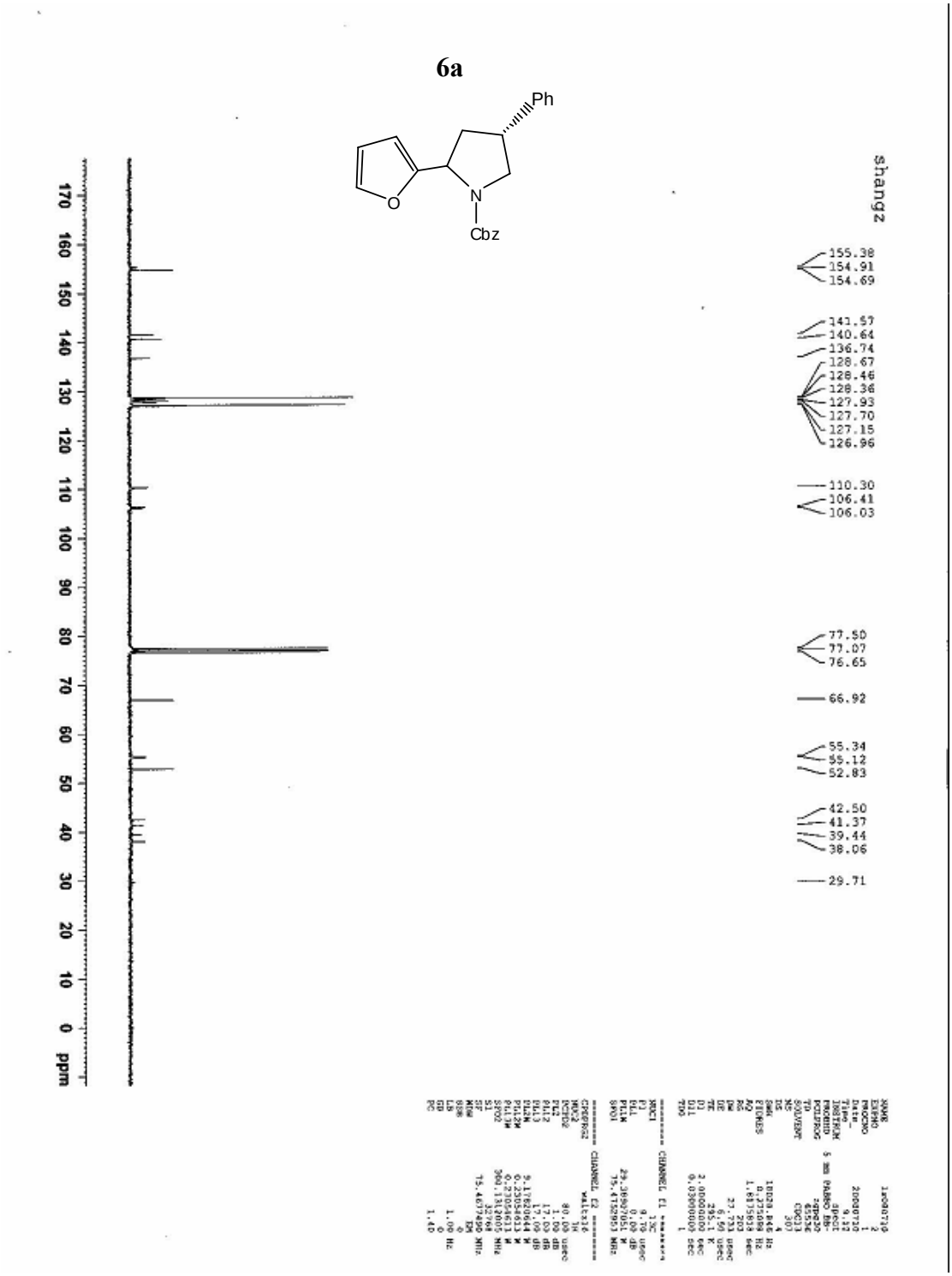
5a


O
OH
Ph
N
Cbz
G5-x
178.15
154.90
142.95
128.63
128.49
128.19
127.94
127.62
127.21
125.31
77.45
77.03
76.60
66.96
60.71
49.44
48.76
41.19
40.54
38.61
37.57
200
180
160
140
120
100
80
60
40
20
0
ppm

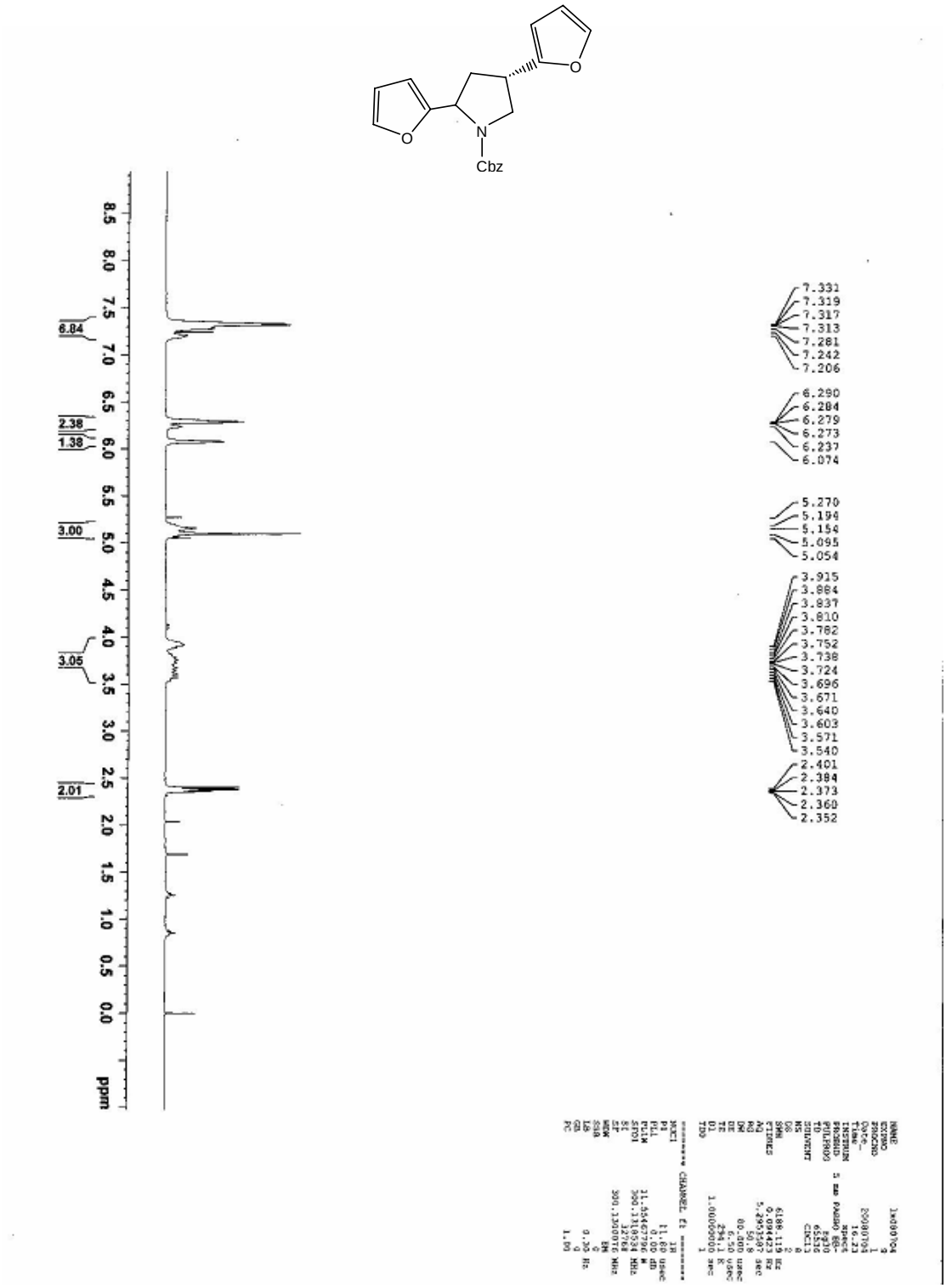
6a


Ph
N
O
Cbz
shangz
7.342
7.332
7.319
7.304
7.296
7.284
7.247
7.232
7.226
7.223
7.207
7.201
7.196
6.313
6.250
6.102
5.257
5.223
5.200
5.162
5.111
5.070
4.070
4.040
4.012
3.981
3.733
3.700
3.675
3.580
3.546
3.500
3.481
3.466
3.434
2.448
2.426
2.408
2.387
2.370
2.344
2.332
2.305
2.290
2.264
10.70
0.95
0.91
2.97
1.00
0.97
1.03
1.99
ppm

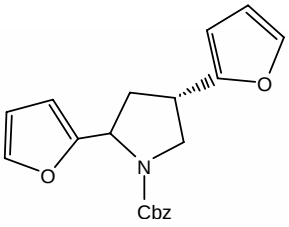
6a

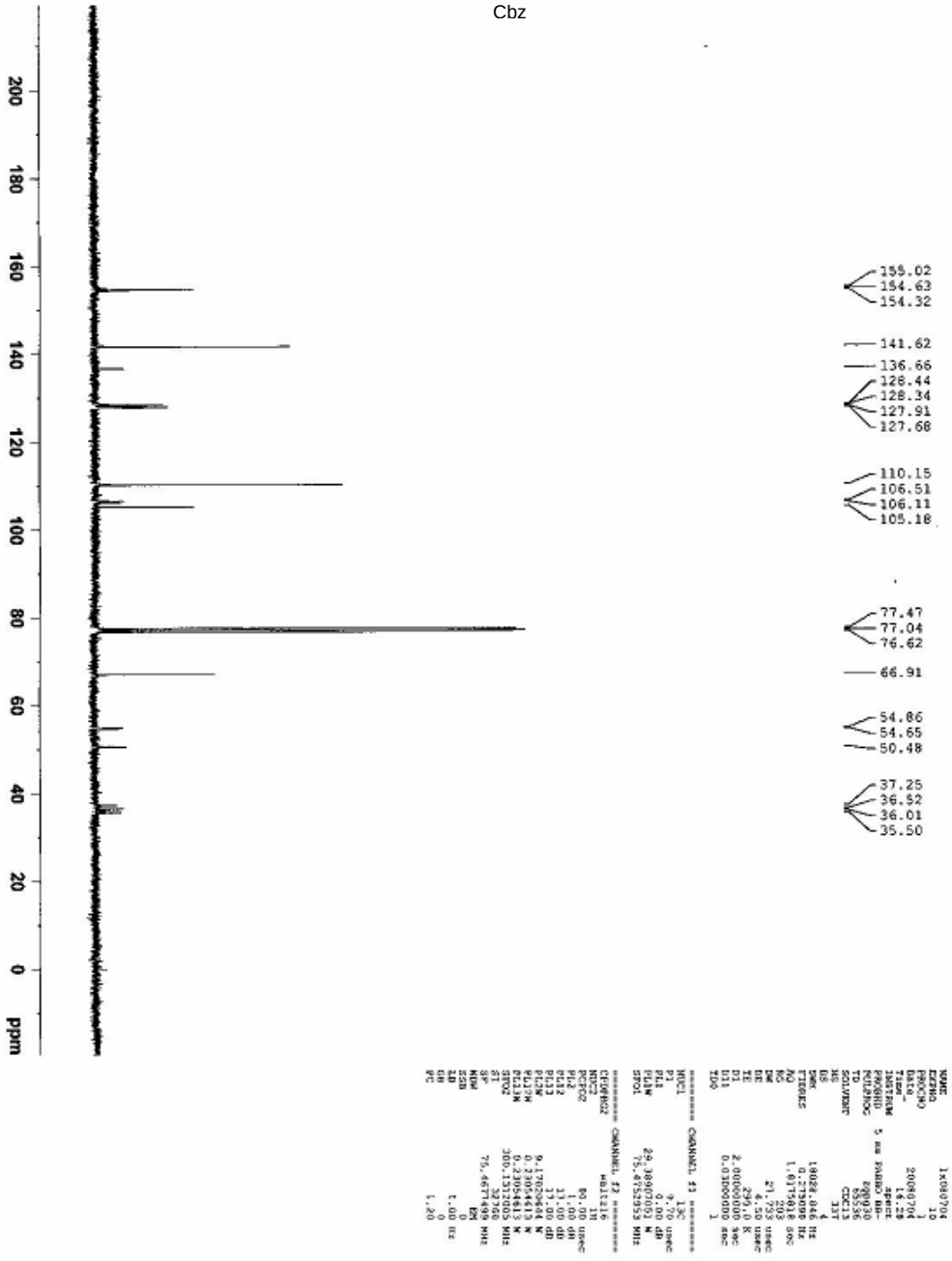

Ph
O
N
Cbz
shangz
155.38
154.91
154.69
141.57
140.64
136.74
128.67
128.46
128.36
127.93
127.70
127.15
126.96
110.30
106.41
106.03
77.50
77.07
76.65
66.92
55.34
55.12
52.83
42.50
41.37
39.44
38.06
29.71
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

7a


O
O
N
Cbz
6.84
2.38
1.38
3.00
3.05
2.01
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 ppm
7.331
7.319
7.317
7.313
7.281
7.242
7.206
6.290
6.284
6.279
6.273
6.237
6.074
5.270
5.194
5.154
5.095
5.054
3.915
3.884
3.837
3.810
3.782
3.752
3.738
3.724
3.696
3.671
3.640
3.603
3.571
3.540
2.401
2.384
2.373
2.360
2.352

7a


O
O
N
Cbz


200
180
160
140
120
100
80
60
40
20
0
ppm
155.02
154.63
154.32
141.62
136.66
128.44
128.34
127.91
127.68
110.15
106.51
106.11
105.18
77.47
77.04
76.62
66.91
54.86
54.65
50.48
37.25
36.52
36.01
35.50