

Highly Enantioselective 1,3-Dipolar Cycloaddition of Azomethine Ylides Catalyzed by Copper(I)/TF-BiphamPhos Complexes

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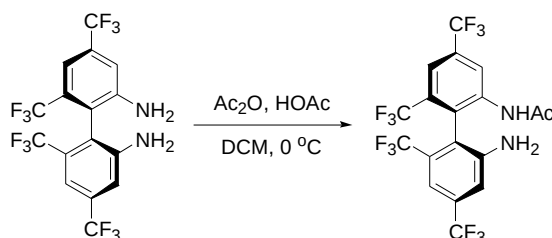
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General Remarks.

^1H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform- d_3 . Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. ^{13}C NMR spectra were recorded on a VARIAN Mercury 75 MHz spectrometer in chloroform- d_3 . Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. ^{31}P NMR spectra were recorded on a VARIAN Mercury 242 MHz spectrometer in chloroform- d_3 , chemical shifts are reported in ppm with the external 85% H_3PO_4 signal at 0.0 ppm as a standard. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Enantiomeric ratios were determined by chiral HPLC using a chiralpak AS-H column, a chiralpak AD-H column, a chiralpak OD-H or a chiralcel OJ-H column with hexane and *i*-PrOH as solvents, or determined by chiral GC using a Supelco chiral Select 1000 column. The absolute configurations of the known products were determined by comparing optical rotation with the reported data. The absolute configurations of the known products **4a-4l** and **4p** were assigned by HPLC and optical rotation comparisons with the reported data,¹⁻³ and those of other adducts were deduced on the basis of these results.

Synthesis of Chiral TF-BiphamPhos 1a-e

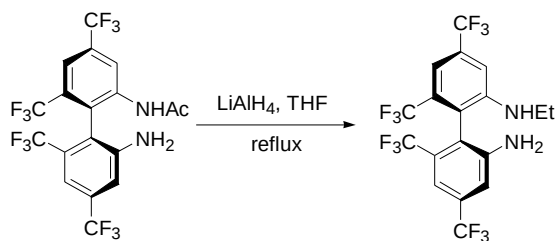
Synthesis of (S)-N-(6'-Amino-4,6,2',4'-tetrakis-trifluoromethyl-biphenyl-2-yl)-acetamide



To a solution of (S)-TF-BIPHAM (456 mg, 1.0 mmol) and AcOH (0.6 mL, 10.0 mmol) in 10 mL of dried CH_2Cl_2 was added acetic anhydride (142 μL , 1.5 mmol) at $0\text{ }^\circ\text{C}$. The resulting solution was stirred for overnight at room temperature, then 2N

NaOH aqueous solution was added until pH >7. The reaction mixture was extracted with CH₂Cl₂ and the combined organic phases were washed with saturated NaHCO₃, brine, and dried over Na₂SO₄. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography. (Ethyl acetate/petroleum ether = 1:2) to afford the product as a white solid (293 mg, 90 % yield based on the recovered starting material). IR (KBr) ν 3584, 3431, 1702, 1530, 1491, 1445, 1370, 1276, 1177, 1129 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.86 (s, 1H), 7.84 (s, 1H), 7.45 (s, 1H), 7.27 (s, 1H), 6.70 (s, 1H), 3.93 (br, 2H), 1.99 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 168.6, 146.4, 138.3, 131.4-133.8 (m), 127.1-129.1 (m), 124.7-125.1 (m), 122.7, 121.1, 116.7, 115.6, 113.0, 24.3; HRMS Calcd. for C₁₈H₁₀F₁₂N₂O: 498.0602, found: 498.0596.

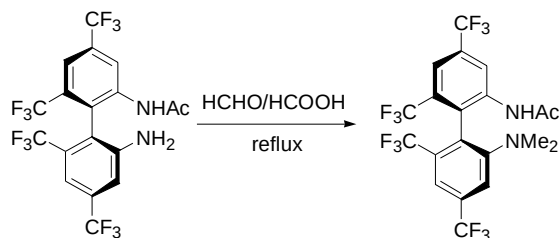
Synthesis of (S)-N-Ethyl-4,6,4',6'-tetrakis-trifluoromethyl-biphenyl-2,2'-diamine



To a stirred suspension of LAH (61 mg, 1.6 mmol) in 5 mL of THF was added dropwise a solution of (S)-N-(6'-Amino-4,6,2',4'-tetrakis-trifluoromethyl-biphenyl-2-yl)-acetamide (270 mg, 0.54 mmol) in 10 mL of THF at 0 °C. After refluxed for 1 h, the mixture was cooled in an ice-bath and the remaining hydride was carefully quenched by dropwise addition of water (10 mL) and then 15% NaOH (5 mL). A white precipitate was filtered off and thoroughly washed with ether. The combined filtrate and ether washings were washed with saturated NaHCO₃, brine and dried over Na₂SO₄. The solvent was removed under reduced pressure, and the residue was purified through flash chromatography to afford the product as yellow oil. (190 mg, 75.2% yield). ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.42 (s, 1H), 7.34 (s, 1H), 7.22 (s, 1H), 7.09 (s, 1H), 3.85 (br, 2H), 3.53 (br, 1H), 3.20 (m, 2H), 1.14 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 147.3, 146.6, 138.5, 131.1-133.2 (m), 124.9-125.4 (m), 121.3-121.8 (m), 118.6, 117.7 (m), 115.3, 113.2, 111.1, 110.1, 38.3,

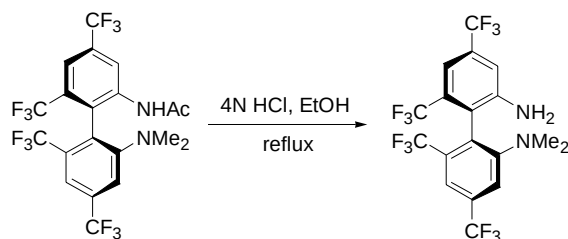
13.9; HRMS Calcd. for C₁₈H₁₂F₁₂N₂: 484.0809, found: 484.0803.

Synthesis of (S)-N-(6'-Dimethylamino-4,6,2',4'-tetrakis-trifluoromethyl-biphenyl-2-yl)-acetamide



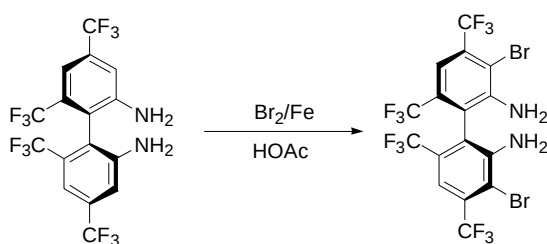
To a three-necked flask containing (S)-N-(6'-Amino-4,6,2',4'-tetrakis-trifluoromethyl-biphenyl-2-yl)-acetamide (300 mg, 0.6 mmol) were added 85% formic acid (0.45 mL, 10.0 mmol) and 37% formaldehyde solution (0.45 mL, 6.0 mmol) under Ar. The flask is connected to a reflux condenser and is placed in an oil bath which has been heated to 100 °C. A vigorous evolution of carbon dioxide begins after 2-3 minutes, at which time the flask is removed from the bath until the gas evolution notably subsides (about 10 minutes), then it is returned to the bath and heated at 100 °C for 24 hour. The reaction mixture was extracted with CH₂Cl₂ and the combined organic phases were washed with saturated NaHCO₃, brine, and dried over Na₂SO₄. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography (ethyl acetate/petroleum ether = 1:5) afford a white solid (278 mg, 88% yield). ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.82 (s, 1H), 7.77 (s, 1H), 7.68 (s, 1H), 7.57(s, 1H), 6.79 (s, 1H), 2.55 (s, 6H), 2.01 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 168.1, 154.6, 138.0, 131.8-133.0 (m), 127.4, 125.0, 121.9, 120.6, 119.5, 116.6, 43.3, 24.4; HRMS Calcd. for C₂₀H₁₄F₁₂NO₂: 526.0915, found: 526.0908.

Synthesis of (S)-N,N'-Dimethyl-4,6,4',6'-tetrakis-trifluoromethyl-biphenyl-2,2'-diamine.



To a solution of (*S*)-*N*-(6'-Dimethylamino-4,6,2',4'-tetrakis-trifluoromethyl-biphenyl-2-yl)-acetamide (220 mg, 0.42 mmol) in 15 ml of EtOH was added 4*N* HCl (6.0 mL), the resulting solution was stirred for overnight at room temperature, the 2*N* NaOH aqueous solution was added until pH > 7. The reaction mixture was extracted by CH₂Cl₂ and the combined organic phases were washed with saturated NaHCO₃, brine, dried over Na₂SO₄. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography (ethyl acetate/petroleum ether = 1:5) to afford a colorless oil. (*S*)-**7** (160 mg, 80% yield). ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.62 (s, 1H), 7.52 (s, 1H), 7.39 (s, 1H), 7.15(s, 1H), 3.85 (br, 2H), 2.61 (s, 6H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 155.1, 146.3, 131.4-132.6 (m), 129.1, 125.0, 122.2, 121.4, 120.4, 116.4, 114.7, 113.4, 43.2; HRMS Calcd. for C₁₈H₁₂F₁₂N₂: 484.0809, found: 484.0807.

Synthesis of (*S*)-3,3'-Dibromo-4,6,4',6'-tetrakis-trifluoromethyl-biphenyl-2,2'-diamine

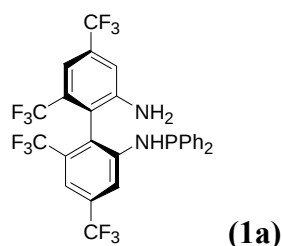


To a round-bottom flask containing (*S*)-TF-BIPHAM (2.28 g, 5.0 mmol) were added 40 mL glacial acetic acid, iron powder (20 mg) and bromine (1.03 mL, 20.0 mmol). The mixture was stirred at room temperature for 4 hour, then 2*N* NaOH aqueous solution was added until pH > 8. The reaction mixture was extracted with CH₂Cl₂, and the combined organic phases were washed with saturated NaHCO₃, brine, and dried over Na₂SO₄. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography (ethyl acetate/petroleum ether =

1:5) to afford a brown solid (2.95 g, 96% yield). $[\alpha]_D^{25} = +126.6$ (c 0.85, CHCl_3); IR (KBr) ν 3504, 1610, 1428, 1378, 1330, 1275, 1198, 1234, 1075, 960, 874 cm^{-1} ; ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.51 (s, 2H), 4.47 (br, 4H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 145.1, 132.5 (q, $J = 32.2$ Hz), 129.8 (q, $J = 34.4$ Hz), 122.6 (q, $J_{\text{C-F}} = 272.7$ Hz), 122.3 (q, $J_{\text{C-F}} = 272.8$ Hz), 120.4, 114.5, 110.4; HRMS Calcd. for $\text{C}_{16}\text{H}_6\text{Br}_2\text{F}_{12}\text{N}_2$: 611.8706, found: 611.8714.

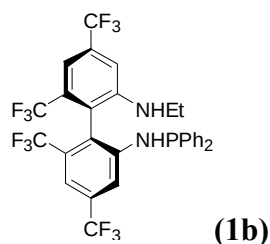
General Procedure for the Synthesis of Chiral TF-BiphamPhos 1a-e

To a solution of corresponding (S)-TF-BIPHAM derivative (1.0 mmol) in CH_2Cl_2 (10 mL) was added DMAP (12.2 mg, 0.1 mmol), Et_3N (1.0 mL, 7.2 mmol) and diphenylphosphine chloride or dicyclohexylphosphine chloride (1.5 mmol) at 0 $^\circ\text{C}$. The reaction mixture was stirred overnight at room temperature. Then the solvent was removed under reduced pressure and the residue was purified by flash alumina chromatography.



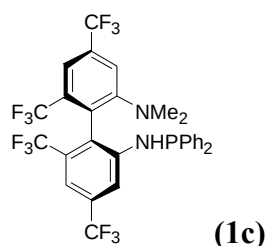
The pure product was obtained as a white solid in 74% yield.

$[\alpha]_D^{25} = +82.2$ (c 0.53, CHCl_3); IR (KBr) ν 3498, 3401, 2925, 1619, 1585, 1508, 1442, 1388, 1276, 1175, 1133, 972, 958, 873, 748, 696, 688 cm^{-1} ; ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.90 (d, $J = 3.6$ Hz, 1H), 7.50 (s, 1H), 7.23-7.34 (m, 11H), 7.10 (s, 1H), 4.21 (d, $J = 6.3$ Hz, 1H), 3.75 (br, 2H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 147.2, 147.0, 146.5, 139.2, 139.0, 138.4, 138.3, 131.8-133.2 (m), 131.4, 130.6-131.4 (m), 129.7, 129.6, 128.8, 128.7, 123.3 (q, $J_{\text{C-F}} = 271.7$ Hz), 123.2 (q, $J_{\text{C-F}} = 271.6$ Hz), 121.9, 121.2, 118.1, 115.8, 115.4, 115.2, 114.2, 113.0; ^{31}P NMR (CDCl_3 , 85% H_3PO_4 , 242.86 MHz) δ 32.17; HRMS Calcd. for $\text{C}_{28}\text{H}_{17}\text{F}_{12}\text{N}_2\text{P}+\text{H}^+$: 641.1011, found: 641.1023.



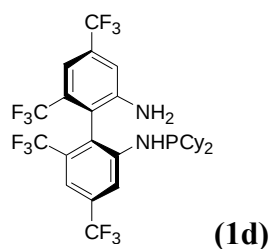
The pure product was obtained as colorless oil in 68% yield.

IR (KBr) ν 3428, 3367, 2977, 1619, 1585, 1521, 1491, 1439, 1390, 1352, 1285, 1177, 1132, 997, 973, 810, 743, 696 cm^{-1} ; ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.94 (s, 1H), 7.49 (s, 1H), 7.16-7.33 (m, 11H), 6.95 (s, 1H), 4.25 (d, J = 6.6 Hz, 1H), 3.40 (s, 1H), 2.92-3.09 (m, 2H), 1.03 (t, J = 6.6, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 147.3, 147.1, 139.1, 139.0, 138.5, 138.4, 128.7-133.4 (m), 124.7-125.2 (m), 121.5, 121.0, 117.9, 115.5, 115.2, 111.1, 111.0, 109.8, 109.7, 38.1, 13.9; ^{31}P NMR (CDCl_3 , 85% H_3PO_4 , 242.86 MHz) δ 31.49; HRMS Calcd. for $\text{C}_{30}\text{H}_{21}\text{F}_{12}\text{N}_2\text{P}+\text{H}^+$: 669.1324, found: 669.1332.



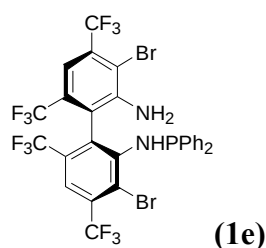
The pure product was obtained as a white solid in 63% yield.

IR (KBr) ν 3483, 3329, 2925, 2853, 1619, 1589, 1503, 1484, 1440, 1384, 1274, 1131, 1000, 982, 878, 726, 684 cm^{-1} ; ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.77 (s, 1H), 7.48 (s, 1H), 7.36 (s, 1H), 7.12-7.29 (m, 11H), 4.84 (d, J = 5.7 Hz, 1H), 2.33 (s, 6H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 154.4, 147.0, 146.8, 138.8-139.3 (m), 128.8-133.0 (m), 125.3, 124.8, 121.8, 121.2, 120.1, 118.1, 116.6, 107.8, 43.5; ^{31}P NMR (CDCl_3 , 85% H_3PO_4 , 242.86 MHz) δ 31.38; HRMS Calcd. for $\text{C}_{30}\text{H}_{21}\text{F}_{12}\text{N}_2\text{P}+\text{H}^+$: 669.1324, found: 669.1332.



The pure product was obtained as a white solid in 48% yield.

IR (KBr) ν 3485, 3370, 2930, 2855, 1618, 1584, 1446, 1389, 1277, 1175, 1133, 999, 974, 958, 869, 730, 684 cm^{-1} ; ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.92 (d, J = 4.2 Hz, 1H), 7.45 (s, 1H), 7.38 (s, 1H), 7.25 (s, 1H), 3.92 (s, 2H), 3.68 (d, J = 9.0 Hz, 1H), 1.46-1.71 (m, 9H), 1.43-1.46 (m, 2H), 0.99-1.28 (m, 9H), 0.75-0.89 (m, 2H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 149.2, 149.0, 146.5, 130.9-133.2 (m), 117.1-128.8 (m), 120.1, 118.6, 36.3, 36.2, 35.6, 29.7, 28.8, 28.6, 28.4, 28.1, 26.6, 26.3, 26.1; ^{31}P NMR (CDCl_3 , 85% H_3PO_4 , 242.86 MHz) δ 32.17.



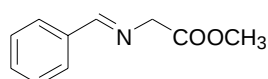
The pure product was obtained as a white solid in 72% yield.

$[\alpha]^{25}_{\text{D}} = +160.8$ (c 0.60, CHCl_3); IR (KBr) ν 3494, 3398, 2928, 1611, 1435, 1378, 1355, 1277, 1195, 1142, 970, 745, 696 cm^{-1} ; ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.70 (s, 1H), 7.23-7.32 (m, 8H), 7.15-7.17 (m, 2H), 7.12 (s, 1H), 4.84 (d, J = 4.5 Hz, 1H), 4.26 (br, 2H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 147.2, 147.0, 145.4, 139.3-139.8 (m), 131.6, 130.5-131.0 (m), 129.8, 129.3, 128.5, 128.4, 126.2, 123.9, 110.3, 119.9, 118.8, 118.3, 113.9, 110.4; ^{31}P NMR (CDCl_3 , 85% H_3PO_4 , 242.86 MHz) δ 41.09; HRMS Calcd. for $\text{C}_{28}\text{H}_{15}\text{Br}_2\text{F}_{12}\text{N}_2\text{P}+\text{H}^+$: 796.9221, found: 796.9214.

General Procedure for the synthesis of α -imino esters.

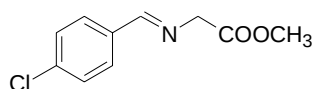
To a suspension of the corresponding amino acid ester hydrochloride (12 mmol) and MgSO_4 (16.0 mmol) in CH_2Cl_2 (20 mL) was added Et_3N (2.0 mL, 14 mmol). The mixture was stirred at room temperature for 1h, and then the corresponding aldehyde

(10.0 mmol) was added. The reaction was stirred at room temperature overnight, and then the resulting precipitate was removed by filtration. The filtrate was washed once with water (30 mL), the aqueous phase was extracted once with CH₂Cl₂ (15 mL) and the combined organic phase was washed with brine 3 times, dried over MgSO₄ and concentrated. The resulting iminoesters were used in 1,3-dipolar cycloadditions without further purification.



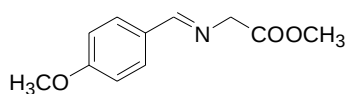
(E)-(Benzylidene-amino)-acetic acid methyl ester:

light yellow oil, yield : 90%; ¹H NMR (CDCl₃, TMS, 400 MHz) δ 8.25 (s, 1H), 7.75-7.77 (m, 2H), 7.37-7.42 (m, 3H), 4.39 (s, 2H), 3.74 (s, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 168.7, 163.6, 133.7, 129.4, 126.8, 126.2, 60.1, 50.3.



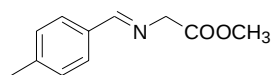
(E)-[(4-Chloro-benzylidene)-amino]-acetic acid methyl ester:

light yellow solid, yield : 85%; ¹H NMR (CDCl₃, TMS, 400 MHz) δ 8.13 (s, 1H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 4.30 (s, 2H), 3.66 (s, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 170.4, 164.0, 137.2, 134.0, 129.7, 128.9, 61.7, 52.1.



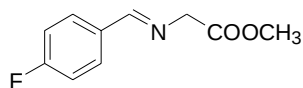
(E)-[(4-Methoxy-benzylidene)-amino]-acetic acid methyl ester:

light yellow solid, yield : 89%; ¹H NMR (CDCl₃, TMS, 400 MHz) δ 8.20 (s, 1H), 7.72 (d, *J* = 8.7 Hz, 2H), 6.92 (d, *J* = 8.7 Hz, 2H), 4.37 (s, 2H), 3.82 (s, 3H), 3.76 (s, 3H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 170.8, 164.7, 162.1, 130.1, 128.5, 114.0, 61.9, 55.3, 52.1.



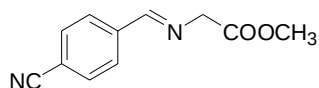
(E)-[(4-Methyl-benzylidene)-amino]-acetic acid methyl ester:

light yellow solid, yield : 92%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.24 (s, 1H), 7.66 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 7.9 Hz, 2H), 4.39 (s, 2H), 3.76 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.7, 165.4, 141.7, 132.9, 129.4, 128.5, 62.0, 52.1, 21.6.



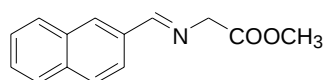
(E)-[(4-Fluorobenzylidene)-amino]-acetic acid methyl ester:

colorless oil, yield : 86%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.25 (s, 1H), 7.76-7.79 (m, 2H), 7.08-7.12 (m, 2H), 4.40 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.5, 163.9, 130.5, 130.4, 116.2, 115.8, 115.6, 61.7, 64.1.



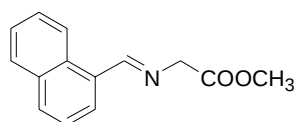
(E)-[(4-Cyanobenzylidene)-amino]-acetic acid methyl ester:

yellow solid, yield : 94%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.35 (s, 1H), 7.90 (d, J = 8.3 Hz, 2H), 7.72 (d, J = 8.2 Hz, 2H), 4.48 (s, 2H), 3.79 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.1, 163.6, 139.2, 132.4, 128.9, 118.4, 114.4, 61.7, 52.3.



(E)-[(Naphthalen-2-ylmethylene)-amino]-acetic acid methyl ester:

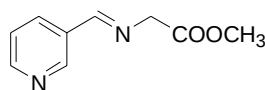
yellow solid, yield : 94%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.36 (s, 1H), 8.00-8.02 (m, 2H), 7.80-7.86 (m, 2H), 7.47-7.51 (m, 2H), 4.44 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.7, 165.5, 134.9, 133.2, 130.0, 128.7, 128.6, 127.9, 127.5, 126.6, 123.8, 62.1, 52.2.



(E)-[(Naphthalen-1-ylmethylene)-amino]-acetic acid methyl ester:

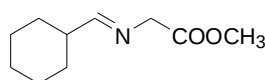
light yellow oil, yield : 87%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.87 (d, J = 8.6 Hz, 1H), 8.83 (s, 1H), 7.81-7.88 (m, 3H), 7.43-7.57 (m, 3H), 4.47 (s, 2H), 3.76 (s, 3H);

^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.7, 165.1, 133.8, 131.7, 131.3, 131.0, 129.4, 128.7, 127.4, 126.2, 125.2, 124.3, 62.7, 52.2.



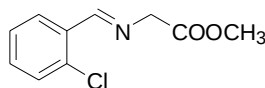
(E)-[(Pyridin-3-ylmethylene)-amino]-acetic acid methyl ester:

light yellow oil, yield : 90%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.90 (s, 1H), 8.66 (d, J = 4.8 Hz, 1H), 8.35 (s, 1H), 8.15-8.18 (d, J = 8.0 Hz, 1H), 7.35-7.38 (m, 1H), 4.46 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.1, 162.6, 151.9, 150.3, 134.6, 131.0, 123.6, 61.7, 52.0.



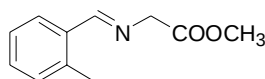
(E)-(Cyclohexylmethylene-amino)-acetic acid methyl ester:

light yellow oil, yield : 83%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.54 (s, 1H), 4.16 (s, 2H), 3.74 (s, 3H), 2.19-2.27 (m, 1H), 1.65-1.88 (m, 5H), 1.18-1.35 (m, 5H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 173.8, 170.7, 61.7, 51.9, 43.5, 29.3, 25.8, 25.2.



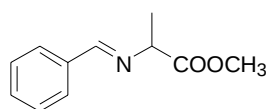
(E)-[(2-Chloro-benzylidene)-amino]-acetic acid methyl ester:

light yellow oil, yield : 90%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.72 (s, 1H), 8.09 (d, J = 7.4 Hz, 1H), 7.29-7.36 (m, 3H), 4.46 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.3, 162.1, 135.4, 132.5, 132.1, 129.8, 128.6, 127.0, 62.0, 52.2.



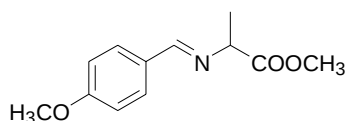
(E)-[(2-Methyl-benzylidene)-amino]-acetic acid methyl ester:

light yellow solid, 95%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.55 (s, 1H), 7.91 (d, J = 7.6 Hz, 1H), 7.26-7.31 (m, 1H), 7.19-7.23 (m, 1H), 7.15 (d, J = 7.5 Hz, 1H), 4.40 (s, 2H), 3.74 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 170.7, 164.0, 138.0, 133.6, 130.8, 127.7, 126.2, 62.4, 52.1, 19.3.



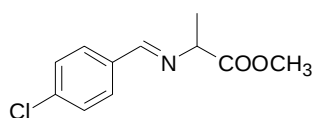
(E)-2-(Benzylidene-amino)-propionic acid methyl ester:

light yellow oil, yield : 87%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.31 (s, 1H), 7.76-7.79 (m, 2H), 7.39-7.44 (m, 3H), 4.16 (q, J = 6.8 Hz, 1H), 3.74 (s, 3H), 1.53 (d, J = 6.9 Hz, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 173.0, 163.0, 131.1, 129.7, 129.0, 128.6, 128.5, 68.0, 52.2, 19.5.



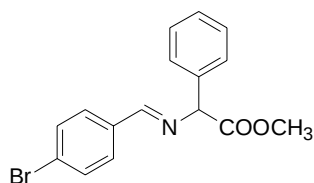
(E)-2-[(4-Methoxy-benzylidene)-amino]-propionic acid methyl ester:

light yellow oil, yield : 87%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.23 (s, 1H), 7.72 (d, J = 8.8 Hz, 2H), 6.92 (d, J = 8.8 Hz, 2H), 4.12 (q, J = 6.8 Hz, 1H), 3.82 (s, 3H), 3.73 (s, 3H), 1.51 (d, J = 6.8 Hz, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 173.2, 162.3, 162.0, 130.1, 129.0, 113.9, 65.0, 55.3, 53.2, 19.6.



(E)-2-[(4-Chloro-benzylidene)-amino]-propionic acid methyl ester:

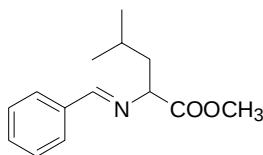
light yellow oil, yield : 81%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.27 (s, 1H), 7.71 (d, J = 8.3 Hz, 2H), 7.36 (d, J = 8.3 Hz, 2H), 4.16 (q, J = 6.8 Hz, 1H), 3.74 (s, 3H), 1.52 (d, J = 6.8 Hz, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 172.8, 161.6, 137.0, 134.1, 130.9, 128.9, 67.9, 52.2, 19.4.



(E)-[(4-Bromo-benzylidene)-amino]-phenyl-acetic acid methyl ester:

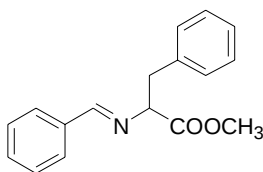
white solid, yield : 81%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.27 (s, 1H), 7.68 (d, J

= 8.4 Hz, 2H), 7.53 (d, J = 8.4 Hz, 2H), 7.49-7.52 (m, 2H), 7.24-7.39 (m, 3H), 5.19 (s, 1H), 3.73 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 171.4, 162.6, 137.9, 134.5, 131.9, 130.1, 128.8, 128.3, 127.9, 125.8, 76.4, 52.6.



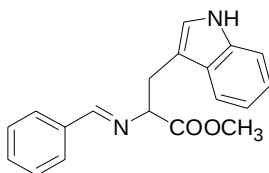
(E)-2-(Benzylidene-amino)-4-methyl-pentanoic acid methyl ester:

light yellow oil, yield : 91%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.28 (s, 1H), 7.77-7.79 (m, 2H), 7.40-7.42 (m, 3H), 7.49-7.52 (m, 2H), 4.09 (t, J = 3.0 Hz, 1H), 3.73 (s, 3H), 1.82-1.88 (m, 2H), 1.55-1.59 (m, 1H), 0.95 (d, J = 6.4 Hz, 3H), 0.90 (d, J = 6.2 Hz, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 172.9, 163.1, 135.6, 131.1, 128.6, 128.5, 71.6, 42.0, 24.4, 23.1, 21.4.



(E)-2-(Benzylidene-amino)-3-phenyl-propionic acid methyl ester:

white solid, yield : 96%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 7.89 (s, 1H), 7.66-7.68 (m, 2H), 7.35-7.40 (m, 3H), 7.14-7.24 (m, 5H), 4.17 (dd, J = 5.0, 5.0 Hz, 1H), 3.72 (s, 3H), 3.37 (dd, J = 5.0, 5.0 Hz, 1H), 3.14 (dd, J = 8.9, 8.9 Hz, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 172.2, 164.0, 137.4, 135.5, 131.2, 129.8, 128.6, 128.5, 128.4, 126.6, 75.1, 52.3, 39.8.

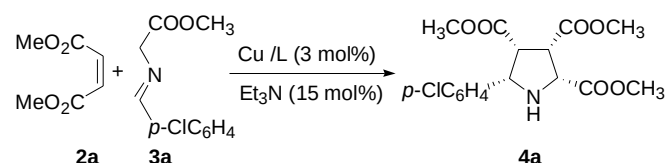


(E)-2-(Benzylidene-amino)-3-(1H-indol-3-yl)-propionic acid methyl ester:

brown oil, yield : 95%; ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.62 (br, 1H), 7.82 (s,

1H), 7.61-7.66 (m, 2H), 7.26-7.36 (m, 4H), 7.09-7.15 (m, 2H), 6.85 (s, 1H), 4.25 (dd, $J = 4.9, 4.9$ Hz, 1H), 3.72 (s, 3H), 3.45 (dd, $J = 4.8, 4.8$ Hz, 1H), 3.22 (dd, $J = 8.6, 8.7$ Hz, 1H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 172.8, 163.7, 136.2, 135.6, 131.1, 128.55, 128.52, 127.3, 123.8, 121.9, 119.3, 118.7, 111.3, 110.9, 73.8, 46.1, 29.7.

Screening Studies of the Asymmetric Cu-Catalyzed 1,3-Dipolar Cycloaddition of Azomethine Ylide **3a** with Dimethyl Maleate **2a**



Entry	Ligand	Cu/L (mol%)	Solvent	Copper ^b	Time (min)	T (°C)	yield (%) ^c	ee (%) ^d
1	1a	3	PhMe	CuClO ₄	10	rt	93	82
2	1a	3	THF	CuClO ₄	60	rt	92	77
3	1a	3	Et ₂ O	CuClO ₄	10	rt	90	79
4	1a	3	CH ₃ CN	CuClO ₄	60	rt	62	70
5	1a	3	DCM	CuClO ₄	10	rt	95	84
6	1a	3	DCM	CuCl	1440	rt	59	85
7	1a	3	DCM	CuI	1440	rt	62	83
8	1a	3	DCM	CuBF ₄	10	rt	97	88
9	1a	3	DCM	Cu(OTf) ₂	1440	rt	11	73
10	1b	3	DCM	CuBF ₄	720	rt	76	57
11	1c	3	DCM	CuBF ₄	720	rt	77	8
12	1d	3	DCM	CuBF ₄	1440	rt	64	28
13	1e	3	DCM	CuBF ₄	10	rt	98	97
14	1e	3	DCM	CuBF ₄	10	0	97	>99
15	1e	1	DCM	CuBF ₄	10	0	97	>99
16	1e	0.5	DCM	CuBF ₄	10	0	96	>99
17	1e	0.1	DCM	CuBF ₄	60	0	85	97

^a All of the reaction was carried out with 0.33 mmol of **2a** and 0.40 mmol of **3a** in 2 mL of solvent. ^b CuClO₄ = Cu(CH₃CN)₄ClO₄; CuBF₄ = Cu(CH₃CN)₄BF₄. ^c Isolated yield. ^d Determined by chiral HPLC analysis.

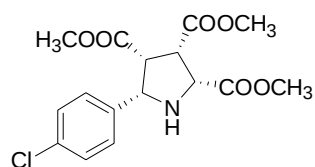
General Procedure for Asymmetric 1,3-Dipolar Cycloaddition of Azomethine Ylides Catalyzed by Cu^I/TF-BiphamPhos **1e** Complex

Under argon atmosphere the solution of ligand (S)-TF-BiphamPhos **1e** (8.0 mg, 0.01 mmol) and Cu(CH₃CN)₄BF₄ (3.1 mg, 0.01 mmol) was stirred at room temperature for about 1h. After it was cooled to the indicated temperature, imine substrate (0.40 mmol) was added as a solution in 0.5 mL CH₂Cl₂ followed by Et₃N (0.06 mmol) and dimethyl maleate (0.33 mmol). Once starting material was consumed

(monitored by TLC), the mixture was filtered through celite and the filtrate was concentrated to dryness. The crude product was analyzed by ^1H NMR to determine the *endo/exo* ratio, and then the residue was purified by column chromatography to give the corresponding cycloaddition product, which was then directly analyzed by chiral HPLC or chiral GC to determine the enantiomeric excess.

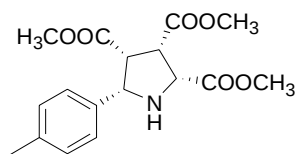
(2R,3S,4R,5S)-trimethyl 5-(4-chlorophenyl)pyrrolidine-2,3,4-tricarboxylate (4a)

(table 2, entry 1)



White solid, yield: 97%; $[\alpha]_D^{25} = -53.5$ (*c* 0.80, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.30 (s, 4H), 4.45 (d, *J* = 5.1 Hz, 1H), 4.15 (d, *J* = 8.1 Hz, 1H), 3.80 (s, 3H), 3.69-3.75 (m, 1H), 3.69 (s, 3H), 3.55-3.60 (m, 1H), 3.28 (s, 3H), 3.06 (br, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.2, 171.0, 136.1, 133.7, 128.7, 128.4, 64.8, 62.3, 52.7, 52.4, 51.7, 51.1; >99% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, t_r = 10.23 and 16.04 min).

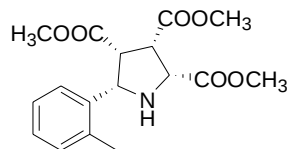
(2R,3S,4R,5S)-trimethyl 5-*p*-tolylpyrrolidine-2,3,4-tricarboxylate (4b) (table 2, entry 2)



White solid, yield: 97%; $[\alpha]_D^{25} = -69.8$ (*c* 0.69, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.22 (d, *J* = 7.5 Hz, 2H), 7.12 (d, *J* = 8.1 Hz, 2H), 4.44 (d, *J* = 6.6 Hz, 1H), 3.80 (s, 3H), 3.69-3.74 (m, 1H), 3.69 (s, 3H), 3.52-3.57 (m, 1H), 3.26 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.4, 171.2, 171.1, 137.6, 134.2, 129.2, 126.8, 65.5, 62.4, 52.7, 52.3, 51.6, 51.3, 21.3; >99% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 1.0 mL/min, t_r = 6.00 and 14.47 min).

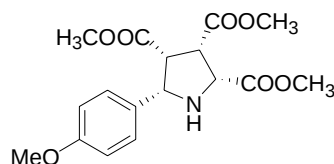
min).

(2R,3S,4R,5S)-trimethyl 5-o-tolylpyrrolidine-2,3,4-tricarboxylate (4c) (table 2, entry 3)



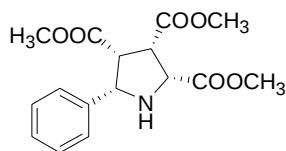
Colorless oil, yield : 98%; $[\alpha]_D^{25} = -74.2$ (c 1.0, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.36-7.39 (m, 1H), 7.14-7.18 (m, 3H), 4.61 (d, $J = 6.6$ Hz, 1H), 4.11 (d, $J = 8.7$ Hz, 1H), 3.83 (s, 3H), 3.71-3.77 (m, 1H), 3.67 (s, 3H), 3.64-3.69 (m, 1H), 3.12 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.5, 171.0, 135.8, 135.1, 130.4, 127.8, 126.2, 62.2, 61.9, 52.6, 52.3, 51.6, 51.4, 51.0; >99% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, $t_r = 7.74$ and 20.69 min).

(2R,3S,4R,5S)-trimethyl 5-(4-methoxyphenyl)pyrrolidine-2,3,4-tricarboxylate (4d) (table 2, entry 4)



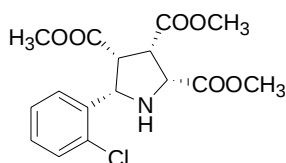
White solid, yield: 98%; $[\alpha]_D^{25} = -68.0$ (c 0.87, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.26 (d, $J = 8.7$ Hz, 2H), 6.85 (d, $J = 8.1$ Hz, 2H), 4.44 (d, $J = 7.2$ Hz, 1H), 4.14 (d, $J = 8.7$ Hz, 1H), 3.80 (s, 3H), 3.78 (s, 3H), 3.68-3.73 (m, 1H), 3.69 (s, 3H), 3.52-3.56 (m, 1H), 3.27 (s, 3H), 3.06 (br, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.3, 171.2, 159.3, 129.3, 128.2, 113.9, 65.1, 65.5, 62.4, 55.4, 52.8, 52.6, 52.3, 51.6, 51.1; >99% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, $t_r = 10.65$ and 21.68 min).

(2R,3S,4R,5S)-trimethyl 5-phenylpyrrolidine-2,3,4-tricarboxylate (4e) (table 2, entry 5)



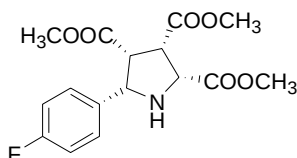
White solid, yield: 93%; $[\alpha]_D^{25} = -68.2$ (c 0.52, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.27-7.34 (m, 5H), 4.49 (d, $J = 6.6$ Hz, 1H), 4.17 (d, $J = 8.7$ Hz, 1H) 3.82 (s, 3H), 3.70-3.75 (m, 1H), 3.70 (s, 3H), 3.55-3.60 (m, 1H), 3.24 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.3, 171.1, 171.0, 137.3, 128.5, 128.0, 126.9, 65.6, 62.4, 52.7, 52.6, 52.3, 51.7, 51.2; >99% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, $t_r = 7.98$ and 17.66 min).

(2R,3S,4R,5S)-trimethyl 5-(2-chlorophenyl)pyrrolidine-2,3,4-tricarboxylate (4f)
(table 2, entry 6)



Colorless oil, yield: 98%; $[\alpha]_D^{25} = -44.1$ (c 0.43, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.48-7.51 (m, 1H), 7.34-7.37 (m, 1H), 7.22-7.28 (m, 2H), 4.75 (d, $J = 6.6$ Hz, 1H), 4.14 (d, $J = 8.7$ Hz, 1H), 3.86-3.91 (m, 1H), 3.83 (s, 3H), 3.75-3.80 (m, 1H), 3.66 (s, 3H), 3.18 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.4, 170.9, 170.7, 134.8, 133.4, 129.4, 129.1, 127.6, 127.0, 62.1, 61.4, 52.6, 52.3, 51.5, 51.0, 50.3; >99% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, $t_r = 9.34$ and 19.99 min).

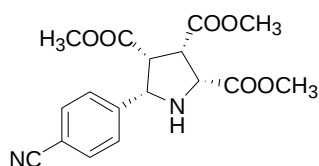
(2R,3S,4R,5S)-trimethyl 5-(4-fluorophenyl)pyrrolidine-2,3,4-tricarboxylate (4g)
(table 2, entry 7)



White solid, yield: 97%; $[\alpha]_D^{25} = -66.0$ (c 0.86, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.31-7.36 (m, 2H), 6.99-7.04 (m, 2H), 4.47 (d, $J = 5.4$ Hz, 1H), 4.15 (d, $J = 9.0$ Hz, 1H), 3.81 (s, 3H), 3.70-3.74 (m, 1H), 3.70 (s, 3H), 3.54-3.59 (m, 1H), 3.27 (s,

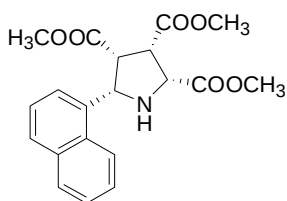
3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.2, 171.1, 164.1, 160.8, 133.3, 128.8, 128.7, 115.6, 115.3, 64.9, 62.4, 52.7, 52.4, 51.7, 51.0; >99% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, t_r = 8.93 and 14.81 min).

(2R,3S,4R,5S)-trimethyl 5-(4-cyanophenyl)pyrrolidine-2,3,4-tricarboxylate (4h)
(table 2, entry 8)



Colorless solid, yield: 96%; $[\alpha]_D^{25} = -62.5$ (c 0.80, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.63 (d, J = 8.1 Hz, 2H), 7.51 (d, J = 7.8 Hz, 2H), 4.54 (d, J = 6.3 Hz, 1H), 4.18 (d, J = 8.7 Hz, 1H), 3.80 (s, 3H), 3.73-3.79 (m, 1H), 3.69 (s, 3H), 3.61-3.67 (m, 1H), 3.27 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.0, 170.8, 170.6, 143.3, 132.2, 128.0, 118.8, 111.7, 64.8, 62.2, 52.7, 52.5, 52.2, 51.8, 51.0; >99% ee, HPLC (Chiralpak AD-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, t_r = 25.71 min).

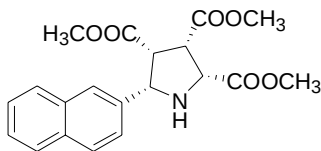
(2R,3S,4R,5S)-trimethyl 5-(1-naphthyl)pyrrolidine-2,3,4-tricarboxylate (4i) (table 2, entry 9)



White solid, yield: 98%; $[\alpha]_D^{25} = -168.0$ (c 0.84, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.93 (d, J = 8.1 Hz, 1H), 7.85 (d, J = 7.2 Hz, 1H), 7.76 (d, J = 8.1 Hz, 1H), 7.61 (d, J = 7.2 Hz, 1H), 7.41-7.51 (m, 3H), 5.17 (d, J = 3.6 Hz, 1H), 4.22 (d, J = 5.1 Hz, 1H), 3.85-3.87 (m, 2H), 3.85 (s, 3H), 3.38 (br, 1H), 2.93 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.6, 171.0, 170.8, 133.7, 132.6, 131.2, 129.2, 128.6, 126.5, 125.8, 125.4, 123.4, 122.8, 61.5, 52.7, 52.3, 52.2, 51.3; >99% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, t_r =

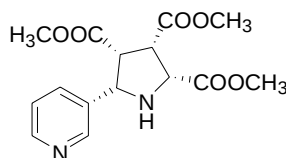
13.73 and 31.14 min).

(2R,3S,4R,5S)-trimethyl 5-(2-naphthyl)pyrrolidine-2,3,4-tricarboxylate (4j) (table 2, entry 10)



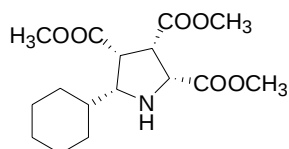
White solid, yield:98%; $[\alpha]_D^{25} = -66.6$ (c 0.62, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.78-7.83 (m, 4H), 7.41-7.48 (m, 3H), 7.76 (d, $J = 8.1$ Hz, 1H), 4.62 (d, $J = 6.6$ Hz, 1H), 4.22 (d, $J = 9.0$ Hz, 1H), 3.83 (s, 3H), 3.75-3.81 (m, 1H), 3.70 (s, 3H), 3.65-3.70 (m, 1H), 3.15 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.4, 171.2, 171.0, 134.7, 133.4, 128.3, 128.2, 127.8, 126.4, 126.3, 125.7, 125.0, 65.7, 62.3, 52.7, 52.4, 51.9, 51.4; >99% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, $t_r = 10.51$ and 25.52 min).

(2R,3S,4R,5S)-trimethyl 5-(pyridin-3-yl)pyrrolidine-2,3,4-tricarboxylate (4k) (table 2, entry 11)



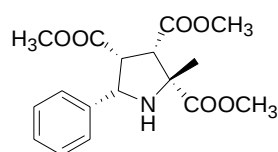
Yellow solid, yield:85%; $[\alpha]_D^{25} = -52.1$ (c 0.68, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 8.59 (s, 1H), 8.51 (d, $J = 3.6$ Hz, 3H), 7.78 (d, $J = 8.1$ Hz, 1H), 7.26-7.30 (m, 1H), 4.53 (d, $J = 7.2$ Hz, 1H), 4.18 (d, $J = 8.7$ Hz, 1H), 3.80 (s, 3H), 3.73-3.79 (m, 1H), 3.62-3.67 (m, 1H), 3.27 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 171.0, 170.9, 171.8, 149.2, 149.0, 134.5, 133.4, 62.9, 52.6, 52.3, 51.7, 50.9; 98% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 205 nm, 0.8 mL/min, $t_r = 11.11$ and 12.99 min).

(2R,3S,4R,5R)-trimethyl 5-cyclohexylpyrrolidine-2,3,4-tricarboxylate (4l) (table 2, entry 12)



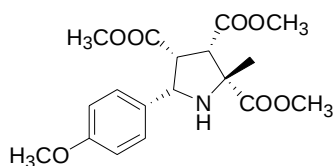
White solid, yield:41%; $[\alpha]_D^{25} = -3.2$ (c 0.64, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 4.07 (d, $J = 9.3$ Hz, 2H), 3.76 (s, 3H), 3.69 (s, 3H), 3.67 (s, 3H), 3.55 (dd, $J = 10.5, 7.2$ Hz, 1H), 3.19 (m, 1H), 2.81 (m, 2H), 2.07 (m, 1H), 1.68-1.80 (m, 4H), 0.95-1.39 (m, 6H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 172.3, 171.4, 170.8, 68.9, 61.5, 52.5, 52.2, 52.0, 51.7, 49.0, 39.3, 31.7, 31.1, 26.4, 26.0, 25.8; 97% ee, Chiral GC: Chiral select 1000, 30 m x 0.25 mm, column temperature: 200 °C, carrier gas: N_2 , 1 mL/min, $t_r = 47.22$ and 48.70 min.

(2R,3S,4R,5S)-trimethyl 2-methyl-5-phenylpyrrolidine-2,3,4-tricarboxylate (4m)
(table 3, entry 1)



Colorless oil, yield: 95%; $[\alpha]_D^{25} = -95.7$ (c 1.39, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.25-7.35 (m, 5H), 4.61 (d, $J = 6.0$ Hz, 1H), 3.80 (s, 3H), 3.75 (s, 3H), 3.67 (d, $J = 10.2$ Hz, 1H), 3.46 (m, 1H), 3.27 (s, 3H), 1.69 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 174.1, 170.8, 137.1, 128.0, 127.4, 126.4, 68.2, 63.3, 57.5, 52.7, 52.3, 51.7, 50.9, 28.1; >99% ee, HPLC (Chiralpak OD-H column, *i*-PrOH/hexane 15/85, detector: 210 nm, 0.8 mL/min, $t_r = 20.56$ and 23.68 min). HRMS Calcd. for $\text{C}_{17}\text{H}_{21}\text{NO}_6$: 335.1369, found: 335.1368.

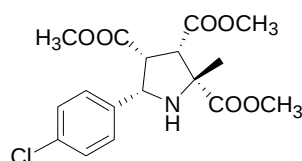
(2R,3S,4R,5S)-trimethyl 5-(4-methoxyphenyl)-2-methylpyrrolidine-2,3,4-tricarboxylate (4n) (table 3, entry 2)



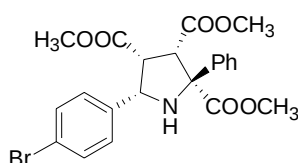
Colorless oil, yield:72%; $[\alpha]_D^{25} = -17.9$ (c 0.68, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.26 (d, $J = 8.7$ Hz, 2H), 6.83 (d, $J = 8.7$ Hz, 2H), 4.55 (d, $J = 5.7$ Hz, 1H),

3.78 (s, 3H), 3.76 (s, 3H), 3.74 (s, 3H), 3.42 (m, 1H), 3.29 (s, 3H), 3.27 (d, $J = 9.6$ Hz, 1H), 1.66 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 174.2, 170.9, 158.8, 129.1, 127.7, 113.4, 68.3, 62.9, 57.4, 54.9, 52.8, 52.3, 51.7, 51.0, 28.1; >99% ee, HPLC (Chiralpak OD-H column, *i*-PrOH/hexane 15/85, detector: 210 nm, 0.8 mL/min, t_r = 15.30 min). HRMS Calcd. for $\text{C}_{18}\text{H}_{23}\text{NO}_7$: 365.1475, found: 365.1473.

(2R,3S,4R,5S)-trimethyl 5-(4-chlorophenyl)-2-methylpyrrolidine-2,3,4- tricarboxylate (4o) (table 3, entry 3)



colorless solid, 92% yield; $[\alpha]_D^{25} = -30.4$ (c 0.30, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.29 (m, 4H), 4.58 (d, $J = 6.0$ Hz, 1H), 3.79 (s, 3H), 3.75 (s, 3H), 3.46 (m, 1H), 3.30 (s, 3H), 3.26-3.30 (m, 1H), 1.67 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 174.1, 170.8, 170.7, 136.0, 133.3, 128.3, 128.2, 128.1, 128.0, 68.4, 62.8, 57.6, 52.6, 52.5, 51.9, 51.2, 28.1; >99% ee, HPLC (Chiralpak OD-H column, *i*-PrOH/hexane 15/85, detector: 210 nm, 0.8 mL/min, t_r = 17.46 min). HRMS Calcd. for $\text{C}_{17}\text{H}_{20}\text{ClNO}_6$: 369.0979, found: 369.0982.

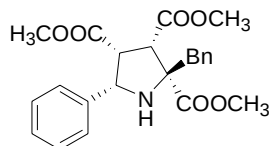


(2S,3S,4R,5S)-trimethyl 5-(4-bromophenyl)-2-phenylpyrrolidine-2,3,4- tricarboxylate (4p) (table 3, entry 4)

Colorless solid, yield: 87%; $[\alpha]_D^{25} = -17.8$ (c 0.57, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.83 (d, $J = 8.1$ Hz, 2H), 7.31-7.47 (m, $J = 8.1$ Hz, 7H), 4.46 (br, 1H), 3.91 (d, $J = 8.1$ Hz, 1H), 3.82 (s, 3H), 3.70 (s, 3H), 3.46 (dd, $J = 8.1$, $J = 8.7$ Hz, 1H), 3.20 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 173.0, 171.3, 170.5, 139.2, 136.9, 131.3, 129.4, 128.6, 128.2, 126.4, 121.7, 75.9, 63.1, 57.0, 53.2, 52.8, 52.3, 51.4; >99% ee, HPLC (Chiralpak OJ-H column, *i*-PrOH/hexane 30/70, detector: 210 nm,

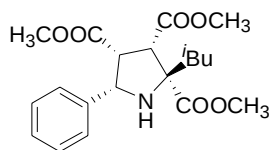
0.8 mL/min, $t_r = 15.30$ min). HRMS Calcd. for $C_{22}H_{22}BrNO_6$: 475.0630, found: 475.0634.

(2R,3S,4R,5S)-trimethyl 2-benzyl-5-phenylpyrrolidine-2,3,4-tricarboxylate (4q)
(table 3, entry 5)



Colorless oil, yield: 90%; $[\alpha]_D^{25} = -43.0$ (c 0.49, $CHCl_3$); 1H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.32-7.46 (m, 2H), 7.23-7.30 (m, 2H), 4.06 (d, $J = 5.7$ Hz, 1H), 3.83 (s, 3H), 3.76 (s, 3H), 3.23-3.33 (m, 1H), 3.23 (s, 3H), 3.17-3.21 (m, 1H), 3.20 (s, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 173.98, 171.38, 170.75, 137.42, 135.90, 131.17, 131.00, 130.97, 130.92, 128.48, 128.43, 128.31, 128.25, 128.20, 128.17, 128.14, 127.97, 127.77, 127.60, 126.94, 126.88, 126.74, 126.48, 126.45, 126.42, 126.36, 72.39, 63.74, 57.42, 54.09, 53.41, 52.52, 52.40, 52.15, 52.06, 51.09, 44.85; 99% ee, HPLC (Chiralpak OD-H column, *i*-PrOH/hexane 15/85, detector: 210 nm, 0.8 mL/min, $t_r = 9.16$ min and 13.38 min). HRMS Calcd. for $C_{23}H_{25}NO_6$: 411.1682, found: 411.1681.

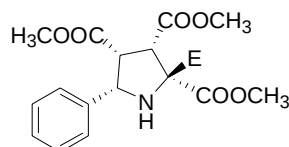
(2R,3S,4R,5S)-trimethyl 2-isobutyl-5-phenylpyrrolidine-2,3,4-tricarboxylate (4r)
(table 3, entry 6)



Colorless oil, yield: 89%; $[\alpha]_D^{25} = +23.0$ (c 0.13, $CHCl_3$); 1H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.25-7.37 (m, 5H), 4.52 (d, $J = 6.0$ Hz, 2H), 3.77 (s, 3H), 3.74 (s, 3H), 3.68-3.72 (m, 1H), 3.43 (m, 1H), 3.25 (s, 3H), 2.07-2.13 (dd, $J = 5.7$, $J = 6.0$ Hz, 1H), 1.77-1.81 (m, 1H), 1.60-1.67 (m, 1H), 1.02 (d, $J = 6.6$ Hz, 3H), 0.88 (d, $J = 6.6$ Hz, 2H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 173.8, 171.3, 171.0, 137.6, 128.3, 127.6, 126.8, 72.4, 64.0, 58.2, 52.8, 52.3, 52.0, 51.1, 48.9, 25.3, 24.3, 23.5; 98% ee, HPLC

(Chiralpak OD-H column, *i*-PrOH/hexane 15/85, detector: 210 nm, 0.8 mL/min, t_r = 7.73 min and 11.93 min). HRMS Calcd. for $C_{20}H_{27}NO_6$: 377.1838, found: 377.1839.

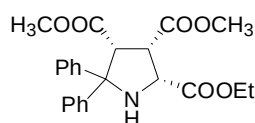
(2R,3S,4R,5S)-trimethyl 2-((1H-indol-3-yl)methyl)-5-phenylpyrrolidine-2,3,4-tricarboxylate (4s) (table 3, entry 7)



E = 3-indolylmethyl

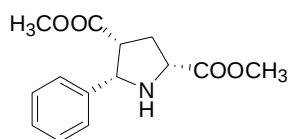
white solid, yield: 80%; $[\alpha]_D^{25} = -60.0$ (*c* 0.11, $CHCl_3$); 1H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.17 (s, 1H), 7.87 (d, $J = 8.1$ Hz, 1H), 7.12-7.38 (m, 9H), 4.08 (d, $J = 5.1$ Hz, 1H), 3.86 (s, 3H), 3.77 (s, 3H), 3.55 (br, 1H), 3.44 (d, $J = 6.6$ Hz, 1H), 3.43 (dd, $J = 14.1, 11.7$ Hz, 2H), 3.29 (s, 3H), 3.16 (dd, $J = 6.0, 5.7$ Hz, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 174.6, 171.7, 171.1, 137.7, 135.8, 128.5, 128.2, 127.5, 126.4, 125.0, 121.8, 119.5, 111.0, 109.7, 72.5, 63.8, 54.1, 52.7, 52.3, 52.1, 51.1, 35.1; 97% ee, (Chiralpak OD-H column, *i*-PrOH/hexane 15/85, detector: 210 nm, 0.8 mL/min, t_r = 25.79 min and 41.75 min). HRMS Calcd. for $C_{23}H_{25}NO_6$: 450.1791, found: 450.1792.

(2R,3S,4R)-2-ethyl 3,4-dimethyl 5,5-diphenylpyrrolidine-2,3,4-tricarboxylate (4t) (table 3, entry 8)



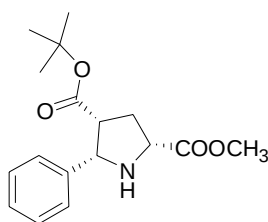
Colorless oil, yield: 75%; $[\alpha]_D^{25} = +7.0$ (*c* 0.05, $CHCl_3$); 1H NMR ($CDCl_3$, TMS, 300 MHz) δ 7.60 (d, $J = 6.9$ Hz, 2H), 7.15-7.45 (m, 8H), 4.30 (d, $J = 3.3$ Hz, 1H), 4.12-4.26 (m, 2H), 3.72 (s, 3H), 3.63 (s, 3H), 3.34-3.54 (m, 1H), 2.81-2.90 (m, 1H), 2.52-2.59 (m, 1H), 1.26 (t, $J = 7.8$ Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 172.3, 172.1, 170.1, 138.9, 135.8, 130.6, 129.0, 128.9, 128.5, 128.0, 127.5, 65.6, 61.3, 52.0, 51.7, 44.8, 32.5, 14.1; 97% ee, (Chiralpak OD-H column, *i*-PrOH/hexane 15/85, detector: 210 nm, 0.8 mL/min, t_r = 8.95 min and 10.35 min).

(2R,4R,5S)-dimethyl 5-phenylpyrrolidine-2,4-dicarboxylate (4u) (table 3, entry 9)



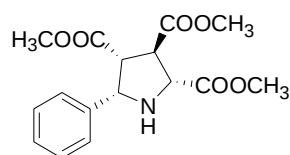
white solid, yield: 90%; $[\alpha]_D^{25} = -9.0$ (*c* 0.57, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.26-7.33 (m, 5H), 4.55 (d, *J* = 7.5 Hz, 1H), 4.00 (t, *J* = 6.3 Hz, 1H), 3.83 (s, 3H), 3.32 (q, *J* = 7.2 Hz, 1H), 3.22 (s, 3H), 2.43 (dd, *J* = 7.2 and 7.5 Hz, 2H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 173.5, 172.8, 138.7, 128.0, 127.4, 126.5, 65.5, 59.6, 52.0, 51.0, 49.4, 33.0; 97% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 210 nm, 1.0 mL/min, *t*_r = 12.93 and 20.83 min).

(2R,4R,5S)-4-butyl 2-methyl 5-phenylpyrrolidine-2,4-dicarboxylate (4v) (table 3, entry 10)



Colorless solid, yield: 94%, $[\alpha]_D^{25} = -45.6$ (*c* 0.44, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.23-7.37 (m, 5H), 4.47 (d, *J* = 8.1 Hz, 1H), 3.95 (t, *J* = 8.1 Hz, 1H), 3.81 (s, 3H), 3.26 (q, *J* = 7.2 Hz, 1H), 2.31-2.45 (m, 2H), 1.02 (s, 9H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 173.7, 171.8, 139.2, 128.1, 127.3, 127.2, 80.6, 65.6, 59.9, 52.2, 50.2, 34.2, 27.5; 97% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 210 nm, 1.0 mL/min, *t*_r = 7.12 and 10.76 min).

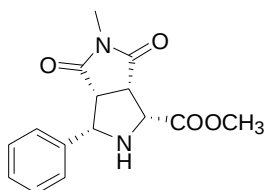
(2R,3R,4R,5S)-trimethyl 5-phenylpyrrolidine-2,3,4-tricarboxylate (4w)



yellow oil, yield: 95%; $[\alpha]_D^{25} = -31.9$ (*c* 0.38, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.26-7.31 (m, 5H), 4.67 (d, *J* = 8.1 Hz, 1H), 4.22 (d, *J* = 7.2 Hz, 1H), 3.85 (s,

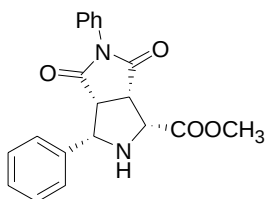
3H), 3.78 (s, 3H), 3.55-3.76 (m, 2H), 3.21 (s, 3H), 2.66 (br, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 172.5, 171.9, 171.5, 137.8, 128.2, 127.8, 126.7, 65.2, 63.1, 53.6, 52.5, 51.5, 50.5; d.r. = 86:14, 90% ee, HPLC (Chiralpak OD-H column, *i*-PrOH/hexane 30/70, detector: 210 nm, 0.8 mL/min, t_r = 18.33 and 28.73 min).

(1R,3S,3aR,6aS)-methyl 5-methyl-4,6-dioxo-3-phenyl-octahydro-pyrrolo[3,4-c]pyrrole-1-carboxylate (4x)



white solid, yield: 85%; $[\alpha]_D^{25} = -34.3$ (*c* 0.80, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.34-7.35 (m, 5H), 4.50 (d, J = 8.7 Hz, 1H), 4.06 (d, J = 6.6 Hz, 1H), 3.89 (s, 3H), 3.59 (dd, J = 6.9, 6.9 Hz, 1H), 3.44 (dd, J = 7.8, 7.8 Hz, 1H), 2.88 (s, 3H), 2.43 (br, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 176.0, 174.7, 170.2, 136.6, 128.4, 128.3, 127.0, 64.0, 61.6, 52.3, 49.5, 48.2, 25.0; 86% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 220 nm, 1.5 mL/min, t_r = 7.15 and 18.50 min).

(1R,3S,3aR,6aS)-methyl 4,6-dioxo-3,5-diphenyl-octahydro-pyrrolo[3,4-c]pyrrole-1-carboxylate (4y)

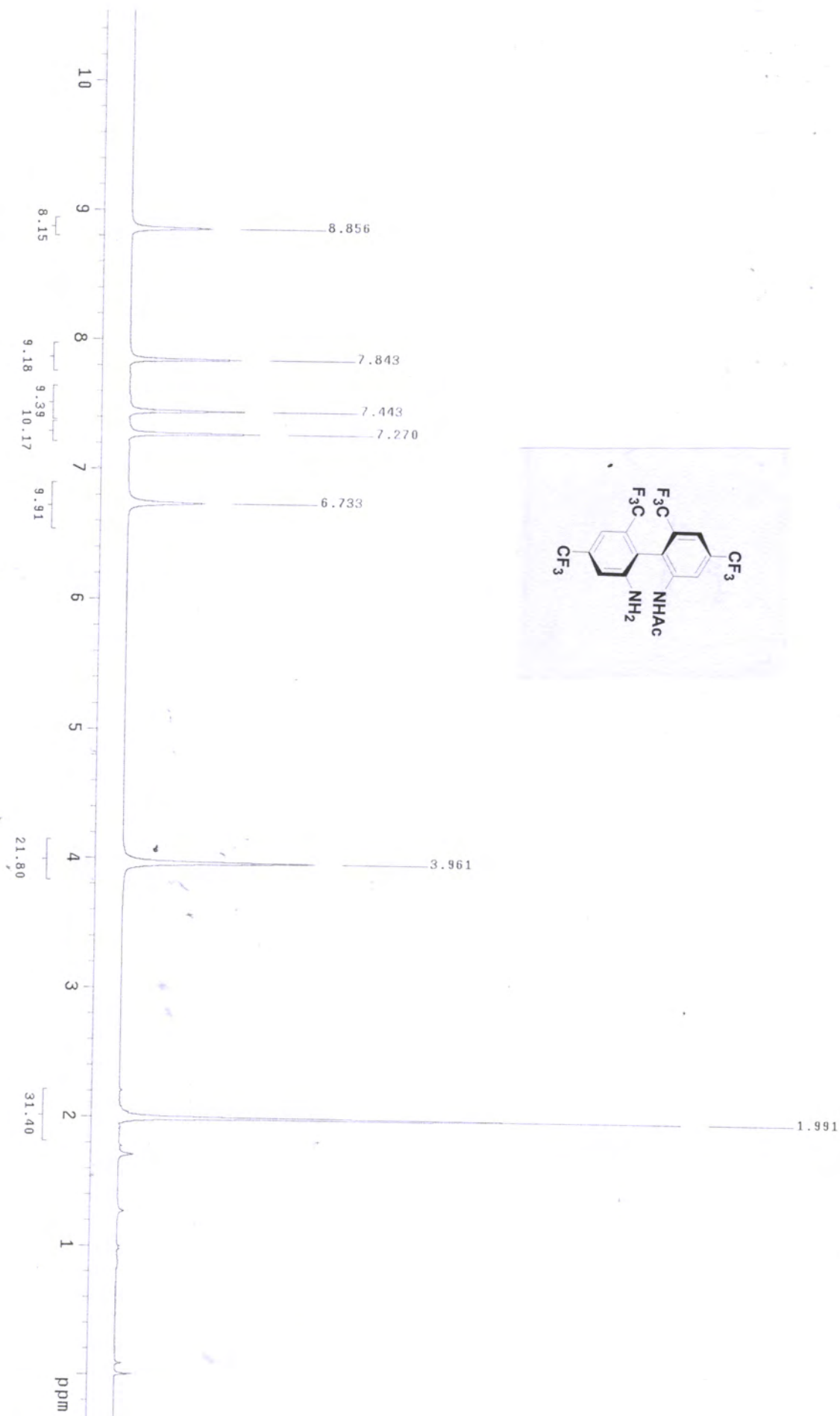
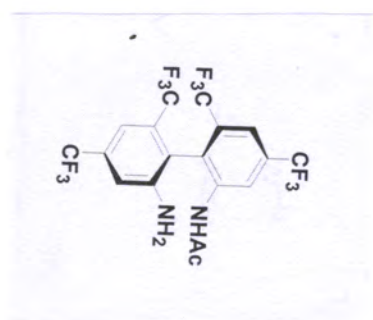


white solid, yield: 95%; $[\alpha]_D^{25} = -59.5$ (*c* 0.72, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.10-7.26 (m, 8H), 6.90-7.12 (m, 2H), 4.36 (d, J = 8.7 Hz, 1H), 3.93 (d, J = 6.6 Hz, 1H), 3.66 (s, 3H), 3.48 (dd, J = 7.2, 7.5 Hz, 1H), 3.31 (dd, J = 8.7, 8.7 Hz, 1H), 2.30 (br, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 175.2, 173.7, 170.2, 136.8, 131.7, 129.1, 128.5, 127.2, 126.2, 64.2, 61.9, 52.4, 49.4, 48.4; 88% ee, HPLC (Chiralpak AS-H column, *i*-PrOH/hexane 50/50, detector: 220 nm, 1.5 mL/min, t_r =

8.66 and 20.64 min).

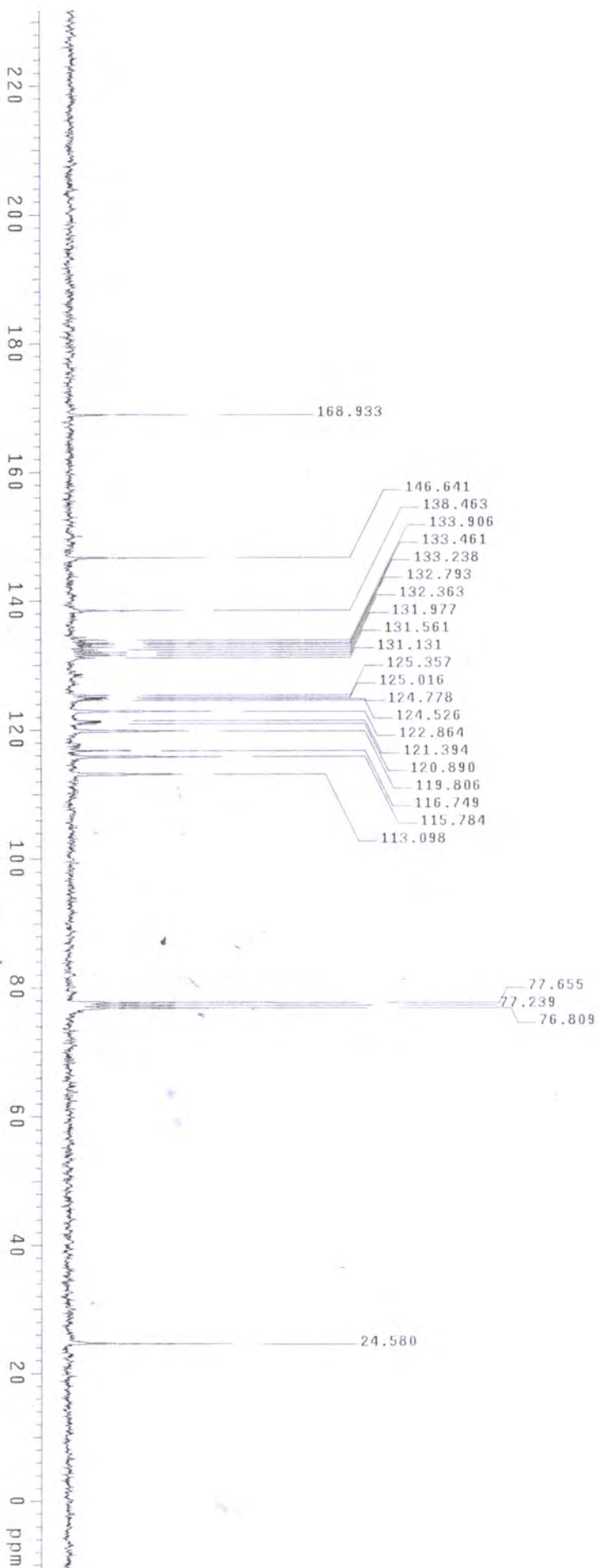
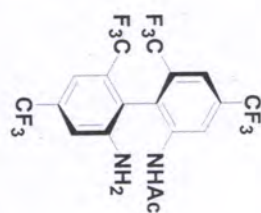
Reference

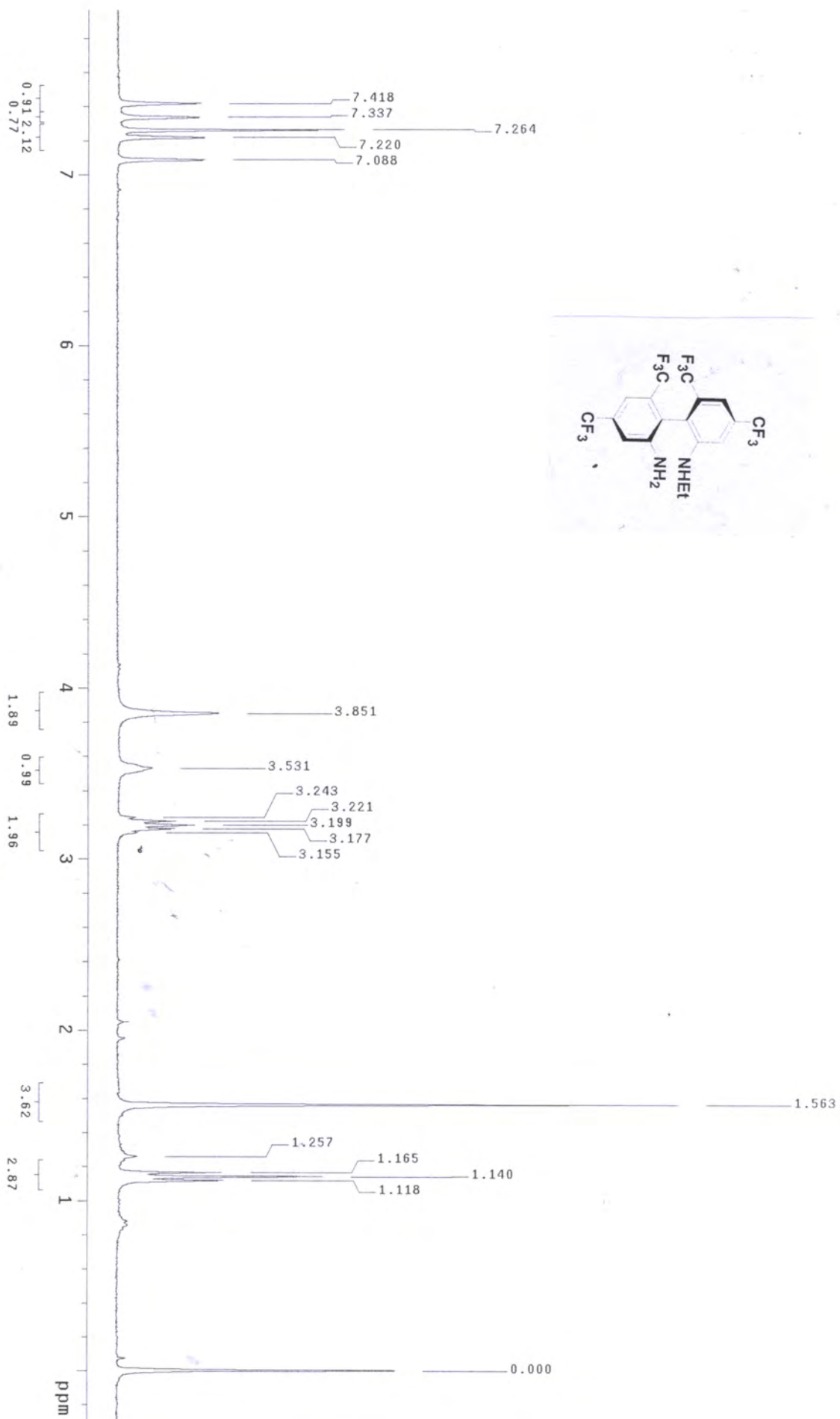
1. Longmire, J. M.; Wang, B.; Zhang, X. *J. Am. Chem. Soc.* **2002**, *124*, 13400.
2. Zeng, W.; Zhou, Y.-G. *Org. Lett.* **2005**, *7*, 5055.
3. Chen, X.-H.; Zhang, W.-Q.; Gong, L.-Z. *J. Am. Chem. Soc.* **2008**, *130*, 5652.



Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

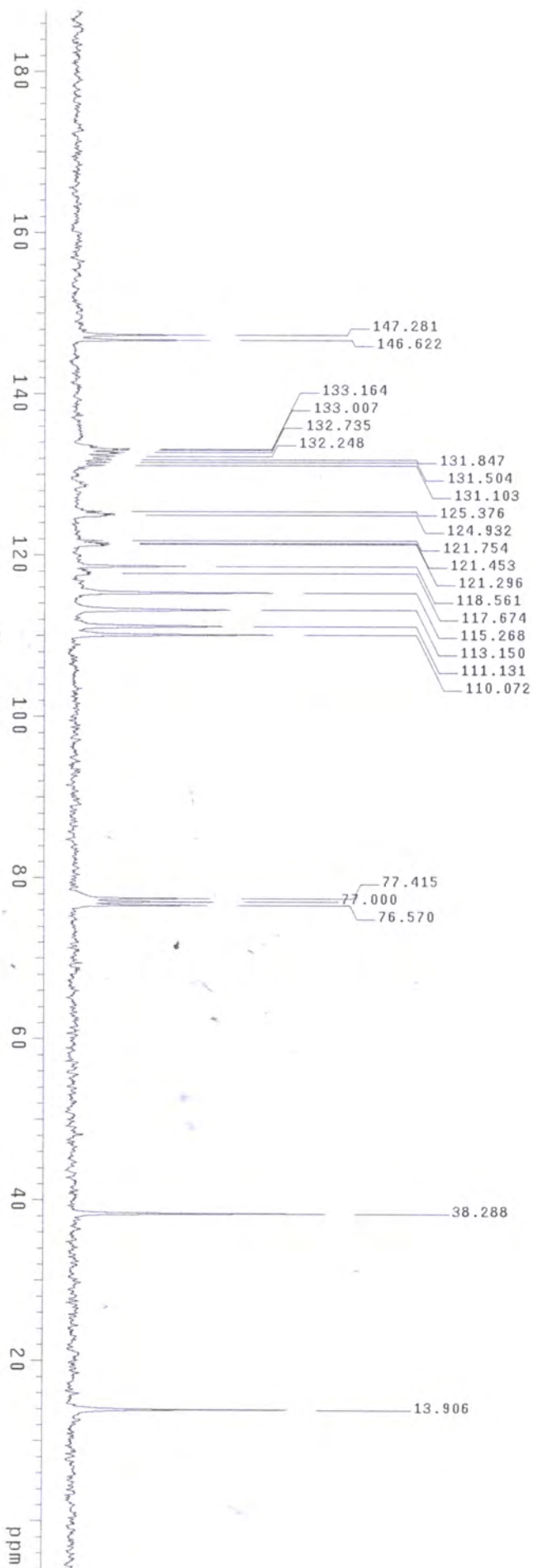
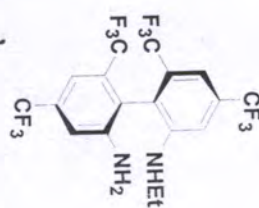
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 18348.6 Hz
336 repetitions
OBSERVE C13, 75.4552576 MHz
DECUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 4.0 Hz
FT size 32768
Total time 3 hr, 40 min, 16 sec

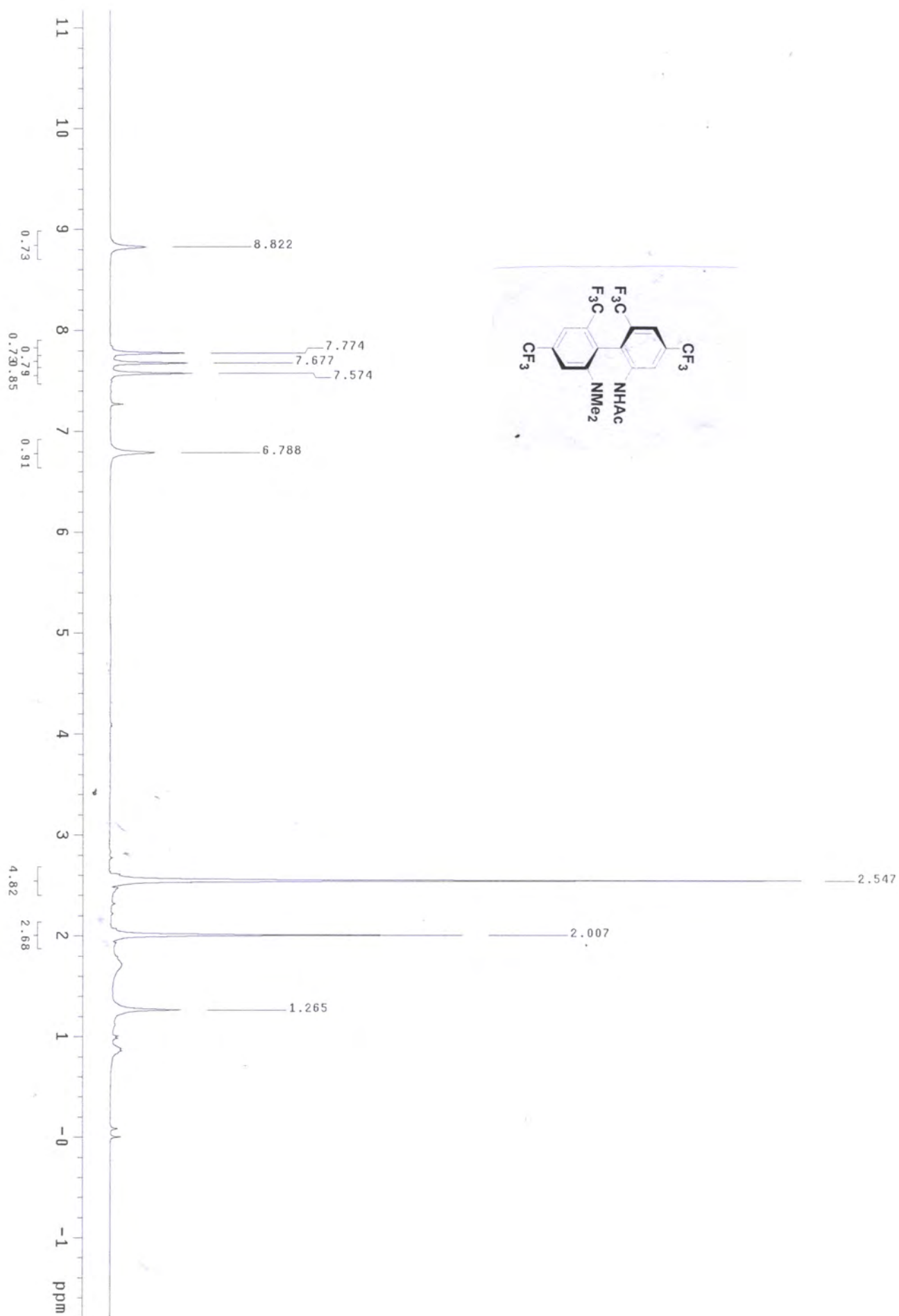




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

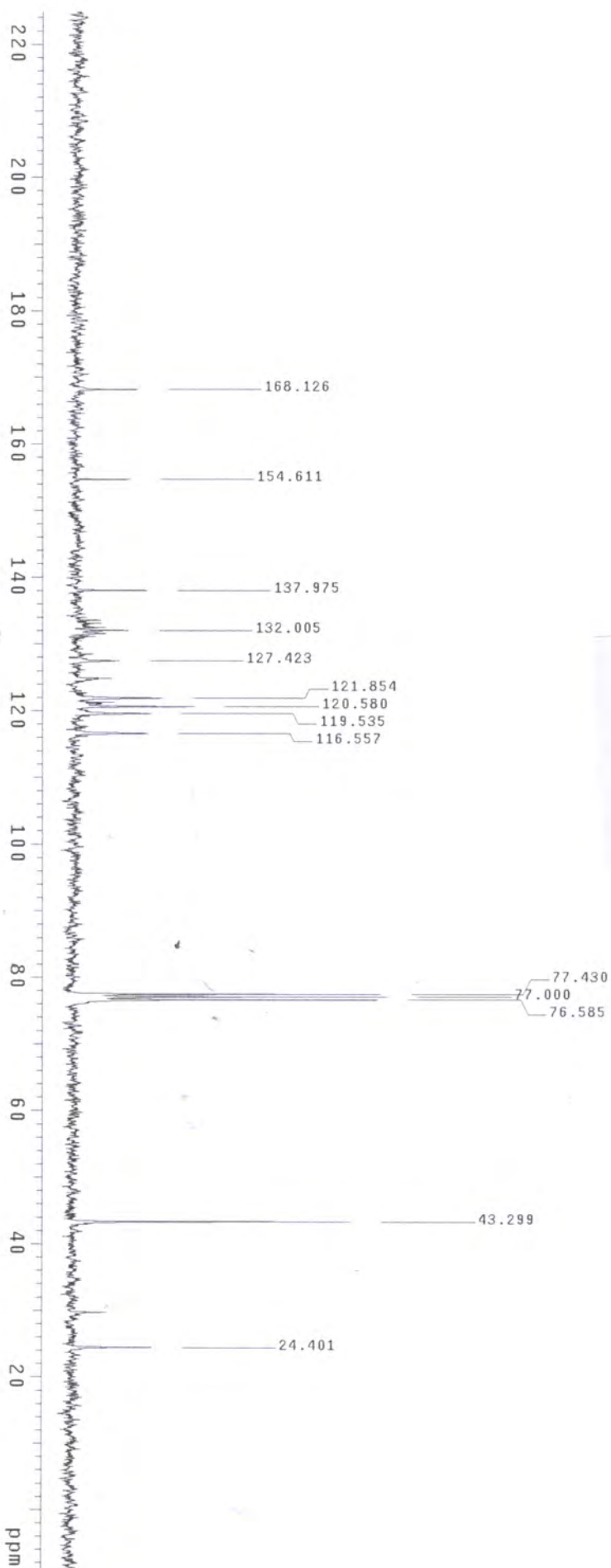
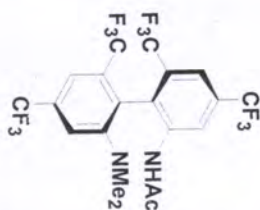
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.439 sec
Width 17699.1 Hz
184 repetitions
OBSERVE C13, 75.4552718 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 8.0 Hz
FT size 32768
Total time 57 min, 43 sec

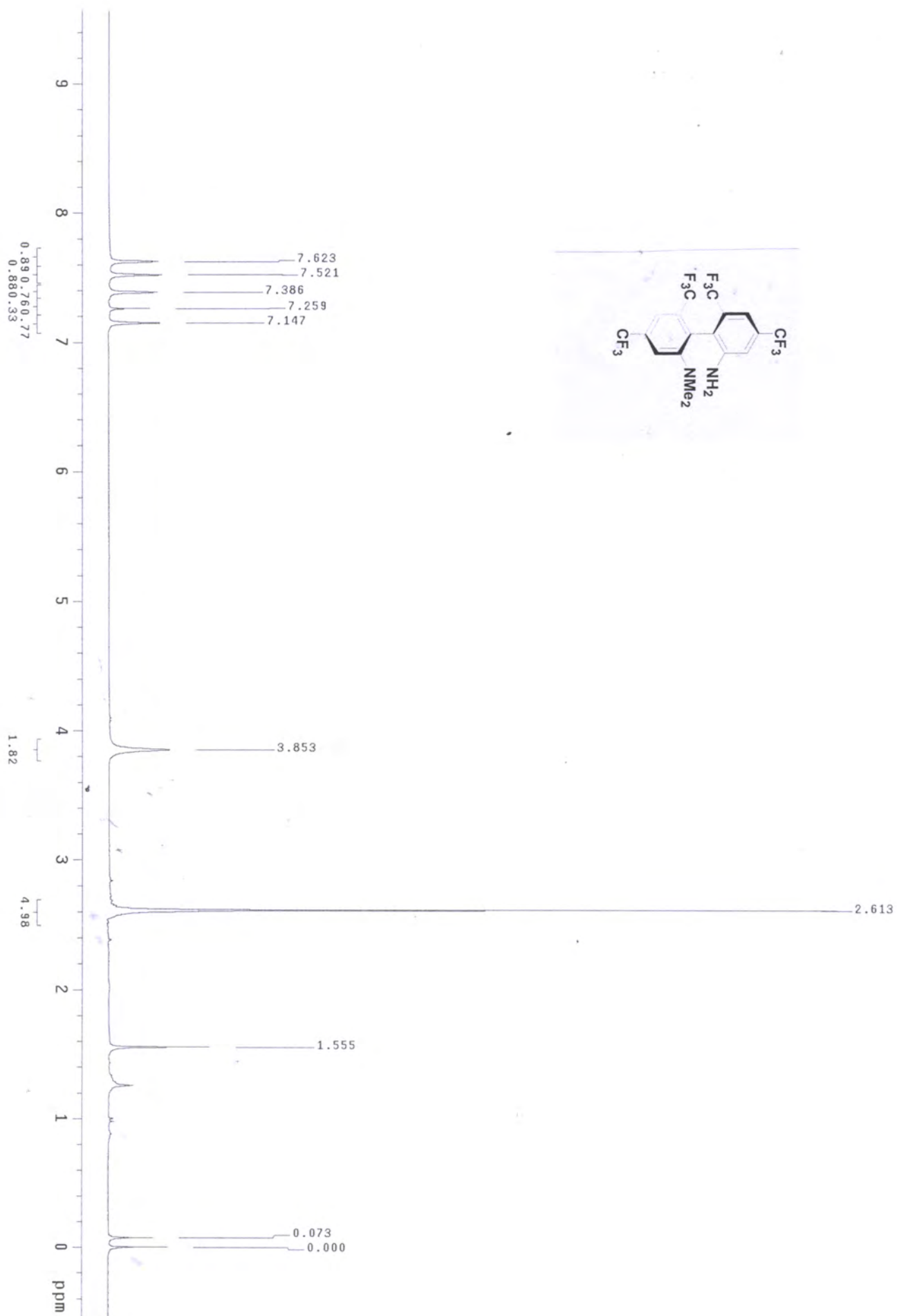




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
256 repetitions
OBSERVE C13, 75.4552740 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 5.0 Hz
FI size 32768
Total time 2 hr, 17 min, 30 sec

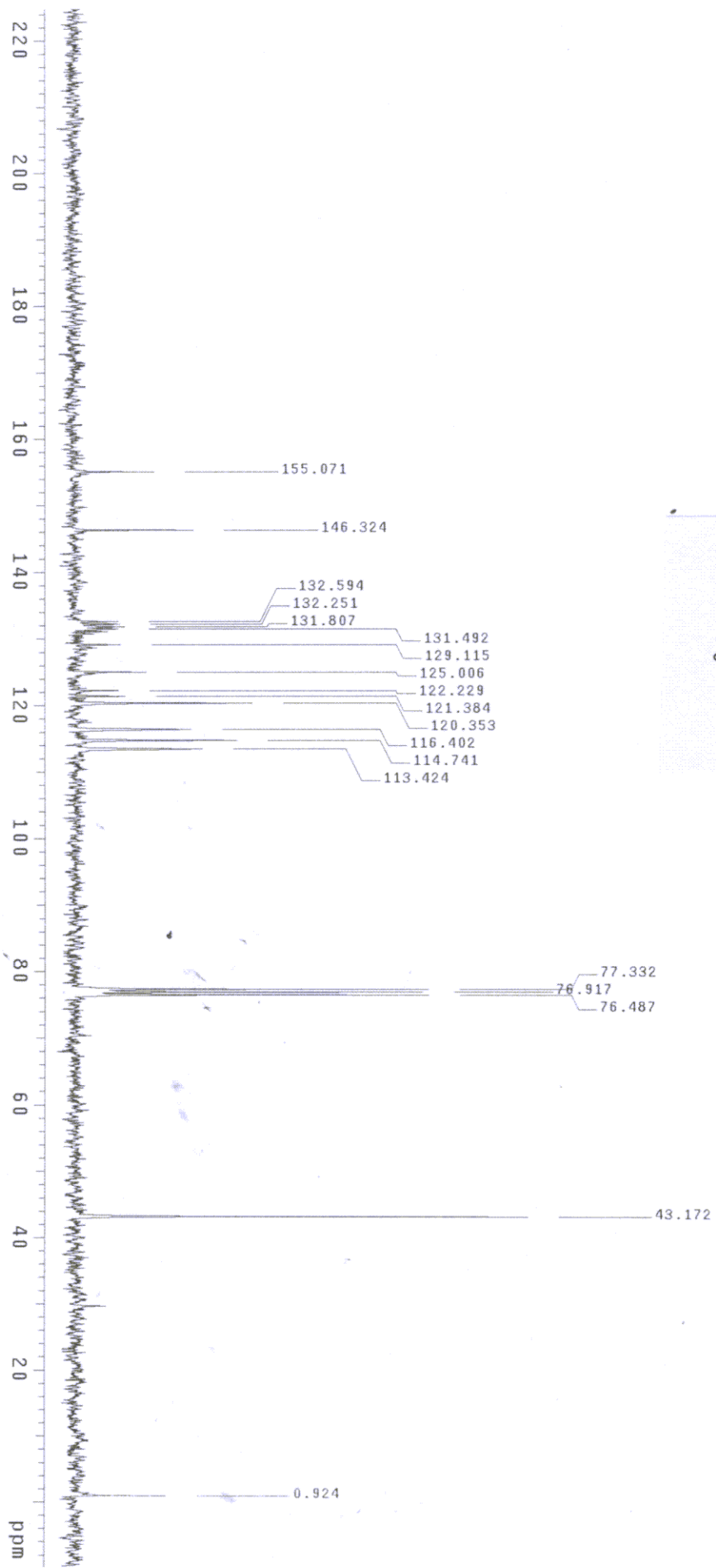
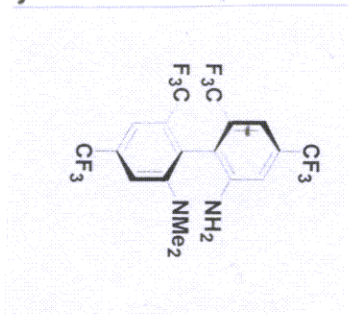


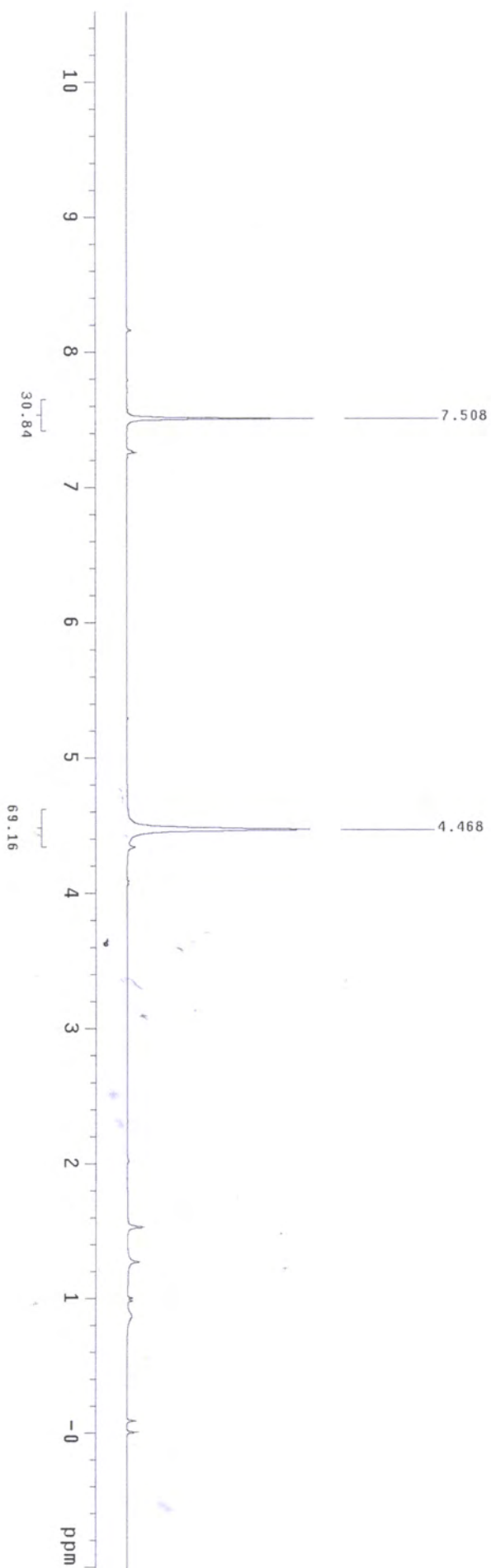
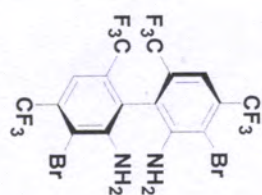


427-3-69

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

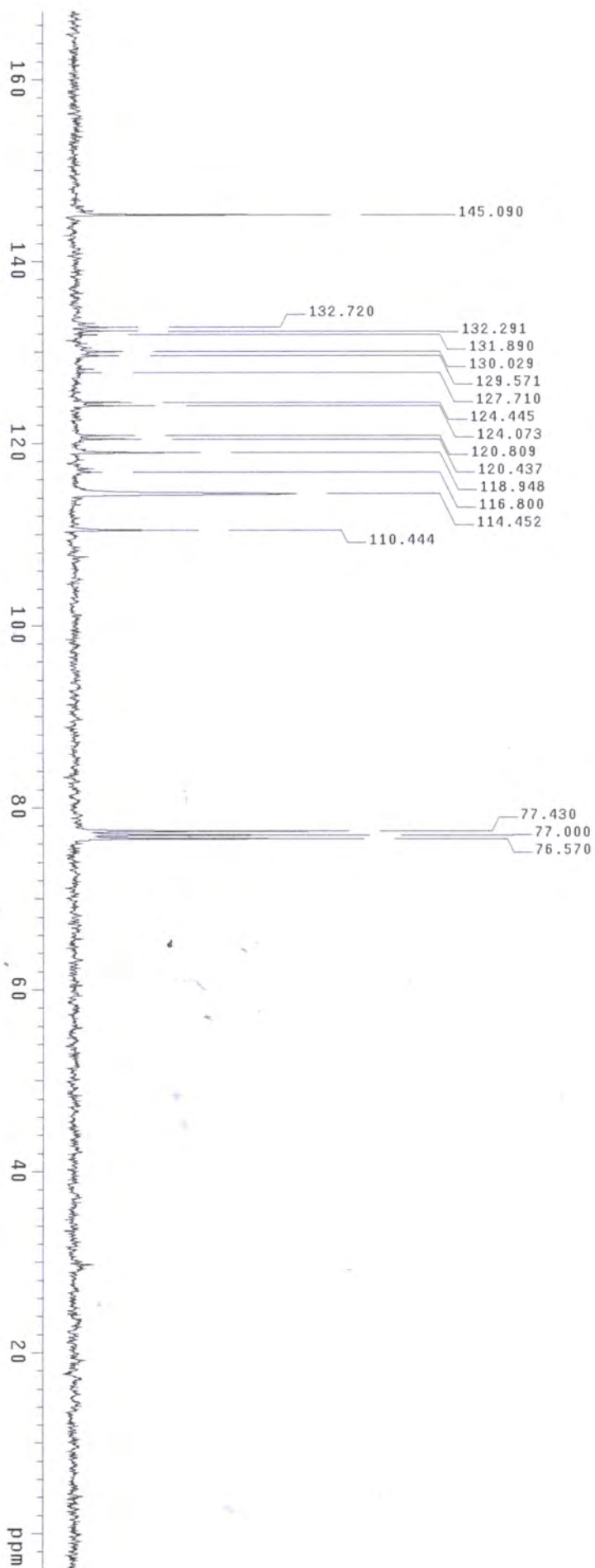
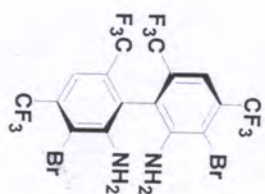
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
264 repetitions
OBSERVE C13 75.4552792 MHz
DECUPLE H1 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 5.0 Hz
FI size 32768
Total time 3 hr, 22 min, 9 sec

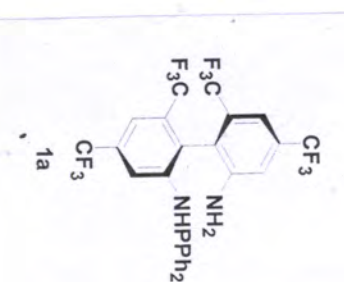
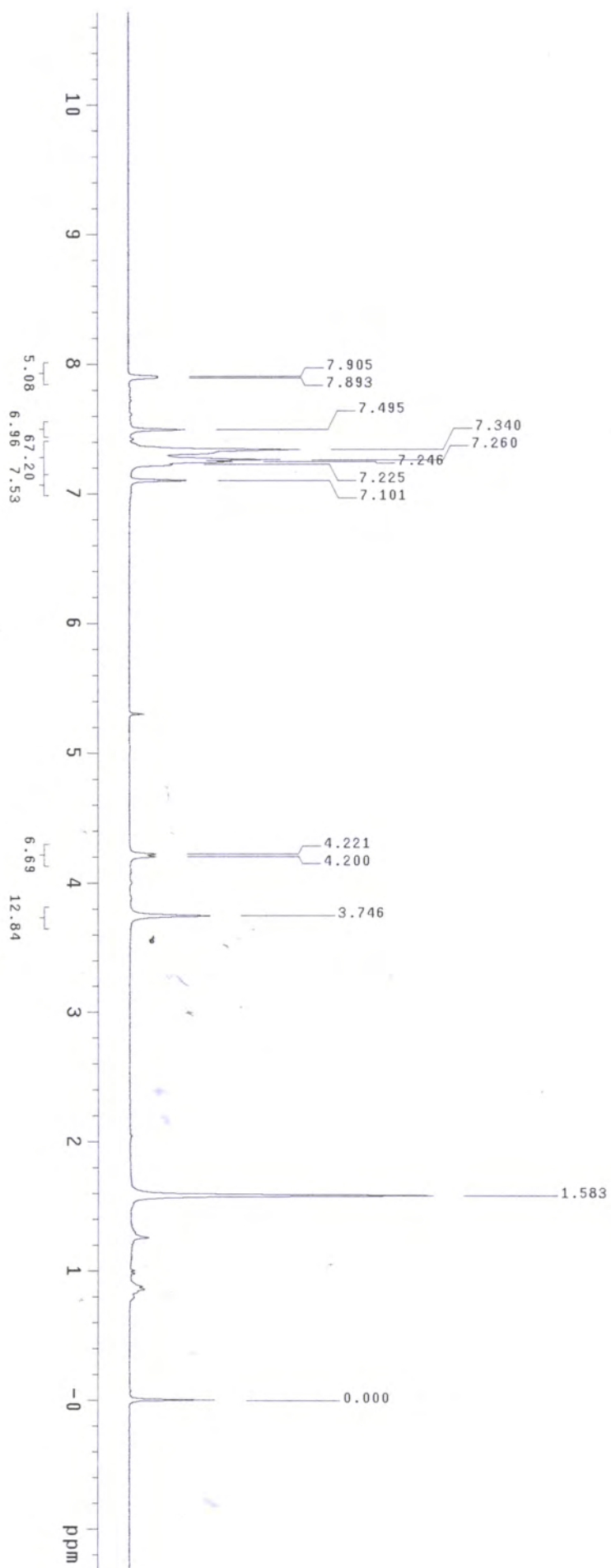




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

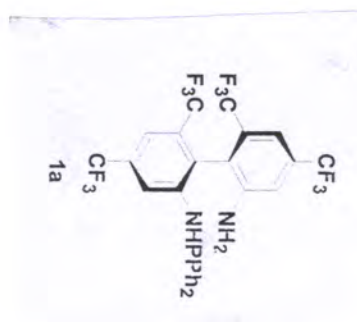
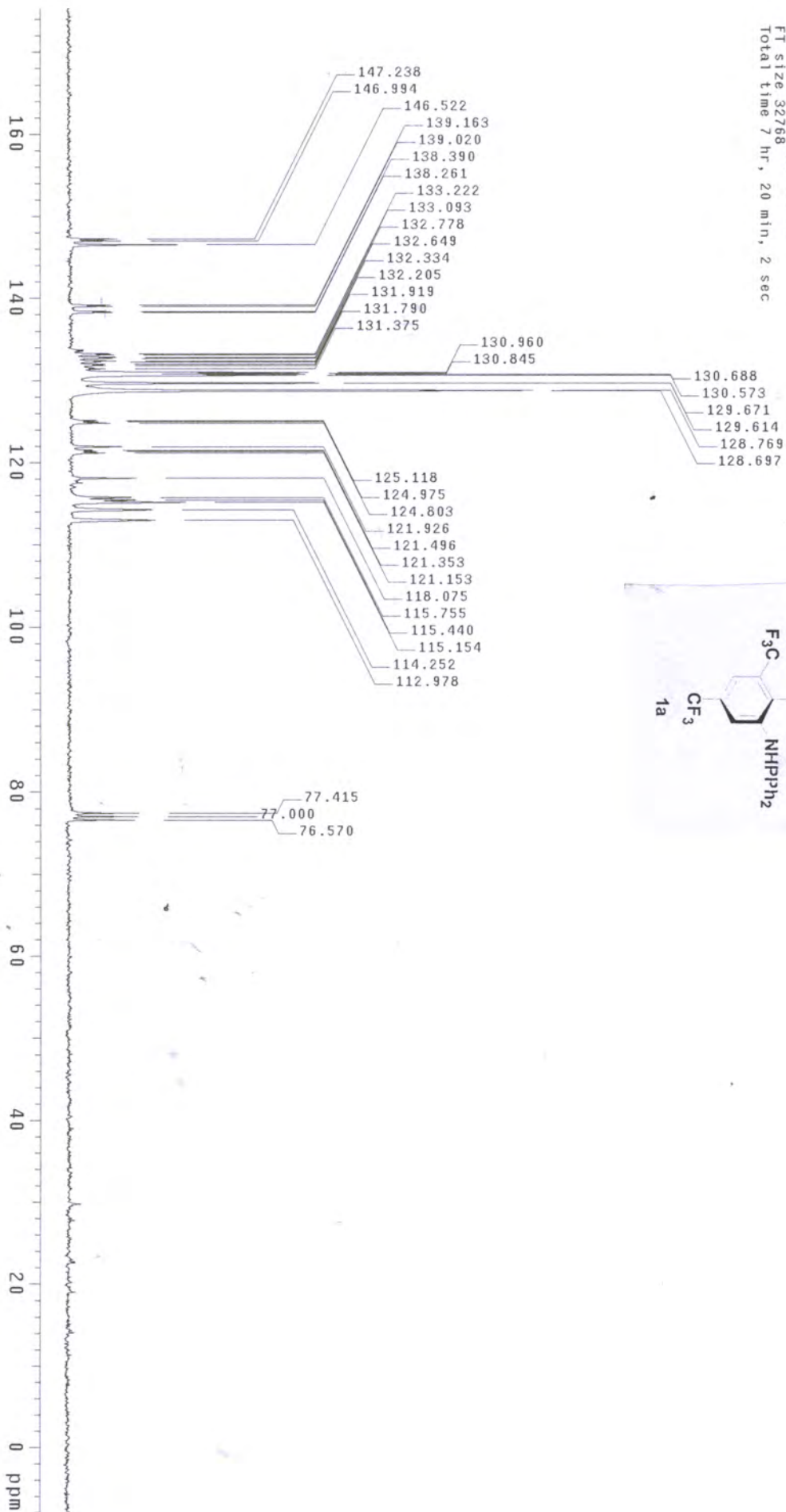
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.400 sec
Width 17699.1 Hz
488 repetitions
OBSERVE C13, 75.4552740 MHz
DECUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 4.0 Hz
FT size 16384
Total time 13 hr, 21 min, 43 sec

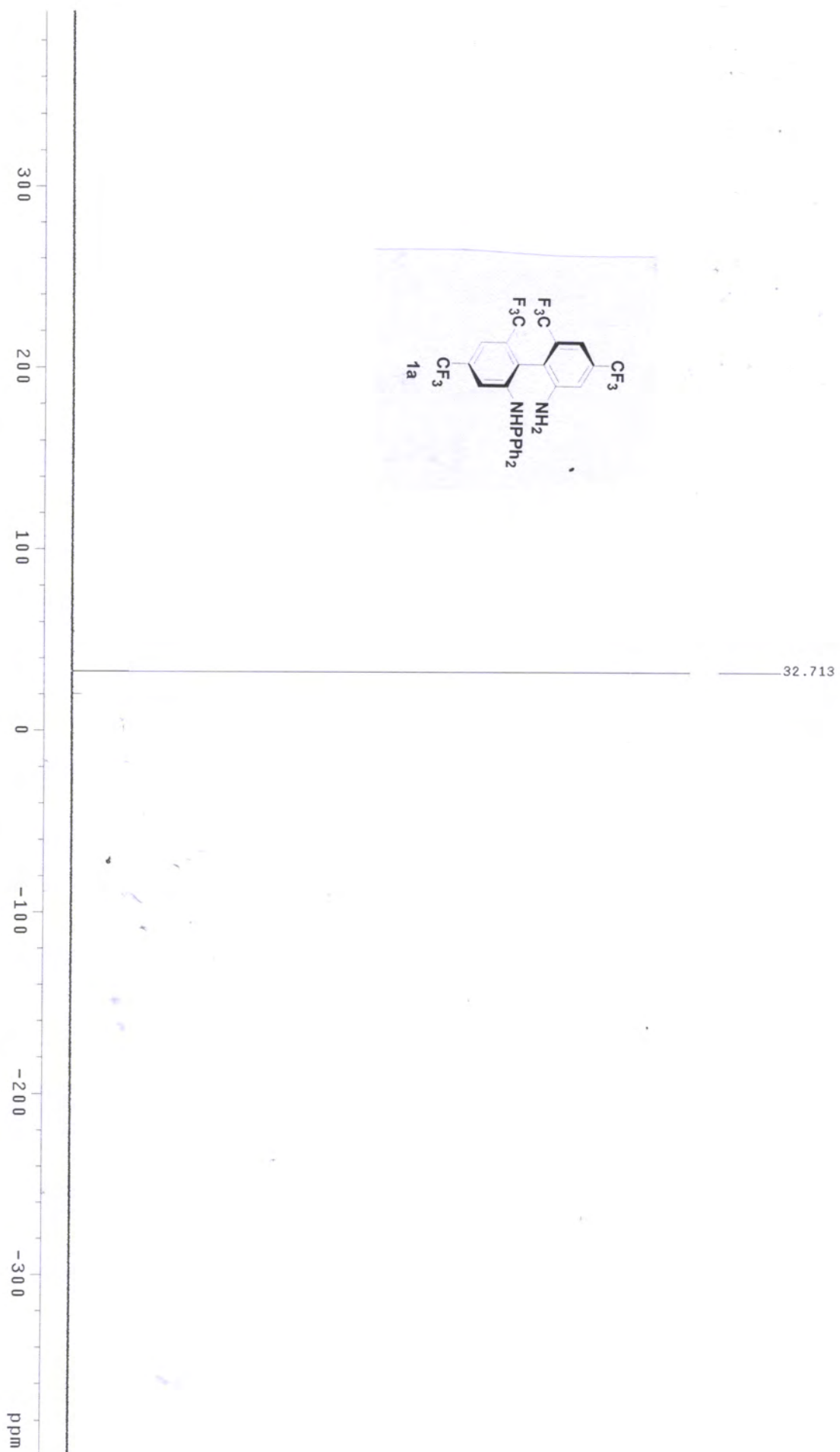


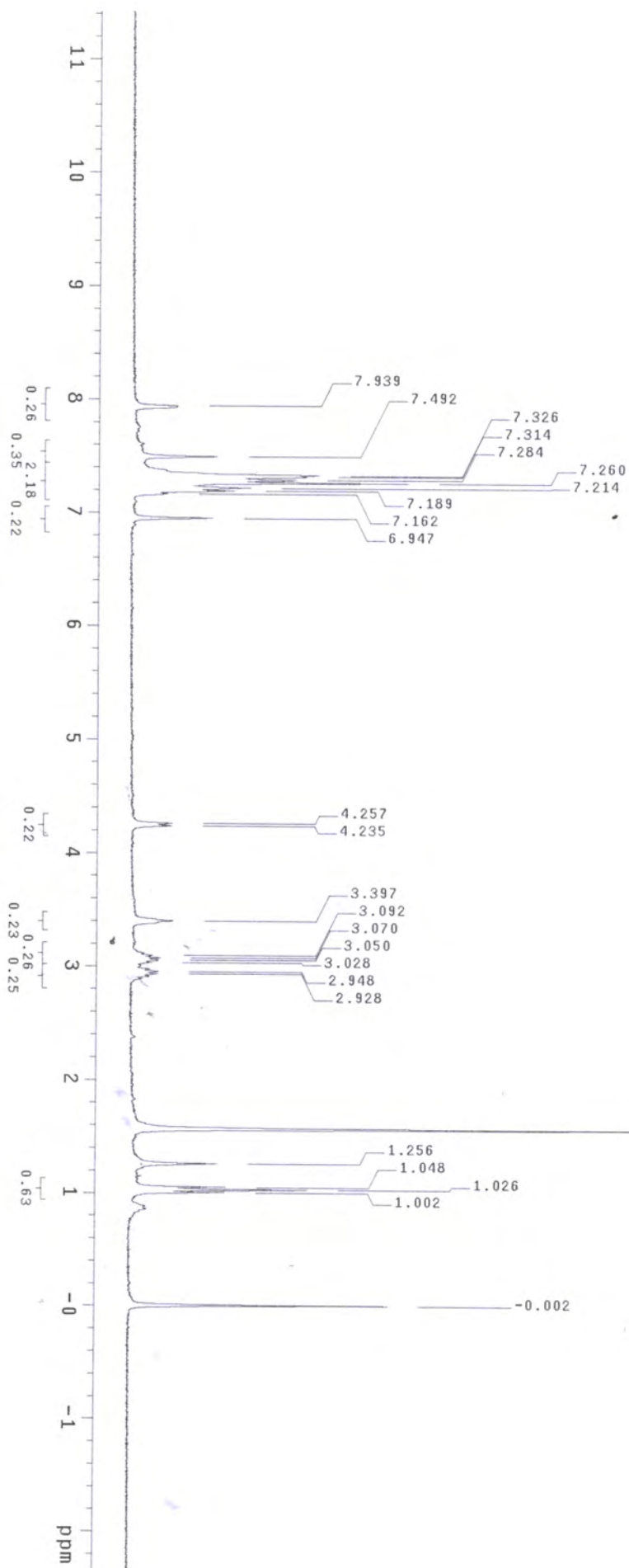
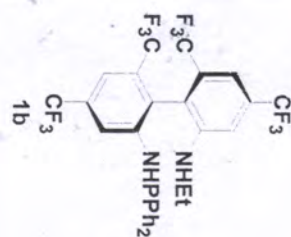


Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
304 repetitions
OBSERVE C13, 75.4552783 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 7 hr, 20 min, 2 sec

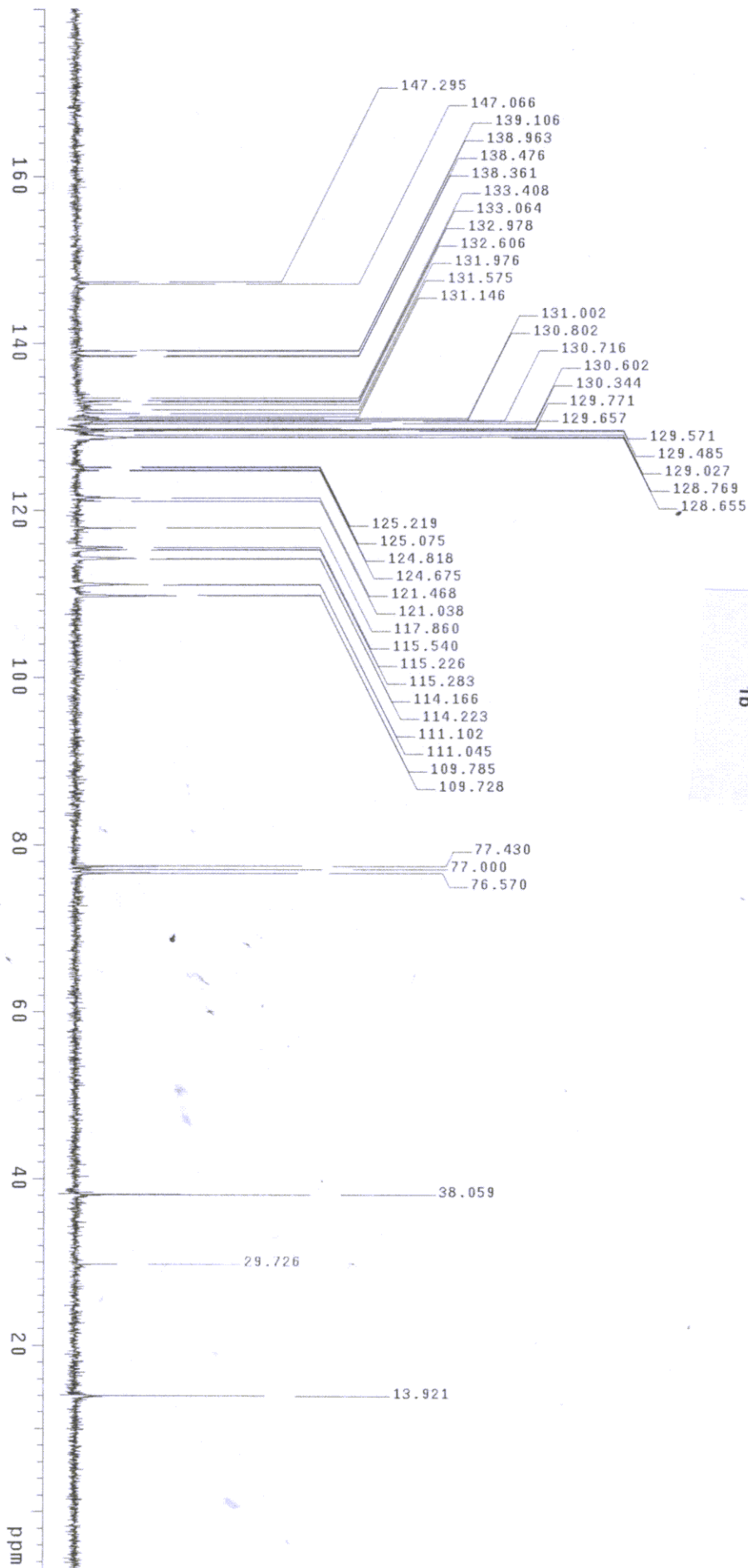
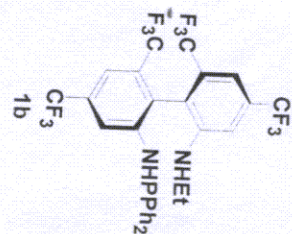


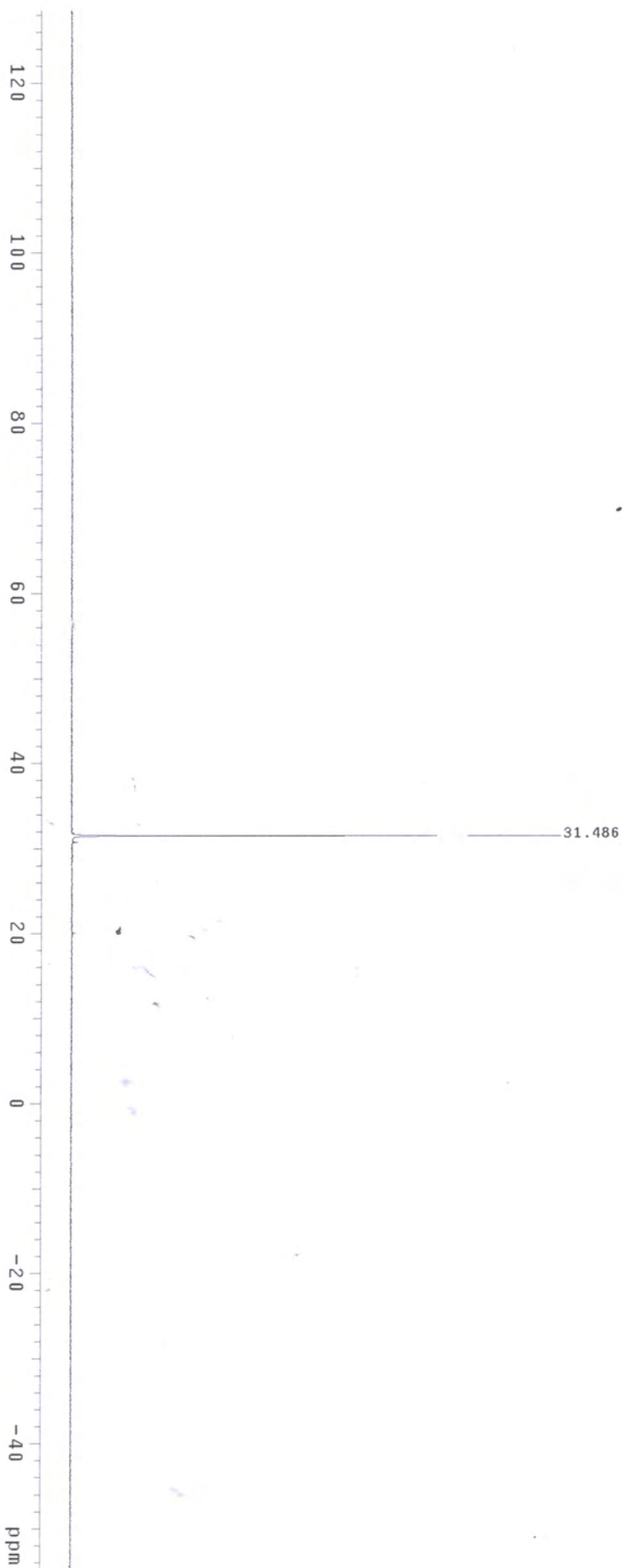
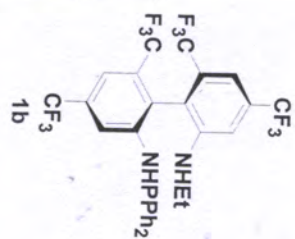


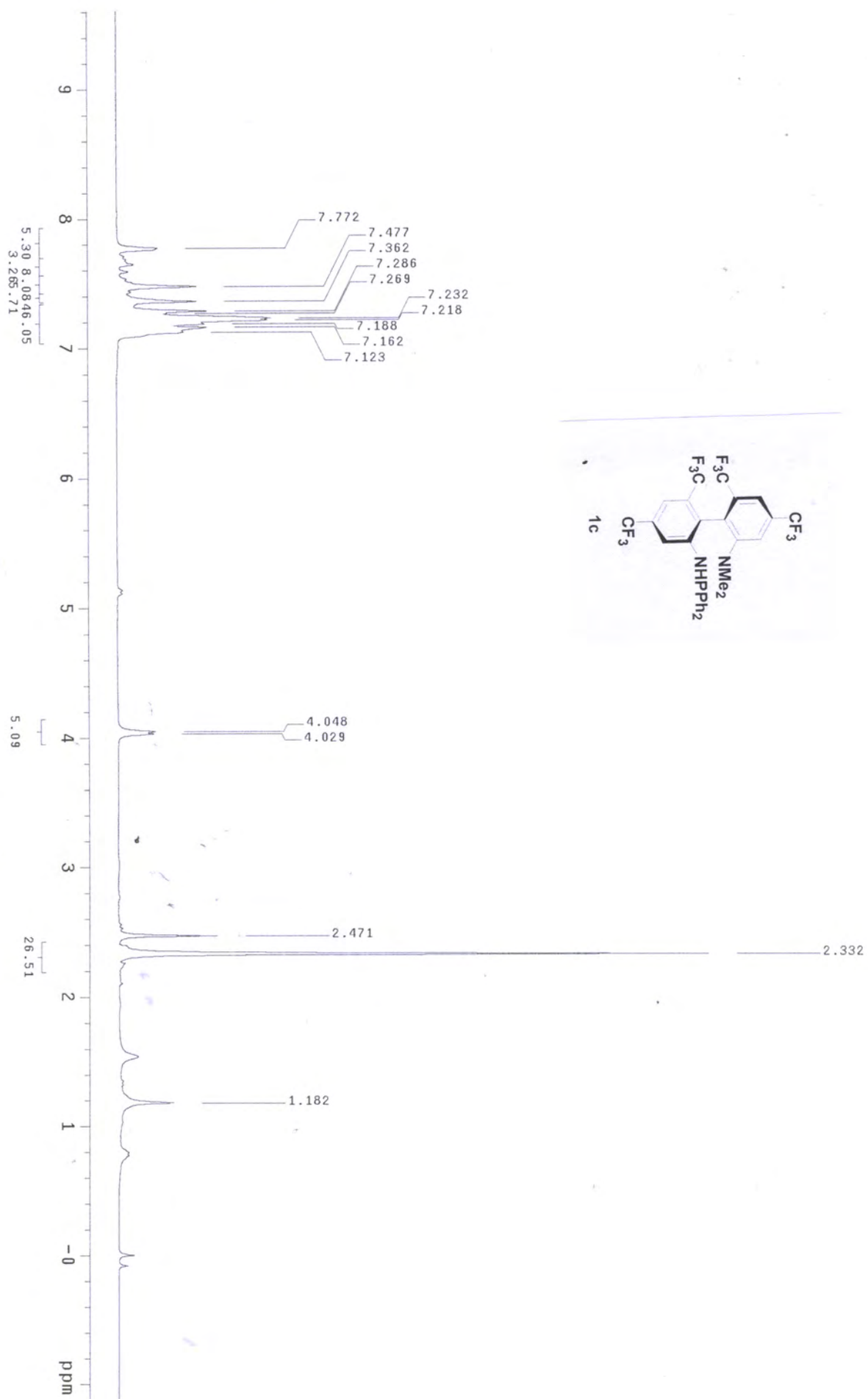


Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"

Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.300 sec
 Width 17699.1 Hz
 424 repetitions
 OBSERVE C13; 75.4552761 MHz
 DECOUPLE H1; 300.0807172 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 FI size 16384
 Total time 49 min, 8 sec

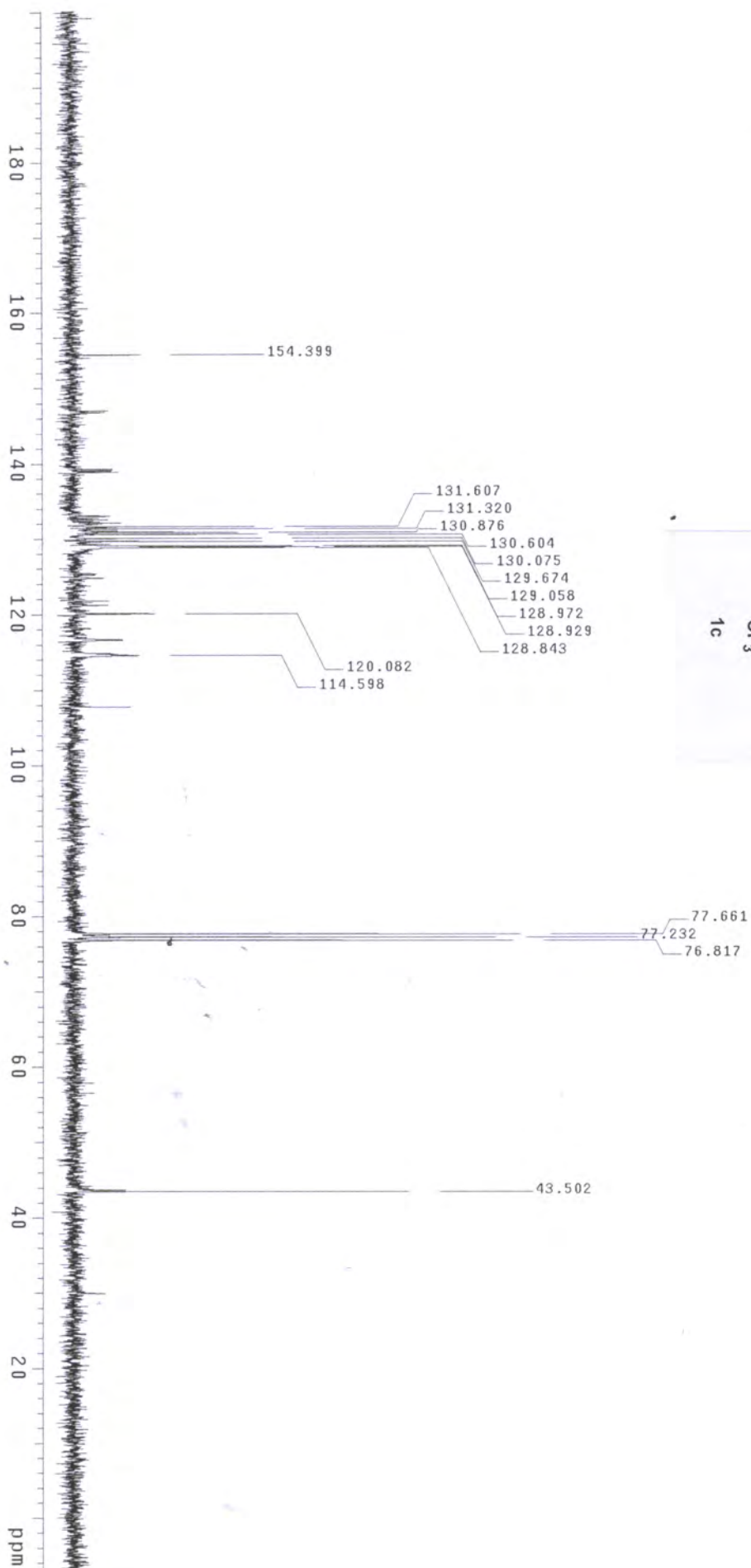
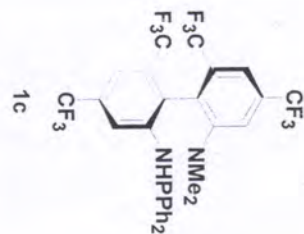


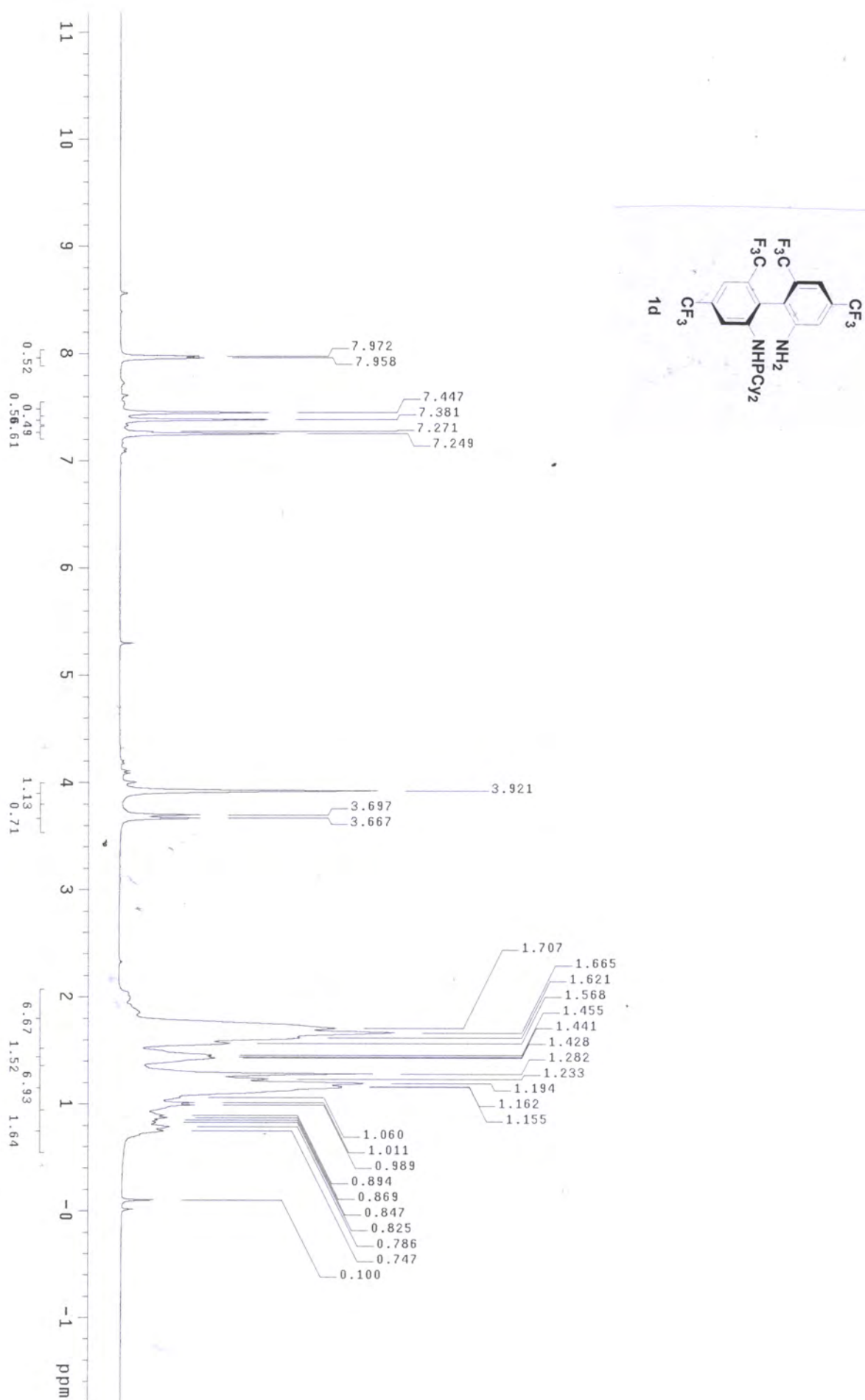




Solvent: CDC13
Ambient temperature
Mercury-300BB "mercury300"

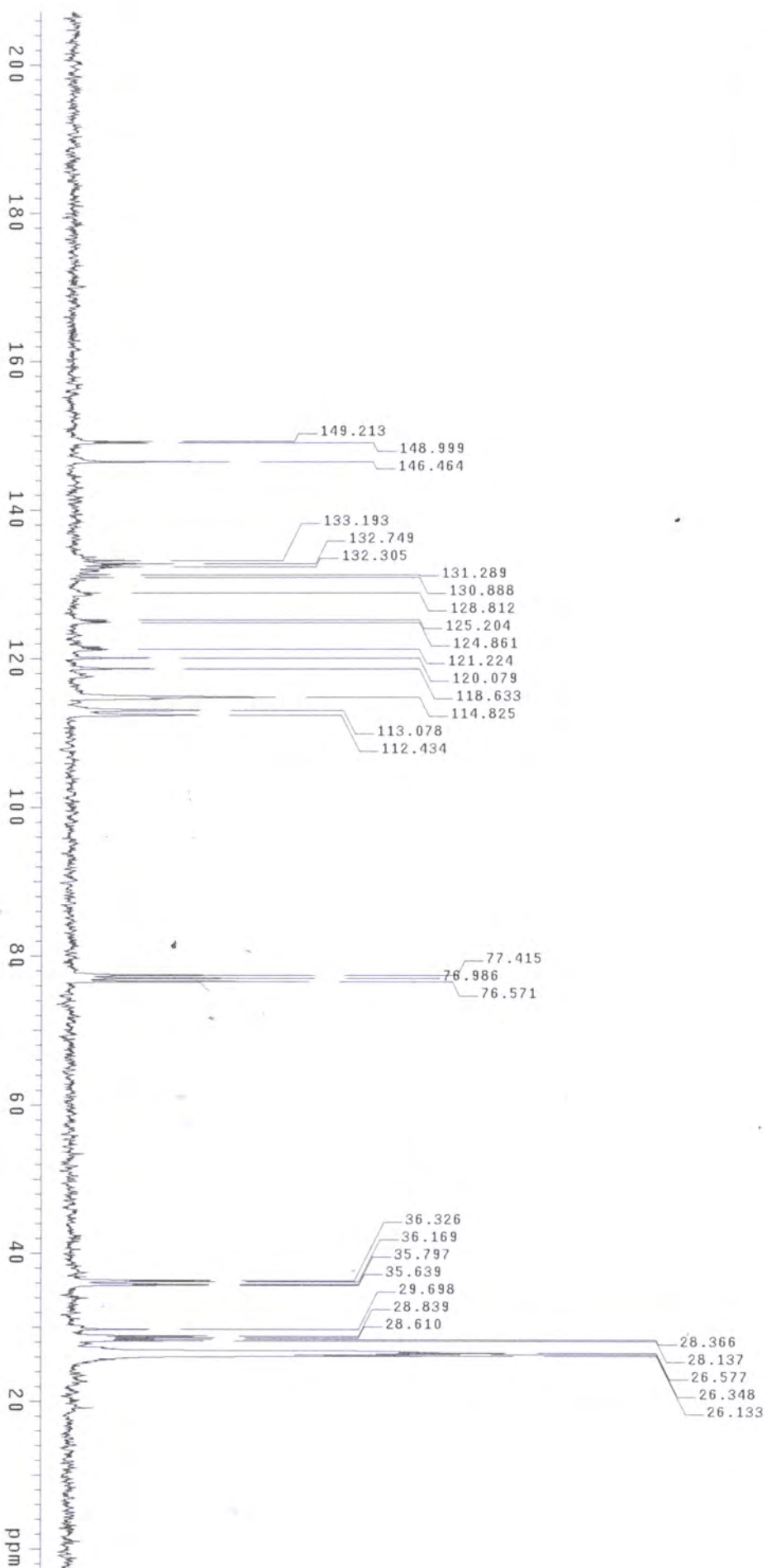
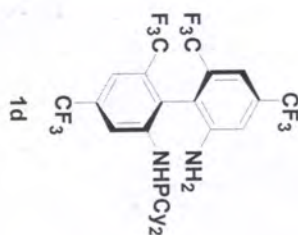
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
304 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 18 hr, 20 min, 6 sec

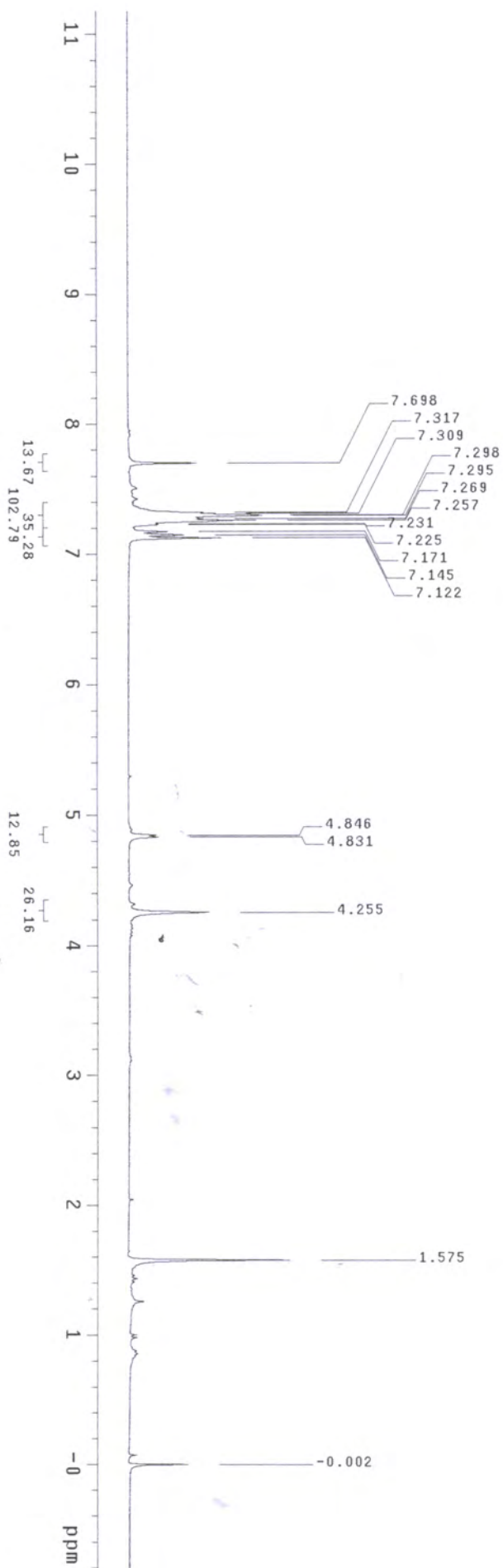
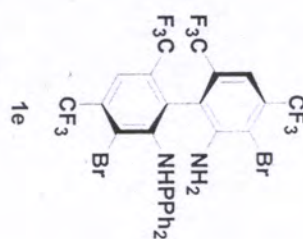




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

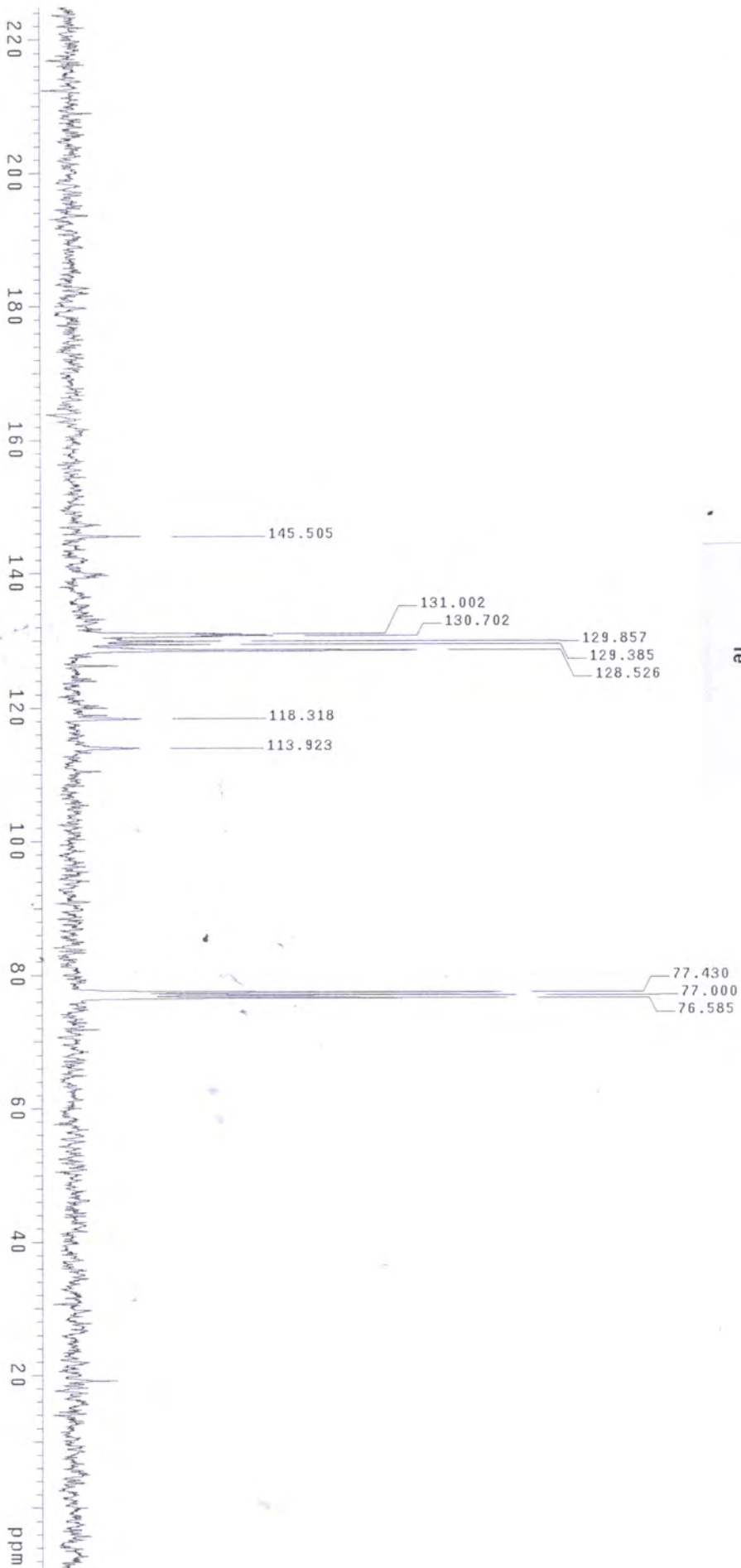
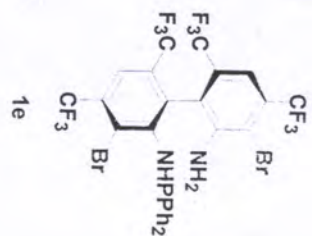
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
336 repetitions
OBSERVE C13, 75.4552740 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 5.0 Hz
FT size 32768
Total time 2 hr, 17 min, 30 sec

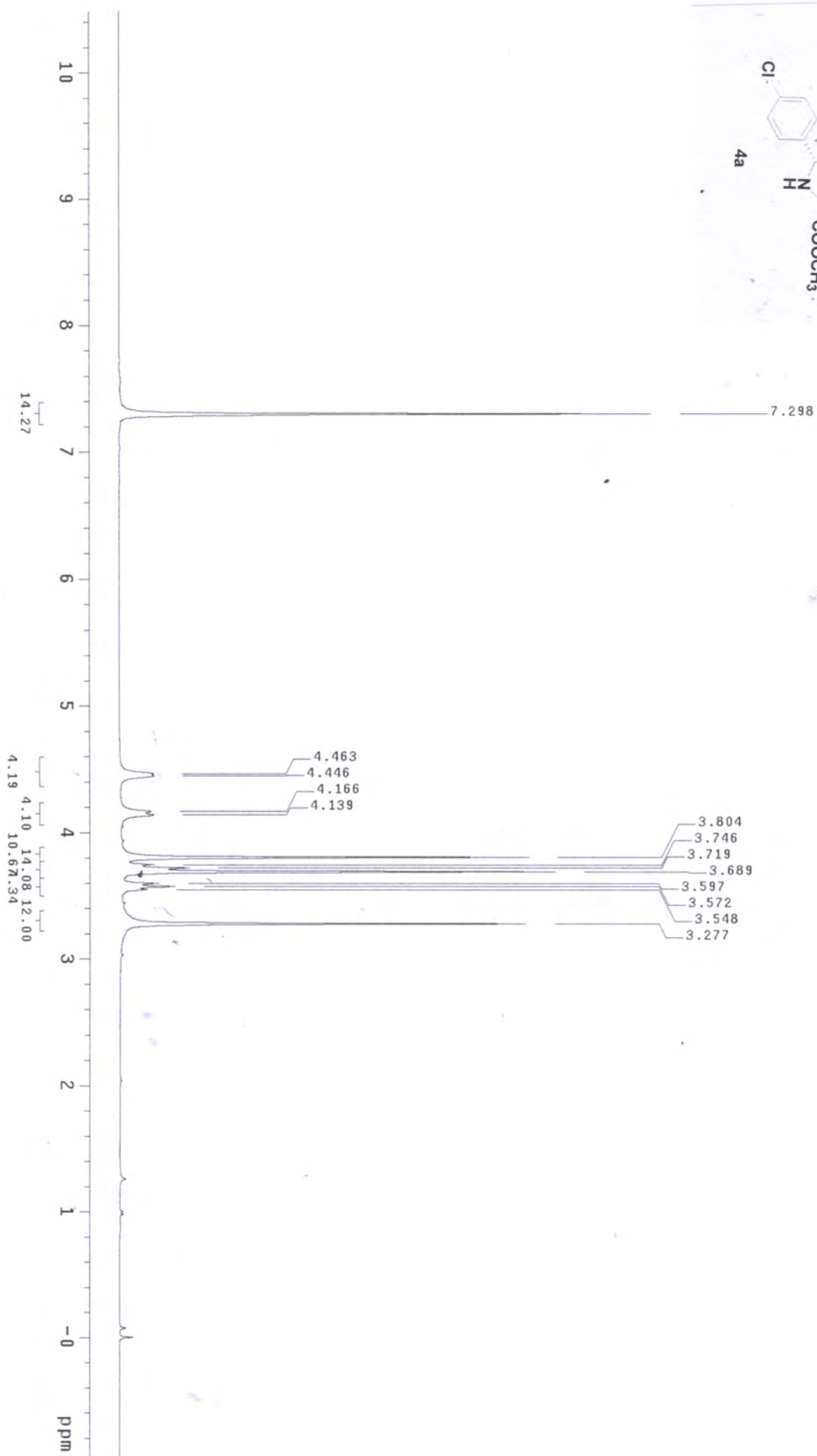
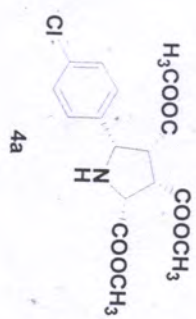




Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"

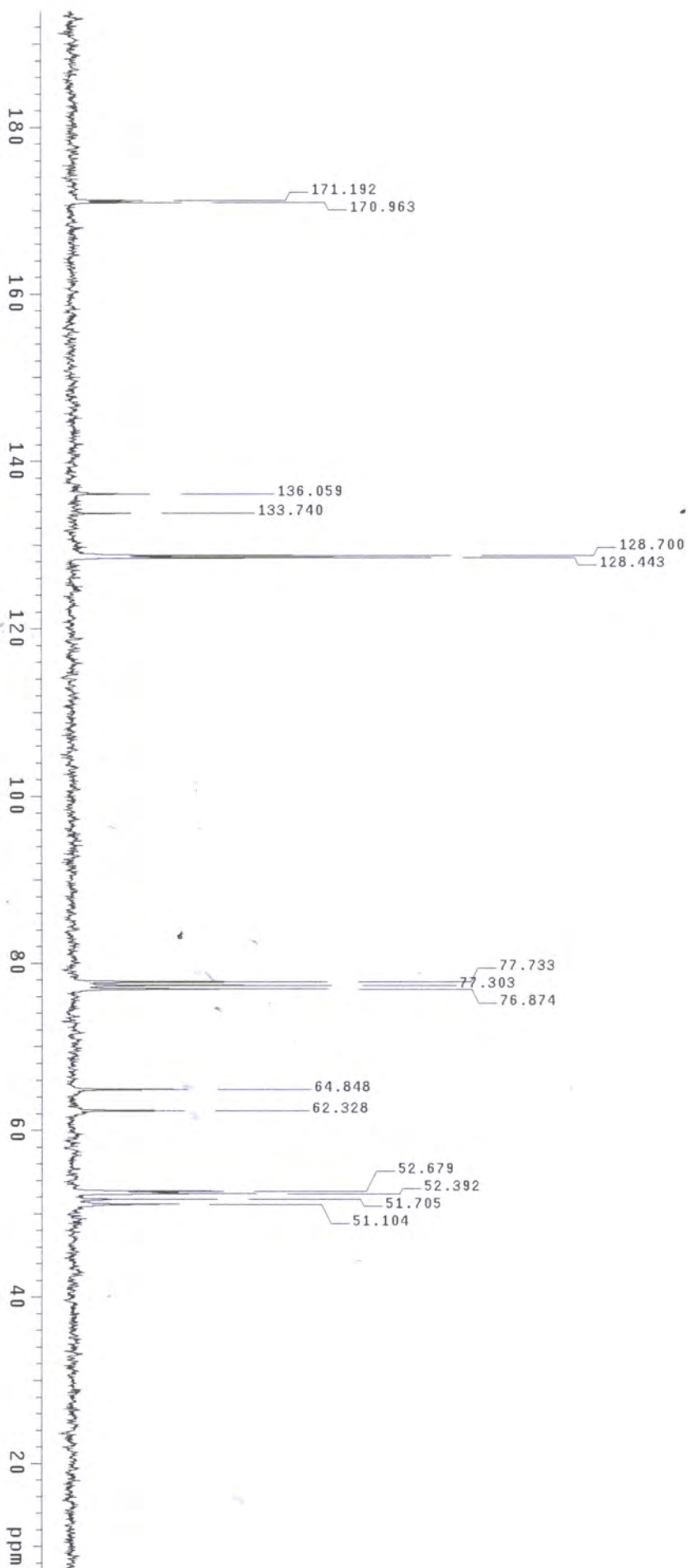
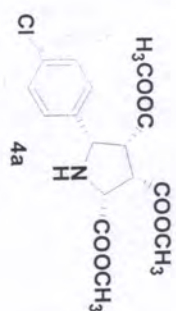
Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.459 sec
 Width 17599.1 Hz
 288 repetitions
 OBSERVE C13, 75.4552761 MHz
 DECOUPLE H1, 300.0807172 MHz
 Power 40 db
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 line broadening 8.0 Hz
 FT size 32768
 Total time 57 min, 43 sec

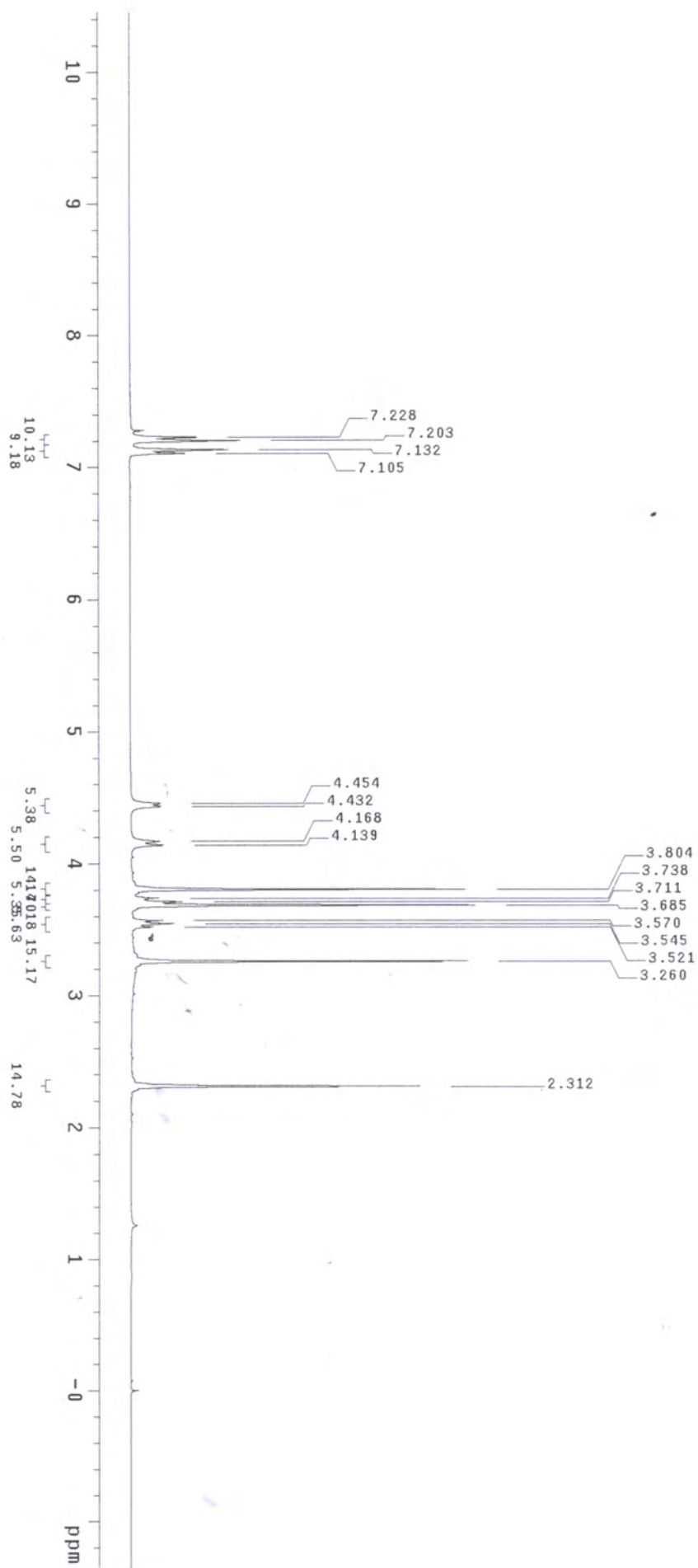
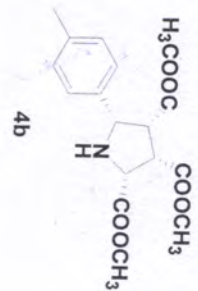




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

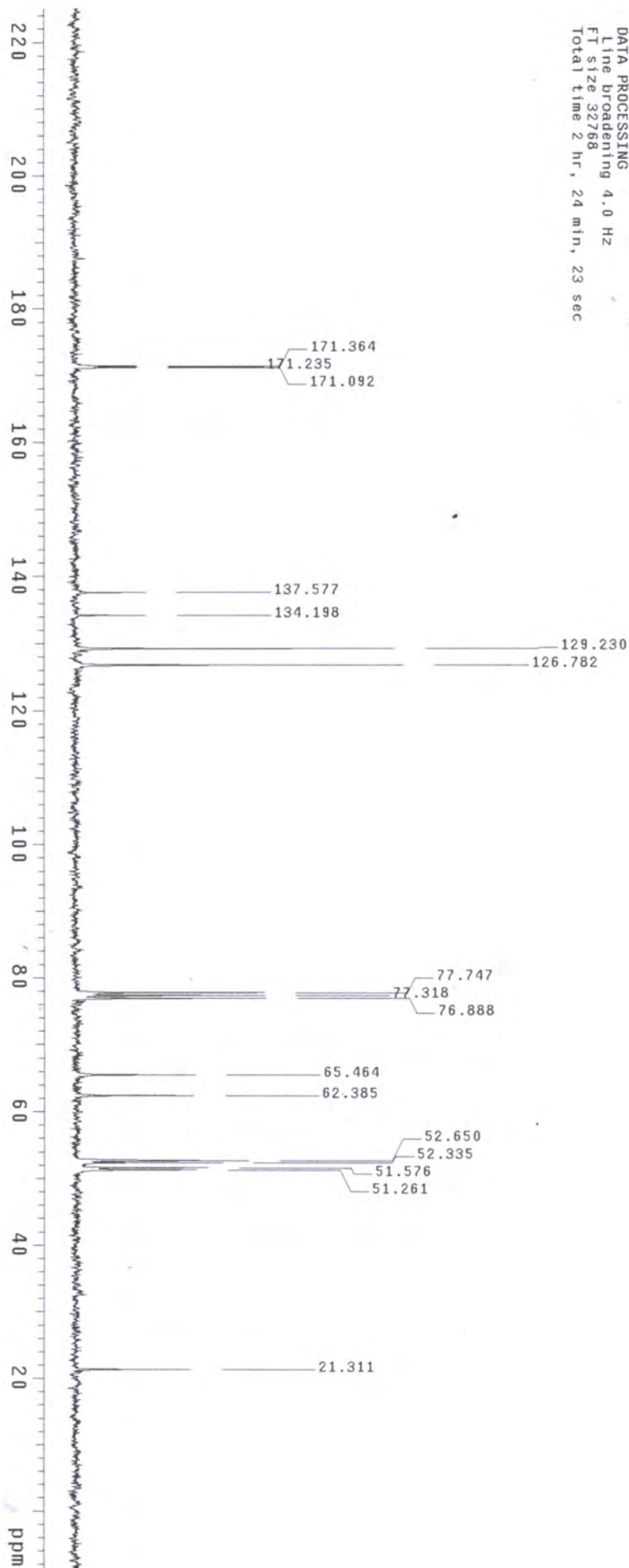
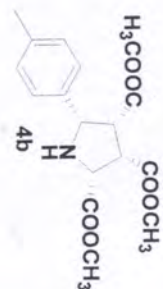
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.450 sec
Width 17699.1 Hz
192 repetitions
OBSERVE C13, 75.4552576 MHz
DECUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
Ft size 16384
Total time 8 hr, 51 min, 4 sec

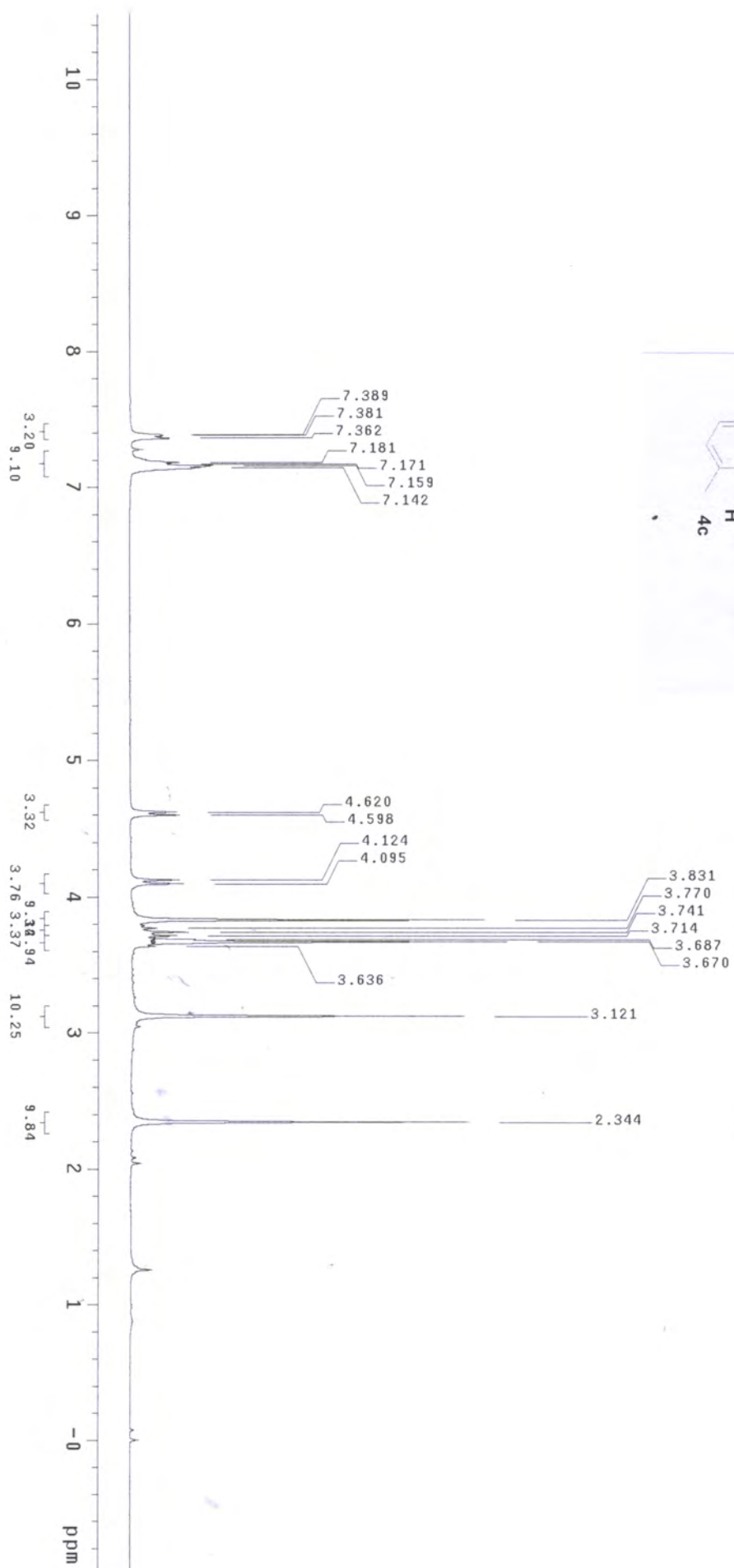




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

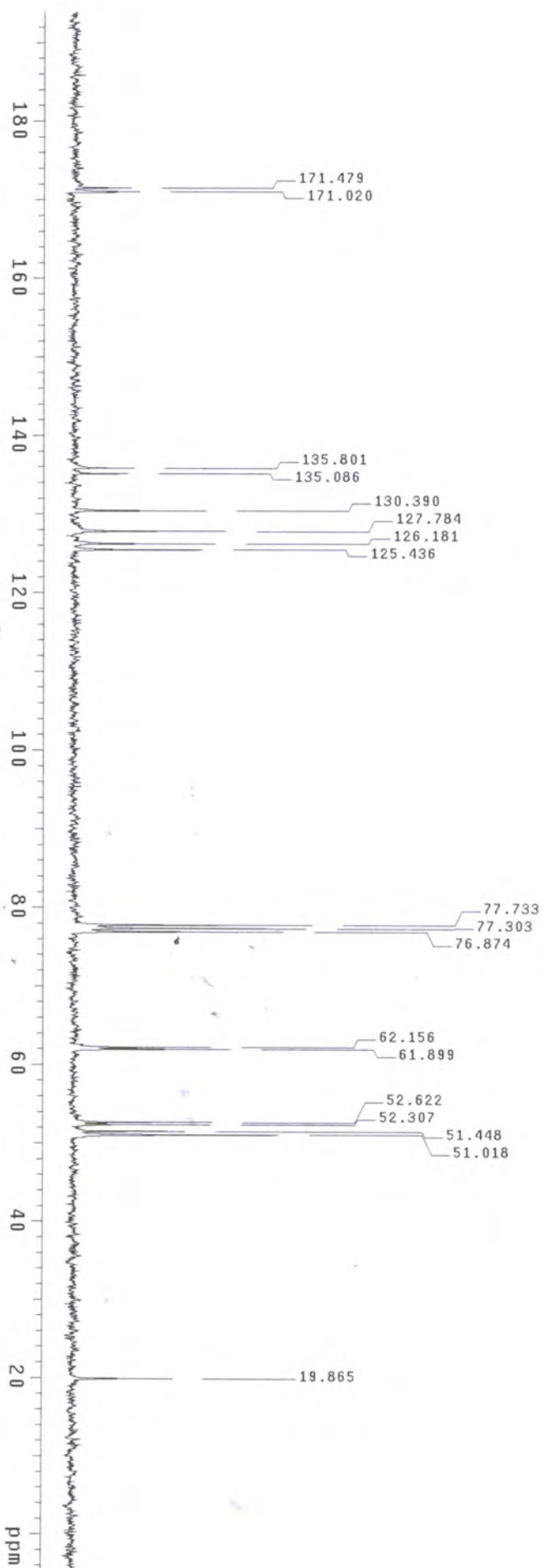
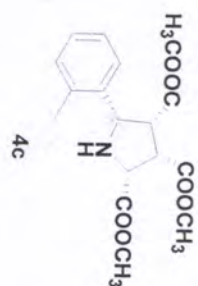
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
240 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FI size 32768
Total time 2 hr, 24 min, 23 sec

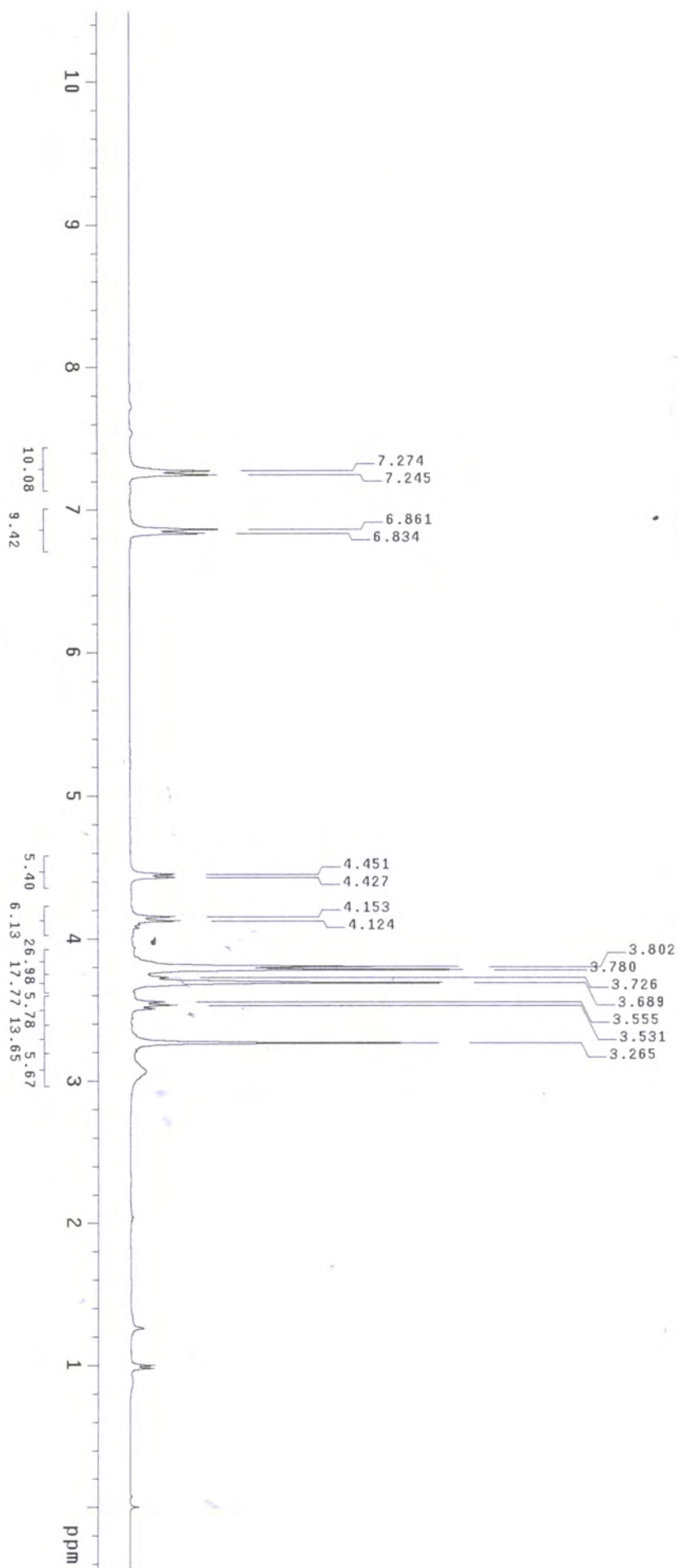
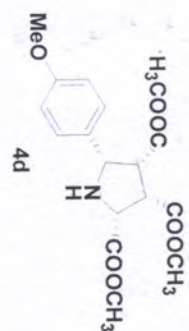




Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"

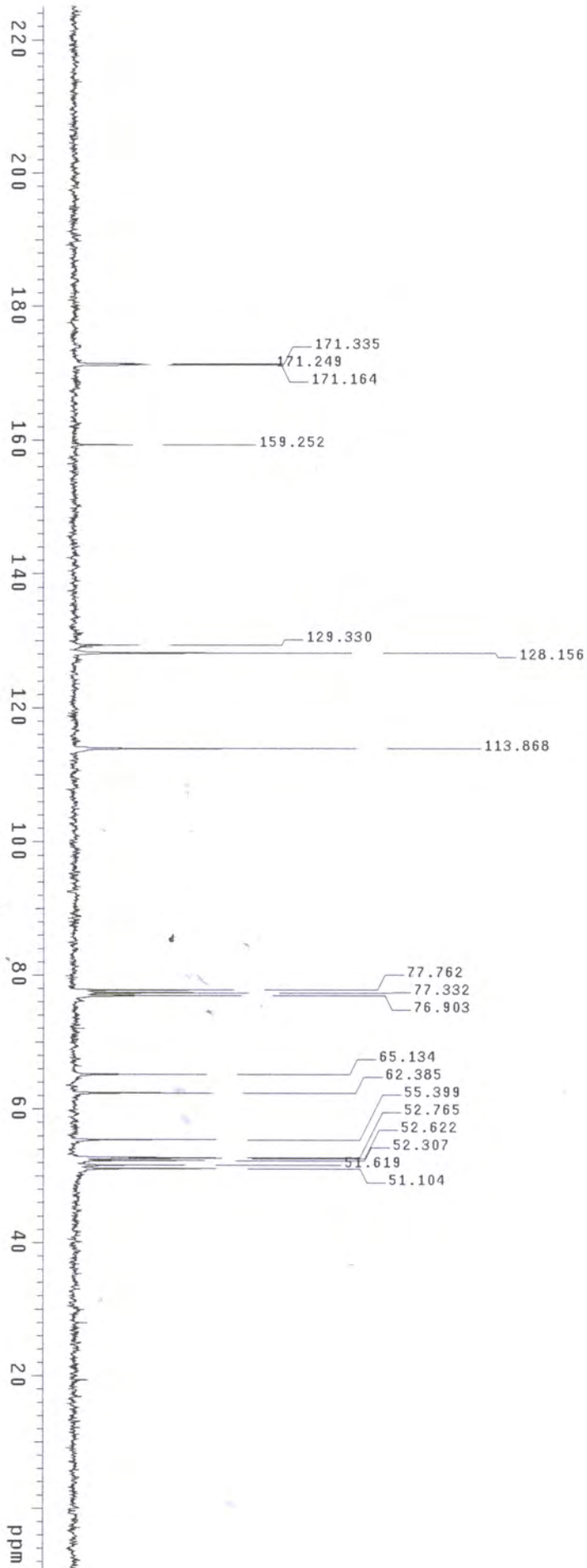
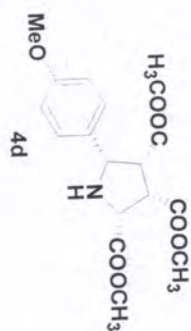
Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.450 sec
 Width 17699.1 Hz
 224 repetitions
 OBSERVE C13, 75.4552576 MHz
 DECOUPLE H1, 300.0807172 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 FT size 16384
 Total time 8 hr, 51 min, 4 sec

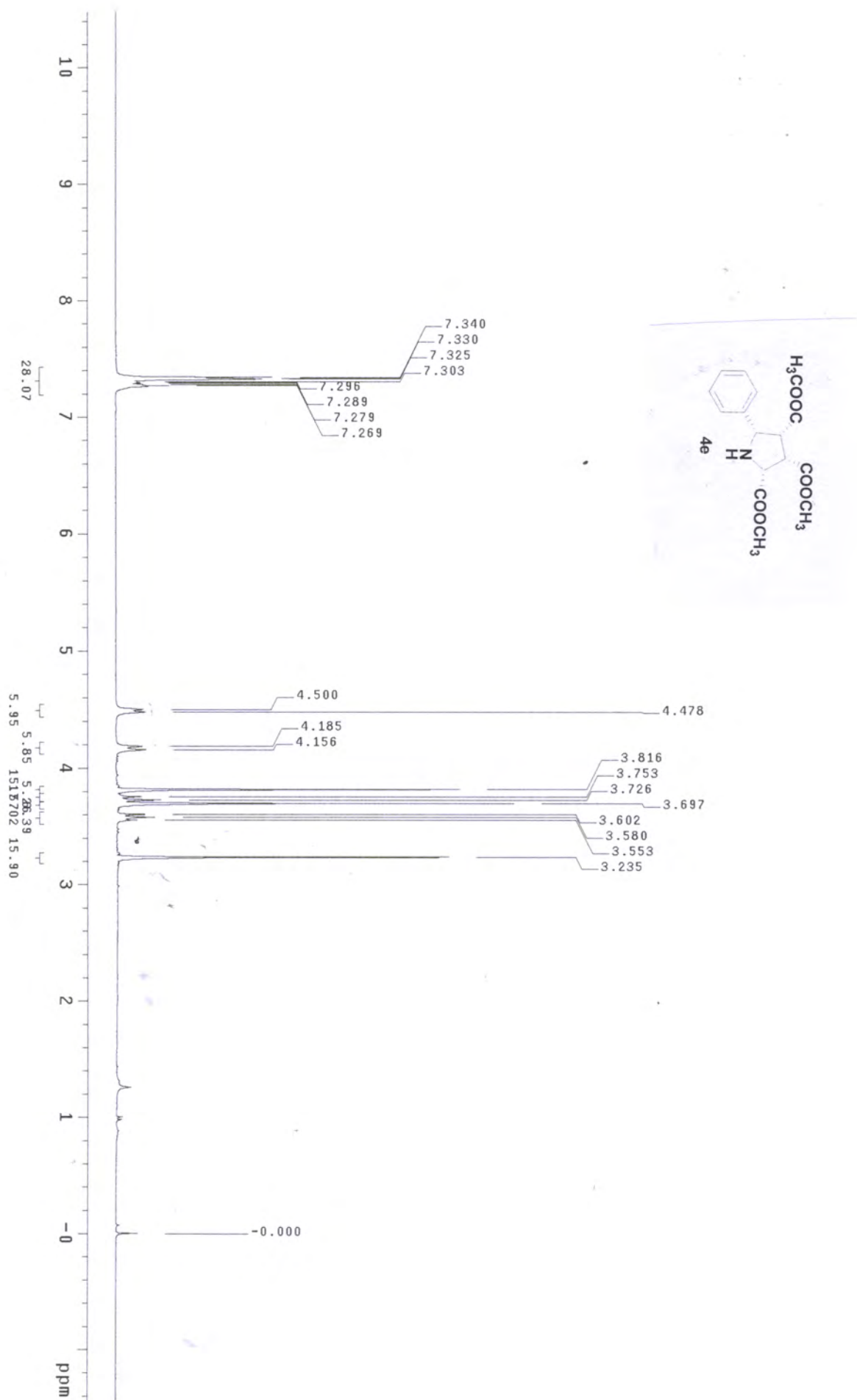




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

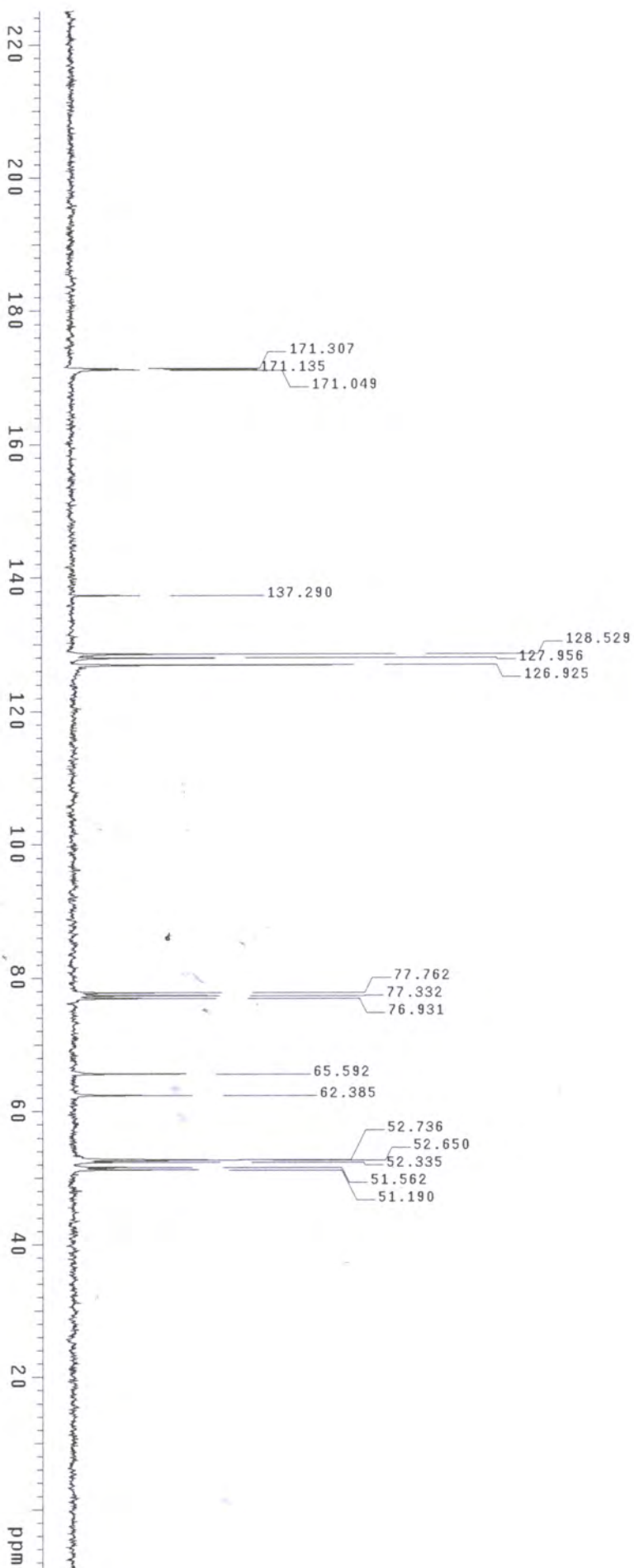
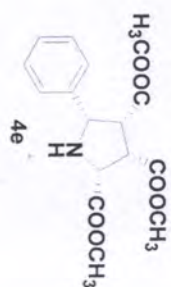
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.450 sec
Width 17699.1 Hz
192 repetitions
OBSERVE C13; 75.4552576 MHz
DECUPLE H1; 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 4.0 Hz
FT size 16384
Total time 8 hr, 51 min, 4 sec

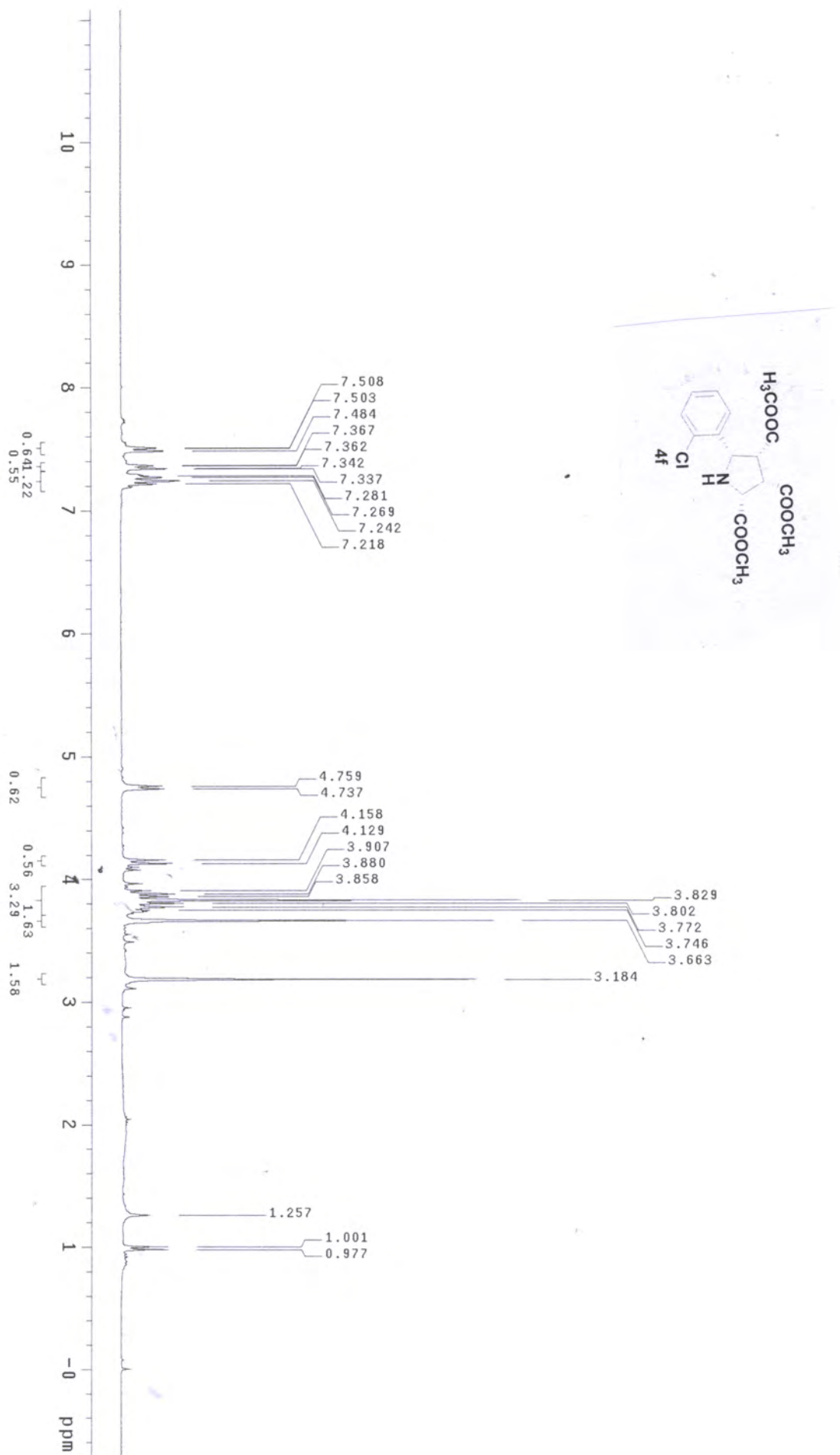




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

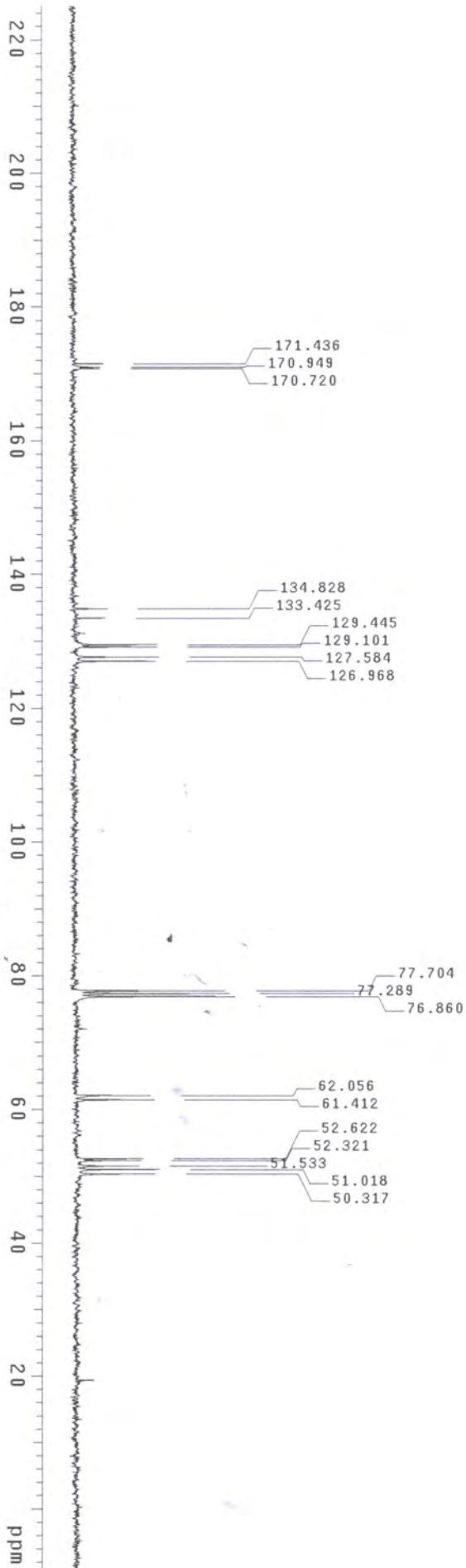
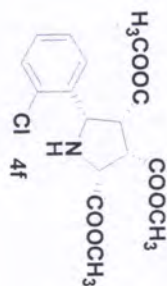
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.450 sec
Width 17699.1 Hz
224 repetitions
OBSERVE C13; 75.4552576 MHz
DECUPLE H1; 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 16384
Total time 8 hr, 51 min, 4 sec

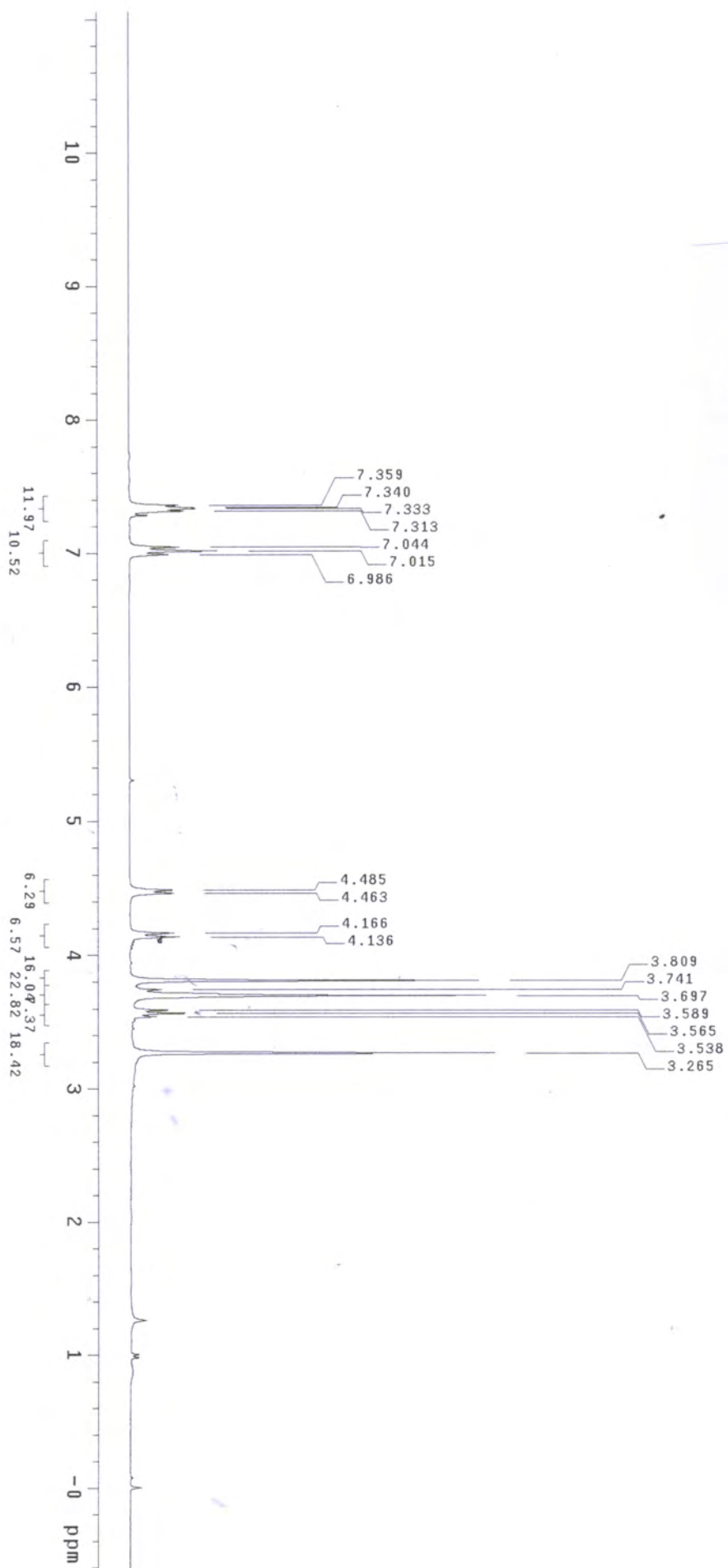
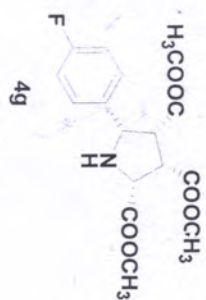




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

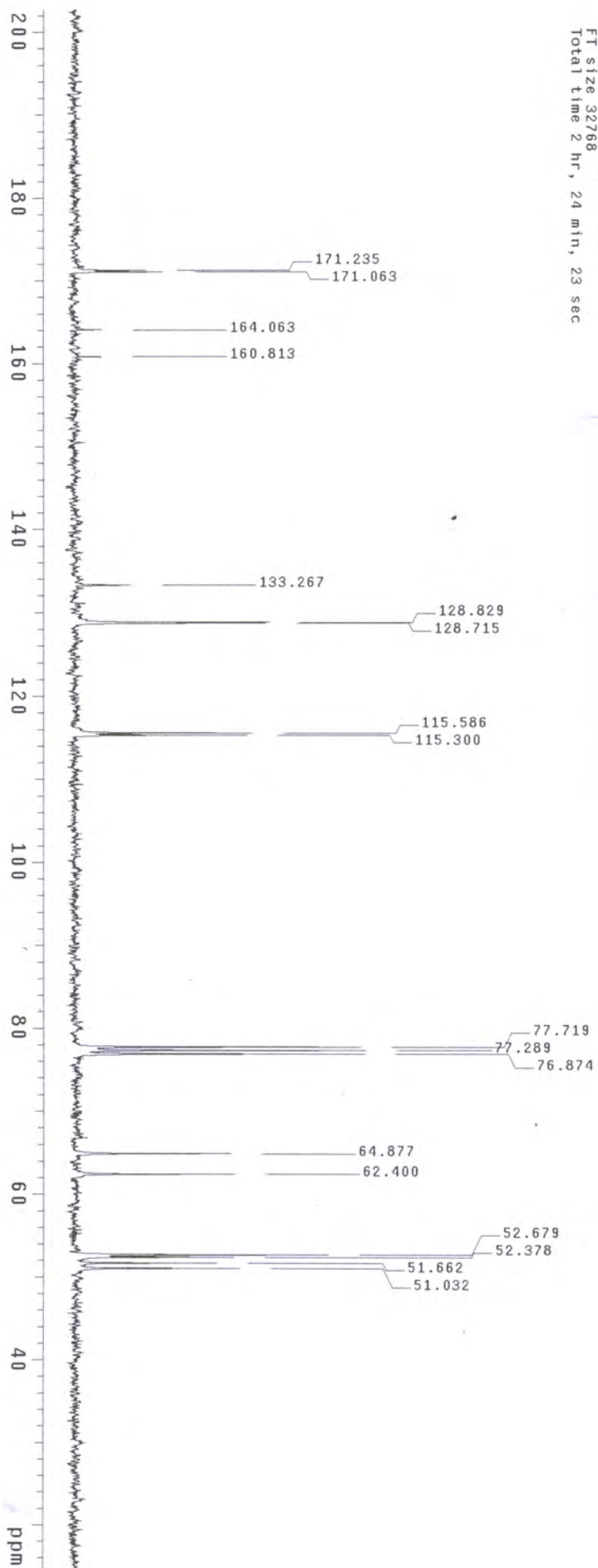
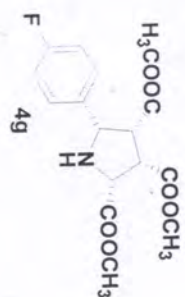
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
240 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 17 min, 30 sec

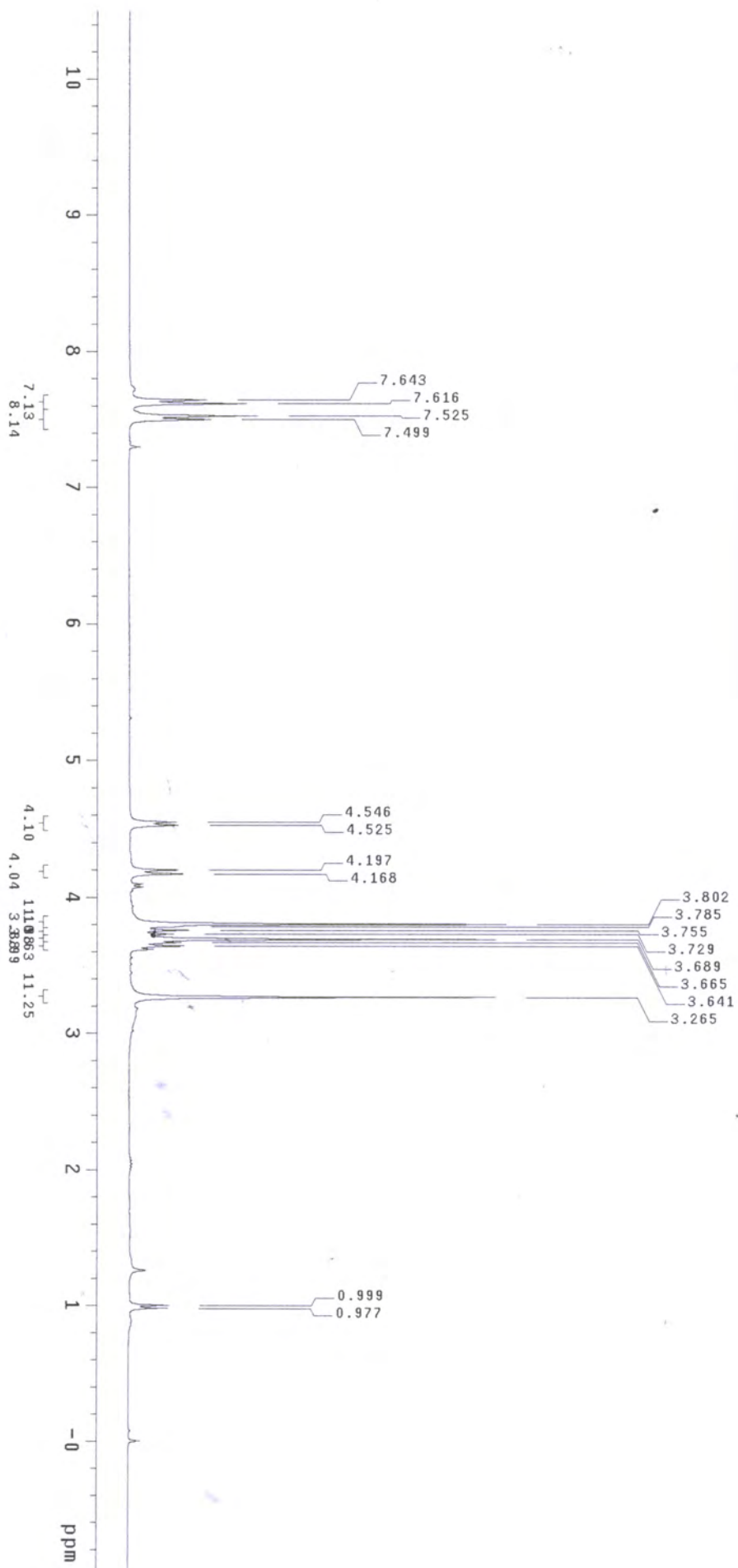
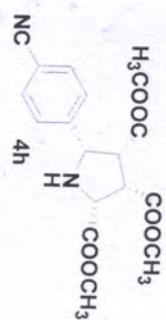




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

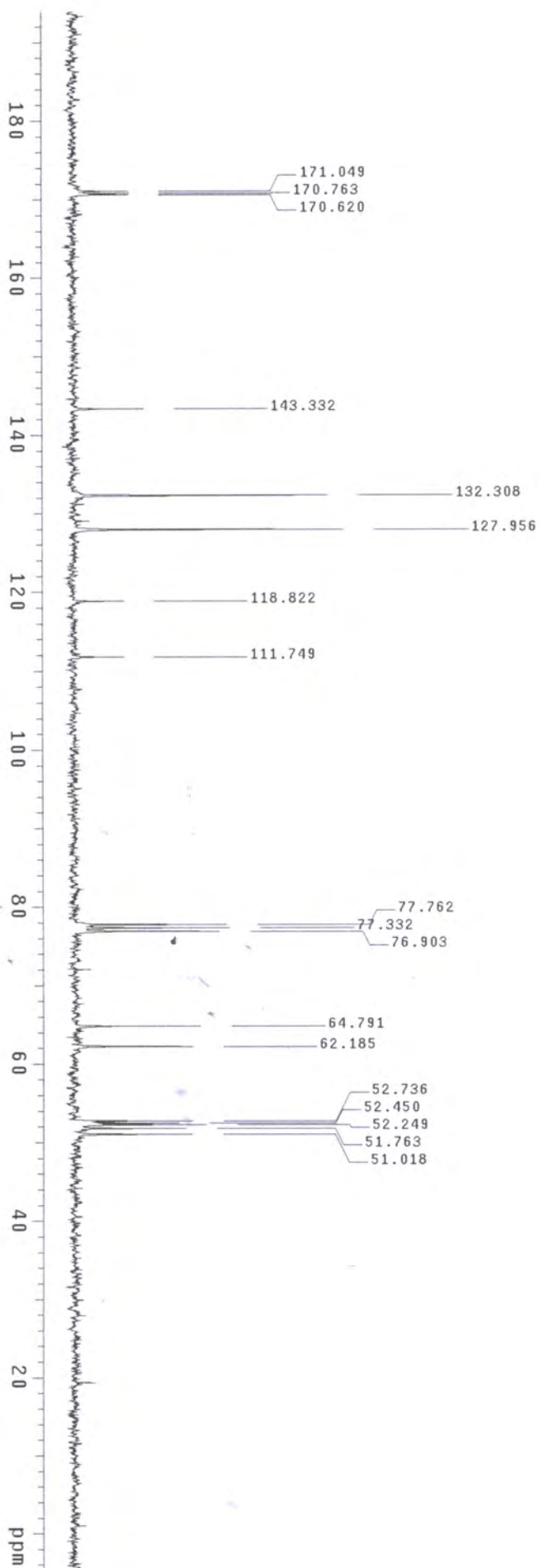
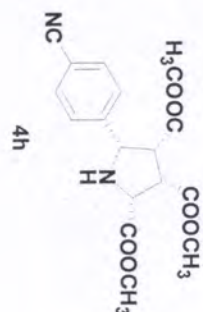
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
376 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FI size 32768
Total time 2 hr, 24 min, 23 sec

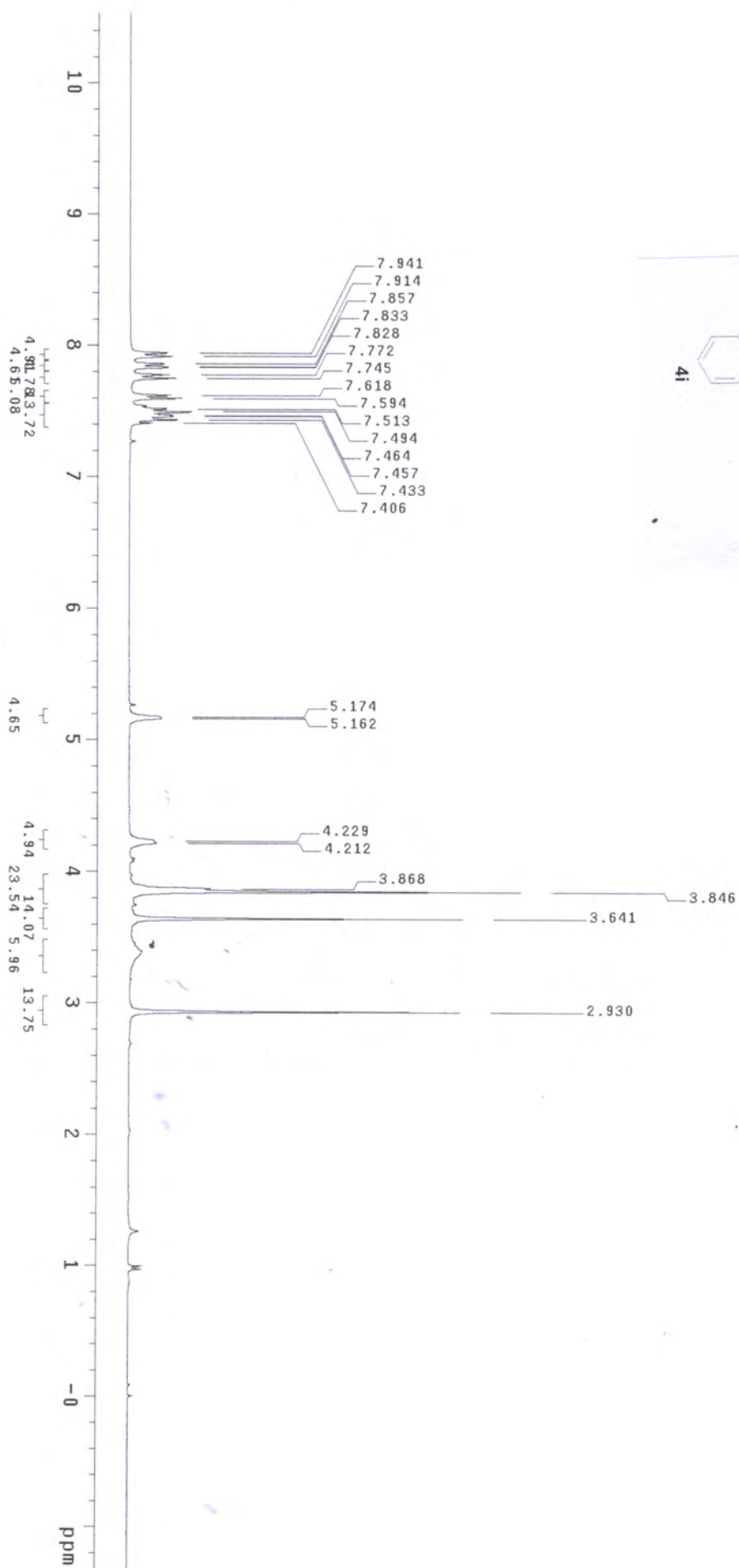




Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"

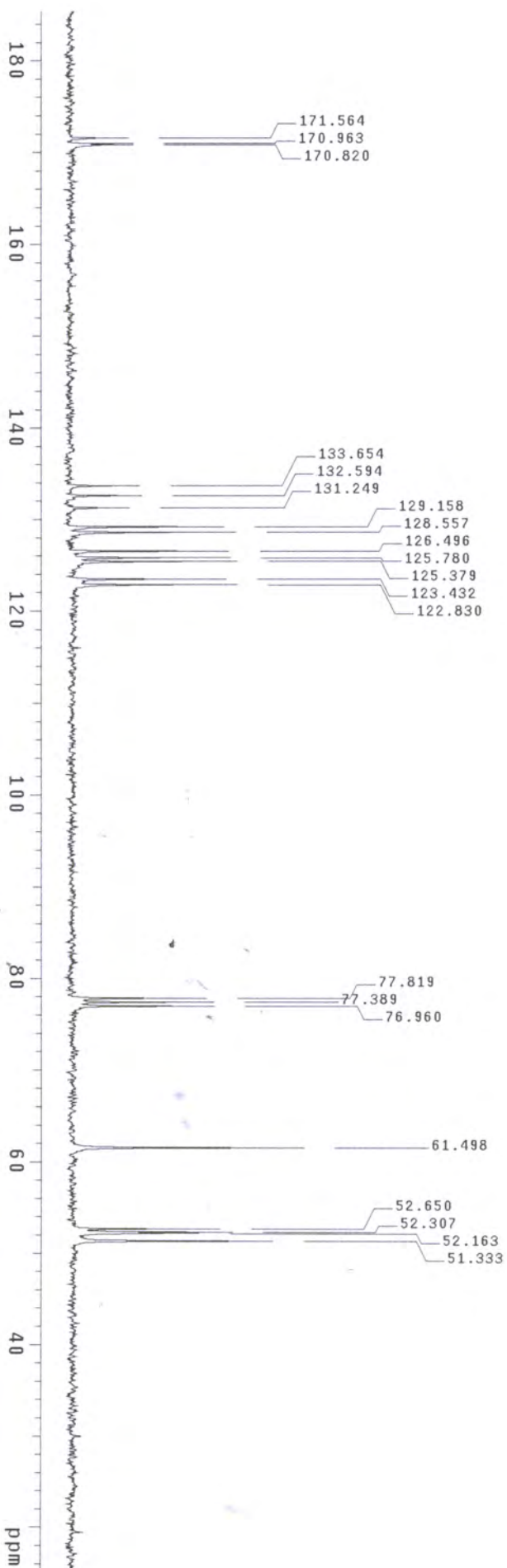
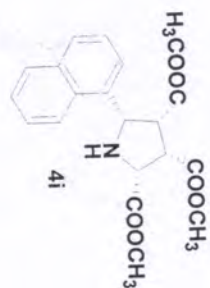
Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.450 sec
 Width 1769.1 Hz
 160 repetitions
 OBSERVE C13, 75.4552576 MHz
 DECOUPLE H1, 300.0807172 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 FI size 16384
 Total time 8 hr, 51 min, 4 sec

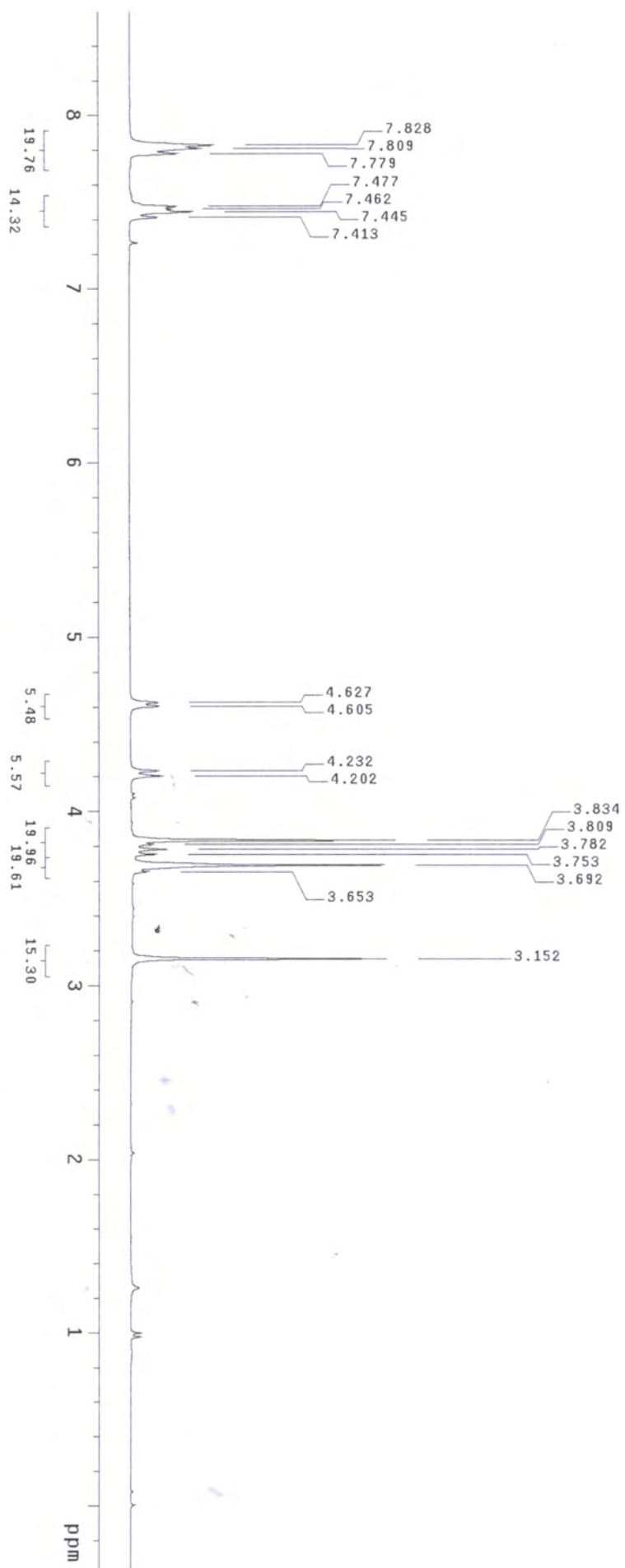
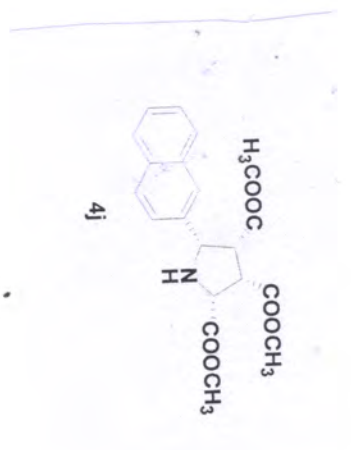




Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"

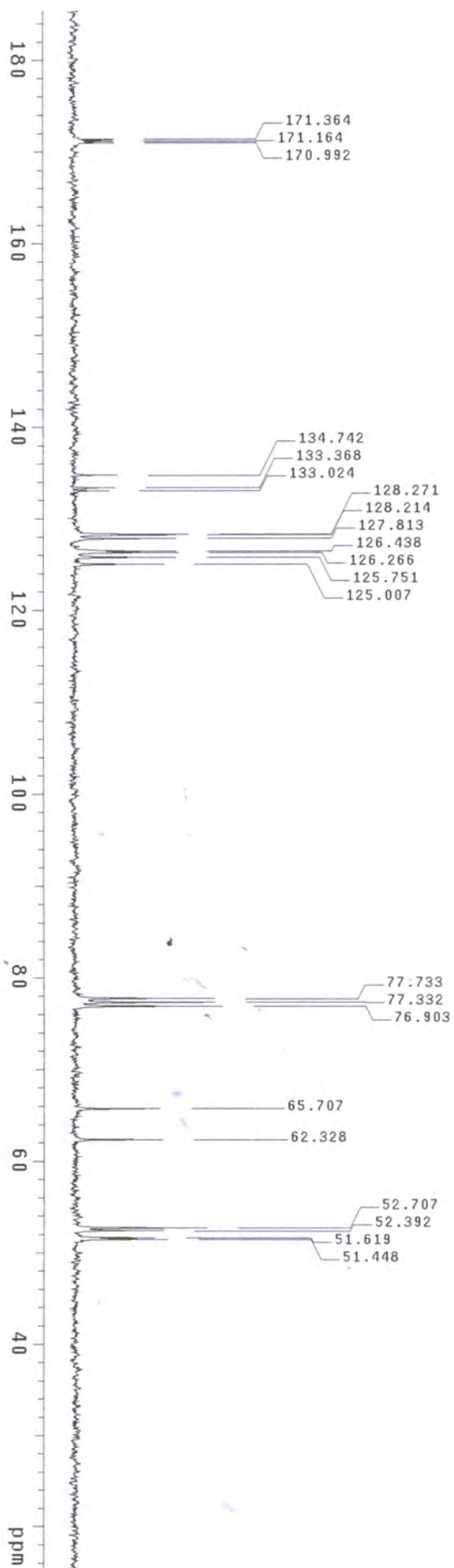
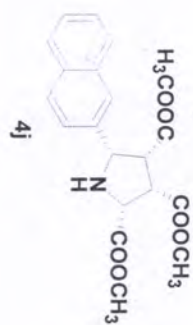
Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.450 sec
 Width 17699.1 Hz
 144 repetitions
 OBSERVE C13, 75.4552576 MHz
 DECOUPLE H1, 300.0807172 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 FT size 16384
 Total time 8 hr, 51 min, 4 sec

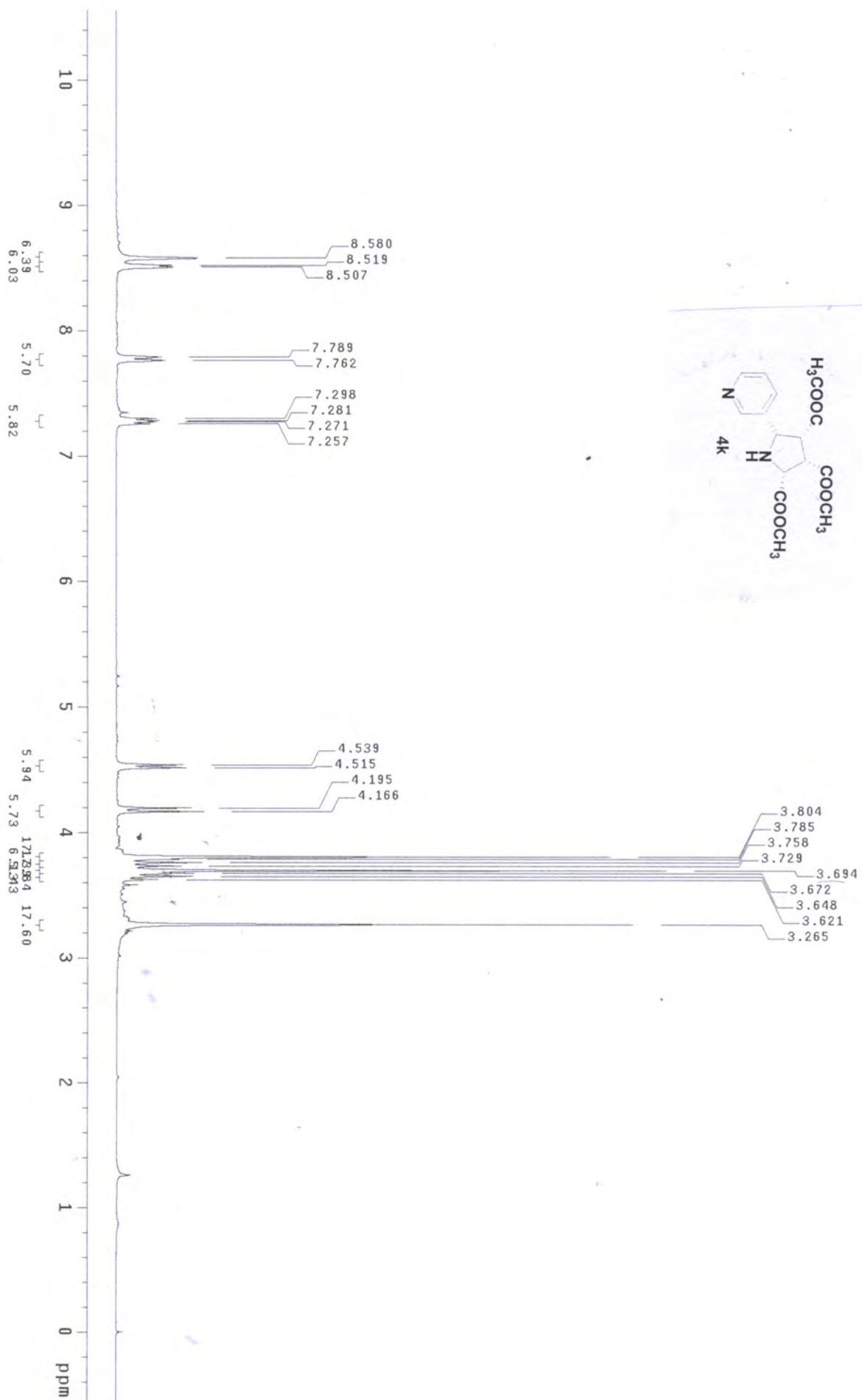




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

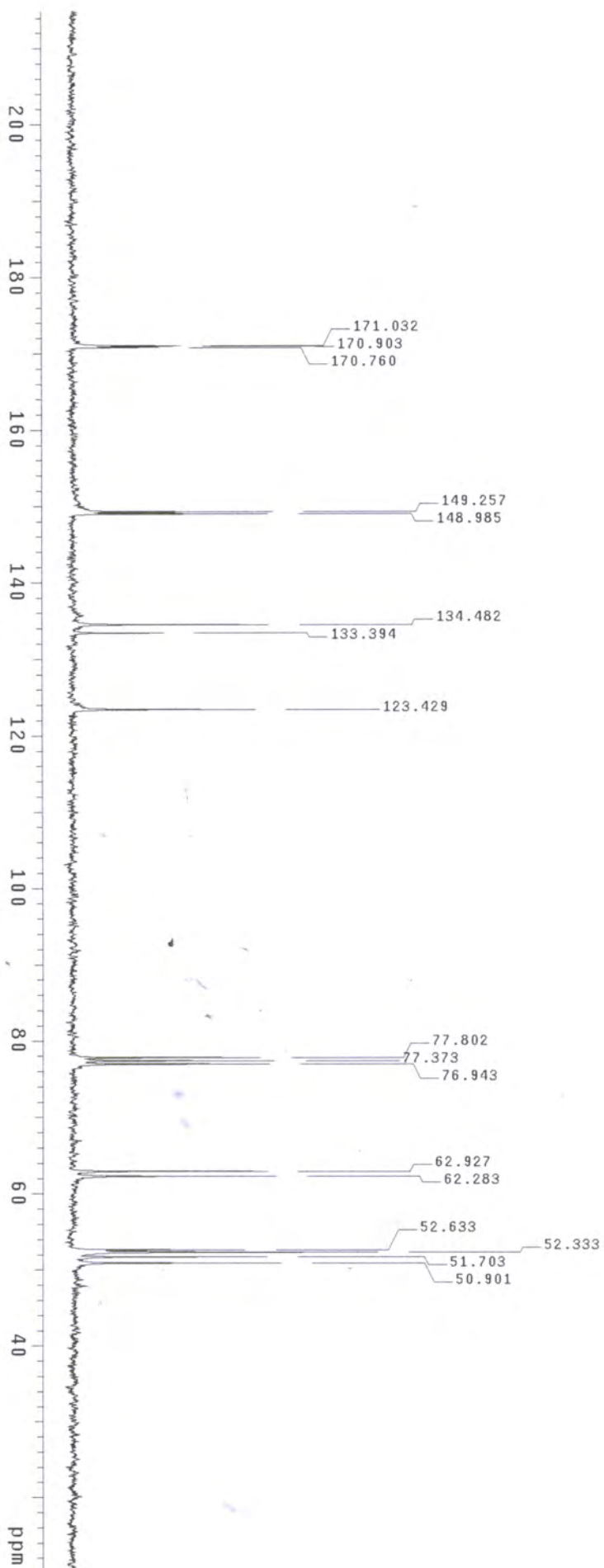
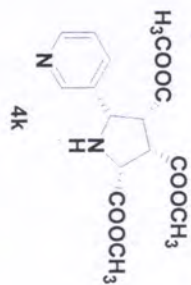
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.450 sec
Width 17599.1 Hz
208 repetitions
OBSERVE C13, 75.4552576 MHz
DECUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 4.0 Hz
FT size 16384
Total time 8 hr, 51 min, 4 sec

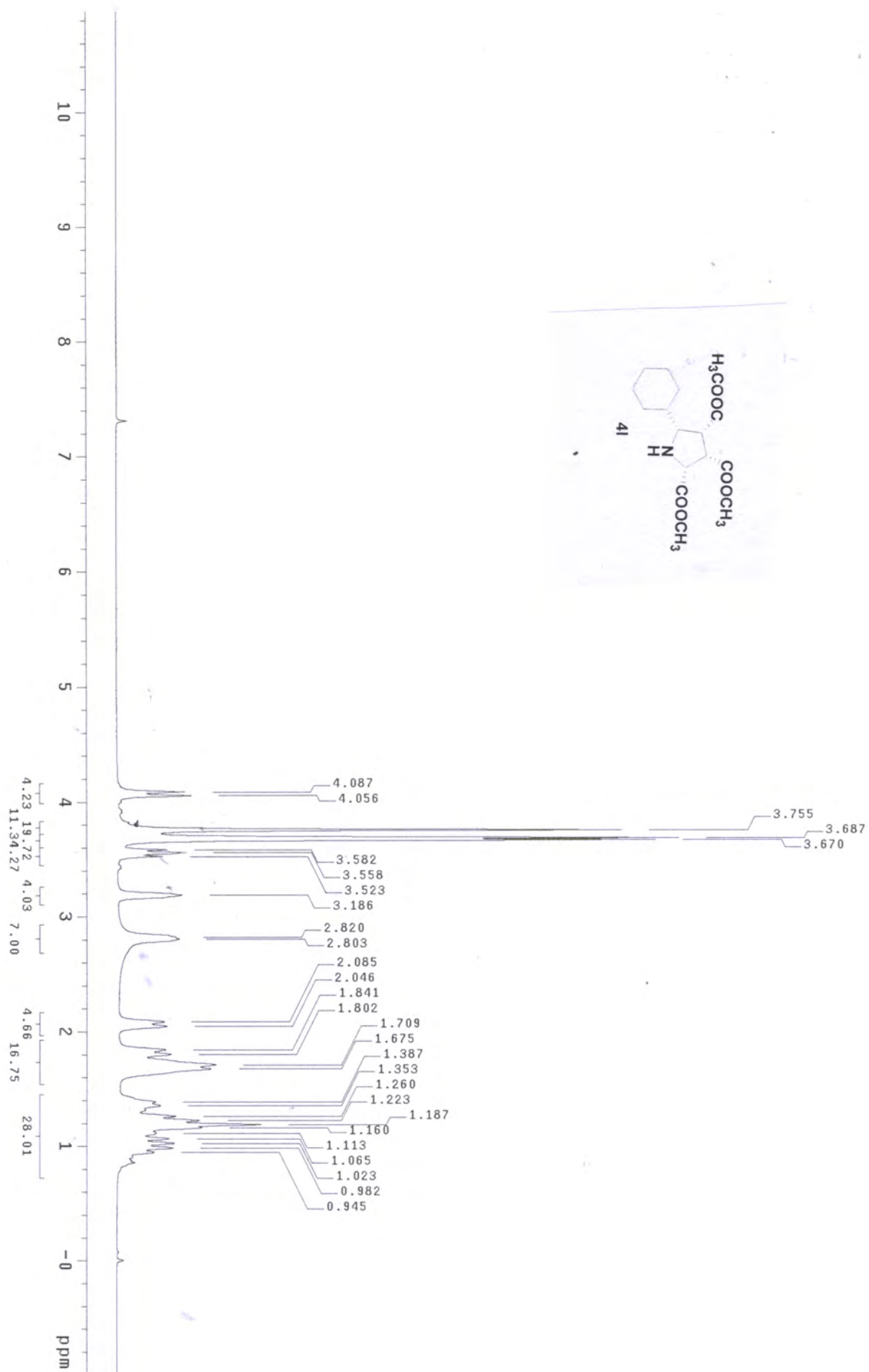




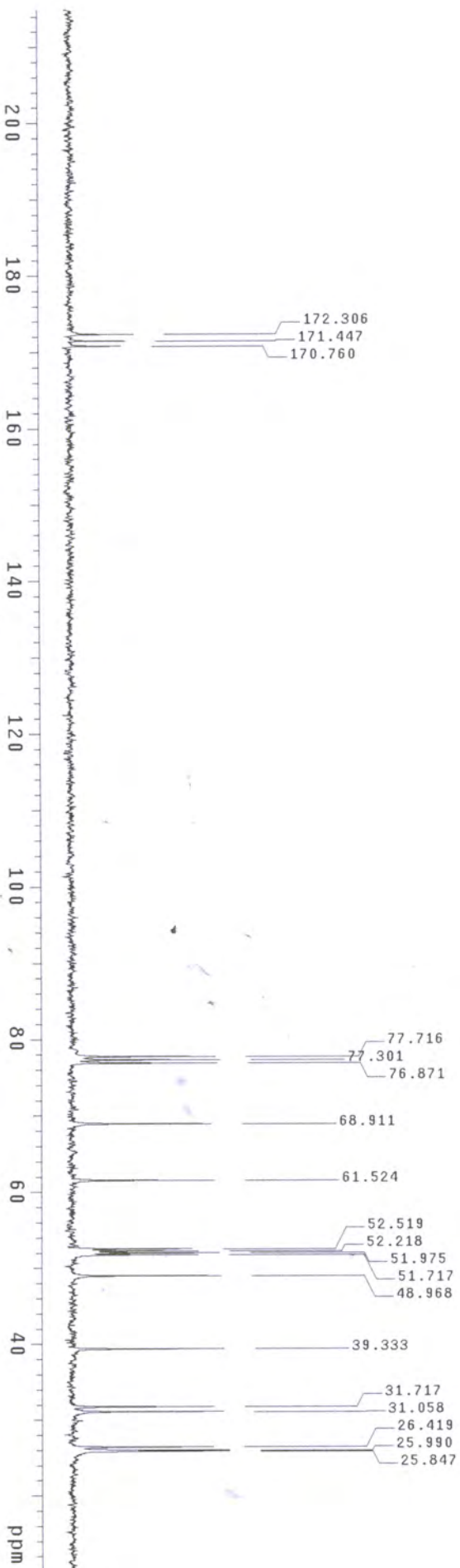
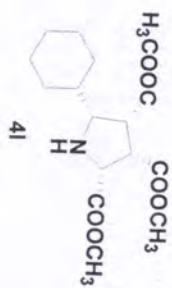
Solvent: DMSO
Ambient temperature
Mercury-300BB "mercury300"

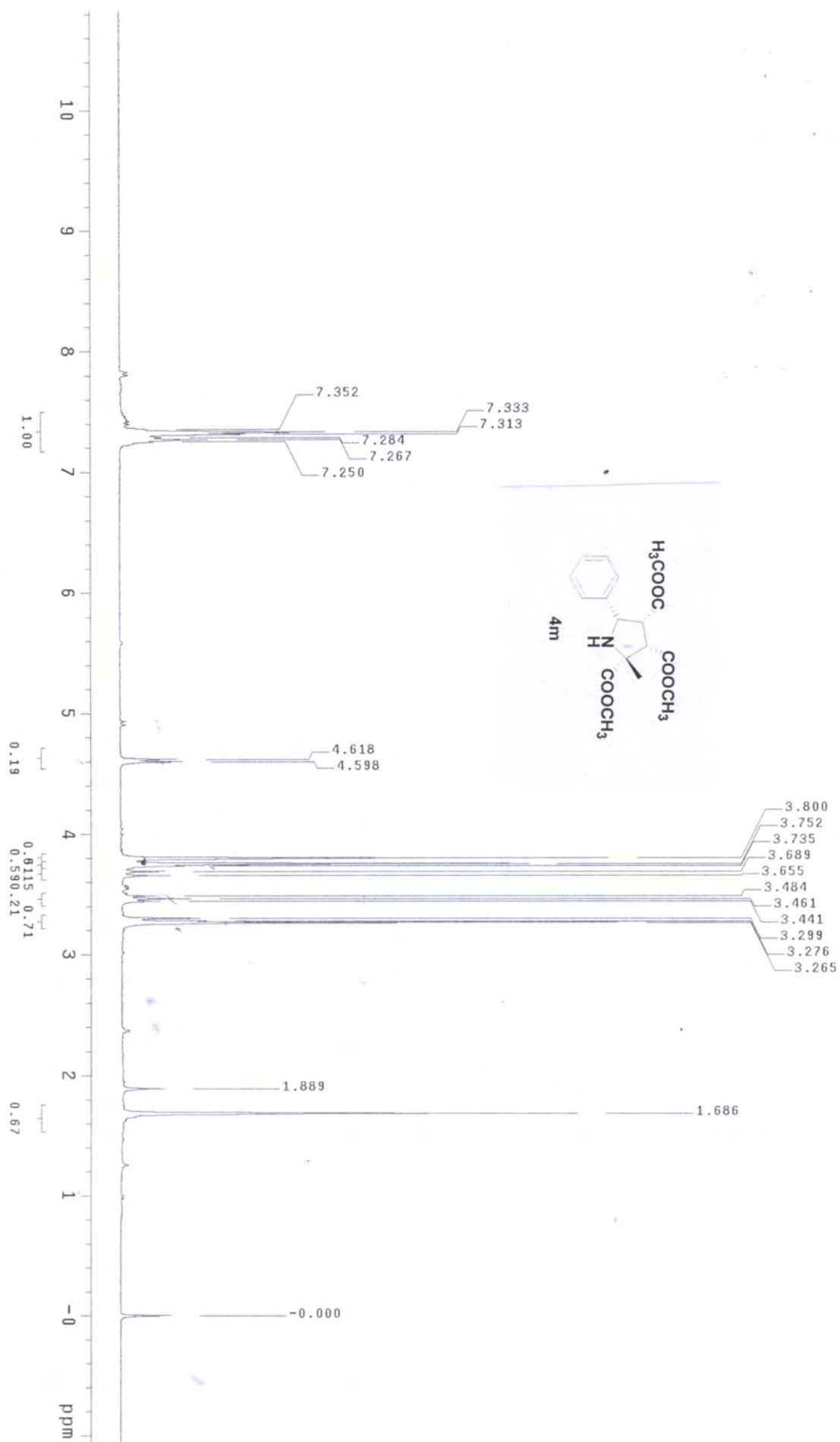
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
208 repetitions
OBSERVE C13, 75.4552576 MHz
DECOUPLE H1, 300.0821426 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
Ft size 32768
Total time 9 hr, 10 min, 3 sec





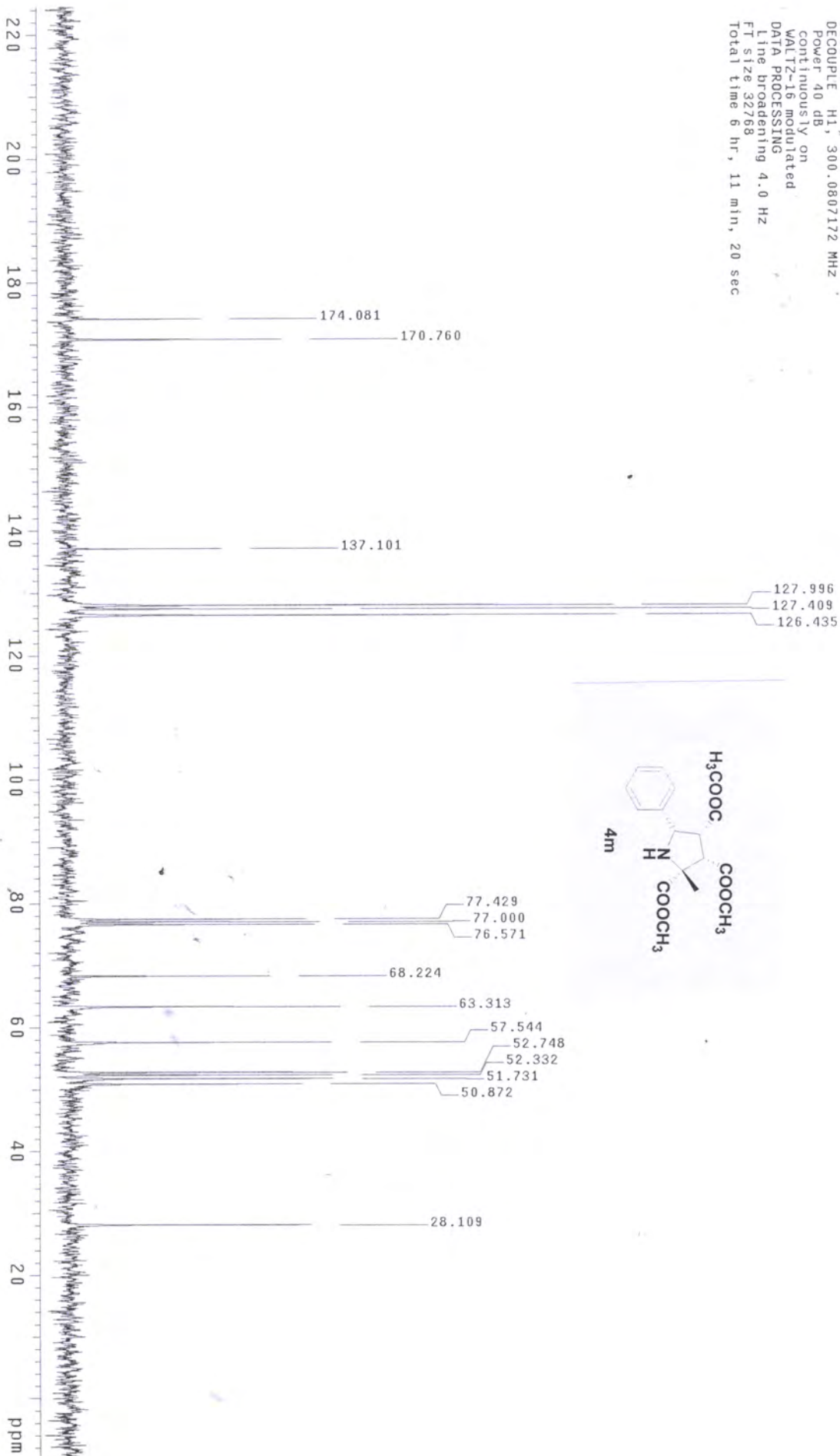
Solvent: DMSO
 Ambient temperature
 Mercury-300BB "mercury300"
 Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.500 sec
 Width 17699.1 Hz
 208 repetitions
 OBSERVE C13, 75.4552576 MHz
 DECOUPLE H1, 300.0821426 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 FT size 32768
 Total time 9 hr, 10 min, 3 sec

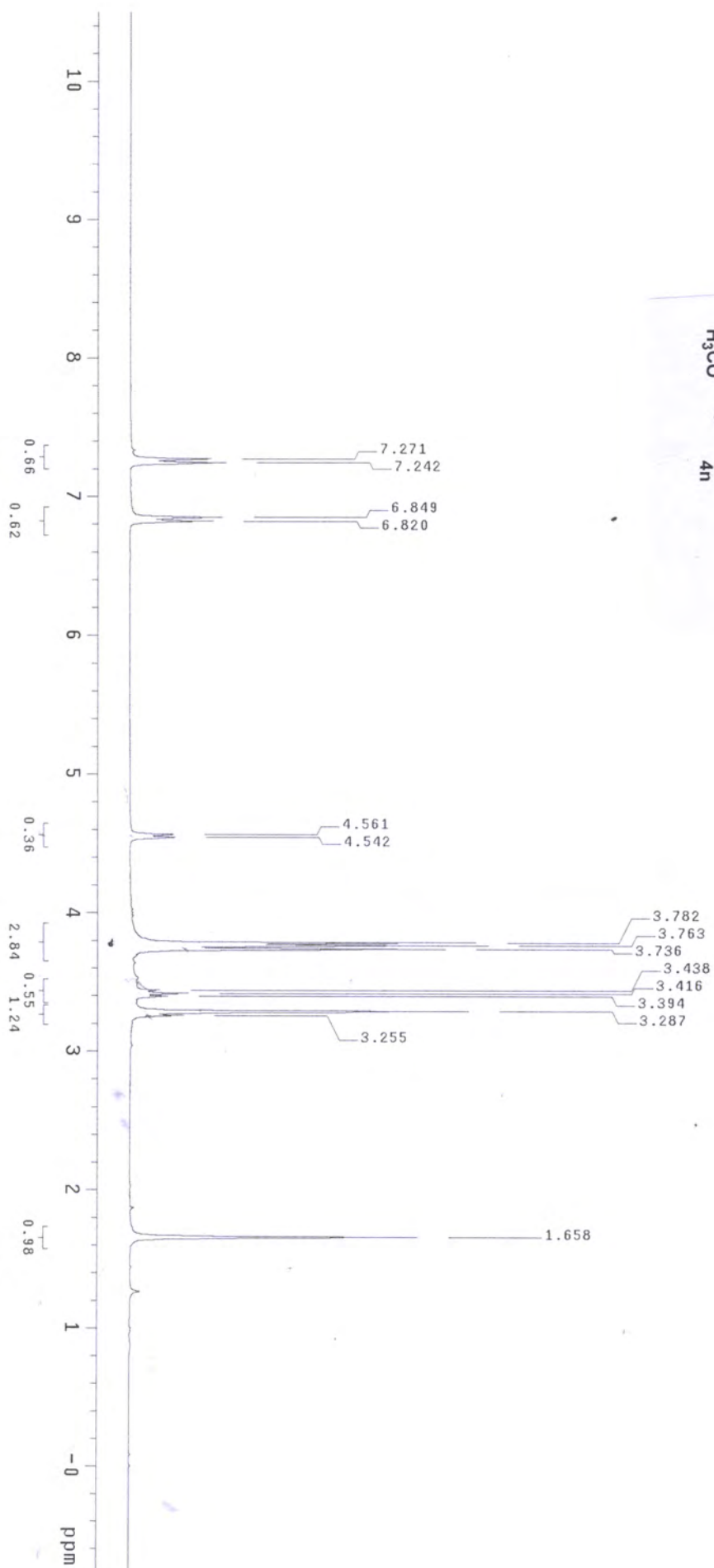




Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"

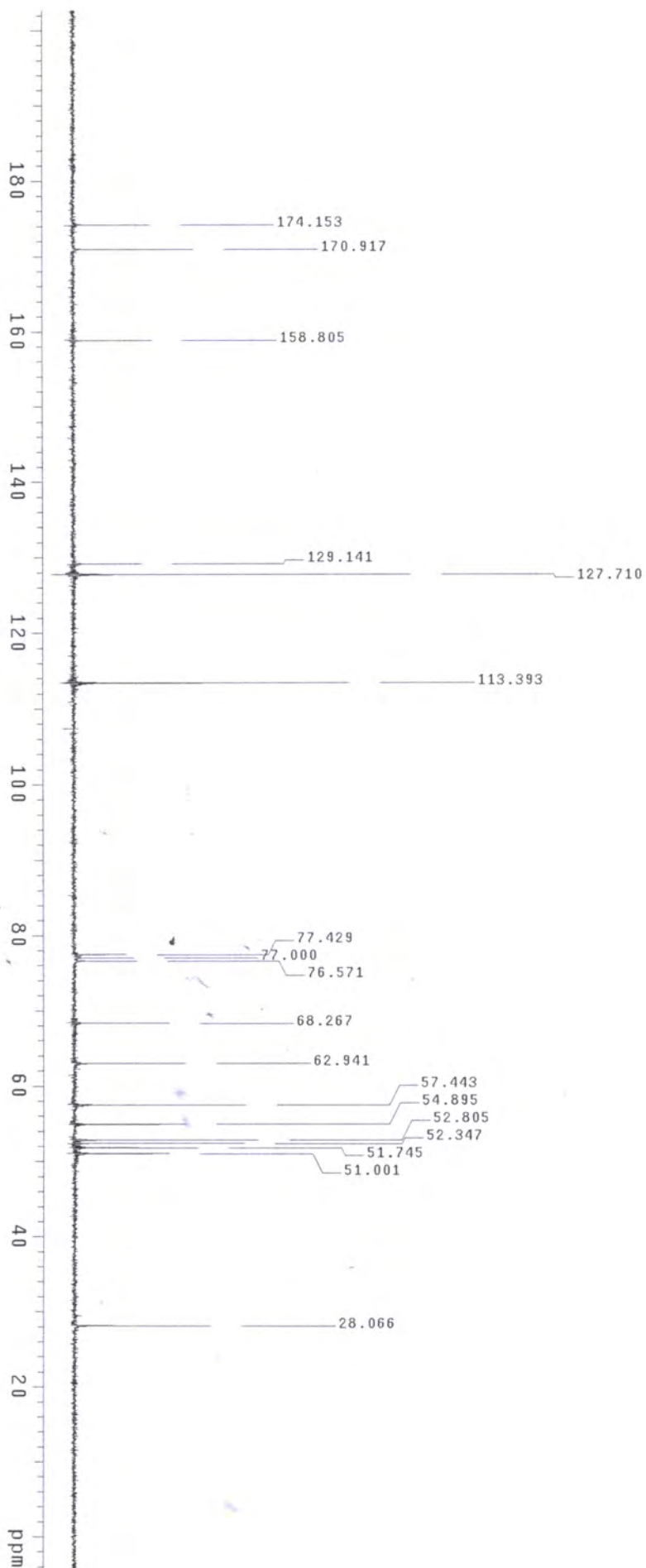
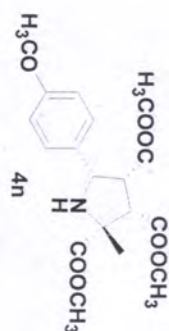
Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.500 sec
 Width 17699.1 Hz
 10240 repetitions
 OBSERVE C13, 75.4552945 MHz
 DECOUPLE H1, 300.0807172 MHz
 Power 40 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 FT size 32768
 Total time 6 hr, 11 min, 20 sec

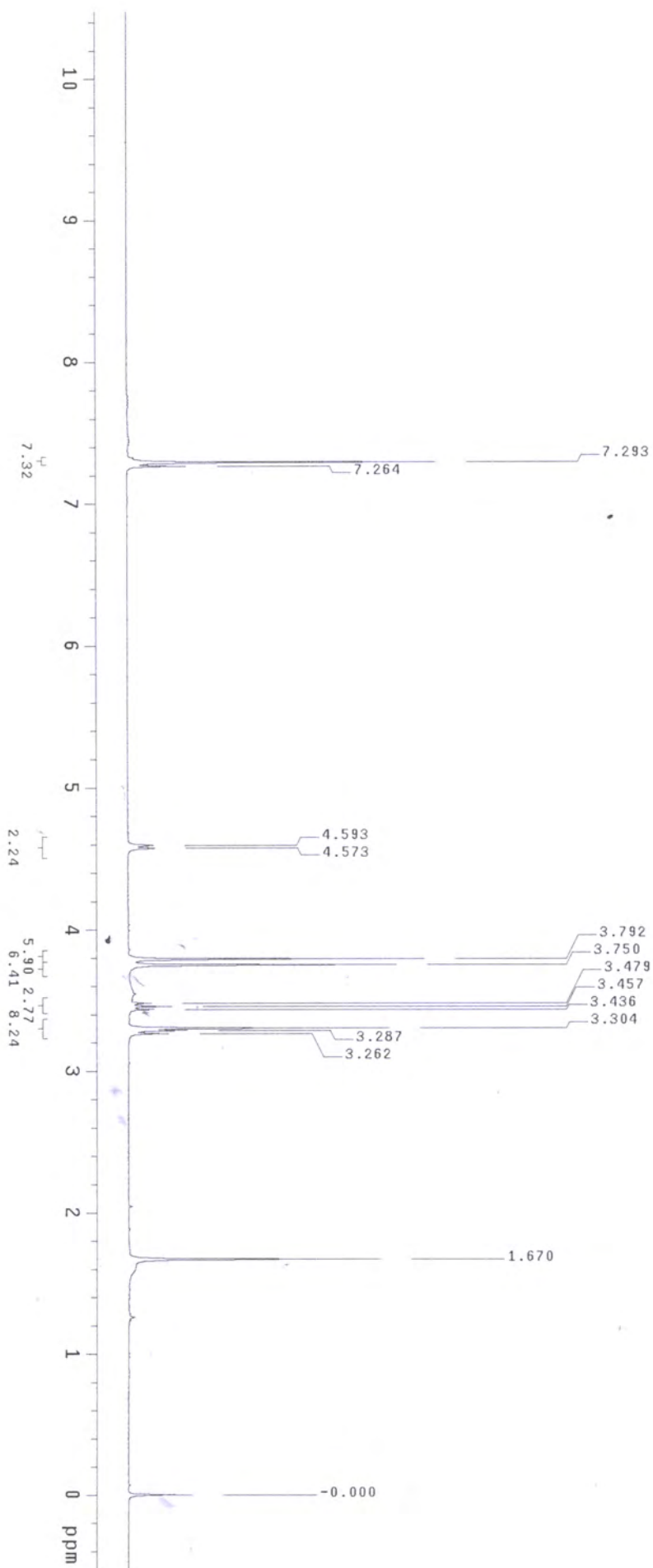
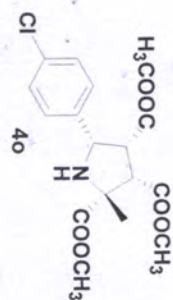




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

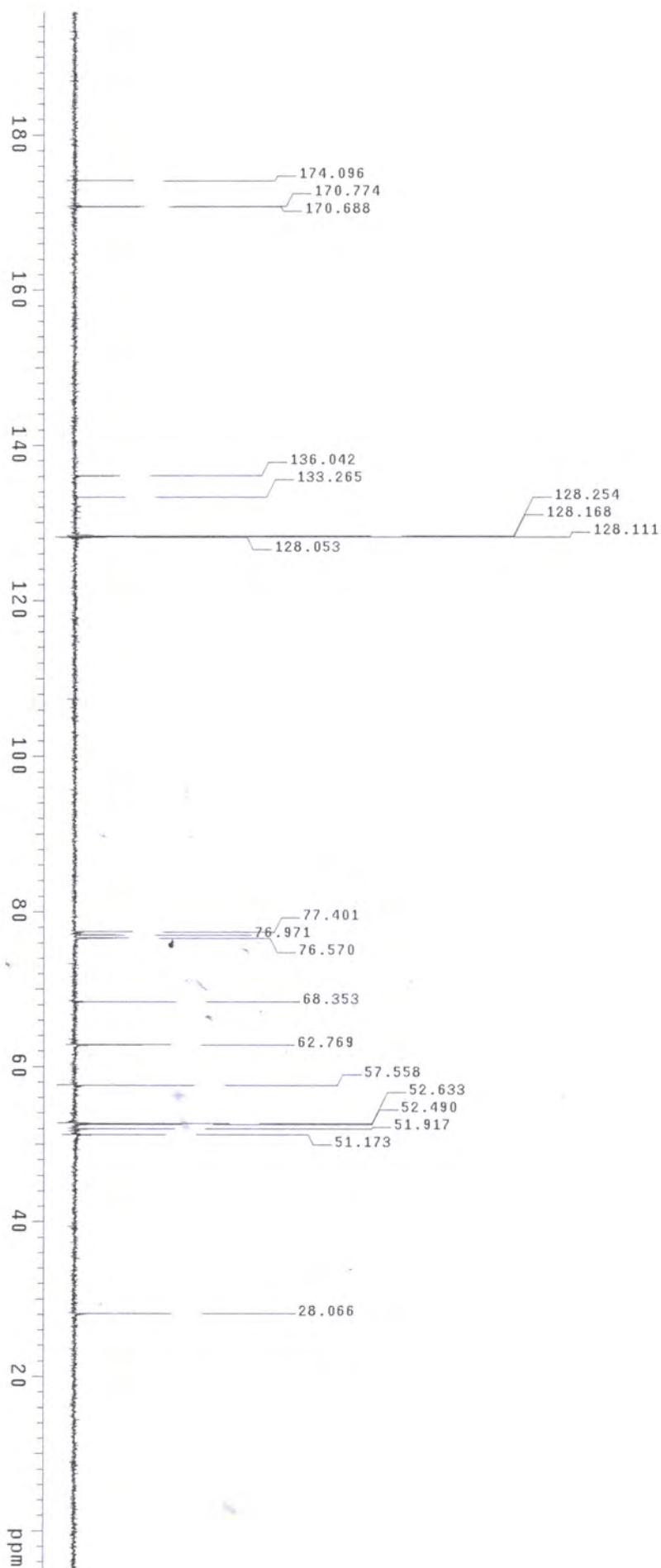
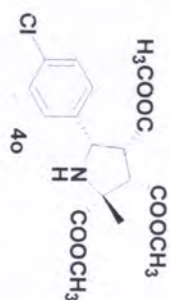
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.300 sec
Width 17699.1 Hz
144 repetitions
OBSERVE C13; 75.4552913 MHz
DECOUPLE H1; 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
F1 size 16384
Total time 49 min, 8 sec

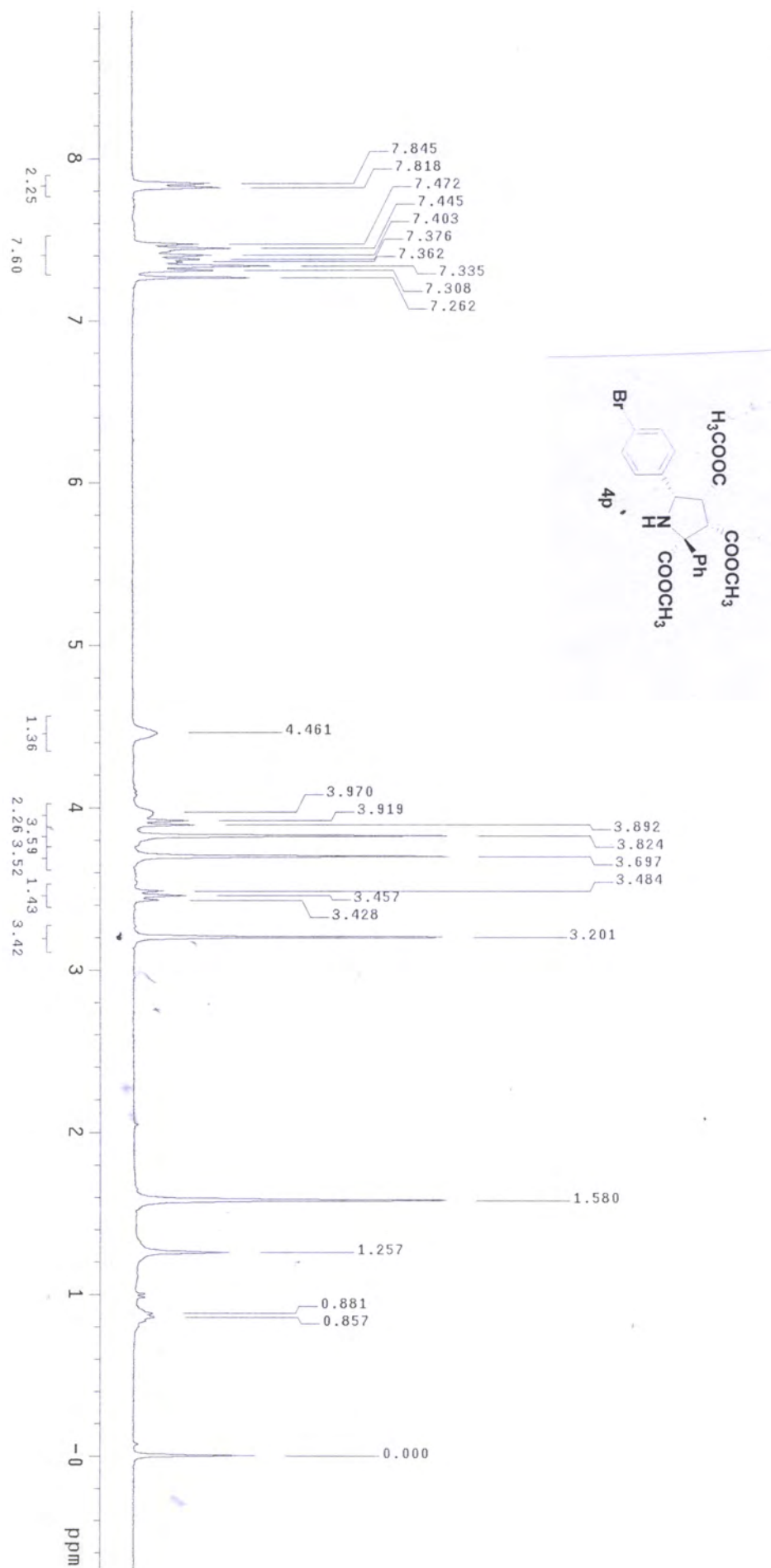




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

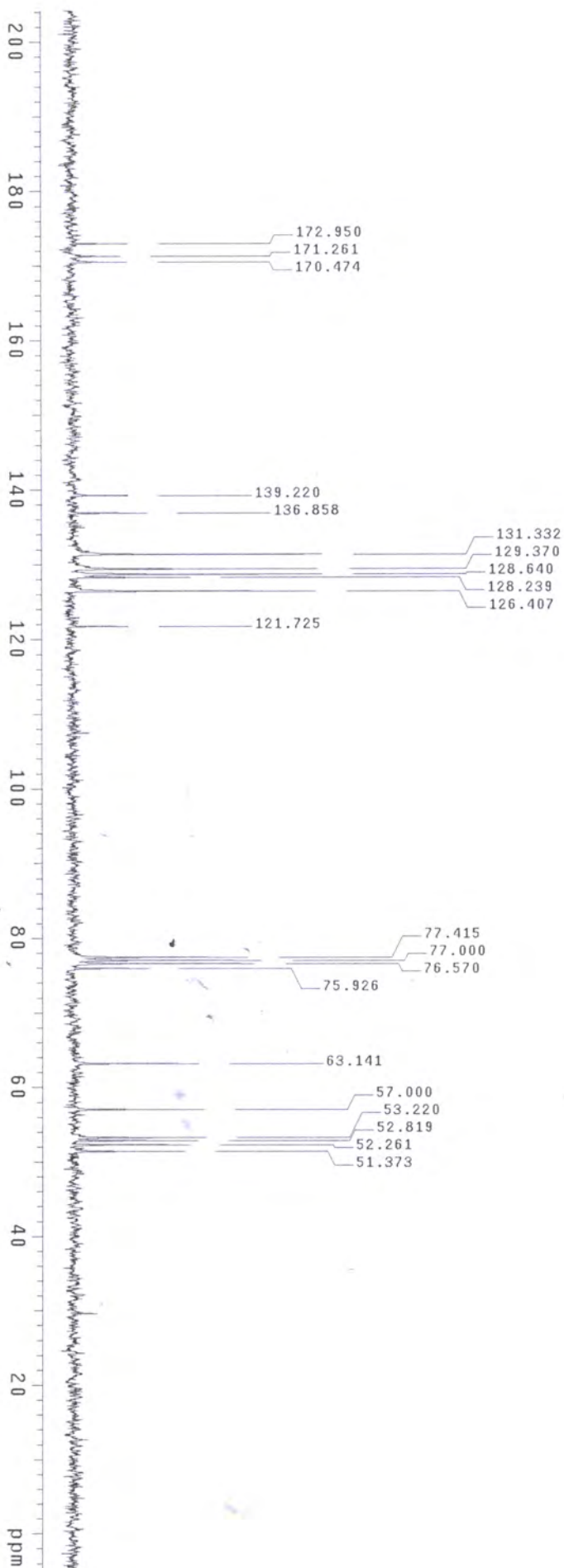
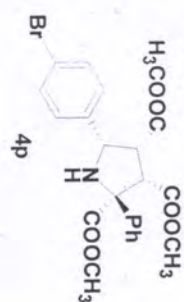
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.300 sec
Width 17599.1 Hz
144 repetitions
OBSERVE C13, 75.4552869 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 16384
Total time 49 min, 8 sec

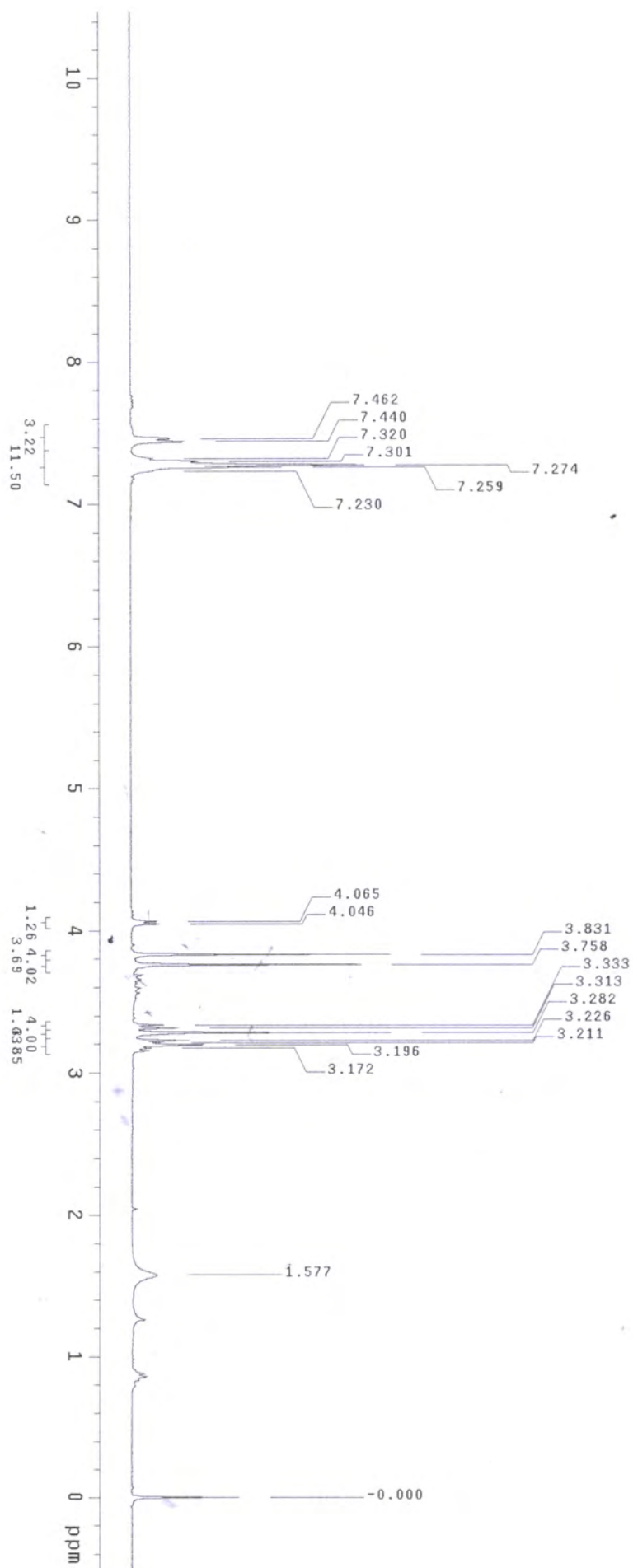




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

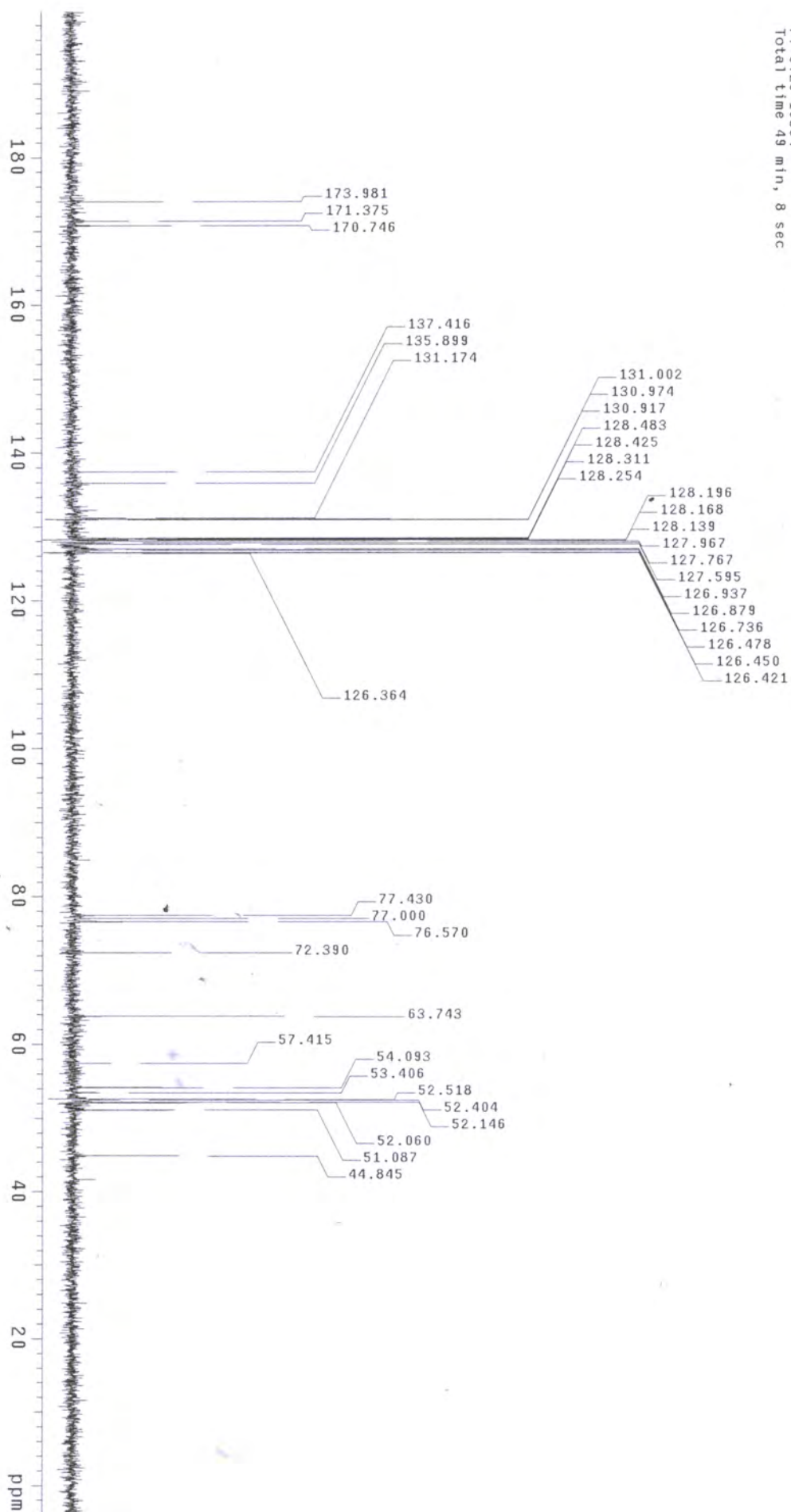
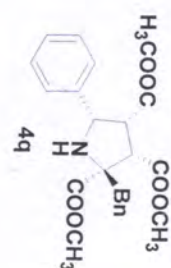
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17693.1 Hz
10240 repetitions
OBSERVE C13, 75.4552826 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 6 hr, 11 min, 20 sec

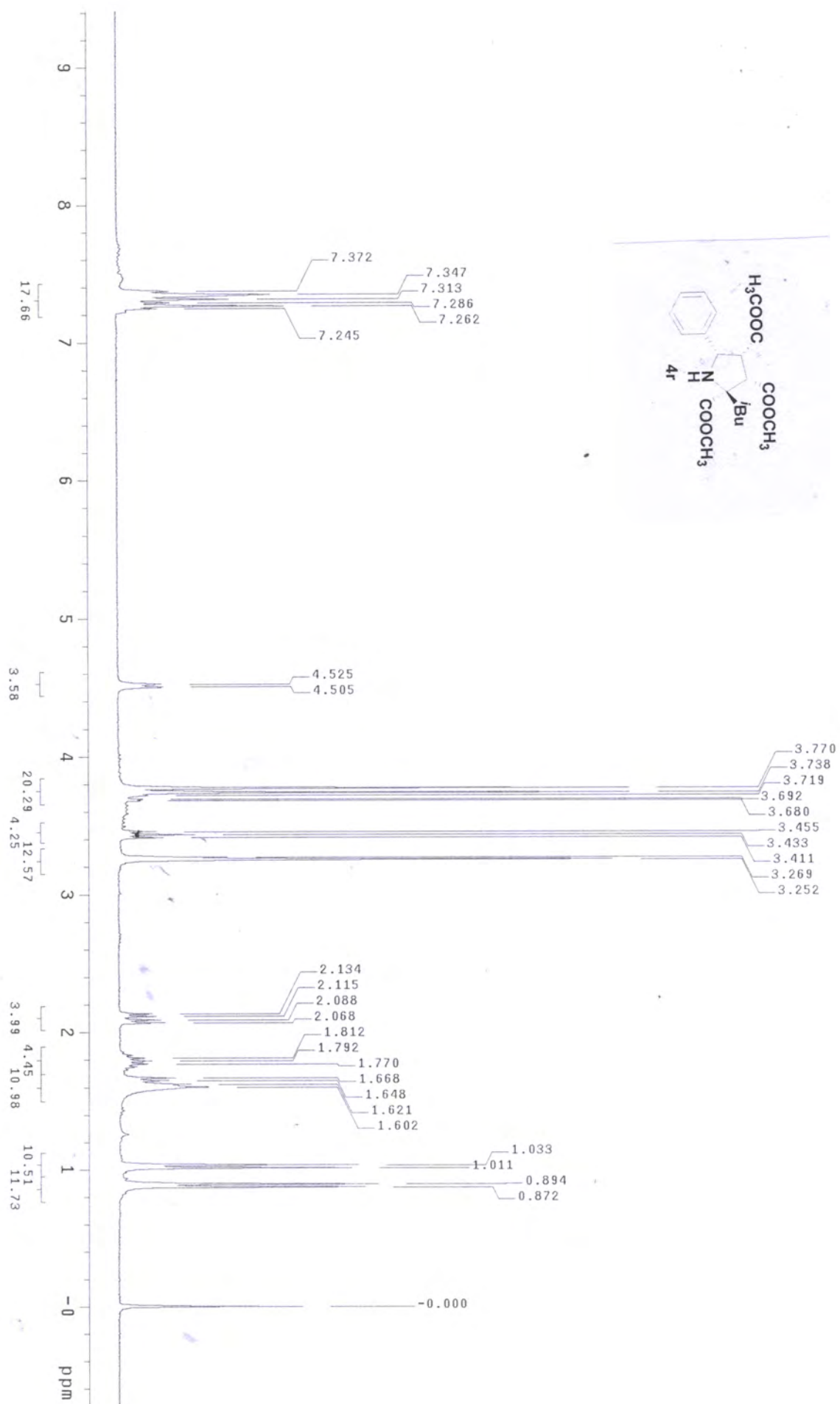




Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"

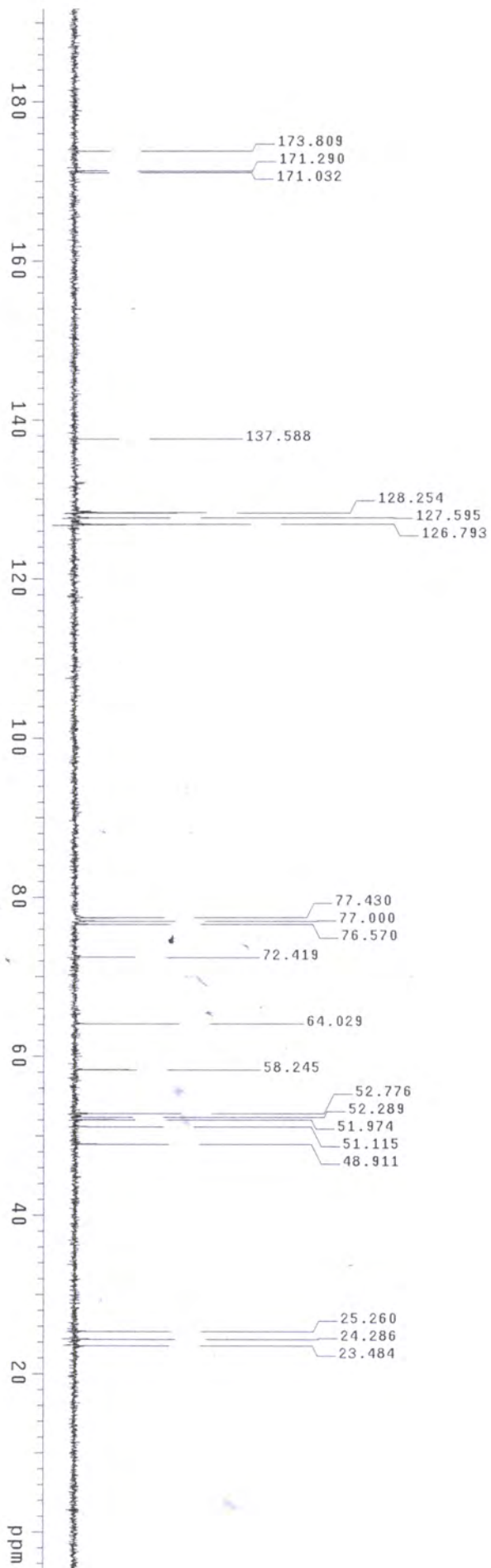
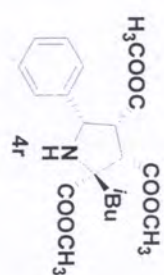
Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.300 sec
 Width 17699.1 Hz
 112 repetitions
 OBSERVE C13; 75.4552848 MHz
 DECOUPLE H1; 300.0807172 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 FT size 16384
 Total time 49 min, 8 sec

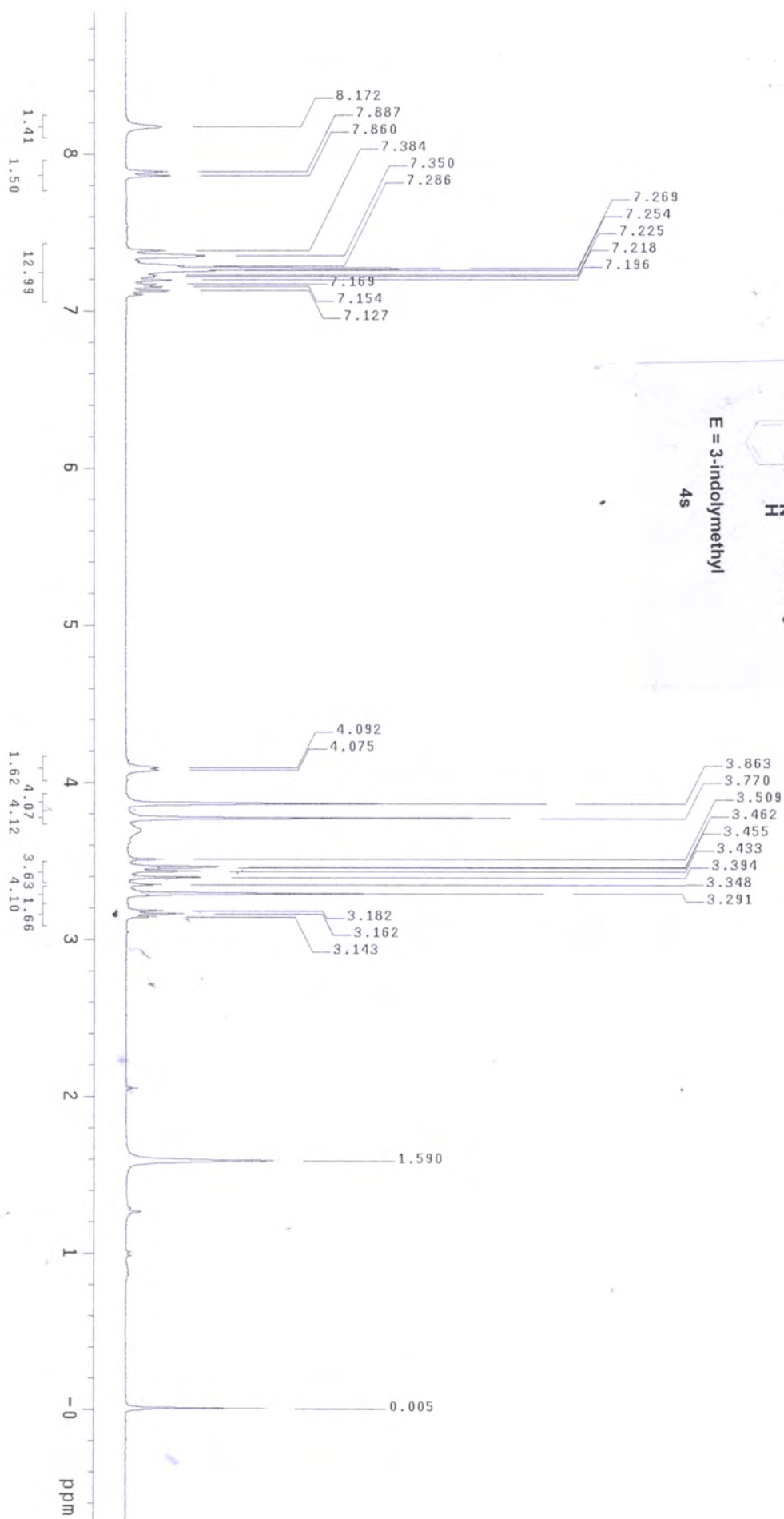




Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"

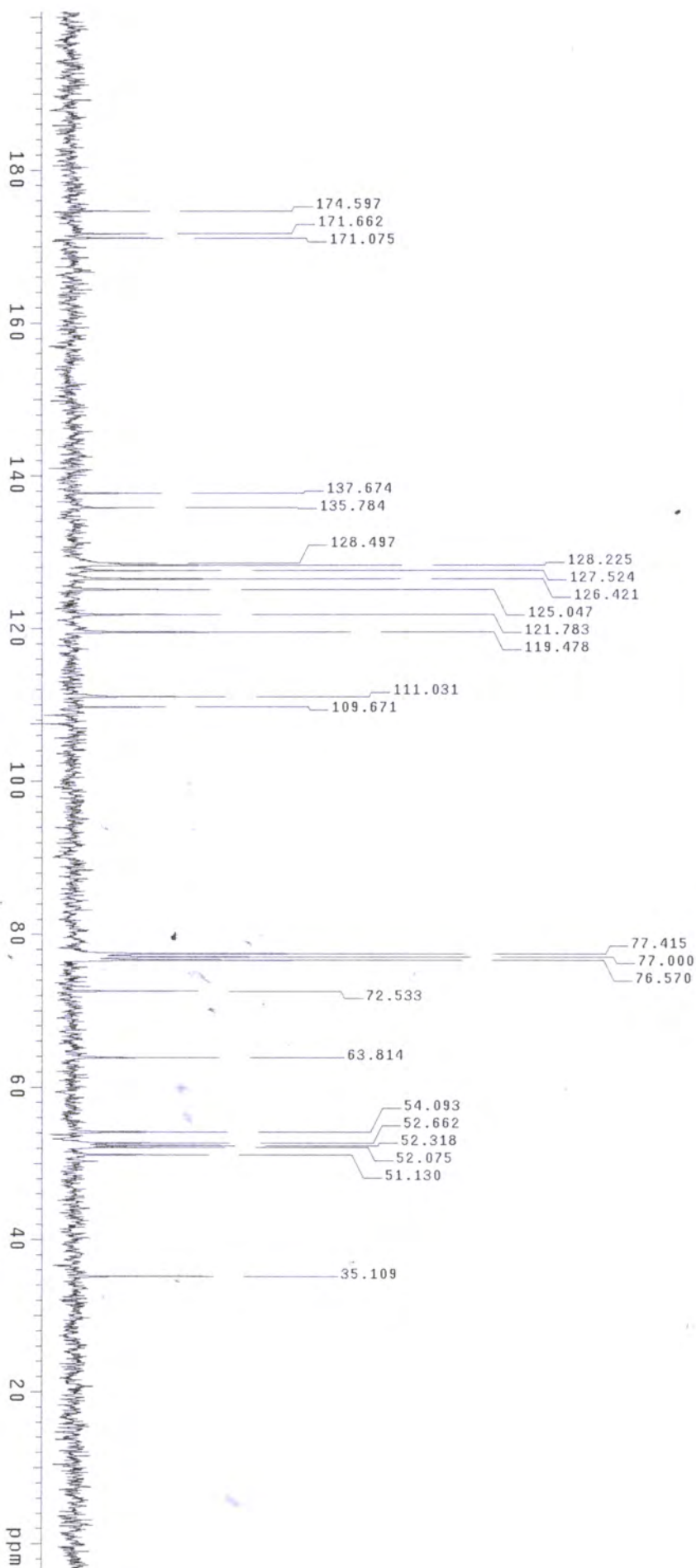
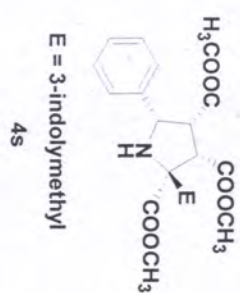
Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.300 sec
 Width 17699.1 Hz
 152 repetitions
 OBSERVE C13; 75.4552805 MHz
 DECOUPLE H1; 300.0807172 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 FT size 16384
 Total time 49 min, 8 sec

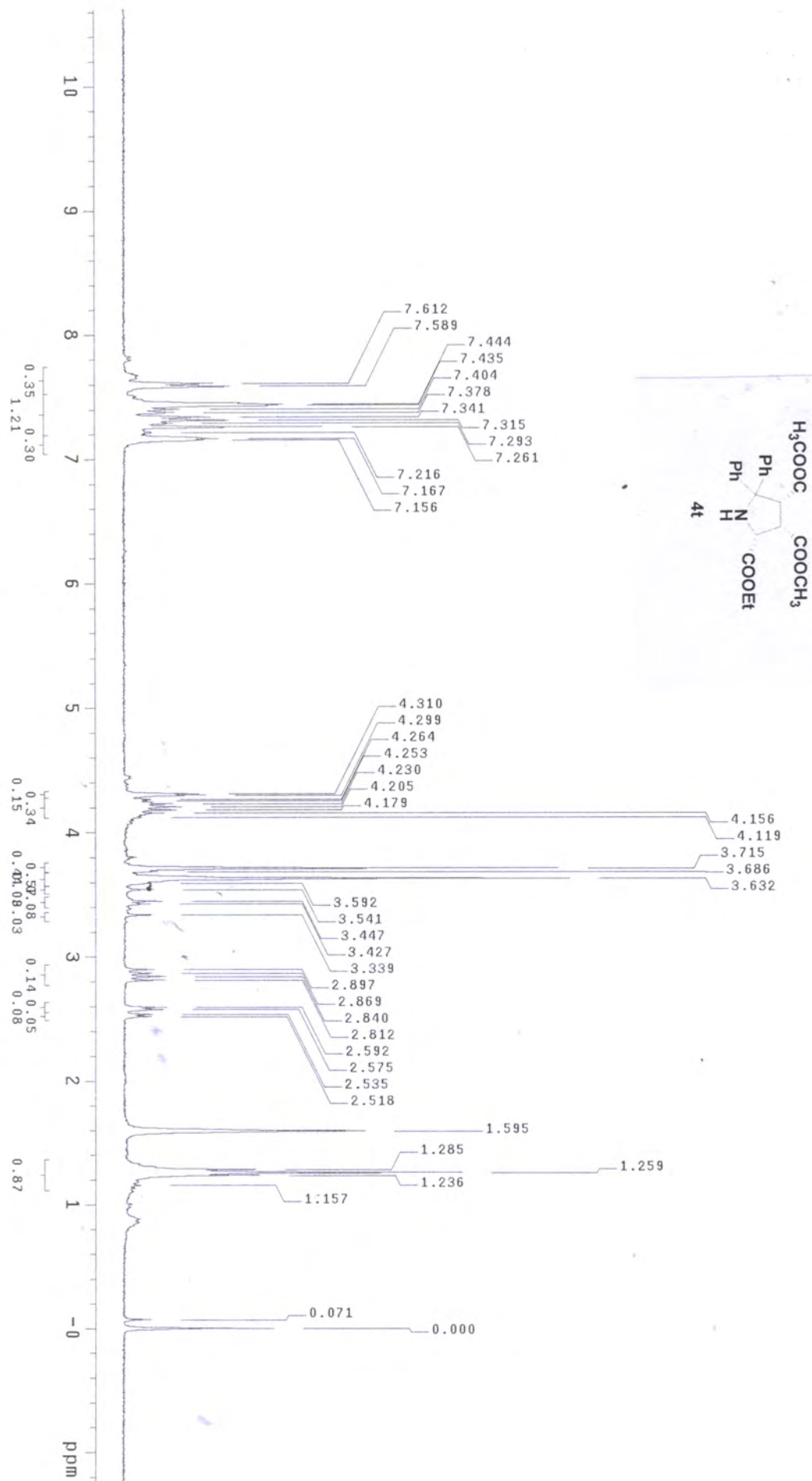




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

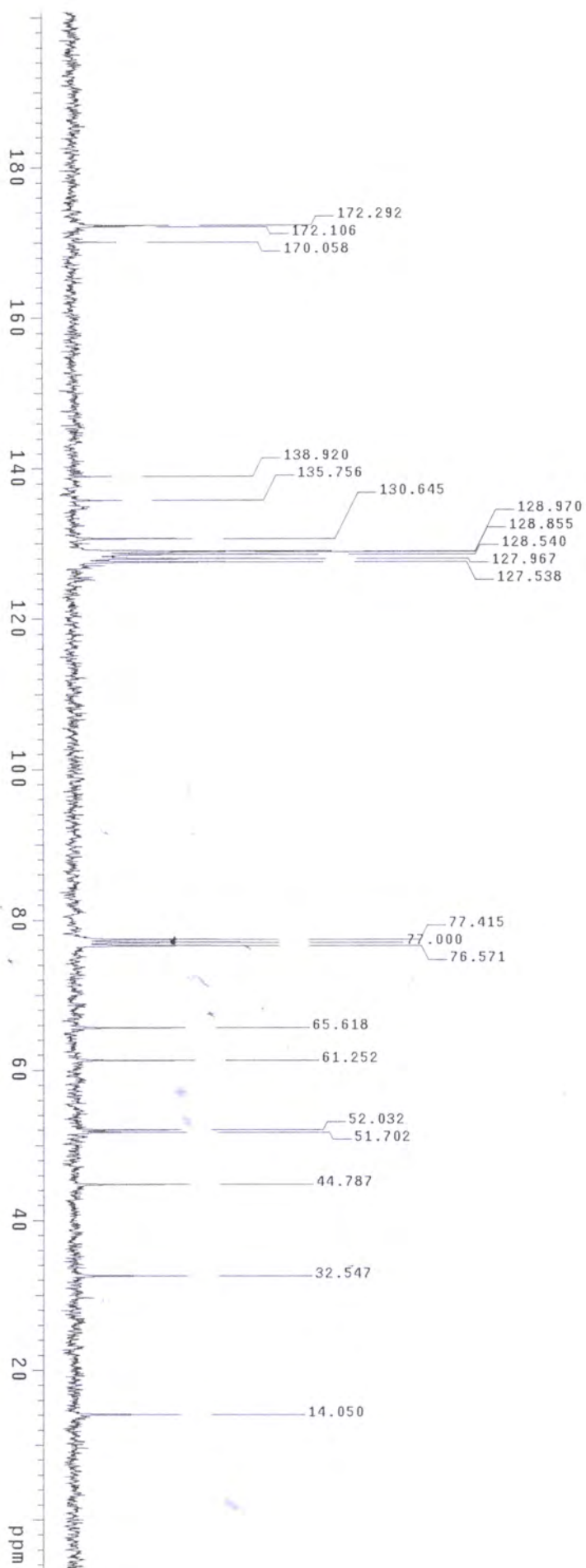
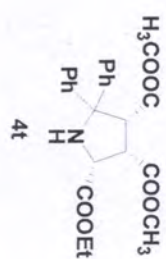
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
10240 repetitions
OBSERVE C13, 75.4552805 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 6 hr, 11 min, 20 sec

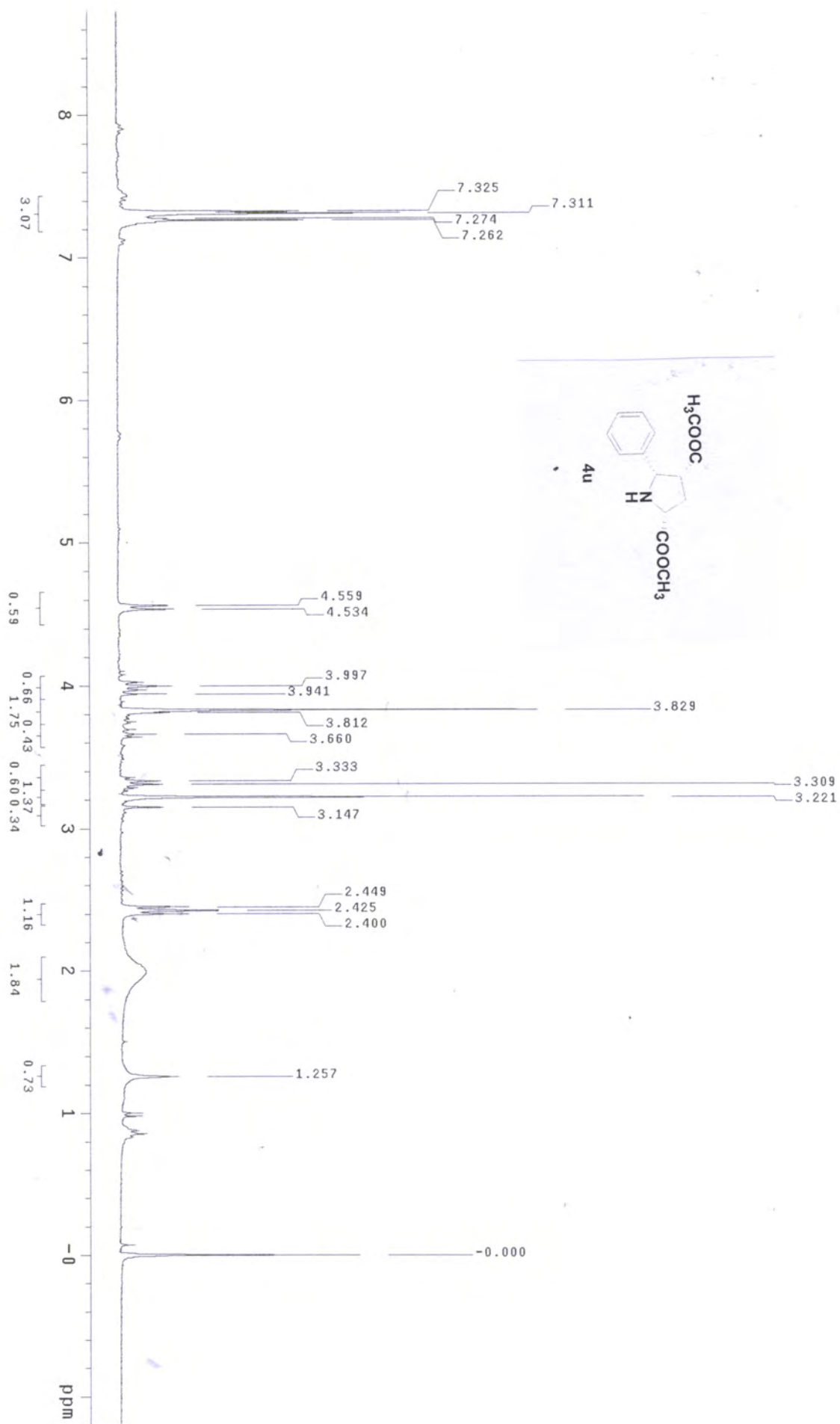




Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"

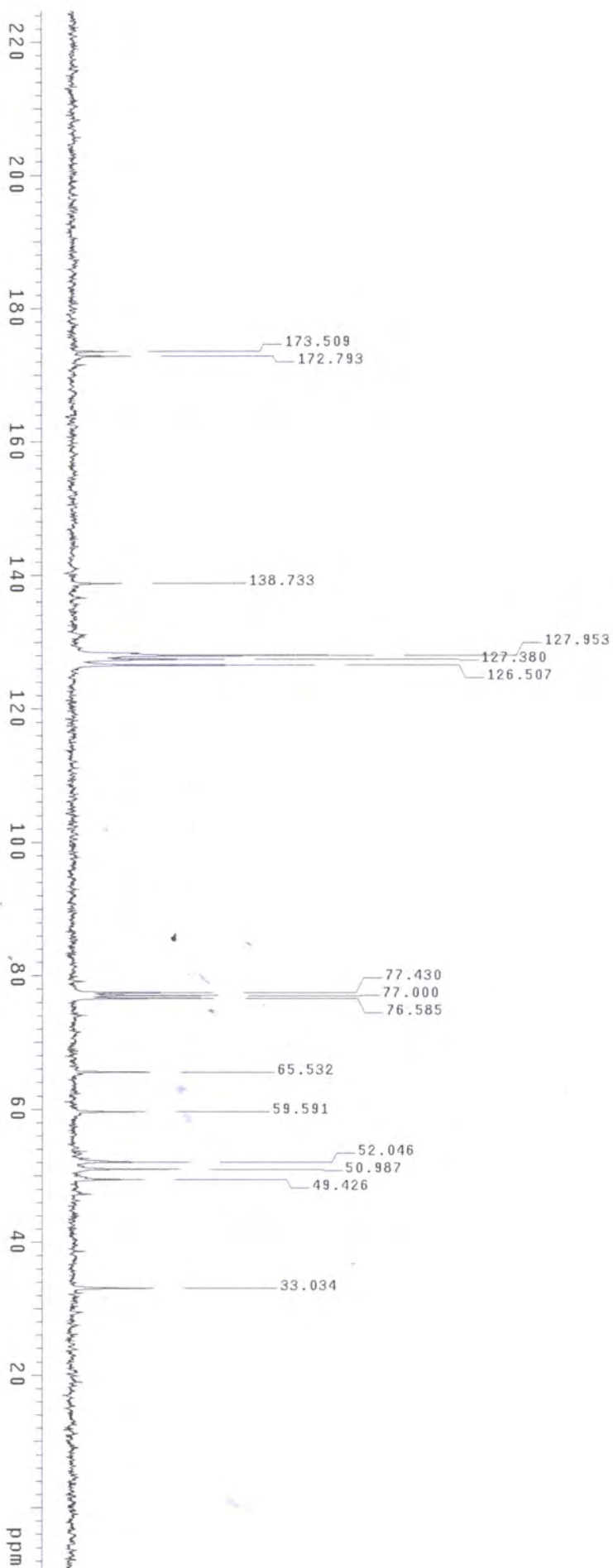
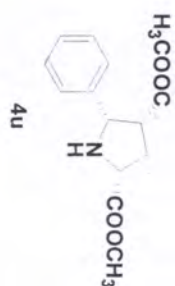
Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.500 sec
 Width 17699.1 Hz
 10240 repetitions
 OBSERVE C13, 75.4552805 MHz
 DECOUPLE H1, 300.0807172 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 FT size 32768
 Total time 6 hr, 11 min, 20 sec

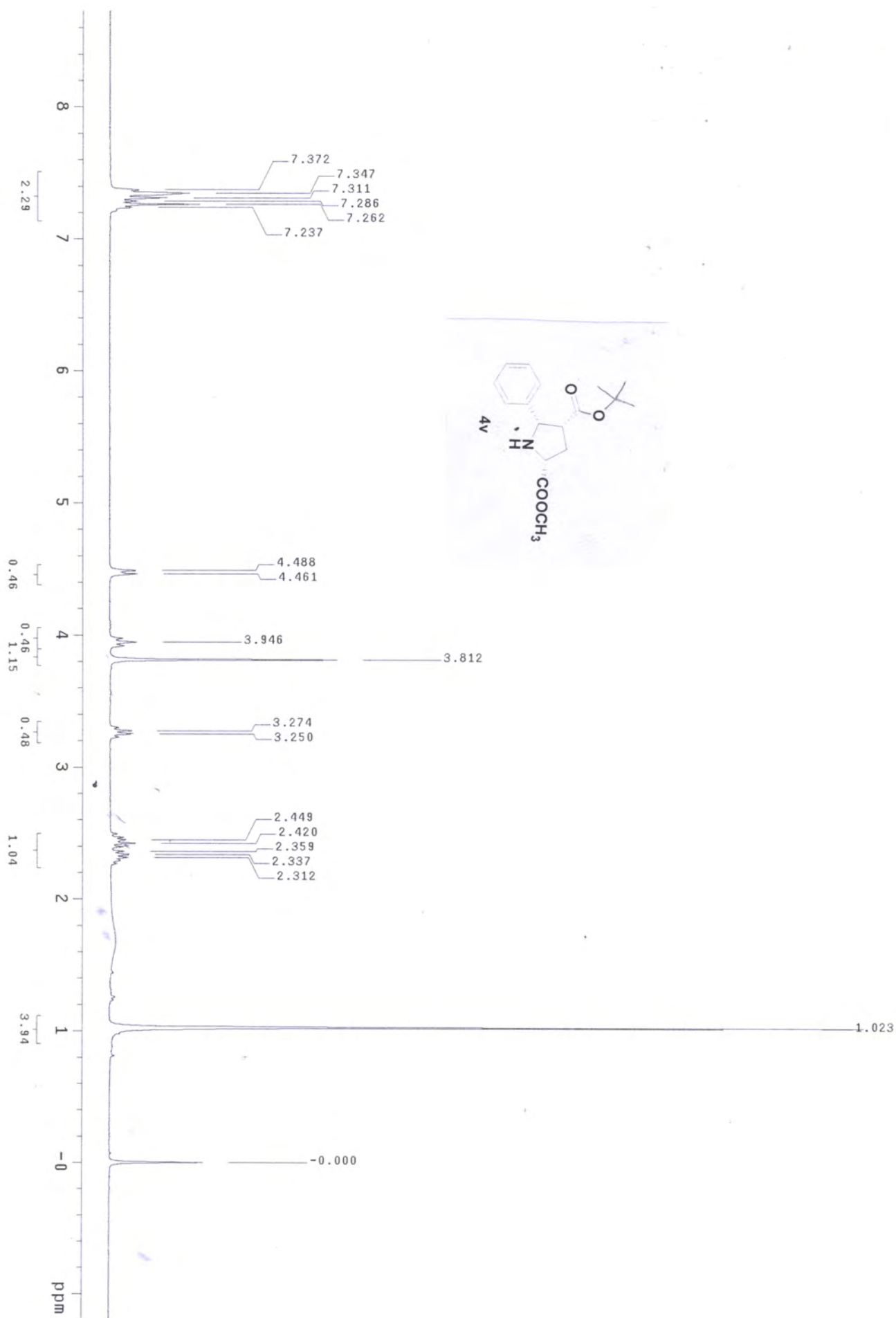




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

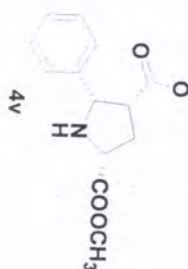
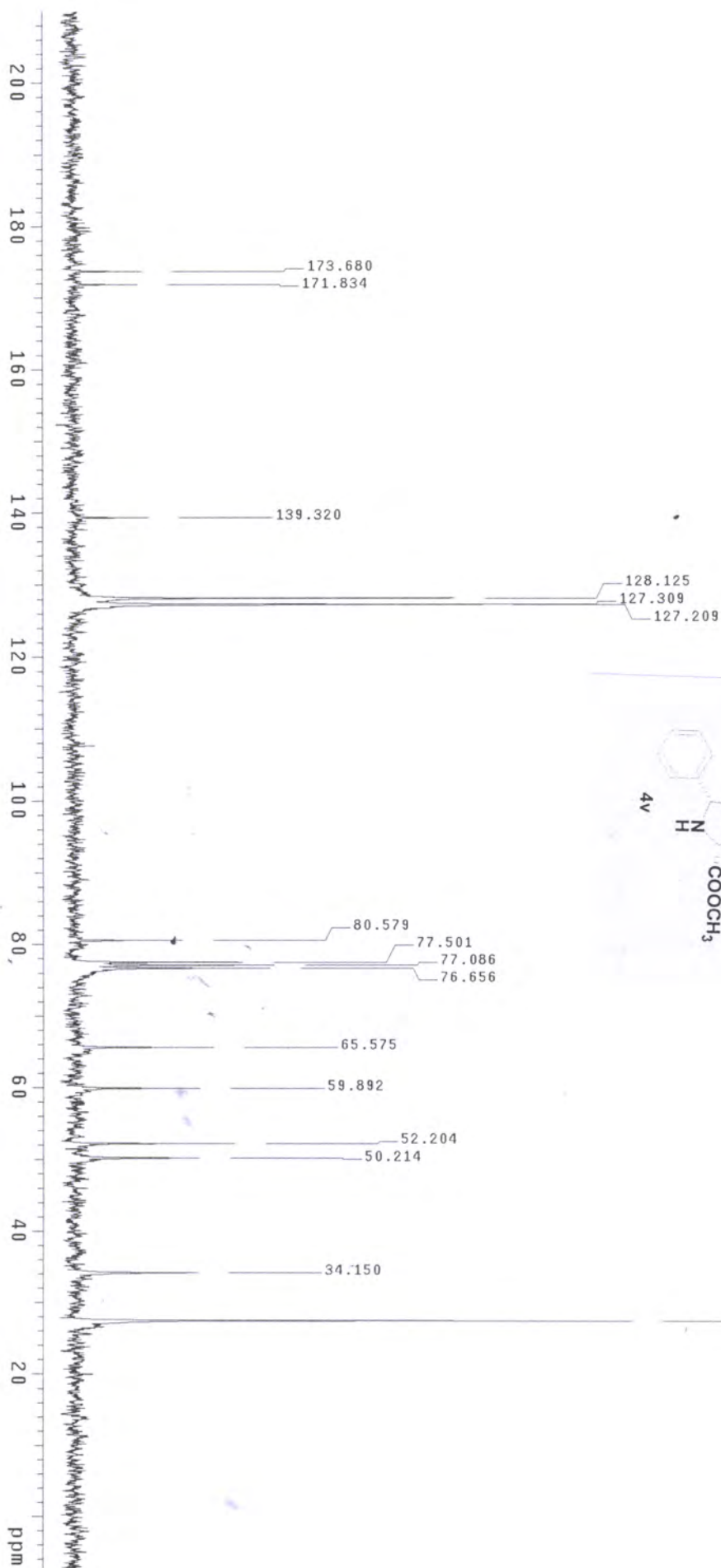
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
160 repetitions
OBSERVE C13, 75.4552902 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 5.0 Hz
FT size 32768
Total time 2 hr, 17 min, 30 sec

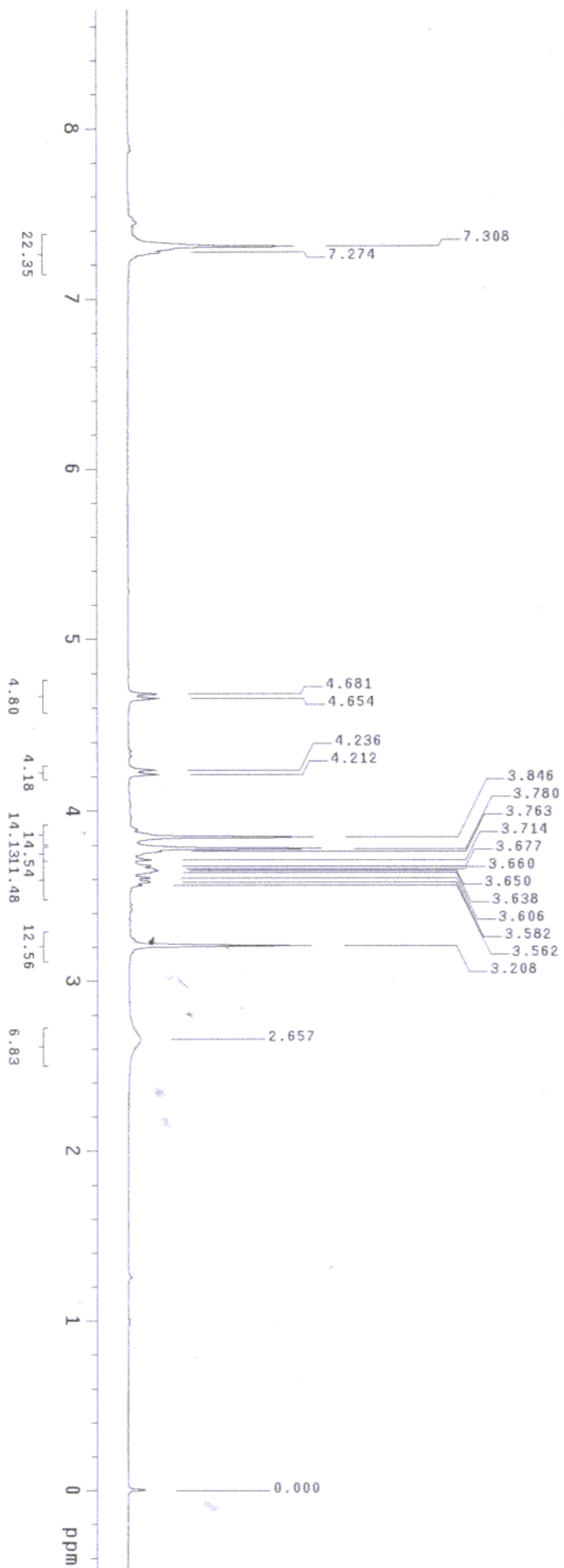
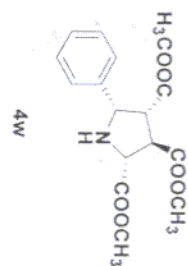




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

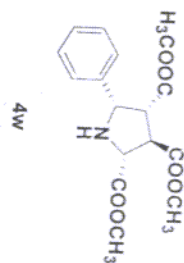
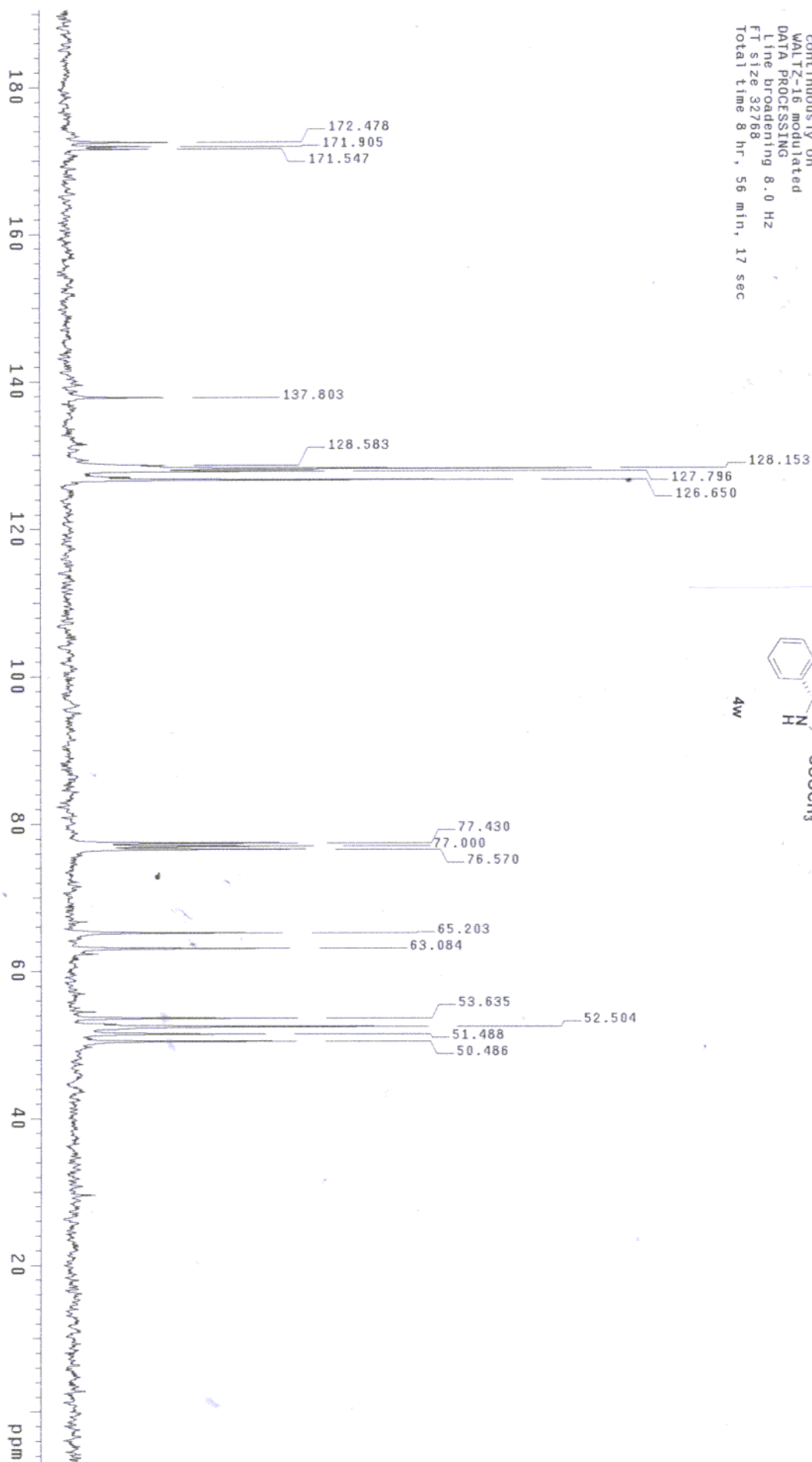
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
152 repetitions
OBSERVE C13, 75.4552718 MHz
DECOUPLE H1, 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

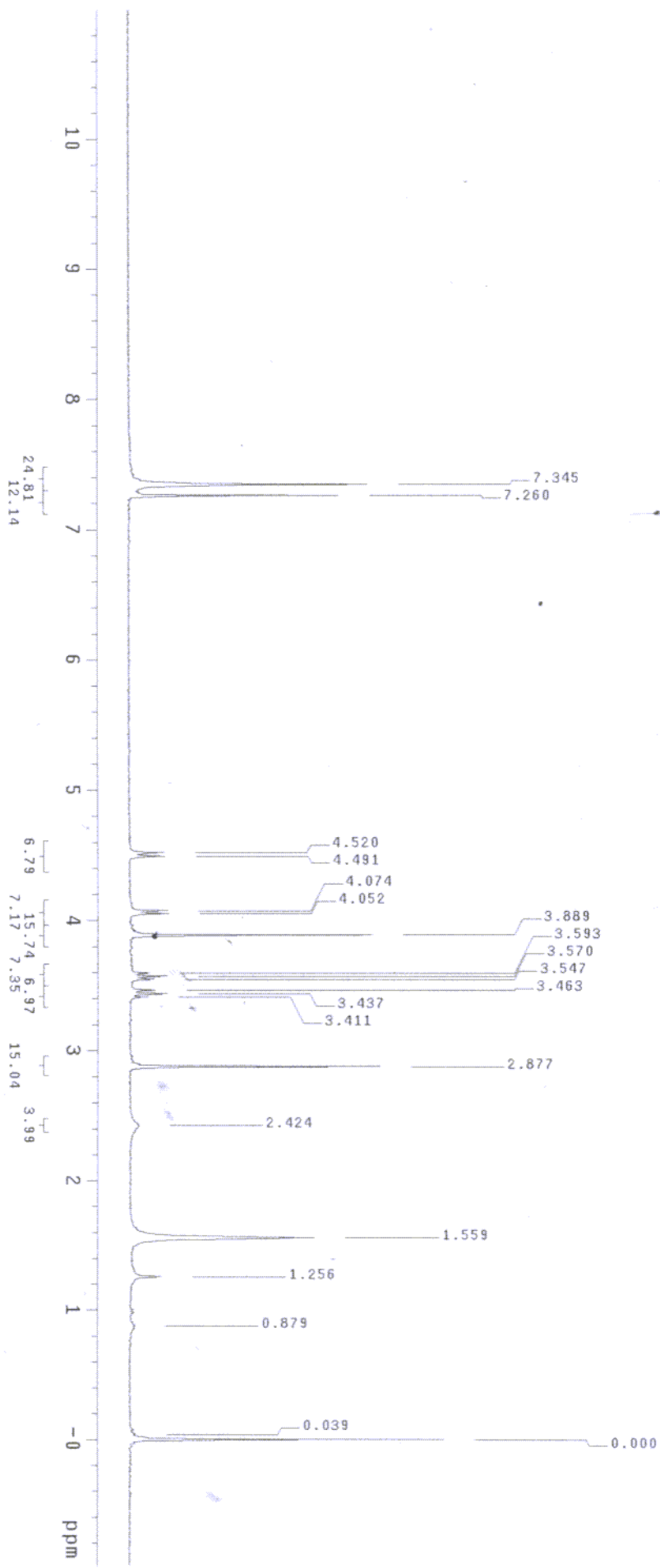




Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

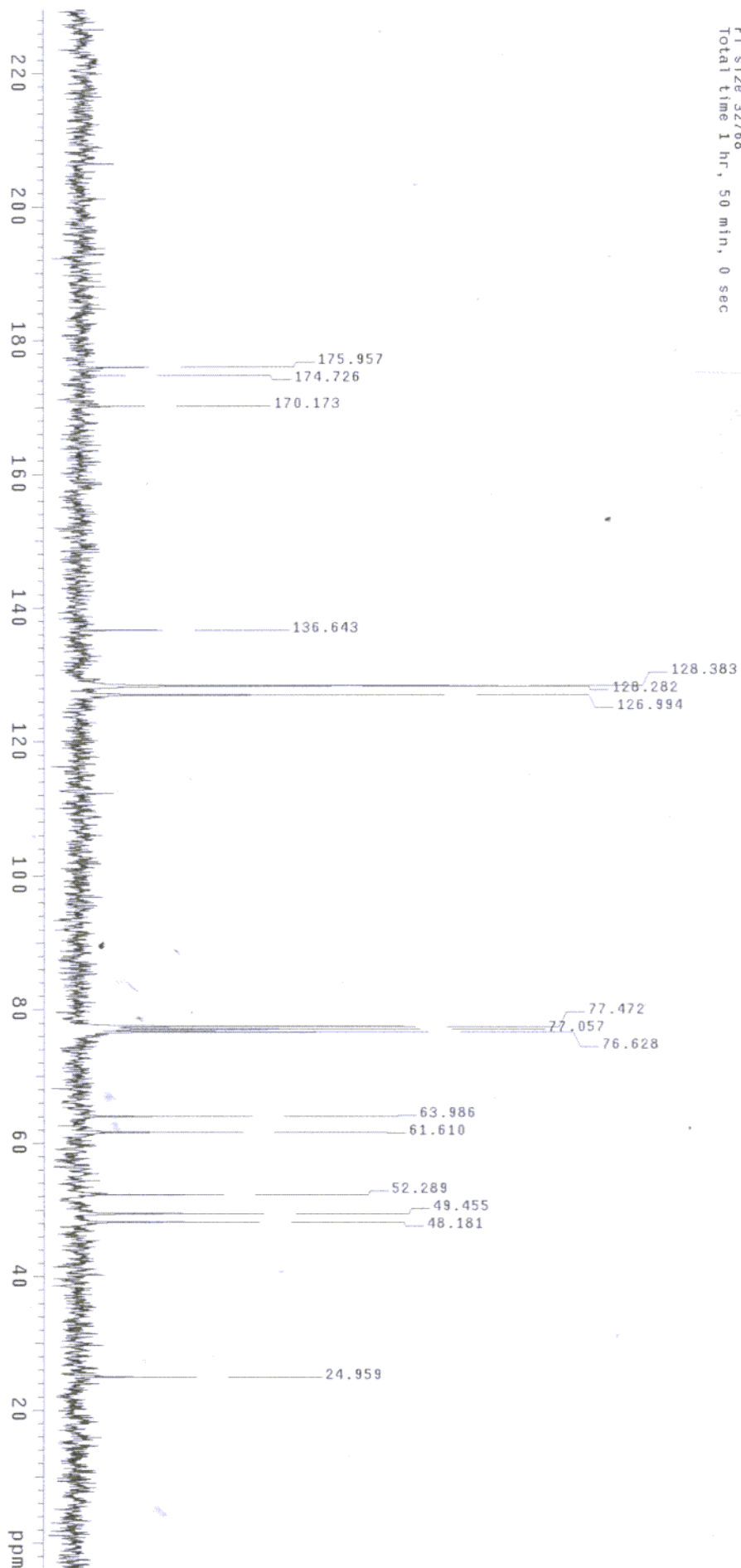
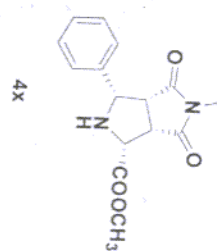
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
320 repetitions
OBSERVE C13; 75.4552848 MHz
DECUPLE H1; 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 8.0 Hz
FT size 32768
Total time 8 hr, 56 min, 17 sec

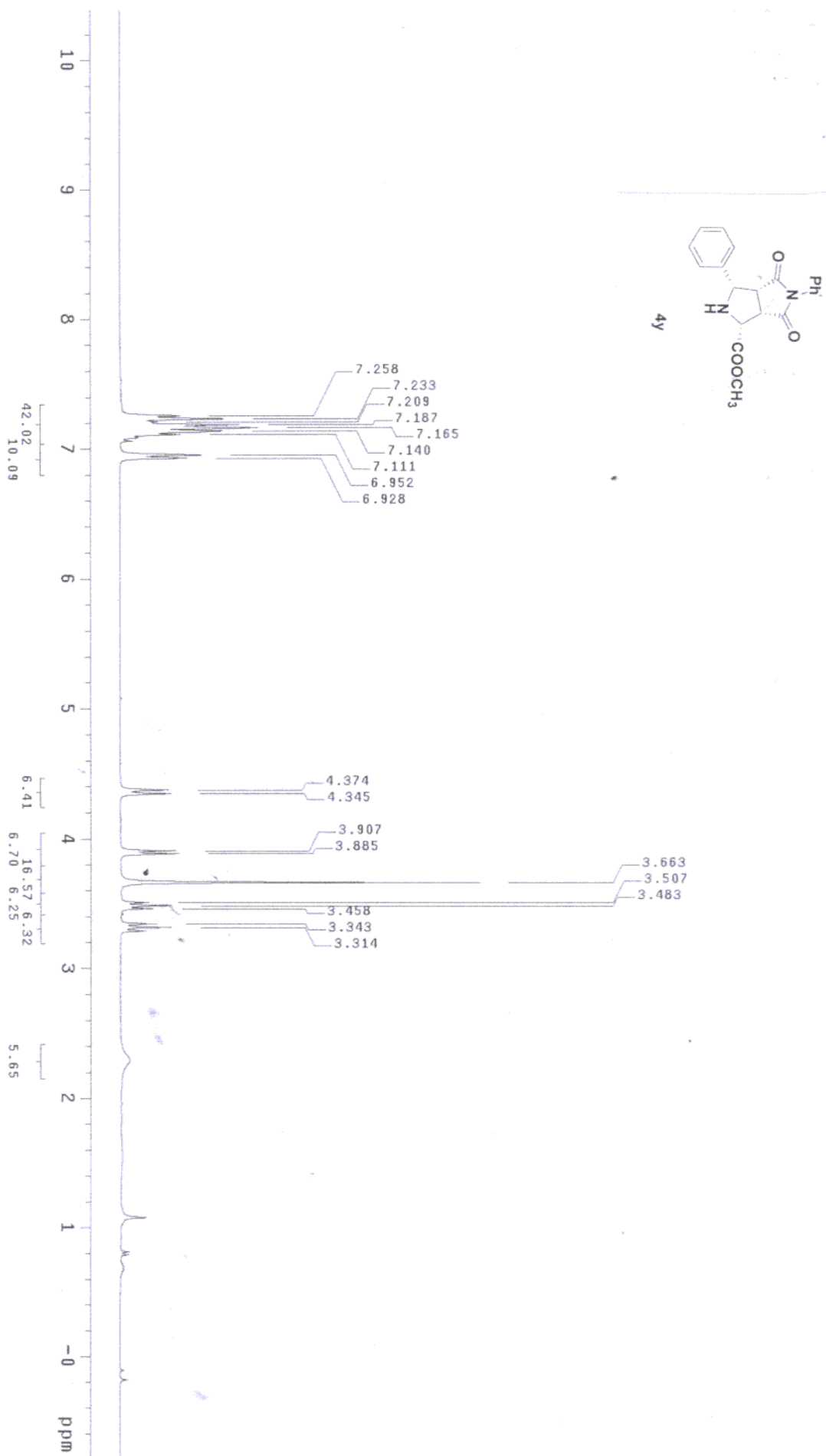




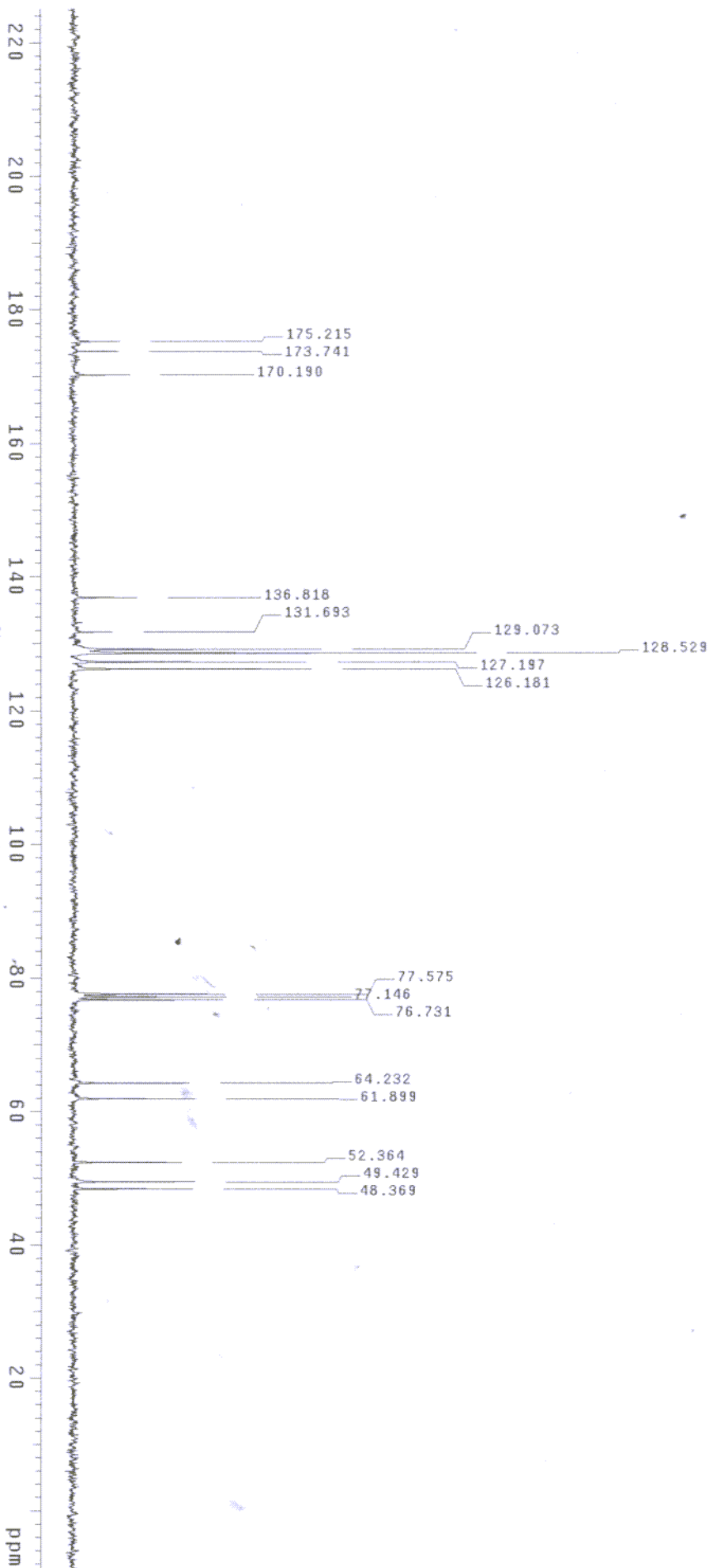
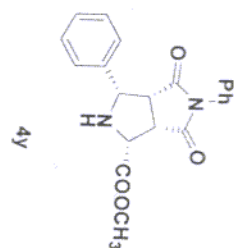
Solvent: DMSO
Ambient temperature
Mercury-300BB "mercury300"

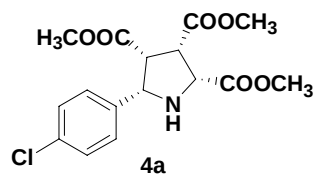
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
160 repetitions
OBSERVE C13, 75.4552759 MHz
DECOUPLE H1, 300.0821426 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 1 hr, 50 min, 0 sec





Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
182 repetitions
OBSERVE C13 75.4552576 MHz
DECOUPLE H1 300.0807172 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FI size 32768
Total time 1 hr, 50 min, 0 sec





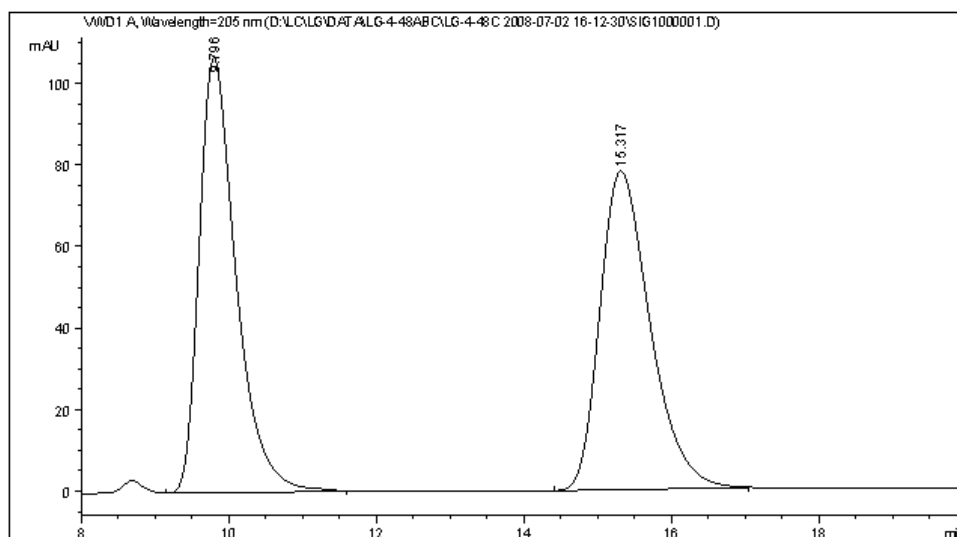
Data File D:\LC\LG\DATA\LG-4-48ABC\LG-4-48C 2008-07-02 16-12-30\SIG1000001.D

Sample Name: rac-lg-4-48c

```

=====
Acq. Operator   : dxq                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 5
Injection Date  : 7/2/2008 4:13:56 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-48ABC\LG-4-48C 2008-07-02 16-12-30\ASH-50-50-205NM-08ML-
                                           25MIN.M
Last changed    : 7/2/2008 4:05:34 PM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-48ABC\LG-4-48C 2008-07-02 16-12-30\SIG1000001.D\DA.M (ASH-
                                           50-50-205NM-08ML-25MIN.M)
Last changed    : 7/2/2008 7:46:49 PM by liang gang
                                           (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-25MIN
=====

```



Area Percent Report

```

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

```

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	9.796	VB	0.5284	3709.50610	106.57875	50.0799
2	15.317	BB	0.7249	3697.67456	78.20860	49.9201

Totals : 7407.18066 184.78735

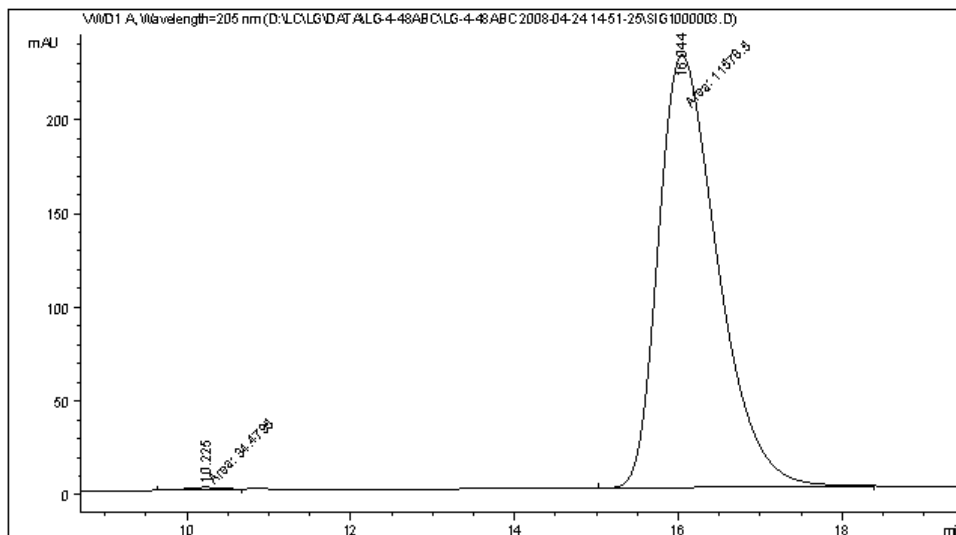
*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-48ABC\LG-4-48ABC 2008-04-24 14-51-25\SIG1000003.D
Sample Name: lg-4-48c

```

=====
Acq. Operator   : liang gang          Seq. Line :    3
Acq. Instrument : Instrument 1         Location  : Vial 53
Injection Date  : 4/24/2008 3:36:08 PM Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-48ABC\LG-4-48ABC 2008-04-24 14-51-25\ASH-50-50-205NM-08ML-
20MIN.M
Last changed    : 4/24/2008 2:39:22 PM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-48ABC\LG-4-48ABC 2008-04-24 14-51-25\SIG1000003.D\DA.M (
ASH-50-50-205NM-08ML-20MIN.M)
Last changed    : 6/14/2008 8:48:54 PM by xzy
(modified after loading)
Method Info     : ASH-50-50-205NM-08ML-20MIN
=====

```



Area Percent Report

```

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

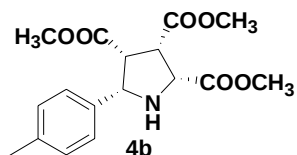
```

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.225	MM	0.4697	34.47951	1.22356	0.2969
2	16.044	MM	0.8370	1.15785e4	230.54886	99.7031

Totals : 1.16129e4 231.77241

*** End of Report ***

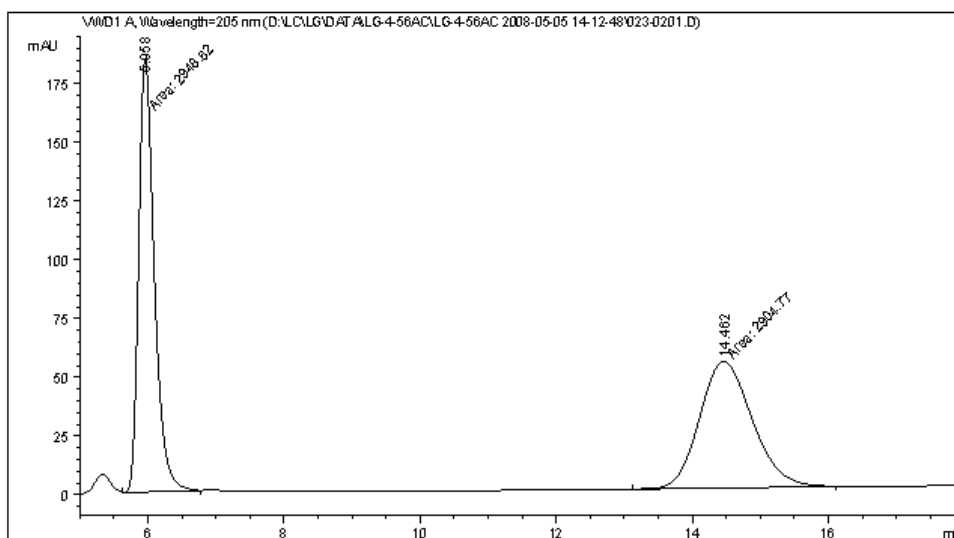


Data File D:\LC\LG\DATA\LG-4-56AC\LG-4-56AC 2008-05-05 14-12-48\023-0201.D
Sample Name: rac-lg-4-56c

```

=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 23
Injection Date  : 5/5/2008 2:40:51 PM              Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-56AC\LG-4-56AC 2008-05-05 14-12-48\ASH-50-50-205NM-10ML-
                                                35MIN.M
Last changed    : 5/5/2008 1:49:38 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-56AC\LG-4-56AC 2008-05-05 14-12-48\023-0201.D\DA.M (ASH-50-
                                                50-205NM-10ML-35MIN.M)
Last changed    : 6/14/2008 9:07:59 PM by xzy
                                                (modified after loading)
Method Info     : ASH-50-50-205NM-1.0ML-35MIN
=====

```



Area Percent Report

```

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

```

Signal 1: VWD1 A, Wavelength=205 nm

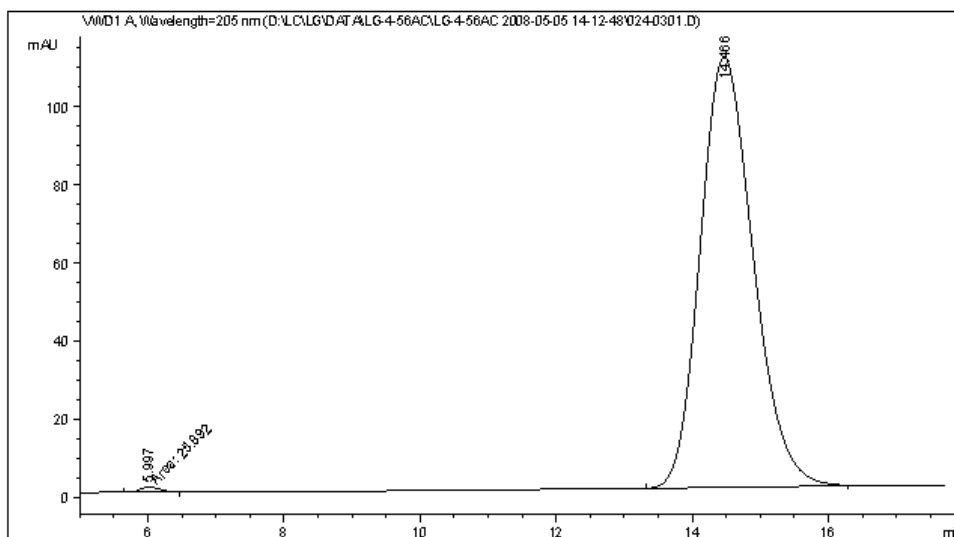
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.958	MM	0.2661	2948.61719	184.70793	50.3745
2	14.462	MM	0.8971	2904.77051	53.96305	49.6255

Totals : 5853.38770 238.67098

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-56AC\LG-4-56AC 2008-05-05 14-12-48\024-0301.D
Sample Name: lg-4-56c

```
=====
Acq. Operator   : liang gang                      Seq. Line :    3
Acq. Instrument : Instrument 1                    Location  : Vial 24
Injection Date  : 5/5/2008 3:22:29 PM              Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-56AC\LG-4-56AC 2008-05-05 14-12-48\ASH-50-50-205NM-10ML-
35MIN.M
Last changed    : 5/5/2008 1:49:38 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-56AC\LG-4-56AC 2008-05-05 14-12-48\024-0301.D\DA.M (ASH-50-
50-205NM-10ML-35MIN.M)
Last changed    : 6/14/2008 9:13:53 PM by xzy
(modified after loading)
Method Info     : ASH-50-50-205NM-1.0ML-35MIN
=====
```



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

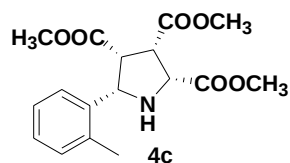
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.997	MM	0.3312	25.89199	1.30311	0.4324
2	14.466	BB	0.8397	5961.98389	110.14528	99.5676

Totals : 5987.87588 111.44839

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

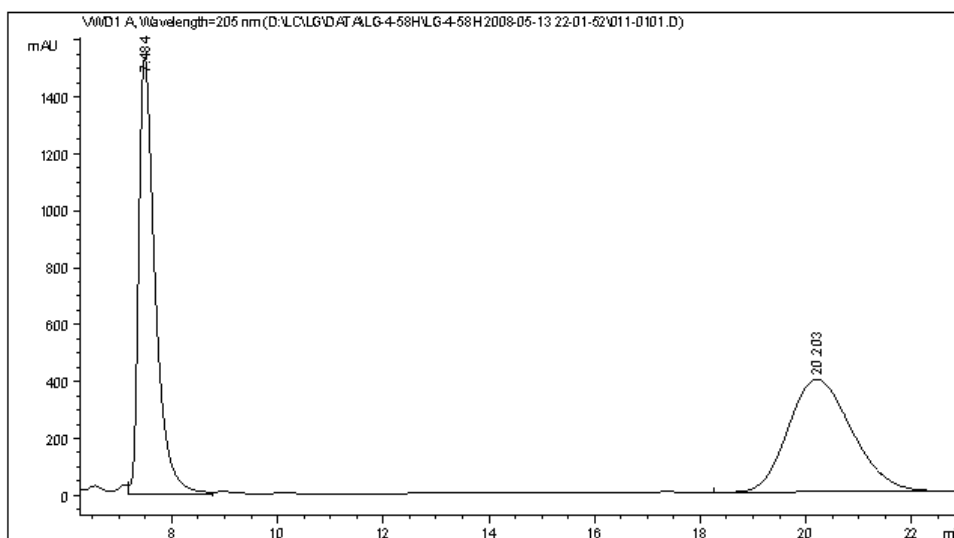


Data File D:\LC\LG\DATA\LG-4-58H\LG-4-58H 2008-05-13 22-01-52\011-0101.D
Sample Name: rac-lg-4-58h

```

=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 11
Injection Date  : 5/13/2008 10:03:11 PM           Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-58H\LG-4-58H 2008-05-13 22-01-52\ASH-50-50-205NM-08ML-
25MIN.M
Last changed    : 5/13/2008 2:59:07 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-58H\LG-4-58H 2008-05-13 22-01-52\011-0101.D\DA.M (ASH-50-
50-205NM-08ML-25MIN.M)
Last changed    : 6/14/2008 9:54:12 PM by xzy
                (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-25MIN
=====

```



Area Percent Report

```

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

```

Signal 1: VWD1 A, Wavelength=205 nm

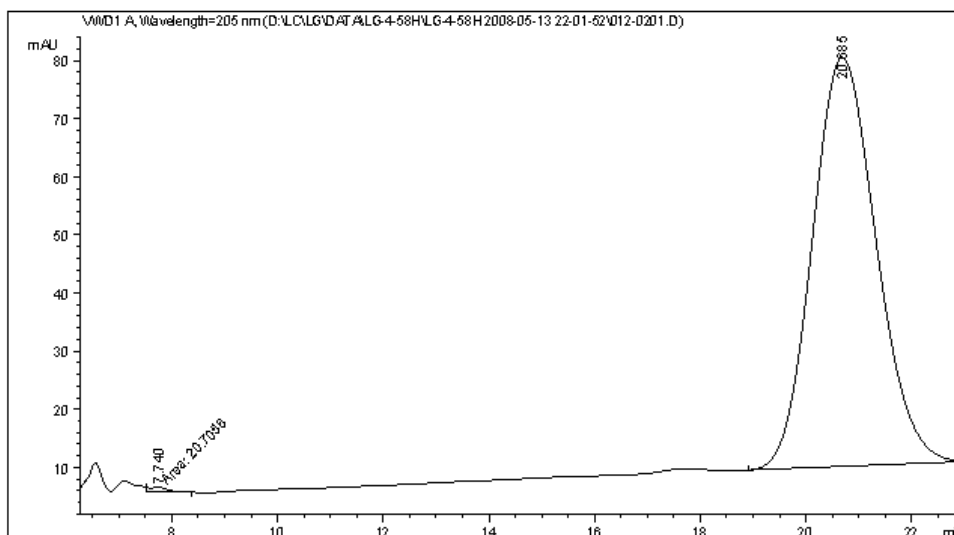
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.484	VV	0.3260	3.32943e4	1534.06287	50.1502
2	20.203	VB	1.3140	3.30949e4	395.52914	49.8498

Totals : 6.63892e4 1929.59201

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-58H\LG-4-58H 2008-05-13 22-01-52\012-0201.D
Sample Name: lg-4-58h

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 12
Injection Date  : 5/13/2008 10:29:48 PM          Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-58H\LG-4-58H 2008-05-13 22-01-52\ASH-50-50-205NM-08ML-
25MIN.M
Last changed    : 5/13/2008 2:59:07 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-58H\LG-4-58H 2008-05-13 22-01-52\012-0201.D\DA.M (ASH-50-
50-205NM-08ML-25MIN.M)
Last changed    : 6/14/2008 10:02:14 PM by xzy
(modified after loading)
Method Info     : ASH-50-50-205NM-08ML-25MIN
=====
```



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

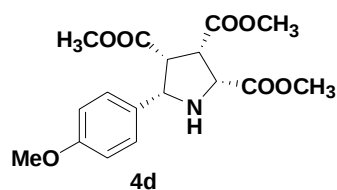
```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.740	MM	0.4364	20.70560	7.90737e-1	0.3501
2	20.685	BB	1.2989	5893.67041	70.47179	99.6499

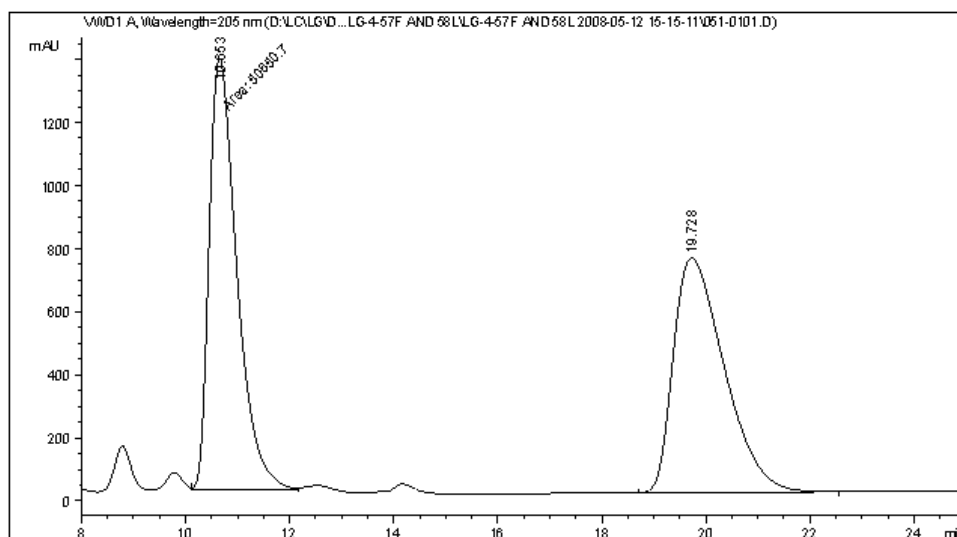
Totals : 5914.37601 71.26253

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



Data File D:\LC\LG\DATA\LG-4-57F AND 58L\LG-4-57F AND 58L 2008-05-12 15-15-11\051-0101.D
 Sample Name: rac-lg-4-57f

```
=====
Acq. Operator   : liang gang          Seq. Line :    1
Acq. Instrument : Instrument 1         Location  : Vial 51
Injection Date  : 5/12/2008 3:16:44 PM Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-57F AND 58L\LG-4-57F AND 58L 2008-05-12 15-15-11\ASH-50-50-
                  205NM-08ML-42MIN.M
Last changed    : 5/12/2008 3:09:33 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-57F AND 58L\LG-4-57F AND 58L 2008-05-12 15-15-11\051-0101.
                  D\DA.M (ASH-50-50-205NM-08ML-42MIN.M)
Last changed    : 6/14/2008 9:39:08 PM by xzy
                  (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-42MIN
=====
```



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

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

Signal 1: VWD1 A, Wavelength=205 nm

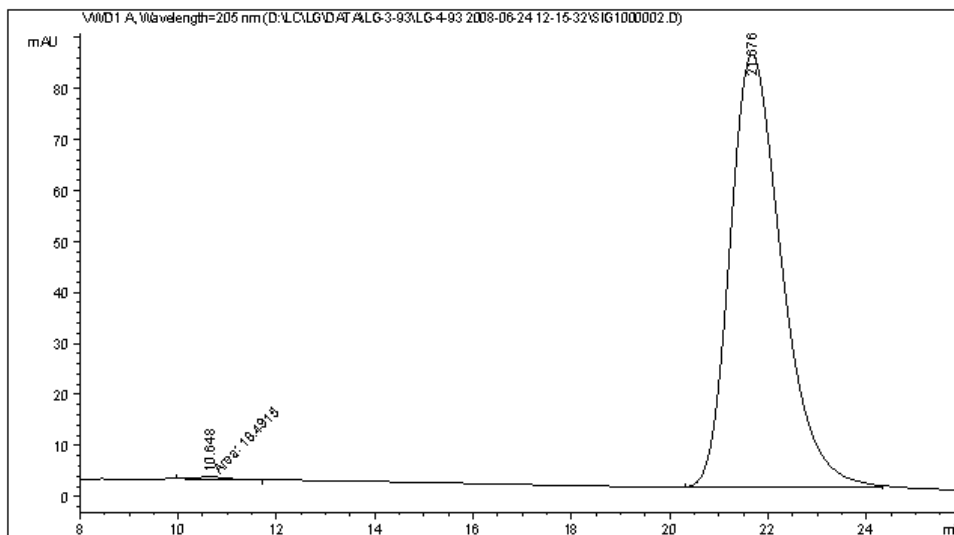
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	10.653	MM	0.6174	5.06507e4	1367.26404	50.0088
2	19.728	BB	1.0503	5.06329e4	745.33575	49.9912

Totals : 1.01284e5 2112.59979

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

Data File D:\LC\LG\DATA\LG-3-93\LG-4-93 2008-06-24 12-15-32\SIG1000002.D
Sample Name: lg-4-57f

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                     Location  : Vial 26
Injection Date  : 6/24/2008 12:48:42 PM           Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-3-93\LG-4-93 2008-06-24 12-15-32\ASH-50-50-205NM-08ML-30MIN.M
Last changed    : 6/24/2008 12:14:14 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-3-93\LG-4-93 2008-06-24 12-15-32\SIG1000002.D\DA.M (ASH-50-
50-205NM-08ML-30MIN.M)
Last changed    : 6/24/2008 1:26:37 PM by liang gang
                  (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-30MIN
=====
```



Area Percent Report

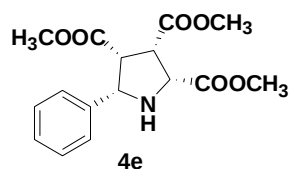
```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	10.648	MM	0.6640	18.49154	4.64134e-1	0.2978
2	21.676	BB	1.1184	6190.06592	84.76894	99.7022

Totals : 6208.55746 85.23307

*** End of Report ***

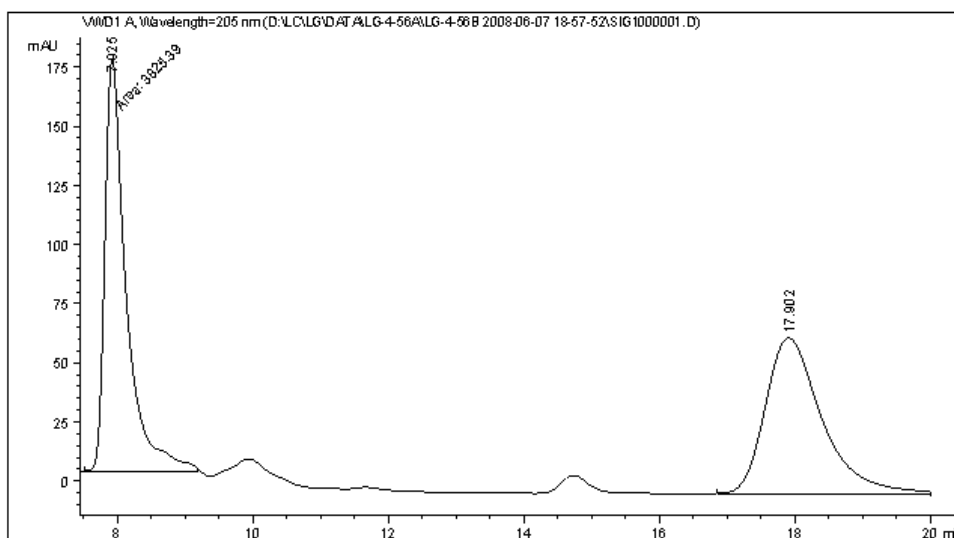


Data File D:\LC\LG\DATA\LG-4-56A\LG-4-56B 2008-06-07 18-57-52\SIG1000001.D
Sample Name: rac-lg-4-56a

```

=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 18
Injection Date  : 6/7/2008 6:59:48 PM             Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-56A\LG-4-56B 2008-06-07 18-57-52\ASH-50-50-205NM-08ML-
20MIN.M
Last changed    : 6/7/2008 4:15:48 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-56A\LG-4-56B 2008-06-07 18-57-52\SIG1000001.D\DA.M (ASH-50-
50-205NM-08ML-20MIN.M)
Last changed    : 6/14/2008 9:02:50 PM by xzy
                  (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-20MIN
=====

```



Area Percent Report

```

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

```

Signal 1: WWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.925	MM	0.3646	3825.38916	174.88838	50.2009
2	17.902	BBA	0.8708	3794.77686	65.80292	49.7991

Totals : 7620.16602 240.69130

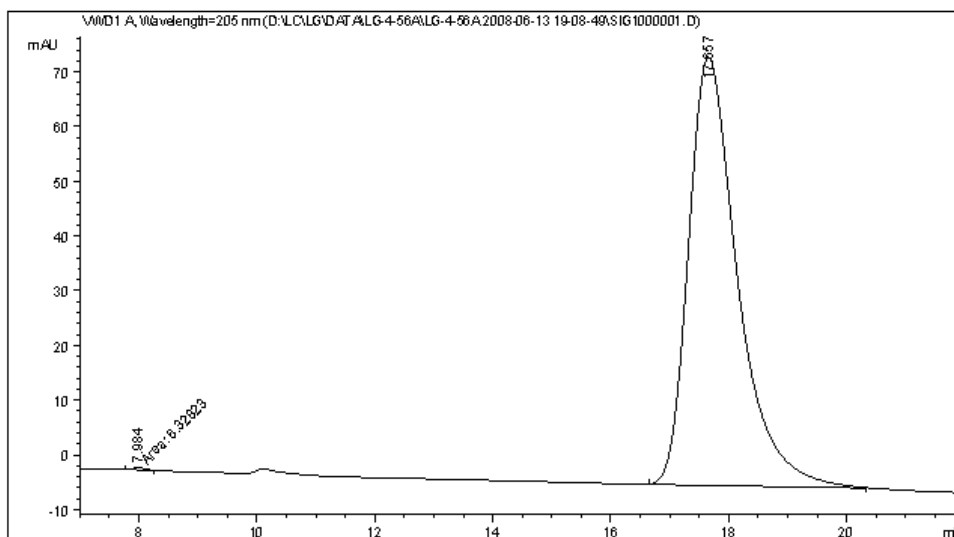
*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-56A\LG-4-56A 2008-06-13 19-08-49\SIG1000001.D
Sample Name: lg-4-56a

```

=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 19
Injection Date  : 6/13/2008 7:10:22 PM           Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-56A\LG-4-56A 2008-06-13 19-08-49\ASH-50-50-205NM-08ML-
25MIN.M
Last changed    : 6/13/2008 4:23:01 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-56A\LG-4-56A 2008-06-13 19-08-49\SIG1000001.D\DA.M (ASH-50-
50-205NM-08ML-25MIN.M)
Last changed    : 6/14/2008 8:56:31 PM by xzy
(modified after loading)
Method Info     : ASH-50-50-205NM-08ML-25MIN
=====

```



Area Percent Report

```

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

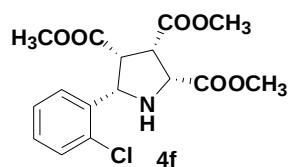
```

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.984	MM	0.2383	6.32823	4.42686e-1	0.1399
2	17.657	BB	0.8681	4517.07861	78.30362	99.8601

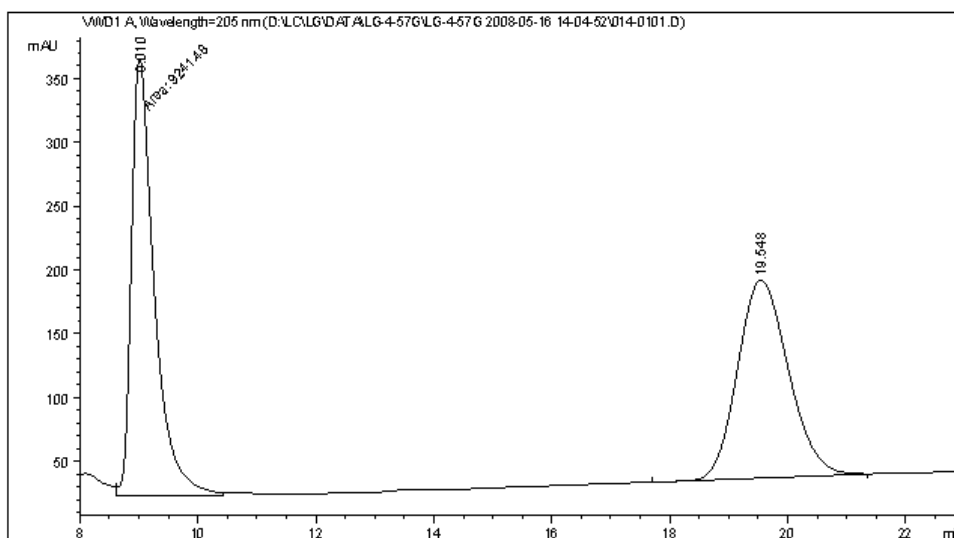
Totals : 4523.40684 78.74631

*** End of Report ***



Data File D:\LC\LG\DATA\LG-4-57G\LG-4-57G 2008-05-16 14-04-52\014-0101.D
Sample Name: rac-lg-4-57g

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 14
Injection Date  : 5/16/2008 2:06:12 PM             Inj       :    1
                                              Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-57G\LG-4-57G 2008-05-16 14-04-52\ASH-50-50-205NM-08ML-
25MIN.M
Last changed    : 5/16/2008 1:53:33 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-57G\LG-4-57G 2008-05-16 14-04-52\014-0101.D\DA.M (ASH-50-
50-205NM-08ML-25MIN.M)
Last changed    : 6/14/2008 9:49:06 PM by xzy
(modified after loading)
Method Info     : ASH-50-50-205NM-08ML-25MIN
=====
```



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.010	MM	0.4490	9241.48145	343.05228	49.8995
2	19.548	BB	0.9289	9278.69141	155.21347	50.1005

Totals : 1.85202e4 498.26575

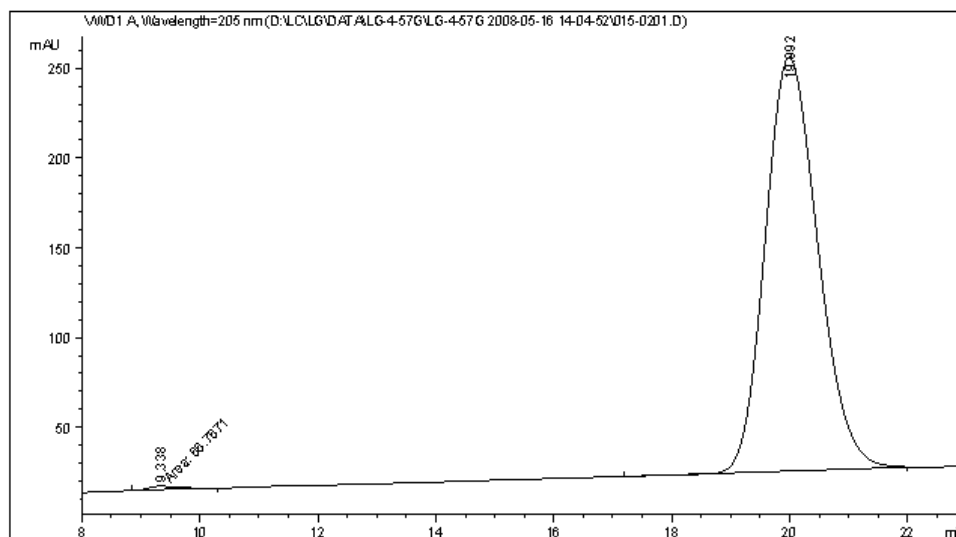
*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-57G\LG-4-57G 2008-05-16 14-04-52\015-0201.D
Sample Name: lg-4-57g

```

=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 15
Injection Date  : 5/16/2008 2:32:51 PM             Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-57G\LG-4-57G 2008-05-16 14-04-52\ASH-50-50-205NM-08ML-
25MIN.M
Last changed    : 5/16/2008 1:53:33 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-57G\LG-4-57G 2008-05-16 14-04-52\015-0201.D\DA.M (ASH-50-
50-205NM-08ML-25MIN.M)
Last changed    : 6/14/2008 9:51:28 PM by xzy
(modified after loading)
Method Info     : ASH-50-50-205NM-08ML-25MIN
=====

```



Area Percent Report

```

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

```

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.338	MM	0.5880	66.76713	1.89251	0.4652
2	19.992	BB	0.9660	1.42848e4	230.56316	99.5348

Totals : 1.43515e4 232.45566

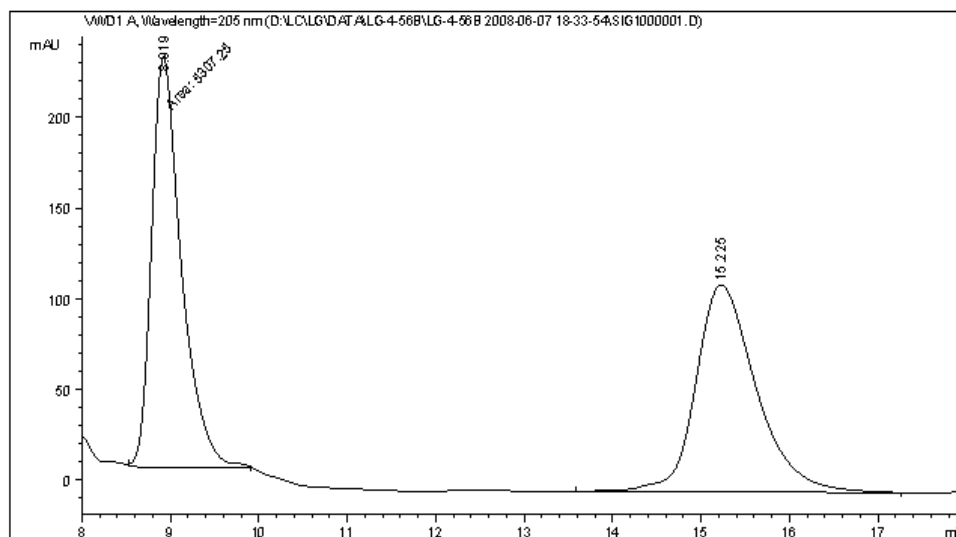
*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-56B\LG-4-56B 2008-06-07 18-33-54\SIG1000001.D
Sample Name: rac-lg-4-56b

```

=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 17
Injection Date  : 6/7/2008 6:35:27 PM             Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-56B\LG-4-56B 2008-06-07 18-33-54\ASH-50-50-205NM-08ML-
20MIN.M
Last changed    : 6/7/2008 4:15:48 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-56B\LG-4-56B 2008-06-07 18-33-54\SIG1000001.D\DA.M (ASH-50-
50-205NM-08ML-20MIN.M)
Last changed    : 6/14/2008 9:17:52 PM by xzy
                  (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-20MIN
=====

```



Area Percent Report

```

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

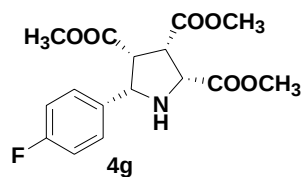
```

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.919	MM	0.3913	5307.25195	226.06686	49.4560
2	15.225	BB	0.7174	5424.00977	114.15173	50.5440

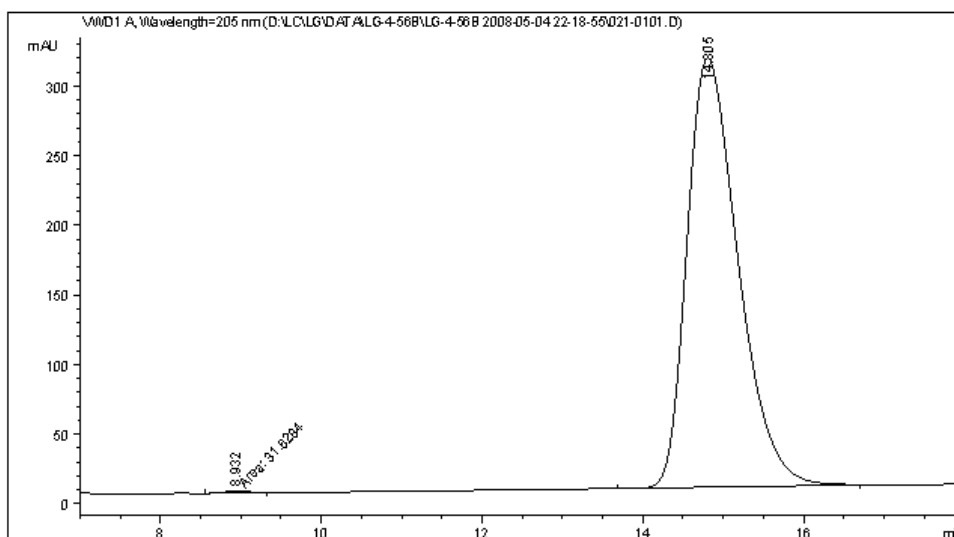
Totals : 1.07313e4 340.21860

*** End of Report ***



Data File D:\LC\LG\DATA\LG-4-56B\LG-4-56B 2008-05-04 22-18-55\021-0101.D
 Sample Name: lg-4-56b

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                     Location  : Vial 21
Injection Date  : 5/4/2008 10:20:14 PM             Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-56B\LG-4-56B 2008-05-04 22-18-55\ASH-50-50-205NM-08ML-
                  25MIN.M
Last changed    : 5/4/2008 9:50:30 PM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-56B\LG-4-56B 2008-05-04 22-18-55\021-0101.D\DA.M (ASH-50-
                  50-205NM-08ML-25MIN.M)
Last changed    : 6/14/2008 9:21:10 PM by xzy
                  (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-25MIN
=====
```



Area Percent Report

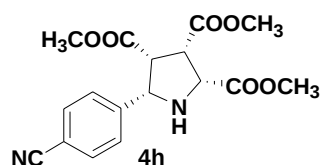
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	8.932	MM	0.3402	31.62838	1.54930	0.2321
2	14.805	BB	0.6792	1.35960e4	308.17331	99.7679

Totals : 1.36276e4 309.72261

*** End of Report ***

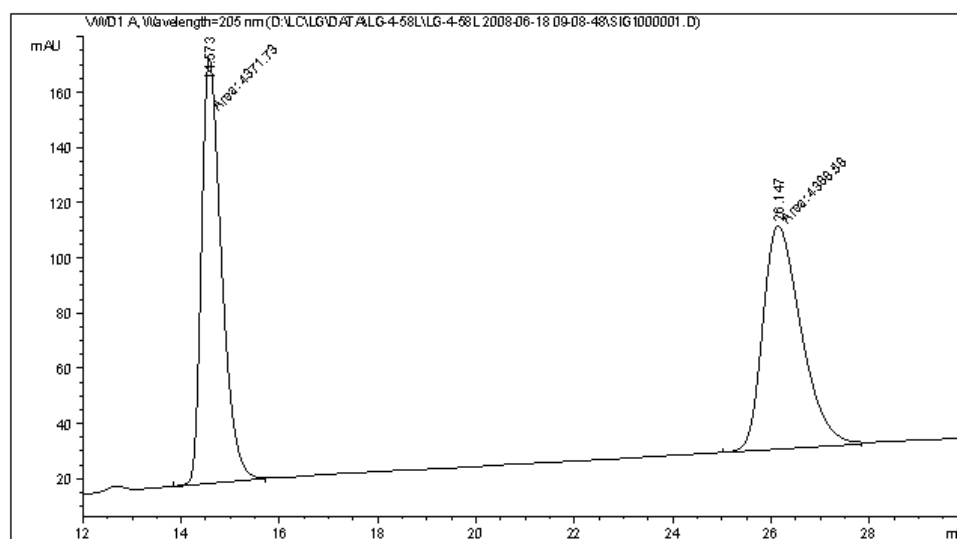


Data File D:\LC\LG\DATA\LG-4-58L\LG-4-58L 2008-06-18 09-08-48\SIG1000001.D
Sample Name: rac-lg-4-58l

```

=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 22
Injection Date  : 6/18/2008 9:10:08 AM             Inj       :    1
                                              Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-58L\LG-4-58L 2008-06-18 09-08-48\AD-H-50-50-205NM-30MIN.M
Last changed    : 6/18/2008 8:58:47 AM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-58L\LG-4-58L 2008-06-18 09-08-48\SIG1000001.D\DA.M (AD-H-
50-50-205NM-30MIN.M)
Last changed    : 6/19/2008 10:14:28 AM by liang gang
                  (modified after loading)
Method Info     : AD-H-50-50-205NM-30MIN
=====

```



Area Percent Report

```

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

```

Signal 1: VWD1 A, Wavelength=205 nm

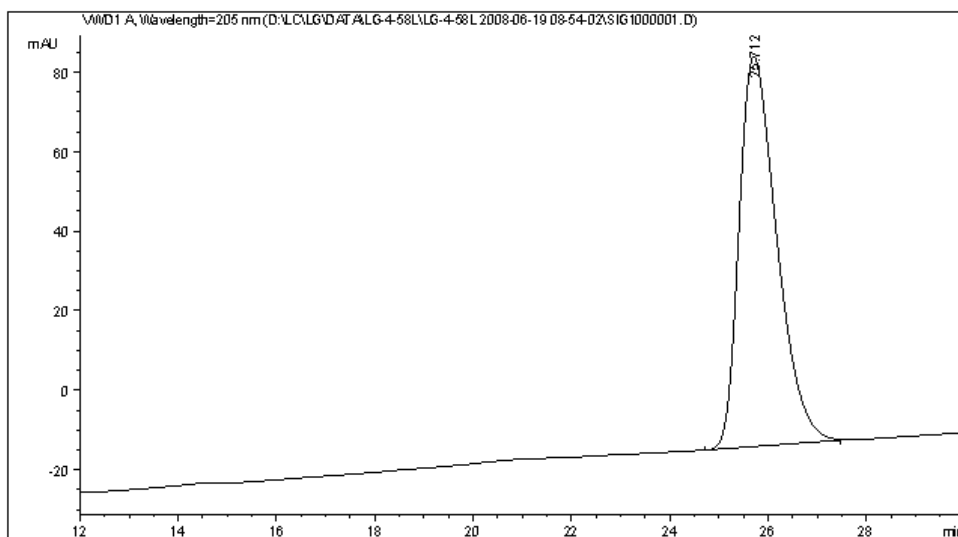
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.573	MM	0.4732	4371.72510	153.97792	49.9038
2	26.147	MM	0.9037	4388.57861	80.94156	50.0962

Totals : 8760.30371 234.91948

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-58L\LG-4-58L 2008-06-19 08-54-02\SIG1000001.D
Sample Name: lg-4-581

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 32
Injection Date  : 6/19/2008 8:55:27 AM             Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-58L\LG-4-58L 2008-06-19 08-54-02\AD-H-50-50-205NM-30MIN.M
Last changed    : 6/18/2008 8:58:47 AM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-58L\LG-4-58L 2008-06-19 08-54-02\SIG1000001.D\DA.M (AD-H-
                    50-50-205NM-30MIN.M)
Last changed    : 6/19/2008 10:16:47 AM by liang gang
                    (modified after loading)
Method Info     : AD-H-50-50-205NM-30MIN
=====
```



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

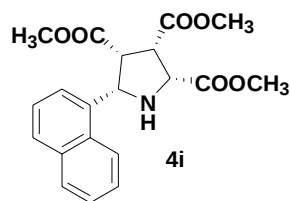
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	25.712	BB	0.8184	5213.74756	98.04409	100.0000

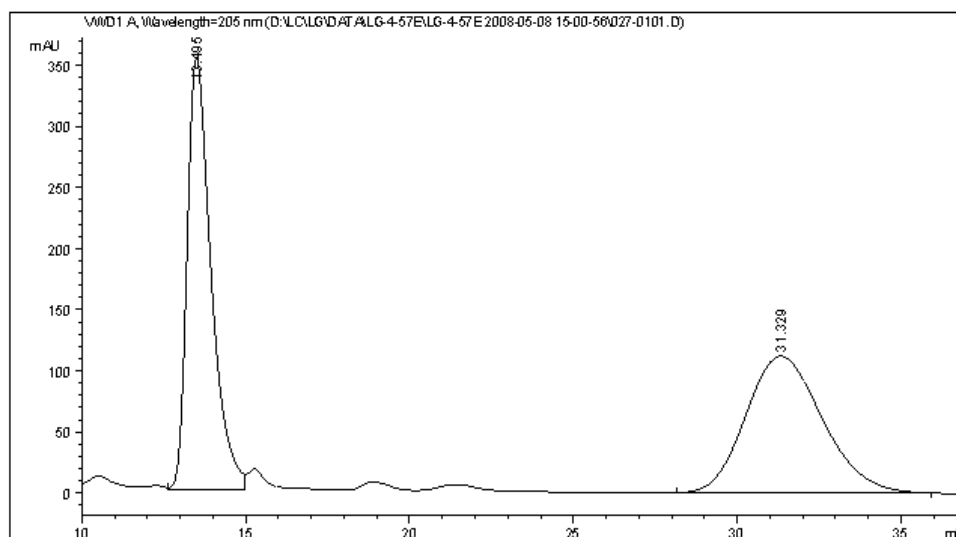
Totals : 5213.74756 98.04409

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



Data File D:\LC\LG\DATA\LG-4-57E\LG-4-57E 2008-05-08 15-00-56\027-0101.D
 Sample Name: rac-lg-4-57e

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                     Location  : Vial 27
Injection Date  : 5/8/2008 3:02:24 PM              Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-57E\LG-4-57E 2008-05-08 15-00-56\ASH-50-50-205NM-08ML-42MIN.M
Last changed    : 5/8/2008 2:44:11 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-57E\LG-4-57E 2008-05-08 15-00-56\027-0101.D\DA.M (ASH-50-50-205NM-08ML-42MIN.M)
Last changed    : 6/14/2008 9:34:07 PM by xzy
                  (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-42MIN
=====
```



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=205 nm

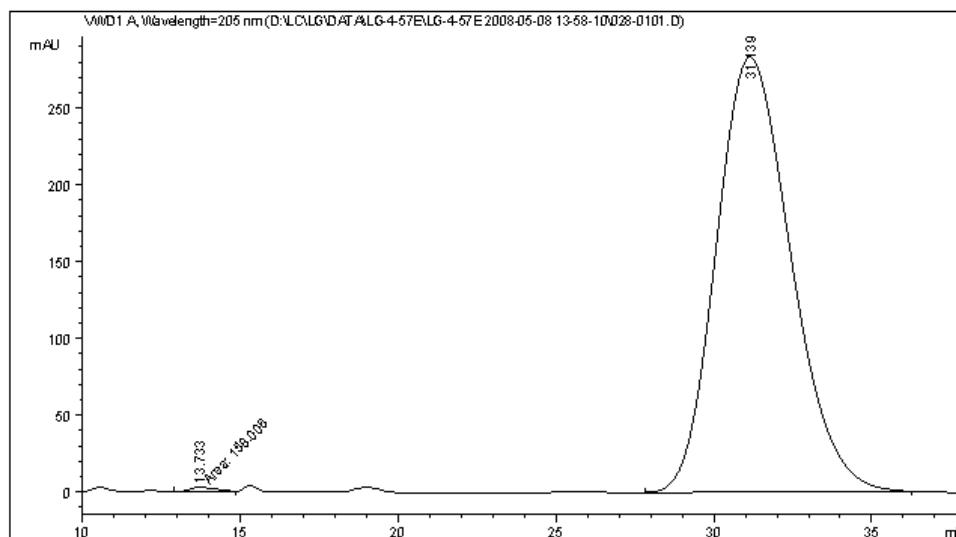
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.495	VV	0.7695	1.79000e4	352.86124	49.5601
2	31.329	BB	2.4192	1.82178e4	111.81341	50.4399

Totals : 3.61178e4 464.67464

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-57E\LG-4-57E 2008-05-08 13-58-10\028-0101.D
Sample Name: lg-4-57e

```
=====
Acq. Operator   : liang gang          Seq. Line :    1
Acq. Instrument : Instrument 1         Location  : Vial 28
Injection Date  : 5/8/2008 1:59:34 PM Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-57E\LG-4-57E 2008-05-08 13-58-10\ASH-50-50-205NM-08ML-
42MIN.M
Last changed    : 5/8/2008 11:49:16 AM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-57E\LG-4-57E 2008-05-08 13-58-10\028-0101.D\DA.M (ASH-50-
50-205NM-08ML-42MIN.M)
Last changed    : 6/14/2008 9:36:30 PM by xzy
                 (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-42MIN
=====
```



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

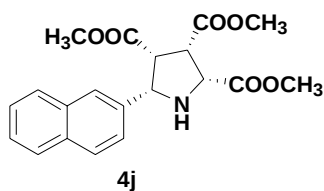
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.733	MM	0.9685	158.00551	2.71916	0.3385
2	31.139	BB	2.5351	4.65267e4	283.47586	99.6615

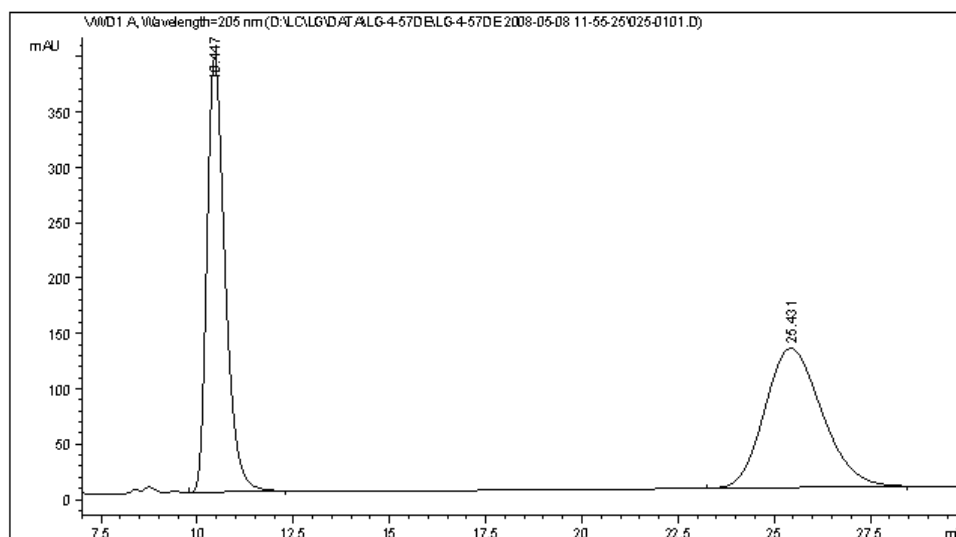
Totals : 4.66847e4 286.19502

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



Data File D:\LC\LG\DATA\LG-4-57DE\LG-4-57DE 2008-05-08 11-55-25\025-0101.D
Sample Name: rac-lg-4-57d

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 25
Injection Date  : 5/8/2008 11:57:14 AM             Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-57DE\LG-4-57DE 2008-05-08 11-55-25\ASH-50-50-205NM-08ML-42MIN.M
Last changed    : 5/8/2008 11:49:16 AM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-57DE\LG-4-57DE 2008-05-08 11-55-25\025-0101.D\DA.M (ASH-50-50-205NM-08ML-42MIN.M)
Last changed    : 6/14/2008 9:28:33 PM by xzy
                  (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-42MIN
=====
```



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=205 nm

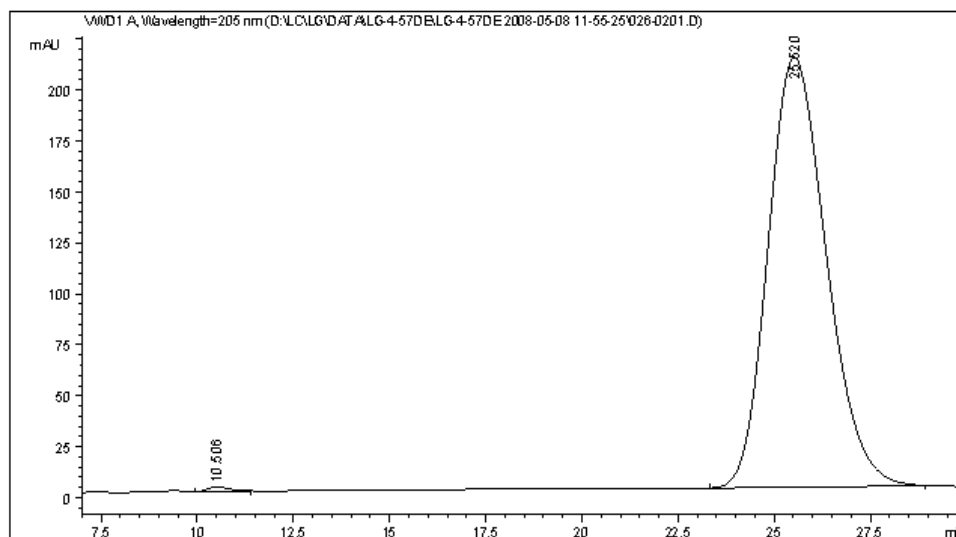
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.447	VB	0.5031	1.28910e4	391.98514	49.8357
2	25.431	BB	1.5966	1.29759e4	125.78681	50.1643

Totals : 2.58669e4 517.77195

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-57DE\LG-4-57DE 2008-05-08 11-55-25\026-0201.D
Sample Name: lg-4-57d

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 26
Injection Date  : 5/8/2008 12:40:26 PM             Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-57DE\LG-4-57DE 2008-05-08 11-55-25\ASH-50-50-205NM-08ML-
42MIN.M
Last changed    : 5/8/2008 11:49:16 AM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-57DE\LG-4-57DE 2008-05-08 11-55-25\026-0201.D\DA.M (ASH-50-
50-205NM-08ML-42MIN.M)
Last changed    : 6/14/2008 9:30:45 PM by xzy
                (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-42MIN
=====
```



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

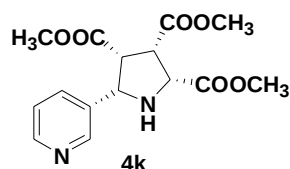
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.506	BB	0.5266	86.94051	2.38820	0.3905
2	25.520	BB	1.6324	2.21771e4	210.99908	99.6095

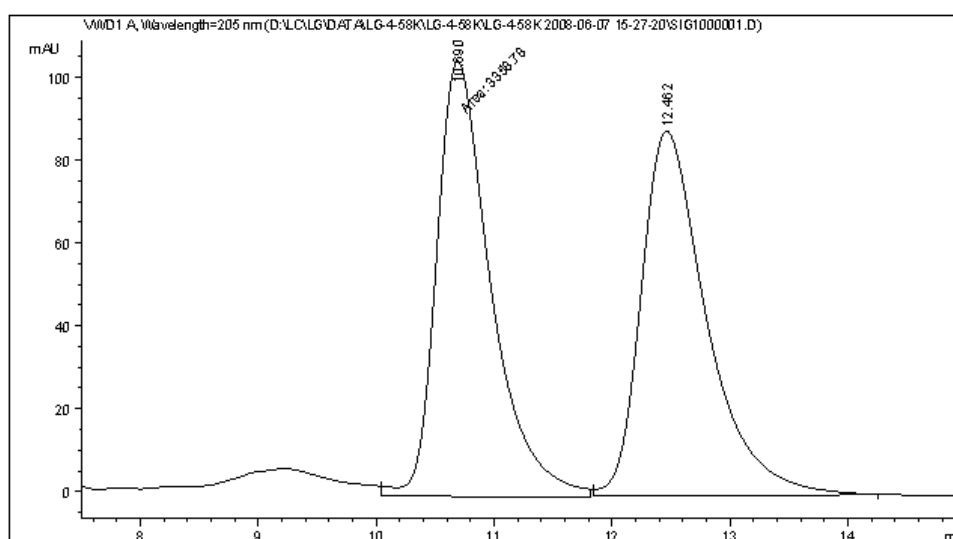
Totals : 2.22640e4 213.38728

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



Data File D:\LC\LG\DATA\LG-4-58K\LG-4-58K 2008-06-07 15-27-20\SIG1000001.D
Sample Name: rac-lg-4-58k

```
=====
Acq. Operator   : liang qiang                      Seq. Line :    1
Acq. Instrument : Instrument 1                      Location  : Vial 11
Injection Date  : 6/7/2008 3:28:44 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\XZP\DATA\LG-4-58K\LG-4-58K 2008-06-07 15-27-20\ASH-50-50-205NM-08ML-
                  15MIN.M
Last changed    : 6/7/2008 3:17:33 PM by xzy
Analysis Method : D:\LC\LG\DATA\LG-4-58K\LG-4-58K 2008-06-07 15-27-20\SIG1000001.D\DA.M
                  (ASH-50-50-205NM-08ML-15MIN.M)
Last changed    : 6/14/2008 10:07:24 PM by xzy
                  (modified after loading)
Method Info     : ASH-50-50-205NM-08ML-15MIN
=====
```



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=205 nm

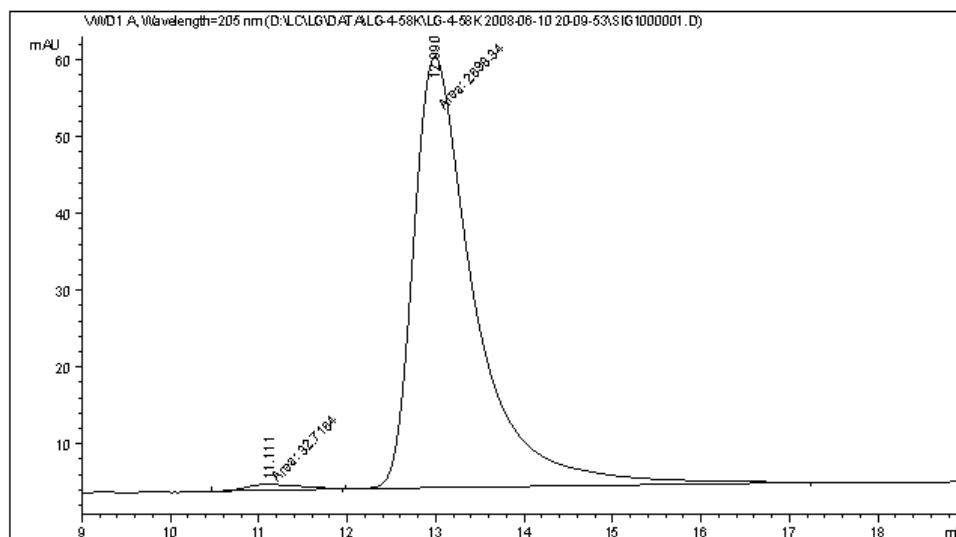
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.690	MM	0.5328	3358.78369	105.07546	49.7767
2	12.462	VB	0.5803	3388.91846	88.00303	50.2233

Totals : 6747.70215 193.07849

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-58K\LG-4-58K 2008-06-10 20-09-53\SIG1000001.D
Sample Name: lg-4-58k

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 7
Injection Date  : 6/10/2008 8:11:22 PM           Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-58K\LG-4-58K 2008-06-10 20-09-53\ASH-50-50-205NM-08ML-
20MIN.M
Last changed    : 6/7/2008 4:15:48 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-58K\LG-4-58K 2008-06-10 20-09-53\SIG1000001.D\DA.M (ASH-50-
50-205NM-08ML-20MIN.M)
Last changed    : 6/14/2008 10:12:32 PM by xzy
(modified after loading)
Method Info     : ASH-50-50-205NM-08ML-20MIN
=====
```



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

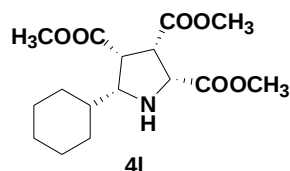
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=205 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.111	MM	0.7021	32.71638	7.76674e-1	1.1979
2	12.990	MM	0.8019	2698.33984	56.08398	98.8021

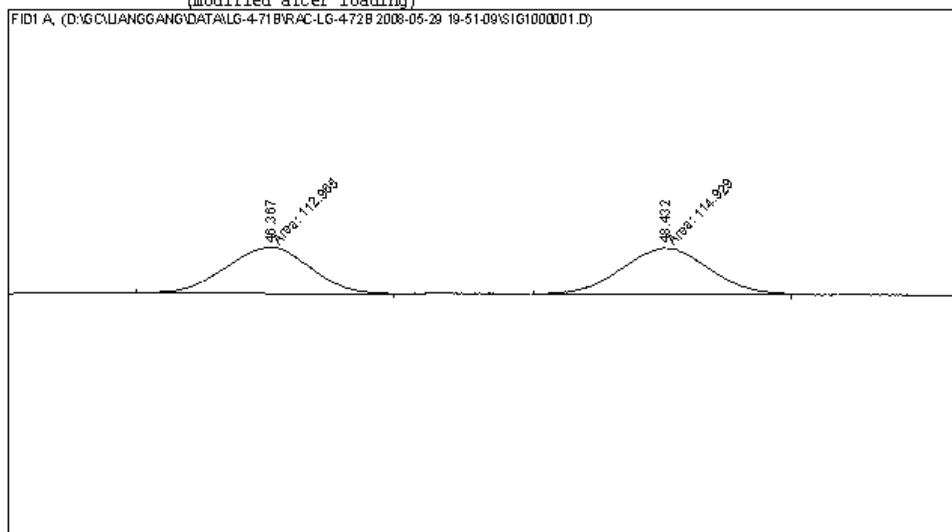
Totals : 2731.05622 56.86066

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



Data File D:\GC\LIANGGANG\DATA\LG-4-71B\RAC-LG-4-72B 2008-05-29 19-51-09\SIG1000001.D
Sample Name: rac-lg-4-72b

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 2                     Location  : Vial 102
Injection Date  : 29-May-08, 19:52:30              Inj       :    1
                                                    Inj Volume: 1 µl
Acq. Method     : D:\GC\lianggang\data\LG-4-71B\RAC-LG-4-72B 2008-05-29 19-51-09\CHIRAL SELECT
                                                    1000 200-100MIN.M
Last changed    : 5/20/2008 8:54:32 PM by gao feng
Analysis Method : D:\GC\LIANGGANG\METHOD\CHIRAL SELECT 1000 200-100MIN.M
Last changed    : 6/14/2008 10:25:32 PM by liang gang
                  (modified after loading)
=====
```



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

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

Signal 1: FID1 A,

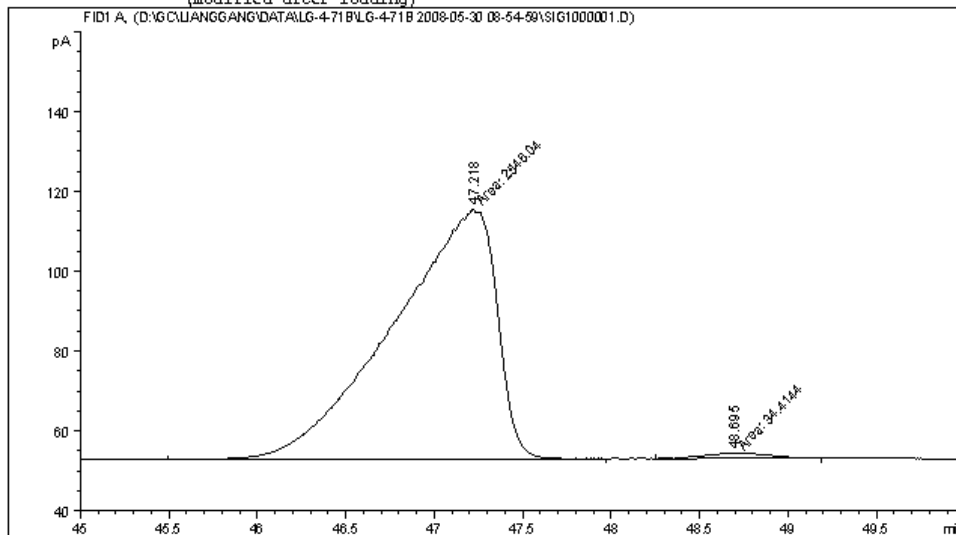
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	46.367	MM	0.5101	112.96505	3.69120	49.56904
2	48.432	MM	0.5189	114.92931	3.69135	50.43096

Totals : 227.89436 7.38256

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

Data File D:\GC\LIANGGANG\DATA\LG-4-71B\LG-4-71B 2008-05-30 08-54-59\SIG1000001.D
Sample Name: lg-4-71b

```
=====
Acq. Operator   : liang gang                Seq. Line :    1
Acq. Instrument : Instrument 2              Location  : Vial 103
Injection Date  : 30-May-08, 08:56:21      Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : D:\GC\lianggang\data\LG-4-71B\LG-4-71B 2008-05-30 08-54-59\CHIRAL SELECT 1000
                                           200-100MIN.M
Last changed    : 5/30/2008 8:48:03 AM by liang gang
Analysis Method : D:\GC\XZP\METHOD\ACHIRAL 75-40.M
Last changed    : 6/14/2008 10:31:58 PM by liang gang
                (modified after loading)
=====
```



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

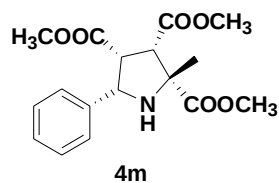
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	47.218	MM	0.6760	2546.04150	62.77246	98.66634
2	48.695	MM	0.4319	34.41440	1.32812	1.33366

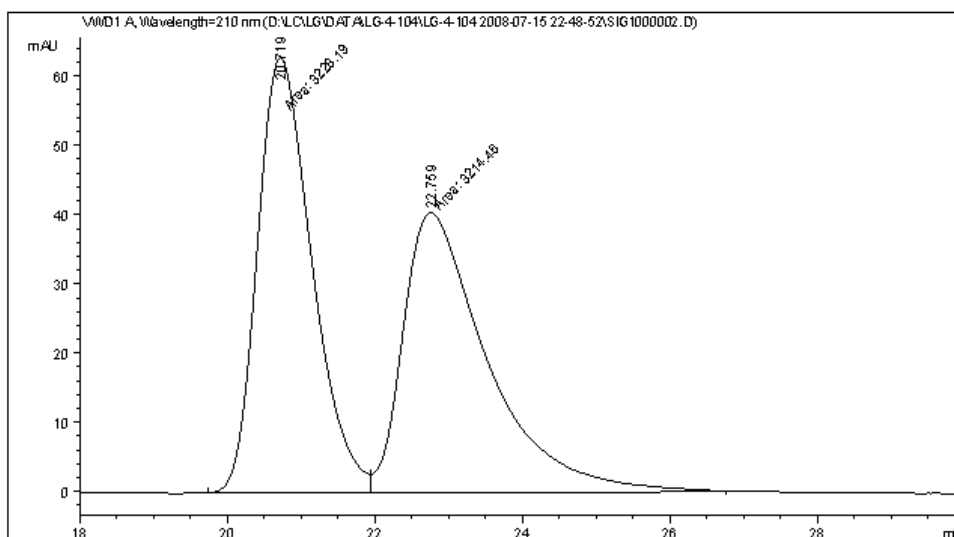
Totals : 2580.45591 64.10057

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



Data File D:\LC\LG\DATA\LG-4-104\LG-4-104 2008-07-15 22-48-52\SIG1000002.D
 Sample Name: rac-1g-4-104

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 21
Injection Date  : 7/15/2008 11:31:47 PM           Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-104\LG-4-104 2008-07-15 22-48-52\ODH-85-15-210NM-08ML-40MIN.M
Last changed    : 7/15/2008 10:45:41 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-104\LG-4-104 2008-07-15 22-48-52\SIG1000002.D\DA.M (ODH-85-15-210NM-08ML-40MIN.M)
Last changed    : 9/4/2008 9:34:47 PM by liang gang
                  (modified after loading)
Method Info     : ODH-85-15-210NM-08ML-40MIN
=====
```



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	20.719	MF	0.8564	3228.19189	62.82407	50.1066
2	22.759	FM	1.3255	3214.45532	40.41879	49.8934

Totals : 6442.64722 103.24286

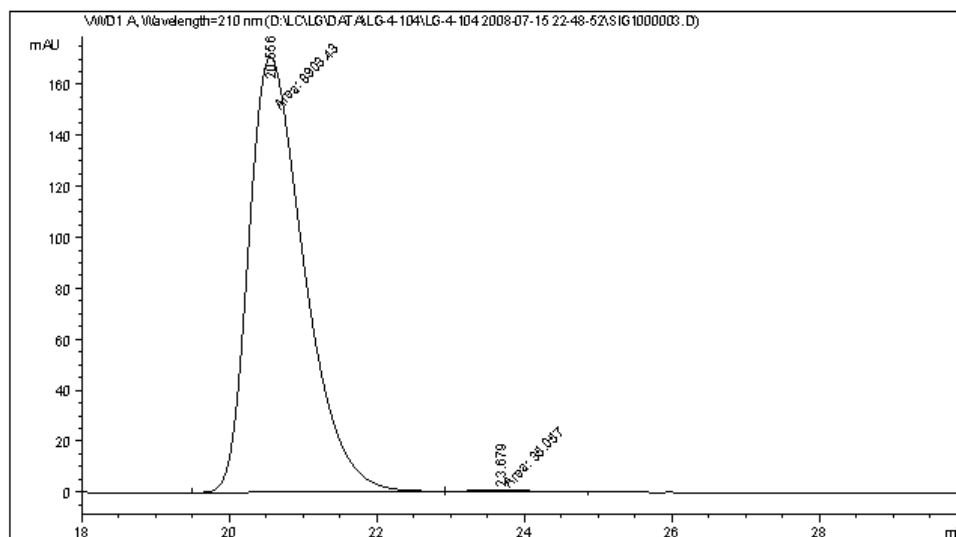
*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-104\LG-4-104 2008-07-15 22-48-52\SIG1000003.D
Sample Name: lg-4-104

```

=====
Acq. Operator   : liang gang                      Seq. Line :    3
Acq. Instrument : Instrument 1                    Location  : Vial 22
Injection Date  : 7/16/2008 12:13:23 AM           Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-104\LG-4-104 2008-07-15 22-48-52\ODH-85-15-210NM-08ML-
40MIN.M
Last changed    : 7/15/2008 10:45:41 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-104\LG-4-104 2008-07-15 22-48-52\SIG1000003.D\DA.M (ODH-85-
15-210NM-08ML-40MIN.M)
Last changed    : 9/4/2008 9:42:06 PM by liang gang
                (modified after loading)
Method Info     : ODH-85-15-210NM-08ML-40MIN
=====

```



Area Percent Report

```

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

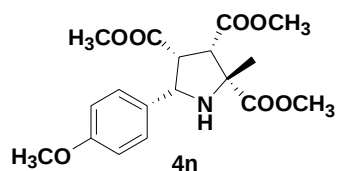
```

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.556	MM	0.8689	8903.42871	170.77814	99.6078
2	23.679	MM	1.2341	35.05701	4.73433e-1	0.3922

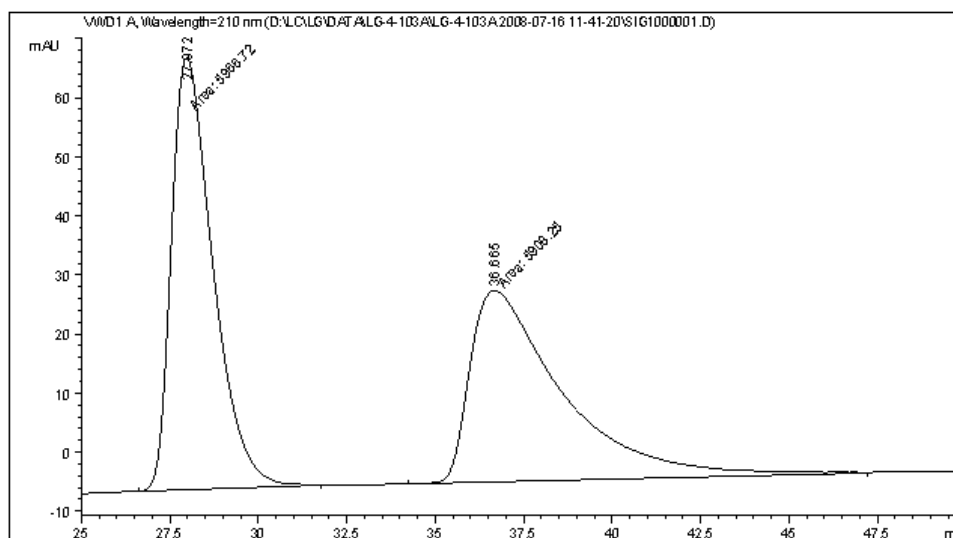
Totals : 8938.48572 171.25157

*** End of Report ***



Data File D:\LC\LG\DATA\LG-4-103A\LG-4-103A 2008-07-16 11-41-20\SIG1000001.D
 Sample Name: rac-lg-4-103a

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 3
Injection Date  : 7/16/2008 11:43:11 AM           Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-103A\LG-4-103A 2008-07-16 11-41-20\ODH-85-15-210NM-08ML-50MIN.M
Last changed    : 7/16/2008 11:40:37 AM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-103A\LG-4-103A 2008-07-16 11-41-20\SIG1000001.D\DA.M (ODH-85-15-210NM-08ML-50MIN.M)
Last changed    : 9/4/2008 9:25:37 PM by liang gang
                  (modified after loading)
Method Info     : ODH-85-15-210NM-08ML-50MIN
=====
```



Area Percent Report

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

Signal 1: WVD1 A, Wavelength=210 nm

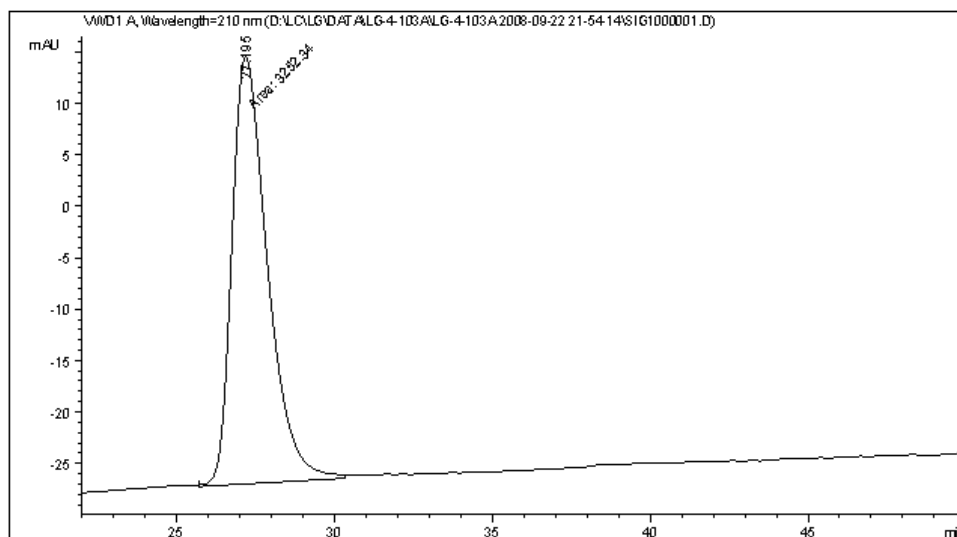
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	27.972	MM	1.3611	5966.72461	73.06190	50.2462
2	36.665	MM	3.0402	5908.24658	32.38983	49.7538

Totals : 1.18750e4 105.45173

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-103A\LG-4-103A 2008-09-22 21-54-14\SIG1000001.D
Sample Name: lg-4-103

```
=====
Acq. Operator   : liangqiang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 25
Injection Date  : 9/22/2008 9:55:35 PM           Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-103A\LG-4-103A 2008-09-22 21-54-14\ODH-85-15-210NM-08ML-
50MIN.M
Last changed    : 9/9/2008 4:36:02 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-103A\LG-4-103A 2008-09-22 21-54-14\SIG1000001.D\DA.M (ODH-
85-15-210NM-08ML-50MIN.M)
Last changed    : 9/28/2008 11:07:20 AM by liang gang
(modified after loading)
Method Info     : ODH-85-15-210NM-08ML-50MIN
=====
```



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

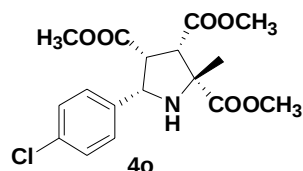
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	27.195	MM	1.3059	3252.34326	41.50711	100.0000

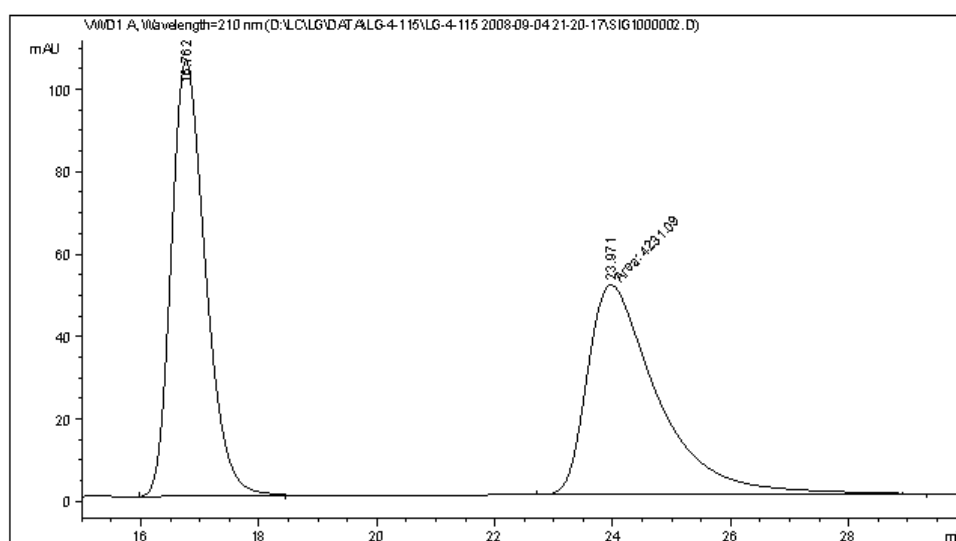
Totals : 3252.34326 41.50711

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



Data File D:\LC\LG\DATA\LG-4-115\LG-4-115 2008-09-04 21-20-17\SIG1000002.D
Sample Name: rac-lg-4-115

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 4
Injection Date  : 9/4/2008 9:32:56 PM             Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-115\LG-4-115 2008-09-04 21-20-17\ODH-85-15-210NM-08ML-40MIN.M
Last changed    : 8/1/2008 4:40:22 PM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-115\LG-4-115 2008-09-04 21-20-17\SIG1000002.D\DA.M (ODH-85-15-210NM-08ML-40MIN.M)
Last changed    : 9/4/2008 10:05:35 PM by liang gang
                  (modified after loading)
Method Info     : ODH-85-15-210NM-08ML-40MIN
=====
```



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

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

Signal 1: VWD1 A, Wavelength=210 nm

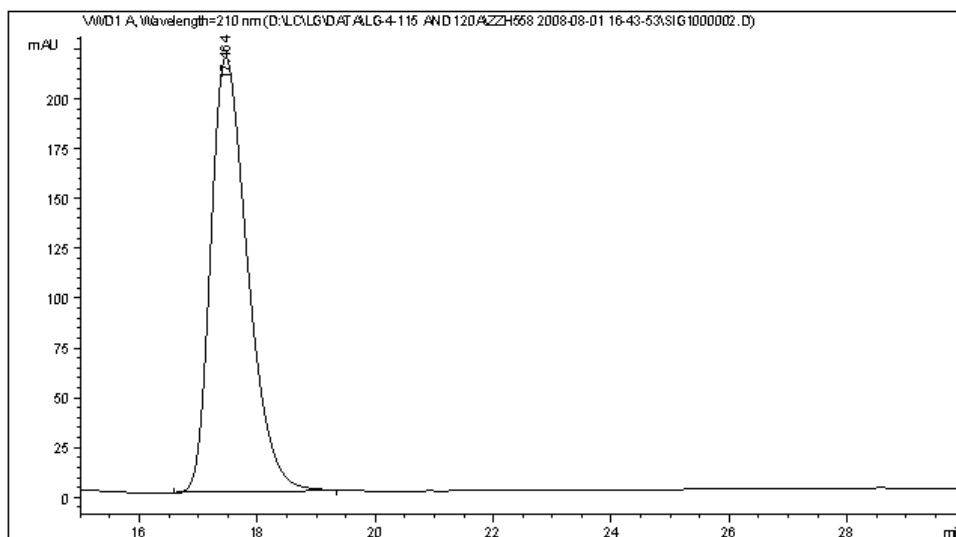
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.762	BB	0.6201	4256.42725	105.63582	50.1493
2	23.971	MM	1.3846	4231.09033	50.93185	49.8507

Totals : 8487.51758 156.56767

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

Data File D:\LC\LG\DATA\LG-4-115 AND 120A\ZZH558 2008-08-01 16-43-53\SIG1000002.D
Sample Name: lg-4-115

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 7
Injection Date  : 8/1/2008 4:56:34 PM              Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-115 AND 120A\ZZH558 2008-08-01 16-43-53\ODH-85-15-210NM-
                  08ML-40MIN.M
Last changed    : 8/1/2008 4:40:22 PM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-115 AND 120A\ZZH558 2008-08-01 16-43-53\SIG1000002.D\DA.M (
                  ODH-85-15-210NM-08ML-40MIN.M)
Last changed    : 9/4/2008 9:21:52 PM by liang gang
                  (modified after loading)
Method Info     : ODH-85-15-210NM-08ML-40MIN
=====
```



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

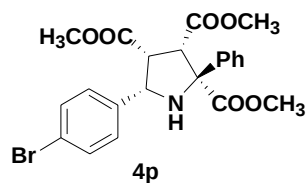
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.464	BB	0.6692	9459.69434	218.06978	100.0000

Totals : 9459.69434 218.06978

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

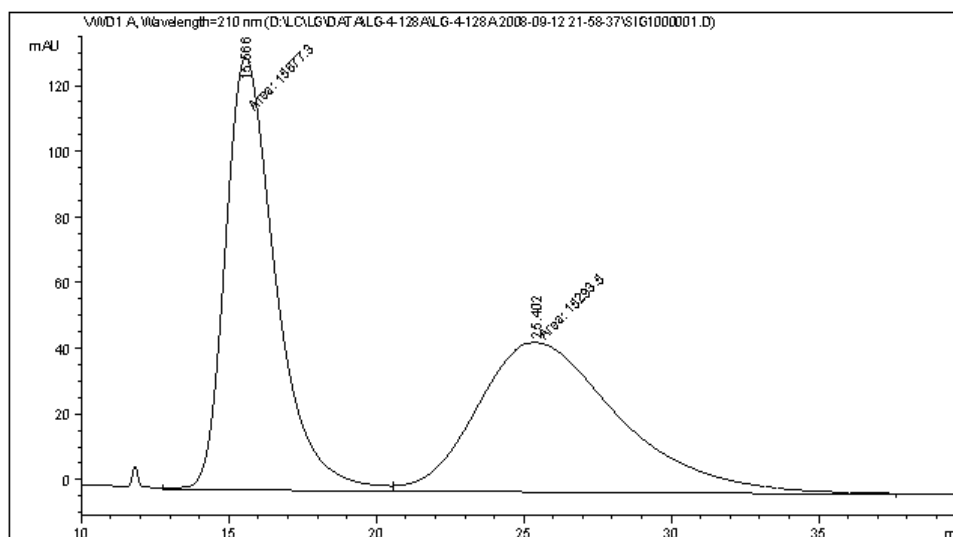


Data File D:\LC\LG\DATA\LG-4-128A\LG-4-128A 2008-09-12 21-58-37\SIG1000001.D

Sample Name: lg-4-128a

```

=====
Acq. Operator   : liang gang          Seq. Line :    1
Acq. Instrument : Instrument 1         Location  : Vial 41
Injection Date  : 9/12/2008 10:00:01 PM Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-128A\LG-4-128A 2008-09-12 21-58-37\0JH-70-30-210NM-08ML-
                    50MIN.M
Last changed    : 9/9/2008 8:57:33 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-128A\LG-4-128A 2008-09-12 21-58-37\SIG1000001.D\DA.M (0JH-
                    70-30-210NM-08ML-50MIN.M)
Last changed    : 9/27/2008 10:35:39 AM by dxq
                    (modified after loading)
Method Info     : 0JH-70-30-210NM-08ML-50MIN
=====
  
```



Area Percent Report

```

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

Signal 1: VWD1 A, Wavelength=210 nm

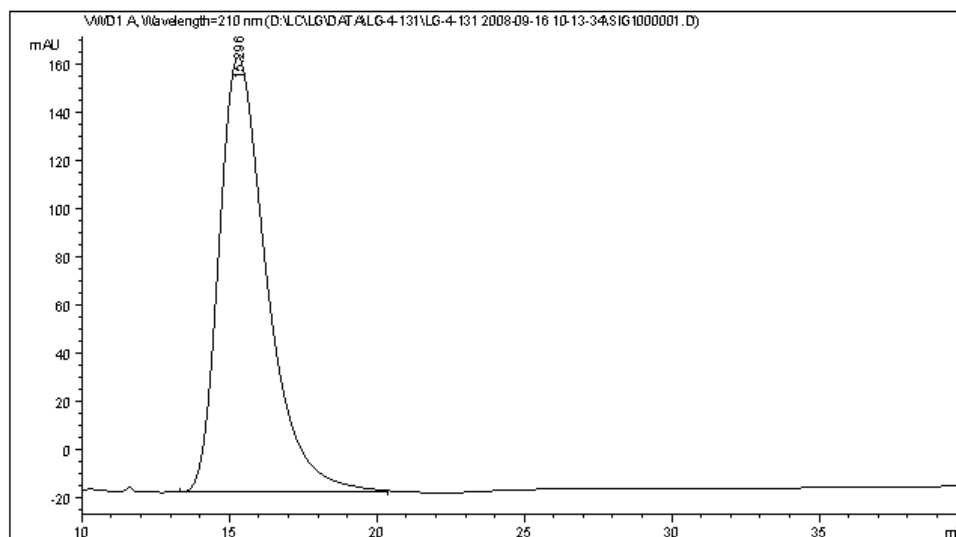
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	15.566	MF	1.9862	1.56773e4	131.55162	50.6196
2	25.402	FM	5.6057	1.52935e4	45.46980	49.3804

Totals : 3.09708e4 177.02142

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-131\LG-4-131 2008-09-16 10-13-34\SIG1000001.D
Sample Name: lg-4-128a

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location : Vial 24
Injection Date  : 9/16/2008 10:14:56 AM           Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-131\LG-4-131 2008-09-16 10-13-34\OJH-70-30-210NM-08ML-
50MIN.M
Last changed    : 9/16/2008 10:08:03 AM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-131\LG-4-131 2008-09-16 10-13-34\SIG1000001.D\DA.M (OJH-70-
30-210NM-08ML-50MIN.M)
Last changed    : 9/27/2008 10:37:56 AM by dxq
                  (modified after loading)
Method Info     : OJH-70-30-210NM-08ML-50MIN
=====
```



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

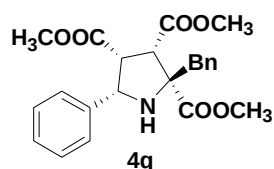
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.296	BB	1.6941	2.01924e4	180.47246	100.0000

Totals : 2.01924e4 180.47246

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

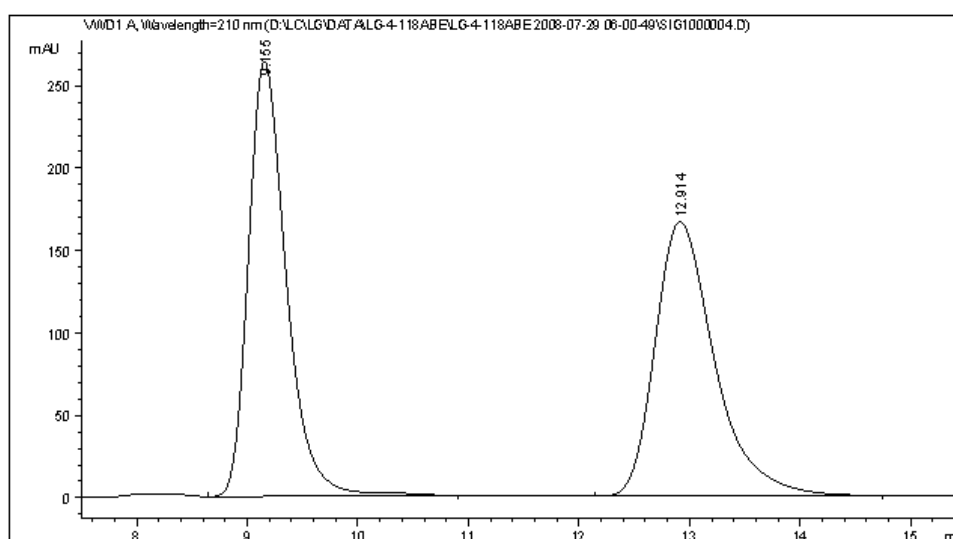


Data File D:\LC\LG\DATA\LG-4-118ABE\LG-4-118ABE 2008-07-29 06-00-49\SIG1000004.D
Sample Name: lg-4-119e

```

=====
Acq. Operator   : liang gang                      Seq. Line :    4
Acq. Instrument : Instrument 1                    Location  : Vial 6
Injection Date  : 7/29/2008 7:56:44 AM           Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-118ABE\LG-4-118ABE 2008-07-29 06-00-49\ODH-85-15-210NM-
                                                08ML-50MIN.M
Last changed    : 7/29/2008 6:00:38 AM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-118ABE\LG-4-118ABE 2008-07-29 06-00-49\SIG1000004.D\DA.M (
                                                ODH-85-15-210NM-08ML-50MIN.M)
Last changed    : 9/4/2008 8:51:54 PM by liang gang
                                                (modified after loading)
Method Info     : ODH-85-15-210NM-08ML-50MIN
=====

```



Area Percent Report

```

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

```

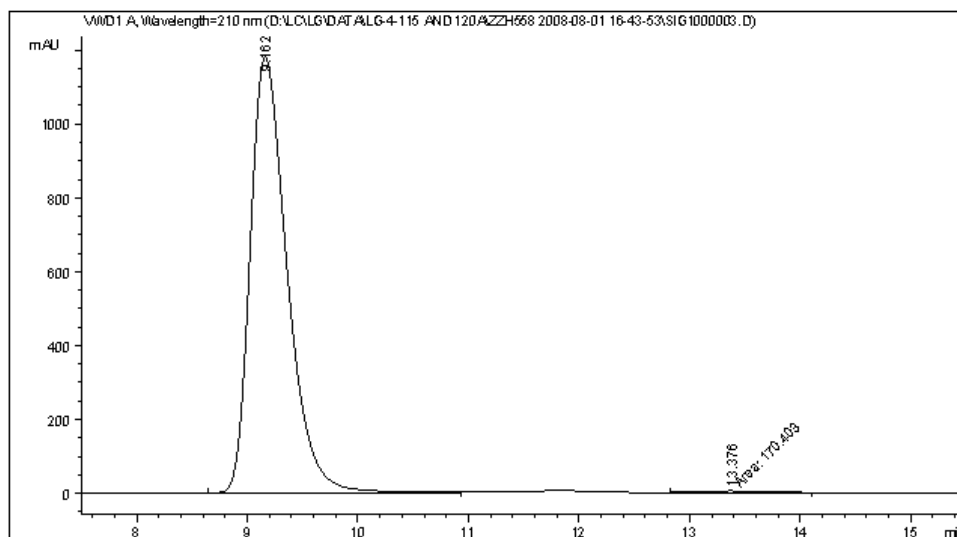
Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	9.155	VB	0.3639	6292.00342	264.07785	50.2450
2	12.914	VB	0.5687	6230.63281	166.64453	49.7550
Totals :				1.25226e4	430.72238	

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-115 AND 120A\ZZH558 2008-08-01 16-43-53\SIG1000003.D
Sample Name: lg-4-120a

```
=====
Acq. Operator   : liang gang                      Seq. Line :    3
Acq. Instrument : Instrument 1                     Location  : Vial 8
Injection Date  : 8/1/2008 5:38:19 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-115 AND 120A\ZZH558 2008-08-01 16-43-53\ODH-85-15-210NM-
08ML-25MIN.M
Last changed    : 8/1/2008 4:41:23 PM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-115 AND 120A\ZZH558 2008-08-01 16-43-53\SIG1000003.D\DA.M (
ODH-85-15-210NM-08ML-25MIN.M)
Last changed    : 9/4/2008 8:56:03 PM by liang gang
(modified after loading)
Method Info     : ODH-85-15-210NM-08ML-25MIN
=====
```



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

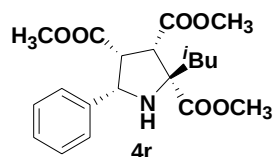
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.162	VB	0.3551	2.73746e4	1179.94116	99.3814
2	13.376	MM	0.6334	170.40274	4.48363	0.6186

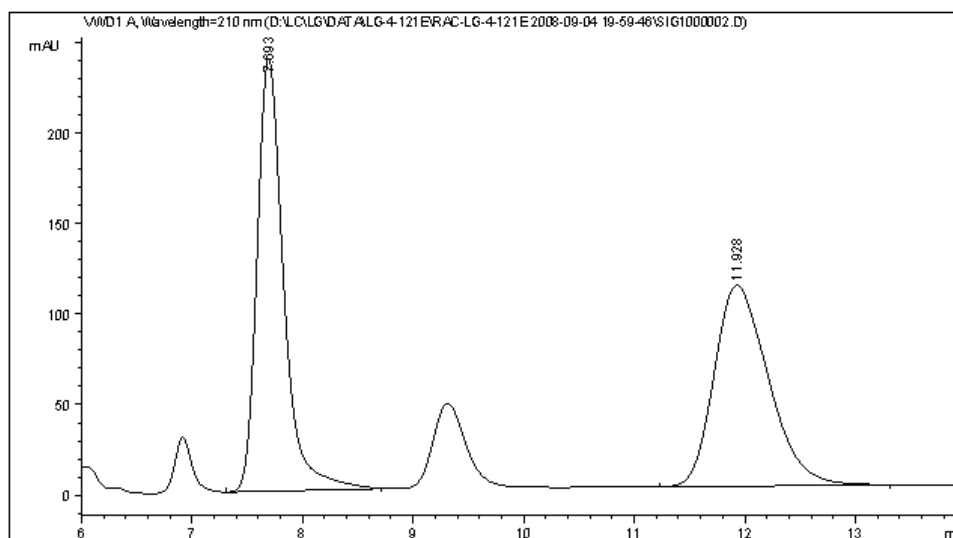
Totals : 2.75450e4 1184.42479

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



Data File D:\LC\LG\DATA\LG-4-121E\RAC-LG-4-121E 2008-09-04 19-59-46\SIG1000002.D
 Sample Name: rac-lg-4-121e

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                     Location  : Vial 18
Injection Date  : 9/4/2008 8:12:29 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-121E\RAC-LG-4-121E 2008-09-04 19-59-46\ODH-85-15-210NM-
08ML-25MIN.M
Last changed    : 8/1/2008 4:41:23 PM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-121E\RAC-LG-4-121E 2008-09-04 19-59-46\SIG1000002.D\DA.M (
ODH-85-15-210NM-08ML-25MIN.M)
Last changed    : 9/4/2008 8:37:39 PM by liang gang
(modified after loading)
Method Info     : ODH-85-15-210NM-08ML-25MIN
=====
```



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=210 nm

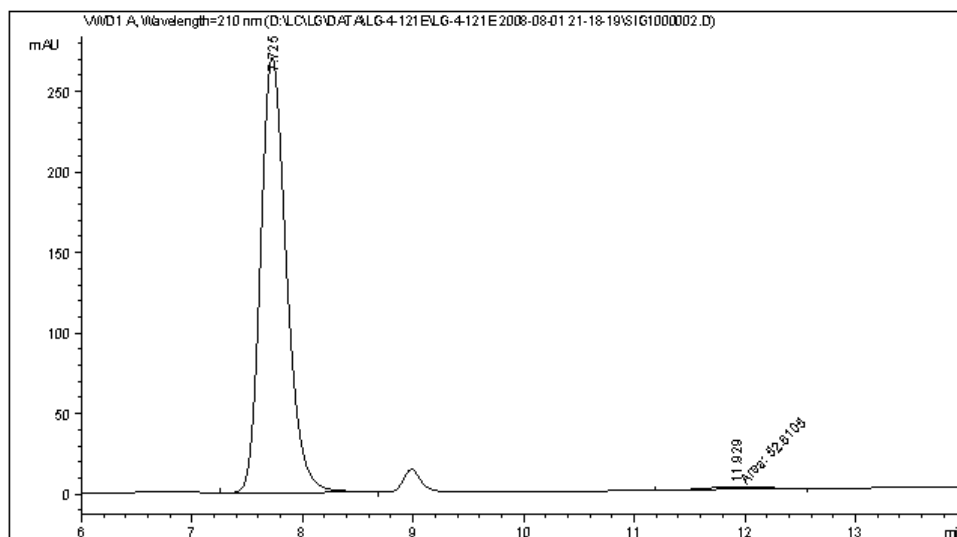
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	7.693	VB	0.2545	3990.94043	239.07321	51.3134
2	11.928	BB	0.5424	3786.63184	110.91858	48.6866

Totals : 7777.57227 349.99179

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-121E\LG-4-121E 2008-08-01 21-18-19\SIG1000002.D
Sample Name: lg-4-121e

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 11
Injection Date  : 8/1/2008 9:30:57 PM             Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-121E\LG-4-121E 2008-08-01 21-18-19\ODH-85-15-210NM-08ML-
40MIN.M
Last changed    : 8/1/2008 4:40:22 PM by dxq
Analysis Method : D:\LC\LG\DATA\LG-4-121E\LG-4-121E 2008-08-01 21-18-19\SIG1000002.D\DA.M (ODH-
85-15-210NM-08ML-40MIN.M)
Last changed    : 9/4/2008 8:49:08 PM by liang gang
(modified after loading)
Method Info     : ODH-85-15-210NM-08ML-40MIN
=====
```



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

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

Signal 1: WVD1 A, Wavelength=210 nm

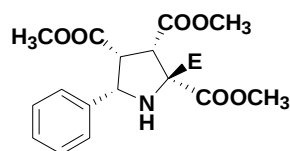
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.725	VB	0.2503	4385.20752	270.43619	98.8145
2	11.929	MM	0.5569	52.61049	1.57442	1.1855

Totals : 4437.81800 272.01061

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

Instrument 1 9/4/2008 8:49:11 PM liang gang

Page 1 of 1



E = 3-indolymethyl

4s

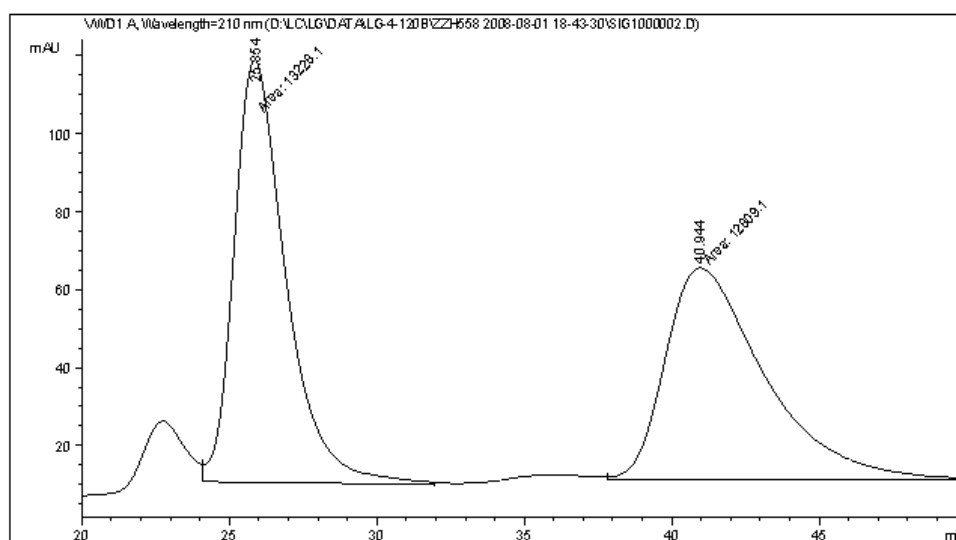
Data File D:\LC\LG\DATA\LG-4-120B\ZZH558 2008-08-01 18-43-30\SIG1000002.D

Sample Name: rac-lg-4-120

```

=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 9
Injection Date  : 8/1/2008 6:56:13 PM              Inj       :    1
                                              Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-120B\ZZH558 2008-08-01 18-43-30\ODH-85-15-210NM-08ML-50MIN.
                                              M
Last changed    : 7/29/2008 6:00:38 AM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-120B\ZZH558 2008-08-01 18-43-30\SIG1000002.D\DA.M (ODH-85-
15-210NM-08ML-50MIN.M)
Last changed    : 9/4/2008 9:03:12 PM by liang gang
                  (modified after loading)
Method Info     : ODH-85-15-210NM-08ML-50MIN
=====

```



Area Percent Report

```

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

```

Signal 1: VWD1 A, Wavelength=210 nm

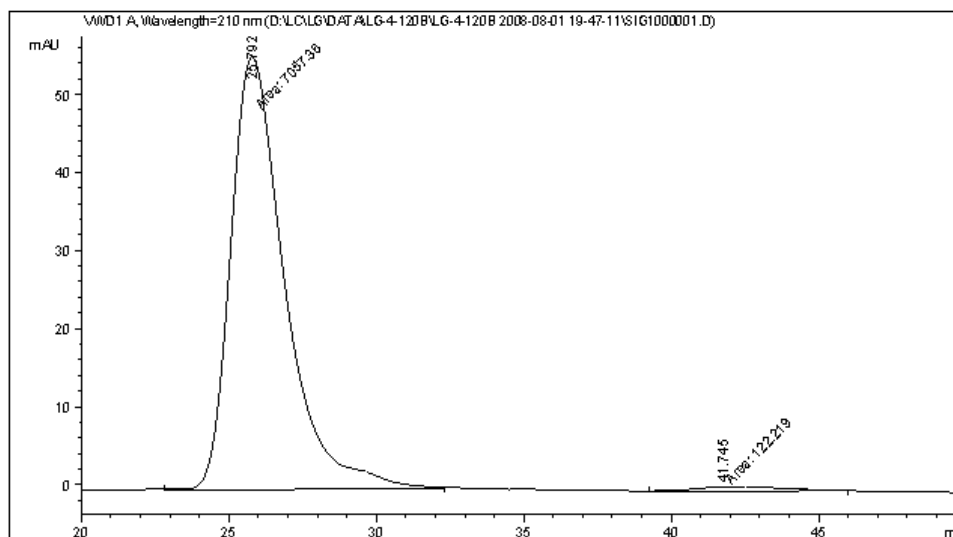
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	25.854	MM	2.0385	1.32281e4	108.15330	50.8045
2	40.944	MM	3.9259	1.28091e4	54.37944	49.1955

Totals : 2.60372e4 162.53274

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-120B\LG-4-120B 2008-08-01 19-47-11\SIG1000001.D
Sample Name: g-4-120

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 10
Injection Date  : 8/1/2008 7:48:44 PM             Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-120B\LG-4-120B 2008-08-01 19-47-11\ODH-85-15-210NM-08ML-
50MIN.M
Last changed    : 7/29/2008 6:00:38 AM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-120B\LG-4-120B 2008-08-01 19-47-11\SIG1000001.D\DA.M (ODH-
85-15-210NM-08ML-50MIN.M)
Last changed    : 9/4/2008 9:07:06 PM by liang gang
(modified after loading)
Method Info     : ODH-85-15-210NM-08ML-50MIN
=====
```



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

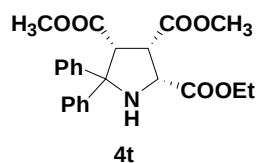
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	25.792	MM	2.1243	7057.35938	55.36968	98.2977
2	41.745	MM	3.7859	122.21851	5.38042e-1	1.7023

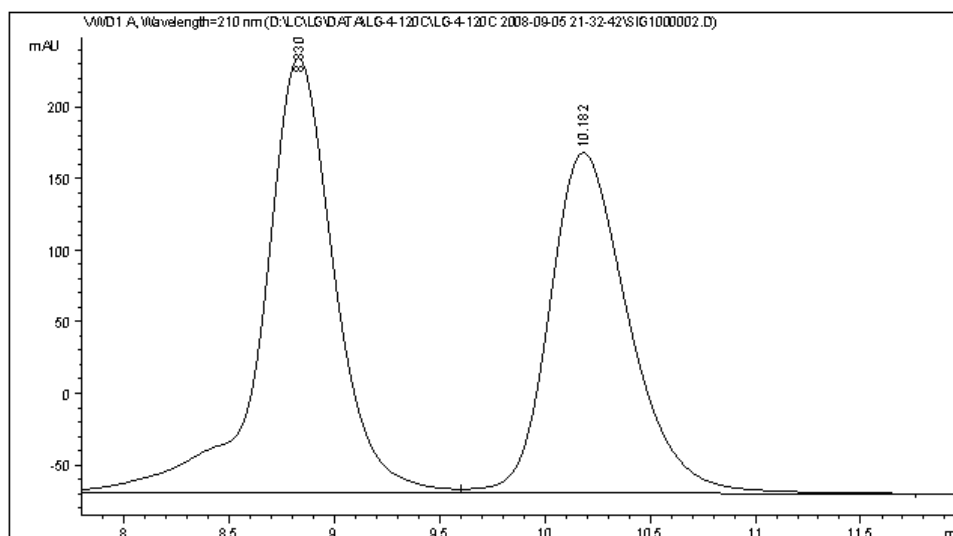
Totals : 7179.57789 55.90772

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



Data File D:\LC\LG\DATA\LG-4-120C\LG-4-120C 2008-09-05 21-32-42\SIG1000002.D
Sample Name: rac-lg-4-120c

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 19
Injection Date  : 9/5/2008 9:45:25 PM             Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-120C\LG-4-120C 2008-09-05 21-32-42\ODH-85-15-210NM-08ML-50MIN.M
Last changed    : 8/28/2008 8:03:58 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-120C\LG-4-120C 2008-09-05 21-32-42\SIG1000002.D\DA.M (ODH-85-15-210NM-08ML-50MIN.M)
Last changed    : 9/27/2008 10:29:29 AM by dxq
                  (modified after loading)
Method Info     : ODH-85-15-210NM-08ML-50MIN
=====
```



Area Percent Report

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

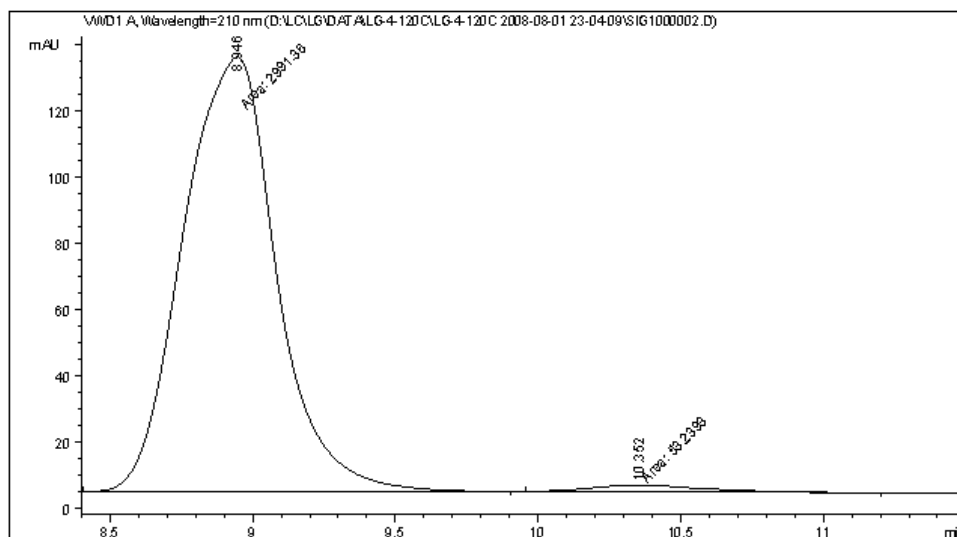
Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	8.830	VV	0.3543	7209.29639	303.51584	53.7866
2	10.182	VB	0.3986	6194.20996	237.74800	46.2134
Totals :				1.34035e4	541.26384	

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-120C\LG-4-120C 2008-08-01 23-04-09\SIG1000002.D
Sample Name: lg-4-120c-a

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                     Location  : Vial 12
Injection Date  : 8/1/2008 11:16:48 PM             Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-120C\LG-4-120C 2008-08-01 23-04-09\ODH-85-15-210NM-08ML-
50MIN.M
Last changed    : 8/1/2008 9:17:30 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-120C\LG-4-120C 2008-08-01 23-04-09\SIG1000002.D\DA.M (ODH-
85-15-210NM-08ML-50MIN.M)
Last changed    : 9/27/2008 10:24:09 AM by dxq
(modified after loading)
Method Info     : ODH-85-15-210NM-08ML-50MIN
=====
```



Area Percent Report

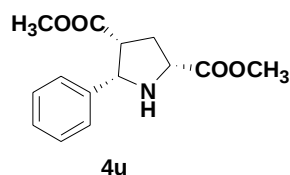
```
=====
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.946	MM	0.3797	2991.35889	131.28754	98.2514
2	10.352	MM	0.4427	53.23930	2.00424	1.7486

Totals : 3044.59819 133.29178

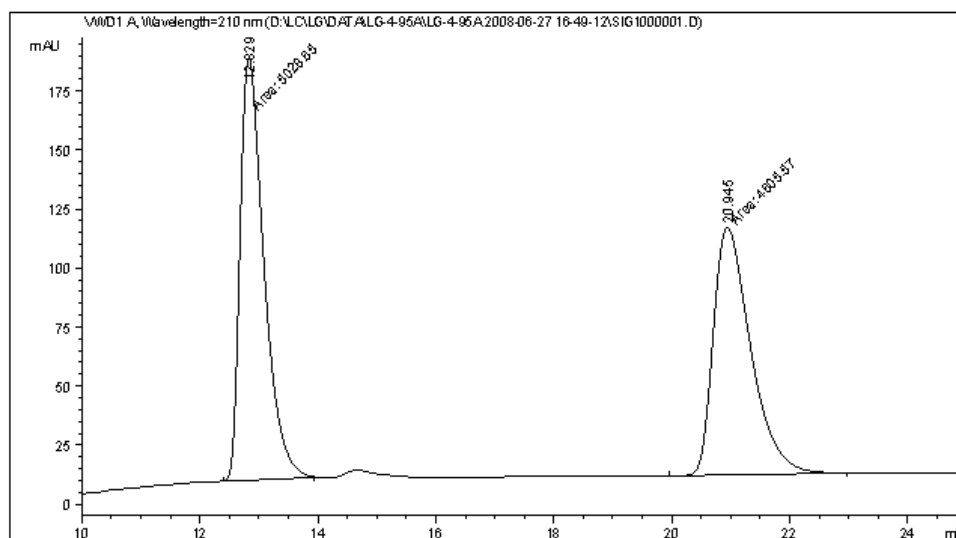
*** End of Report ***



Data File D:\LC\LG\DATA\LG-4-95A\LG-4-95A 2008-06-27 16-49-12\SIG1000001.D
 Sample Name: lg-4-95a

```

=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 3
Injection Date  : 6/27/2008 4:50:35 PM             Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-95A\LG-4-95A 2008-06-27 16-49-12\ASH-50-50-210NM-10ML-40MIN.M
Last changed    : 6/27/2008 4:44:41 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-95A\LG-4-95A 2008-06-27 16-49-12\SIG1000001.D\DA.M (ASH-50-50-210NM-10ML-40MIN.M)
Last changed    : 9/4/2008 8:19:25 PM by liang gang
                  (modified after loading)
Method Info     : ASH-50-50-210NM-10ML-40MIN
  
```



Area Percent Report

```

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

Signal 1: VWD1 A, Wavelength=210 nm

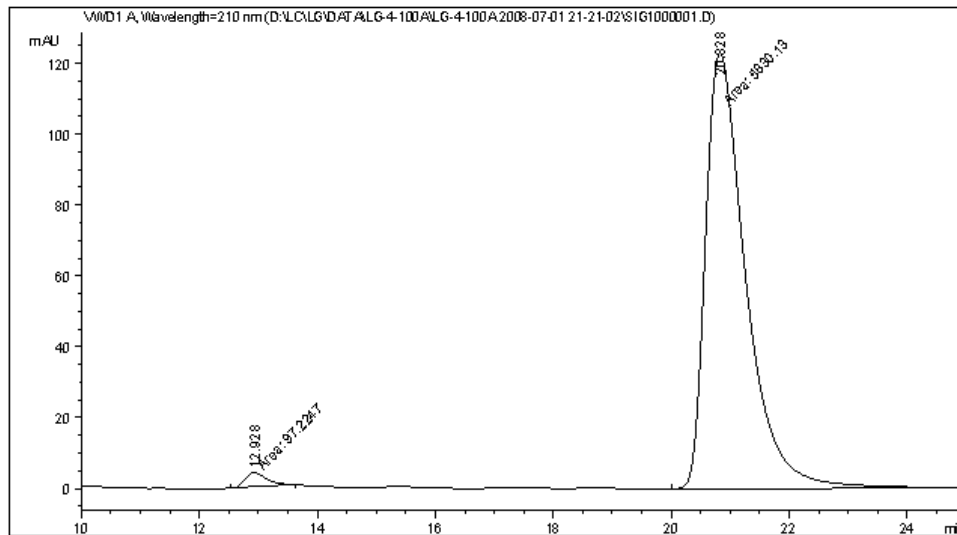
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.829	MM	0.4684	5028.65332	178.94913	52.1957
2	20.945	MM	0.7319	4605.57422	104.87179	47.8043

Totals : 9634.22754 283.82092

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-100A\LG-4-100A 2008-07-01 21-21-02\SIG1000001.D
Sample Name: lg-4-100a

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 4
Injection Date  : 7/1/2008 9:22:39 PM              Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-100A\LG-4-100A 2008-07-01 21-21-02\ASH-50-50-210NM-10ML-
40MIN.M
Last changed    : 7/1/2008 8:45:52 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-100A\LG-4-100A 2008-07-01 21-21-02\SIG1000001.D\DA.M (ASH-
50-50-210NM-10ML-40MIN.M)
Last changed    : 9/4/2008 8:29:20 PM by liang gang
                (modified after loading)
Method Info     : ASH-50-50-210NM-10ML-40MIN
=====
```



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

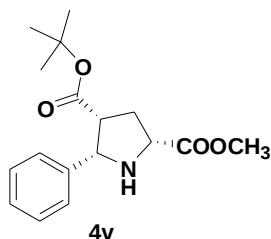
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.928	MM	0.4120	97.22474	3.93323	1.6403
2	20.828	MM	0.7907	5830.12891	122.88190	98.3597

Totals : 5927.35365 126.81513

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

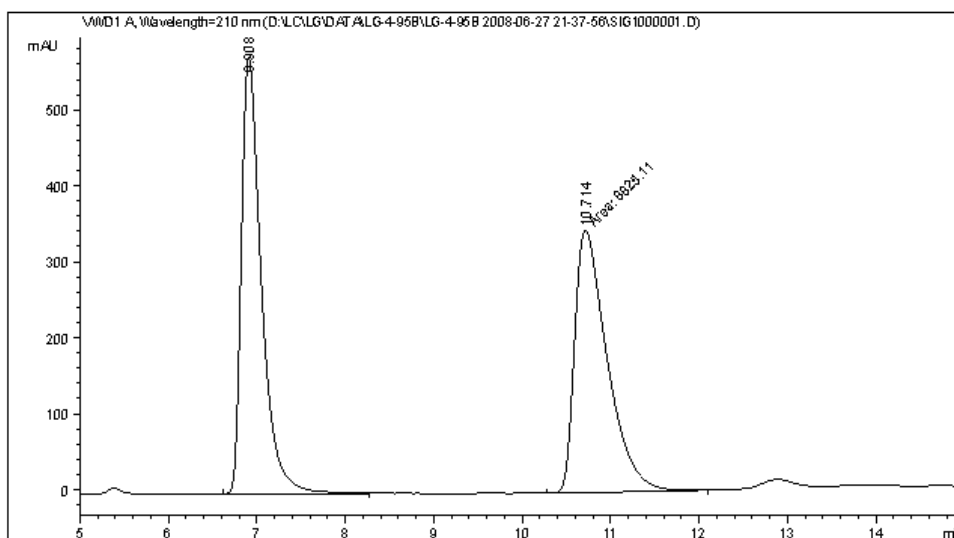


Data File D:\LC\LG\DATA\LG-4-95B\LG-4-95B 2008-06-27 21-37-56\SIG1000001.D
Sample Name: rac-lg-4-95b

```

=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 5
Injection Date  : 6/27/2008 9:39:19 PM             Inj       :    1
                                              Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-95B\LG-4-95B 2008-06-27 21-37-56\ASH-50-50-210NM-10ML-
                  40MIN.M
Last changed    : 6/27/2008 9:33:11 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-95B\LG-4-95B 2008-06-27 21-37-56\SIG1000001.D\DA.M (ASH-50-
                  50-210NM-10ML-40MIN.M)
Last changed    : 9/4/2008 8:24:29 PM by liang gang
                  (modified after loading)
Method Info     : ASH-50-50-210NM-10ML-40MIN
=====

```



Area Percent Report

```

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

```

Signal 1: VWD1 A, Wavelength=210 nm

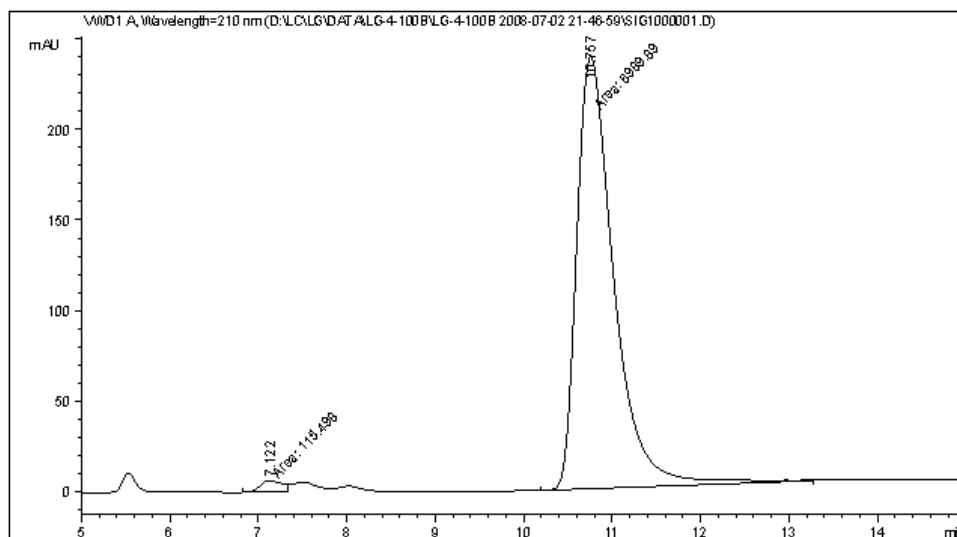
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.908	BB	0.2368	8991.80371	573.54755	50.4678
2	10.714	MM	0.4254	8825.11230	345.75031	49.5322

Totals : 1.78169e4 919.29785

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-100B\LG-4-100B 2008-07-02 21-46-59\SIG1000001.D
Sample Name: lg-4-100b-CH2c12

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location : Vial 6
Injection Date  : 7/2/2008 9:48:27 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-100B\LG-4-100B 2008-07-02 21-46-59\ASH-50-50-210NM-10ML-
                  16MIN.M
Last changed    : 7/2/2008 9:40:37 PM by zhang zhihai
Analysis Method : D:\LC\LG\DATA\LG-4-100B\LG-4-100B 2008-07-02 21-46-59\SIG1000001.D\DA.M (ASH-
                  50-50-210NM-10ML-16MIN.M)
Last changed    : 9/4/2008 8:45:51 PM by liang gang
                  (modified after loading)
Method Info     : ASH-50-50-210NM-10ML-16MIN
=====
```



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

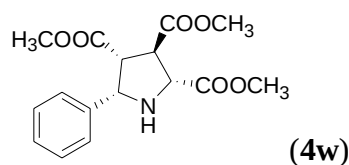
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.122	MM	0.2961	115.49764	6.50171	1.6255
2	10.757	MM	0.4882	6989.89111	238.64571	98.3745

Totals : 7105.38876 245.14742

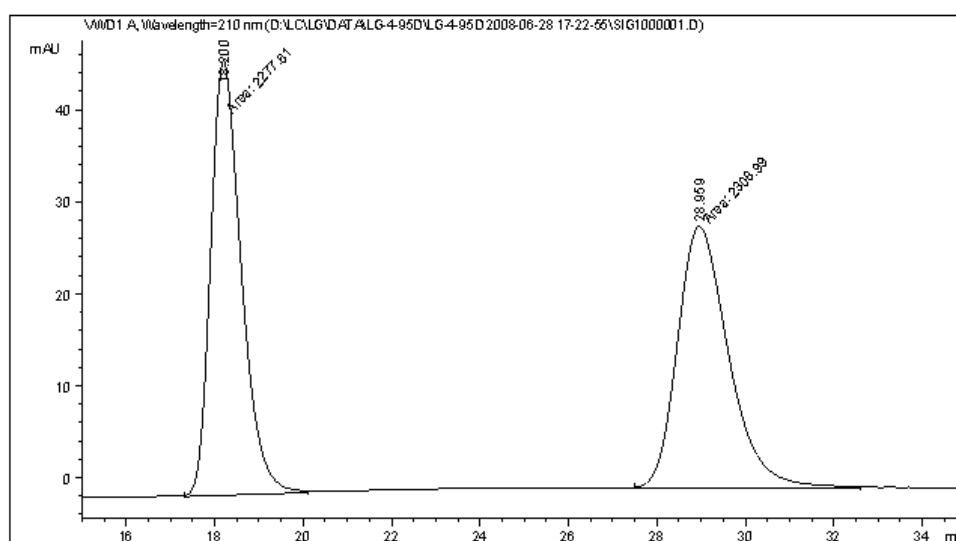
=====
*** End of Report ***



Data File D:\LC\LG\DATA\LG-4-95D\LG-4-95D 2008-06-28 17-22-55\SIG1000001.D
Sample Name: rac-lg-4-95d

```

=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 9
Injection Date  : 6/28/2008 5:24:25 PM             Inj       :    1
                                              Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-95D\LG-4-95D 2008-06-28 17-22-55\ODH-70-30-210NM-08ML-
40MIN.M
Last changed    : 6/28/2008 5:16:42 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-95D\LG-4-95D 2008-06-28 17-22-55\SIG1000001.D\DA.M (ODH-70-
30-210NM-08ML-40MIN.M)
Last changed    : 11/8/2008 6:31:13 PM by liang gang
                  (modified after loading)
Method Info     : ODH-70-30-210NM-08ML-40MIN
  
```



Area Percent Report

```

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

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.200	MM	0.8041	2277.60669	47.21064	49.6579
2	28.959	MM	1.3489	2308.98560	28.53025	50.3421
Totals :				4586.59229	75.74089	

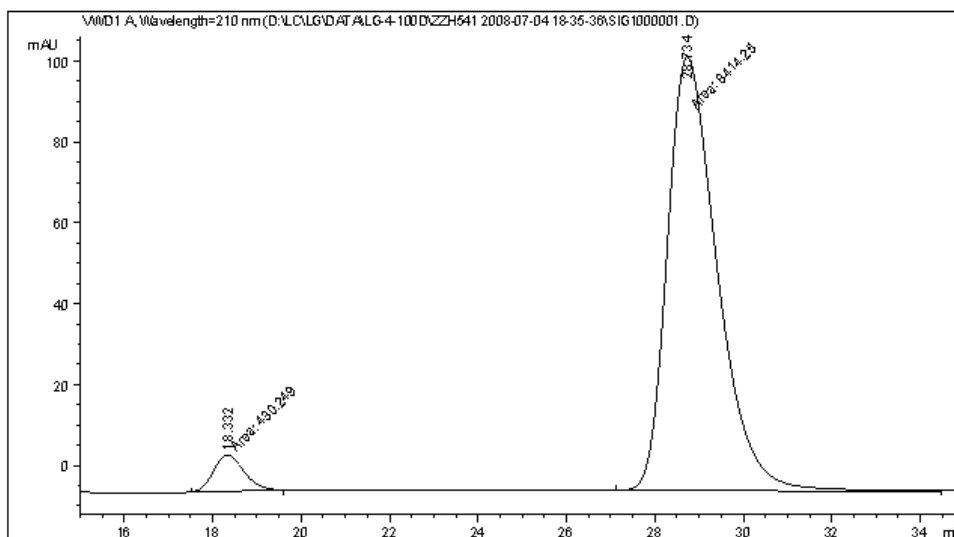
*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-100D\ZZH541 2008-07-04 18-35-36\SIG1000001.D
Sample Name: lg-4-100d

```

=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                    Location  : Vial 10
Injection Date  : 7/4/2008 6:37:07 PM              Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-100D\ZZH541 2008-07-04 18-35-36\ODH-70-30-210NM-08ML-40MIN.
M
Last changed    : 6/28/2008 5:16:42 PM by liang gang
Analysis Method : D:\LC\LG\DATA\LG-4-100D\ZZH541 2008-07-04 18-35-36\SIG1000001.D\DA.M (ODH-70-
30-210NM-08ML-40MIN.M)
Last changed    : 11/8/2008 6:26:56 PM by liang gang
(modified after loading)
Method Info     : ODH-70-30-210NM-08ML-40MIN
=====

```



Area Percent Report

```

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

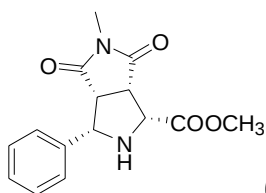
```

Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.332	MM	0.7947	430.24924	9.02360	4.8646
2	28.734	MM	1.3078	8414.24707	107.23325	95.1354

Totals : 8844.49631 116.25685

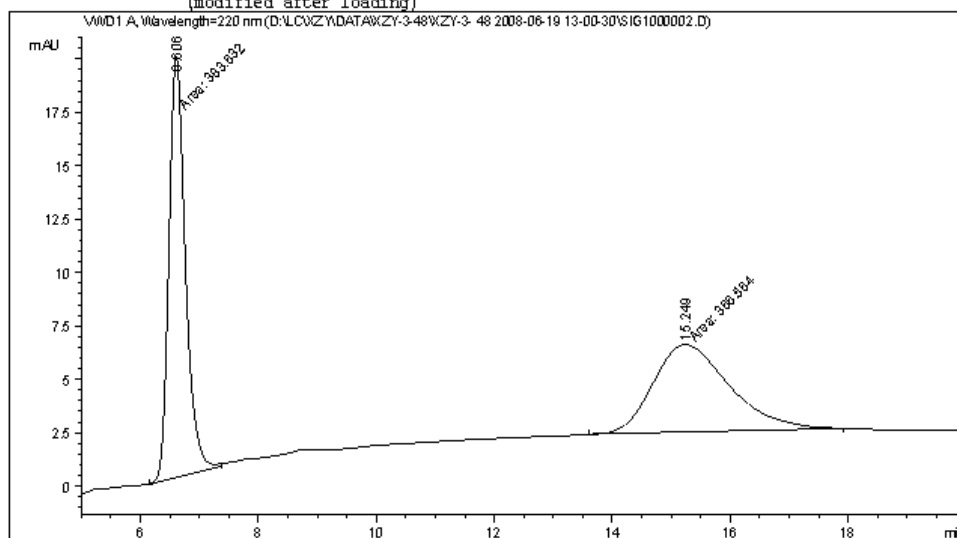
*** End of Report ***



(4x)

Data File D:\LC\XYZ\DATA\XYZY-3-48\XYZY-3- 48 2008-06-19 13-00-30\SIG1000002.D
Sample Name: xzy-3-48d

```
=====
Acq. Operator   : xzy                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 51
Injection Date  : 6/19/2008 1:13:08 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\XYZ\DATA\XYZY-3-48\XYZY-3- 48 2008-06-19 13-00-30\ASH-50-50-15ML-220NM.M
Last changed    : 5/27/2008 4:07:32 PM by xzy
Analysis Method : D:\LC\XYZ\DATA\XYZY-3-48\XYZY-3- 48 2008-06-19 13-00-30\SIG1000002.D\DA.M (ASH-
50-50-15ML-220NM.M)
Last changed    : 11/10/2008 8:39:40 PM by liang gang
                  (modified after loading)
=====
```



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

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

Signal 1: VWD1 A, Wavelength=220 nm

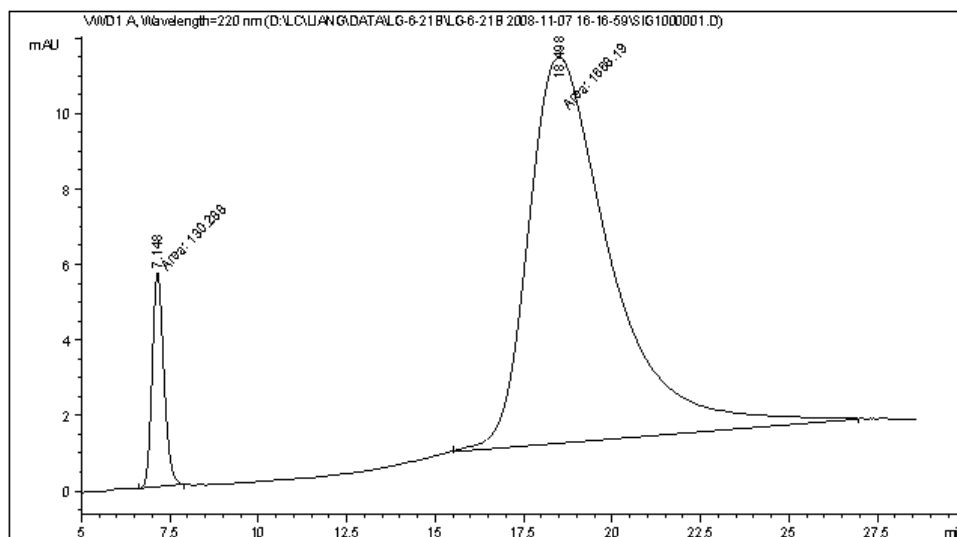
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.606	MM	0.3248	383.63168	19.68369	51.1376
2	15.249	MM	1.4963	366.56360	4.08305	48.8624

Totals : 750.19528 23.76674

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

Data File D:\LC\LIANG\DATA\LG-6-21B\LG-6-21B 2008-11-07 16-16-59\SIG1000001.D
Sample Name: lg-6-21b

```
=====
Acq. Operator   : liang gang                      Seq. Line :    1
Acq. Instrument : Instrument 1                     Location  : Vial 11
Injection Date  : 11/7/2008 4:18:22 PM             Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\liang\data\LG-6-21B\LG-6-21B 2008-11-07 16-16-59\ASH-50-50-220NM-15ML-30MIN.M
Last changed    : 11/7/2008 4:13:27 PM by liang gang
Analysis Method : D:\LC\LIANG\DATA\LG-6-21B\LG-6-21B 2008-11-07 16-16-59\SIG1000001.D\DA.M (ASH-50-50-220NM-15ML-30MIN.M)
Last changed    : 11/10/2008 8:43:57 PM by liang gang
                  (modified after loading)
Method Info     : ASH-50-50-220NM-15ML-30MIN
=====
```



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

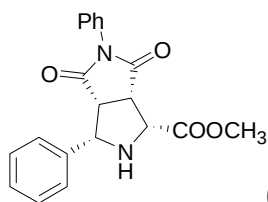
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: WVD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.148	MM	0.3837	130.28781	5.65971	7.2444
2	18.498	MM	2.7171	1668.18591	10.23281	92.7556

Totals : 1798.47372 15.89252

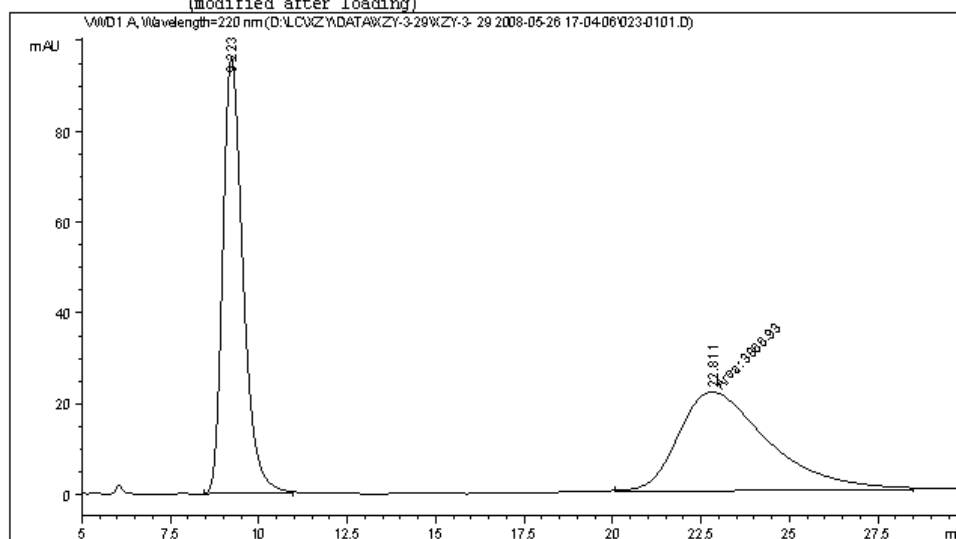
=====
*** End of Report ***



(4y)

Data File D:\LC\XYZ\DATA\XYZ-3-29\XYZ-3- 29 2008-05-26 17-04-06\023-0101.D
Sample Name: xzy-3-29c

```
=====
Acq. Operator   : xzy                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 23
Injection Date  : 5/26/2008 5:05:34 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\XYZ\DATA\XYZ-3-29\XYZ-3- 29 2008-05-26 17-04-06\ASH-50-50-15ML-220NM.M
Last changed    : 5/13/2008 10:33:13 AM by xzy
Analysis Method : D:\LC\XYZ\DATA\XYZ-3-29\XYZ-3- 29 2008-05-26 17-04-06\023-0101.D\DA.M (ASH-50-
50-15ML-220NM.M)
Last changed    : 11/10/2008 8:35:30 PM by liang gang
                  (modified after loading)
=====
```



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=220 nm

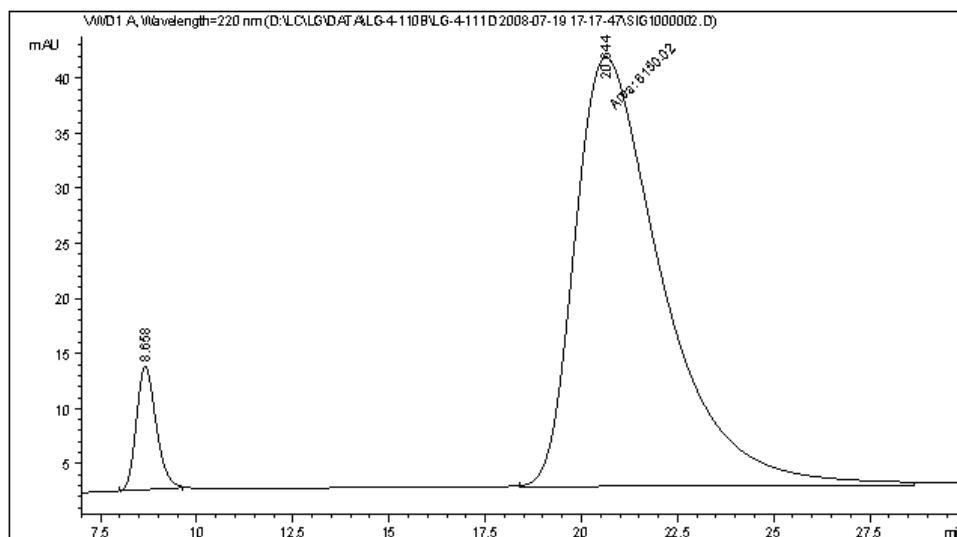
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.223	BB	0.6069	3800.09961	95.81161	49.5642
2	22.811	MM	2.9636	3866.92847	21.74714	50.4358

Totals : 7667.02808 117.55875

*** End of Report ***

Data File D:\LC\LG\DATA\LG-4-110B\LG-4-111D 2008-07-19 17-17-47\SIG1000002.D
Sample Name: lg-4-110b

```
=====
Acq. Operator   : liang gang                      Seq. Line :    2
Acq. Instrument : Instrument 1                    Location  : Vial 67
Injection Date  : 7/19/2008 5:30:53 PM           Inj       :    1
                                                Inj Volume: 5 µl
Acq. Method     : D:\LC\LG\DATA\LG-4-110B\LG-4-111D 2008-07-19 17-17-47\ASH-50-50-220NM-15ML-
50MIN.M
Last changed    : 7/19/2008 5:15:09 PM by xzy
Analysis Method : D:\LC\LG\DATA\LG-4-110B\LG-4-111D 2008-07-19 17-17-47\SIG1000002.D\DA.M (ASH-
50-50-220NM-15ML-50MIN.M)
Last changed    : 11/8/2008 6:33:50 PM by liang gang
(modified after loading)
Method Info     : ASH-50-50-220NM-1.5ML-50MIN
=====
```



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

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

Signal 1: WVD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.658	BB	0.5443	398.50516	11.17030	6.0854
2	20.644	MM	2.6307	6150.01758	38.96358	93.9146

Totals : 6548.52274 50.13388

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