(i) Supporting Information for the manuscript :

Title: "Syntheses and reactivities of non-symmetrical "active ester" bi-dentate cross-linking reagents having a phthalimidoyl and acid chloride, 2-benzothiazole, or 1-benzotriazole group"

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1. Experimental Section

1.1. General

All the melting points were uncorrected using a micro melting point apparatus. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$ using TMS as an internal standard. All the reactions were monitored with TLC and the products were separated by column chromatography using Silica Gel 60 and by preparative layer chromatography using Silica Gel 60 PF_{254} with UV or PMA and DNP detection. Mass spectra were obtained on a JEOL-JMS-D300 mass spectrometer. The elemental analyses were performed at Micro Analytical Laboratory of the Department of Material Systems Engineering and Life Science, University of Toyama. All the reagents were the highest quality and were further purified by distillation, or re-crystallization.

1.2. Preparation of Phthalimido 4-chloroformylbutanoate (9)

Method (A): Glutaryl dichloride **17** (1457.4 mg, 8.62 mmol) was added to a stirred solution of *N*-hydroxyphthalimide (469.8 mg, 2.87 mmol) and pyridine (749.0 μL, 2.87 mmol) in CH_2Cl_2 (7 mL) at rt under N_2, and the reaction mixture was stirred for 3 h. Then, hexane was added to the reaction mixture and the precipitate was removed by glass funnel. The solution was concentrated by evaporation and purification by kugelrohr distillation and finally repeated re-crystallization from AcOEt/hexane to yield acid chloride **9** (373.8 mg, 44%) as a colorless solid; *Method (B)*: Thionyl chloride (316.1 μL, 4.33 mmol) was added to a stirred solution of **10** (1.00 g, 3.60 mmol) in $ClCH_2CH_2Cl$ (13 mL) under N_2 and the reaction mixture was refluxed with stirring for 3 h. Then, the solvent was removed under vacuum. The residue was purified by repeated re-crystallization from AcOEt/hexane to yield acid chloride **9** (979.0 mg, 92%) as a colorless solid; *Method (C)*: Compound **10** (50.0 mg, 0.18 mmol) was added to a stirred solution of dichloromethyl methyl ether (47.8 μL, 0.54 mmol) in CH_2Cl_2 (1 mL) and C_6H_6 (1 mL) at reflux under N_2, and stirred for 5 h. Then, the solvent was removed under vacuum. The residue was purified by repeated re-crystallization from AcOEt/hexane to yield acid chloride **9** (46.8 mg, 88%) as a colorless solid; *Method (D)*: Compound **10** (115.0 mg, 0.42 mmol) was added to a stirred solution of trichloroacetic acid ethyl ester (114.3 μL, 0.83 mmol) in the presence of Ph_3P

(217.7 mg, 0.83 mmol) in $ClCH_2CH_2Cl$ (3 mL) at rt under N_2, and the reaction mixture was stirred for 5 h. Then, the solvent was removed under vacuum. The residue was purified by repeated re-crystallization from AcOEt/hexane to yield acid chloride **9** (111.7 mg, 90%) as a colorless solid; mp 88-90 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.13-2.21 (m, 2H), 2.80 (t, J = 7.9 Hz, 2H), 3.13 (t, J = 7.2 Hz, 2H), 7.79-7.83 (m, 2H), 7.88-7.92 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.9, 29.2, 45.2, 123.9, 124.0, 128.7, 134.7, 134.8, 161.7, 168.5, 173.1; IR (KBr) 1805, 1791, 1741 cm^{-1}. Anal. Calcd for $C_{13}H_{10}ClNO_5$: C, 52.81; H, 3.41; N, 4.74. Found: C, 53.02; H, 3.56; N, 4.78.

1.3. Preparation of Phthalimido 4-carboxybutanoate (10)

To a stirred solution of *N*-hydroxyphthalimide (1.00 g, 6.13 mmol) and 4-DMAP (898.6 mg, 7.36 mmol) in CH_2Cl_2 (8 mL) was added to a stirred solution of glutaric anhydride (768.7 mg, 6.74 mmol) in CH_2Cl_2 (4 mL) and the reaction mixture was stirred for 4 h under N_2 at 0 °C. Then, the reaction mixture was neutralized by 1N HCl solution and extracted with CH_2Cl_2 and H_2O, washed with 1N HCl (4×), dried over anhydrous $MgSO_4$, and concentrated under vacuum, to give **10** (1478 mg, 87%) as a colorless solid; mp 119-119.8 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.09-2.16 (m, 2H), 2.58 (t, J = 7.2 Hz, 2H), 2.80 (t, J = 7.2 Hz, 2H), 7.77-7.82 (m, 2H), 7.87-7.91 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.5, 29.9, 32.3, 124.0, 124.0, 128.8, 134.7, 161.8, 168.9, 177.8; IR (KBr) 3131, 1787, 1741, 1704 cm^{-1}. Anal. Calcd for $C_{13}H_{11}NO_6$: C, 56.32; H, 4.00; N, 5.05. Found: C, 55.89; H, 4.02; N, 5.18.

1.4. Preparation of N-(3-Phenylpropionyloxy)benzotriazole (11)

DCC (892.9 mg, 4.32 mmol) was added to a stirred solution of hydrocinnamic acid (500.0 mg, 3.32 mmol) and 1-hydroxybenzotriazole (584.8 mg, 4.32 mmol) in CH_2Cl_2 (8 mL) at 0 ˚C under N_2 and stirred for 3 h. Then, the precipitate was filtered and washed with CH_2Cl_2. Purification by TLC on silica gel (AcOEt/hexane; 1:1) gave the title compound **11** (851.8 mg, 96%) as a colorless solid; mp 92-93 °C (from AcOEt-hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ3.17 (t, J = 7.6 Hz, 2H), 3.48 (t, J = 7.6 Hz, 2H), 7.22-7.31 (m, 5H), 7.54-7.58 (m, 1H), 7.75-7.79 (m, 1H), 8.00 (d, J = 8.4 Hz, 1H), 8.40 (d, J = 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ29.9, 36.5, 115.5, 116.0, 126.5, 126.7, 128.3, 128.6, 132.5, 133.0, 139.4, 169.1; IR (KBr) 1736 cm^{-1}. Anal. Calcd for $C_{15}H_{13}N_3O_2$: C, 67.40; H, 4.90; N, 15.72. Found: C, 67.68; H, 5.01; N, 15.80.

1.5. Preparation of. *N*-(3-Phenylpropionyloxy)phthalimide (12)

DCC (1786.8 mg, 8.66 mmol) was added to a stirred solution of hydrocinnamic acid (1.00 g, 6.66 mmol) and *N*-hydroxyphthalimide (1412.2 mg, 8.66 mmol) in CH_2Cl_2 (15 mL) at 0 ˚C under N_2 and stirred for 3 h. Then, the precipitate was filtered and washed with CH_2Cl_2. Purification by TLC on silica gel (AcOEt/hexane; 1:1) gave the title compound **12** (1.81 g, 92%) as a colorless solid; mp 84-85 °C (from CH_2Cl_2-hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ2.96-3.00 (m, 2H), 3.11 (t, J = 7.2 Hz, 2H), 7.23-7.27 (m, 3H), 7.31-7.35 (m, 2H), 7.77-7.81 (m, 2H), 7.86-7.91 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ30.5, 32.7, 123.9, 126.7, 128.3, 128.7, 128.9, 134.7, 139.1, 161.9, 168.8; IR (KBr) 1789, 1740 cm^{-1}. Anal. Calcd for $C_{17}H_{13}NO_4$: C, 69.15; H, 4.44; N, 4.74. Found: C, 69.14; H, 4.52; N, 4.69.

1.6. Preparation of 3-Phenylpropionyloxybenzothiazole (13)

DCC (44.3 mg, 0.21 mmol) was added to a stirred solution of hydrocinnamic acid (25.0 mg, 0.16 mmol) and 2-hydroxybenzothiazole (32.0 mg, 0.21 mmol) in CH_2Cl_2 (1 mL) at rt under N_2 and stirred for 3 h. Then, the precipitate was filtered and washed with CH_2Cl_2. Purification by TLC on

silica gel (CH_2Cl_2) gave the title compound **8**[l] (29.0 mg, 64%) as a colorless solid; mp 83-84 ˚C (from CH_2Cl_2-hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ3.09 (t, J = 7.4 Hz, 2H), 3.45 (t, J = 7.6 Hz, 2H), 7.18-7.37 (m, 7H), 8.25-8.27 (dd, J = 8.2, 8.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ30.4, 40.6, 117.7, 121.8, 125.4, 126.3, 126.9, 128.5, 128.5, 134.6, 140.2, 170.9, 173.3; IR (KBr) 1732 cm^{-1}. Anal. Calcd for $C_{16}H_{13}NO_2S$: C, 67.82; H, 4.62; N, 4.94. Found: C, 67.89; H, 4.75; N, 4.98.

1.7. Preparation of *N*-(3-Phenylpropionyl)benzotriazole (14)

Method A: A mixture of hydrocinnamic acid (53.0 mg, 0.35 mmol) and 1-(methanesulfonyl)benzotriazole (70 mg, 0.35 mmol) and Et_3N (50 mg, 0.48 mmol) were refluxed in THF (2 mL) under N_2 for overnight. The solvent was evaporated and the residue was dissolved in $CHCl_3$. The organic layer was washed with water, dried over anhydrous $MgSO_4$, and evaporated to give a solid crude product, which was purified by TLC on silica gel (AcOEt/hexane; 1:1) gave the title compound **14** (74.7 mg, 85%) as a colorless solid; *Method B*: Benzotriazole (158.4 mg, 1.33 mmol) was added to a stirred solution of hydrocinnamic acid (200.0 mg, 1.33 mmol) and DCC (328.0 mg, 1.59 mmol) in CH_2Cl_2 (4 mL) at rt under N_2 and stirred for over night. The precipitate was filtered and washed with CH_2Cl_2. Purification by TLC on silica gel (AcOEt/hexane; 1:1) gave the title compound **9** (220.5 mg, 66%) as a colorless solid. mp 58-59 ˚C (from AcOEt-hexane); ^{1}H NMR (400 MHz, $CDCl_3$) δ3.23 (t, J = 7.6 Hz, 2H), 3.76 (t, J = 3.0 Hz, 2H), 7.19-7.32 (m, 5H), 7.47-7.51 (m, 1H), 7.62-7.66 (m, 1H), 8.11-8.13 (m, 1H), 8.28-8.31 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ30.1, 37.0, 114.3, 120.1, 126.1, 126.4, 128.4, 128.6, 130.3, 131.0, 139.7, 146.1, 171.5; IR (KBr) 1755 cm^{-1}.

1.8. Modified procedure for the synthesis of Phthalimido 4-(2-benzothiazolyloxycarbonyl)butanoate (15)

DCC (96.5 mg, 0.46 mmol) was added to a stirred solution of **10** (100.0 mg, 0.36 mmol) and 2-hydroxybenzothiazole (54.4 mg, 0.36 mmol) in CH_2Cl_2 (2 mL) and the reaction mixture was stirred for 3 h under N_2 at 0 °C. Then, the reaction mixture was neutralized by dil. AcOH solution and extracted with CH_2Cl_2. The organic layer was washed with water, dried over anhydrous $MgSO_4$, and concentrated under vacuum. The residue was purified after repeated recrystalization to yield **15** (115.1 mg, 78%) as a colorless solid; mp 150-151 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.23-2.30 (m, 2H), 2.86 (t, J = 7.4 Hz, 2H), 3.32 (t, J = 7.0 Hz, 2H), 7.25-7.28 (m, 1H), 7.32-7.39 (m, 2H), 7.78-7.81 (m, 2H), 7.87-7.90 (m, 2H), 8.33 (d, J = 8.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.3, 29.9, 37.5, 117.8, 121.8, 121.9, 123.9,125.5, 127.0, 128.8, 134.7, 161.8, 169.0, 172.9; IR (KBr) 1787, 1739, 1714 cm^{-1}. Anal. Calcd for $C_{20}H_{14}N_2O_6S$: C, 58.53; H, 3.44; N, 6.83. Found: C, 57.94; H, 3.52; N, 6.81.

1.9. Preparation of Phthalimido 4-(1-benzotriazolylcarbonyl)butanoate (16)

Method (A): To a stirred solution of **26** (156.2 mg, 0.79 mmol) in CH_2Cl_2 (4 mL) was added to a solution of **10** (200.0 mg, 0.72 mmol) in CH_2Cl_2 (4 mL) in the presence of pyridine (75.5 µL, 0.93 mmol) at rt under N_2, and stirred for 9 h. Then, the solvent was removed under vacuum. The residue was purified by repeated re-crystallization from CH_2Cl_2/hexane to yield **16** (145.6 mg, 55%) as a colorless solid; *Method (B)*: Compound **10** (1.00 g, 3.82 mmol) was dissolved in CH_2Cl_2 (8 mL) and this solution was added to a solution of benzotriazole (456.4 mg, 3.82 mmol) and DCC (948.1 mg, 4.59 mmol) in CH_2Cl_2 (17 mL) and stirred for 1 h under N_2 at 0 °C. The precipitate was filtered and washed with CH_2Cl_2. Then, the removal of solvent afforded crude

product, which were purified by flash chromatography (AcOEt/hexane; 1:1) to yield **16** (1215.0 mg, 84%) as a colorless solid; mp 155.5-157 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.37-2.44 (m, 2H), 2.95 (t, J = 7.2 Hz, 2H), 3.65 (t, J = 7.2 Hz, 2H), 7.50-7.54 (m, 1H), 7.65-7.69 (m, 1H), 7.78-7.82 (m, 2H), 7.87-7.90 (m, 2H), 8.12-8.15 (m, 1H), 8.29-8.31(m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.1, 30.0, 34.1, 114.3, 120.2, 124.0, 126.2, 128.8, 130.5, 134.7, 146.1, 161.8, 168.8, 171.3; IR (KBr) 1812, 1785, 1752 cm^{-1}. Anal. Calcd for $C_{19}H_{14}N_4O_5$: C, 60.32; H, 3.73; N, 14.81. Found: C, 60.46; H, 3.73; N, 14.85. HRMS (EI) calcd for $C_{19}H_{14}N_4O_5$: 378.0964; found: m/z 378.0949.
X-ray crystal data; Empirical formula: $C_{19}H_{14}N_4O_4$; Formula weight 362.34; Crystal system = triclinic; Space group P$\bar{1}$ (#2); Lattice parameters: a = 8.781(2) Å; b = 14.847(2) Å, c = 7.735(2) Å; α = 90.96(2)˚, β = 114.70(2)˚, γ = 74.50(1)˚; V = 877.7(3) Å^3; T = 23.0 ˚C; Z = 2; m (M_oKa) = 0.99 cm^{-1}; 5427 reflections measured, 2839 unique (R_{int} = 0.048); final R value 0.066. Crystallographic data has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 751600

1.10. Bis(1,3-diphthalimidyl glutarate) (18)

Colorless solid; mp 216.5-217.5 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.24-2.31 (m, 2H), 2.89 (t, J = 7.4 Hz, 2H), 7.78-7.82 (m, 2H), 7.87-7.92 (m, 2H); ^{13}C NMR ($CDCl_3$,100 MHz) δ19.7, 29.7, 124.0, 128.9, 134.8, 161.8, 168.7; IR (KBr) 1816, 1787, 1741 cm^{-1}. Anal. Calcd for $C_{21}H_{14}N_2O_8$: C, 59.72; H, 3.34; N, 6.63. Found: C, 59.77; H, 3.58; N, 6.66.

1.11. General procedure for the reaction of model "active ester" compound 11, 12, 13, and 14 with several nucleophiles (product 19 as an example)

Typical procedure: To a stirred solution of 4-DMAP (86.1 mg, 0.70 mmol) was added to a stirred solution of 4-methylbenzyl alcohol (86.1 mg, 0.70 mmol) and **13** (200.0 mg, 0.70 mmol) in CH_2Cl_2 (4 mL) under N_2 at rt and stirred for 2 h. Then, the reaction mixture was neutralized by dil. AcOH solution and extracted with CH_2Cl_2. The organic layer was separated, successively washed with water and brine, and dried over anhydrous $MgSO_4$. Removal of solvent in vacuum, to give oil crude product, which was purified by flash chromatography yielded 3-phenylpropionic acid 4-methylbenzyl ester **19** (159.7 mg, 89%) as a colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.31 (s, 3H), 2.61-2.65 (m, 2H), 2.93 (t, J = 7.8 Hz, 2H), 5.04 (s, 2H), 7.11-7.26 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ21.0, 30.8, 35.7, 66.0, 126.1, 128.1, 128.2, 128.3, 129.0, 132.8, 137.8, 140.3, 172.5; IR (neat) 1735 cm^{-1}. HRMS (EI) calcd for $C_{17}H_{18}O_2$: 254.1307; found: m/z 254.1304.

1.11.1. 3-Phenylpropionic acid 4-chlorobenzyl ester (20)

Colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.66 (t, J = 7.6 Hz, 2H), 2.94 (t, J = 7.8 Hz, 2H), 5.03 (s, 2H), 7.15-7.29 (m, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ30.8, 35.6, 65.2, 126.2, 128.1, 128.4, 128.5, 129.4, 133.9, 134.3, 140.1, 172.4; IR (neat) 1736 cm^{-1}. HRMS (EI) calcd for $C_{16}H_{15}ClO_2$: 274.0761; found: m/z 274.0741.

1.11.2. 3-Phenyl propionic acid 2 phenylethyl ester (21)

Colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.49-2.54 (m, 2H), 2.79-2.84 (m, 4H), 4.17-4.21 (m, 2H), 7.06-7.21 (m, 10H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ30.8, 35.0, 35.8, 64.8, 126.1, 126.4, 128.2, 128.4, 128.8, 137.7, 140.4, 172.7; IR (neat) 1734 cm^{-1}. HRMS (EI) calcd for $C_{17}H_{18}O_2$: 254.1307; found: m/z 254.1282.

1.11.3. *N*-Benzyl-3-phenylpropionamide (22)

Colorless solid; mp 76.5-77.0 ˚C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.51 (t, J = 7.6 Hz, 2H), 2.99 (t, J = 6.8 Hz, 2H), 4.39 (d, J = 6.0 Hz, 2H), 5.62 (s, 1H), 7.13-7.31 (m, 10H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ31.6, 38.5, 43.6, 126.2, 127.4, 127.7, 128.3, 128.5, 128.6, 138.1, 140.7, 171.8; IR (KBr) 3292, 1639 cm^{-1}. Anal. Calcd for $C_{16}H_{17}NO$: C, 80.30; H, 7.16; N, 5.85; found: C, 80.34; H, 7.16; N, 5.90.

1.11.4. *N*-4-Chlorobenzyl-3-phenylpropionamide (23)

Colorless solid; mp 114.5-115.5 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.48 (t, J = 7.8 Hz, 2H), 2.93 (t, J = 7.8 Hz, 2H), 4.28 (s, 2H), 6.11 (s, 1H), 6.96-6.99 (m, 2H); 7.00-7.27 (m, 7H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ31.5, 38.1, 42.5, 126.1, 128.2, 128.4, 128.5, 128.7, 132.9, 136.6, 140.5, 172.0; IR (KBr) 3282, 1636 cm^{-1}. HRMS (EI) calcd for $C_{17}H_{16}ClNO$: 273.0920; found: m/z 273.0917.

1.11.5. 3-Phenylthiopropionic S-benzyl ester (24)

Colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.85-2.89 (m, 2H), 2.99 (t, J = 7.6 Hz, 2H), 4.12 (s, 2H), 7.15-7.31 (m, 10H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ31.3, 33.1, 45.2, 126.3, 127.2, 128.2, 128.5, 128.6, 128.7, 137.5, 139.9, 197.8; IR (neat) 1686 cm^{-1}. HRMS (EI) calcd for $C_{16}H_{16}OS$: 256.0922; found: m/z 256.0920.

1.12. *N*-(1-Methanesulphonyl)benzotriazole (26)

To a ice-cold solution of benzotriazole (1.00 g, 8.4 mmol) and pyridine (1215.1 μL, 15.1 mmol) in dry toluene (5 mL) was added dropwise methanesulfonyl chloride (977.4 μL, 12.6 mmol) in toluene (10 mL) and the reaction mixture was stirred at rt under N_2 for 12 h. Then, AcOEt and H_2O were added and organic layer was separated, successively washed with H_2O and brine, and dried over anhydrous $MgSO_4$. Removal of solvents in vacuum, to give a solid crude product which was separated by column (Hexane/AcOEt; 1:1) to give **26** (1538 mg, 93%) as a colorless solid; mp[9-10] 110-111.5 °C (from CH_2Cl_2/hexane).

1.13. General procedure for the reactions of linker 9, 15, or 16 with various nucleophiles in the present of 4-DMAP (product 27 as an example)

Typical procedure: To a stirred solution of 4-DMAP (29.3 mg, 0.24 mmol) and benzyl alcohol (25.3 μL, 0.24 mmol) in CH_2Cl_2 (1 mL) dropwise added to a stirred solution of **15** (100.0 mg, 0.24 mmol) in CH_2Cl_2 (1 mL) at rt under N_2, and stirred for 1 h. Then, the reaction mixture was neutralized by $NaHCO_3$ solution and extracted with CH_2Cl_2, and dried over anhydrous $MgSO_4$, and concentrated under vacuum, to give crude product which was purified by flash chromatography yielded, phthalimido 4-benzyloxycarbonylbutanoate **27** (69.6 mg, 79%) as a colourless solid; mp 33.5-35 ˚C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.09-2.16 (m, 2H), 2.55 (t, J = 7.2 Hz, 2H), 2.76 (t, J = 7.2 Hz, 2H), 5.15 (s, 2H), 7.30-7.38 (m, 5H), 7.76-7.80 (m, 2H), 7.85-7.90 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.8, 29.9, 32.6, 66.3, 123.9, 128.1, 128.2, 128.5, 128.8, 134.7, 135.7, 161.8, 168.9, 172.3; IR (KBr) 1814, 1787, 1743 cm^{-1}. Anal. Calcd for $C_{20}H_{17}NO_6$: C, 65.39; H, 4.66; N, 3.81. Found: C, 65.71; H, 4.75; N, 3.72.

1.13.1. Phthalimido 4-(4-methylbenzyloxycarbonyl)butanoate (28)

Colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ1.54-1.61 (m, 3H), 2.08-2.13 (m, 2H), 2.50-2.54 (m, 2H), 2.69-2.75 (m, 2H), 5.88-5.91 (m, 1H), 7.25-7.37 (m, 5H), 7.77-7.80 (m, 2H), 7.87-7.90 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.8, 22.2, 29.9, 32.9, 72.6, 123.9, 126.0, 126.1, 127.9, 128.5, 128.6, 128.8, 134.7, 141.5, 161.8, 169.0, 171.7; IR (neat) 1814, 1788, 1745 cm^{-1}. HRMS (EI) calcd for $C_{21}H_{19}NO_6$: 381.1212; found: m/z 381.1198.

1.13.2. Phthalimido 4-(1-methylbenzyloxycarbonyl)butanoate (29)

Colorless solid; mp 48.5-50.5 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.98-2.05 (m, 2H), 2.25 (s, 3H), 2.44 (t, *J* = 7.2 Hz, 2H), 2.66 (t, *J* = 7.2 Hz, 2H), 7.05-7.08 (m, 2H), 7.12-7.17 (m, 2H), 7.67-7.70 (m, 2H), 7.75-7.79 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.7, 21.0, 29.9, 32.6, 66.2, 123.8, 128.3, 128.7, 129.0, 129.1, 132.7, 134.6, 138.0, 161.7, 168.9, 172.2; IR (KBr) 1817, 1787, 1738 cm^{-1}. HRMS (EI) calcd for $C_{21}H_{19}NO_6$: 381.1212; found: m/z 381.1220.

1.13.3. Phthalimido 4-(4-chlorobenzyloxycarbonyl)butanoate (30)

Colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ1.99-2.06 (m, 2H), 2.46 (t, *J* = 7.4 Hz, 2H), 2.67 (t, *J* = 8.0 Hz, 2H), 5.01 (s, 2H), 7.18-7.24 (m, 5H), 7.67-7.71 (m, 2H), 7.75-7.79 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.7, 29.8, 32.4, 65.4, 123.8, 128.4, 128.6, 129.5, 134.0, 134.2, 134.7, 161.7, 168.8, 172.1; IR (neat) 1813, 1788, 1744 cm^{-1}. HRMS (EI) calcd for $C_{20}H_{16}ClNO_6$: 401.0666; found: m/z 401.0668.

1.13.4. Phthalimido 4-phenylethyloxycarbonylbutanoate (31)

Colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.03-2.12 (m, 2H), 2.45-2.49 (m, 2H), 2.68-2.72 (m, 2H), 2.95 (t, *J* = 7.2 Hz, 2H), 4.30-4.34 (m, 2H), 7.21-7.31 (m, 5H), 7.77-7.80 (m, 2H), 7.87-7.89 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.7, 29.9, 32.6, 35.0, 123.9, 126.5, 128.4, 128.7, 128.8, 134.7, 137.6, 161.8, 168.9, 172.3; IR (neat) 1814, 1788, 1745 cm^{-1}. HRMS (EI) calcd for $C_{21}H_{19}NO_6$: 381.1212; found: m/z 381.1194.

1.13.5. Phthalimido 4-phenyloxycarbonylbutanoate (32)

Colorless solid; mp 105-106 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.19-2.26 (m, 2H), 2.78 (t, *J* = 7.2 Hz, 2H), 2.86 (t, *J* = 7.2 Hz, 2H), 7.08-7.12 (m, 2H), 7.21-7.26 (m, 2H), 7.21-7.26 (m, 1H), 7.36-7.41 (m, 2H), 7.77-7.82 (m, 1H), 7.87-7.91 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ19.7, 29.9, 32.6, 121.5, 124.0, 125.8, 128.8, 129.4, 134.8, 150.5, 161.8, 168.9, 171.0; IR (KBr) 1808, 1781, 1751 cm^{-1}. Anal. Calcd for $C_{19}H_{15}NO_6$: C, 64.59; H, 4.28; N, 3.96. Found: C, 64.34; H, 4.33; N, 4.04. HRMS (EI) calcd for $C_{19}H_{15}NO_6$: 353.0899; found: m/z 353.0868.

1.13.6. Phthalimido 4-(S-benzyloxycarbonyl)butanoate (33)

Colorless solid; mp 49-50 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.11-2.19 (m, 2H), 2.73-2.78 (m, 4H), 4.14 (s, 2H), 7.21-7.31 (m, 5H), 7.77-7.81 (m, 2H), 7.86-7.90 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 20.3 , 29.9, 33.2, 41.9, 123.9, 127.3, 128.6, 128.7, 128.8, 134.7, 137.3, 161.8, 168.8, 197.5; IR (KBr) 1810, 1745, 1683 cm^{-1}. Anal. Calcd for $C_{20}H_{17}NO_5S$: C, 62.65; H, 4.47; N, 3.65. Found: C, 63.04; H, 4.54; N, 3.59.

1.13.7. Phthalimido 4-(*N*-benzyloxycarbonyl)butanoate (34)

Colorless solid; mp 149.0-149.5 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.14-2.21 (m, 2H), 2.40 (t, *J* = 7.2 Hz, 2H), 2.74 (t, *J* = 6.8 Hz, 2H), 4.46 (d, *J* = 5.6 Hz, 2H), 6.27 (s, 1H), 7.21-7.32 (m, 5H), 7.78-7.82 (m, 2H), 7.85-7.89 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ20.9, 29.9, 34.4, 43.6, 124.0, 127.4, 127.8, 128.6, 128.7, 134.8, 138.1, 162.0, 169.3, 171.5; IR (KBr) 3316, 1808, 1787, 1747 cm^{-1}. Anal. Calcd for $C_{20}H_{18}N_2O_5$: C, 65.57; H, 4.95; N, 7.65. Found: C, 65.13; H, 4.88; N, 7.52.

1.13.8. Phthalimido 4-(*N*-4-cholorobenzyloxycarbonyl)butanoate (35)

Colorless solid; mp 144.5-147 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.12-2.22 (m, 2H), 2.40 (t, *J* = 7.0 Hz, 2H), 2.74 (t, *J* = 6.6 Hz, 2H), 4.43 (d, *J* = 5.6 Hz, 2H), 6.33 (s, 1H), 7.21-7.27 (m, 4H), 7.80-7.83 (m, 2H), 7.86-7.89 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ20.9, 29.8, 34.3, 42.9, 123.5, 124.0, 128.7, 129.2, 134.3, 134.9, 171.6, 147.9; IR (KBr) 3293, 1807, 1783, 1743 cm^{-1}. HRMS (EI) calcd for $C_{20}H_{17}ClN_2O_5$: 400.0826; found: m/z 400.0868.

1.13.9. Phthalimido 4-(*N*-phenylethyloxycarbonyl)butanoate (36)

Colorless solid; mp 100.5-102 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ1.99-2.06 (m, 2H), 2.22 (t, *J* = 7.0 Hz, 2H), 2.60 (t, *J* = 6.8 Hz, 2H), 2.75 (t, *J* = 7.0 Hz, 2H), 3.48 (q, *J* = 12.8 Hz, 2H), 6.00 (s, 1H), 7.03-7.19 (m, 5H), 7.70-7.77 (m, 2H), 7.78-7.81 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ20.8, 29.8, 34.4, 35.4, 40.4, 123.9, 126.3, 128.4, 128.6, 128.7, 133.9, 134.7, 134.8, 138.6, 161.9, 169.2, 171.6; IR (KBr) 3312, 1816, 1791, 1750 cm^{-1}. HRMS (EI) calcd for $C_{21}H_{20}N_2O_5$: 380.1372; found: m/z 380.1414.

1.13.10. Phthalimido 4-(N-phenylalaninocarbonyl methyl ester)butanoate (37)

Colorless solid; mp 94.5-95.5 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ2.04-2.12 (m, 2H), 2.33-2.37 (m, 2H), 2.59-2.75 (m, 2H), 3.06 (dd, *J* = 6.8 Hz, 14.0 Hz, 1H), 3.19 (dd, *J* = 5.6 Hz, 14.0 Hz, 1H), 3.73 (s, 3H), 4.93 (q, *J* = 13.6 Hz, 1H), 6.21 (d, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 7.2 Hz, 2H), 7.18-7.29 (m, 3H), 7.80-7.82 (m, 2H), 7.87-7.91 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ20.7, 29.8, 34.2, 37.8, 52.3, 53.0, 124.0, 127.0, 128.5, 128.8, 129.1, 134.8, 135.8, 161.9, 169.2, 171.2, 172.0; IR (KBr) 3293, 1785, 1745, 1639 cm^{-1}. Anal. Calcd for $C_{23}H_{22}N_2O_7$: C, 63.01; H, 5.06; N, 6.39. Found: C, 63.02; H, 5.10; N, 6.40.

1.13.11. Phthalimido 4-(N-diphenylalaninocarbonyl benzyl ester)butanoate (38)

Colorless solid; mp 134-136 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ1.99-2.05 (m, 2H), 2.29 (t, *J* = 8.0 Hz, 2H), 2.57-2.64 (m, 2H), 2.97-3.10 (m, 4H), 4.64-4.70 (m, 1H), 4.78-4.85 (m, 1H), 5.10 (s, 2H), 6.34 (d, *J* = 8.0 Hz, 1H), 6.40 (d, *J* = 8.0 Hz, 1H), 6.93-6.96 (m, 2H), 7.16-7.37 (m, 13H), 7.80-7.83 (m, 2H), 7.83-7.91 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ20.6, 29.6, 29.8, 34.2, 37.8, 53.3, 54.2, 67.2, 124.0, 127.0, 127.0, 128.5, 128.5, 128.6, 128.6, 128.8, 129.2, 129.2, 134.8, 135.0, 135.5, 136.4, 161.9, 169.2, 170.4, 170.7, 171.6; IR (KBr) 3289, 1813, 1788, 1747 cm^{-1}. HRMS (EI) calcd for $C_{38}H_{35}N_3O_8$: 661.2424; found: m/z 661.2433.

1.14. Typical procedure for the preparation of 3β-Cholesteryl 4-(phthalimidoyloxycarbonyl)butyrate (39)

To a stirred solution of 4-DMAP (59.8 mg, 0.48 mmol) and cholesterol (188.3 mg, 0.48 mmol) in CH_2Cl_2 (2 mL) was added dropwise to a stirred solution of **15** (200 mg, 0.48 mmol) in CH_2Cl_2 (2 ml) at rt under N_2 and stirred for 19 h. Then, the reaction mixture was neutralized by $NaHCO_3$ solution and extracted with CH_2Cl_2, and dried over anhydrous $MgSO_4$, and concentrated under vacuum, to give crude product which was purified by flash chromatography yielded **39** (248.4 mg, 79%) as a colorless solid; mp 83-85 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ0.67 (s, 3H), 0.86 (dd, *J* = 1.6 Hz, 6.4 Hz, 6H), 0.91 (d, *J* = 6.4 Hz, 3H), 0.94-1.04 (m, 5H), 1.05-1.17 (m, 6H), 1.23-1.37 (m, 6H), 1.42-1.58 (m, 7H), 1.78-1.88 (m, 3H), 1.93-2.03 (m, 2H), 2.06-2.13 (m, 2H), 2.33 (d, *J* = 7.6 Hz, 2H), 2.47 (t, *J* = 7.2 Hz, 2H), 2.76 (t, *J* = 7.2 Hz, 2H), 4.60-4.68 (m, 1H), 5.38 (d, *J* = 4 Hz, 1H), 7.77-7.81 (m, 2H), 7.86-7.91 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ11.8, 18.7, 19.3, 19.9, 21.0, 22.5, 22.8, 23.8, 24.3, 27.8, 27.9, 28.2, 30.07, 31.8, 31.9, 33.1, 35.8, 36.2, 36.6, 36.9, 38.1, 39.5, 39.7, 42.3, 49.9, 56.1, 56.7, 74.2, 122.7, 123.9, 128.8, 134.8, 139.6, 161.8, 169.1, 171.9; IR (KBr) 1814, 1789, 1741 cm^{-1}. Anal. Calcd for $C_{40}H_{55}NO_6$: C, 74.38; H, 8.58; N, 2.17. Found: C, 74.79; H, 8.71; N, 2.12.

1.15. General procedure for the reaction of 39 with several amines (product 40 as an example)

Typical procedure: To a stirred solution of benzylamine (17 μL, 0.15 mmol) in CH_2Cl_2 (1 mL) was added dropwise to a stirred solution of **39** (100 mg, 0.15 mmol) in CH_2Cl_2 (1 mL) at rt under N_2, and stirred for 10.5 h. Then, the reaction mixture was neutralized by dil. AcOH solution and extracted with CH_2Cl_2, and dried over anhydrous $MgSO_4$, and concentrated under vacuum, to give solid crude product which was purified by flash chromatography yielded, 3β-cholesteryl 4-(benzylaminocarbonyl)butyrate **40** (74.4 mg, 82%) as a colorless solid; mp 115-117 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ0.67 (s, 3H), 0.86 (dd, *J* = 1.6 Hz, 7.2 Hz, 6H), 0.91 (d, *J* = 6.4 Hz, 3H), 0.94-1.04 (m, 5H), 1.08-1.21 (m, 6H), 1.24-1.39 (m, 6H), 1.42-1.61 (m, 7H), 1.78-1.87 (m, 3H), 1.94-2.02 (m, 4H), 2.25-2.29 (m, 4H), 2.35 (t, *J* = 7.0 Hz, 2H), 4.43 (d, *J* = 5.6 Hz, 1H), 4.55-4.63 (m, 1H), 5.36 (d, *J* = 4.4 Hz, 2H), 5.86 (s, 1H), 7.26-7.37 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ11.8, 18.6, 19.2, 20.9, 22.5, 22.8, 23.7, 24.2, 27.7, 27.9, 28.2, 31.8, 31.9, 33.6, 35.4, 35.7, 36.1, 36.5, 36.9, 38.1, 39.4, 39.6, 42.2, 43.6, 49.9, 56.0, 56.6, 74.0, 122.6, 127.5, 127.8, 128.7, 138.2, 139.5, 171.9, 172.5; IR (KBr) 3276, 1729, 1639 cm^{-1}. Anal. Calcd for $C_{39}H_{59}NO_3$: C, 79.41; H, 10.08; N, 2.37. Found: C, 78.93; H, 9.87; N, 2.35.

1.15.1. 3β-Cholesteryl 4-(4-cholorobenzylaminocarbonyl)butyrate (41)

Colorless solid; mp 122-124 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ0.67 (s, 3H), 0.86 (dd, *J* = 2.0 Hz, 6.4 Hz, 6H), 0.91 (d, *J* = 6.4 Hz, 3H), 0.94-1.0 (m, 6H), 1.05-1.16 (m, 7H), 1.20-1.35 (m, 5H), 1.41-1.58 (m, 7H), 1.80-1.90 (m, 3H), 1.95-2.02 (m, 4H), 2.27-2.37 (m, 6H), 4.41 (d, *J* = 5.6 Hz, 1H), 4.40-4.61 (m, 1H), 5.36 (d, *J* = 4.8 Hz, 1H), 6.38 (s, 1H), 7.20-7.31 (m, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ11.8, 18.7, 19.2, 20.9, 21.0, 22.5, 22.8, 23.8, 24.2, 27.7, 27.9, 28.2, 31.8, 31.9, 33.4, 35.1, 35.7, 36.1, 36.5, 36.9, 38.0, 39.5, 39.7, 42.2, 43.1, 49.9, 56.1, 56.6, 74.3, 122.7, 128.8, 129.2, 133.4, 136.3, 139.4, 172.8; IR (KBr) 3262, 1724, 1638 cm^{-1}. HRMS (EI) calcd for $C_{39}H_{58}ClNO_3$: 623.4105; found: m/z 623.4081.

1.15.2. 3β-Cholesteryl 4-(2-phenylethylaminocarbonyl)butyrate (42)

Colorless solid; mp 128-129.5 °C; (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ0.67 (s, 3H), 0.86 (dd, *J* = 1.6 Hz, 6.6 Hz, 6H), 0.91 (d, *J* = 6.4 Hz, 4H), 0.92-1.03 (m, 6H), 1.04-1.20 (m, 7H), 1.20-1.34 (m, 4H), 1.40-1.58 (m, 7H), 1.78-1.99 (m, 7H), 2.19 (t, *J* = 7.4 Hz, 2H), 2.30 (t, *J* = 7.4 Hz, 4H), 2.81 (t, *J* = 6.8 Hz, 2H), 4.55-4.65 (m, 1H), 5.36 (d, *J* = 3.6 Hz, 2H), 5.65 (s, 1H),

7.17-7.32 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 11.8, 18.6, 19.2, 20.9, 21.0, 22.5, 22.7, 23.7, 24.2, 27.7, 28.1, 31.7, 31.8, 33.5, 35.4, 35.6, 36.1, 36.5, 36.9, 38.0, 39.4, 39.6, 40.5, 42.2, 49.9, 56.0, 56.6, 73.9, 122.6, 126.4, 128.5, 128.6, 138.7, 139.5, 172.1, 172.5; IR (KBr) 3292, 1729, 1637 cm^{-1}. HRMS (EI) calcd for $C_{40}H_{61}NO_3$: 603.4651; found: m/z 603.4631.

1.15.3. 3β-Cholesteryl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (43)

Colorless solid; mp 80-82 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.60 (s, 3H), 0.78 (dd, *J* = 1.6 Hz, 6.8 Hz, 6H), 0.84 (d, *J* = 6.4 Hz, 3H), 0.87-0.97 (m, 5H), 0.98-1.09 (m, 6H), 1.10-1.27 (m, 6H), 1.36-1.51 (m, 7H), 1.73-1.95 (m, 7H), 2.16 (t, *J* = 7.0 Hz, 2H), 2.19-2.24 (m, 4H), 2.98 (dd, *J* = 6.0 Hz, 14.2 Hz, 1H), 3.10 (dd, *J* = 6.0 Hz, 13.8 Hz, 1H), 3.65 (s, 3H), 4.49-4.57 (m, 1H), 4.79-4.84 (m, 1H), 5.30 (d, *J* = 4.4 Hz, 1H), 5.90 (d, *J* = 8.0 Hz, 2H), 7.01-7.03 (m, 2H), 7.15-7.24 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 11.8, 18.6, 19.2, 20.7, 20.9, 22.5, 22.8, 24.2, 27.7, 27.9, 28.2, 31.8, 31.9, 33.5, 35.2, 36.1, 36.9, 37.8, 38.0, 39.4, 39.6, 42.2, 49.9, 52.3, 52.9, 56.0, 56.6, 73.9, 122.6, 123.4, 127.1, 128.5, 128.6, 128.8, 129.1, 134.0, 135.8, 139.5, 171.7, 172.0, 172.4; IR (KBr) 3431, 1789, 1733, 1716 cm^{-1}. HRMS (EI) Calcd for $C_{42}H_{63}NO_5$: 661.4706; found: m/z 661.4717.

1.15.4. 3β-Cholesteryl 4-(diphenylalaninocarbonyl benzyl ester)butyrate (44)

Colorless solid; mp 145.5-147 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.67 (s, 3H), 0.86 (dd, *J* = 2.0 Hz, 6.8 Hz, 6H), 0.91 (d, *J* = 6.4 Hz, 3H), 0.93-1.04 (m, 6H), 1.05-1.21 (m, 7H), 1.20-1.34 (m, 5H), 1.41-1.58 (m, 7H), 1.80-1.87 (m, 5H), 1.90-2.02 (m, 2H), 2.16-2.22 (m, 4H), 2.30 (d, *J* = 8.0 Hz, 2H), 2.99 (d, *J* = 6.8 Hz, 3H), 3.08 (dd, *J* = 14.0 Hz, 6.0 Hz, 1H), 4.55-4.65 (m, 2H), 4.77 (q, *J* = 6.4 Hz, 1H), 5.09 (s, 2H), 5.36 (d, *J* = 4.4 Hz, 1H), 6.31 (s, 1H), 6.42 (s, 1H), 6.94 (t, *J* = 3.4 Hz, 2H), 7.13-7.27 (m, 10H), 7.34-7.36 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 11.8, 18.6, 19.2, 20.7, 21.0, 22.5, 23.8, 24.2, 27.7, 27.9, 28.2, 31.8, 31.9, 33.4, 35.0, 35.7, 36.1, 36.9, 37.7, 38.0, 38.1, 39.4, 39.6, 42.2, 49.9, 53.5, 54.4, 56.1, 56.6, 67.2, 74.0, 122.7, 127.0, 127.1, 128.5, 128.5, 128.6, 129.2, 129.3, 135.0, 135.4, 136.2, 139.5, 170.5, 170.6, 172.4; IR (KBr) 3295, 1728, 1638 cm^{-1}. HRMS (FAB) Calcd for $C_{42}H_{63}NO_5$ (M+1): 885.5703; found (M+1): 885.6105.

1.16. General procedure for the reaction of 37 with various nucleophiles (product 45 as an example)

4-DMAP (55.7 mg, 0.45 mmol) was added to a stirred solution of **37** (200 mg, 0.45 mmol) and benzyl alcohol (47.1 μL, 0.45 mmol) in CH_2Cl_2 (2 mL) at rt under N_2, and stirred for 32 h. Then, the reaction mixture was neutralized by dil. AcOH solution and extracted with CH_2Cl_2, and dried over anhydrous $MgSO_4$, and concentrated under vacuum, to give oil crude product which was purified by flash column chromatography yielded, *O*-benzyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (**45**) (130.8 mg, 75%) as a colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.89-1.97 (m, 2H), 2.21 (t, *J* = 7.2 Hz, 2H), 2.34-2.40 (m, 2H), 3.05 (dd, *J* = 6.0 Hz, 14.0 Hz, 1H), 3.14 (dd, *J* = 5.6 Hz, 14.0 Hz, 1H), 3.72 (s, 3H), 4.85-4.90 (m, 1H), 5.10 (s, 2H), 5.92 (d, *J* = 7.6 Hz, 1H), 7.07-7.09 (m, 2H), 7.20-7.38 (m, 8H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 20.6, 33.1, 35.0, 37.8, 52.3, 52.9, 66.2, 127.1, 128.2, 128.2, 128.5, 128.6, 129.1, 135.7, 135.8, 171.5, 172.0, 172.8; IR (neat) 3301, 1814, 1787, 1743 cm^{-1}. HRMS (EI) Calcd for $C_{22}H_{25}NO_5$: 383.1733; found: m/z 383.1734.

1.16.1. *O*-4-Methlbenzyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (46)

Colorless solid; mp 53.5-55 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.88-1.96 (m, 2H), 2.21 (t, *J* = 7.0 Hz, 2H), 2.30-2.39 (m, 2H), 2.33 (s, 3H), 3.05 (dd, *J* = 6.0 Hz, 13.8 Hz, 1H), 3.14 (dd, *J* = 6.0 Hz, 13.8 Hz, 1H), 3.72 (s, 3H), 4.85-4.90 (m, 1H), 5.06 (s, 2H), 5.91 (d, *J* = 7.2 Hz, 1H), 7.06-7.08 (m, 2H), 7.16 (d, *J* = 7.2 Hz, 2H), 7.23-7.29 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 20.6, 21.1, 33.1, 35.1, 37.8, 52.3, 52.9, 66.1, 127.1, 128.3, 128.5, 129.1, 129.2, 132.8, 135.7, 138.1, 171.6, 172.0, 172.9; IR (KBr) 3299, 1733, 1652 cm^{-1}. HRMS (EI) Calcd for $C_{23}H_{27}NO_5$: 397.1889; found: m/z 397.1886.

1.16.2. *O*-Phenyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (47)

Colorless solid; mp 43-45 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.00-2.07 (m, 2H), 2.30-2.33 (m, 2H), 2.55-2.59 (m, 2H), 3.07 (dd, *J* = 6.0 Hz, 13.8 Hz, 1H), 3.17 (dd, *J* = 5.6 Hz, 14.0 Hz, 1H), 3.73 (s, 3H), 4.88-4.93 (m, 1H), 5.97 (d, *J* = 7.6 Hz, 1H), 7.04-7.11 (m, 4H), 7.20-7.30 (m, 4H), 7.35-7.39 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 20.5, 33.1, 34.9, 37.8, 52.3, 52.9, 121.4, 125.7, 127.1, 128.5, 129.1, 129.3, 135.7, 150.5, 171.5, 171.6, 172.0; IR (KBr) 3315, 1749, 1646 cm^{-1}. HRMS (EI) Calcd for $C_{21}H_{23}NO_5$: 369.1576; found: m/z 369.1579.

1.16.3. *N*-Benzyl 4-(L-phenylalaninocarbonyl methyl ester)butanamide (48)

Colorless solid; mp 100-101 °C (from CH_2Cl_2/hexane); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.90-1.98 (m, 2H), 2.14-2.25 (m, 4H), 2.92 (dd, *J* = 7.2 Hz, 14.0 Hz, 1H), 3.09 (dd, *J* = 5.6 Hz, 14.0 Hz, 1H), 3.72 (s, 3H), 4.33-4.45 (m, 2H), 4.83 (q, *J* = 6.9 Hz 1H), 5.92 (d, *J* = 7.6 Hz, 1H), 6.28 (s, 1H), 7.07 (d, *J* = 6.8 Hz, 2H), 7.20-7.35 (m, 8H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.7, 34.8, 34.9, 37.6, 43.4, 52.4, 52.9, 127.1, 127.5, 127.8, 128.6, 128.7, 129.0, 135.8, 138.5, 172.2, 172.3, 172.4; IR (KBr) 3289, 1747, 1643, 1546 cm^{-1}. Anal. Calcd for $C_{22}H_{26}N_2O_4$: C, 69.09; H, 6.85; N, 7.32; found: C, 68.81; H, 6.64; N, 7.23.

1.16.4. *S*-Benzyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (49)

Colorless syrup; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.92-1.99 (m, 2H), 2.18-2.22 (m, 2H), 2.50-2.62 (m, 2H), 3.05 (dd, *J* = 6.0 Hz, 13.8 Hz, 1H), 3.15 (dd, *J* = 5.6 Hz, 14.0 Hz, 1H), 3.72 (s, 3H), 4.11 (s, 2H), 4.85-4.90 (m, 1H), 5.96 (d, *J* = 8 Hz, 1H), 7.07-7.20 (m, 2H), 7.20-7.31 (m, 8H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.2, 33.1, 34.8, 37.8, 42.3, 52.3, 52.9, 127.1, 127.2, 128.5, 128.6, 128.7, 129.1, 135.7, 137.4, 171.3, 172.0, 198.2; IR (KBr) 3291, 1743, 1683, 1652 cm^{-1}. Anal. Calcd for $C_{22}H_{25}NO_4S$: C, 66.14; H, 6.31; N, 3.51. Found: C, 66.35; H, 6.10; N, 3.61. HRMS (EI) Calcd for $C_{22}H_{25}NO_4S$: 399.1504; found: m/z 399.1519.

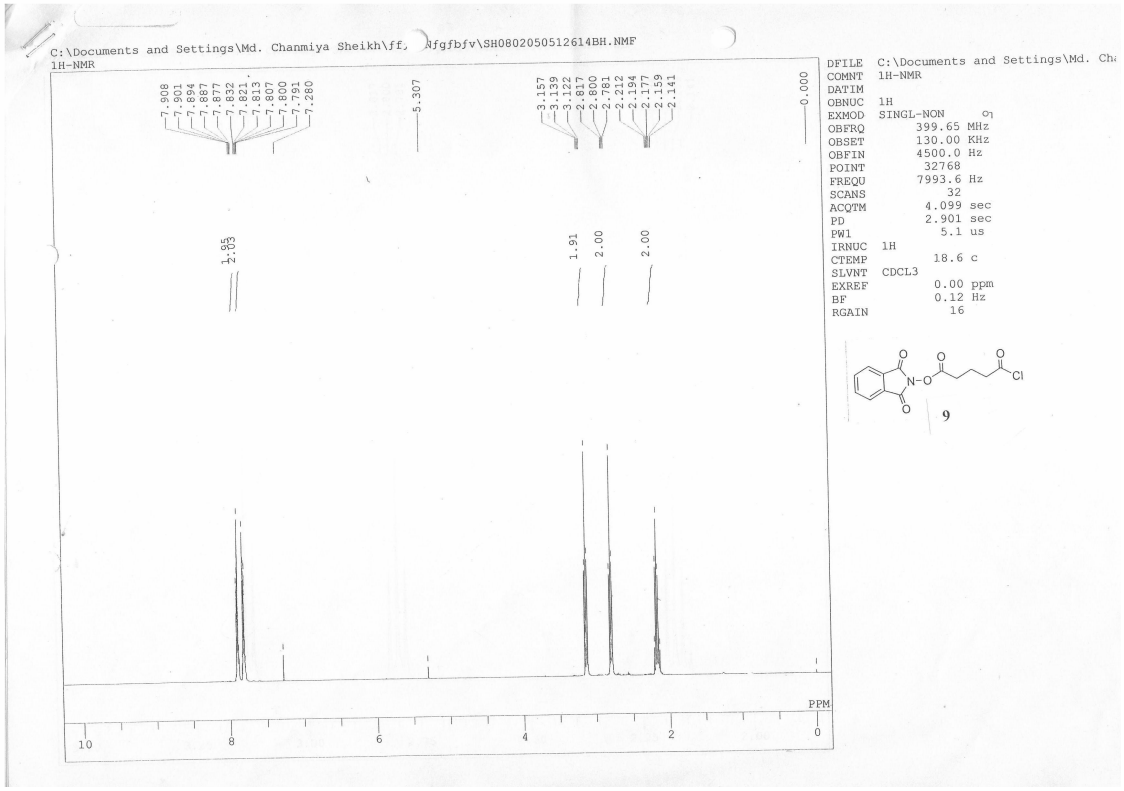
References and Notes

1. For reviews and recent examples, see: (a) G. M. Dubowchick, M. A. Walker, *Pharmacol. Ther.* 1999, **83**, 67-123; (b) V. K. Rusiecki, S. A. Warne, *Bioorg. Med. Chem. Lett*. 1993, **3**, 707-710; (c) K. D. Janda, J. A. Ashley, T. M. Jones, D. A. McLeod, D. M. Schloeder, M. I. Weinhouse, *J. Am. Chem. Soc*. 1990, **112**, 8886-8888; (d) G. A. Pietersz, *Bioconjugate Chem*. 1990, **1**, 89-95; (e) K. D. Janda, D. Schloeder, S. J. Benkovic, R. A. Lerner, *Science*, 1988, **241**, 1188-1191; (f) B. Frisch, C. Boeckler, F. Schuber, *Bioonjugate Chem*. 1996, **7**, 180-186; (g) X. Chen, Y. H. Chen, V. E. Anderson, *Anal. Biochemm.* 1999, **273**, 192-203; (h) D. M. Schulz, C. Ihling, G. M. Clore, A. Sinz, *Biochemistry* 2004, **43**, 4703-4715; (i) B. Ekman, C. Lofter, I. Sjoholm, *Biochemistry*. 1976, **15**, 5115-5120; (j) C. Renner, R. Behrendt, N. Heim, I. Moroder, *Biopolymers*. 2002, **63**, 382-393; (k) S. M. Standley, Y. J. Kwon, N. Murthy, J. Kunisawa, N. Shastri, S. J. Guillaudeu, L. Lau, J. M. J. Frechet, *Bioconjugate Chem*. 2004, **15**, 1281-1288; (l) X. Liang, H. Asanuma, M. Komiyama, *J. Am. Chem. Soc*. 2002, **124**, 1877-1883; (m) K. V. Sing, J. Kaur, G. C. Varshney, M. Raje, C. R.

Suri, *Bioconjugate Chem*. 2004, **15**, 168-173.
2. K. H. Dalton, G. M. Dubowchik, A. W. Michael, *Tetrahedron Lett*. 2002, **43**, 1987-1990.
3. (a) H. Morita, J. K. Byung, *Chem. Lett*. 2000, **1**, 42-43; (b) H. Morita, J. K. Byung, S. Yamada, T. Funada, Y. Kadoma, *Bioor. Med. Chem. Lett*. 2000, **10**, 357-358.
4. H. Morita, M. C. Sheikh, J. K. Byung, S. Takagi, unpublished work.
5. (a) M. C. Sheikh, S. Takagi, M. Sakai, H. Abe, H. Morita, *J. Org. Biomol. Chem.* 2008, **6**, 4505-4508; (b) M. C. Sheikh, S. Takagi, A. Ogasawara, M. Ohira, R. Miyatake, H. Abe, T. Yoshimura, H. Morita, *Tetrahedron* 2010, **66**, 2132-2140.
6. A. Arrieta, T. Garcia, C. Palomo, *Synth. Commun*. 1982, **12**, 681-690.
7. D. H. R. Barton, P. Blundell, J. C. Jaszberenyi, *Tetrahedron Lett*. 1989, **30**, 2341-2344.
8. (a) A. R. Katrizky, A. Vakulenko, R. Jain, ARKIVON (Gainesville, FL, United states) 2003, **14**, 131-139; (b) A. R. Katrizky, K. H. Suzuki, *J. Org. Chem*. 2000, **65**, 8210-8213; (c) M. C. Sheikh, S. Takagi, T. Yoshimura, H. Morita, *Tetrahedron* 2010, **66**, 7272-7278.
9. A. R. Kartritzky, N. Shobana, J. Prnak, A. S. Afridi, F. Wei-Qiang, *Tetrahedron* 1992, **48**, 7817-7822.
10. A. R. Katritzky, X. Lan, J. Z. Yang, V. Olga, *Chem. Rev*. 1998, **98**, 409-548.
11. A. R. Katritzky, S. Ledoux, R. M. Witek, S. K. Nair, *J. Org. Chem*. 2004, **69**, 2979-2982.
12. N. Mori, H. Togo, *Tetrahedron* 2005, **61**, 5915-5925.
13. Q. Liu, J. Li, Xiao-X. Shen, Rui-G. XING, J. Yang, Z. Liu, B. Zhou, *Tetrahedron Lett*. 2009, **50**, 1026-1028.
14. L. Mercs, G. Pozzi, S. Quici, *Tetrahedron Lett*. 2007, **48**, 3053-3056.
15. A. J. A. Wason, A. C. Maxwell, J. M. A. Williams, *Org. Lett*. 2009, **11**, 2667-2670.
16. H. Nakatsuji, M. Morimoto, T. Misaki, Y. Tanabe, *Tetrahedron* 2007, **63**, 12071-12080.

^{1}H and ^{13}C NMR Spectrum:

Phthalimido 4-chloroformylbutanoate (9) (^{1}H-NMR)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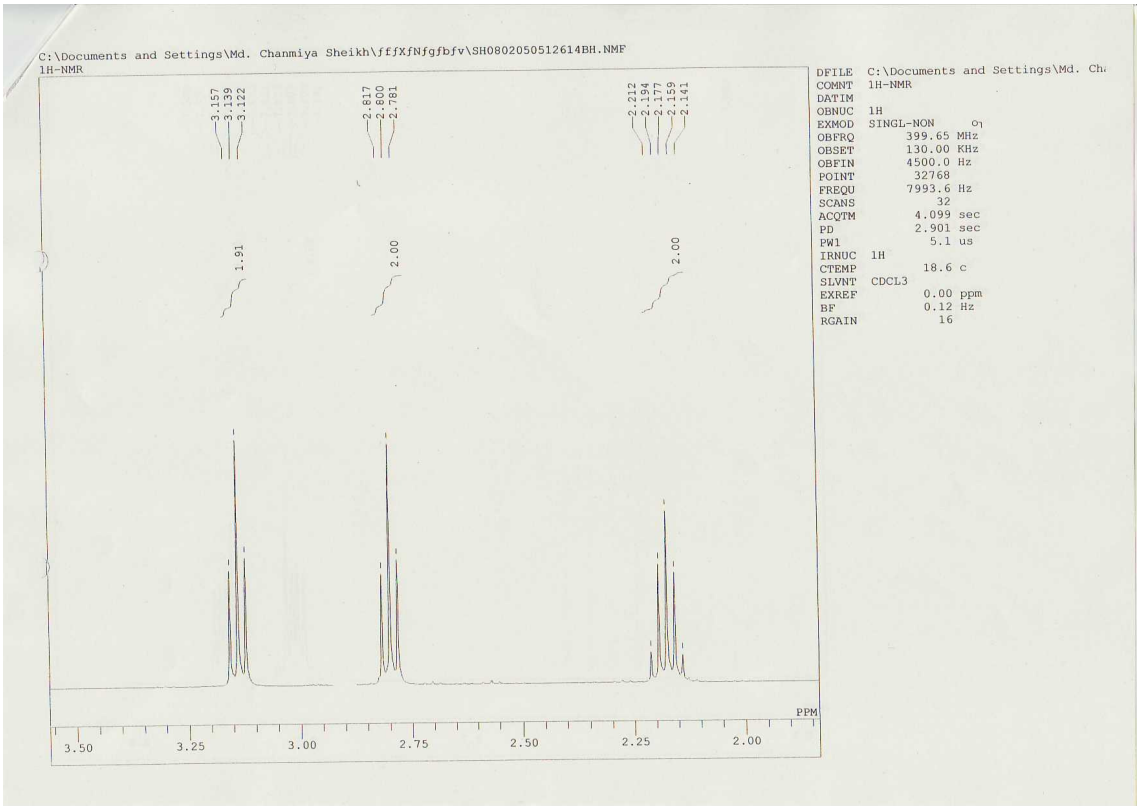
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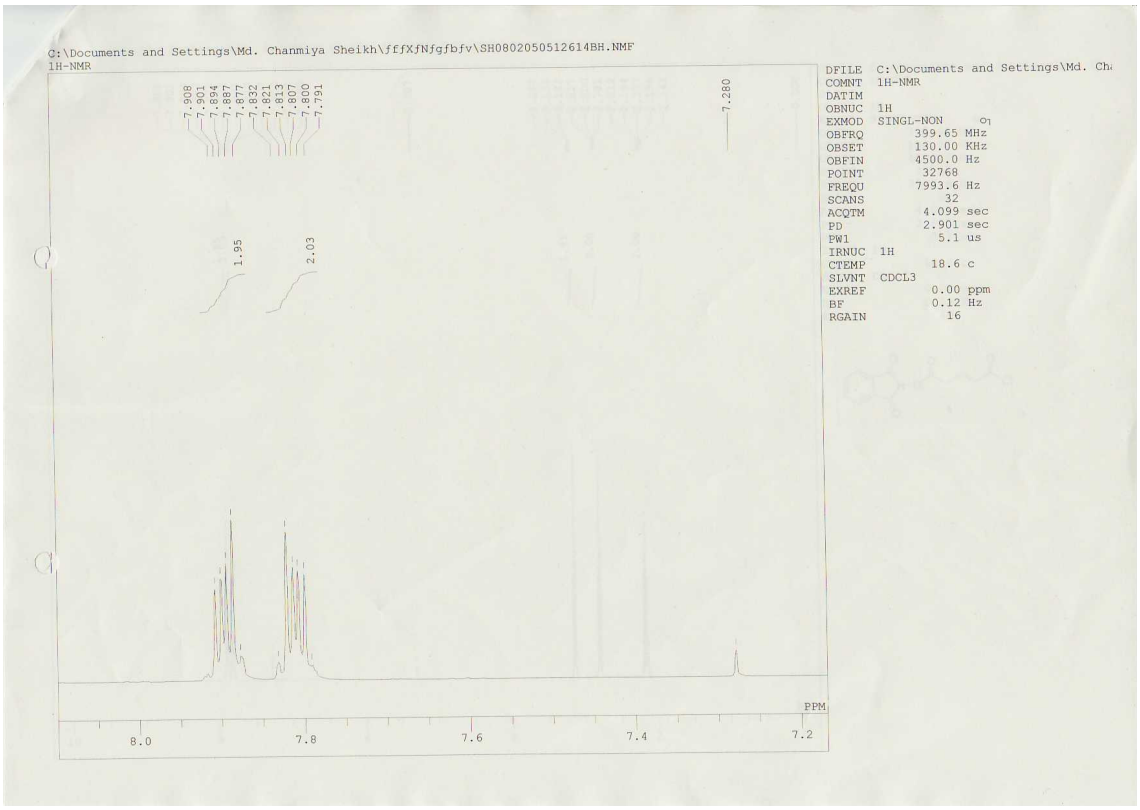

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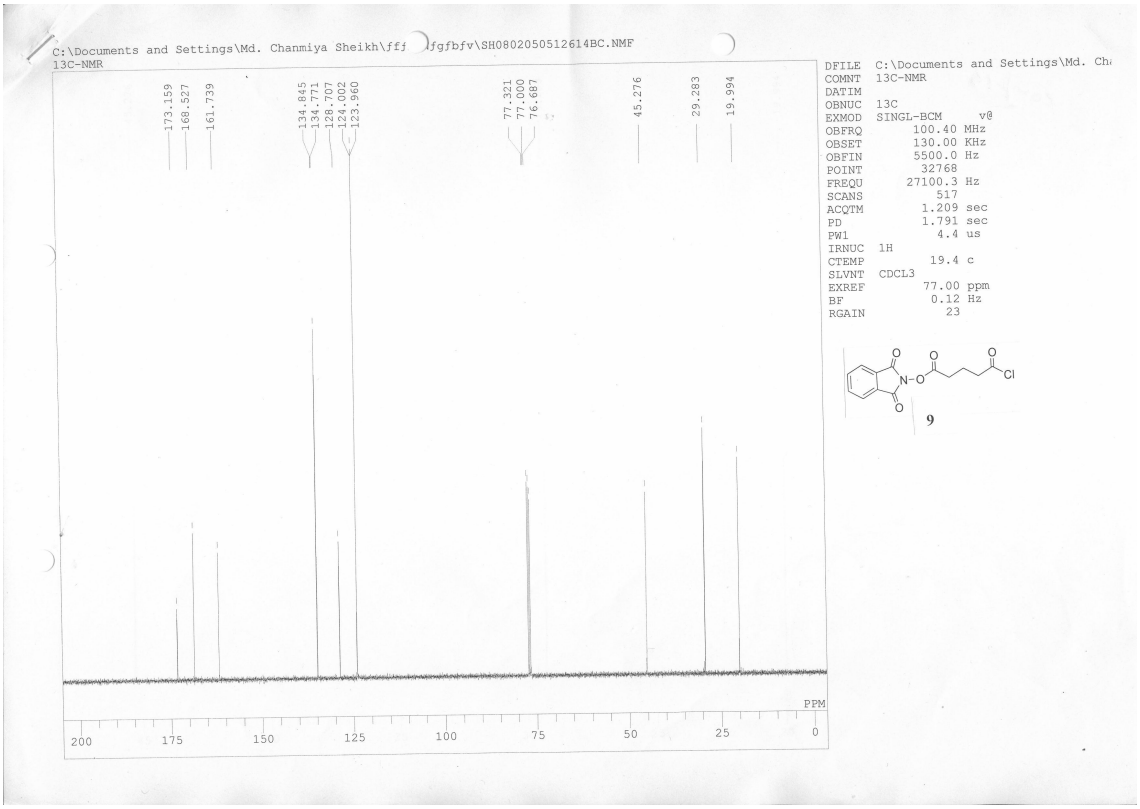

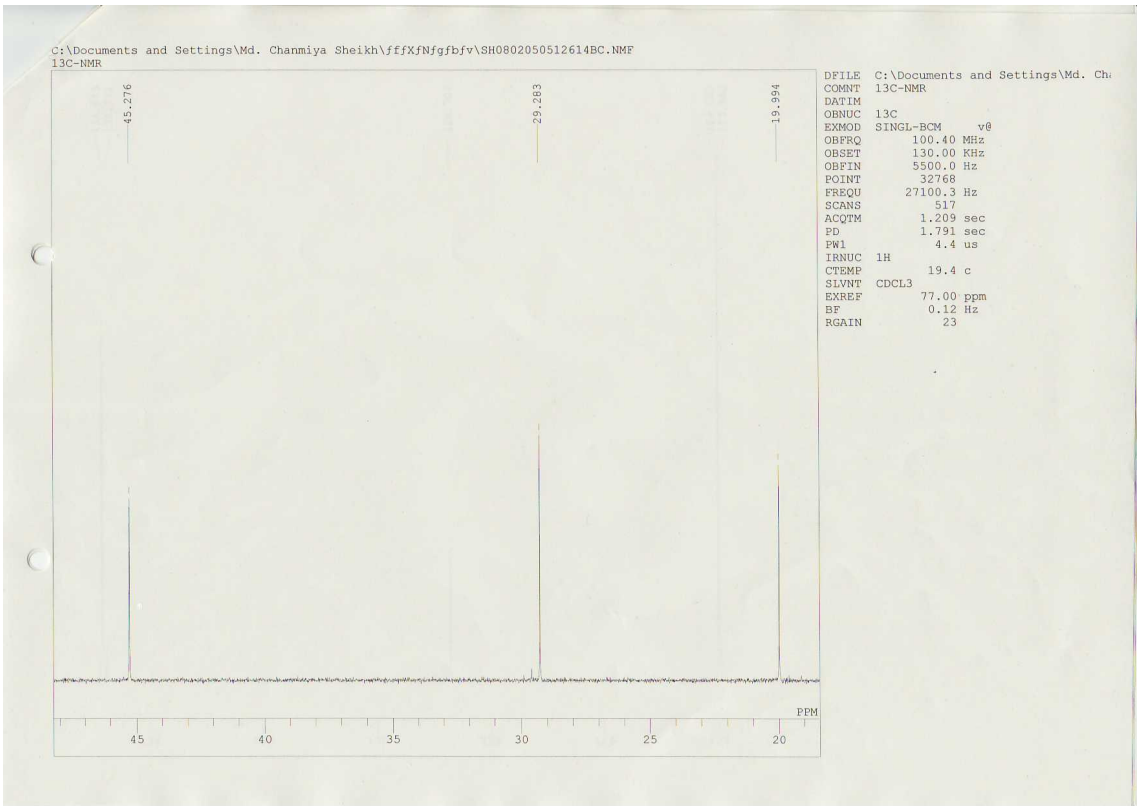
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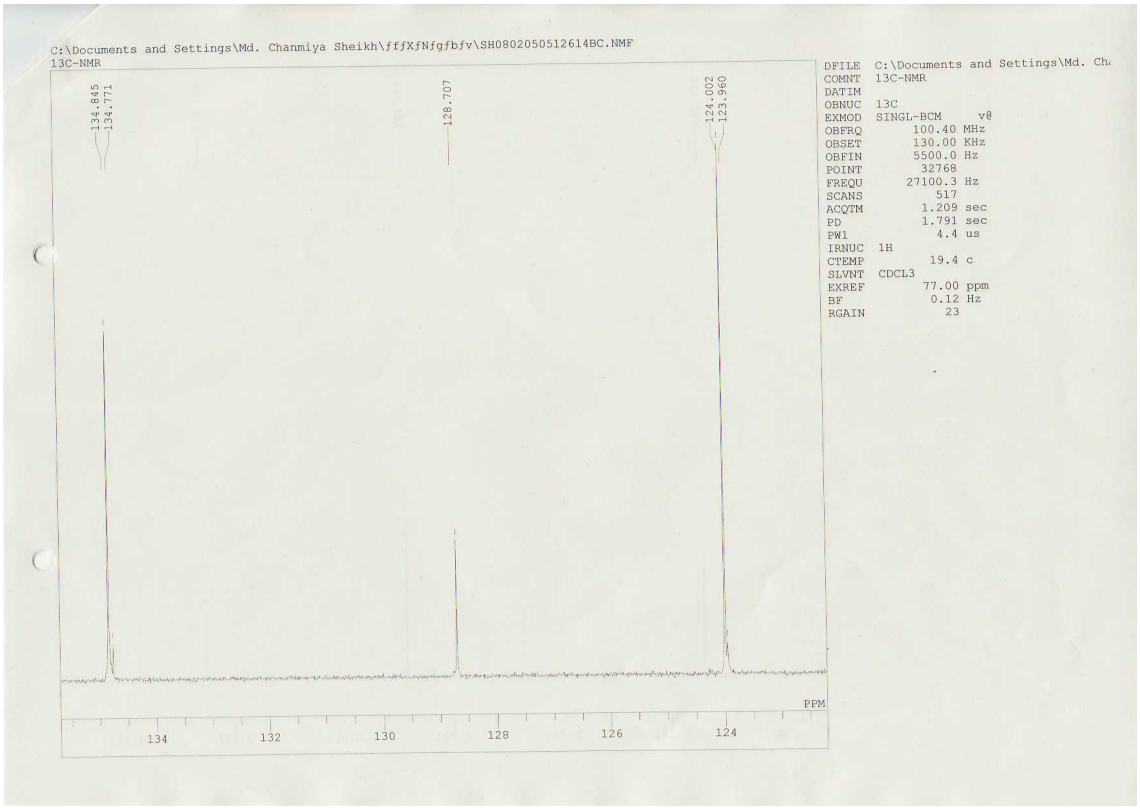
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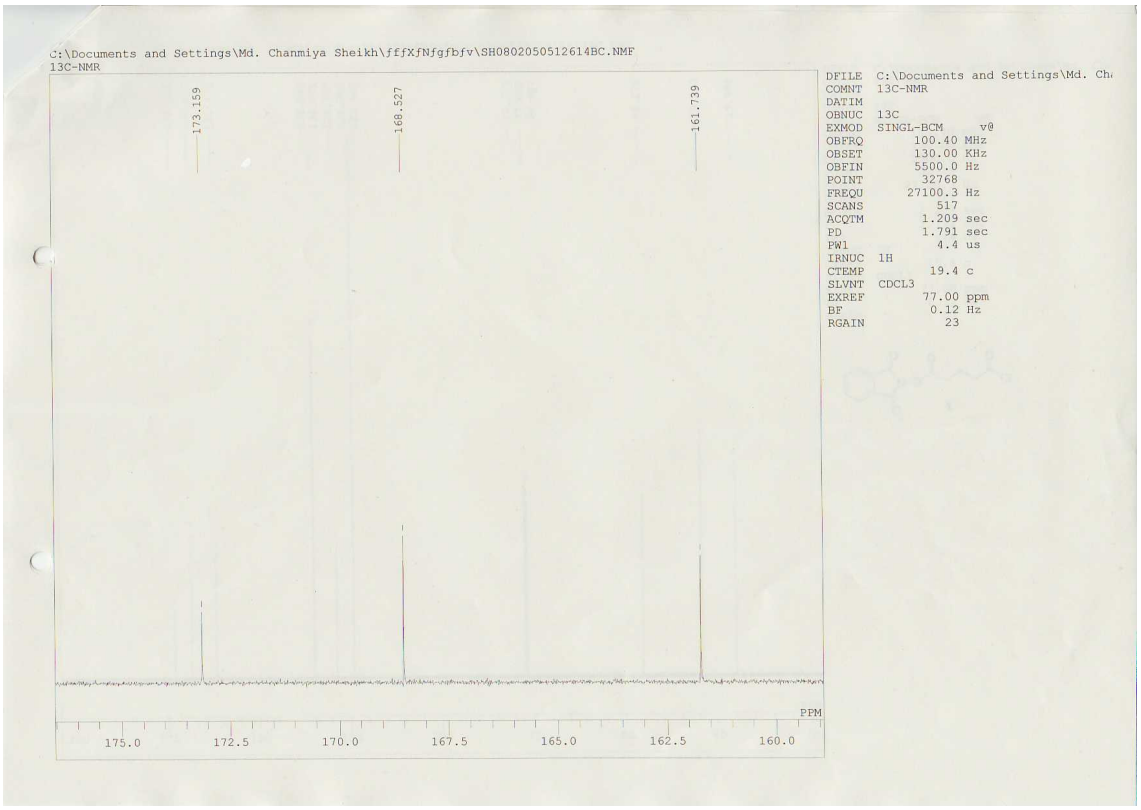

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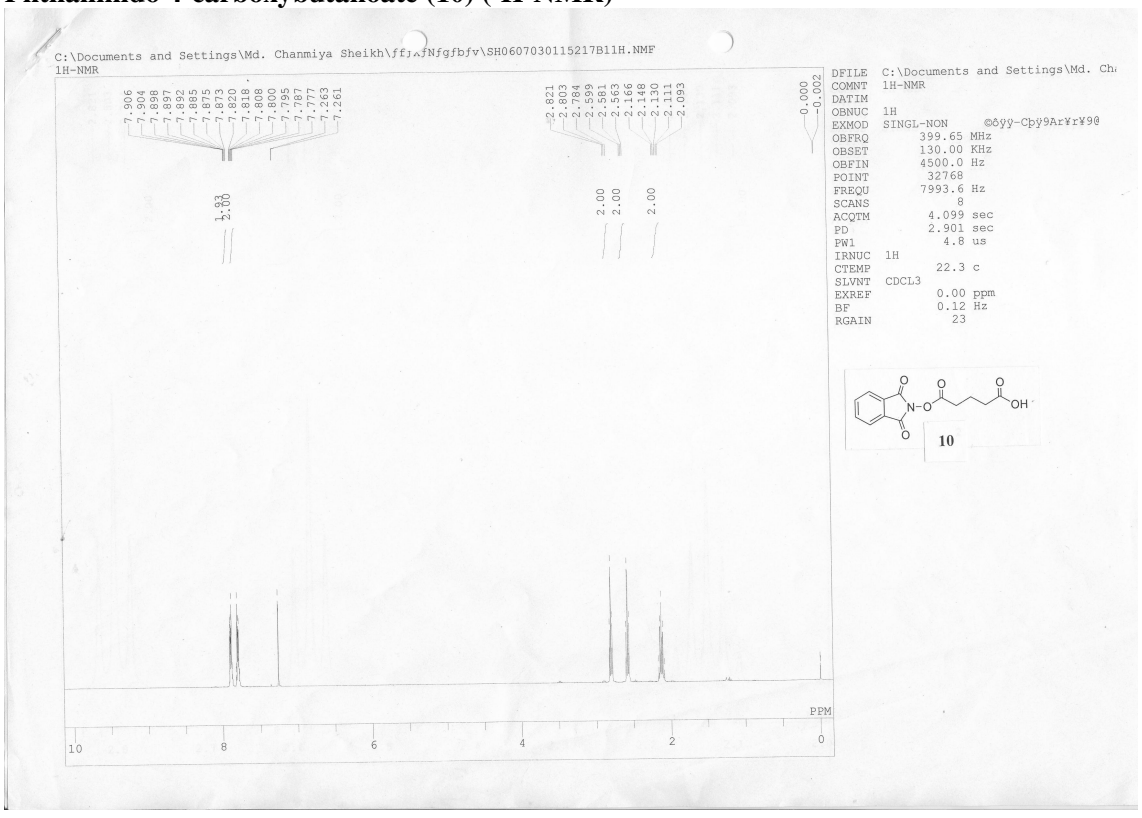

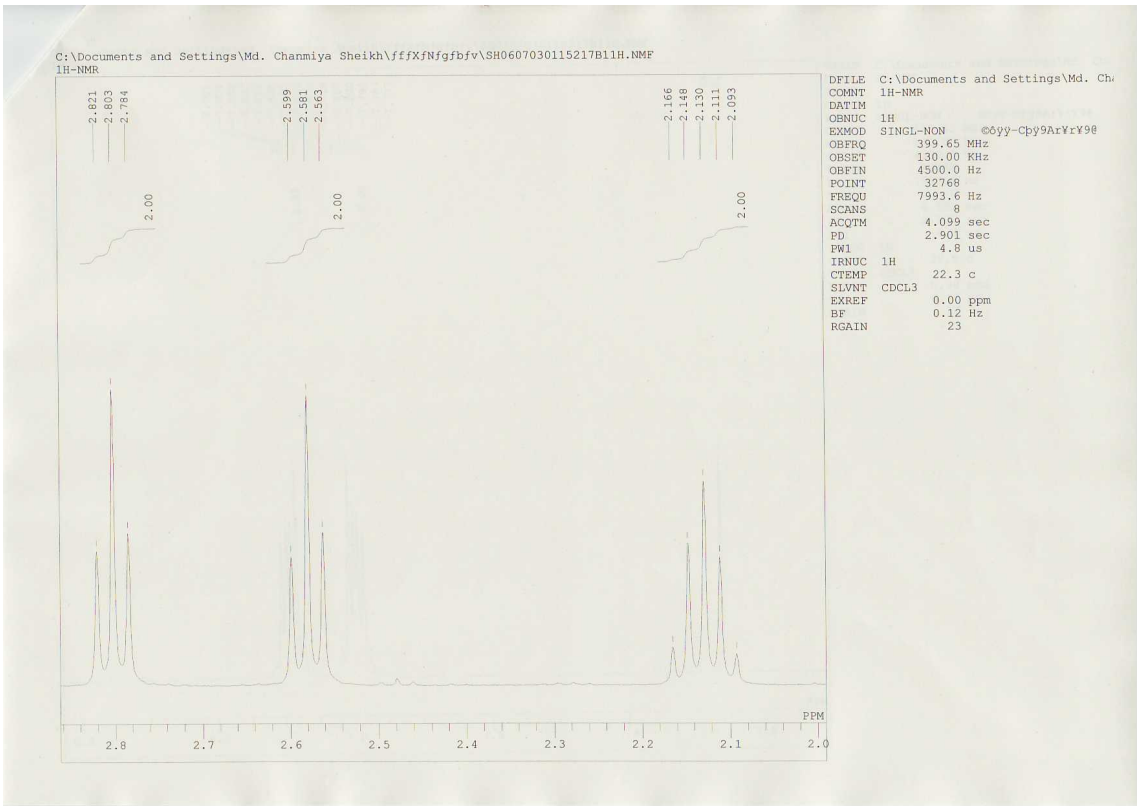

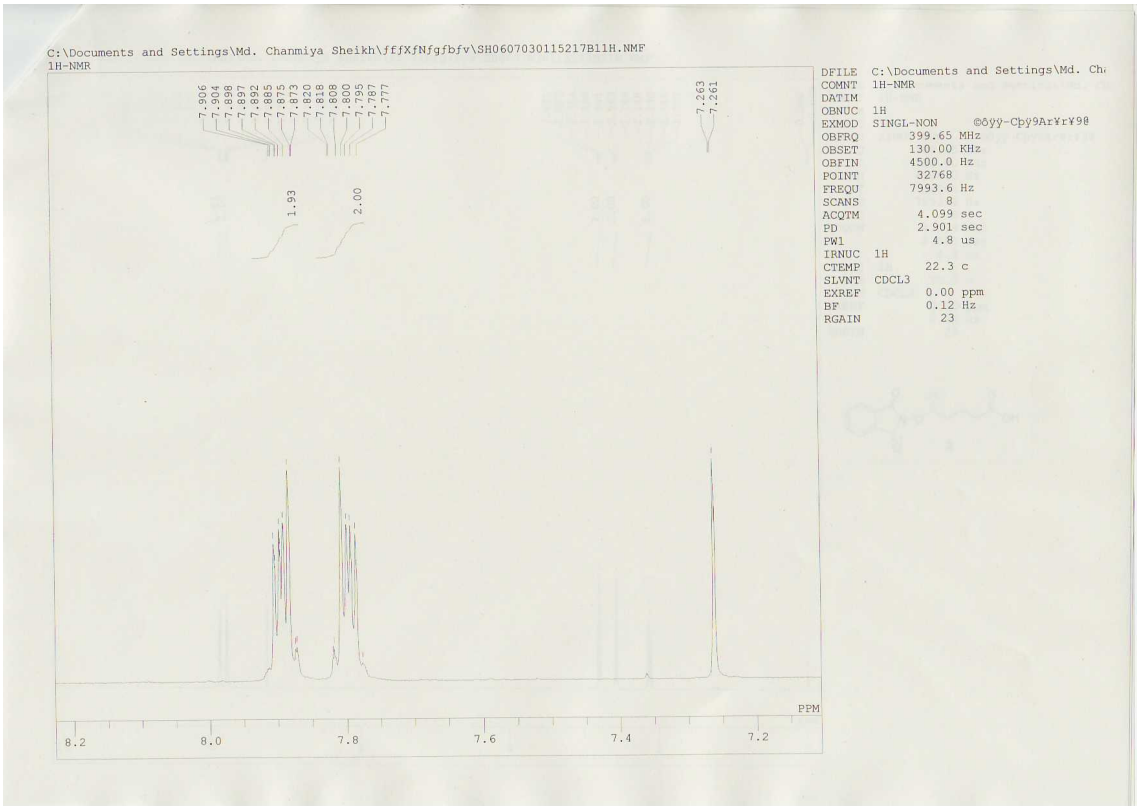

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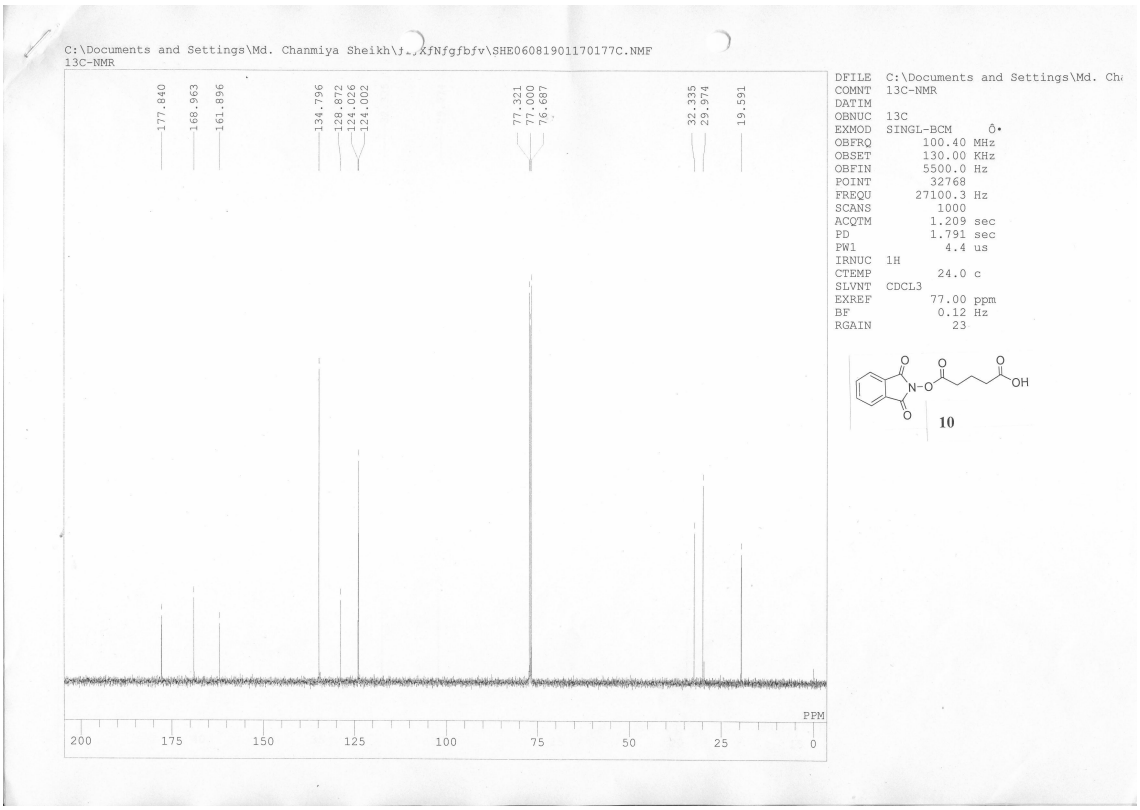
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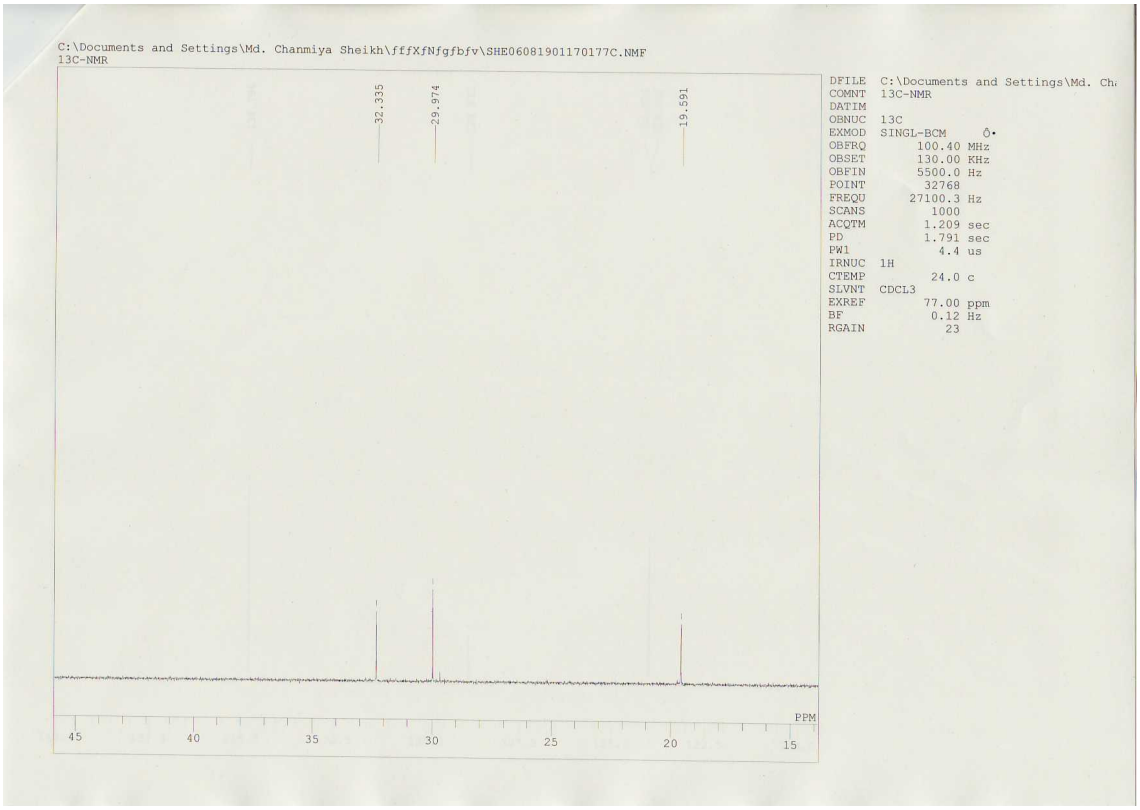
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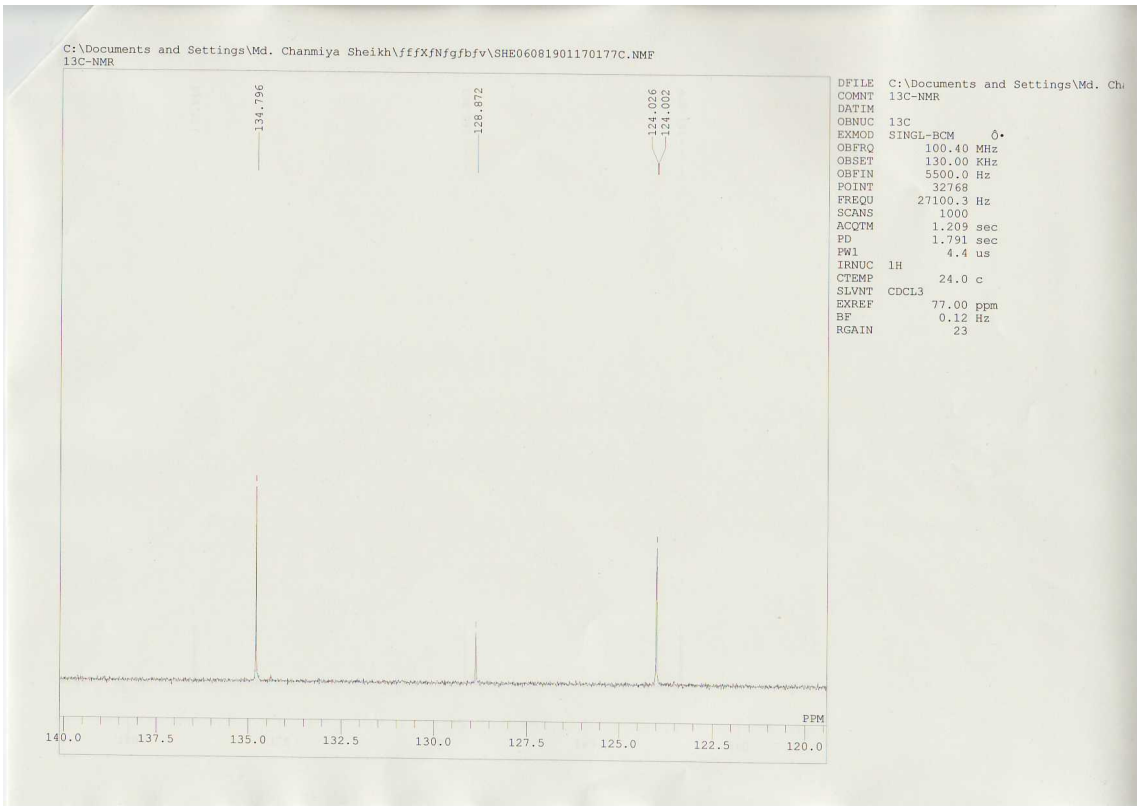

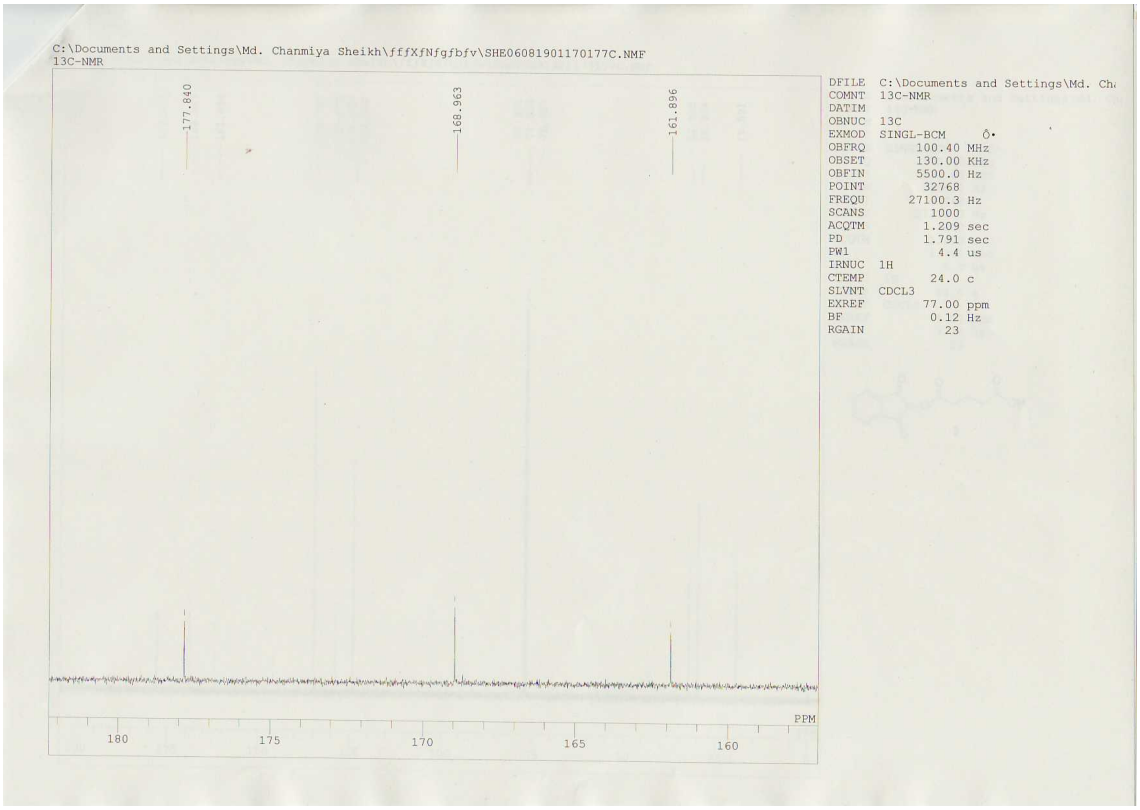

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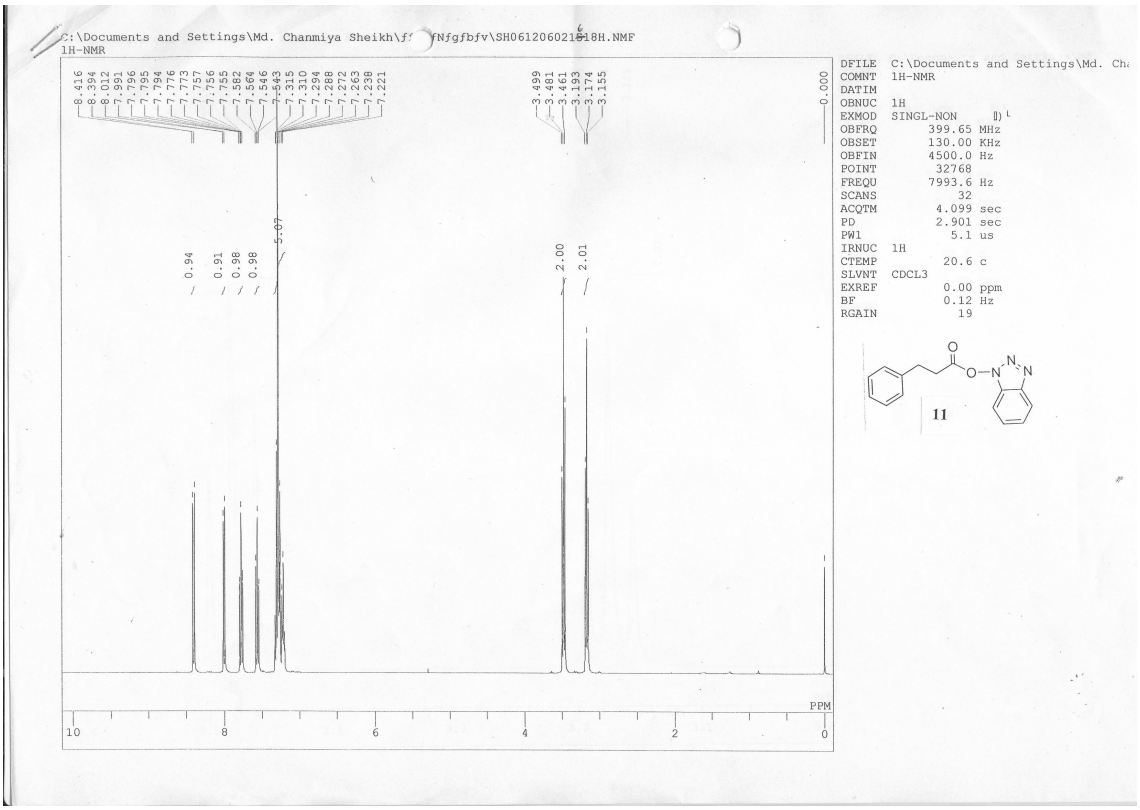
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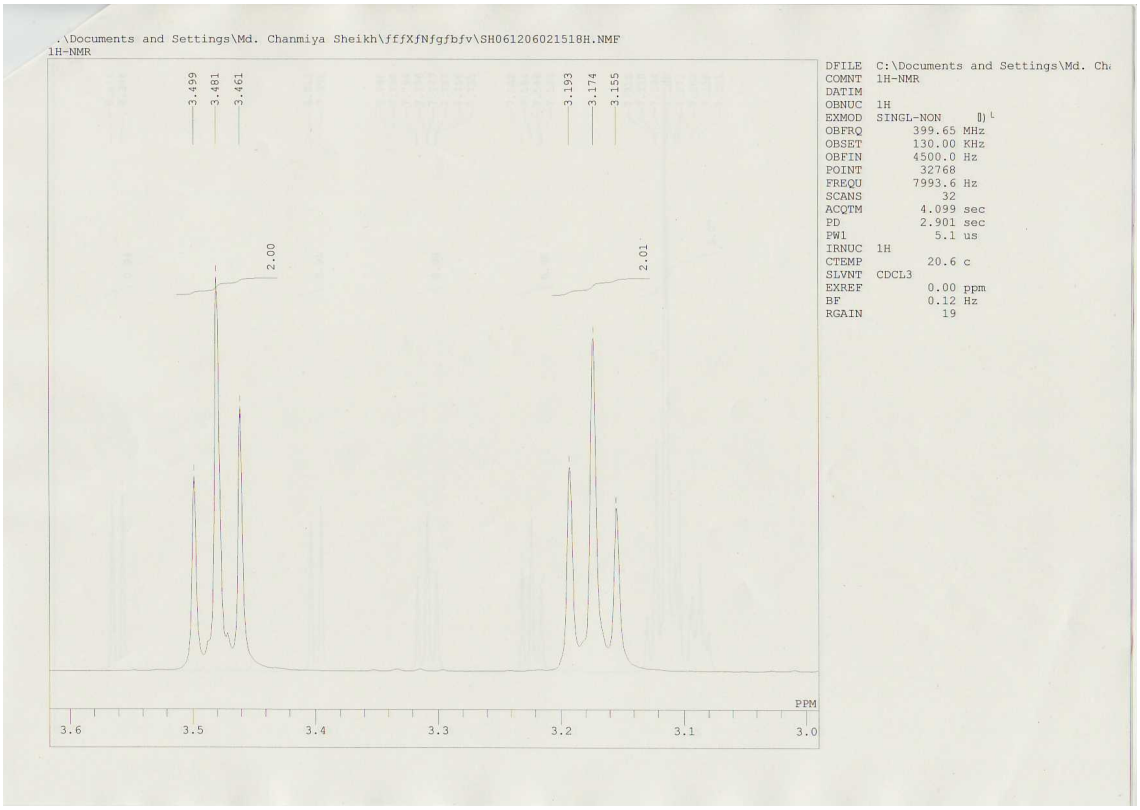
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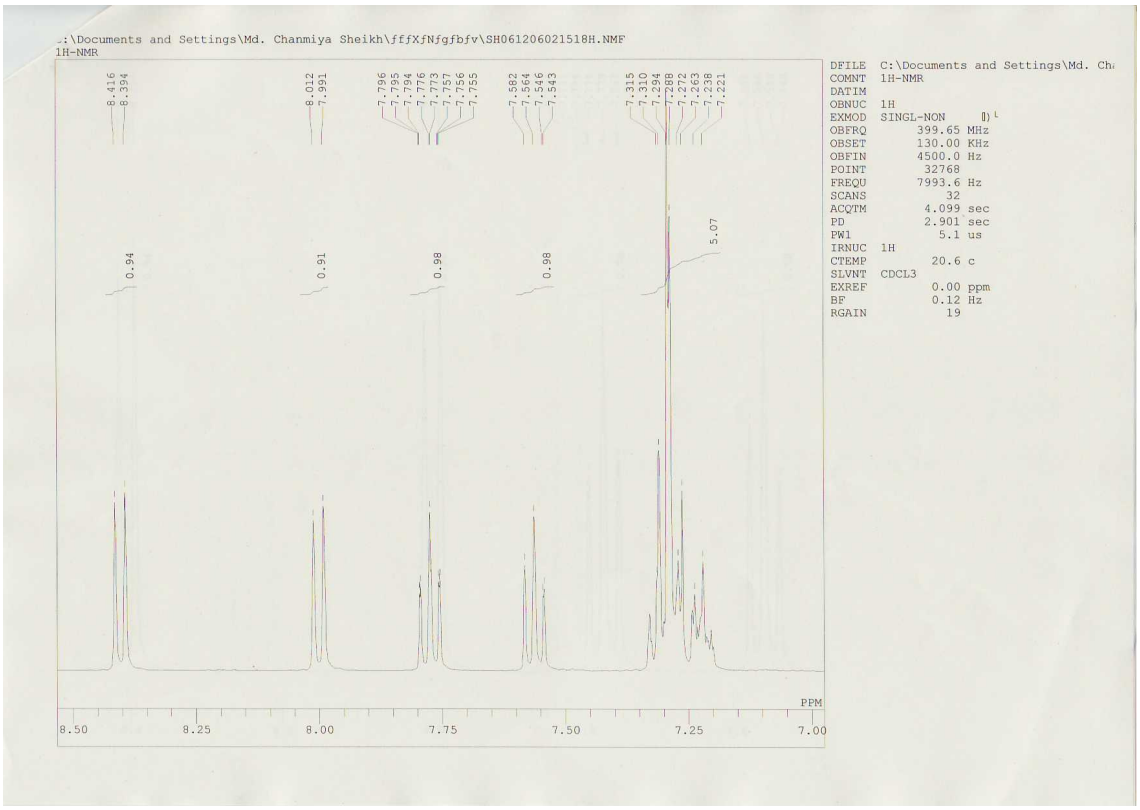
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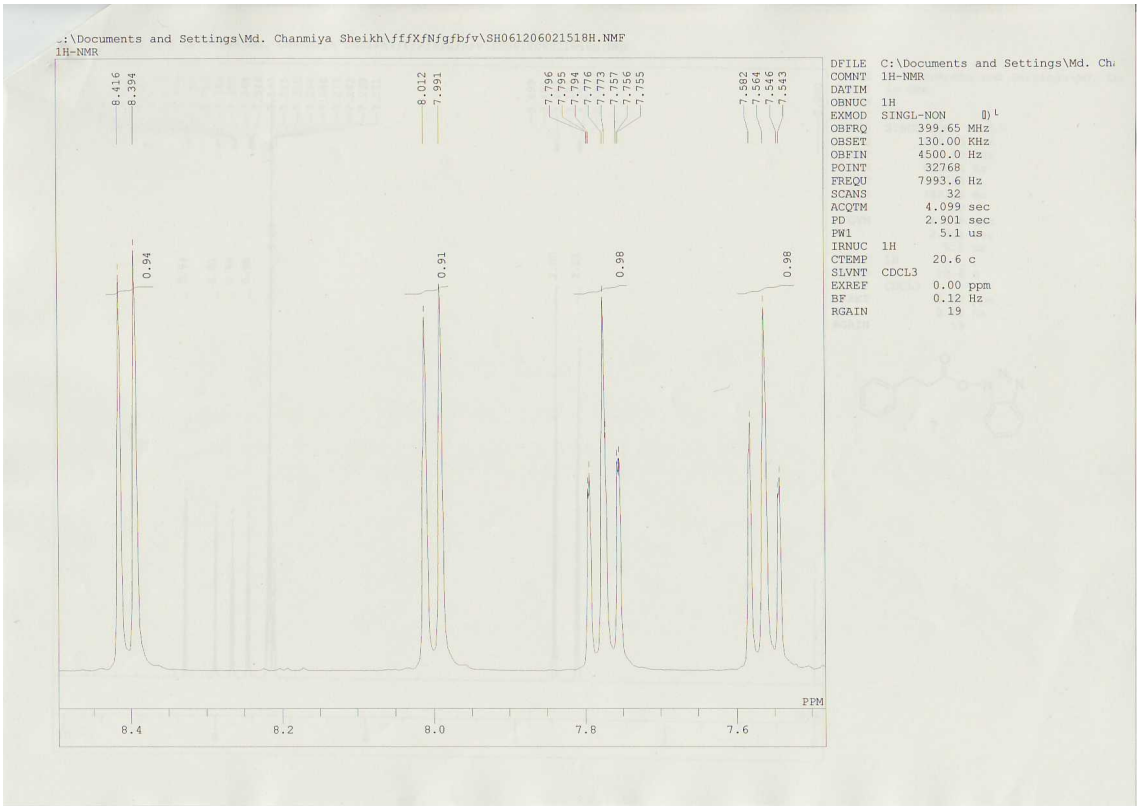
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19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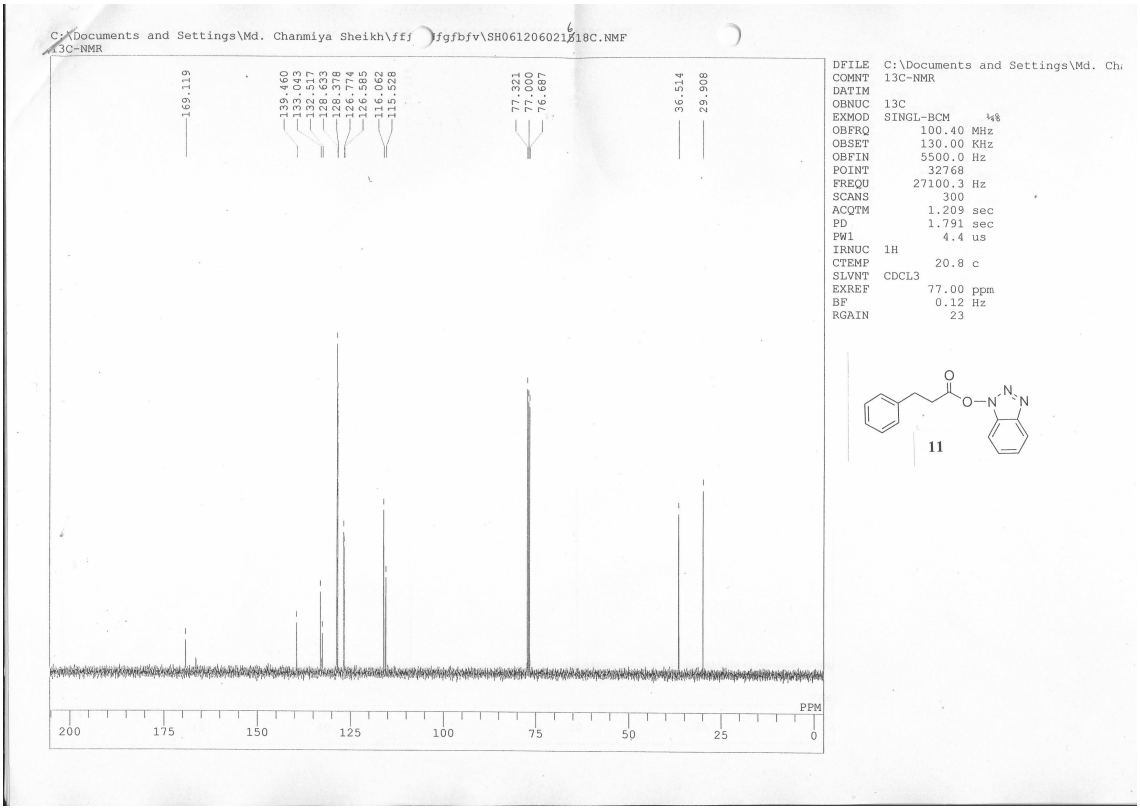
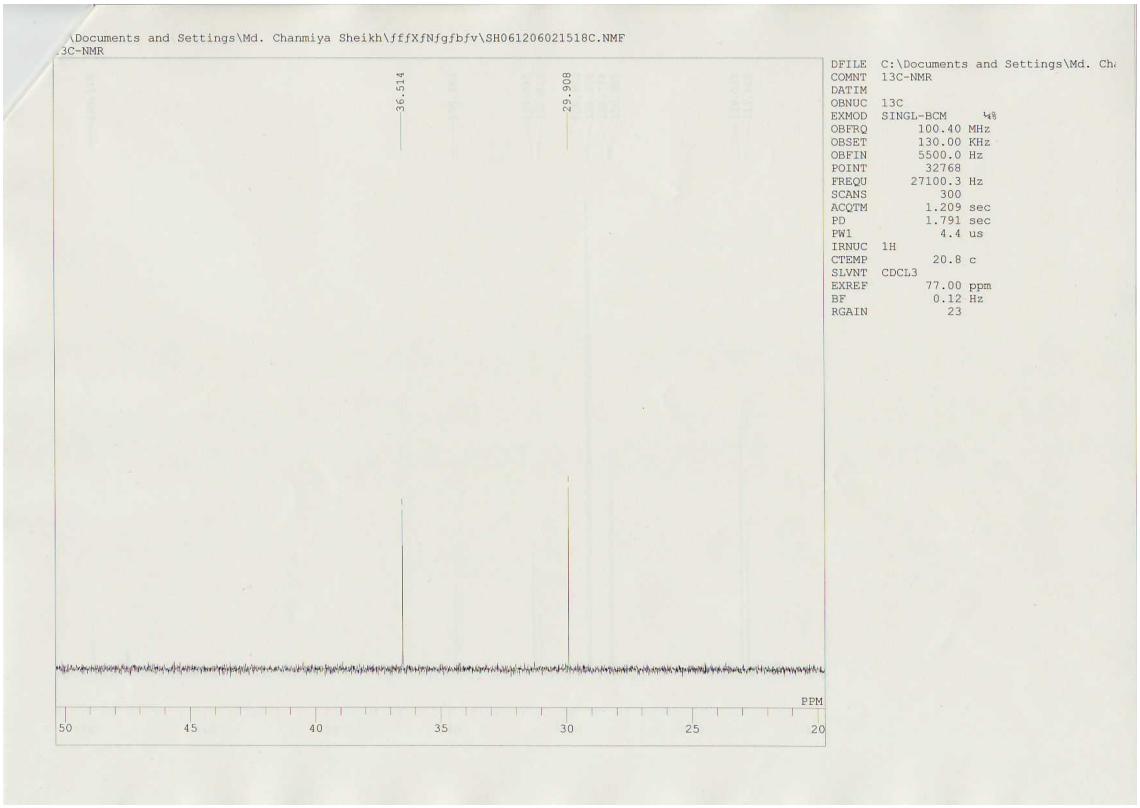
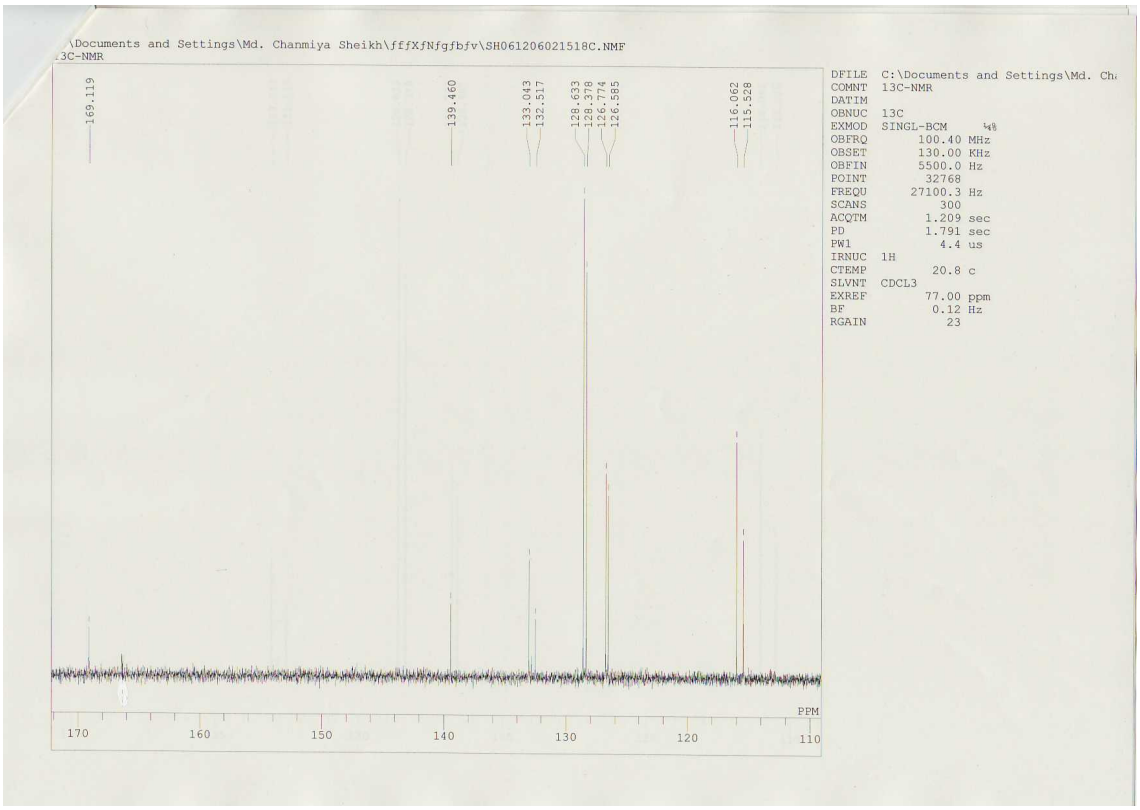
1H-NMR
8.416
8.394
8.012
7.991
7.796
7.795
7.794
7.776
7.773
7.757
7.756
7.755
7.582
7.564
7.546
7.543
0.94
0.91
0.98
0.98
PPM
8.4
8.2
8.0
7.8
7.6
DFILE C:\Documents and Settings\Md. Ch
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL-NON
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 32
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.1 us
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19

Phenylpropionyloxy)benzotriazole (11) (^{13}C-NMR)



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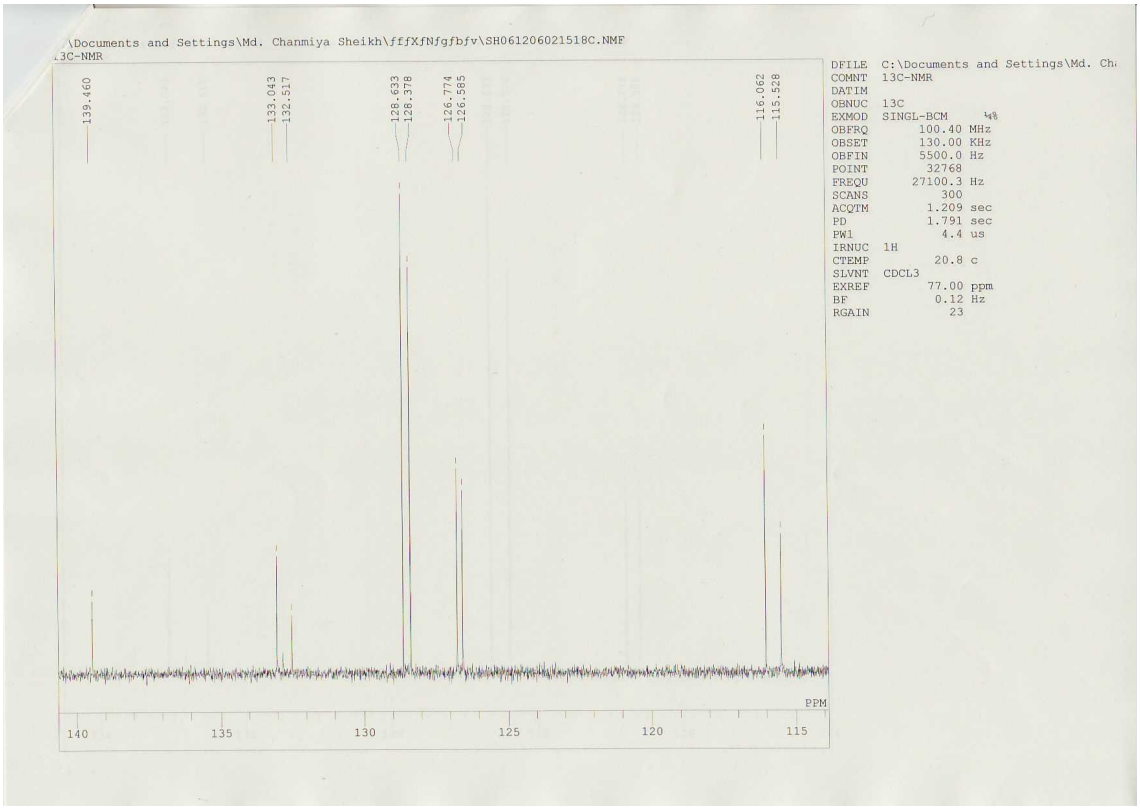
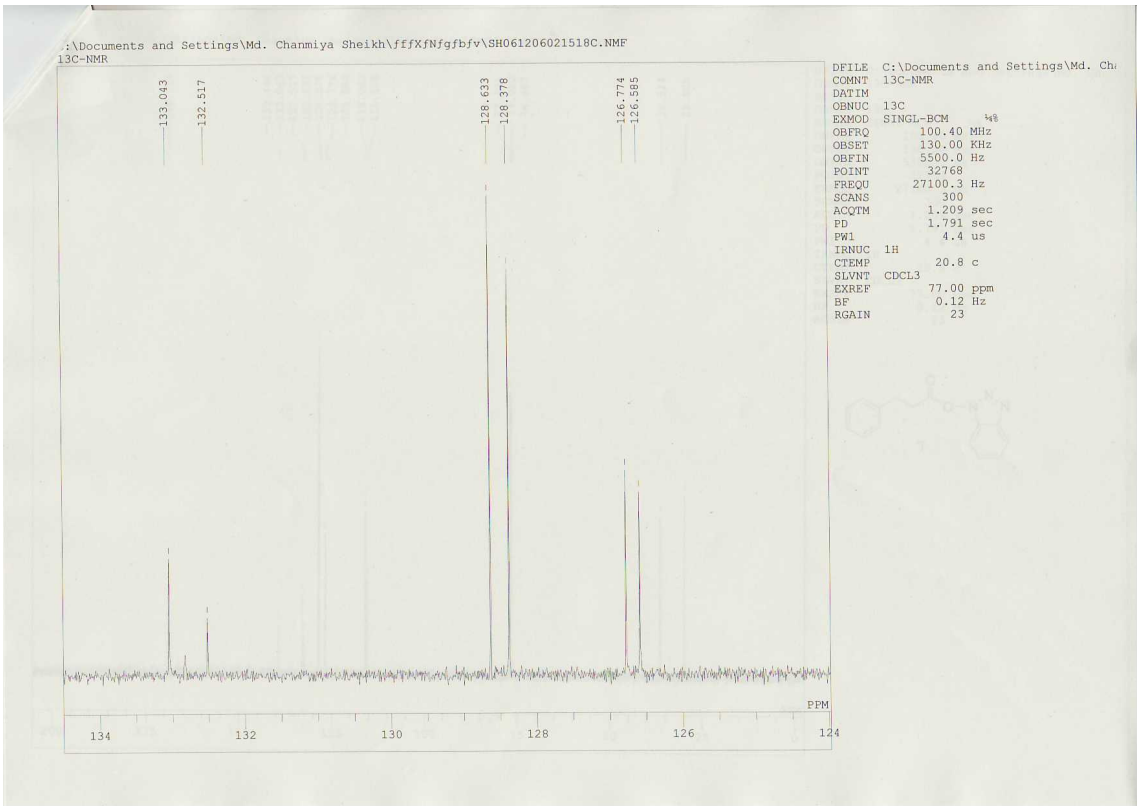
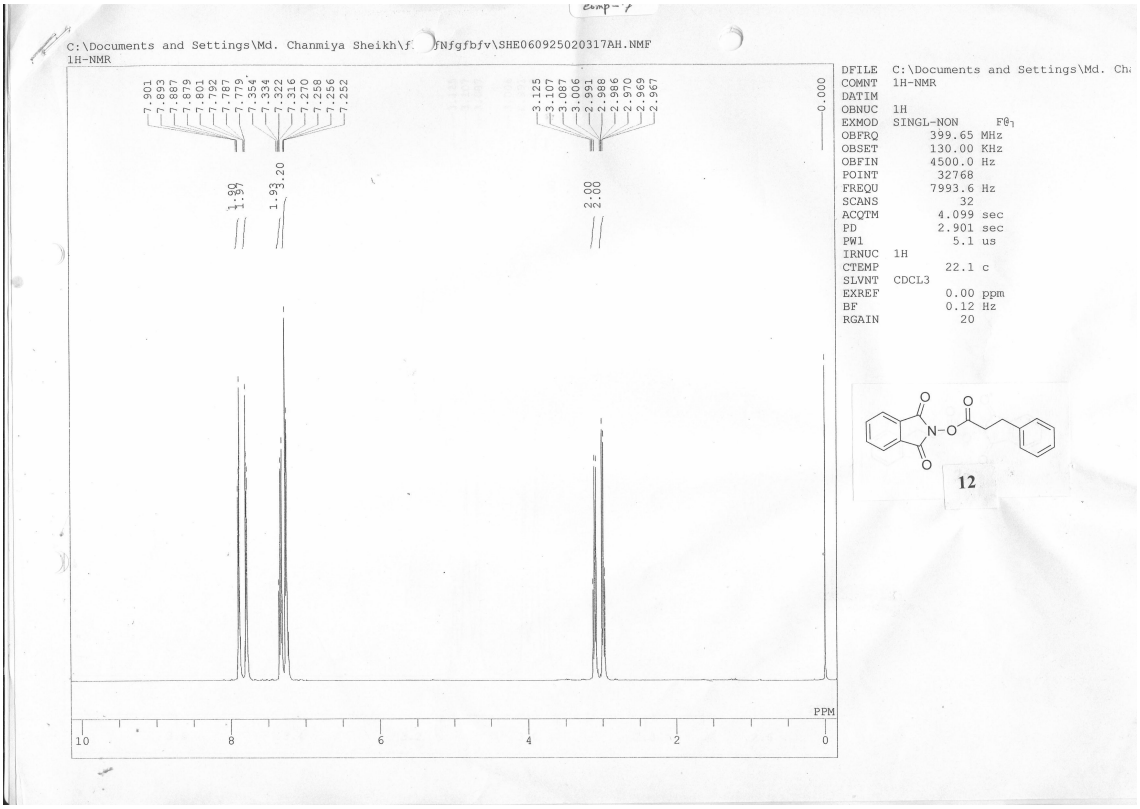
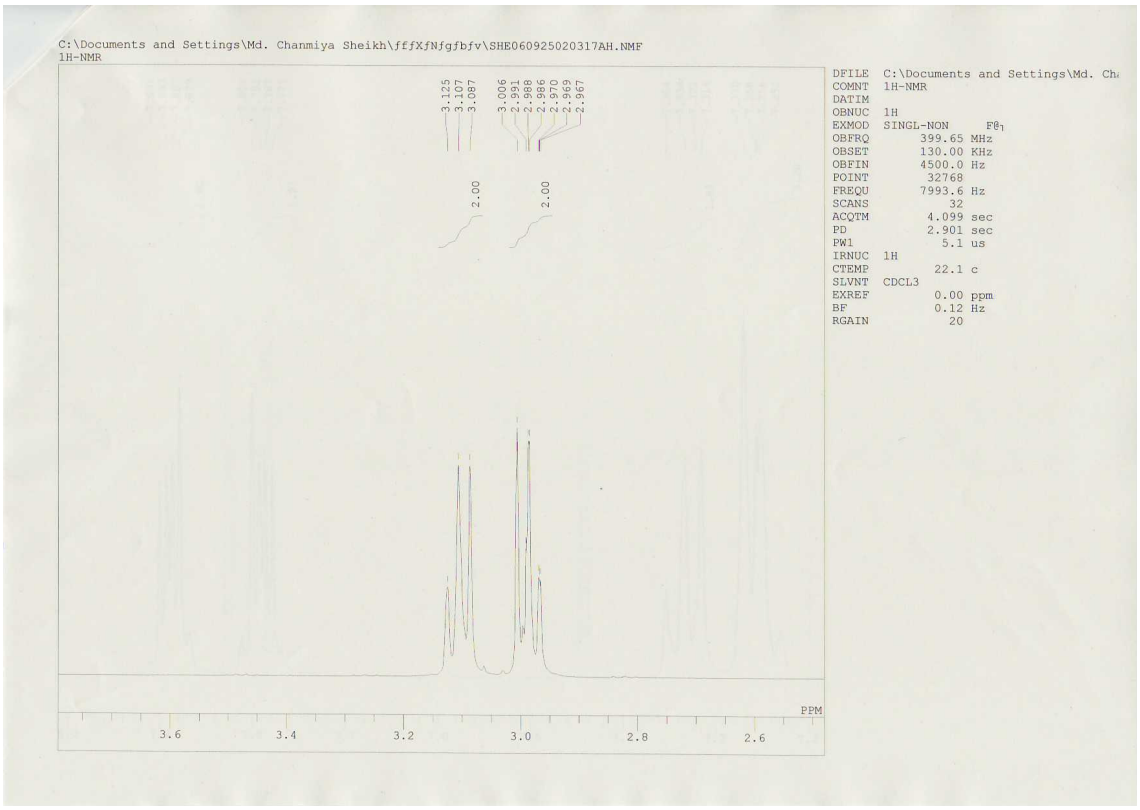
13C-NMR
169.119
139.460
133.043
132.517
128.633
128.378
126.774
126.585
116.062
115.528
PPM
170
160
150
140
130
120
110
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 300
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

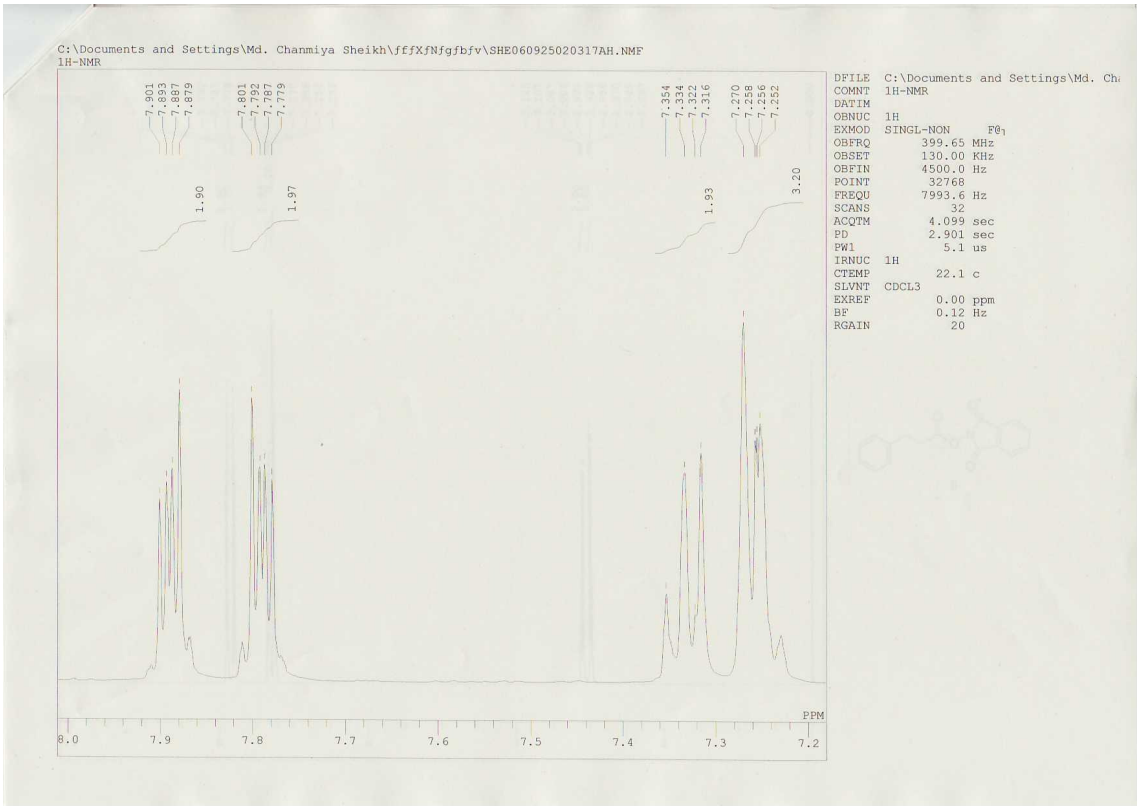

\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SH061206021518C.NMF
13C-NMR
139.460
133.043
132.517
128.633
128.378
126.774
126.585
116.062
115.528
PPM
140
135
130
125
120
115
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 300
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23



Phenylpropionyloxy)phthalimide (12) (^{1}H-NMR)







Phenylpropionyloxy)phthalimide (12) (^{13}C-NMR)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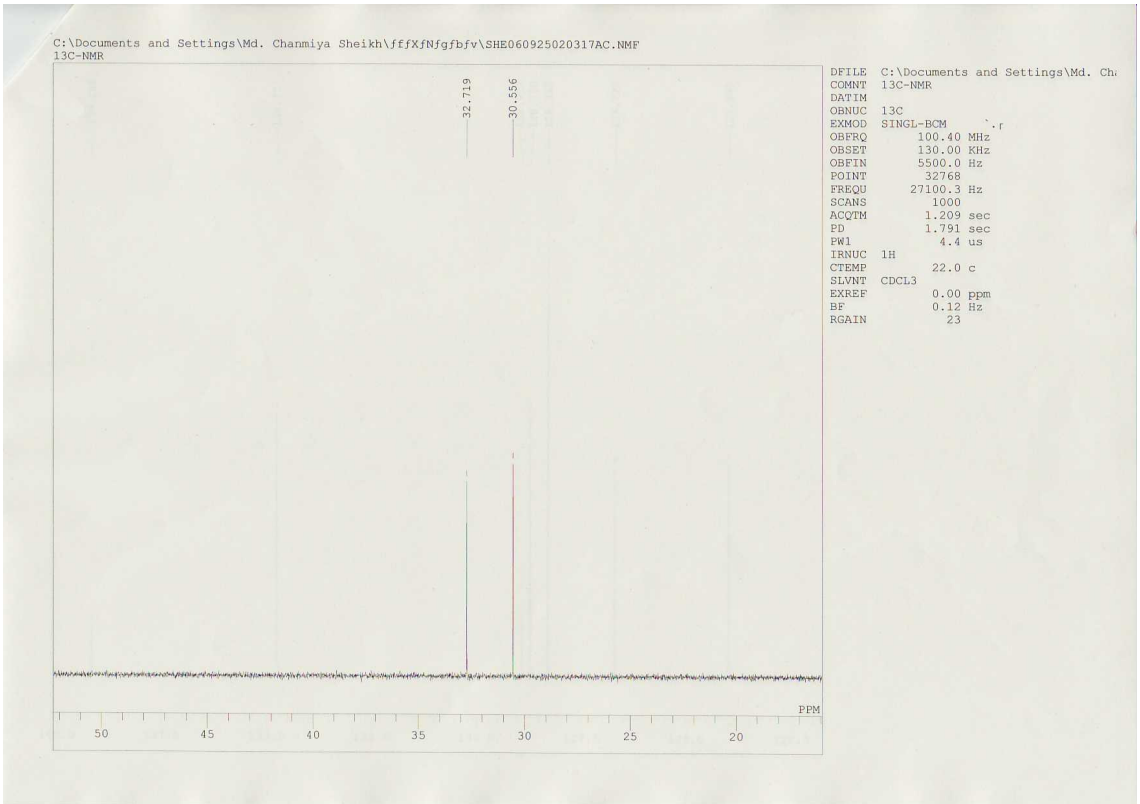
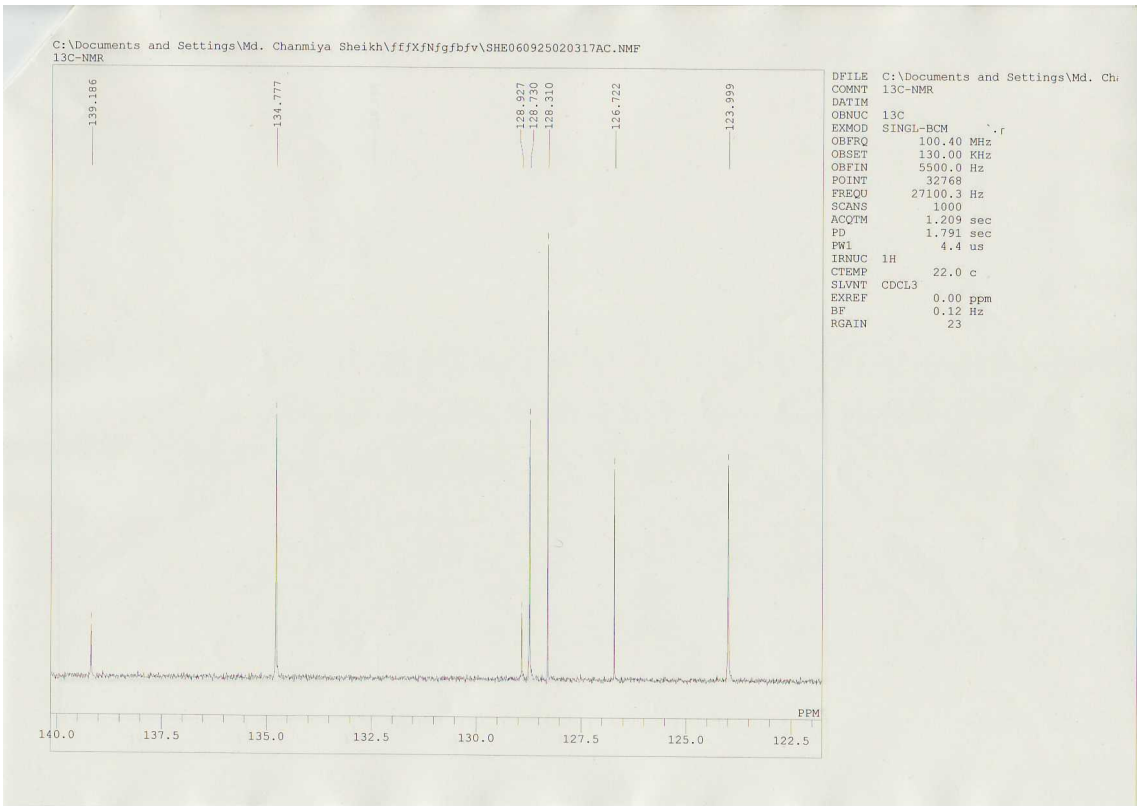
13C-NMR
168.895
161.902
139.186
134.777
128.927
128.730
128.310
126.722
123.999
77.343
77.031
76.710
32.719
30.556
0.000
PPM
200
175
150
125
100
75
50
25
0
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM `.r
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23
12


C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SHE060925020317AC.NMF
13C-NMR
32.719
30.556
PPM
50
45
40
35
30
25
20
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM `.r
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23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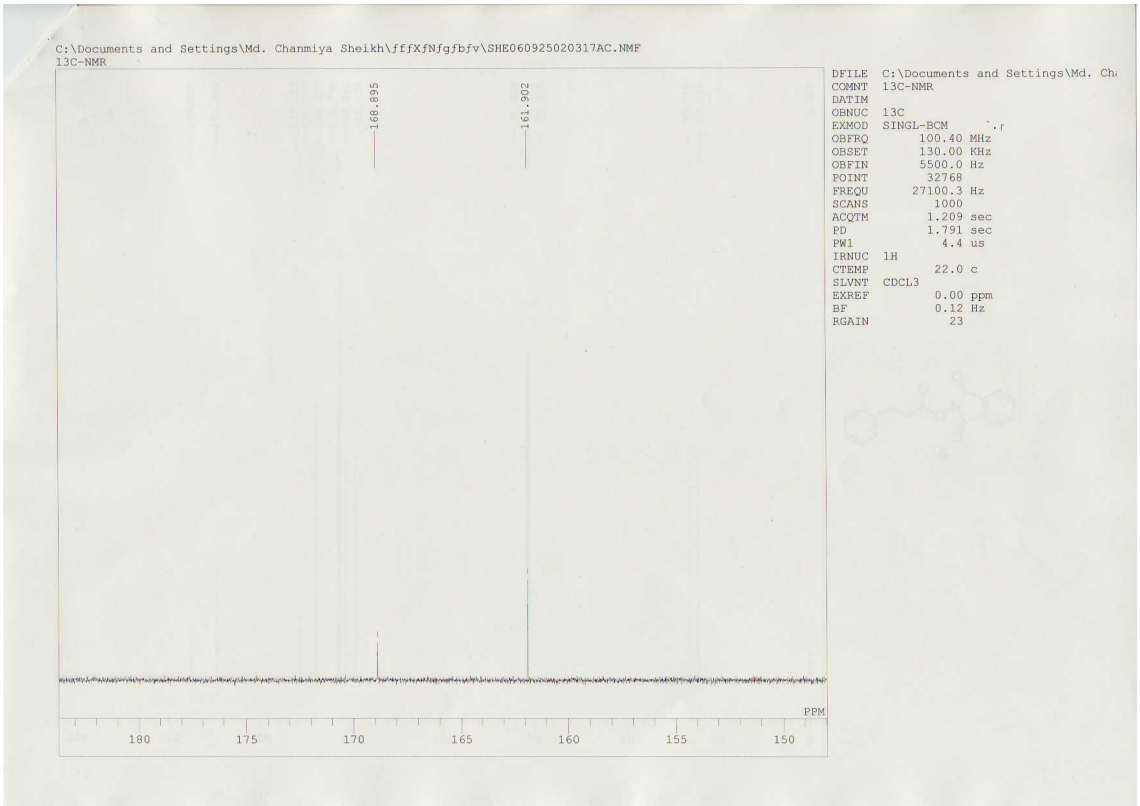
13C-NMR
139.186
134.777
128.927
128.730
128.310
126.722
123.999
PPM
140.0
137.5
135.0
132.5
130.0
127.5
125.0
122.5
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM `.r
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23

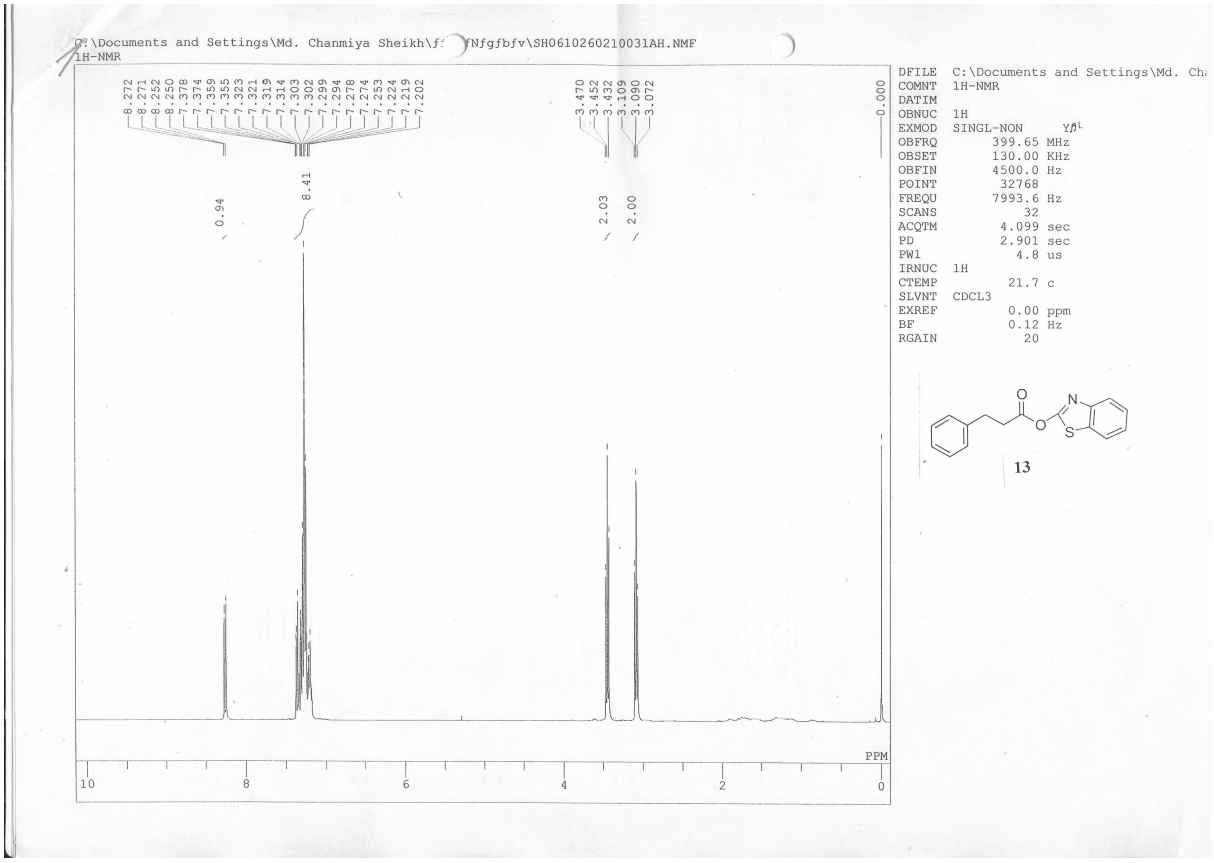

C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SHE060925020317AC.NMF
13C-NMR
168.895
161.902
PPM
180
175
170
165
160
155
150
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM `.r
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23

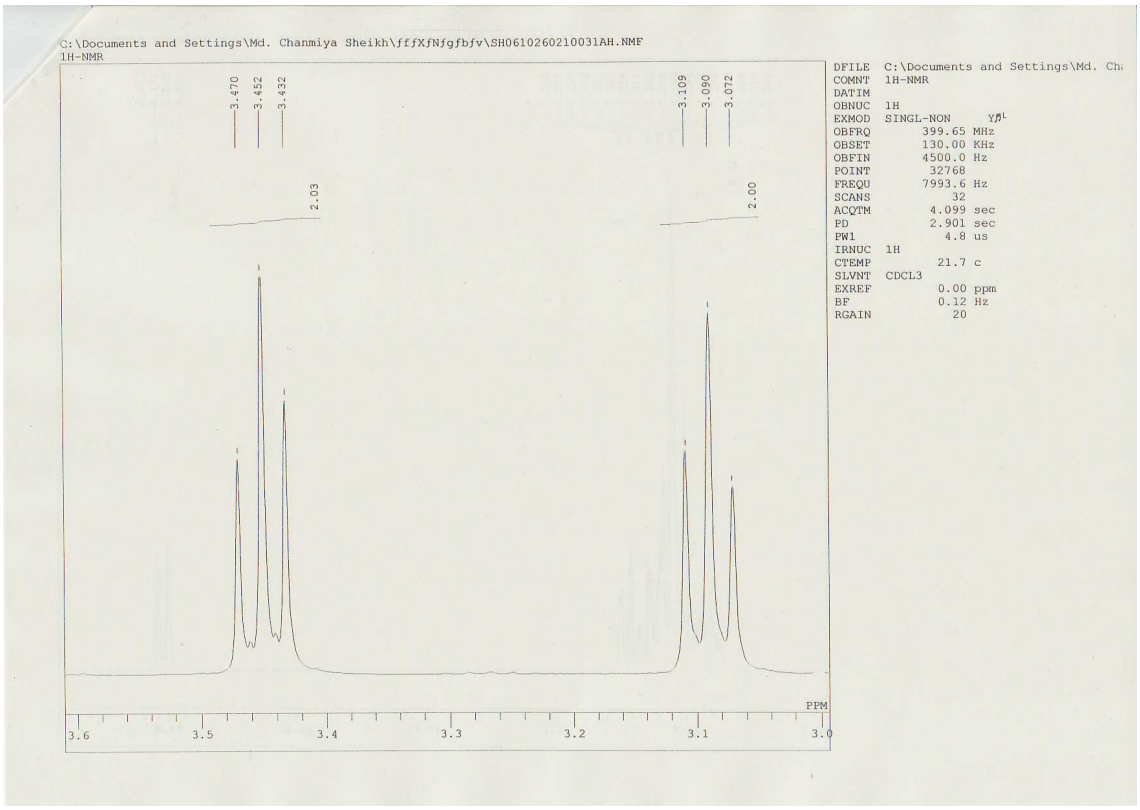
(ii) Supporting Information for the manuscript:

Title: “Syntheses and reactivities of non-symmetrical "active ester" bi-dentate cross-linking reagents having a phthalimidoyl and acid chloride, 2-benzothiazole, or 1-benzotriazole group”

(^{1}H –NMR and ^{13}C-NMR)

3-Phenylpropionyloxy)benzothiazole (**13**) (**^{1}H-NMR)**

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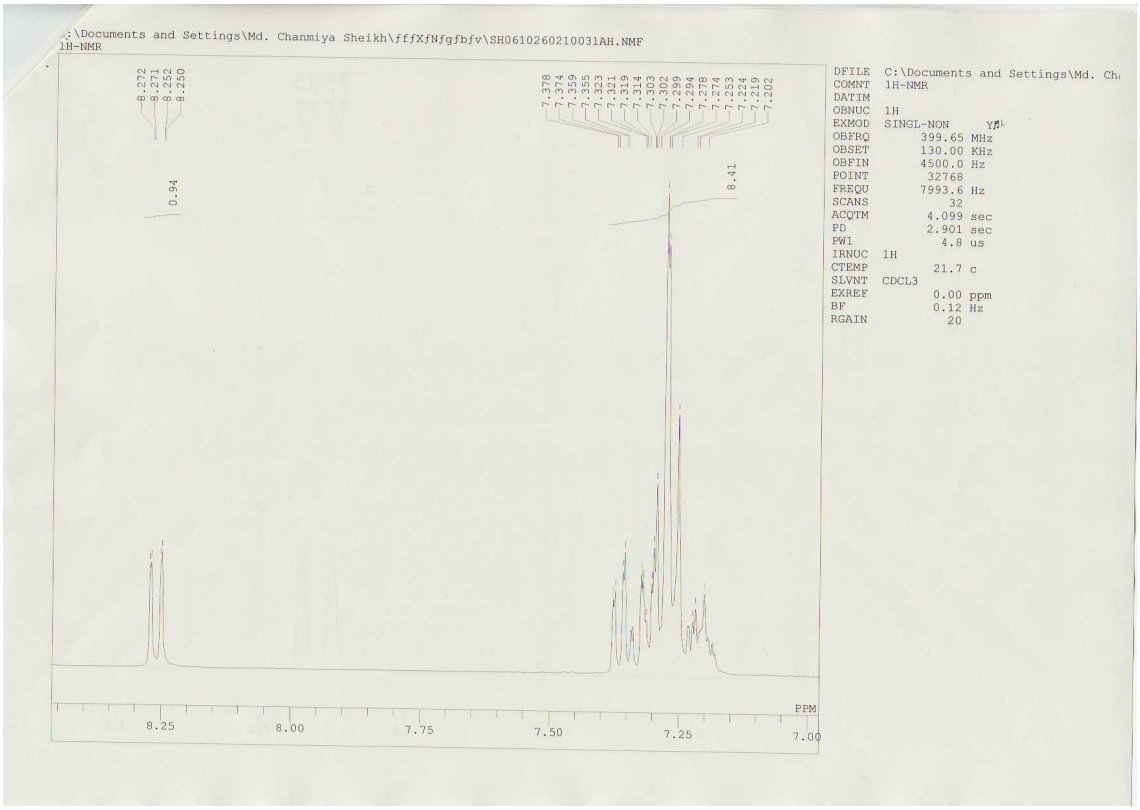
1H-NMR
3.470
3.452
3.432
3.109
3.090
3.072
2.03
2.00
PPM
3.6
3.5
3.4
3.3
3.2
3.1
3.0
DFILE C:\Documents and Settings\Md. Ch
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL-NON
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 32
ACQTM 4.099 sec
PD 2.901 sec
PW1 4.8 us
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20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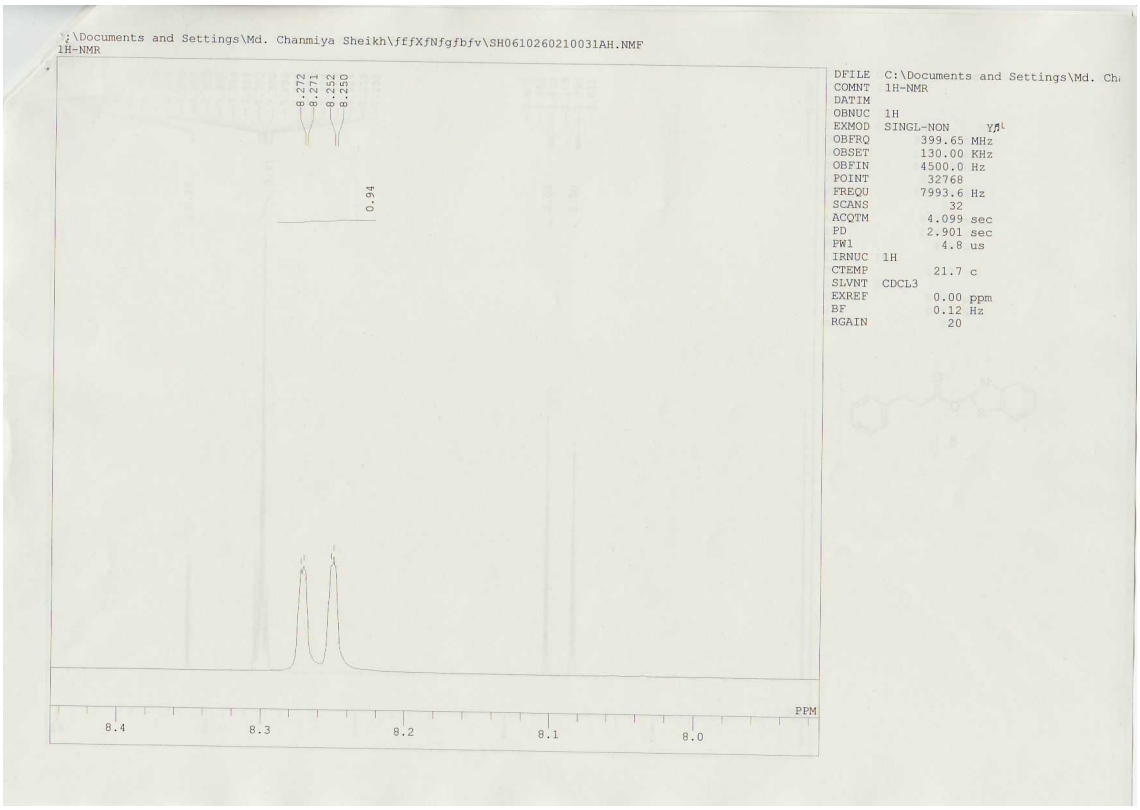
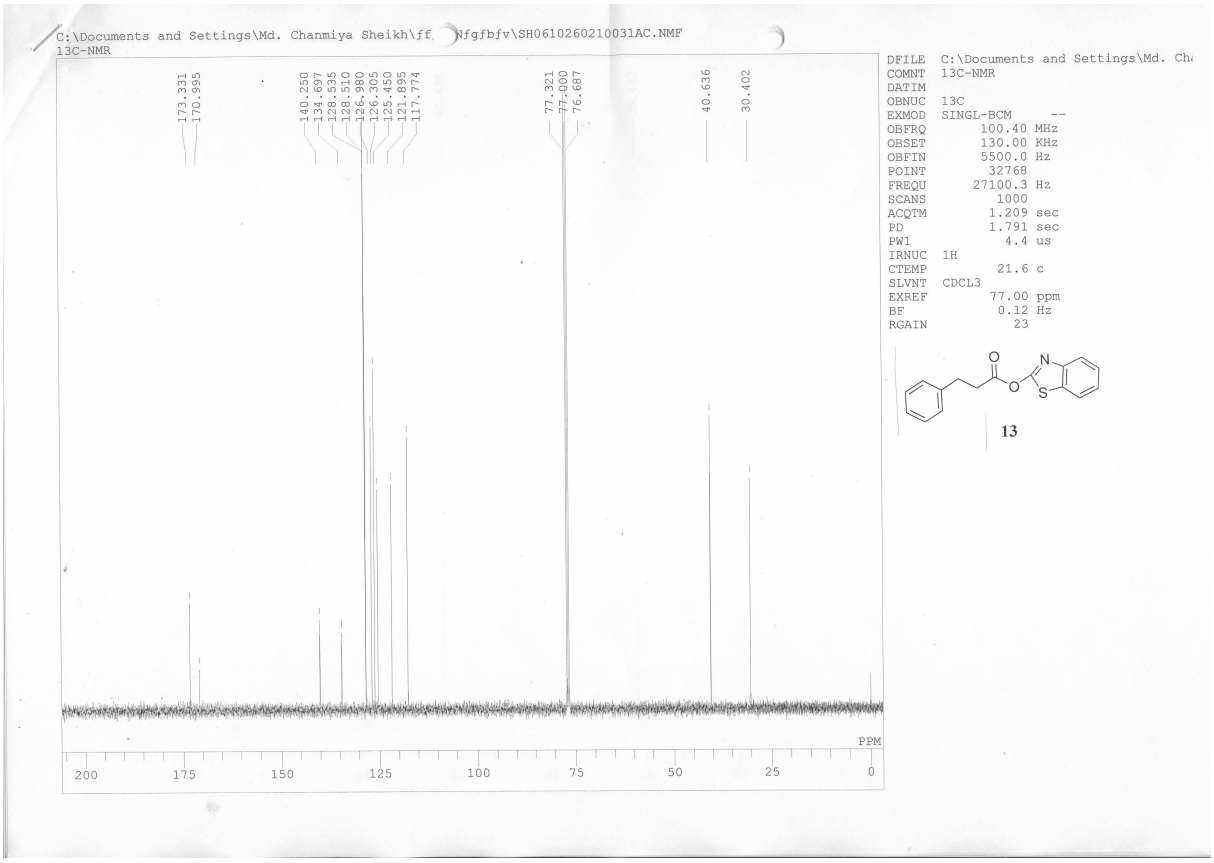
1H-NMR
8.272
8.271
8.252
8.250
7.378
7.374
7.359
7.355
7.323
7.321
7.319
7.314
7.303
7.302
7.299
7.294
7.278
7.274
7.253
7.234
7.219
7.202
0.94
8.41
PPM
8.25
8.00
7.75
7.50
7.25
7.00
DFILE C:\Documents and Settings\Md. Ch
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL-NON
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 32
ACQTM 4.099 sec
PD 2.901 sec
PW1 4.8 us
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20



3-Phenylpropionyloxy)benzothiazole (**13**) (**^{13}C-NMR)**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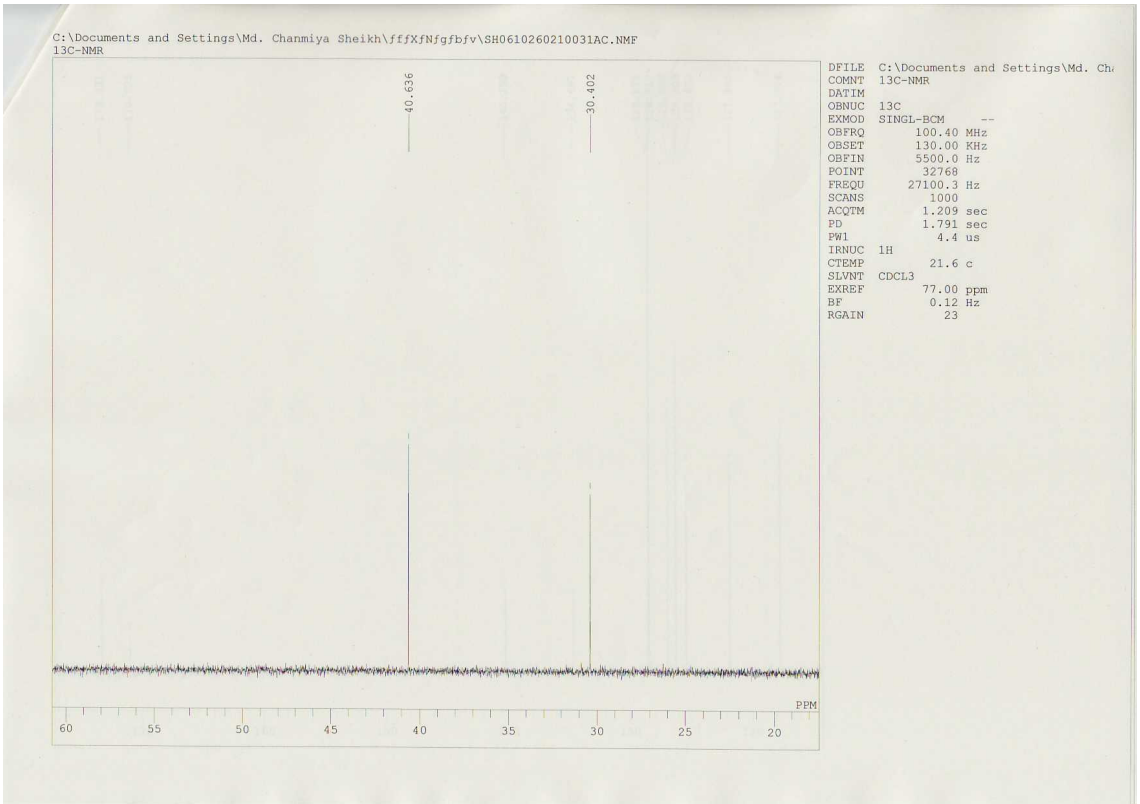
13C-NMR
173.331
170.995
140.250
134.697
128.535
128.510
126.980
126.305
125.450
121.895
117.774
77.321
77.000
76.687
40.636
30.402
PPM
200
175
150
125
100
75
50
25
0
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM --
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
O
N
O
S
13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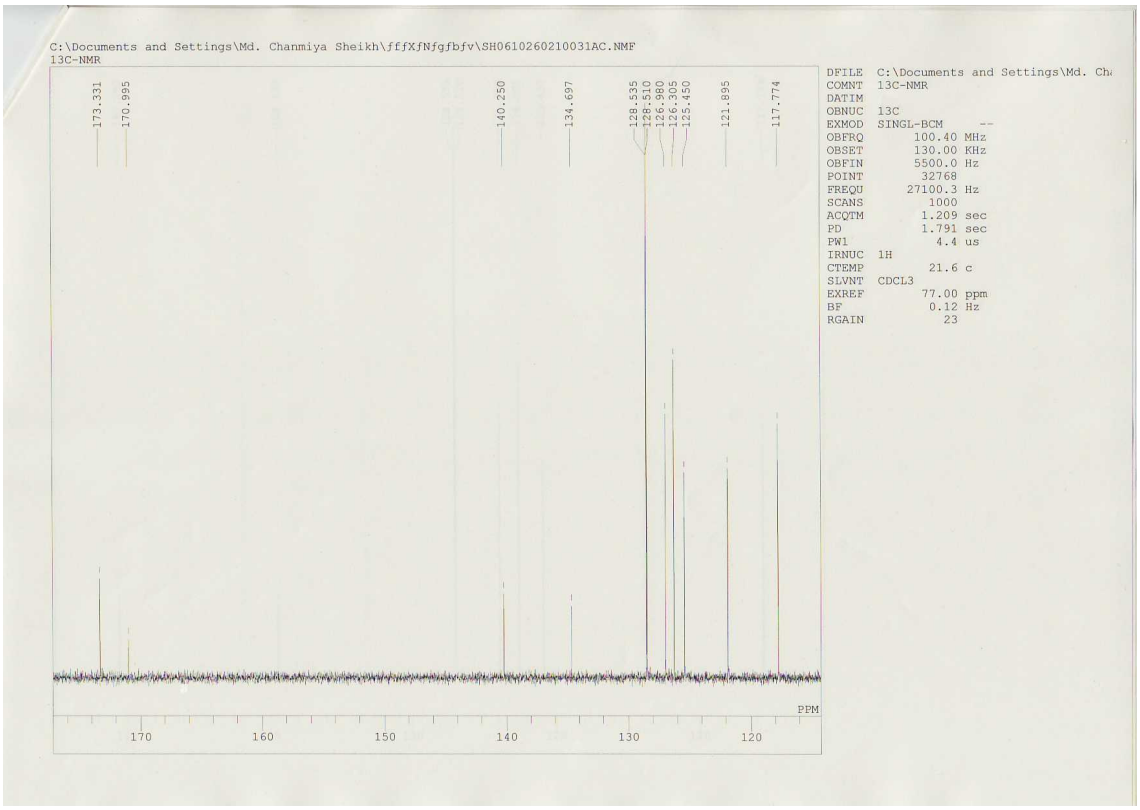
13C-NMR
40.636
30.402
PPM
60
55
50
45
40
35
30
25
20
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM --
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23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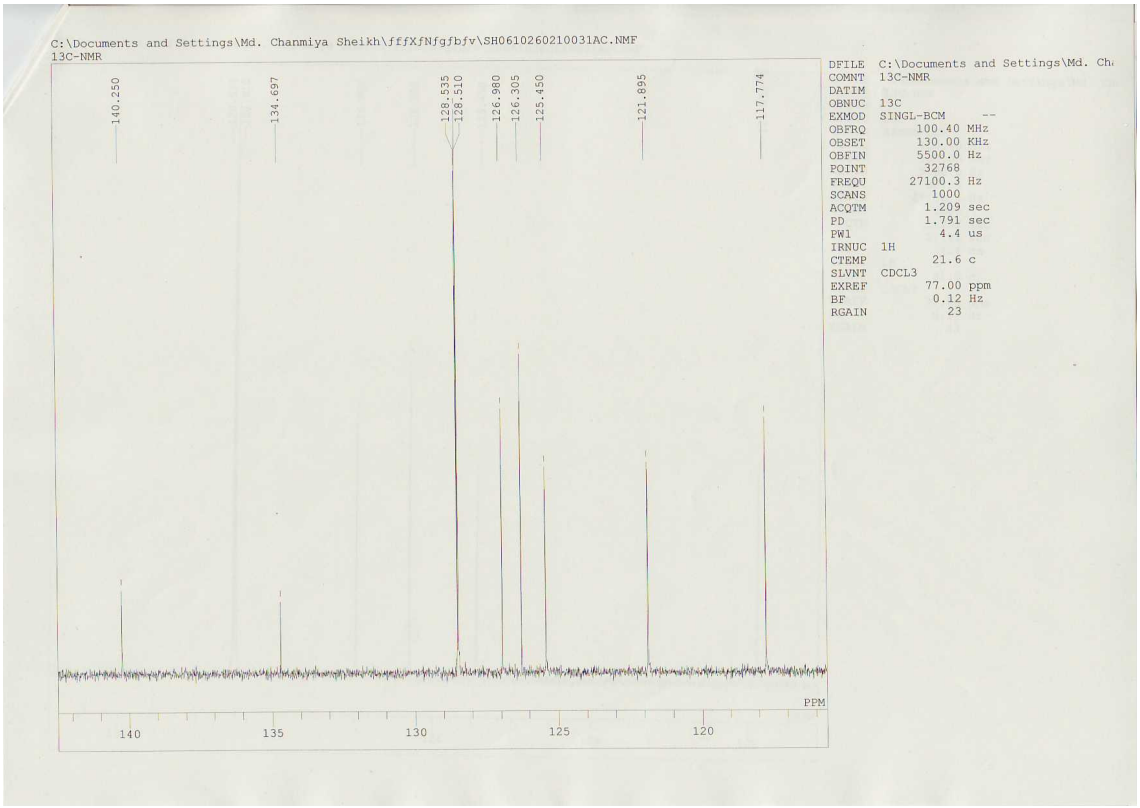
13C-NMR
173.331
170.995
140.250
134.697
128.535
128.510
126.980
126.305
125.450
121.895
117.774
PPM
170
160
150
140
130
120
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM --
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23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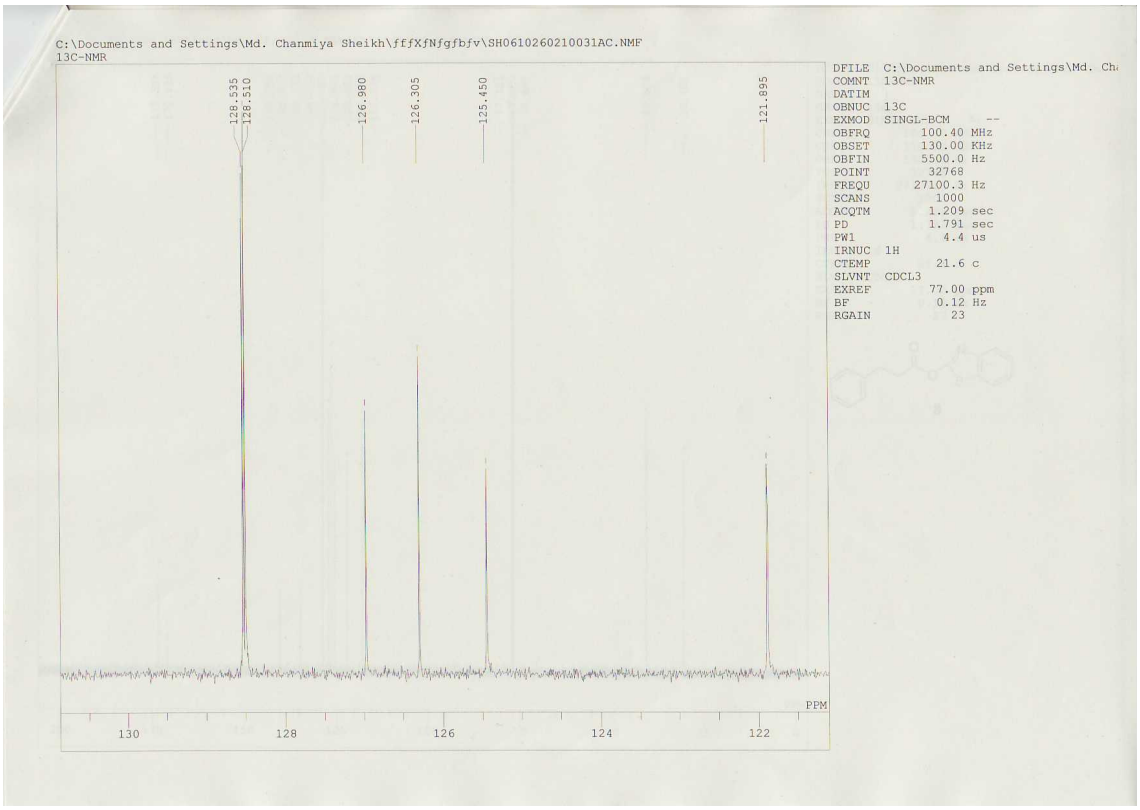
13C-NMR
140.250
134.697
128.535
128.510
126.980
126.305
125.450
121.895
117.774
PPM
140
135
130
125
120
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM --
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23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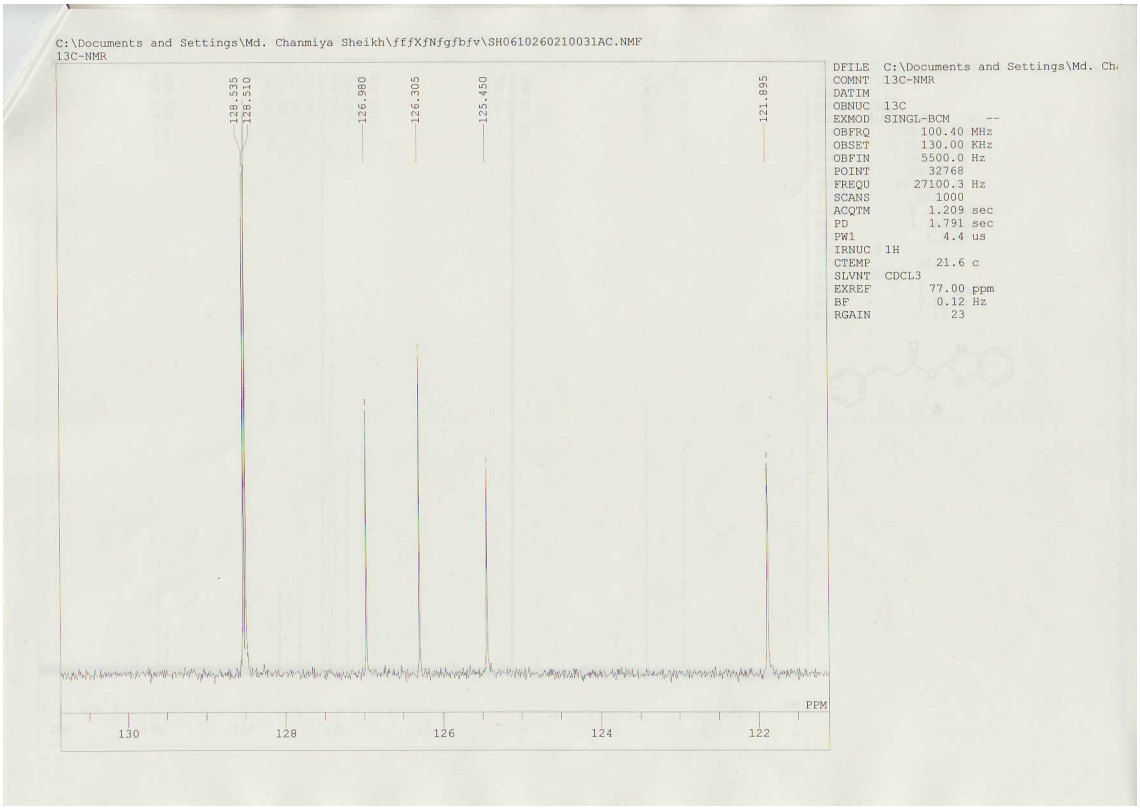
13C-NMR
128.535
128.510
126.980
126.305
125.450
121.895
PPM
130
128
126
124
122
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM --
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23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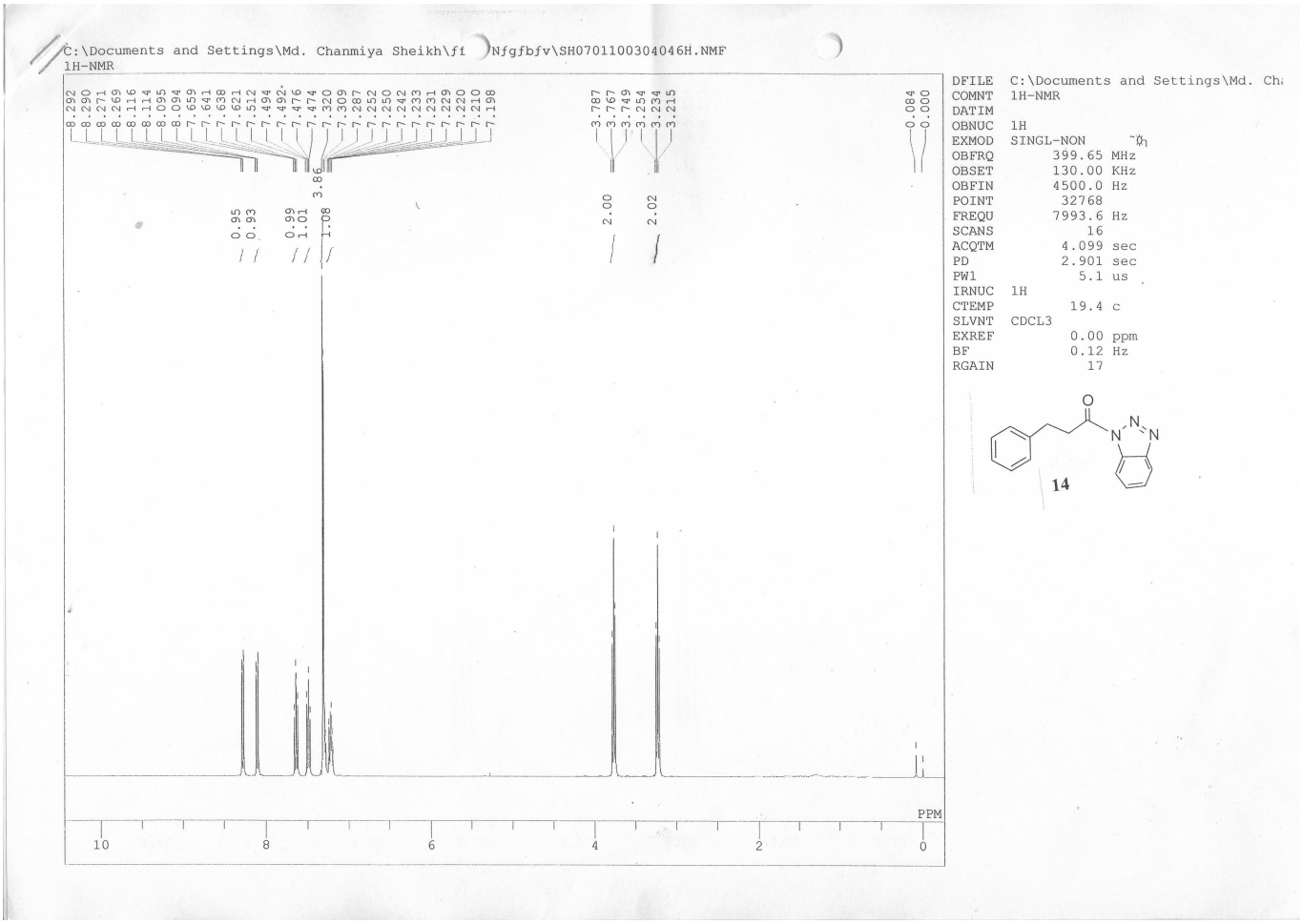
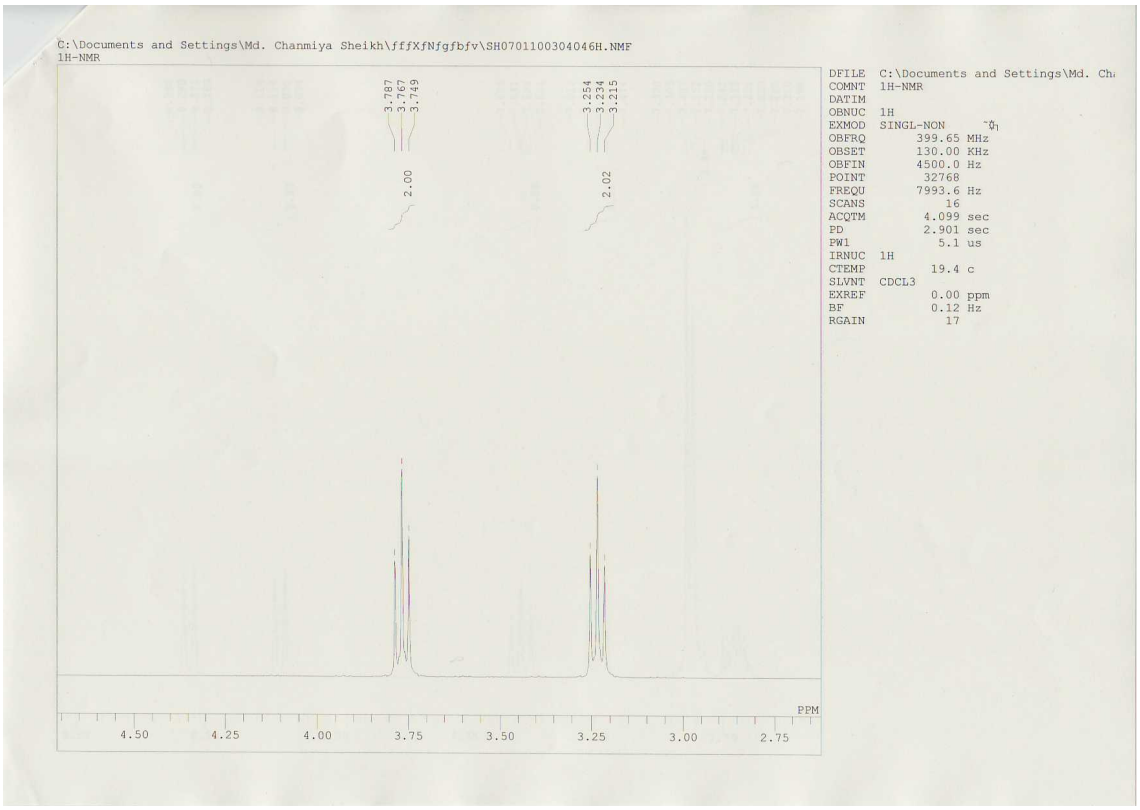
13C-NMR
128.535
128.510
126.980
126.305
125.450
121.895
PPM
130
128
126
124
122
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM --
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

N-(3-Phenylpropionyl)benzotriazole (**14**) (**^{1}H-NMR)**




C:\Documents and Settings\Md. Chanmiya Sheikh\fffXfNfgfbfv\SH0701100304046H.NMF
1H-NMR
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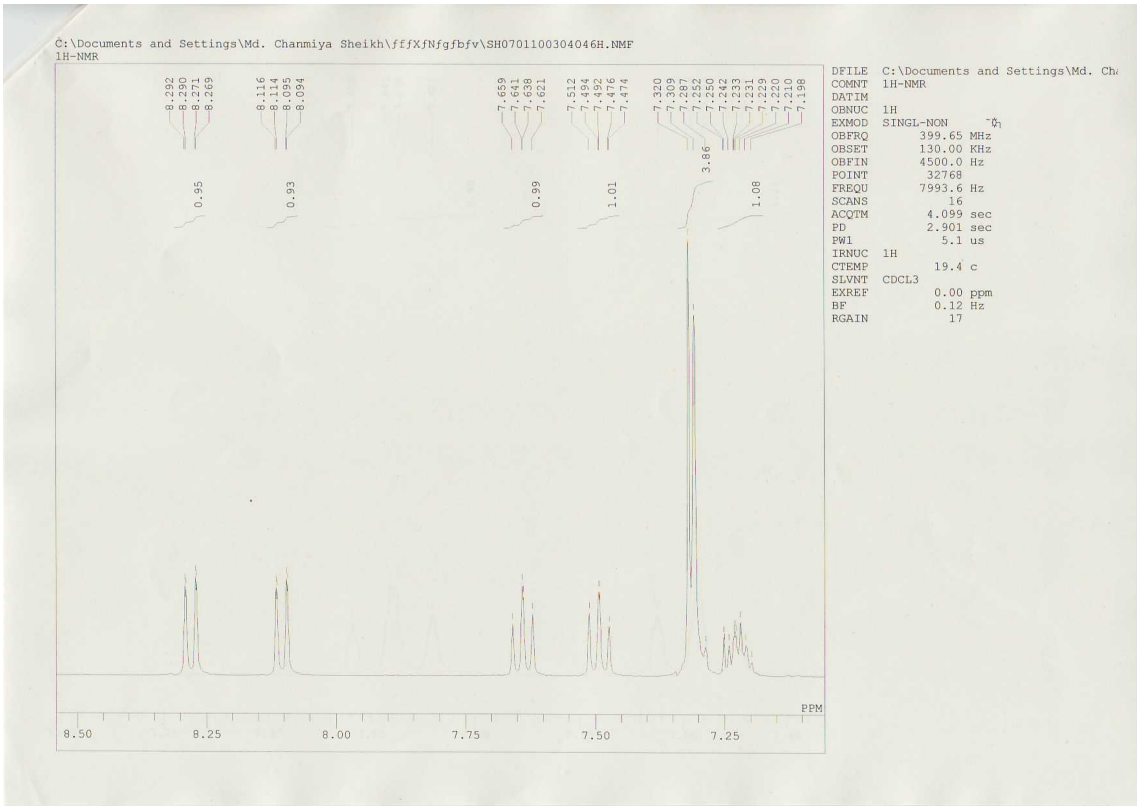
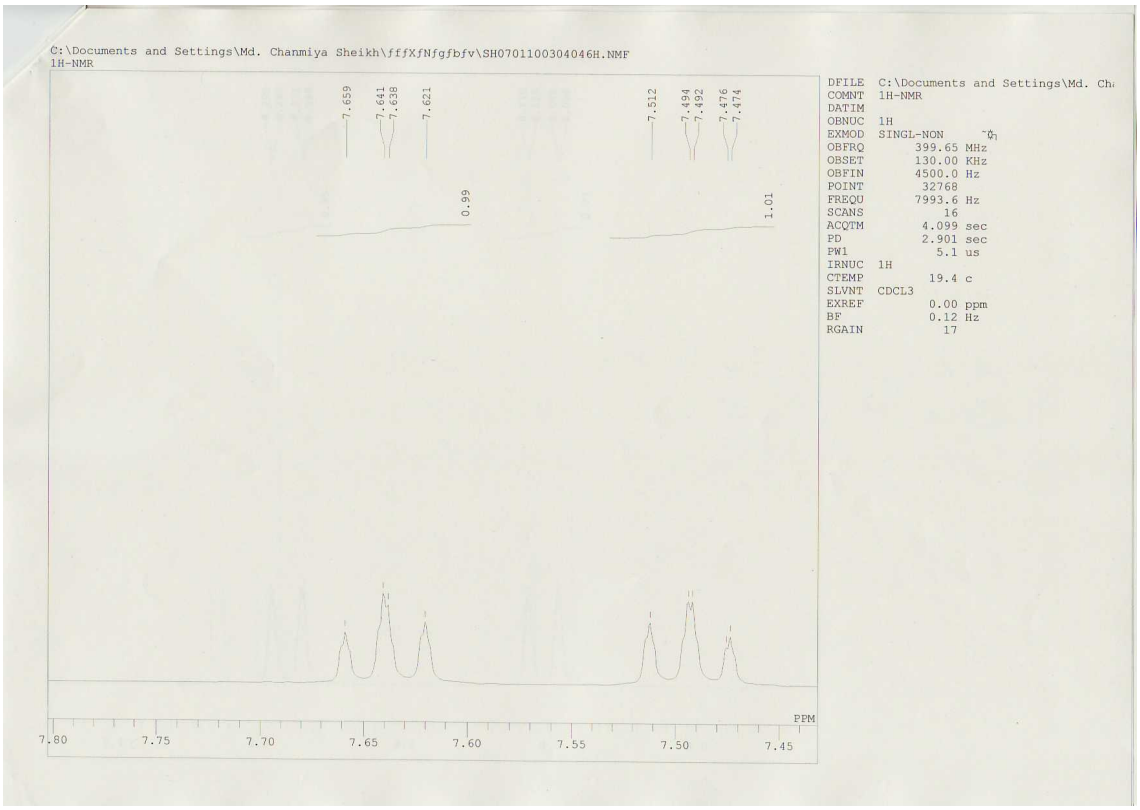
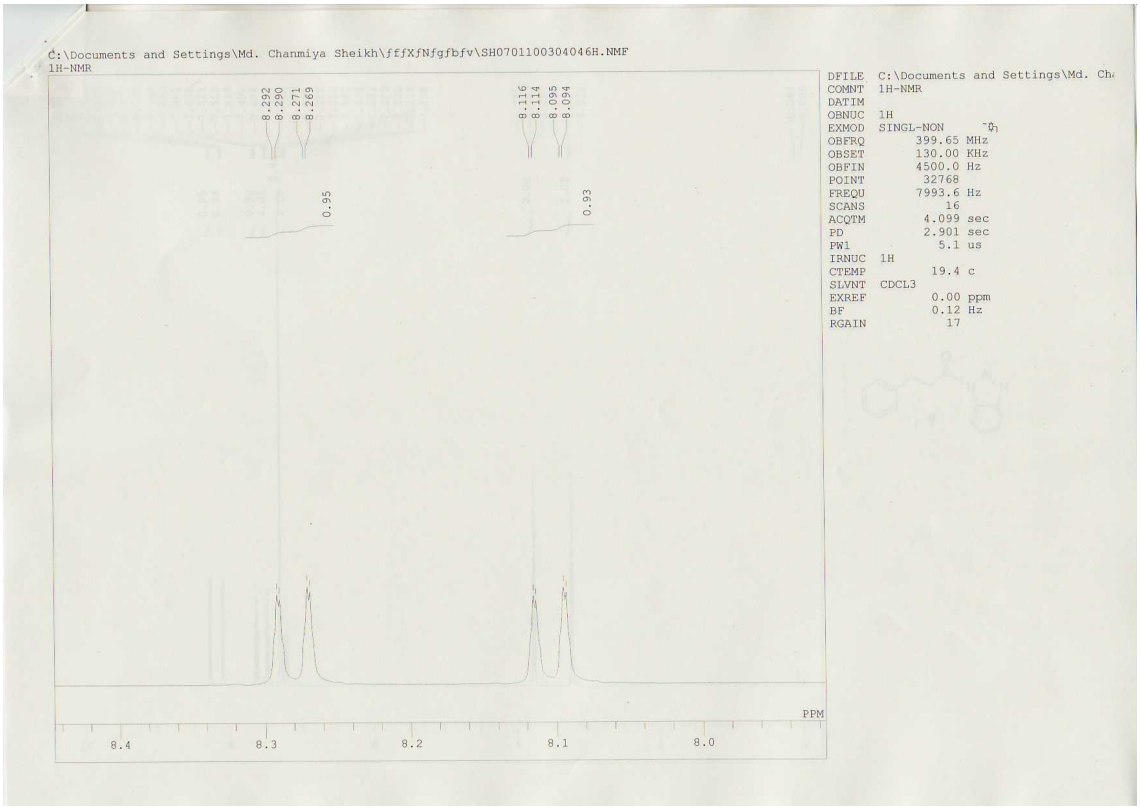
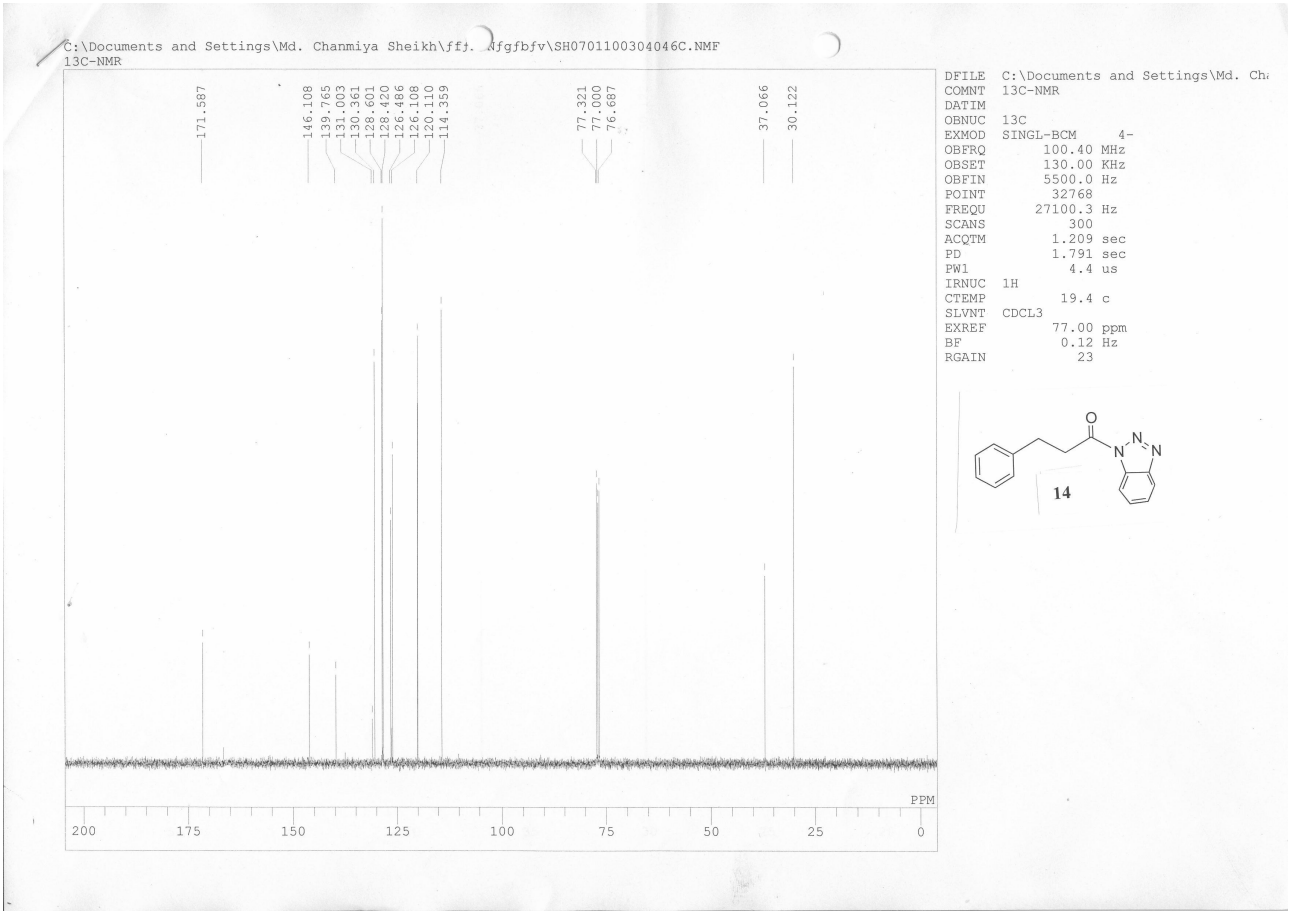
3.767
3.749
3.254
3.234
3.215
2.00
2.02
PPM
4.50
4.25
4.00
3.75
3.50
3.25
3.00
2.75
DFILE C:\Documents and Settings\Md. Ch
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL-NON
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 16
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.1 us
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

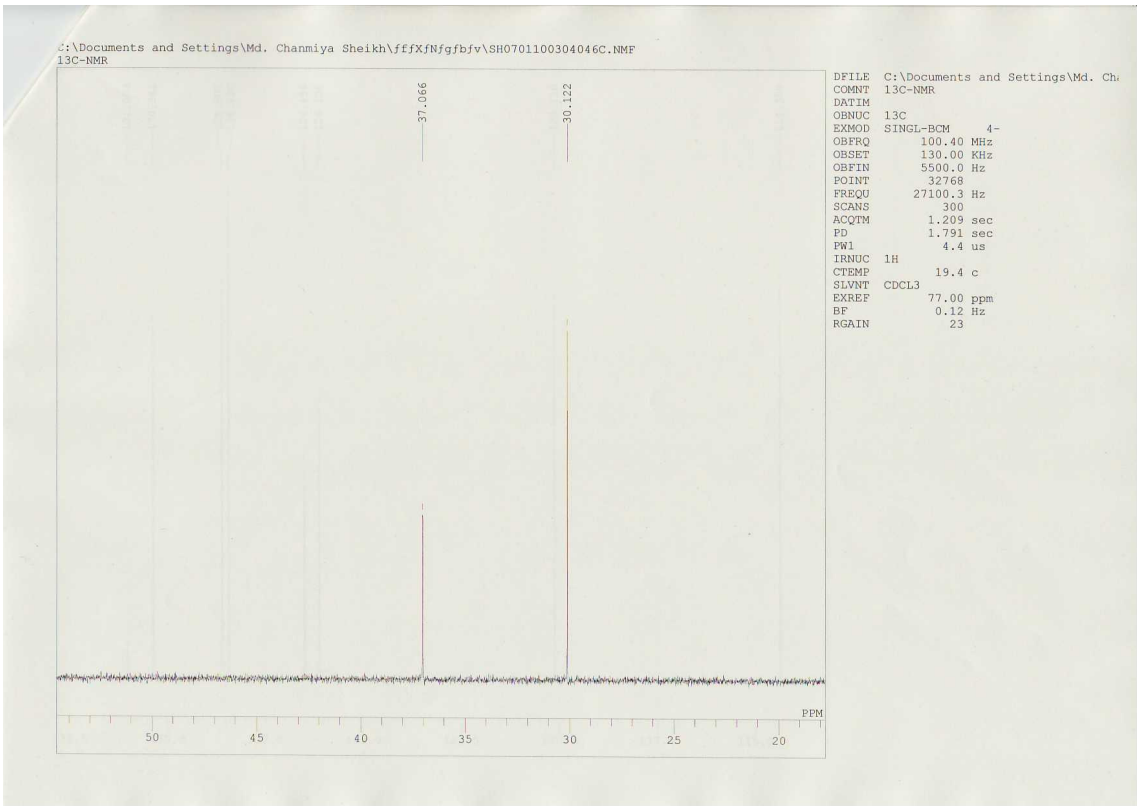

C:\Documents and Settings\Md. Chanmiya Sheikh\fffXfNfgfbfv\SH0701100304046H.NMF
1H-NMR
8.292
8.290
8.271
8.269
8.116
8.114
8.095
8.094
7.659
7.641
7.638
7.621
7.512
7.494
7.492
7.476
7.474
7.320
7.309
7.287
7.252
7.250
7.242
7.233
7.231
7.229
7.220
7.210
7.198
0.95
0.93
0.99
1.01
3.86
1.08
PPM
8.50
8.25
8.00
7.75
7.50
7.25
DFILE C:\Documents and Settings\Md. Ch
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL-NON
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 16
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.1 us
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

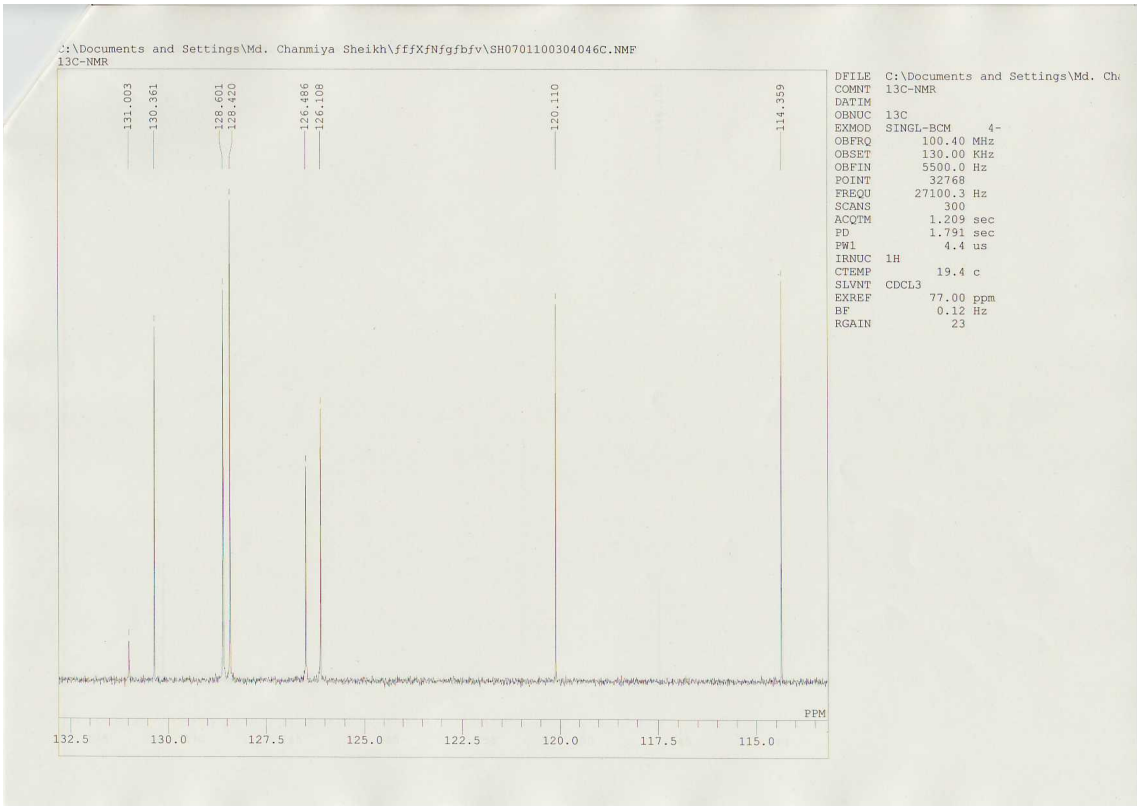

C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SH0701100304046H.NMF
1H-NMR
7.659
7.641
7.638
7.621
7.512
7.494
7.492
7.476
7.474
0.99
1.01
PPM
7.80
7.75
7.70
7.65
7.60
7.55
7.50
7.45
DFILE C:\Documents and Settings\Md. Ch
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL-NON
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 16
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.1 us
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

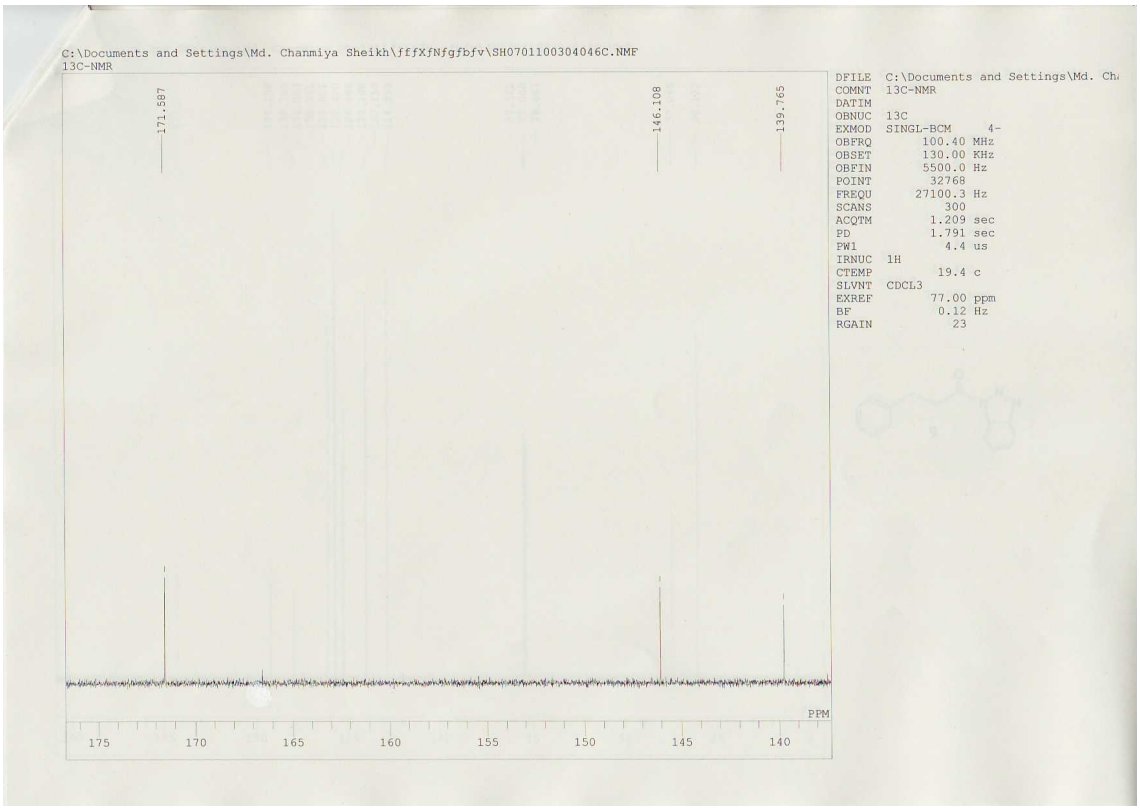

C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SH0701100304046H.NMF
1H-NMR
8.292
8.290
8.271
8.269
8.116
8.114
8.095
8.094
0.95
0.93
PPM
8.4
8.3
8.2
8.1
8.0
DFILE C:\Documents and Settings\Md. Ch
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL-NON
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 16
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.1 us
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

N-(3-Phenylpropionyl)benzotriazole (**14**) (**^{13}C-NMR)**

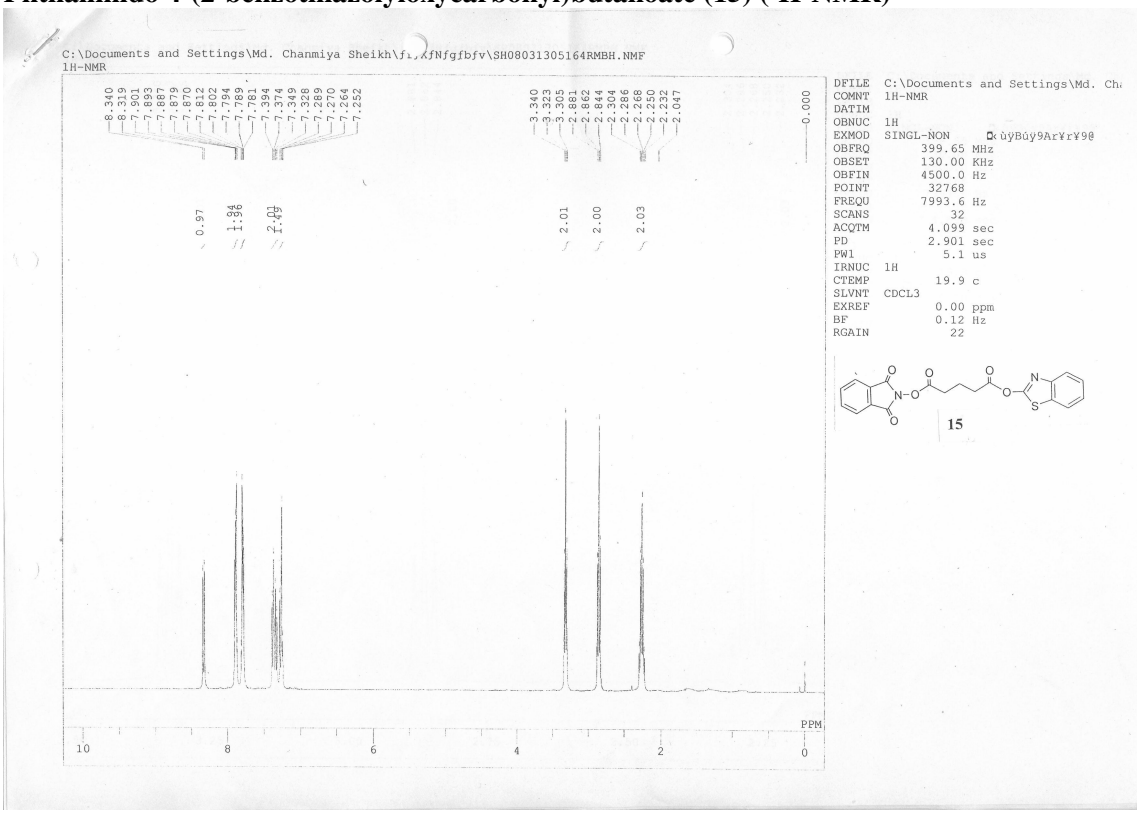


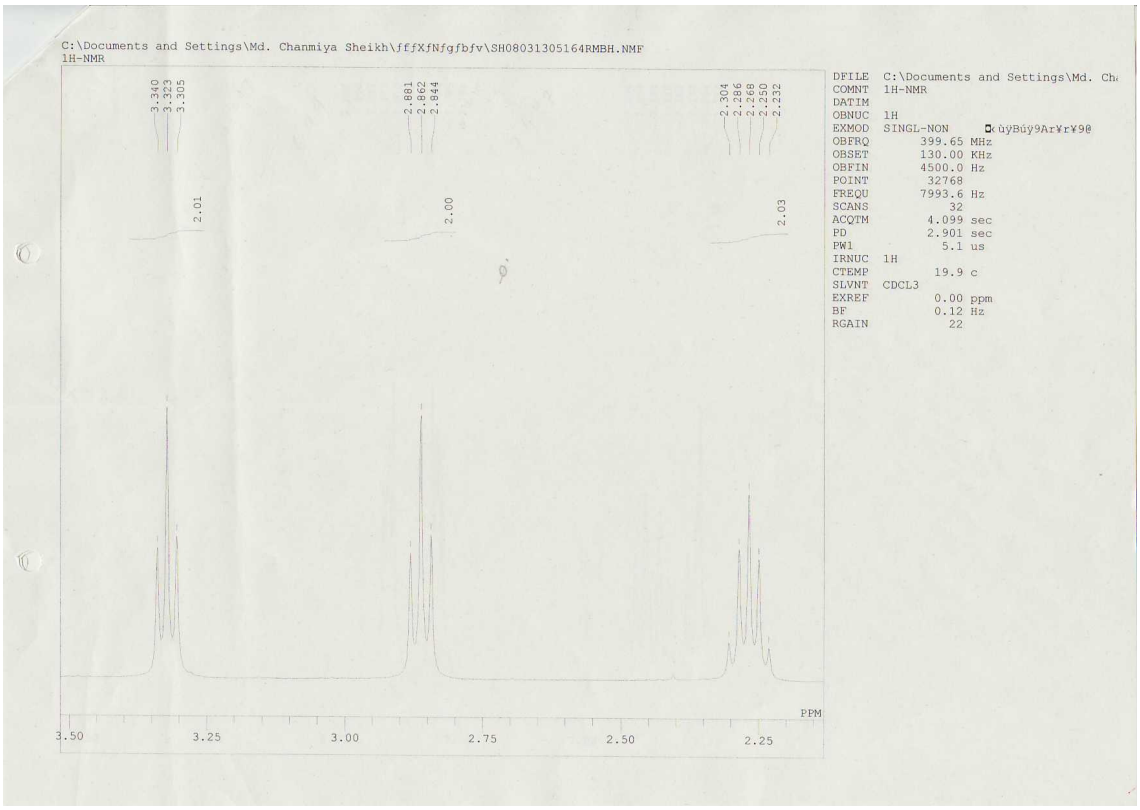

C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SH0701100304046C.NMF
13C-NMR
37.066
30.122
PPM
50
45
40
35
30
25
20
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM 4-
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 300
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

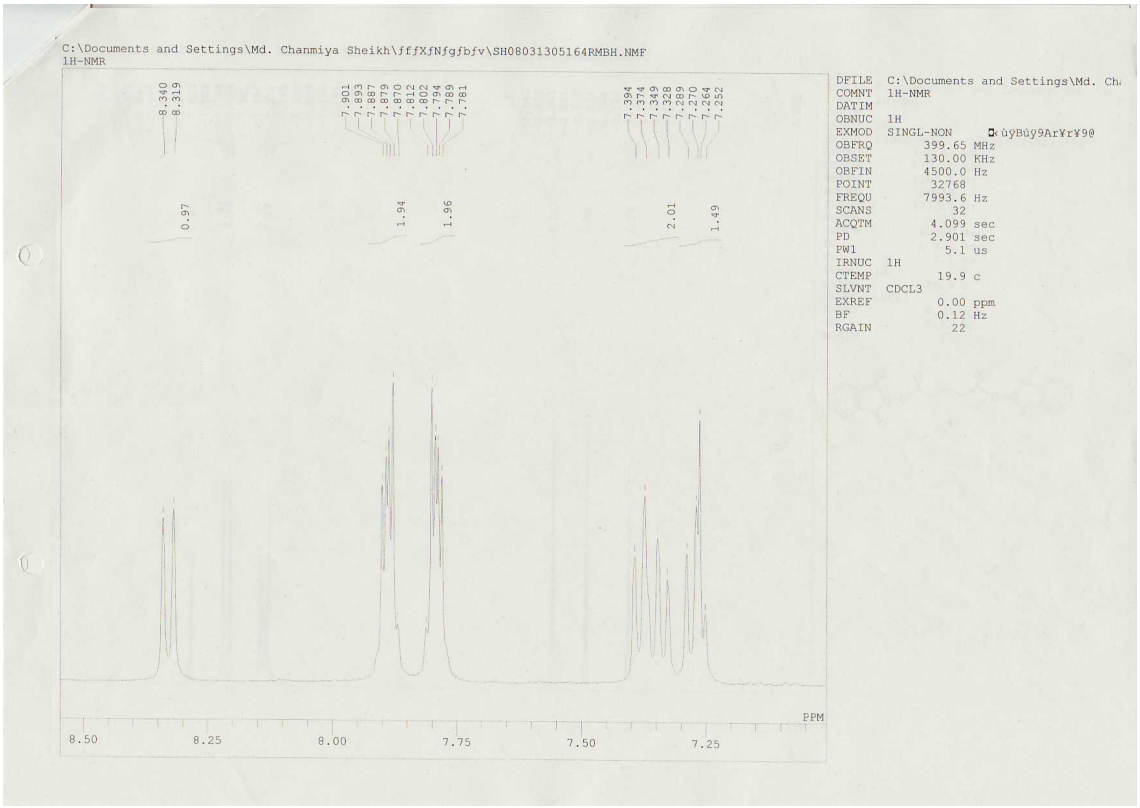

C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SH0701100304046C.NMF
13C-NMR
131.003
130.361
128.601
128.420
126.486
126.108
120.110
114.359
PPM
132.5
130.0
127.5
125.0
122.5
120.0
117.5
115.0
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM 4-
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 300
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23



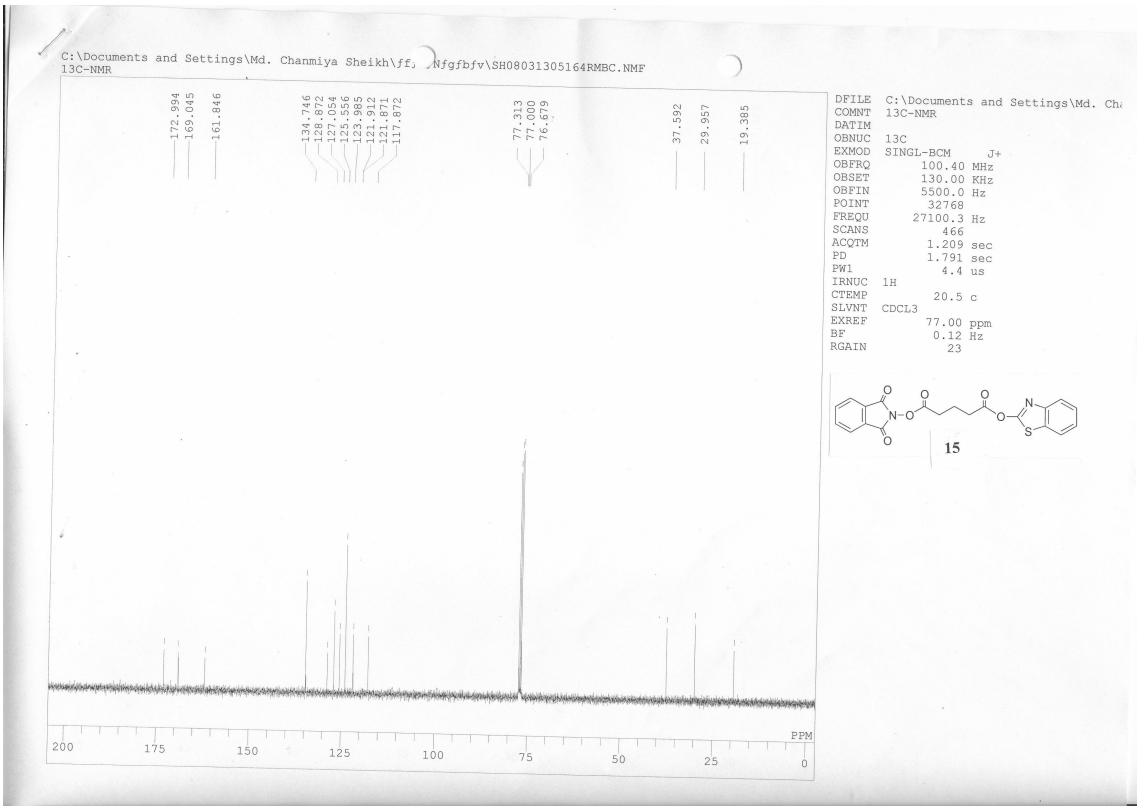
Phthalimido 4-(2-benzothiazolyloxycarbonyl)butanoate (15) (^{1}H-NMR)

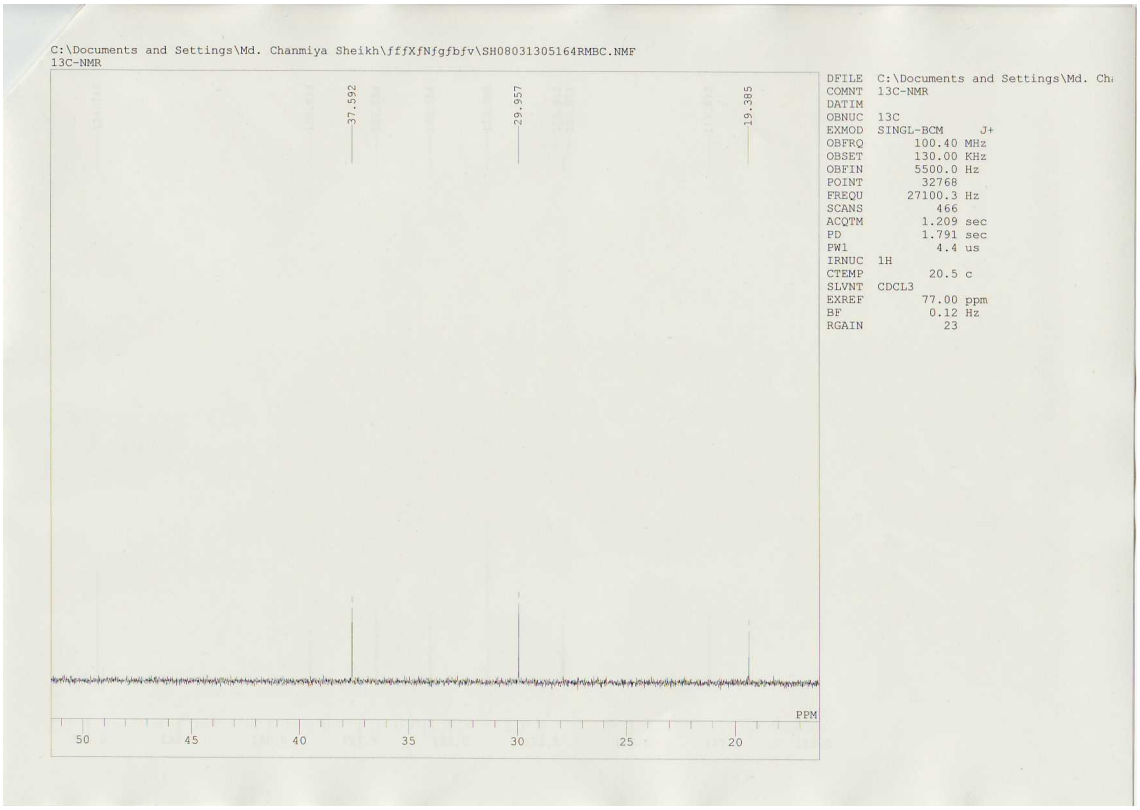


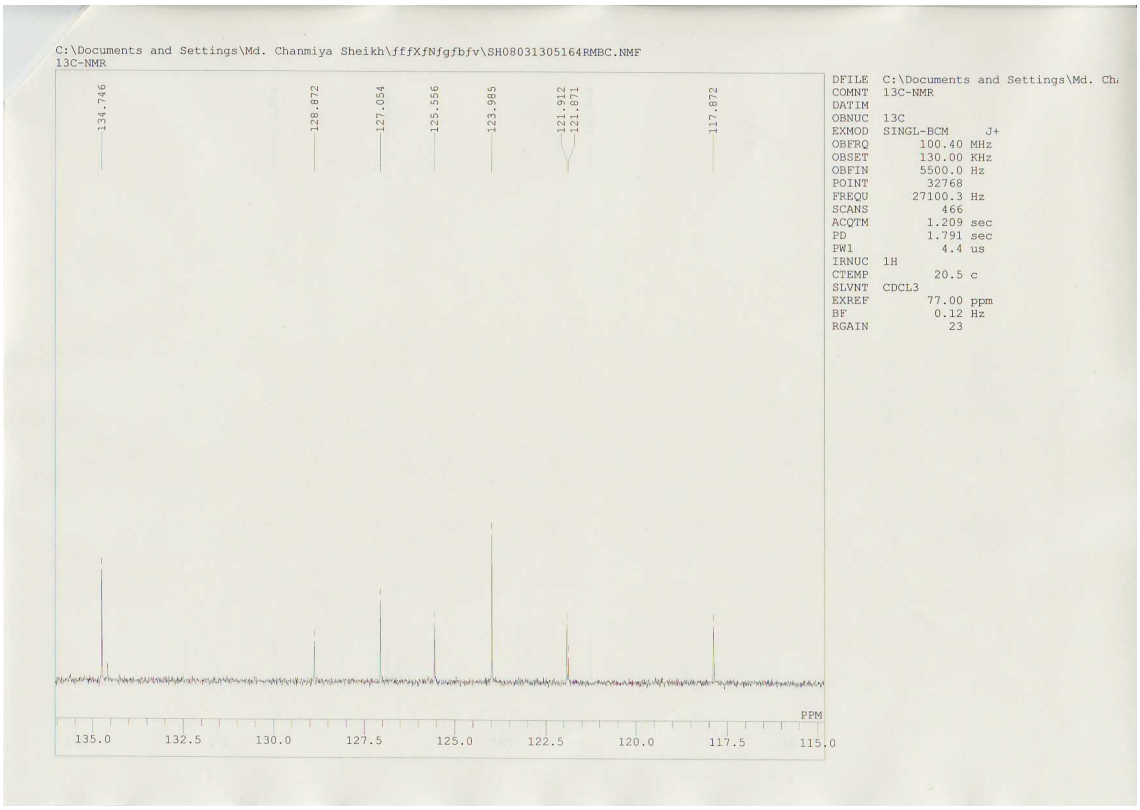


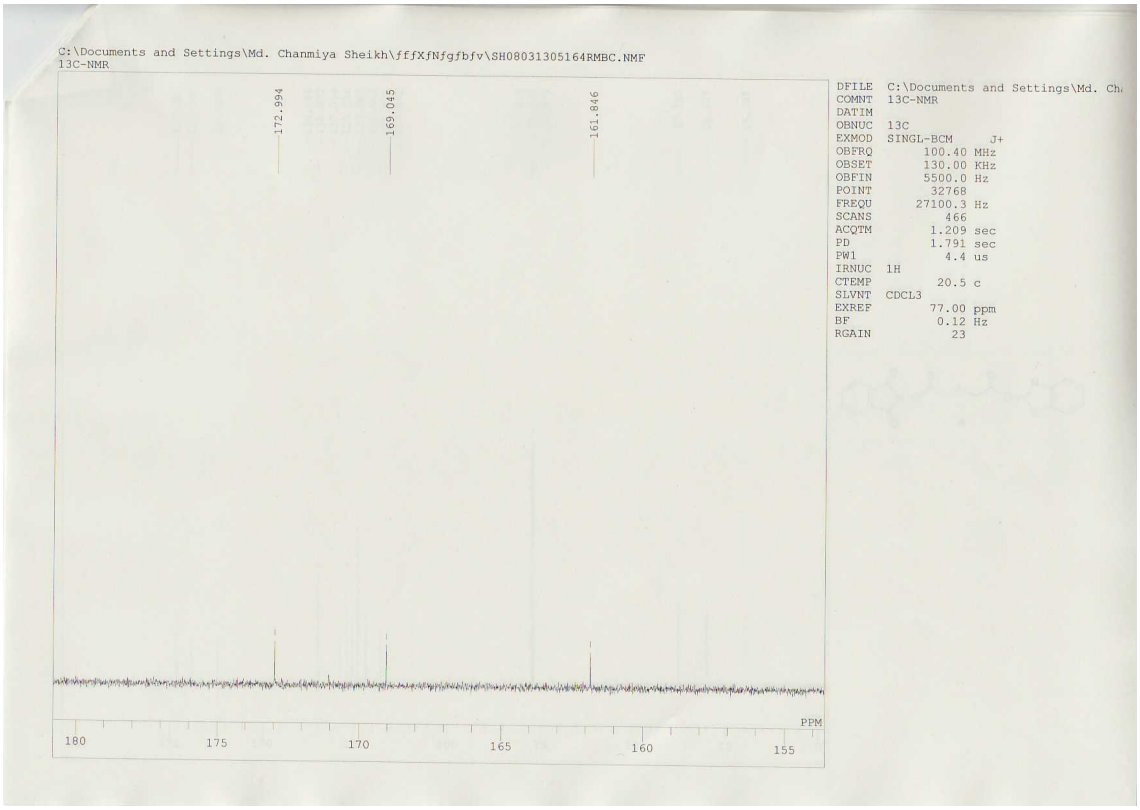


Phthalimido 4-(2-benzothiazolyloxycarbonyl)butanoate (15) (^{13}C-NMR)

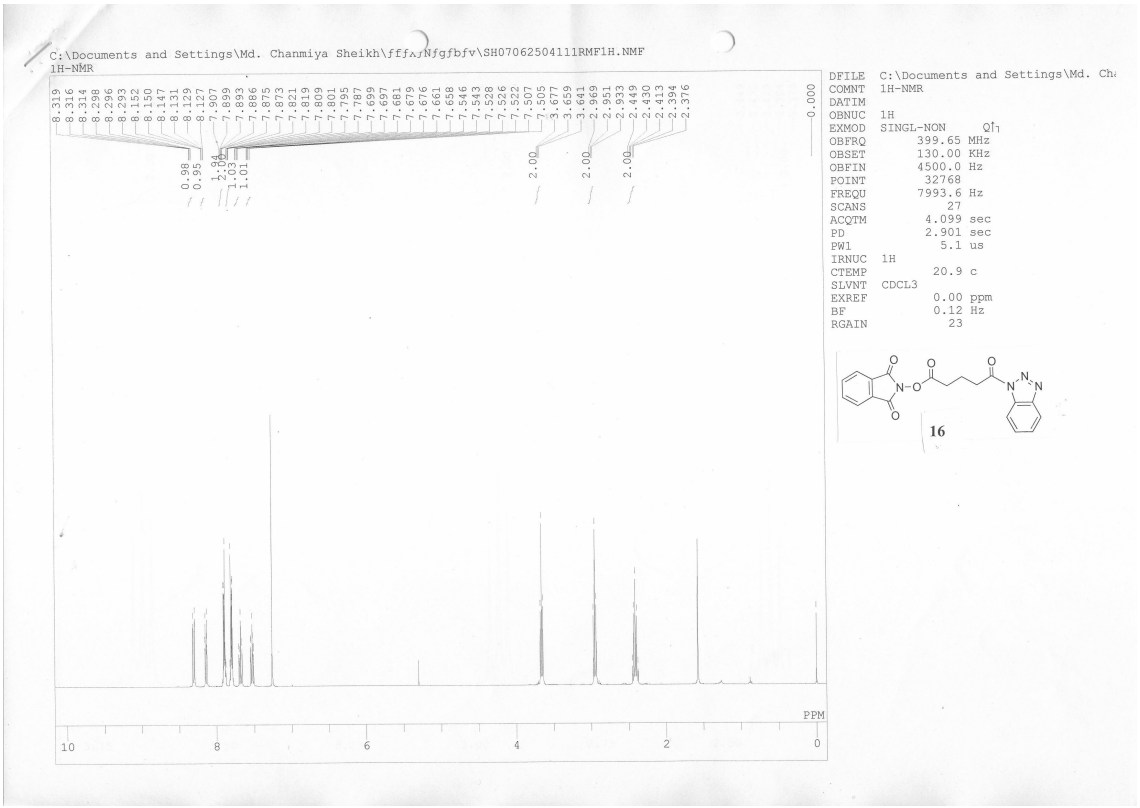

C:\Documents and Settings\Md. Chanmiya Sheikh\ff, Nfgfbfv\SH08031305164RMBC.NMF
13C-NMR
172.994
169.045
161.846
134.746
128.872
127.054
125.556
123.985
121.912
121.871
117.872
77.313
77.000
76.679
37.592
29.957
19.385
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM J+
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 466
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
15
PPM
200
175
150
125
100
75
50
25
0

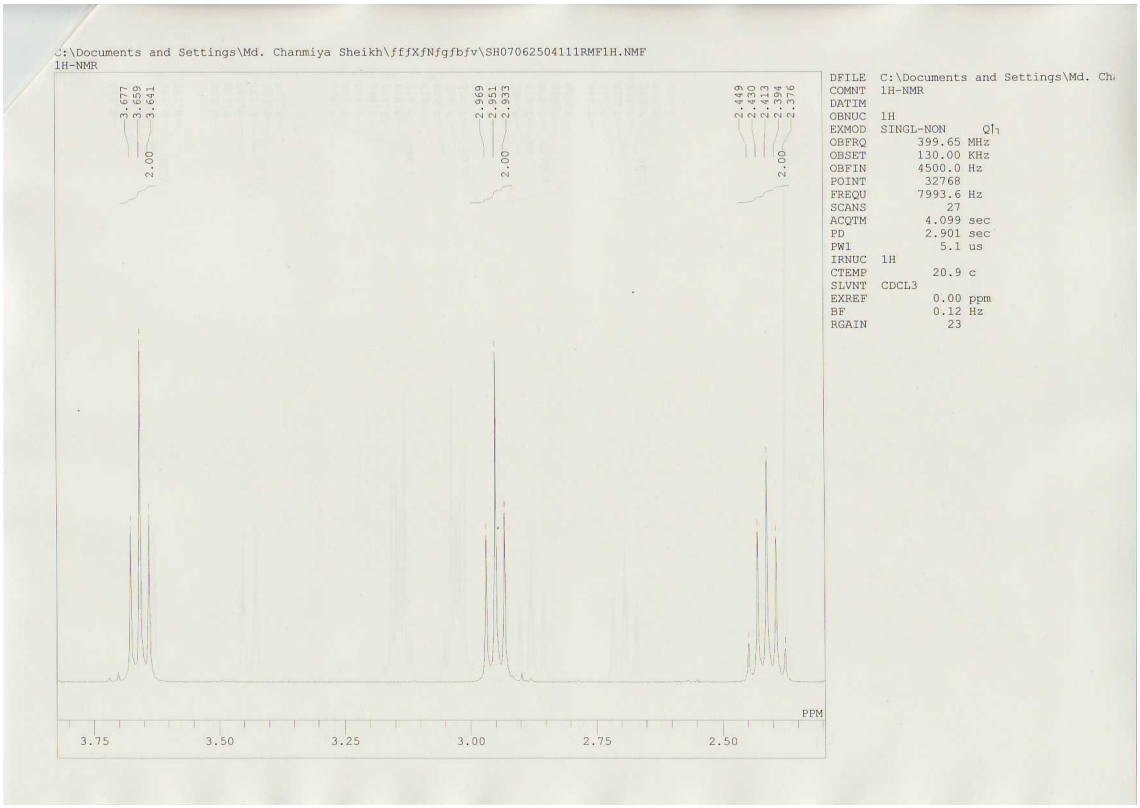

C:\Documents and Settings\Md. Chanmiya Sheikh\fffXfNfgfbfv\SH08031305164RMBC.NMF
13C-NMR
37.592
29.957
19.385
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM J+
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 466
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
PPM
50
45
40
35
30
25
20

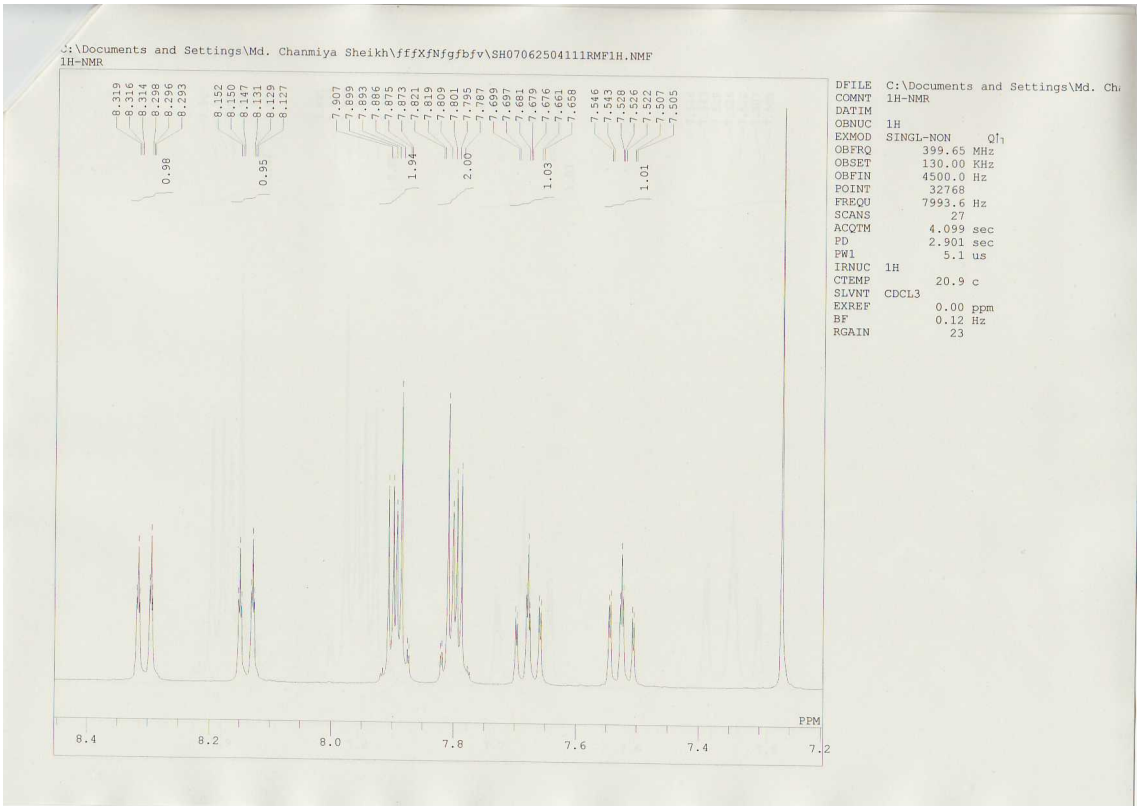

C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SH08031305164RMBC.NMF
13C-NMR
134.746
128.872
127.054
125.556
123.985
121.912
121.871
117.872
PPM
135.0
132.5
130.0
127.5
125.0
122.5
120.0
117.5
115.0
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM J+
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 466
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

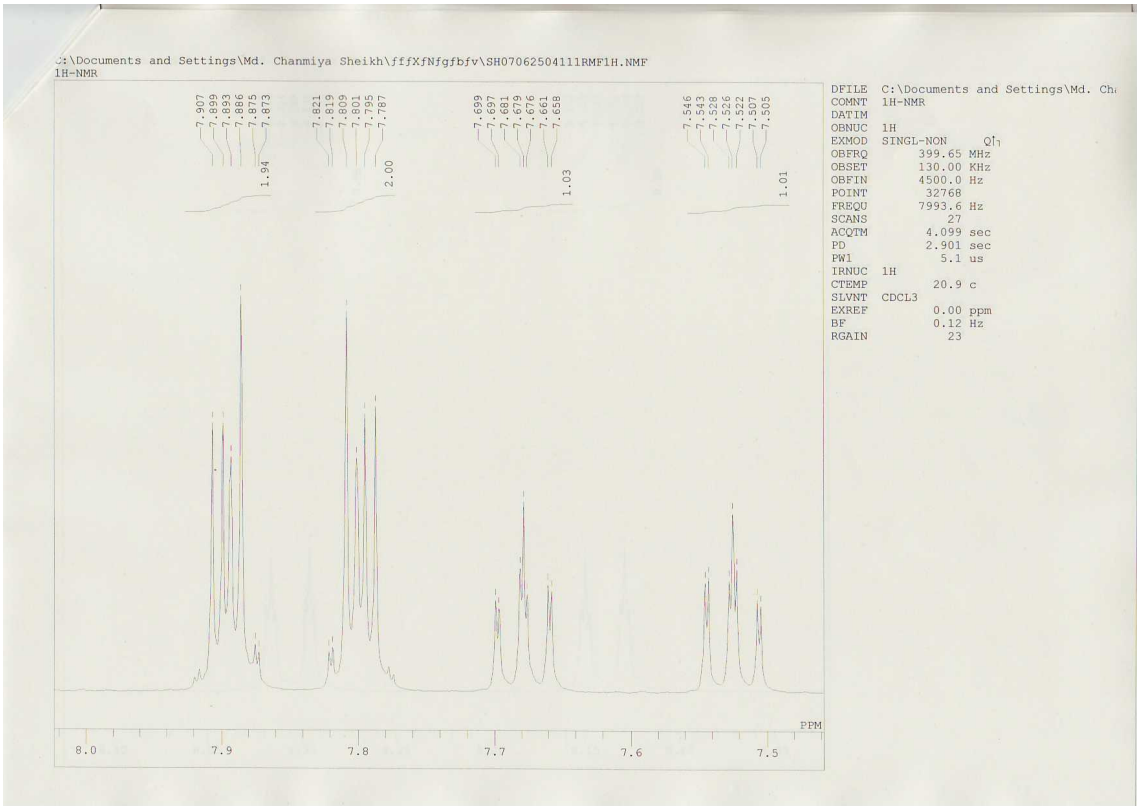

C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SH08031305164RMBC.NMF
13C-NMR
172.994
169.045
161.846
PPM
180
175
170
165
160
155
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM J+
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 466
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

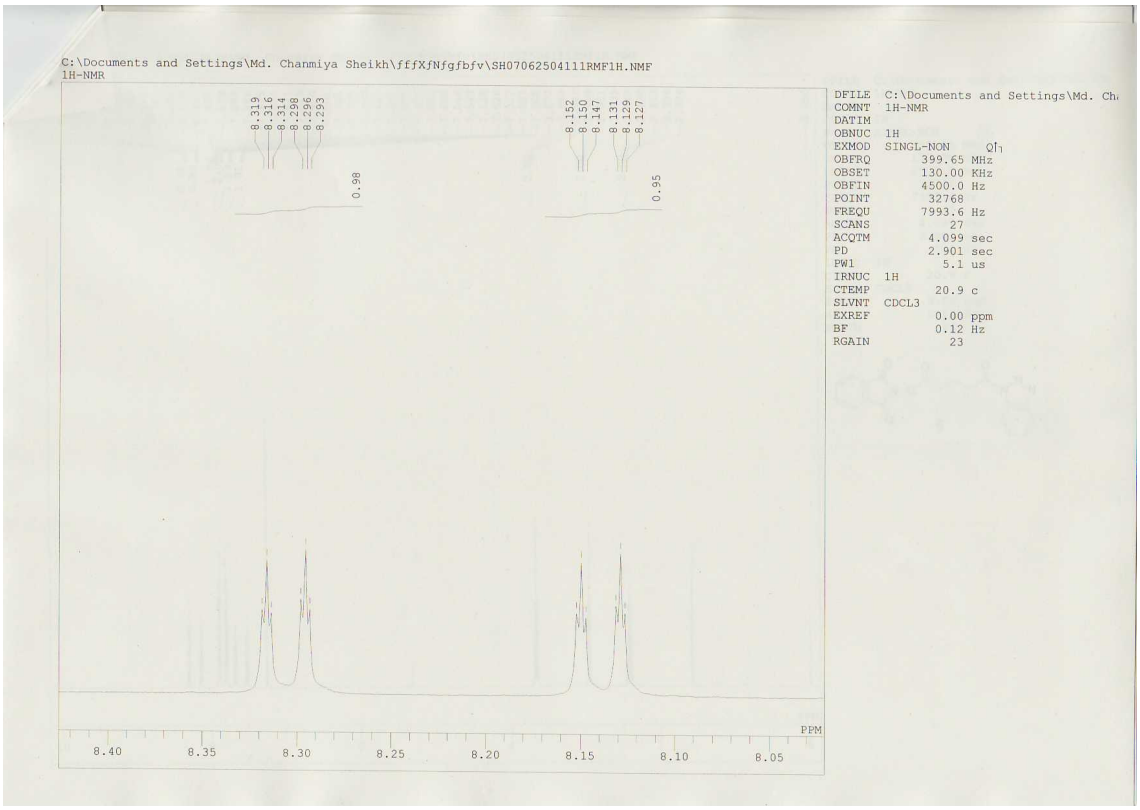
Phthalimido 4-(1-benzotriazolylcarbonyl)butanoate (16) (^{1}H-NMR)



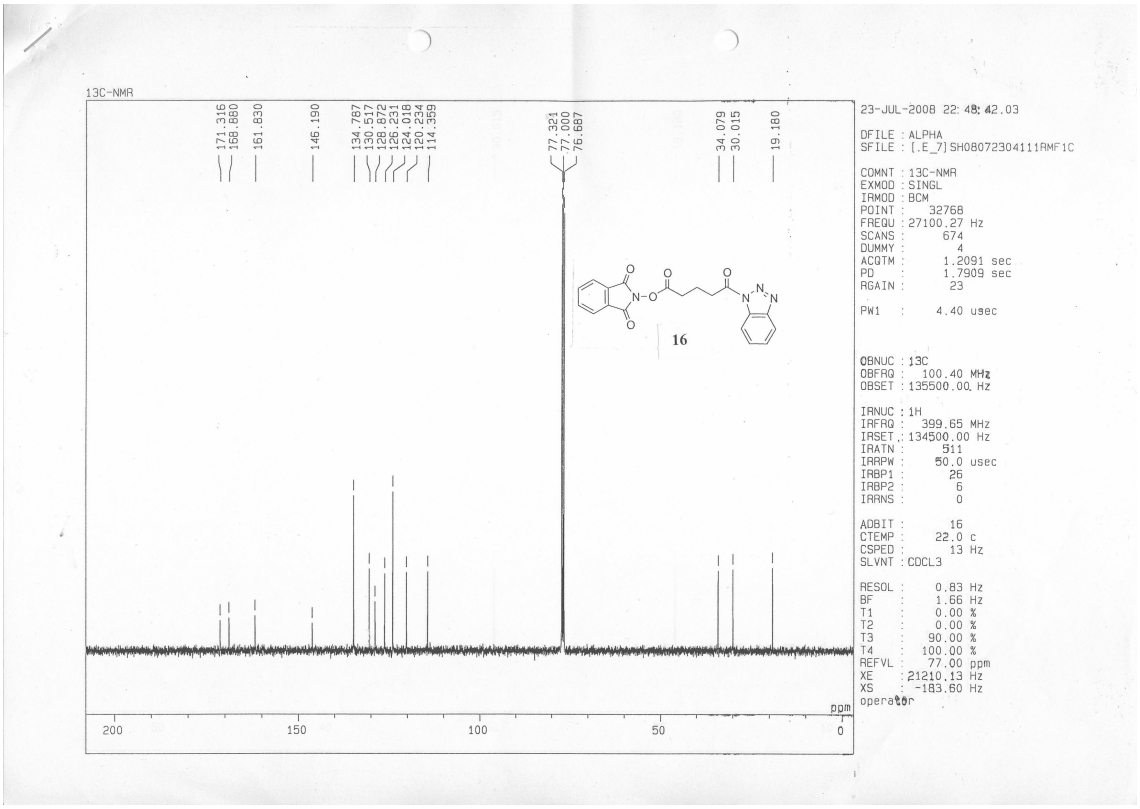


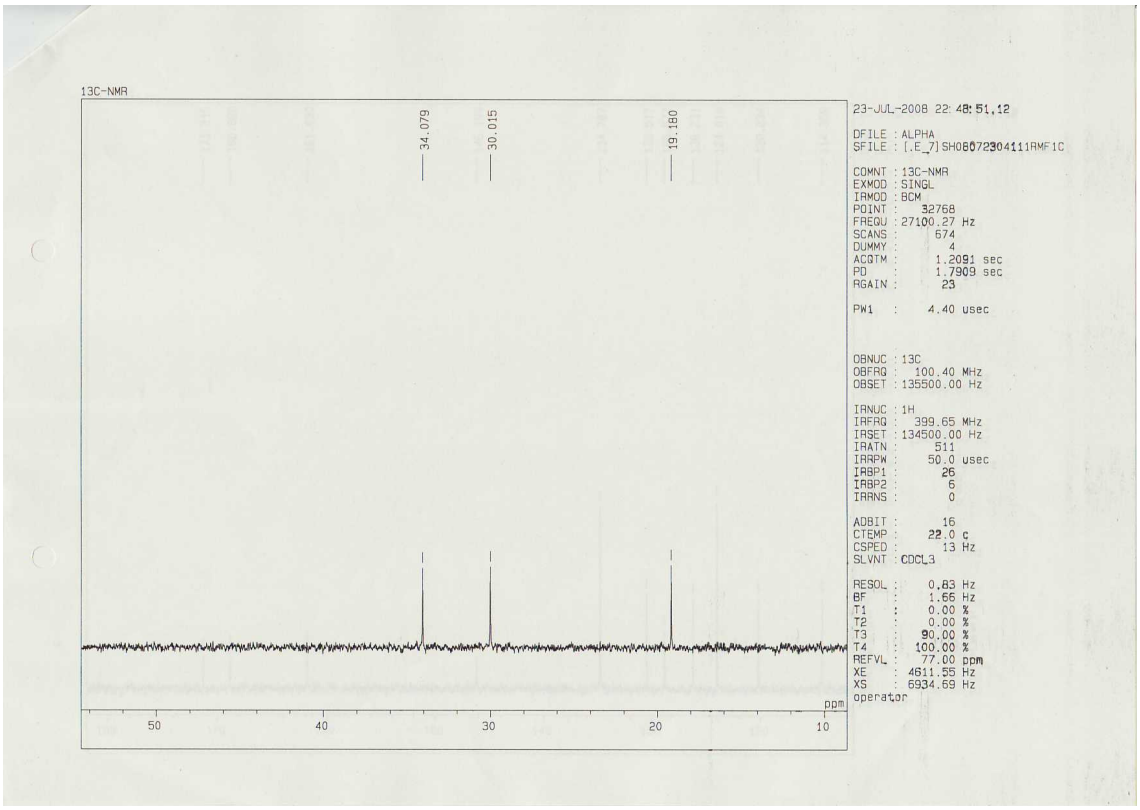

C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SH07062504111RMF1H.NMF
1H-NMR
8.319
8.316
8.314
8.298
8.296
8.293
8.152
8.150
8.147
8.131
8.129
8.127
7.907
7.899
7.893
7.886
7.875
7.873
7.821
7.819
7.809
7.801
7.795
7.787
7.699
7.697
7.681
7.679
7.676
7.661
7.658
7.546
7.543
7.528
7.526
7.522
7.507
7.505
0.98
0.95
1.94
2.00
1.03
1.01
PPM
8.4
8.2
8.0
7.8
7.6
7.4
7.2
DFILE C:\Documents and Settings\Md. Ch
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL-NON
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 27
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.1 us
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23

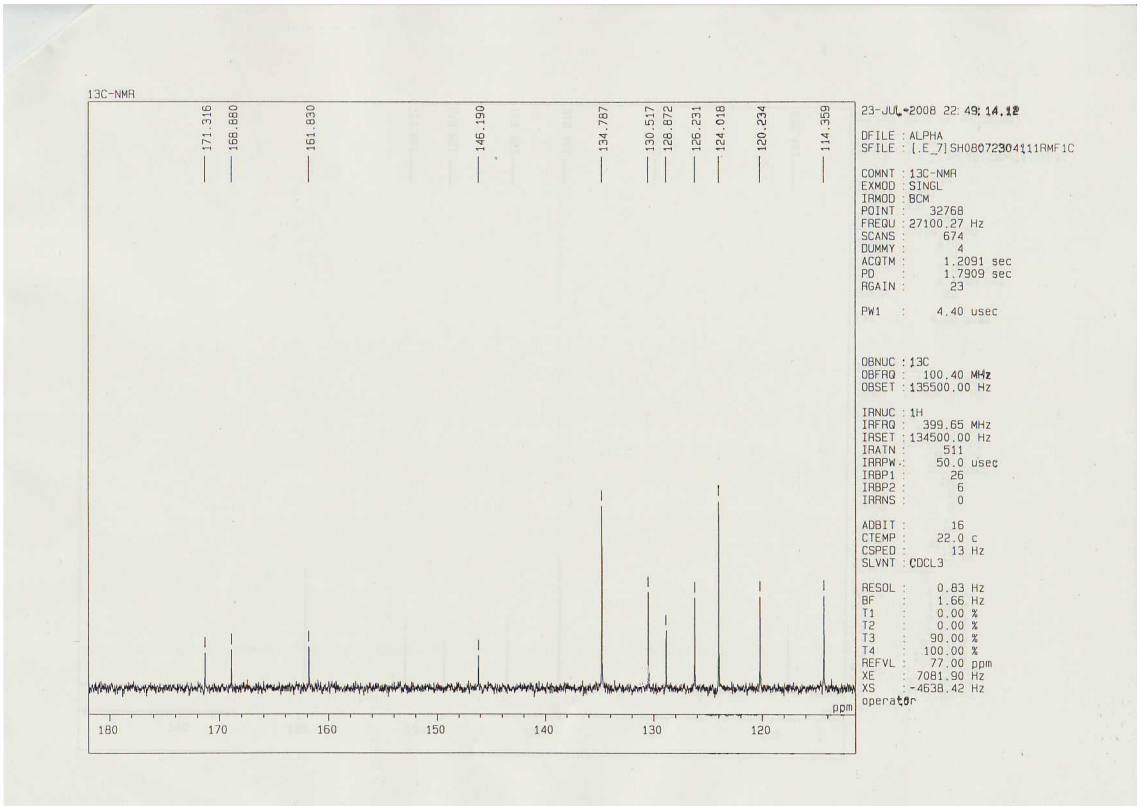

C:\Documents and Settings\Md. Chanmiya Sheikh\ffXfNfgfbfv\SH07062504111RMF1H.NMF
1H-NMR
7.907
7.899
7.893
7.886
7.875
7.873
7.821
7.819
7.809
7.801
7.795
7.787
7.699
7.697
7.681
7.679
7.676
7.661
7.658
7.546
7.543
7.528
7.526
7.522
7.507
7.505
1.94
2.00
1.03
1.01
PPM
8.0
7.9
7.8
7.7
7.6
7.5
DFILE C:\Documents and Settings\Md. Ch
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL-NON
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.0 Hz
POINT 32768
FREQU 7993.6 Hz
SCANS 27
ACQTM 4.099 sec
PD 2.901 sec
PW1 5.1 us
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23

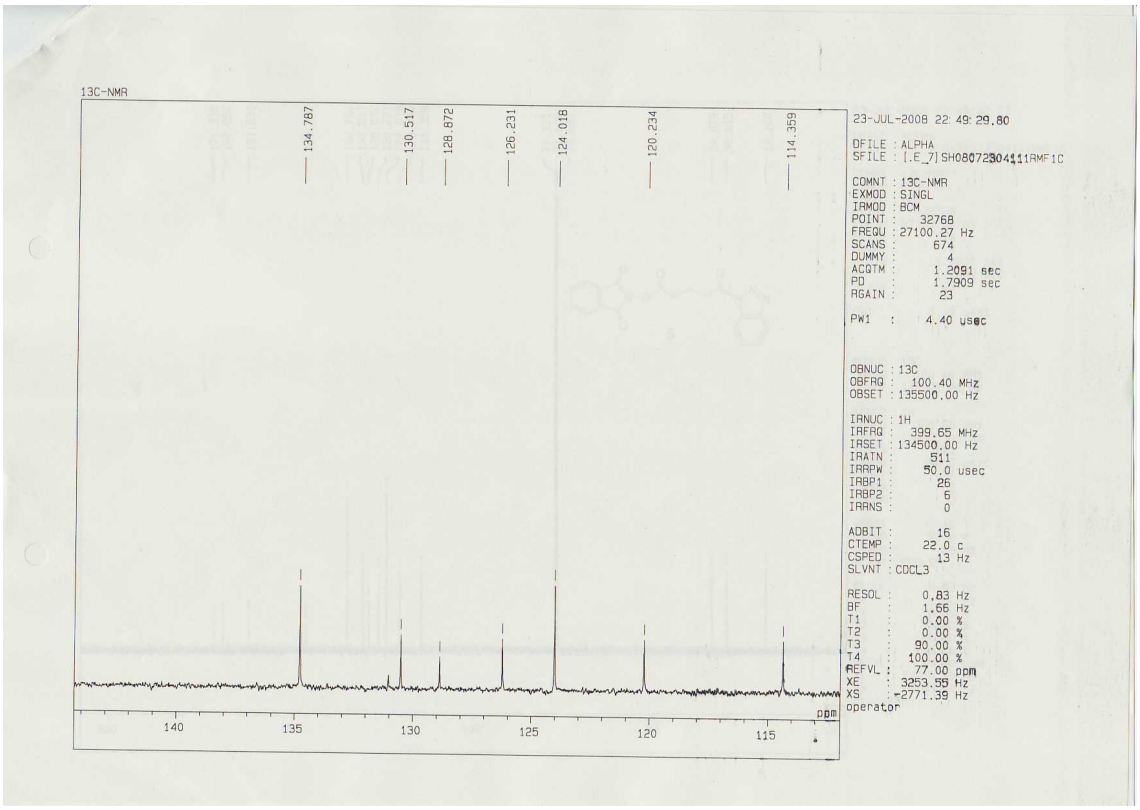


Phthalimido 4-(1-benzotriazolylcarbonyl)butanoate (16) (^{13}C-NMR)

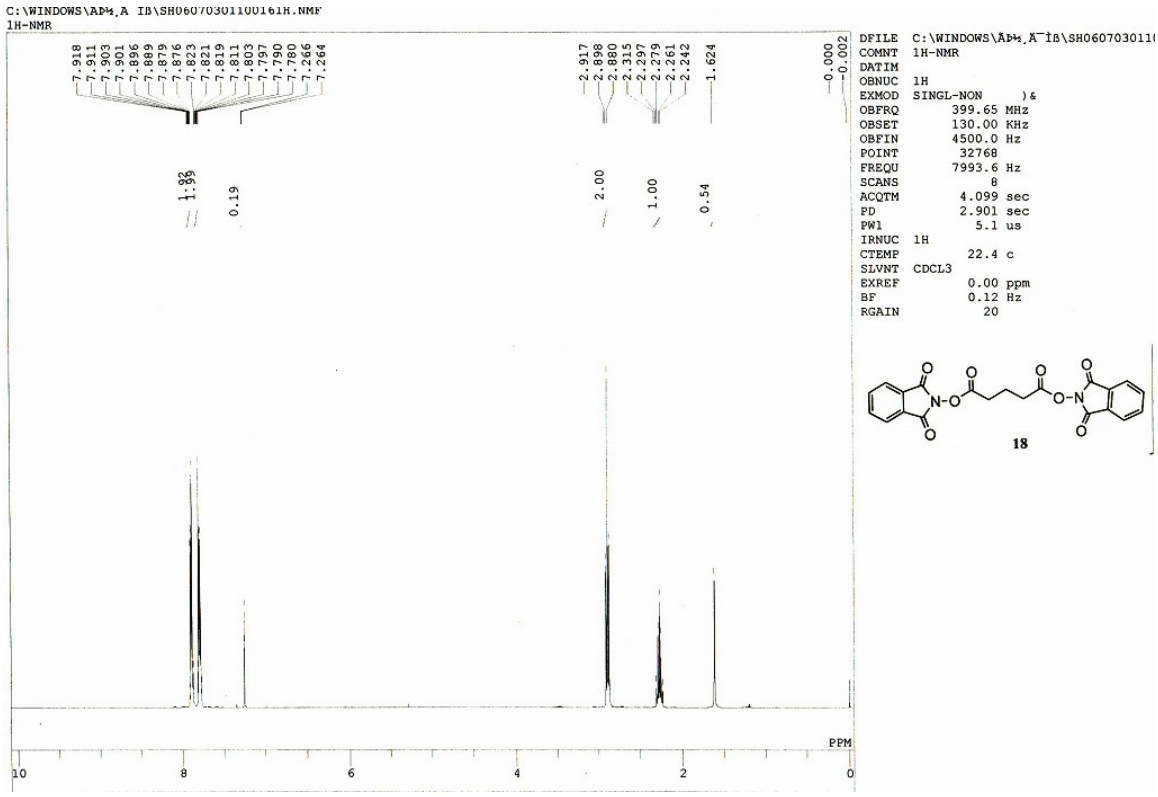


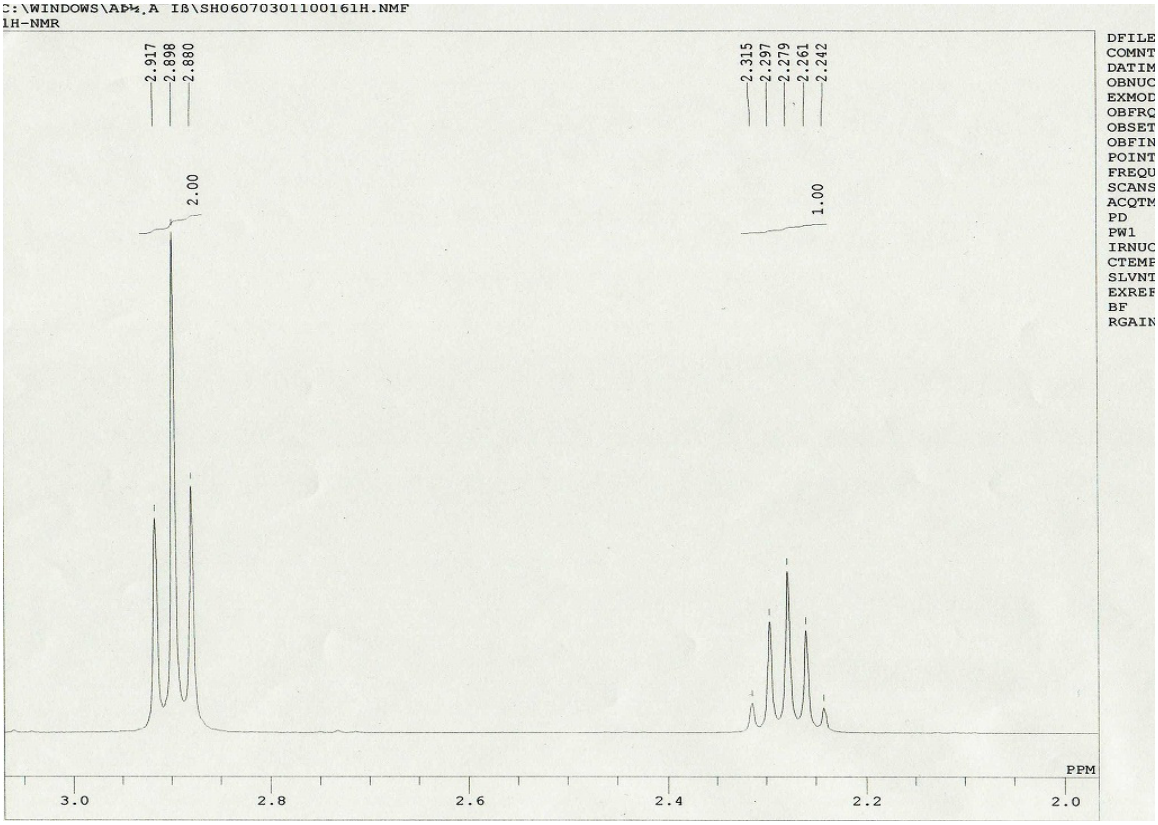

13C-NMR
34.079
30.015
19.180
ppm
50
40
30
20
10
23-JUL-2008 22:48:51.12
DFILE : ALPHA
SFILE : [.E_7]SH08072304111RMF1C
COMNT : 13C-NMR
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQU : 27100.27 Hz
SCANS : 674
DUMMY : 4
ACQTM : 1.2091 sec
PD : 1.7909 sec
RGAIN : 23
PW1 : 4.40 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 26
IRBP2 : 6
IRRNS : 0
ADBIT : 16
CTEMP : 22.0 c
CSPED : 13 Hz
SLVNT : CDCL3
RESOL : 0.83 Hz
BF : 1.66 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 4611.55 Hz
XS : 6934.69 Hz
operator

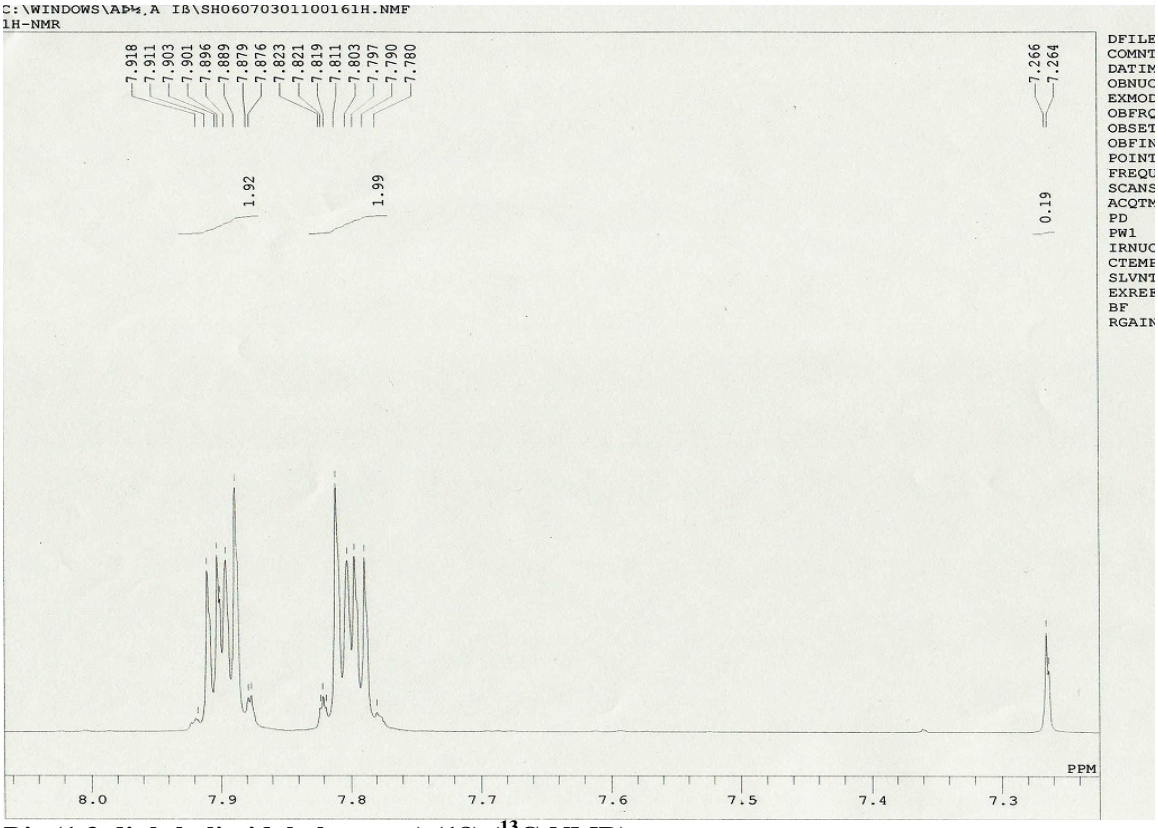

13C-NMR
171.316
168.880
161.830
146.190
134.787
130.517
128.872
126.231
124.018
120.234
114.359
ppm
180
170
160
150
140
130
120
23-JUL-2008 22:49:14.12
DFILE : ALPHA
SFILE : [.E_7]SH08072304111RMF1C
COMNT : 13C-NMR
EXMOD : SINGL
IRMOD : BCM
POINT : 32768
FREQU : 27100.27 Hz
SCANS : 674
DUMMY : 4
ACQTM : 1.2091 sec
PD : 1.7909 sec
RGAIN : 23
PW1 : 4.40 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134500.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 26
IRBP2 : 6
IRRNS : 0
ADBIT : 16
CTEMP : 22.0 c
CSPED : 13 Hz
SLVNT : CDCL3
RESOL : 0.83 Hz
BF : 1.66 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 77.00 ppm
XE : 7081.90 Hz
XS : -4638.42 Hz
operator



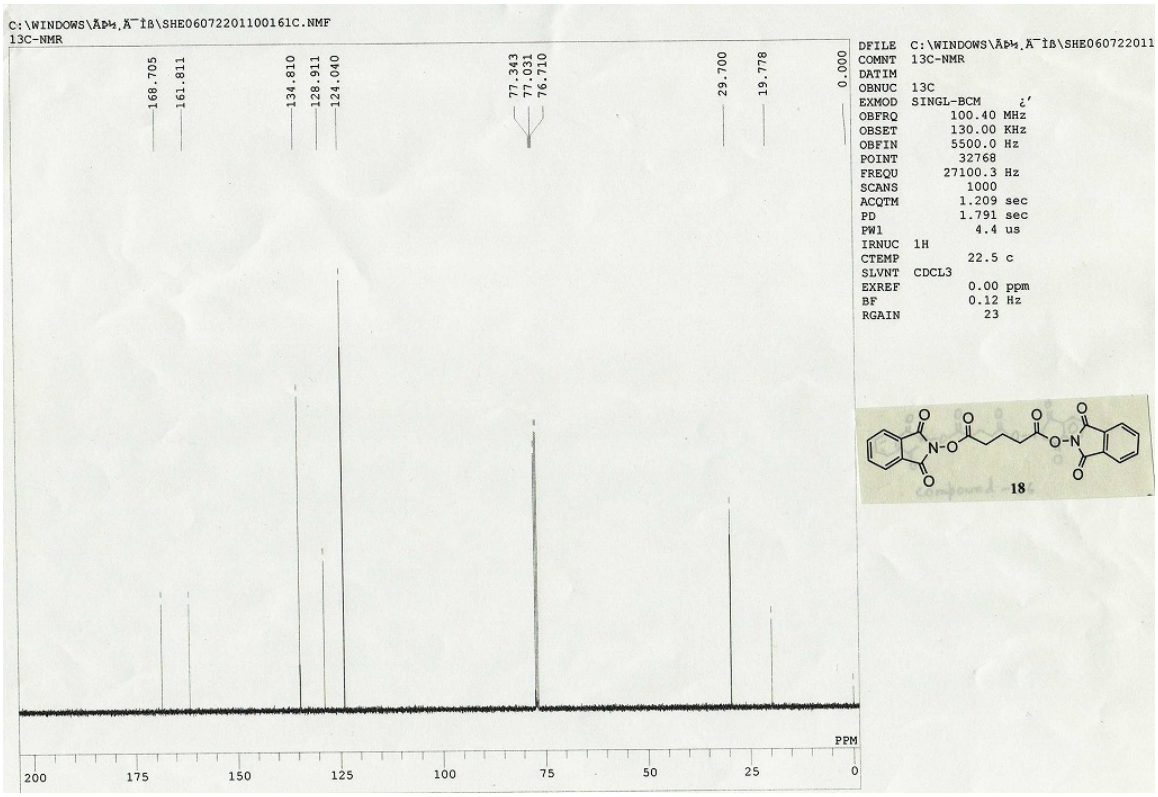
Bis-(1,3-diphthalimidyl glutarate) (18) (^{1}H-NMR)

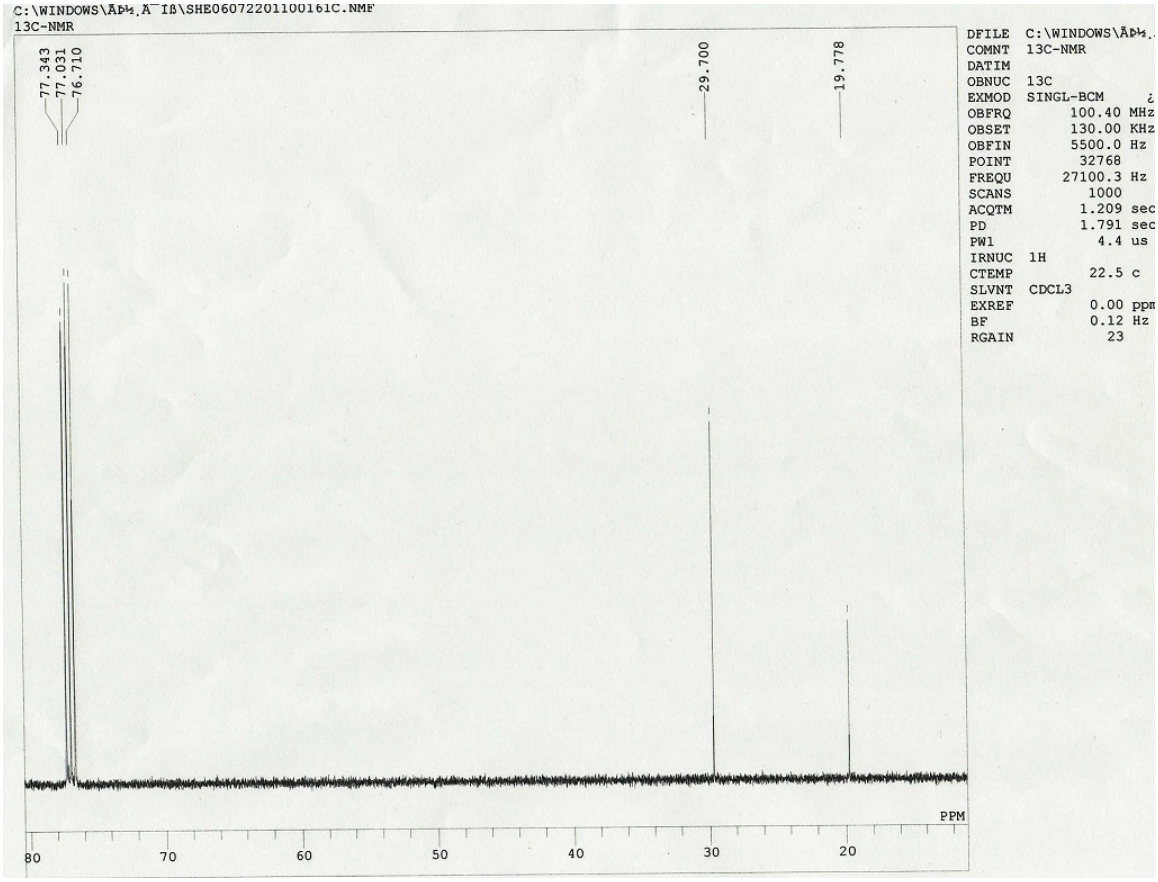


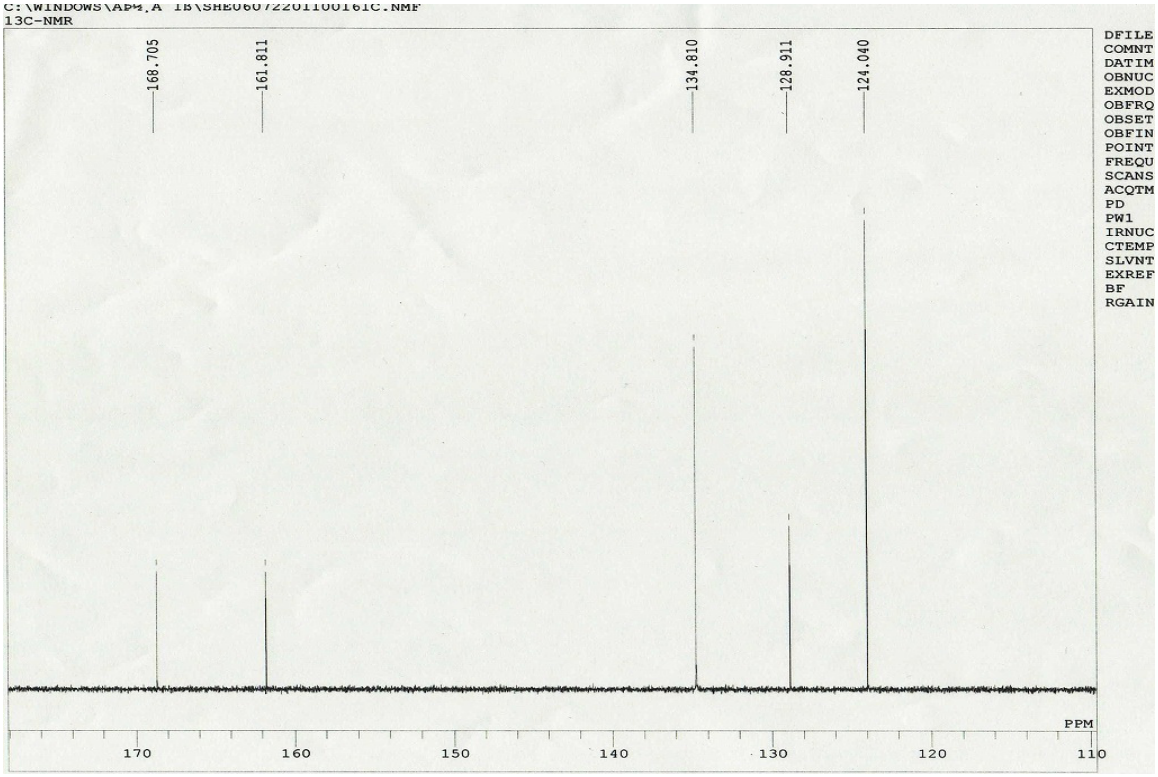




Bis-(1,3-diphthalimidyl glutarate) (18) (^{13}C-NMR)


C:\WINDOWS\ÃÞ½.Ä¯ÍßSHE06072201100161C.NMF
13C-NMR
168.705
161.811
134.810
128.911
124.040
77.343
77.031
76.710
29.700
19.778
0.000
PPM
200
175
150
125
100
75
50
25
0
DFILE C:\WINDOWS\ÃÞ½.Ä¯ÍßSHE060722011
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM ¿'
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23
18


C:\WINDOWS\ÃÞ½.Ä¯ÍßSHE06072201100161C.NMF
13C-NMR
77.343
77.031
76.710
29.700
19.778
PPM
80
70
60
50
40
30
20
DFILE C:\WINDOWS\ÃÞ½.
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM ¿
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 1000
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23

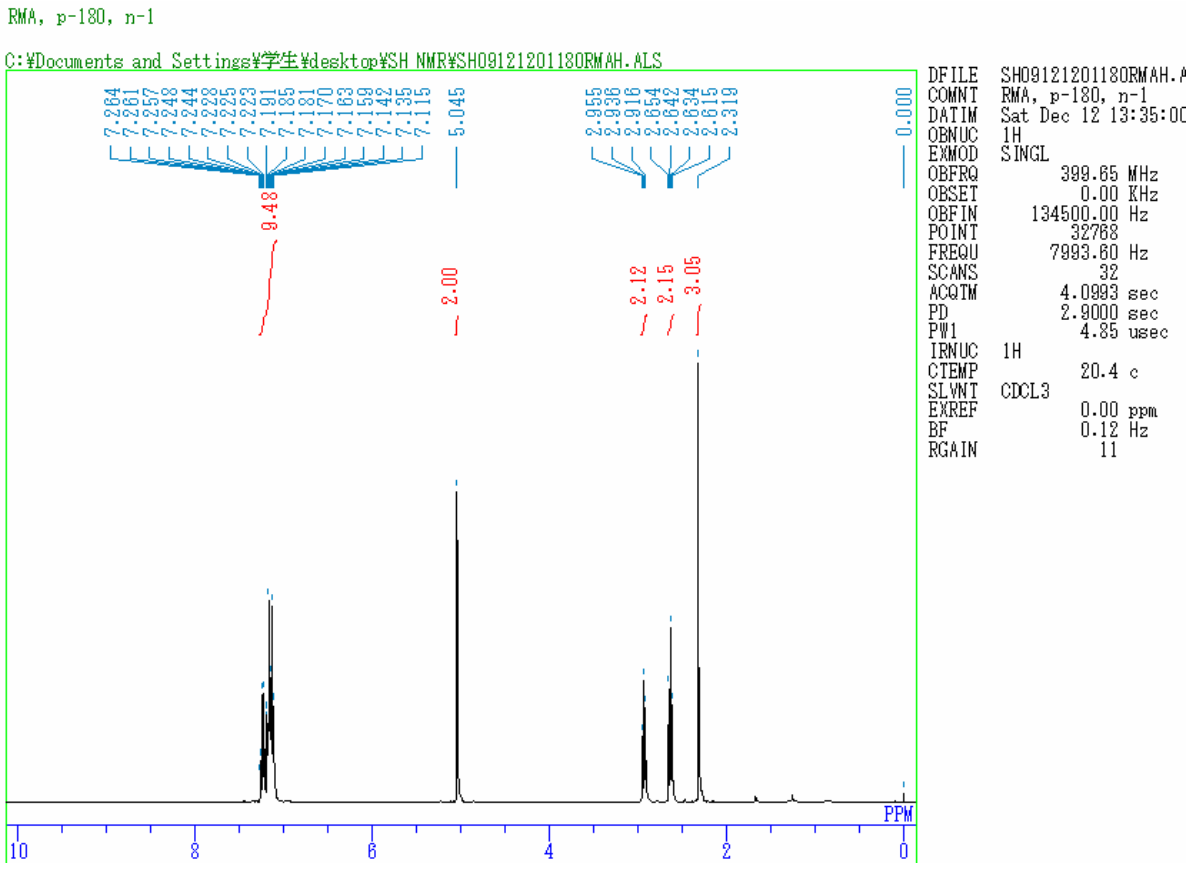

13C-NMR
168.705
161.811
134.810
128.911
124.040
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
170
160
150
140
130
120
110

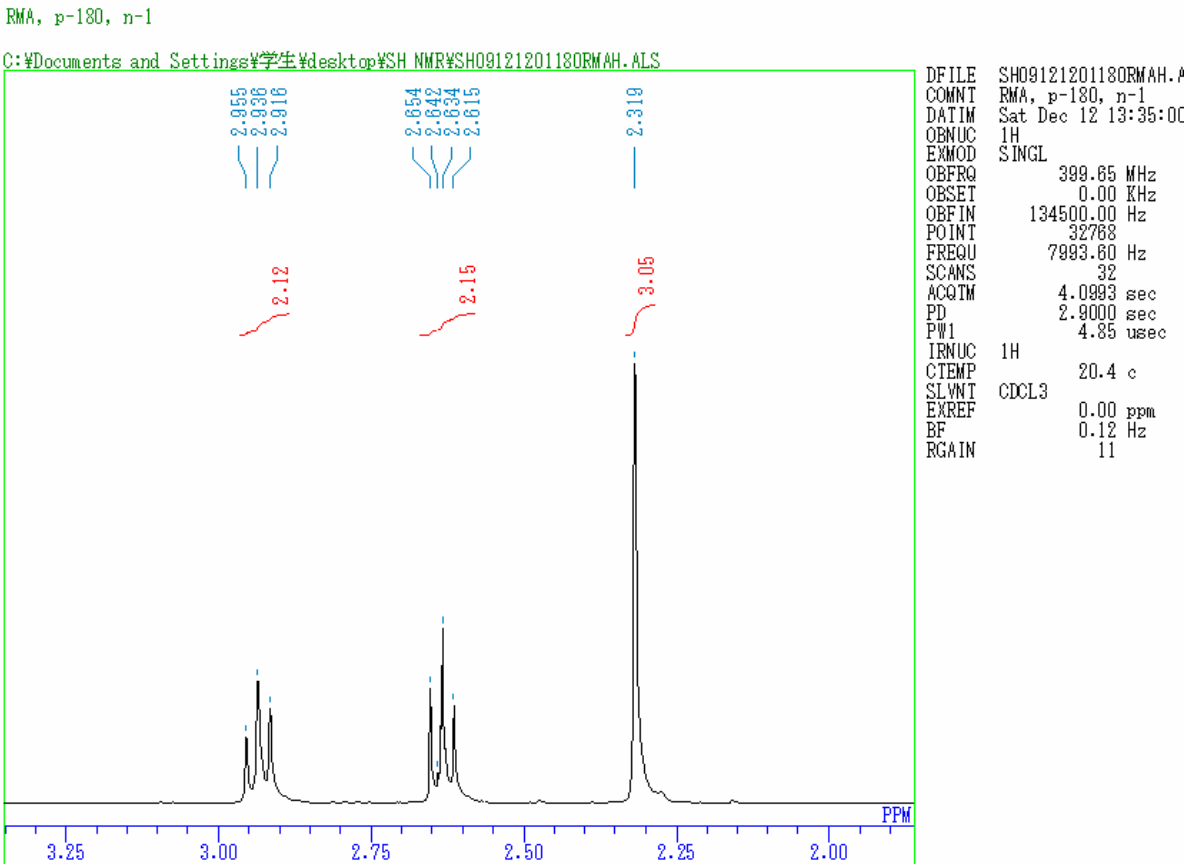
(iii) Supporting Information for the manuscript:

Title: “Syntheses and reactivities of non-symmetrical "active ester" bi-dentate cross-linking reagents having a phthalimidoyl and acid chloride, 2-benzothiazole, or 1-benzotriazole group”

(^{1}H –NMR and ^{13}C-NMR)

3-phenylpropionic acid 4-methylbenzyl ester (19) (^{1}H-NMR)




RMA, p-180, n-1
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2.955
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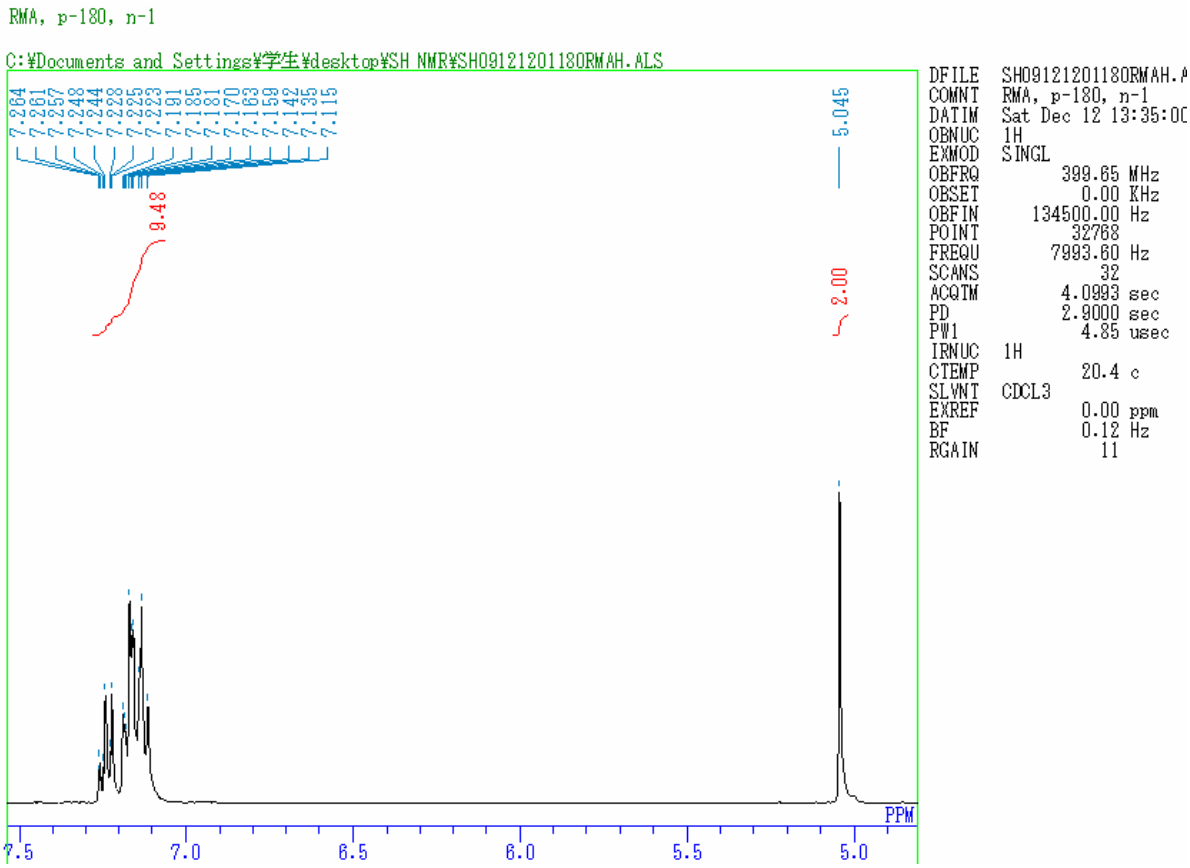
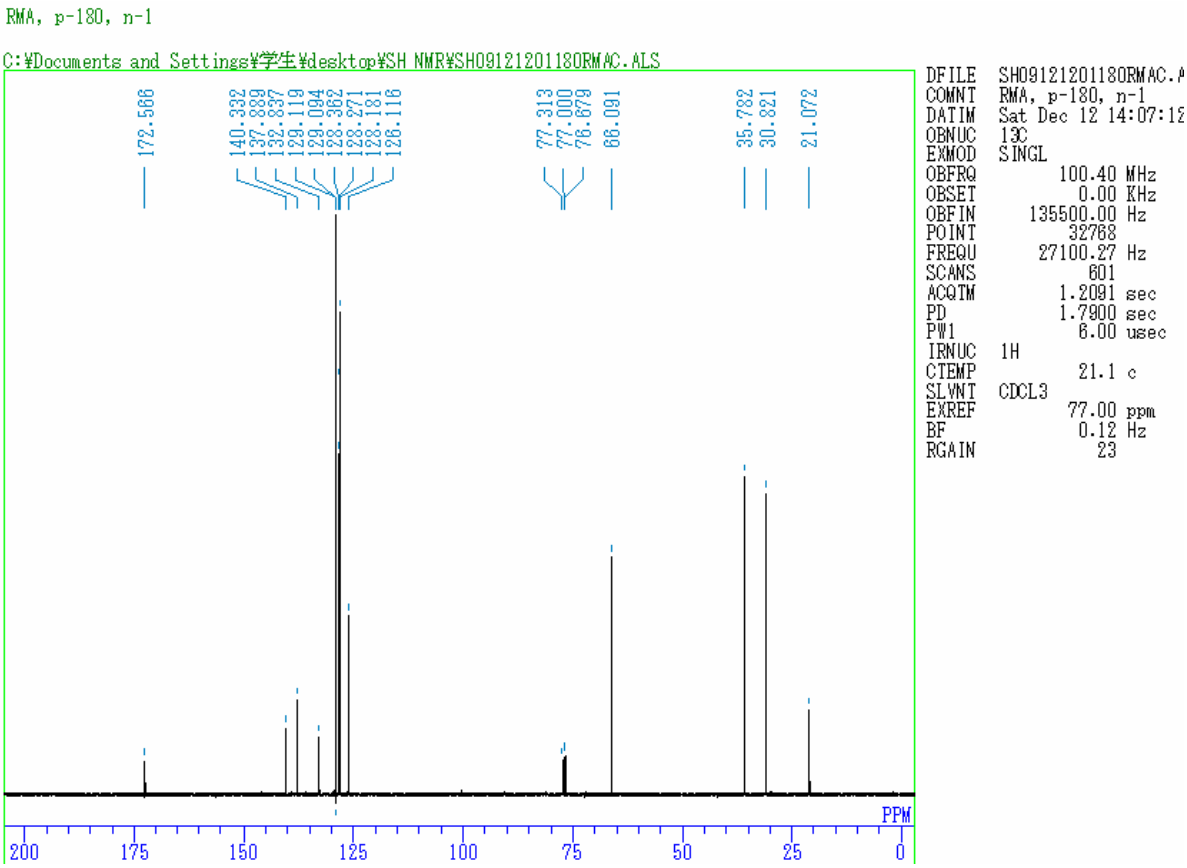
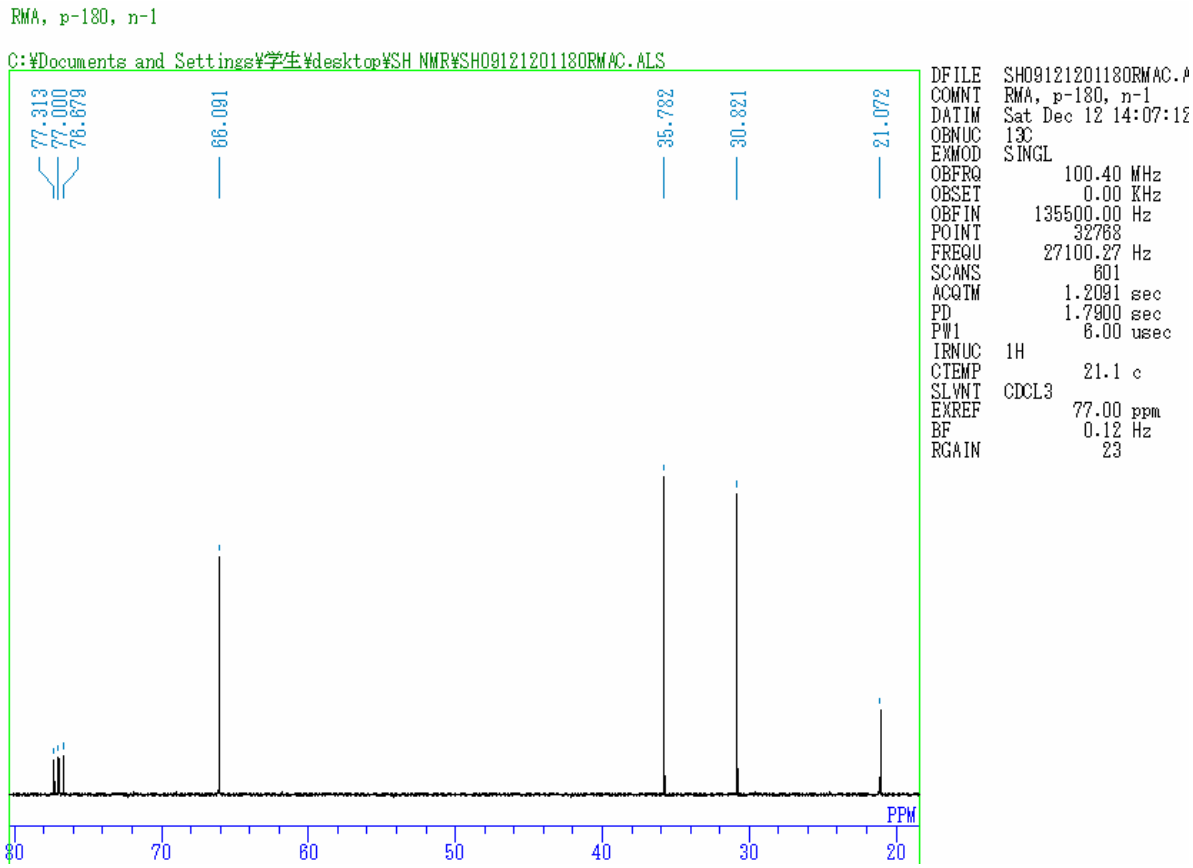
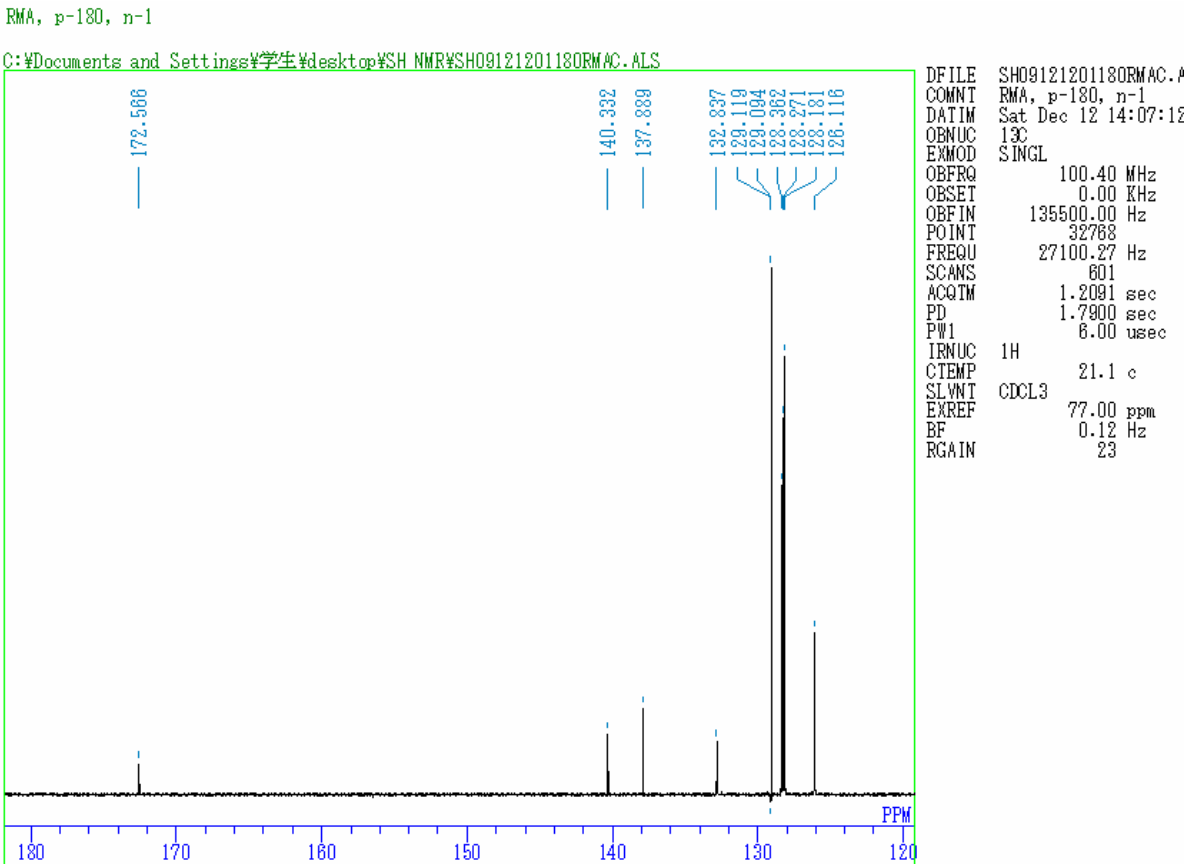
2.916
2.654
2.642
2.634
2.615
2.319
2.12
2.15
3.05
PPM
3.25
3.00
2.75
2.50
2.25
2.00
DFILE SH09121201180RMAH.A
COMNT RMA, p-180, n-1
DATIM Sat Dec 12 13:35:0C
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 11

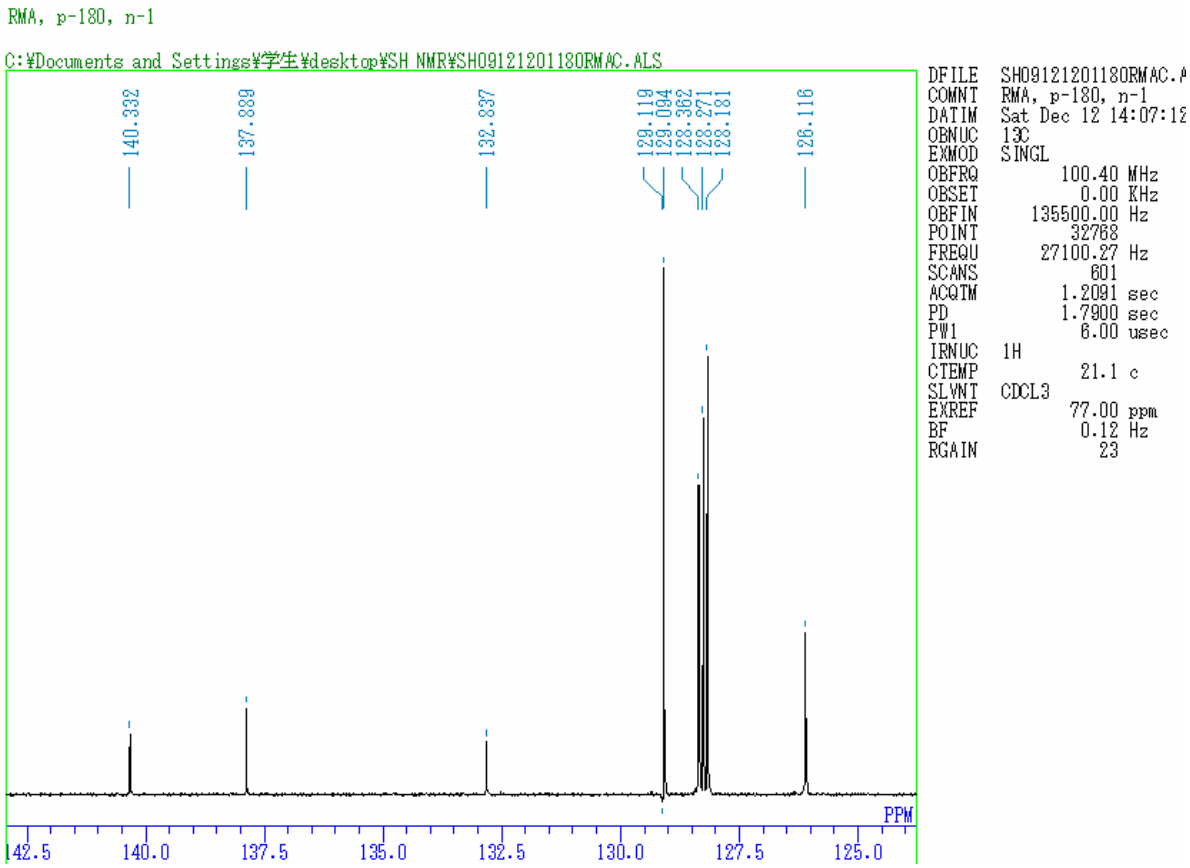


3-phenylpropionic acid 4-methylbenzyl ester (19) (^{13}C-NMR)

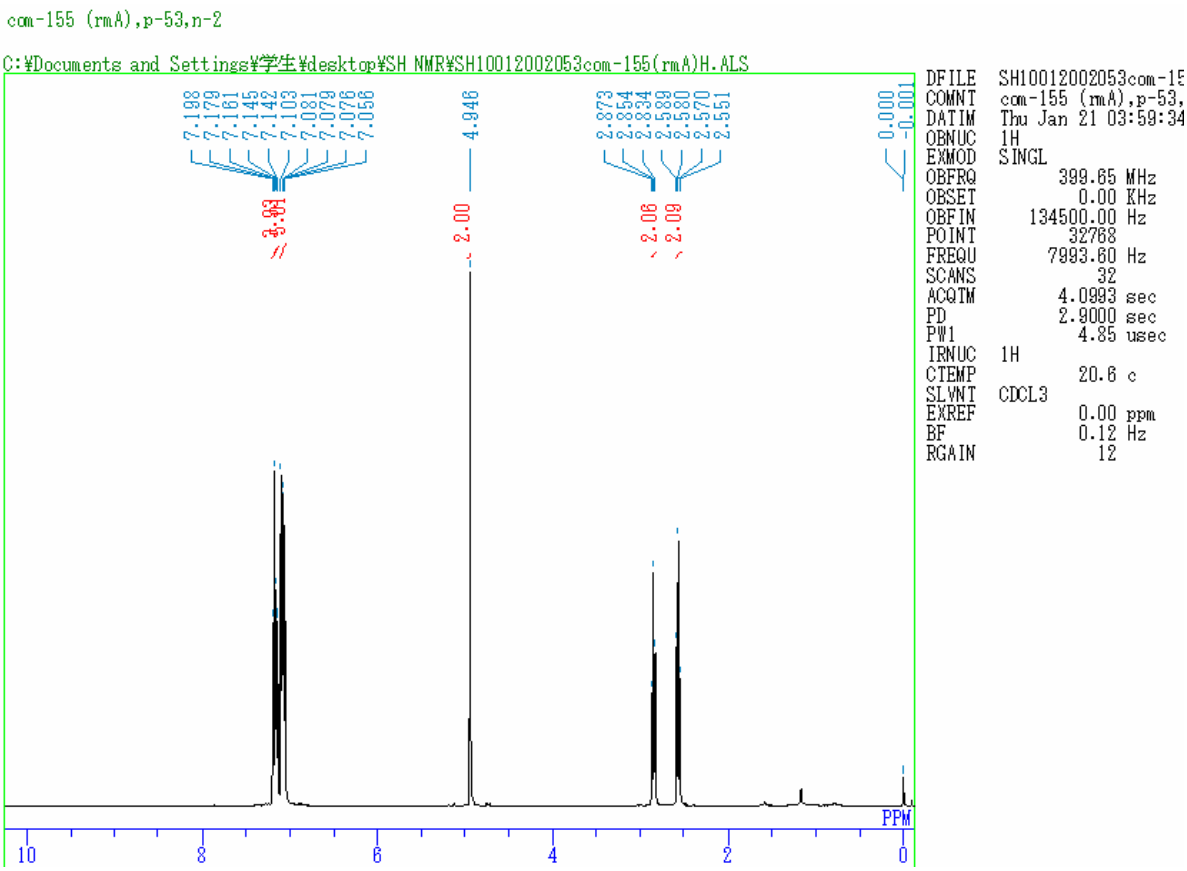

RMA, p-180, n-1
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH09121201180RMAC.ALS
172.566
140.332
137.889
132.837
129.119
129.094
128.362
128.271
128.181
126.116
77.313
77.000
76.679
66.091
35.782
30.821
21.072
PPM
200
175
150
125
100
75
50
25
0
DFILE SH09121201180RMAC.A
COMNT RMA, p-180, n-1
DATIM Sat Dec 12 14:07:12
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 601
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

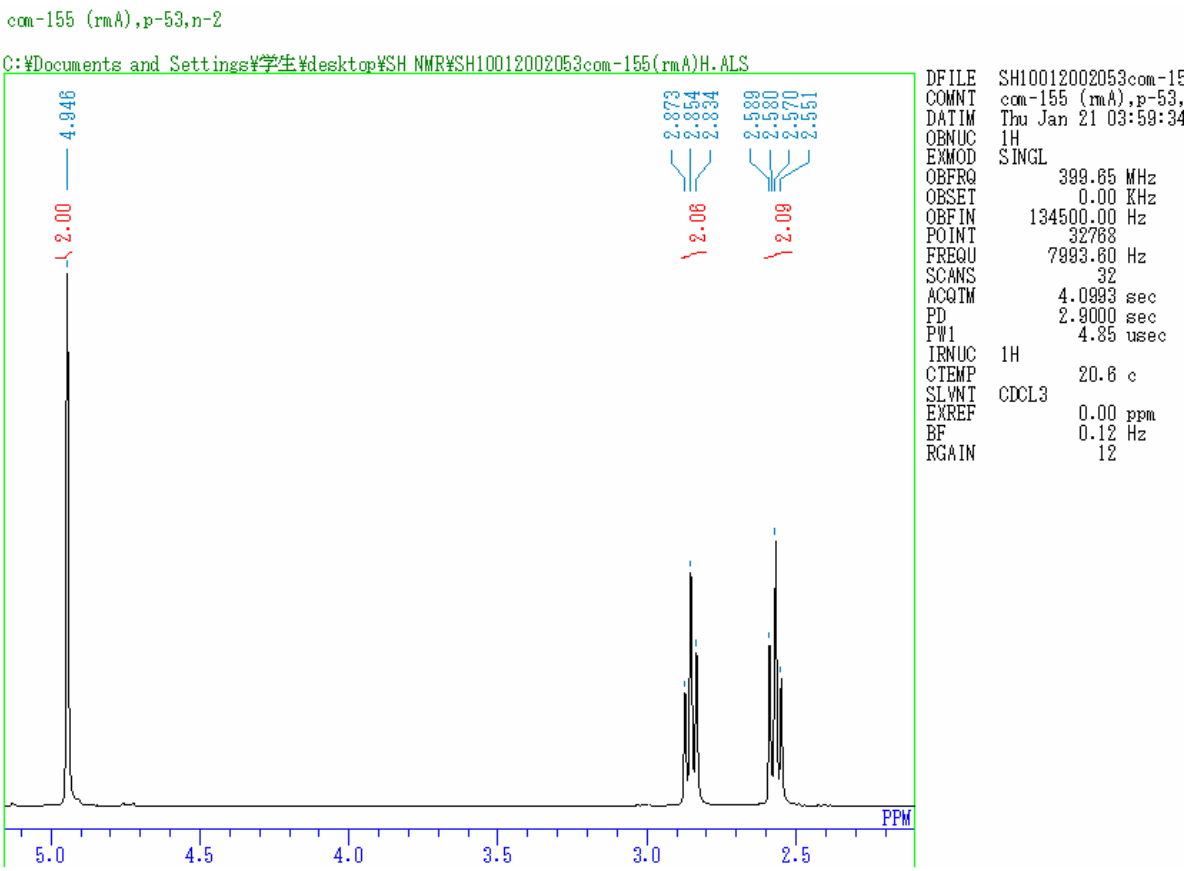

RMA, p-180, n-1
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH09121201180RMAC.ALS
77.313
77.000
76.679
66.091
35.782
30.821
21.072
PPM
80
70
60
50
40
30
20
DFILE SH09121201180RMAC.A
COMNT RMA, p-180, n-1
DATIM Sat Dec 12 14:07:12
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 601
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

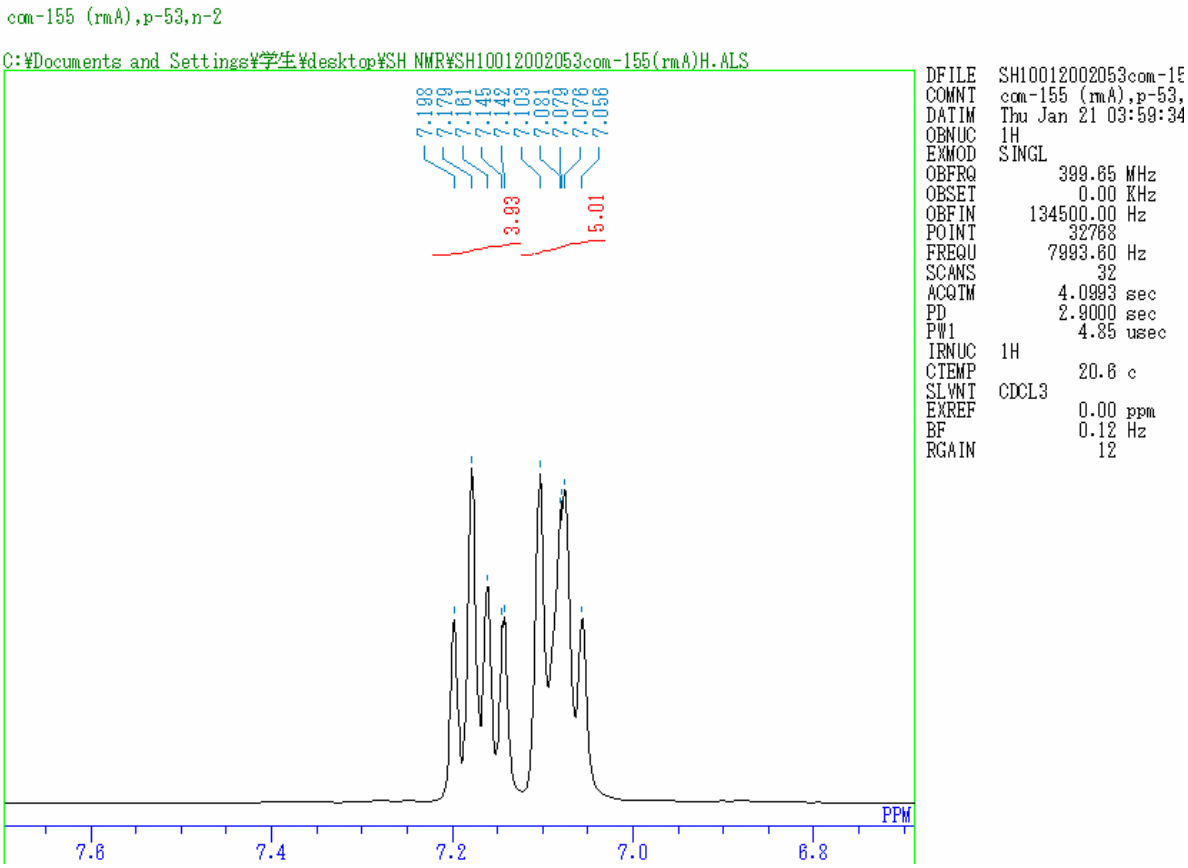

RMA, p-180, n-1
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH09121201180RMAC.ALS
172.566
140.332
137.889
132.837
129.119
129.094
128.362
128.271
128.181
126.116
PPM
180
170
160
150
140
130
120
DFILE SH09121201180RMAC.A
COMNT RMA, p-180, n-1
DATIM Sat Dec 12 14:07:12
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 601
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23



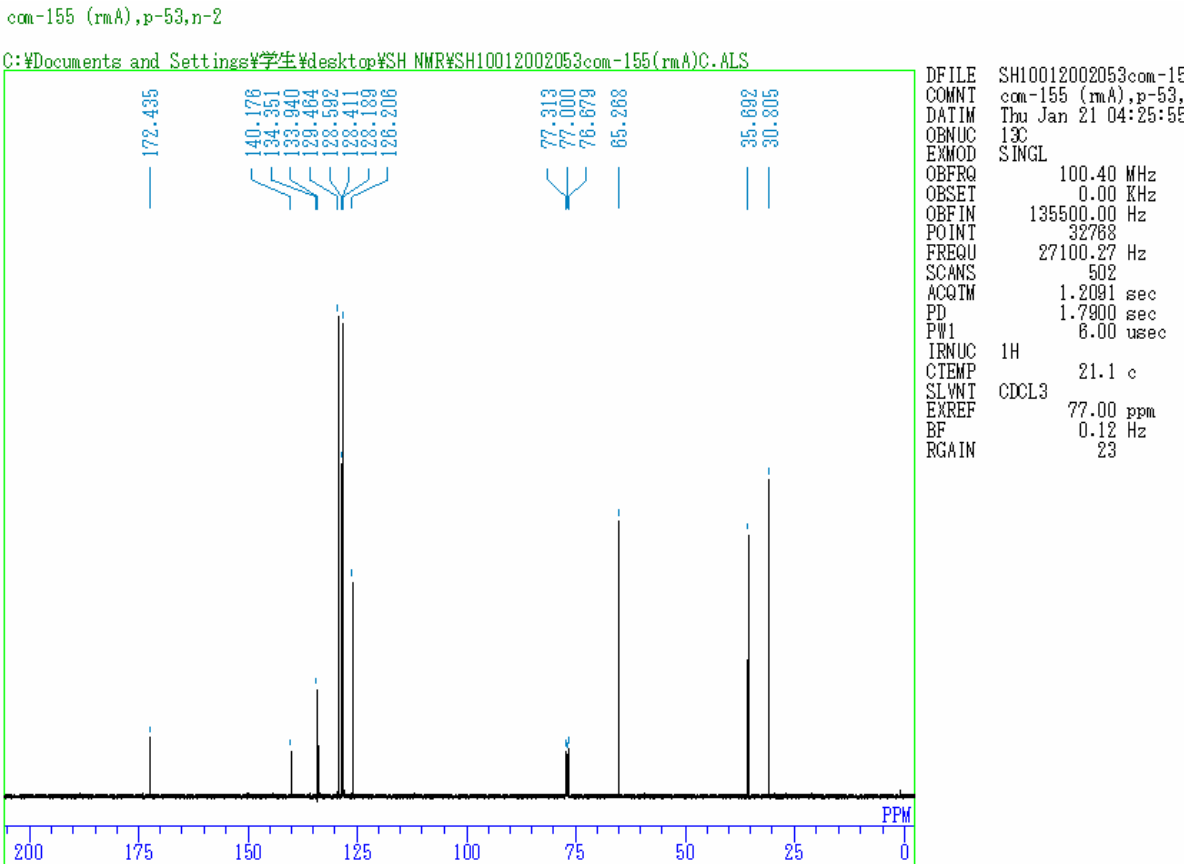
3-phenylpropionic acid 4-chlorobenzyl ester (20) (^{1}H-NMR)

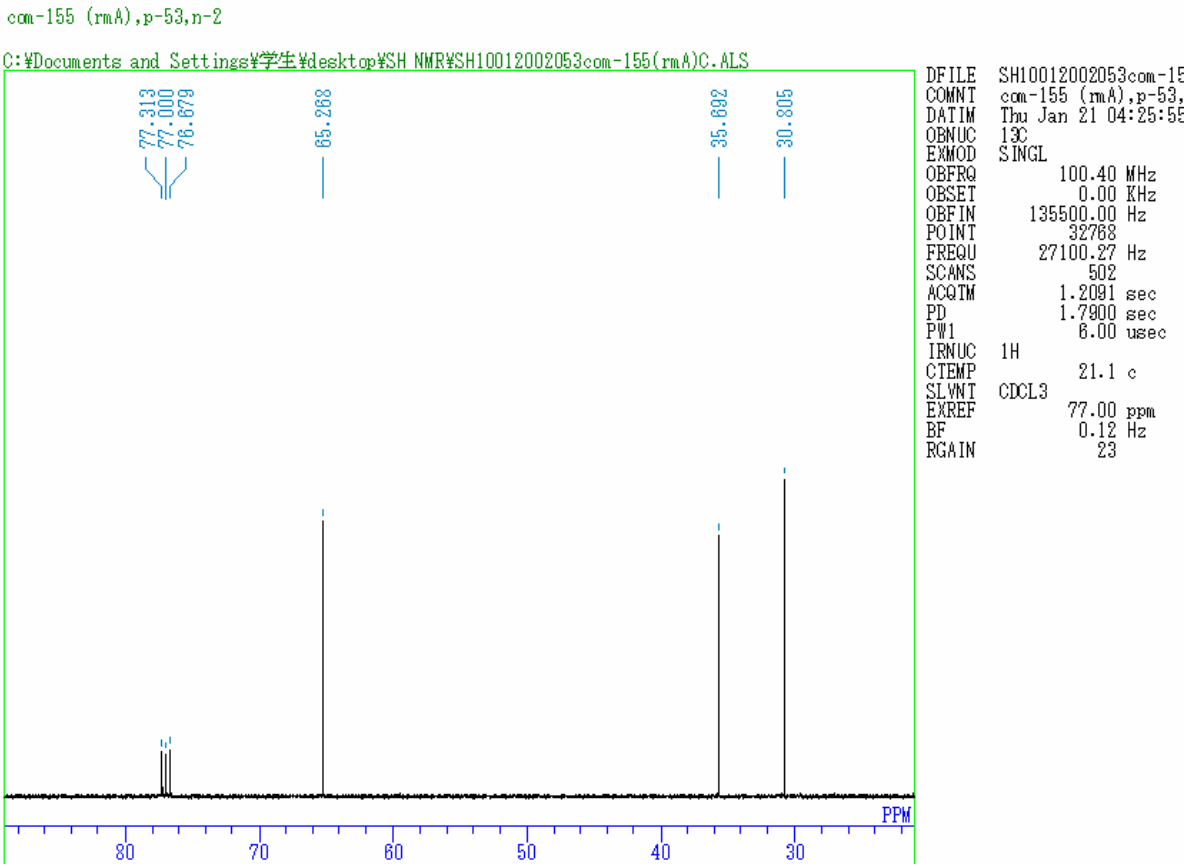

com-155 (rmA),p-53,n-2
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH10012002053com-155(rmA)H.ALS
7.198
7.179
7.161
7.145
7.142
7.103
7.081
7.079
7.076
7.056
4.946
2.873
2.854
2.834
2.589
2.580
2.570
2.551
0.000
-0.001
3.93
2.00
2.08
2.09
PPM
10
8
6
4
2
0
DFILE SH10012002053com-15
COMNT com-155 (rmA),p-53,
DATIM Thu Jan 21 03:59:34
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12

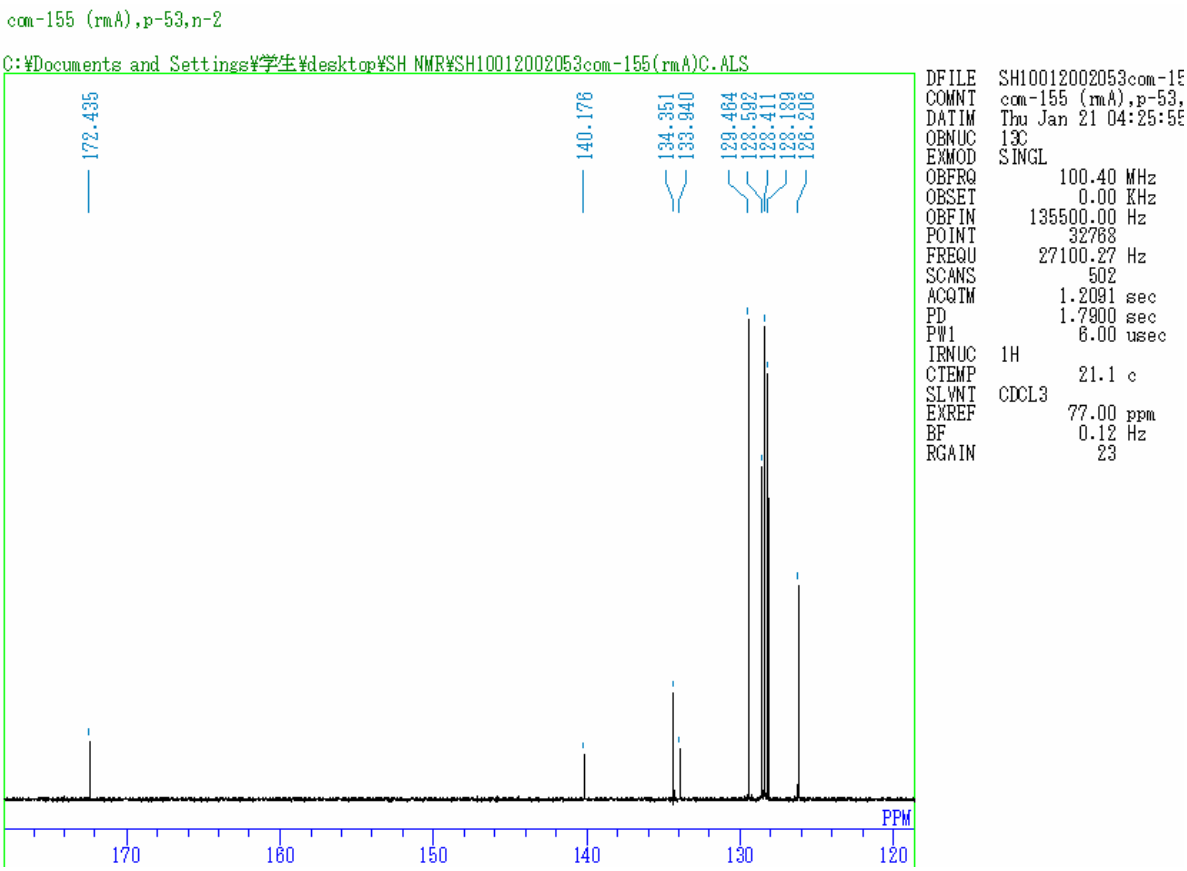

com-155 (rmA),p-53,n-2
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH10012002053com-155(rmA)H.ALS
4.946
2.873
2.864
2.834
2.589
2.580
2.570
2.551
2.00
2.06
2.09
PPM
5.0
4.5
4.0
3.5
3.0
2.5
DFILE SH10012002053com-15
COMNT com-155 (rmA),p-53,
DATIM Thu Jan 21 03:59:34
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12



3-phenylpropionic acid 4-chlorobenzyl ester (20) (^{13}C-NMR)


com-155 (rmA),p-53,n-2
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH10012002053com-155(rmA)C.ALS
172.435
140.176
134.351
133.940
129.464
128.592
128.411
128.139
126.206
77.313
77.000
76.679
65.268
35.692
30.805
PPM
200
175
150
125
100
75
50
25
0
DFILE SH10012002053com-15
COMNT com-155 (rmA),p-53,
DATIM Thu Jan 21 04:25:55
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 502
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23


com-155 (rmA),p-53,n-2
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH10012002053com-155(rmA)C.ALS
77.313
77.000
78.679
65.268
35.892
30.805
PPM
80
70
60
50
40
30
DFILE SH10012002053com-15
COMNT com-155 (rmA),p-53,
DATIM Thu Jan 21 04:25:55
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 502
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23


com-155 (rmA),p-53,n-2
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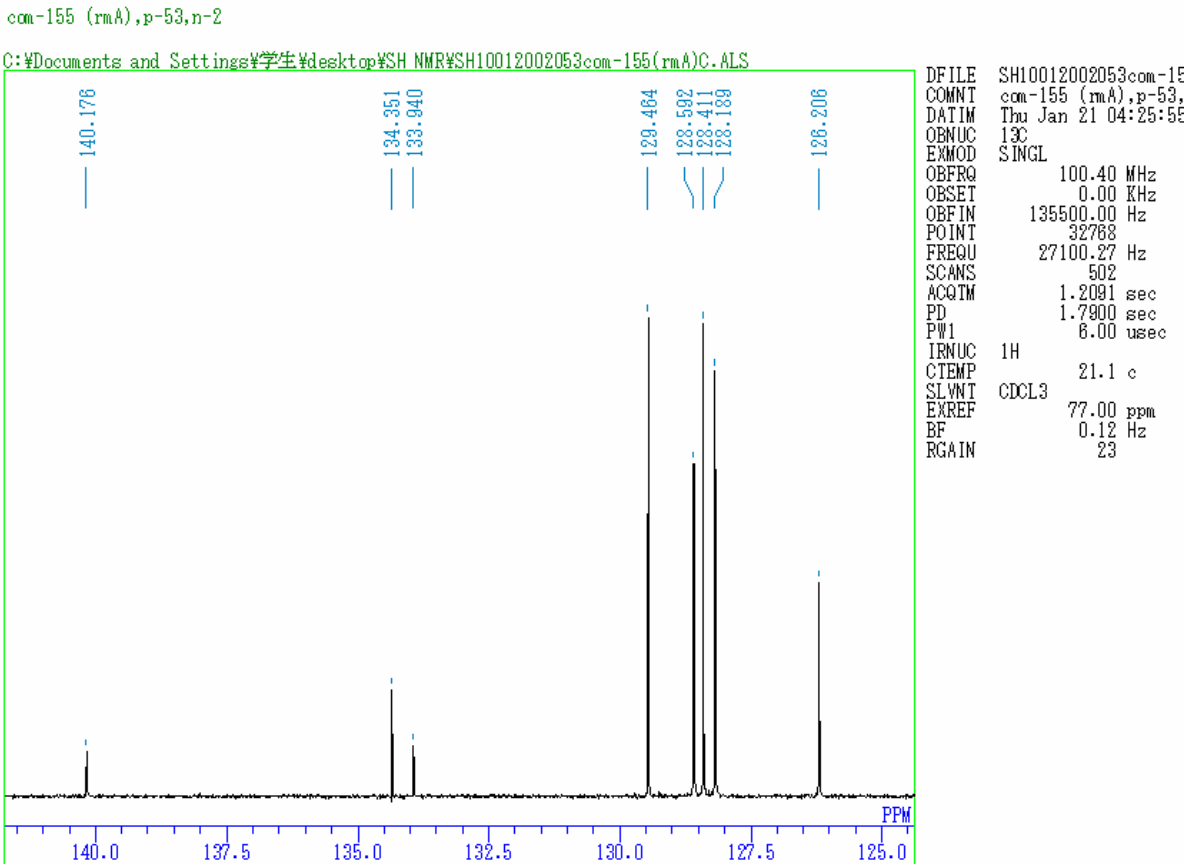
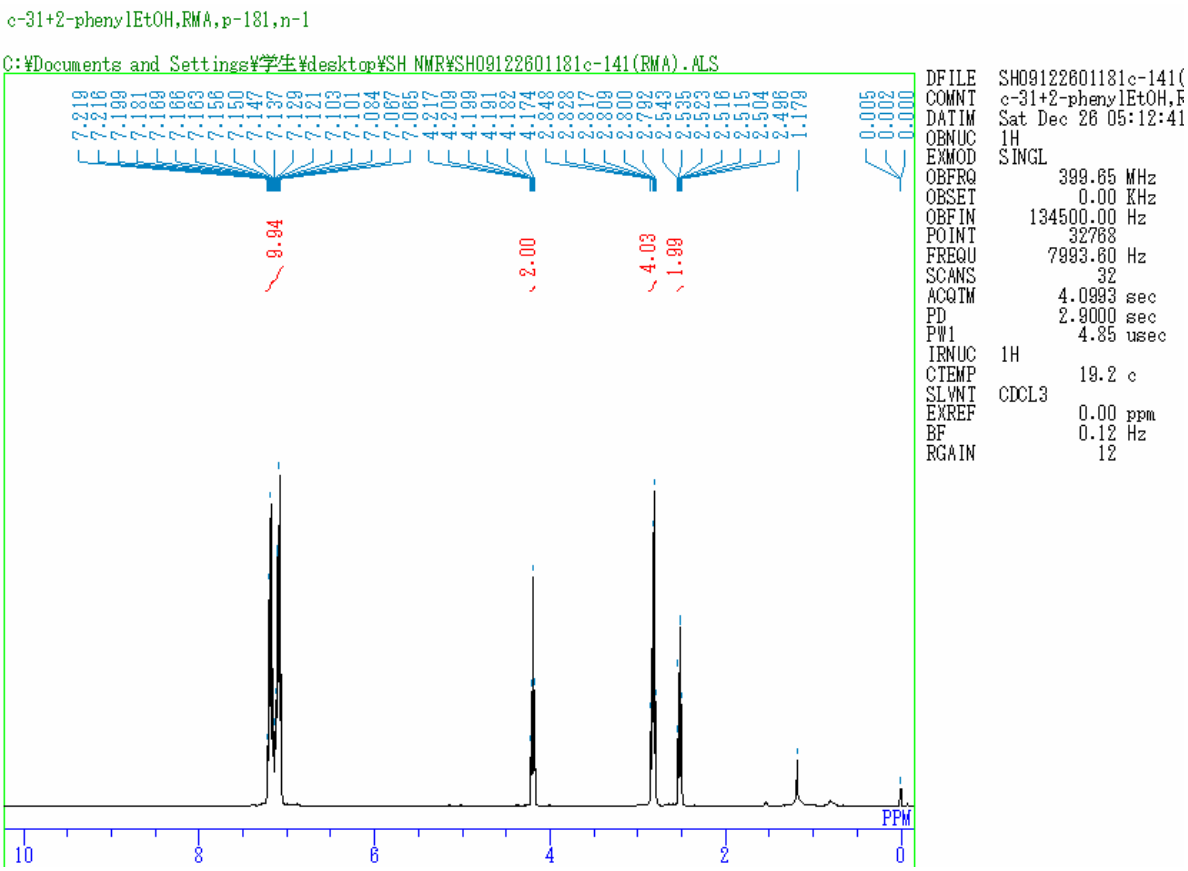
172.435
140.176
134.351
133.940
129.464
128.592
128.411
128.189
126.206
PPM
170
160
150
140
130
120
DFILE SH10012002053com-15
COMNT com-155 (rmA),p-53,
DATIM Thu Jan 21 04:25:55
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 502
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23



3-phenyl propionic acid 2 phenylethyl ester (21) (^{1}H-NMR)


c-31+2-phenylEtOH,RMA,p-181,n-1
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH09122601181c-141(RMA).ALS
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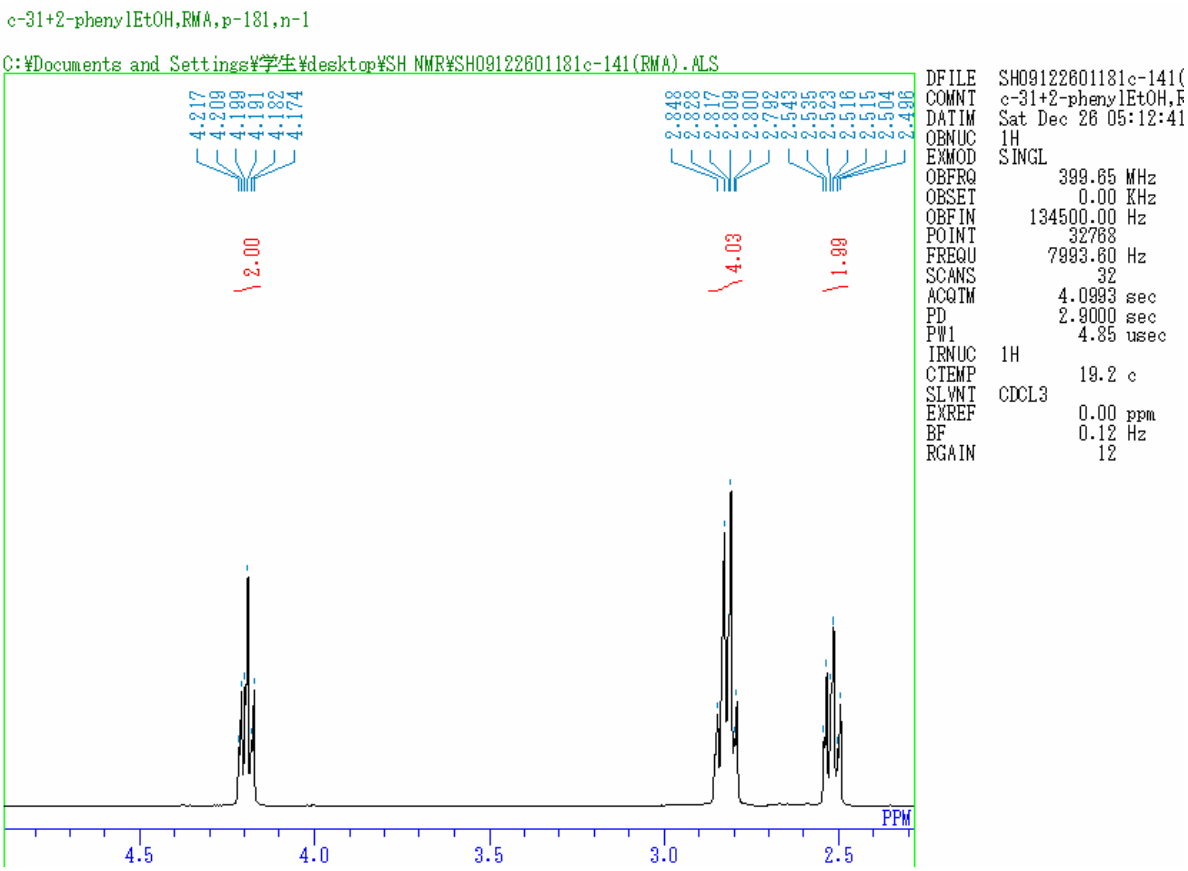
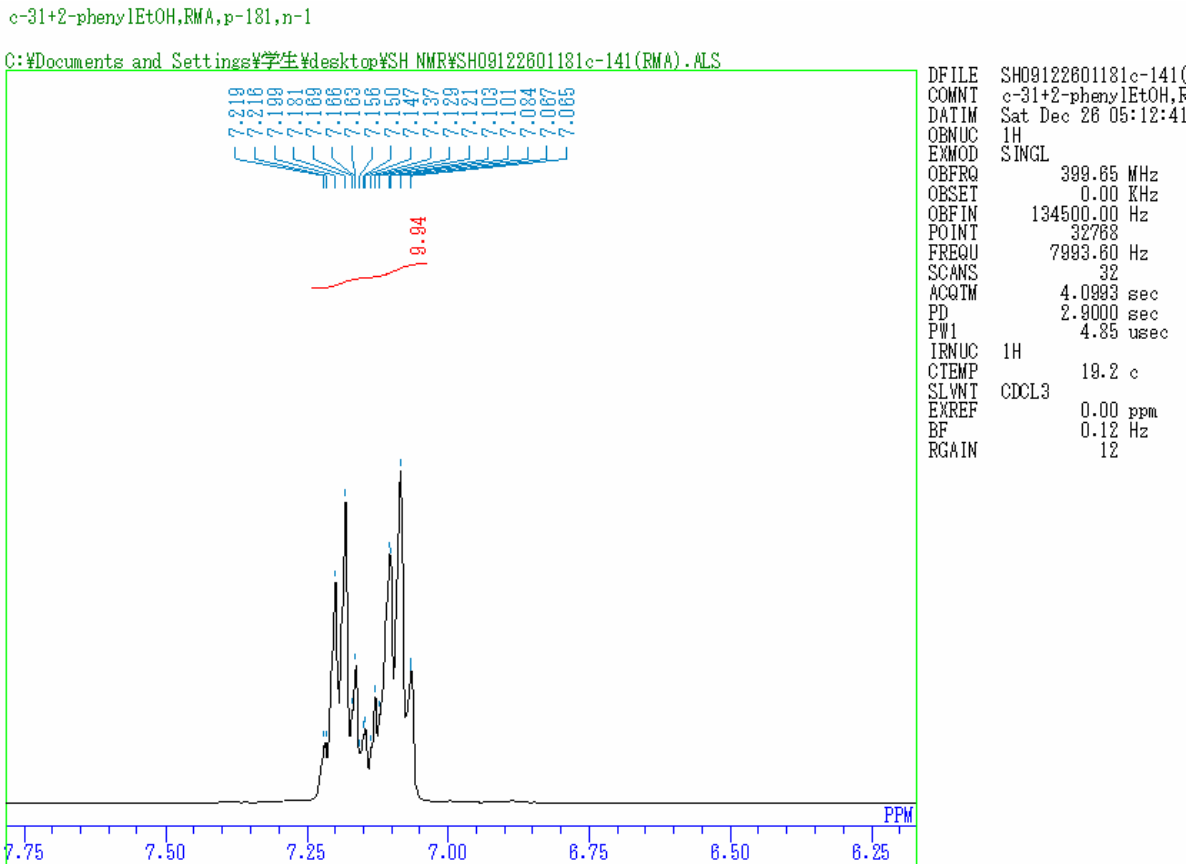
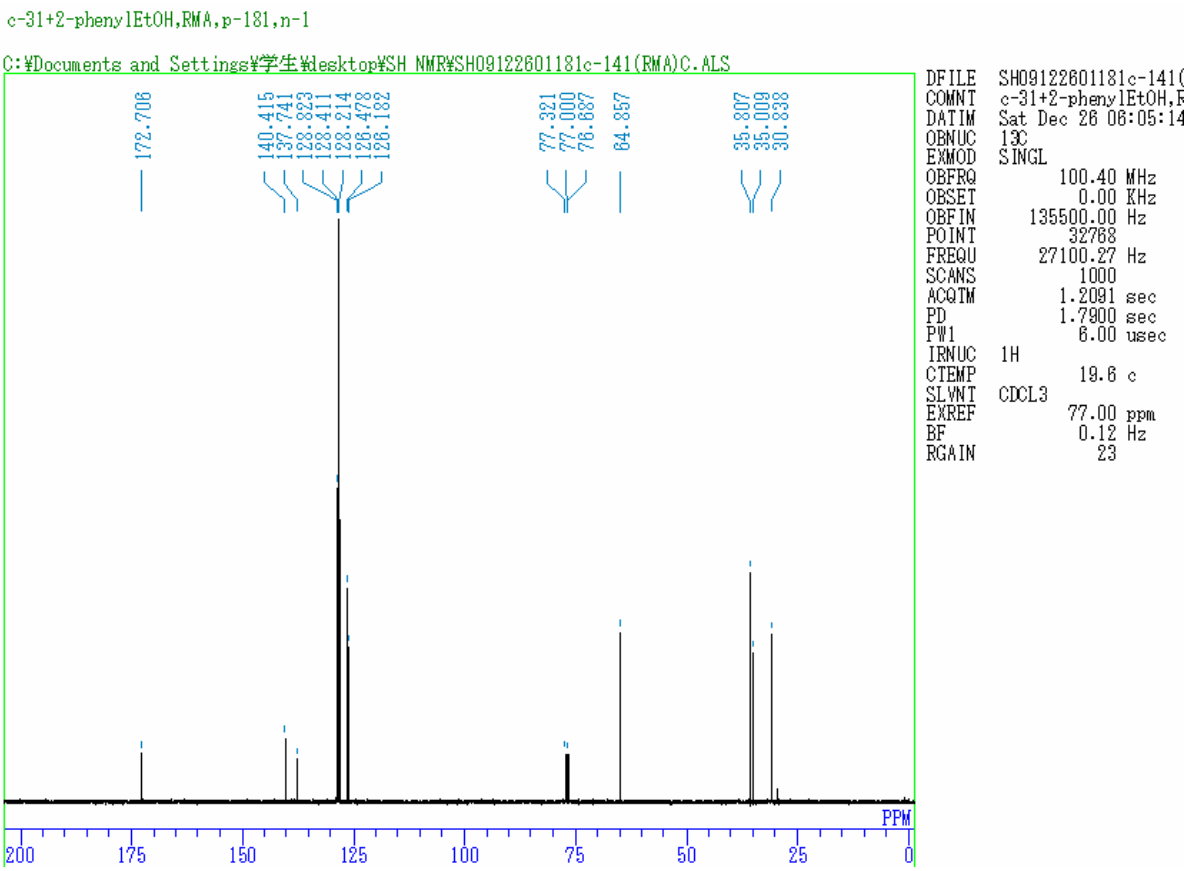
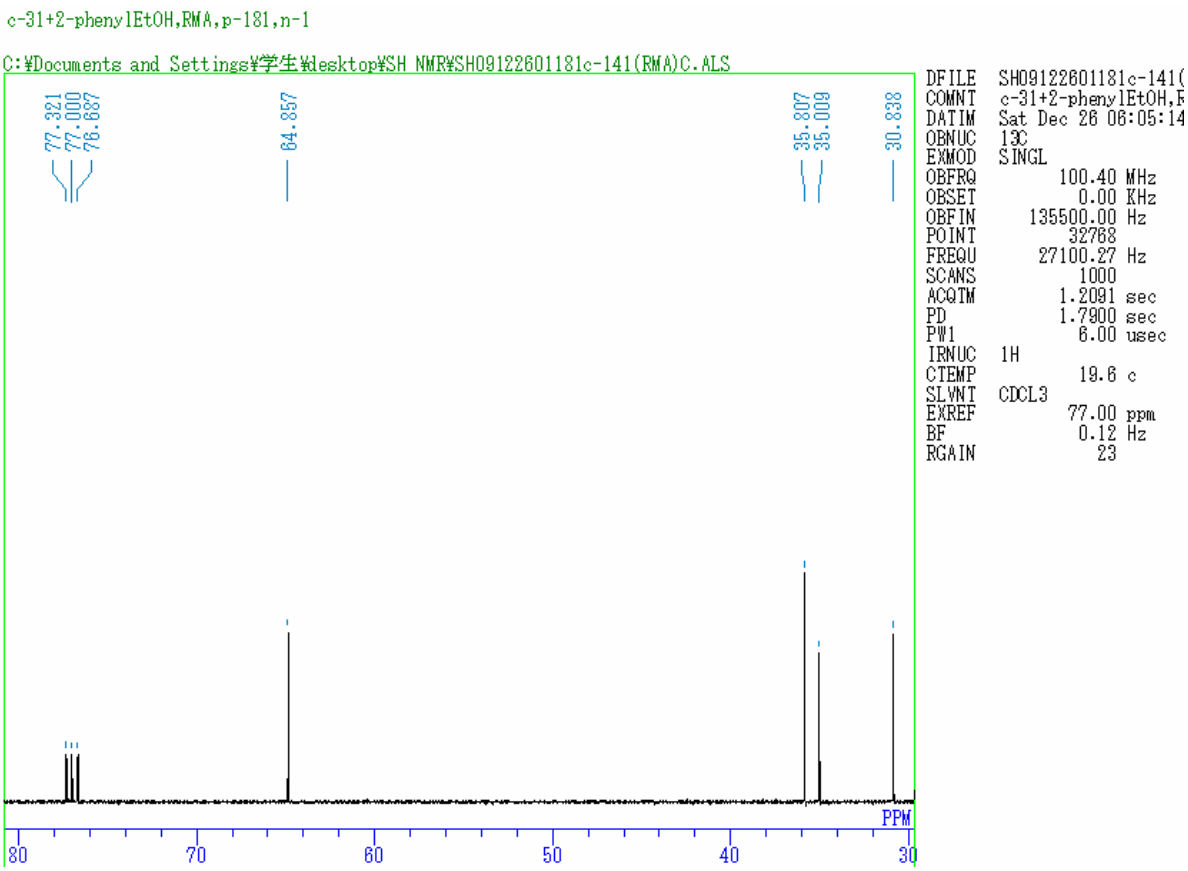
7.216
7.199
7.181
7.169
7.166
7.163
7.156
7.150
7.147
7.137
7.129
7.121
7.103
7.101
7.084
7.067
7.065
4.217
4.209
4.199
4.191
4.182
4.174
2.848
2.828
2.817
2.809
2.800
2.792
2.543
2.535
2.523
2.516
2.515
2.504
2.486
1.179
0.005
0.002
0.000
9.94
2.00
4.03
1.99
PPM
10
8
6
4
2
0
DFILE SH09122601181c-141(
COMNT c-31+2-phenylEtOH,R
DATIM Sat Dec 26 05:12:41
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12

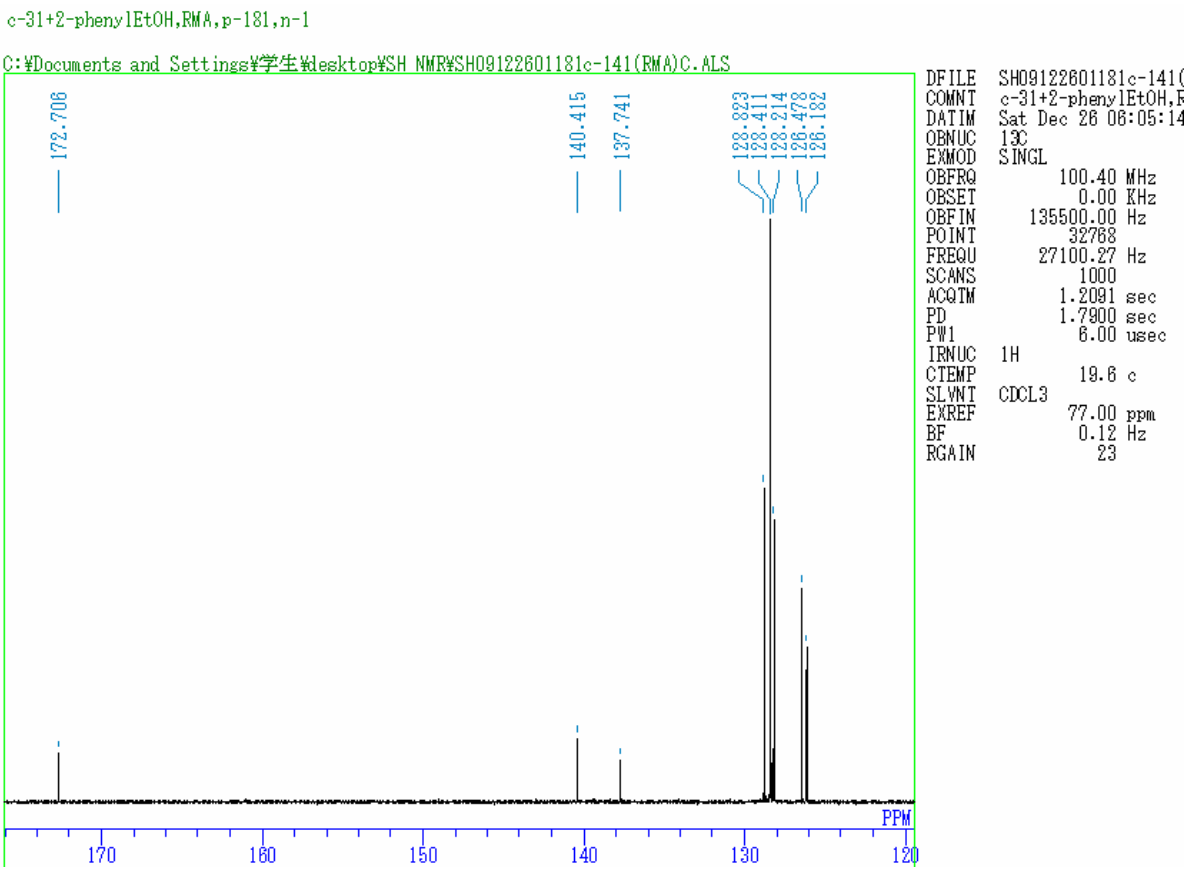

c-31+2-phenylEtOH,RMA,p-181,n-1
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH09122601181c-141(RMA).ALS
4.217
4.209
4.199
4.191
4.182
4.174
2.848
2.828
2.817
2.809
2.800
2.792
2.543
2.535
2.523
2.516
2.515
2.504
2.496
2.00
4.03
1.99
PPM
4.5
4.0
3.5
3.0
2.5
DFILE SH09122601181c-141(
COMNT c-31+2-phenylEtOH,R
DATIM Sat Dec 26 05:12:41
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12



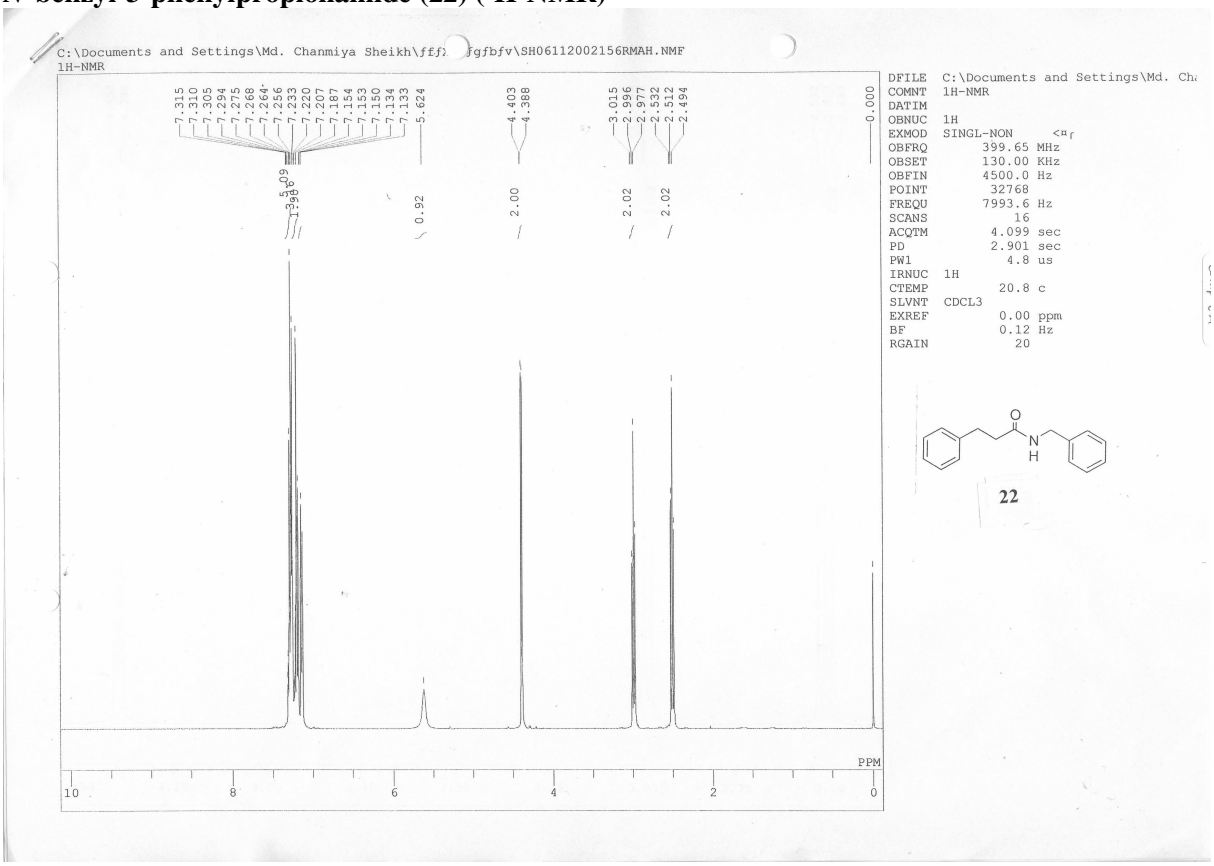
3-phenyl propionic acid 2 phenylethyl ester (21) (^{13}C-NMR)

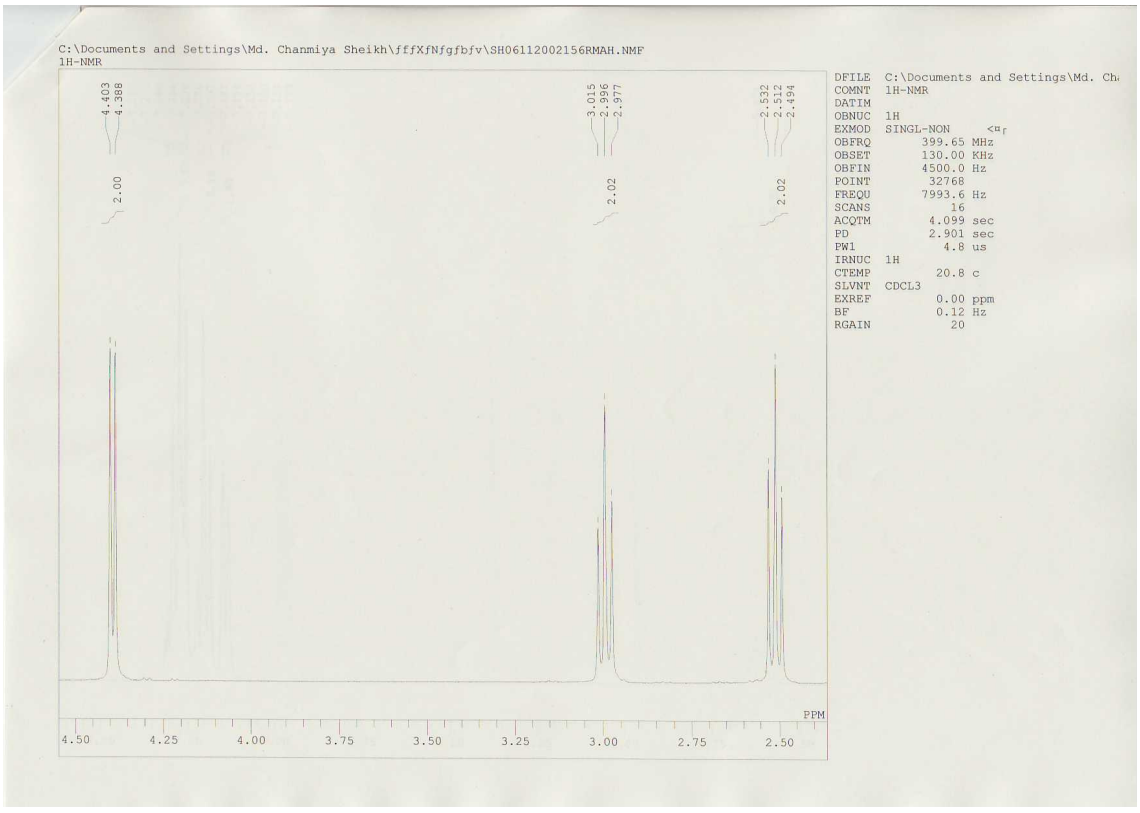

c-31+2-phenylEtOH,RMA,p-181,n-1
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH09122601181c-141(RMA)C.ALS
172.706
140.415
137.741
128.823
128.411
128.214
126.478
126.182
77.321
77.000
76.687
64.857
35.807
35.009
30.838
DFILE SH09122601181c-141(
COMNT c-31+2-phenylEtOH,F
DATIM Sat Dec 26 08:05:14
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 1000
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
PPM
200
175
150
125
100
75
50
25
0

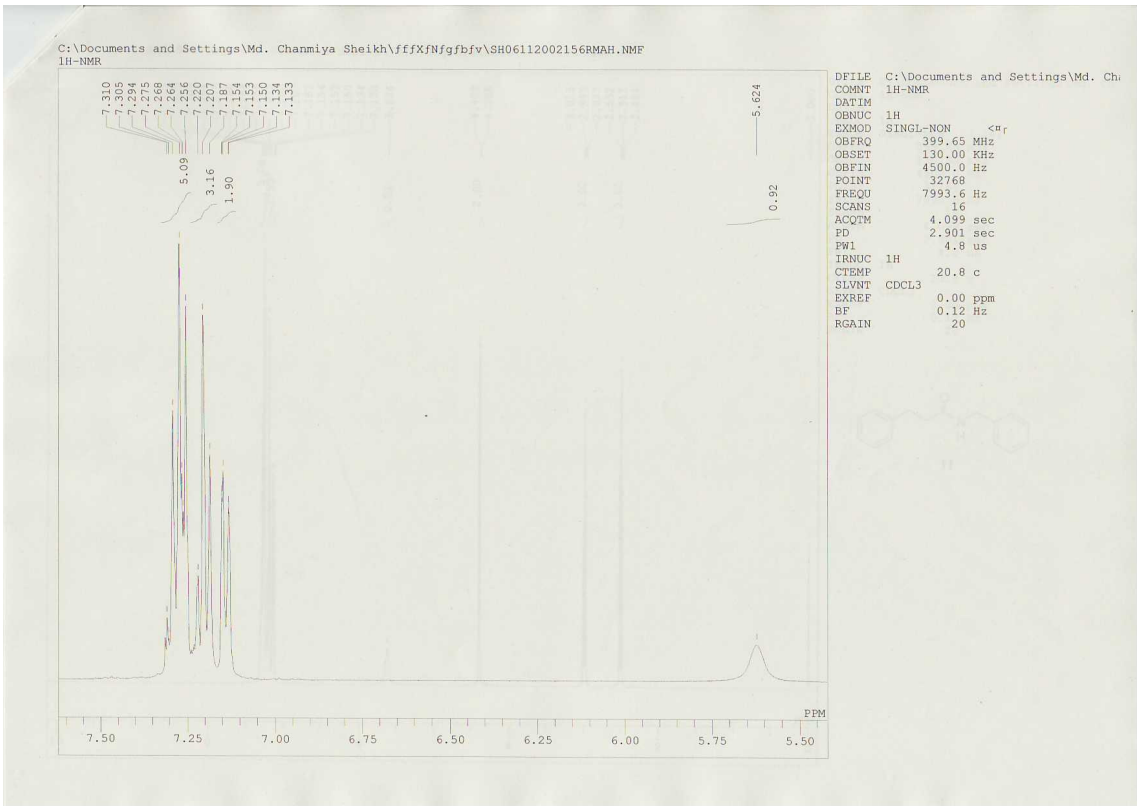

c-31+2-phenylEtOH,RMA,p-181,n-1
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH09122601181c-141(RMA)C.ALS
77.321
77.000
76.687
64.857
35.807
35.009
30.838
PPM
80
70
60
50
40
30
DFILE SH09122601181c-141(
COMNT c-31+2-phenylEtOH,R
DATIM Sat Dec 26 08:05:14
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 1000
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23


c-31+2-phenylEtOH,RMA,p-181,n-1
C:¥Documents and Settings¥学生¥desktop¥SH NMR¥SH09122601181c-141(RMA)C.ALS
172.706
140.415
137.741
128.823
128.411
128.214
126.478
126.182
PPM
170
160
150
140
130
120
DFILE SH09122601181c-141(
COMNT c-31+2-phenylEtOH,R
DATIM Sat Dec 26 08:05:14
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 1000
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

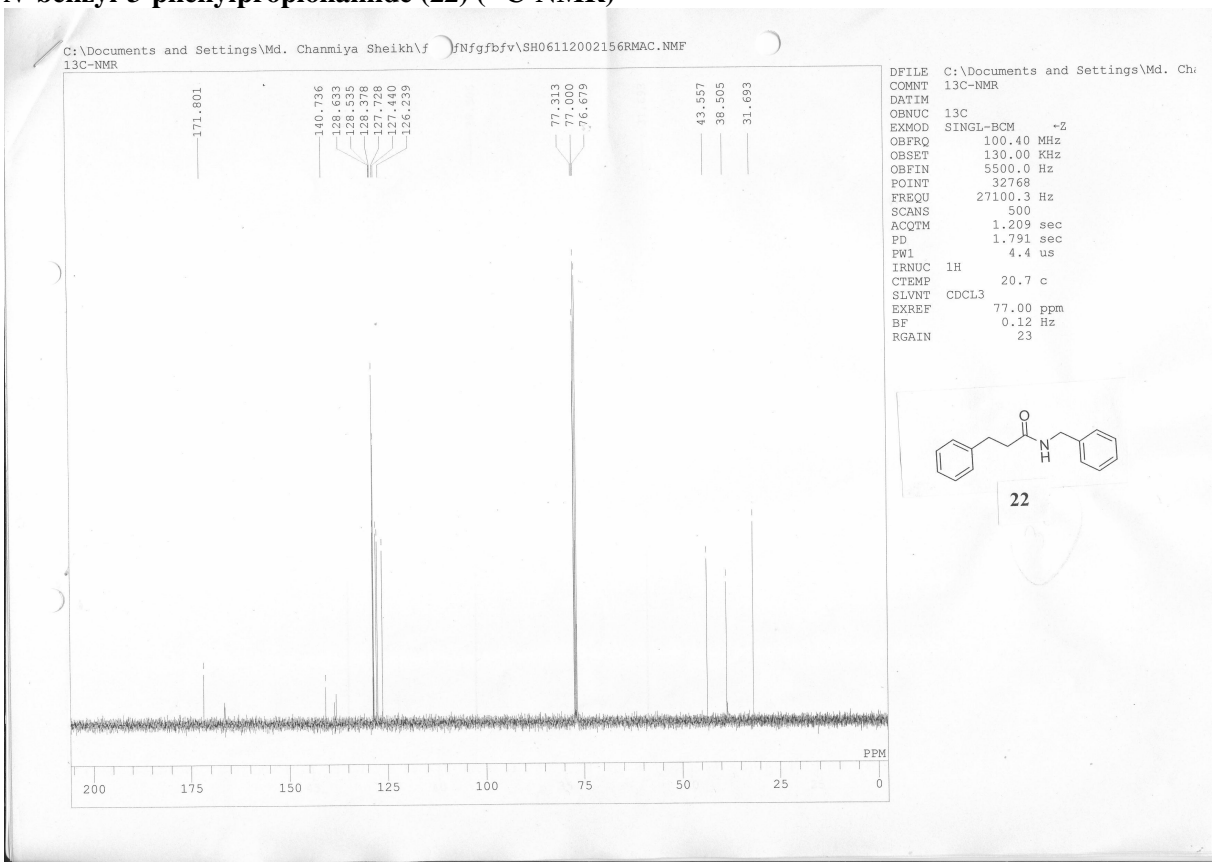
N-benzyl-3-phenylpropionamide (22) (^{1}H-NMR)

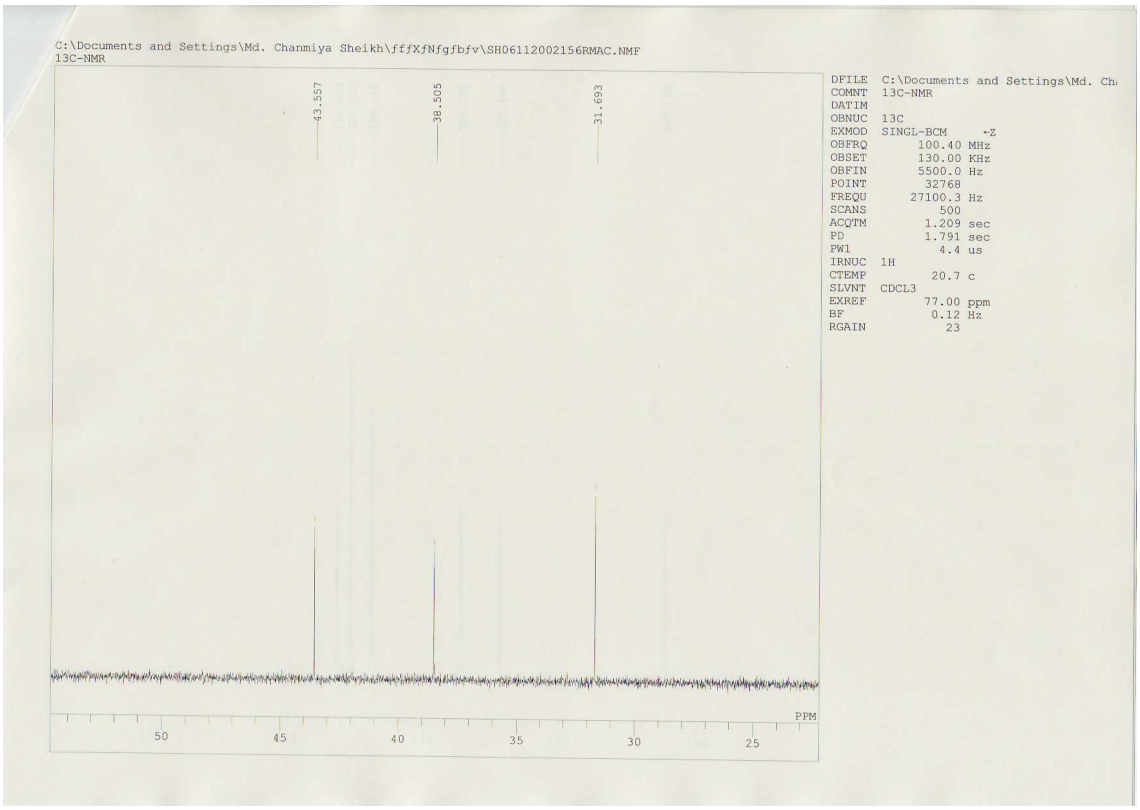






N-benzyl-3-phenylpropionamide (22) (^{13}C-NMR)

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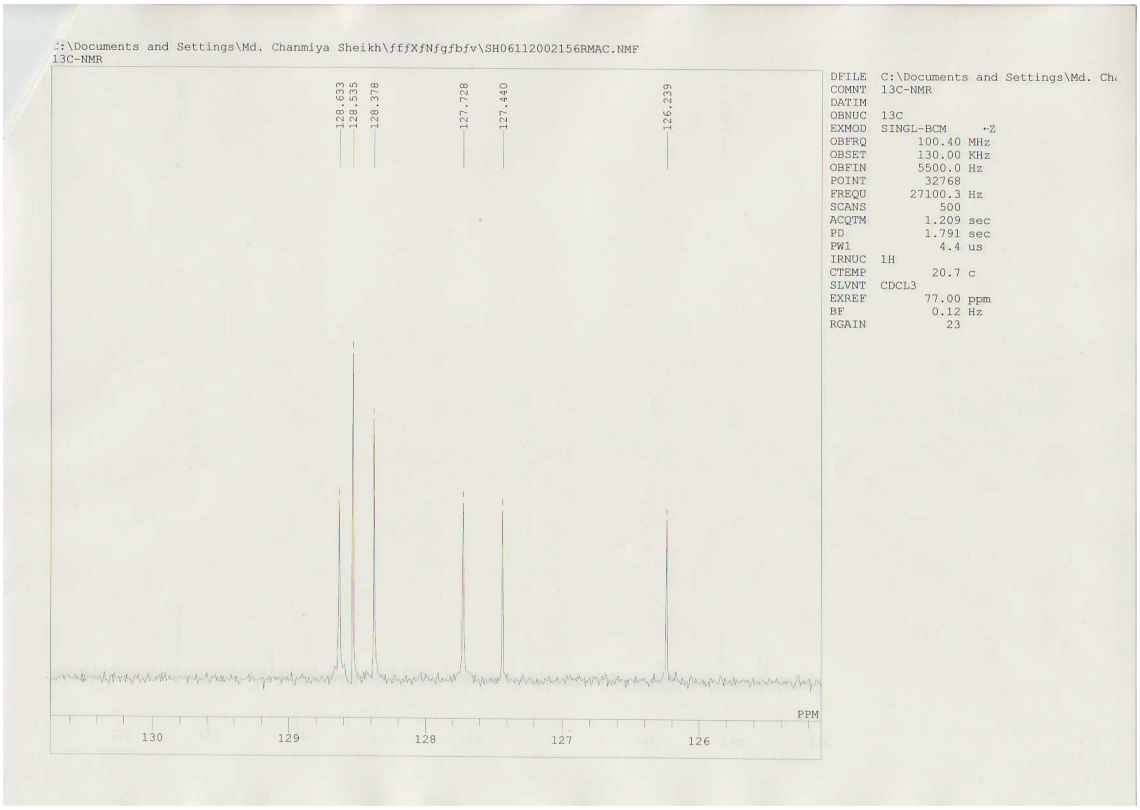
13C-NMR
43.557
38.505
31.693
PPM
50
45
40
35
30
25
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM +Z
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 500
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23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13C-NMR
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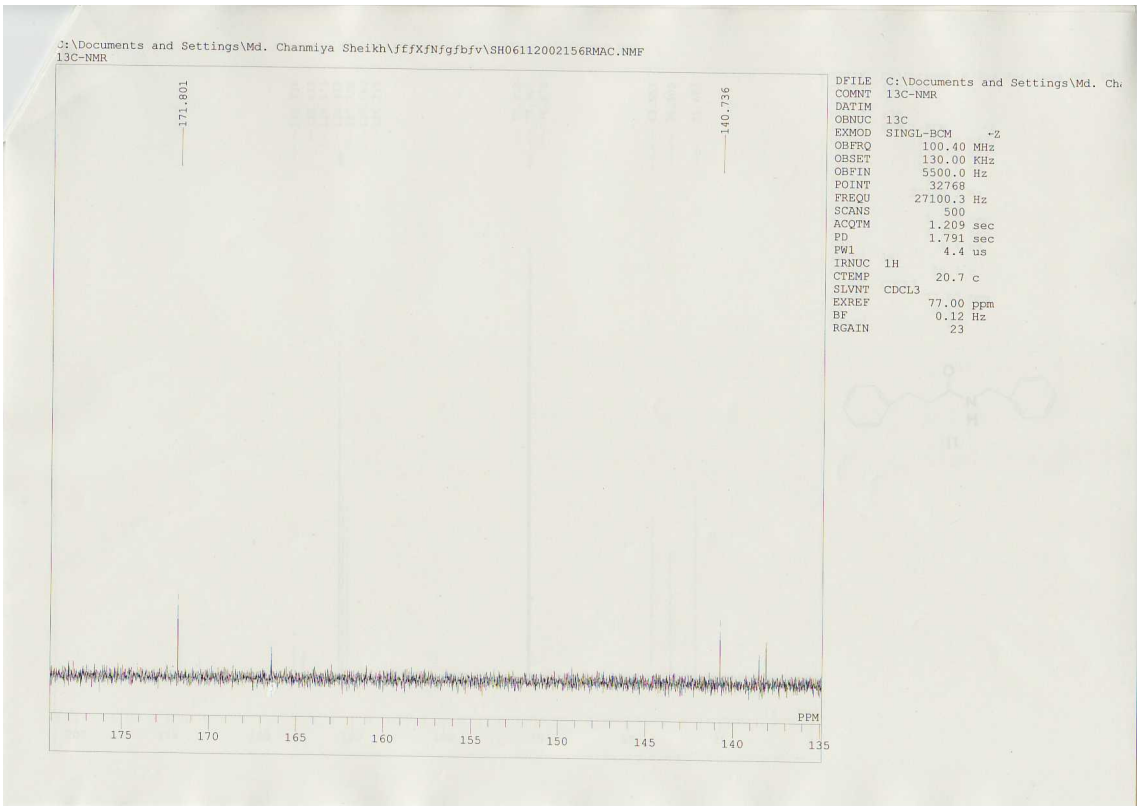
128.535
128.378
127.728
127.440
126.239
PPM
130
129
128
127
126
DFILE C:\Documents and Settings\Md. Ch
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL-BCM +Z
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 500
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23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DATIM
OBNUC 13C
EXMOD SINGL-BCM
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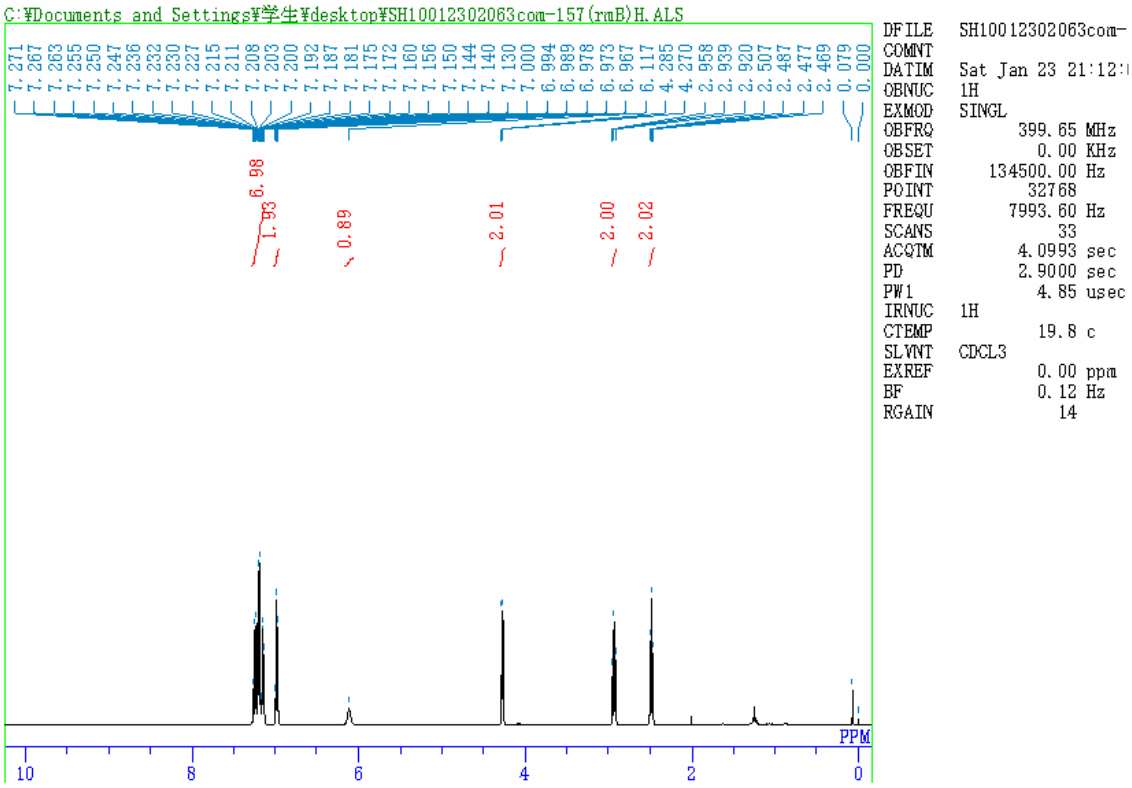
OBFIN 5500.0 Hz
POINT 32768
FREQU 27100.3 Hz
SCANS 500
ACQTM 1.209 sec
PD 1.791 sec
PW1 4.4 us
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
PPM
175
170
165
160
155
150
145
140
135

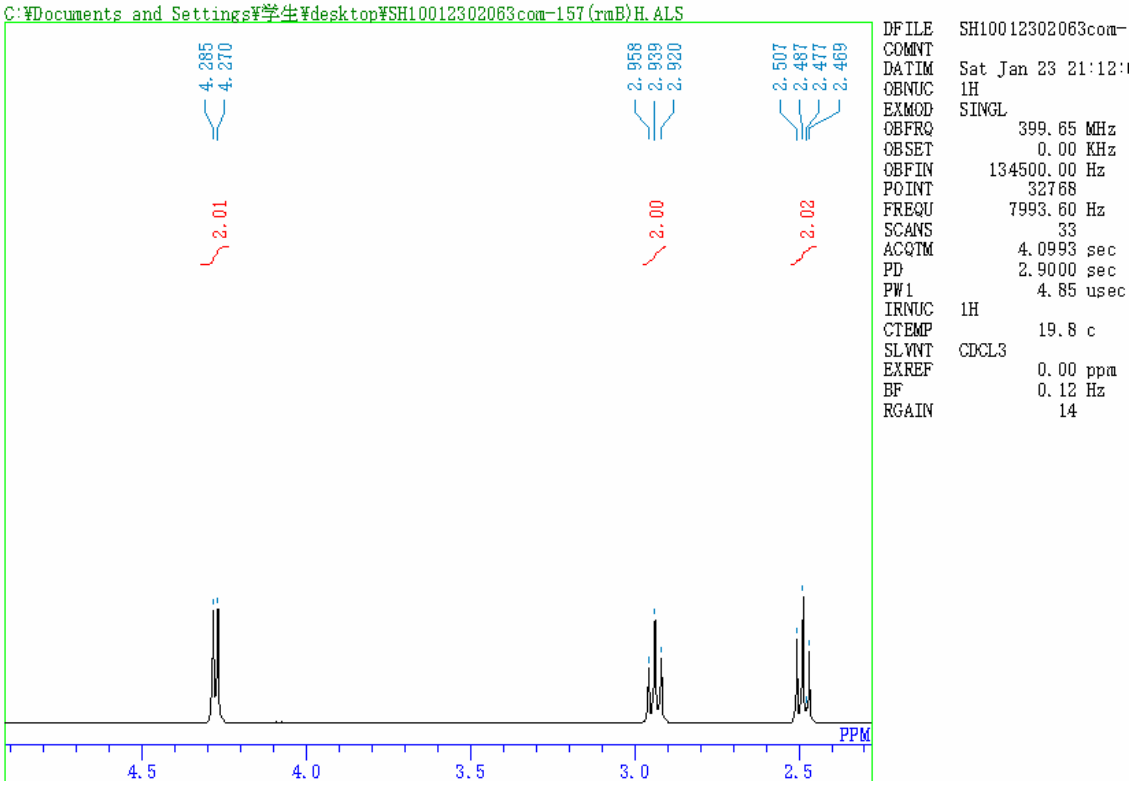
(iv) Supporting Information for the manuscript:

Title: “Syntheses and reactivities of non-symmetrical "active ester" bi-dentate cross-linking reagents having a phthalimidoyl and acid chloride, 2-benzothiazole, or 1-benzotriazole group”

(*^{1}H –NMR and ^{13}C–NMR)*

N-4-chlorobenzyl-3-phenylpropionamide (23) (^{1}H–NMR)

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2.958
2.939
2.920
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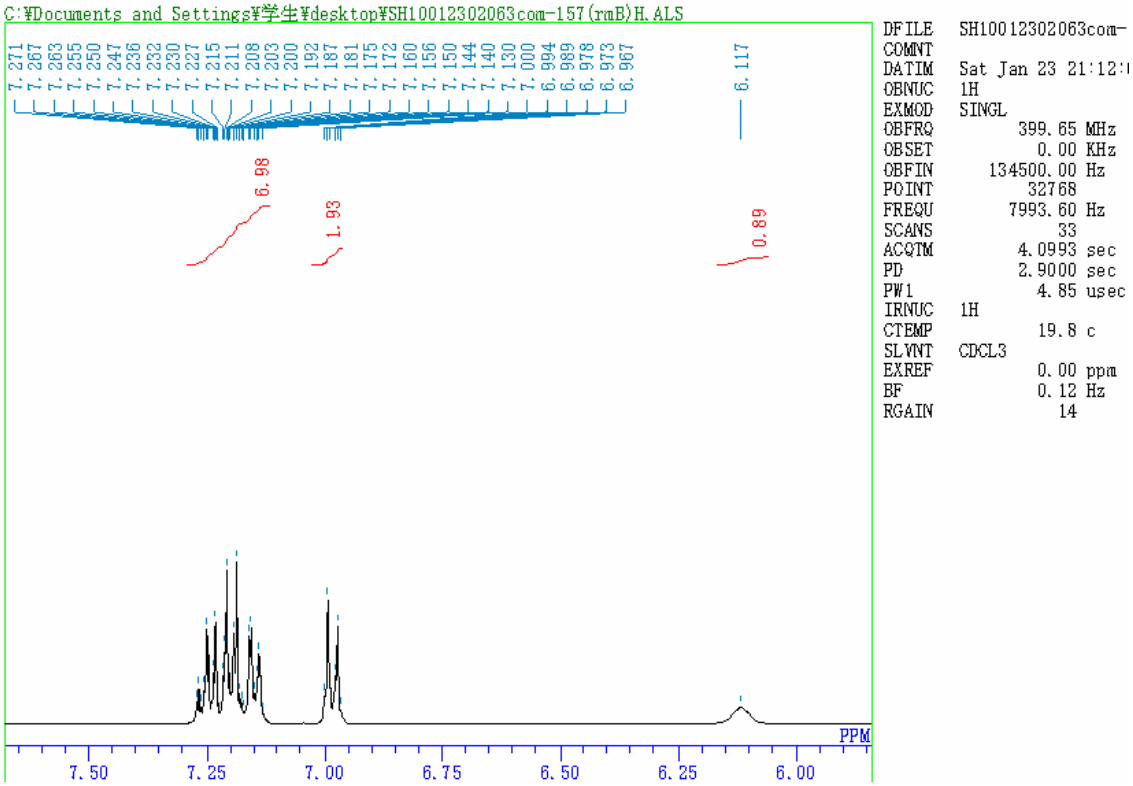
2.469
2.01
2.00
2.02
PPM
4.5
4.0
3.5
3.0
2.5
DFILE SH10012302063com-
COMNT
DATIM Sat Jan 23 21:12:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 33
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14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7.263
7.255
7.250
7.247
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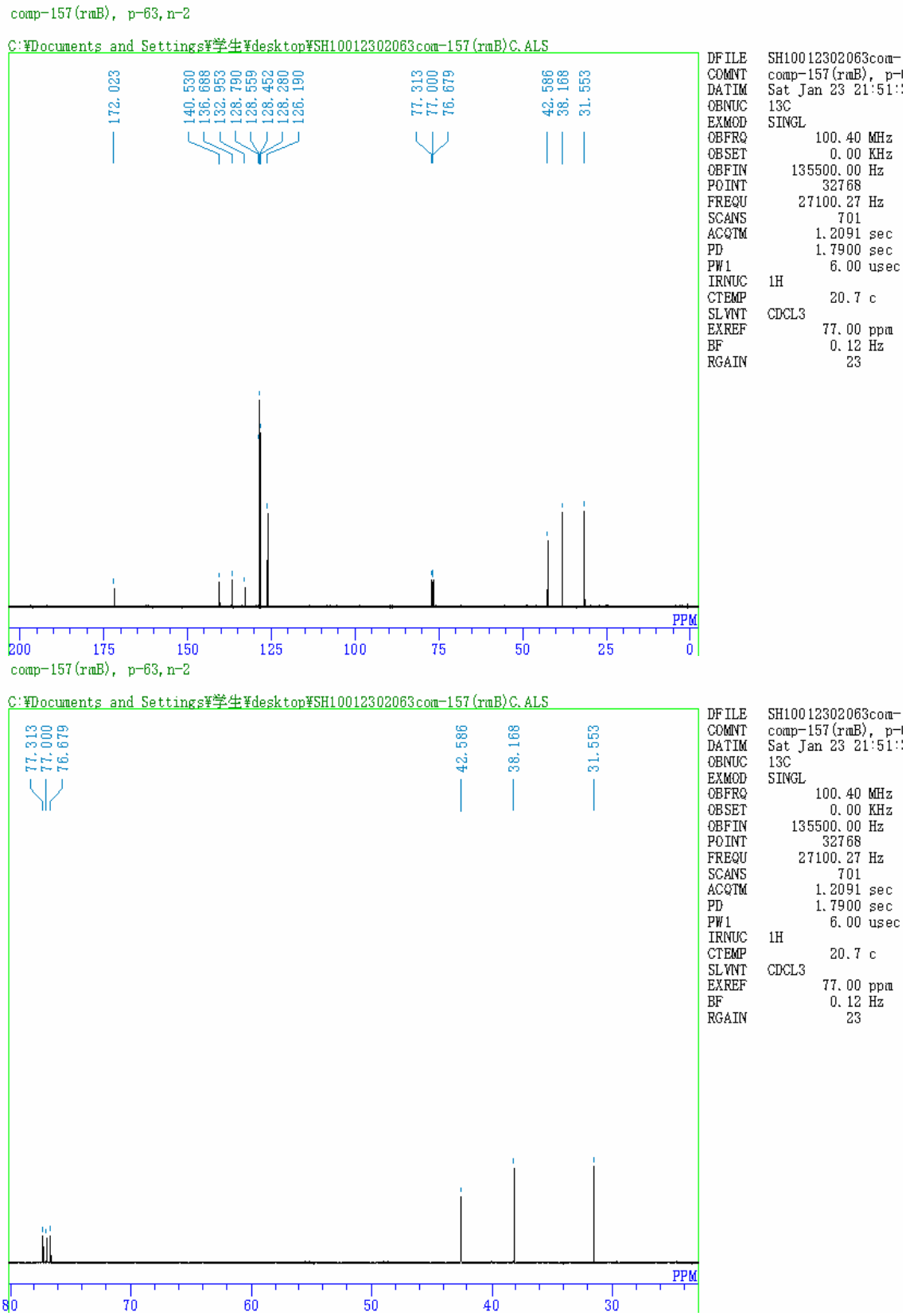
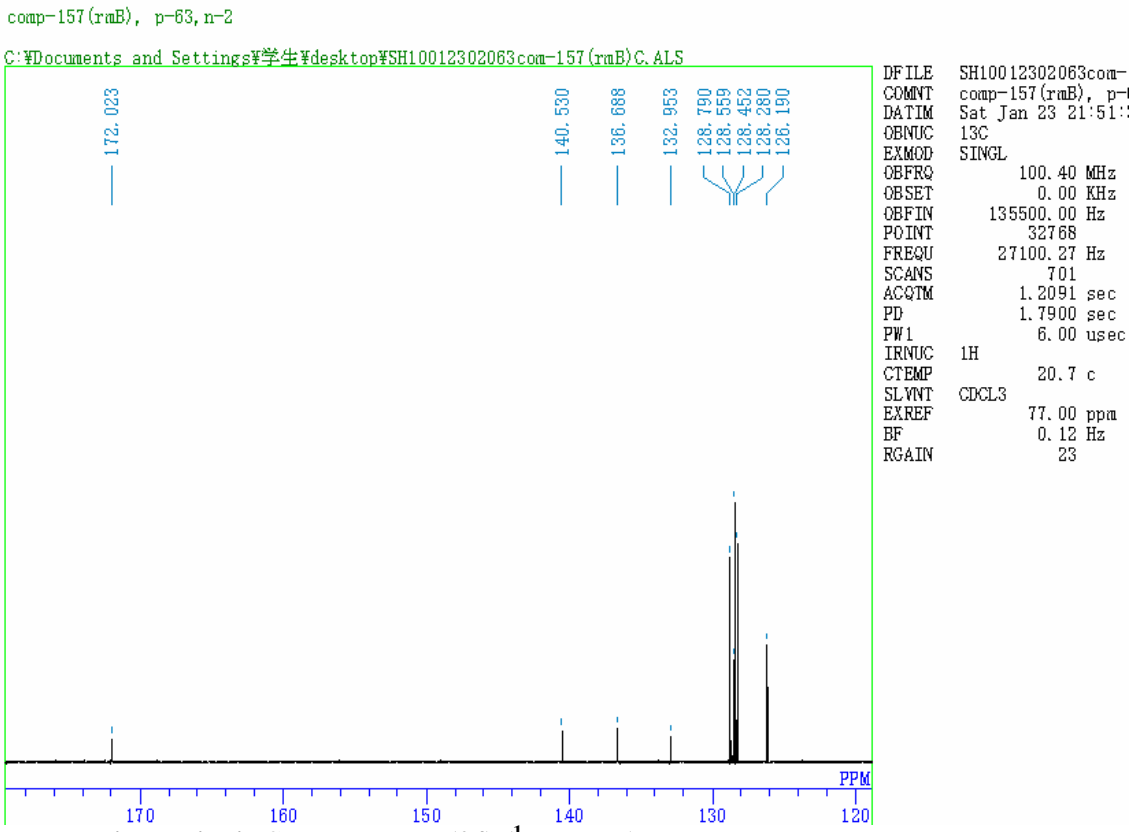
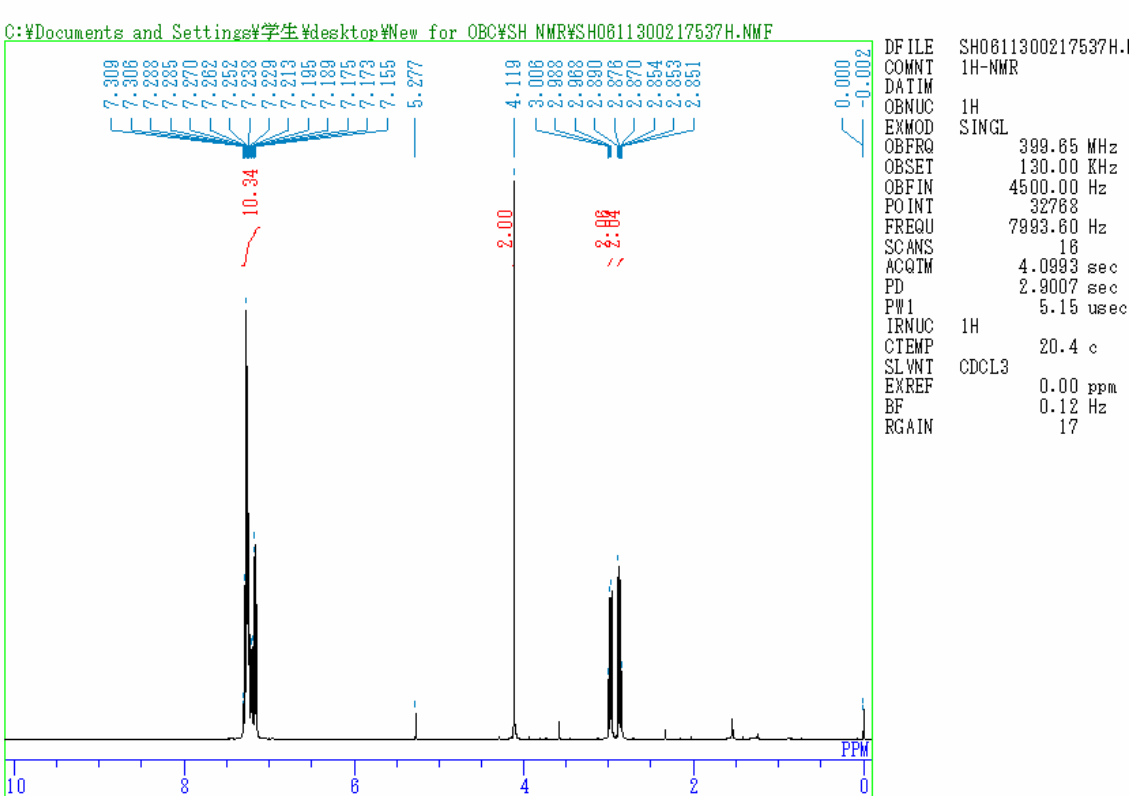
7.232
7.230
7.227
7.215
7.211
7.208
7.203
7.200
7.192
7.187
7.181
7.175
7.172
7.160
7.156
7.150
7.144
7.140
7.130
7.000
6.994
6.989
6.978
6.973
6.967
6.117
6.98
1.93
0.89
PPM
7.50
7.25
7.00
6.75
6.50
6.25
6.00
DFILE SH10012302063com-
COMNT
DATIM Sat Jan 23 21:12:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 33
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14

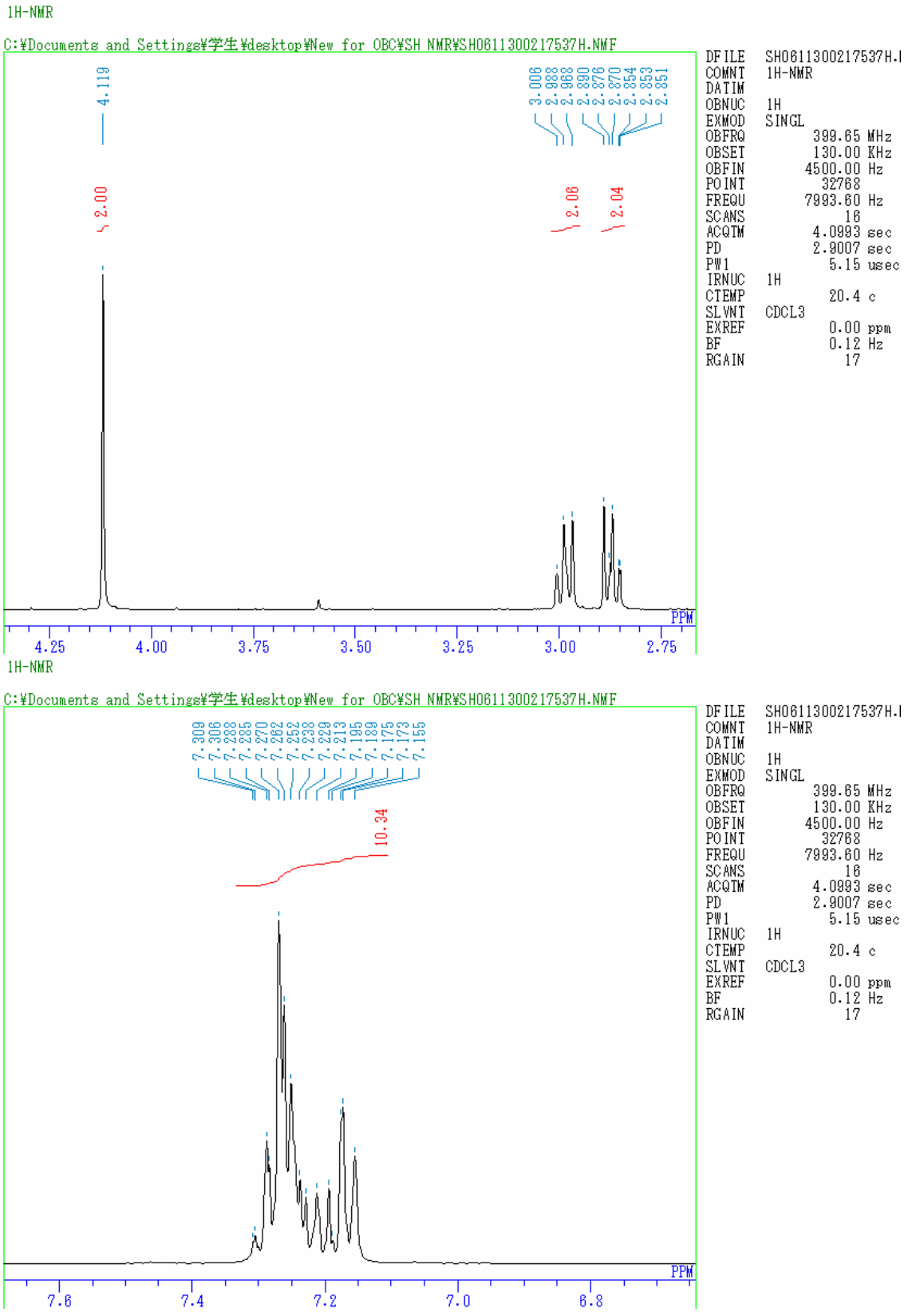
N-4-chlorobenzyl-3-phenylpropionamide (23) (^{13}C–NMR)





3-phenylthiopropionic *S*-benzyl ester (24) (^{1}H–NMR)

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2.890
2.876
2.870
2.854
2.853
2.851
2.00
2.06
2.04
PPM
4.25
4.00
3.75
3.50
3.25
3.00
2.75
DFILE SH0611300217537H.I
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17
1H-NMR
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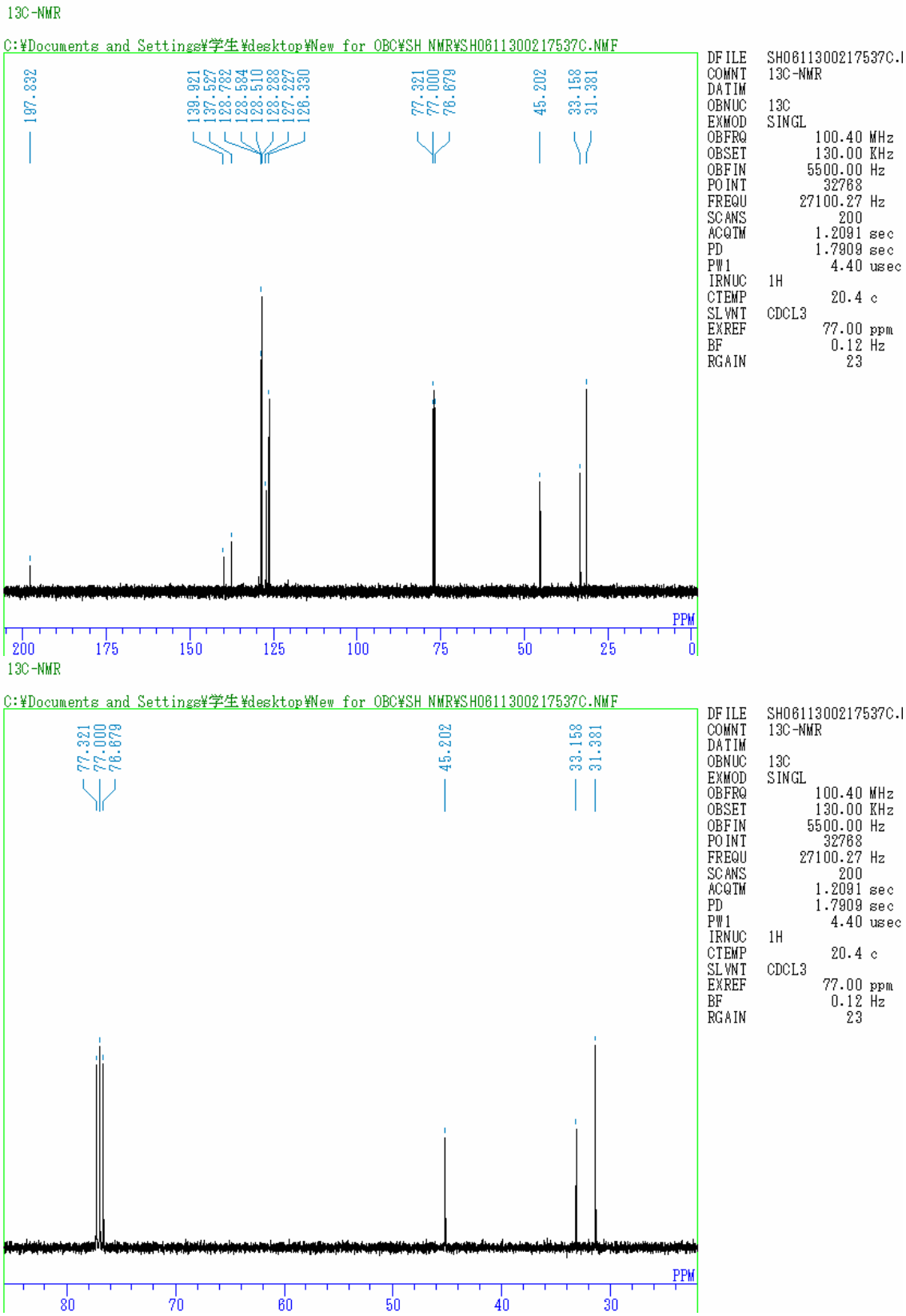
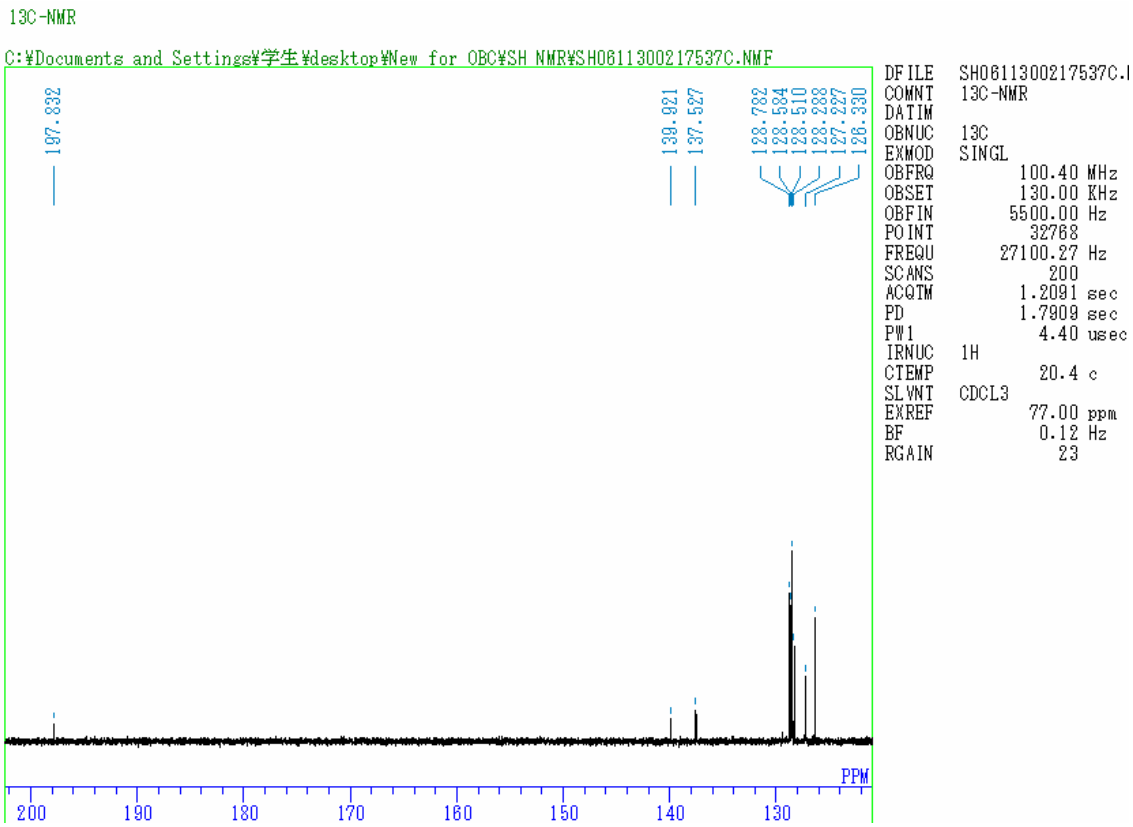
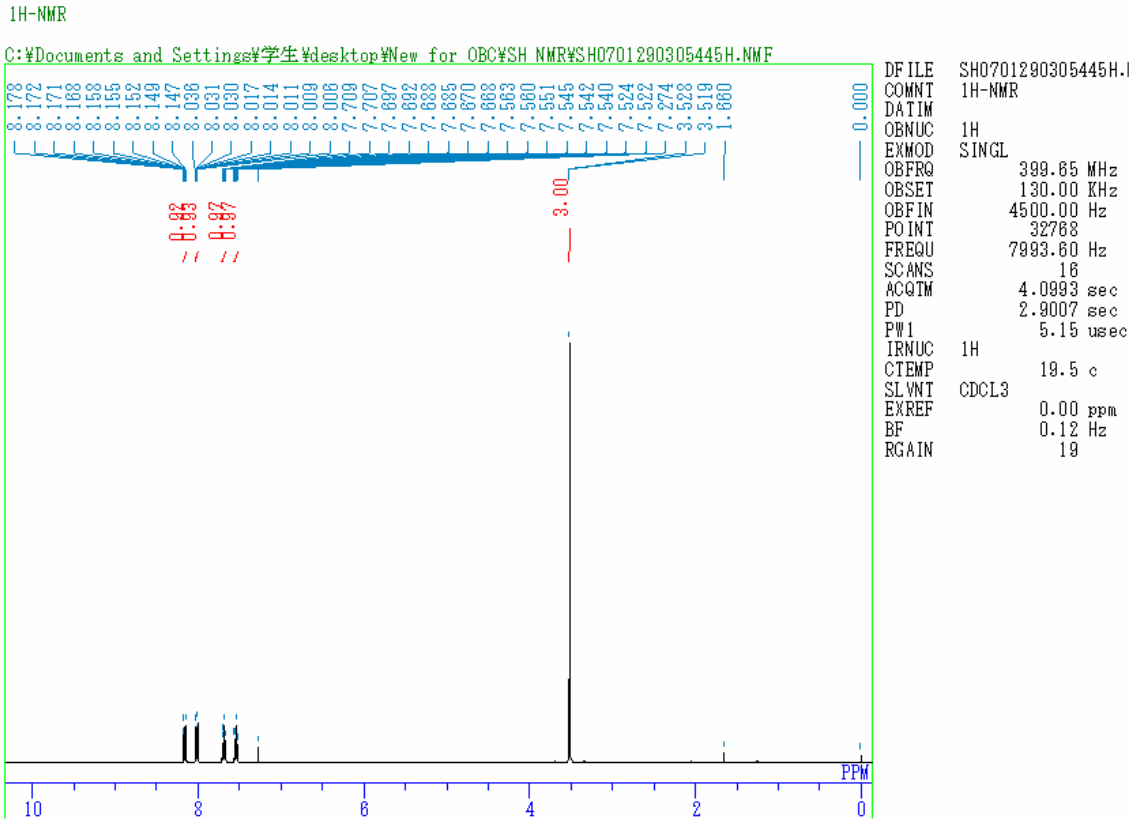
7.306
7.288
7.285
7.270
7.262
7.252
7.238
7.229
7.213
7.195
7.189
7.175
7.173
7.155
10.34
PPM
7.6
7.4
7.2
7.0
6.8
DFILE SH0611300217537H.I
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

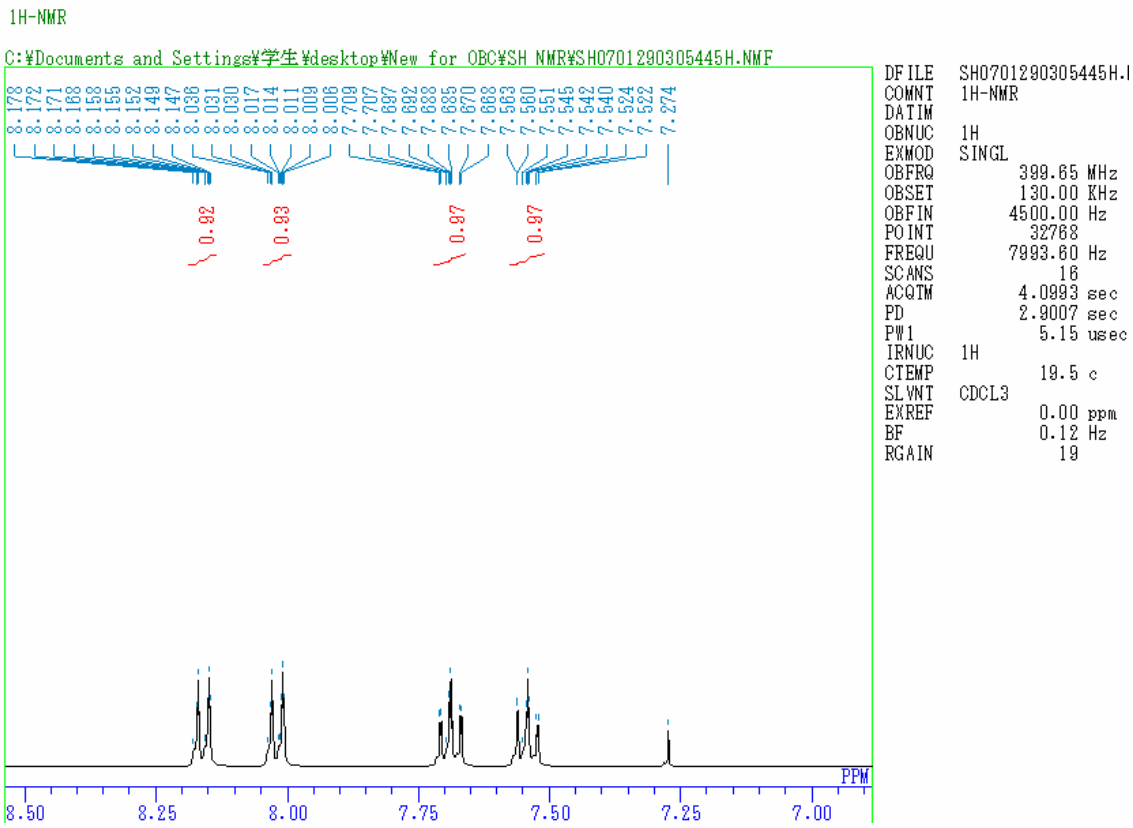
3-phenylthiopropionic *S*-benzyl ester (24) (^{13}C–NMR)



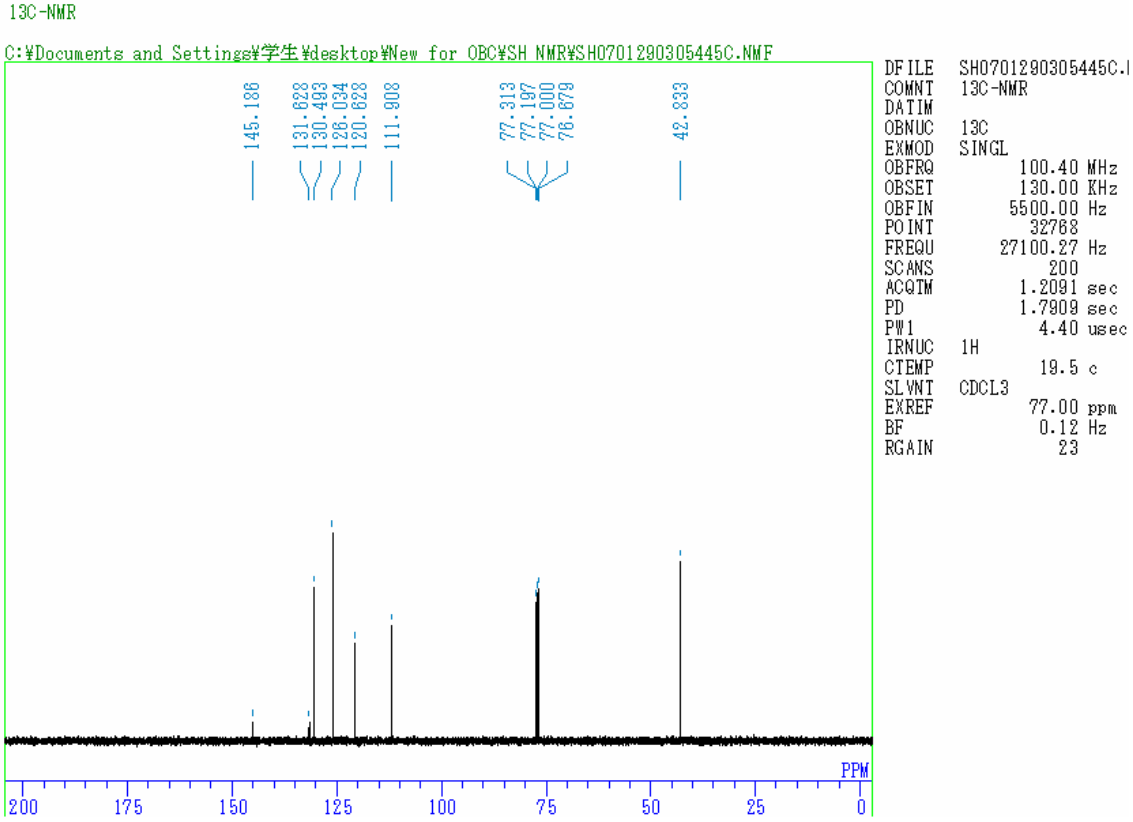


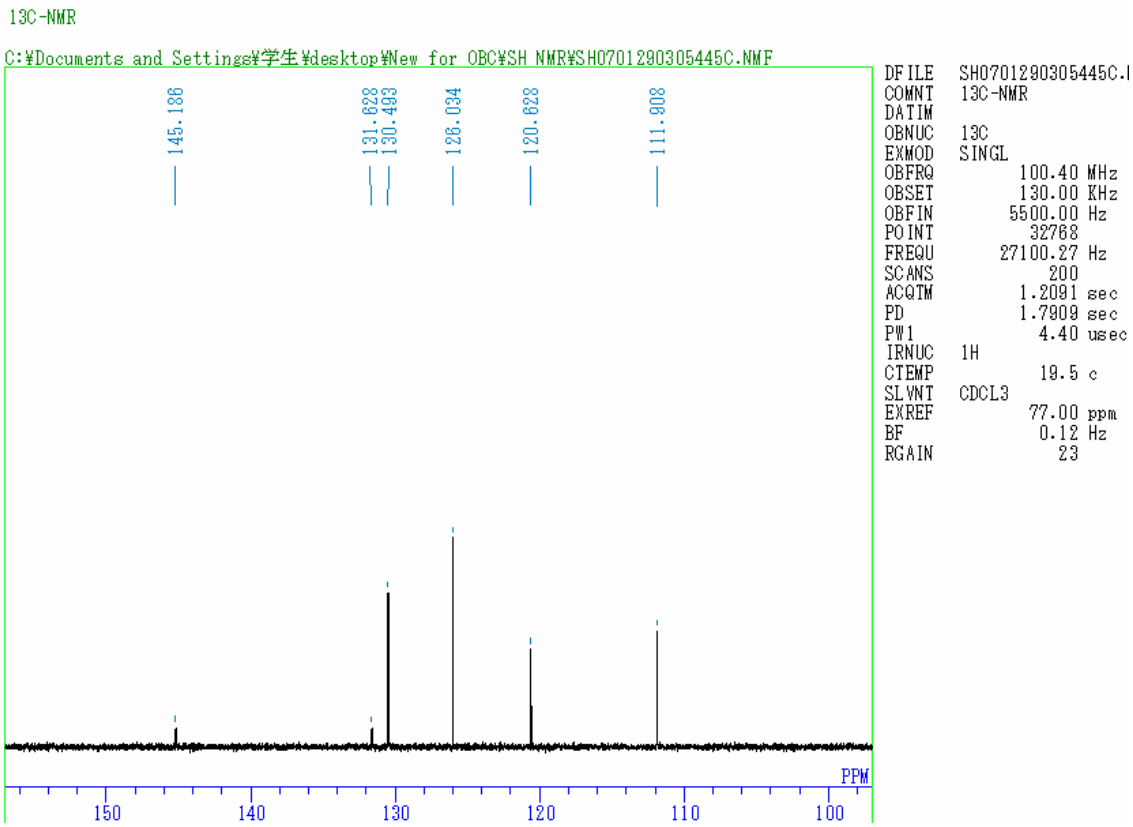
1-(methanesulfonyl)benzotriazole (26) (^{1}H–NMR)



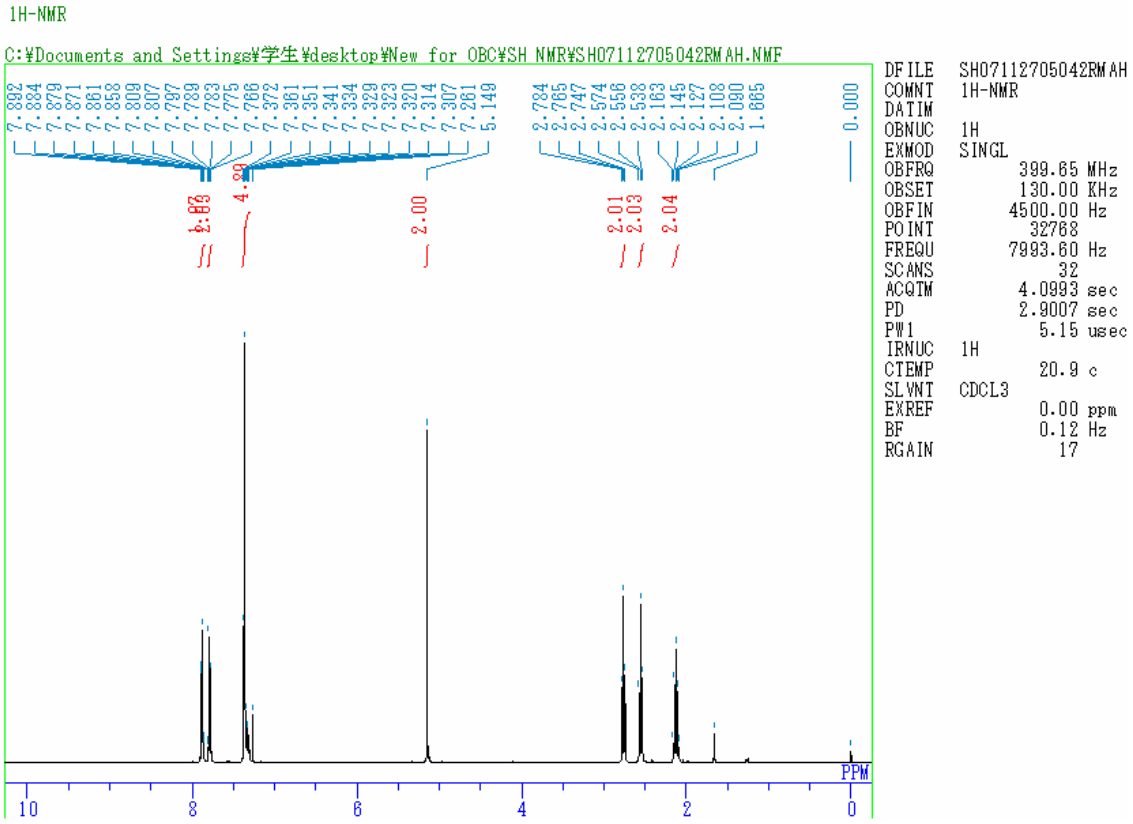


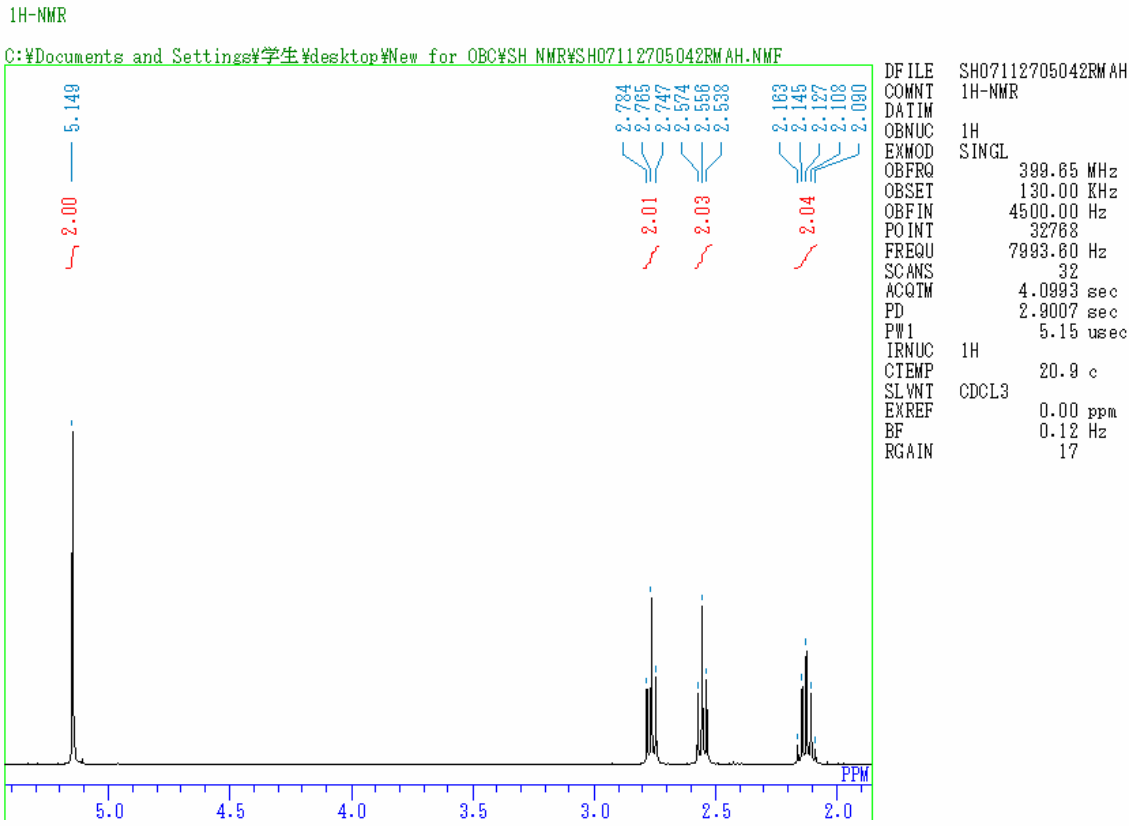
1-(methanesulfonyl)benzotriazole (26) (^{13}C–NMR)





Phthalimido 4-benzyloxycarbonylbutanoate (27) (^{1}H–NMR)

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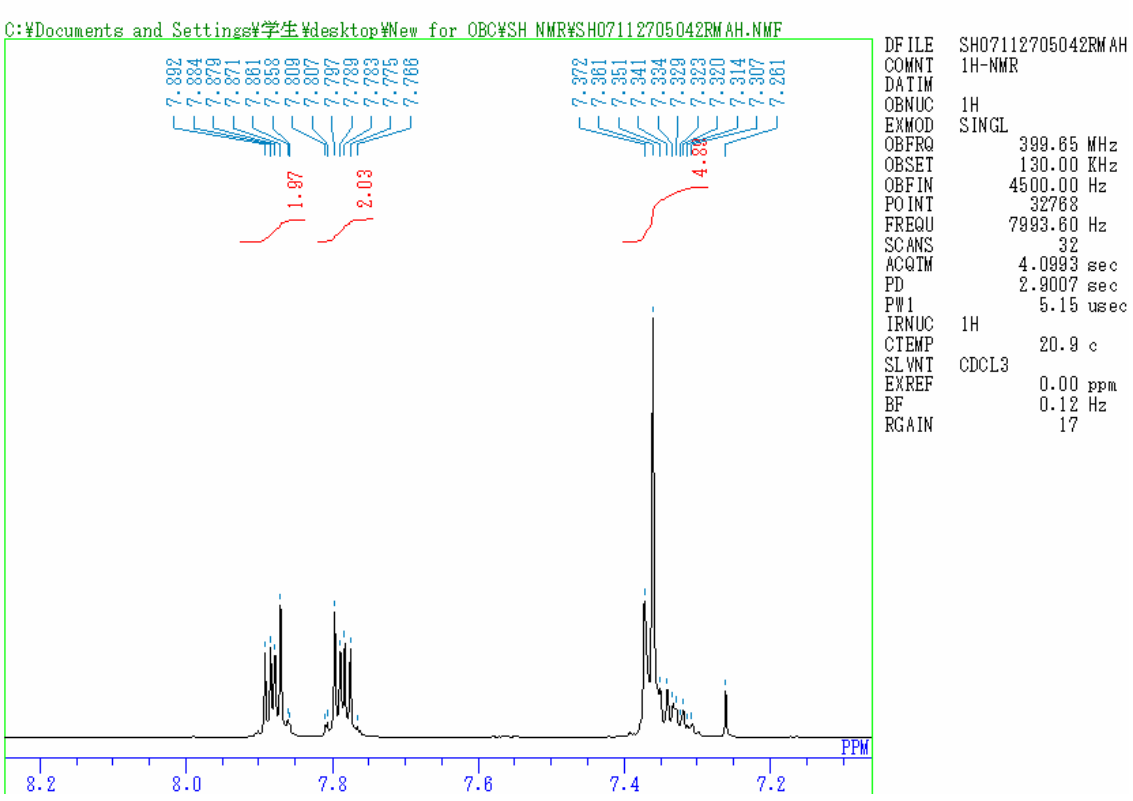
2.538
2.163
2.145
2.127
2.108
2.090
2.00
2.01
2.03
2.04
PPM
5.0
4.5
4.0
3.5
3.0
2.5
2.0
DFILE SH07112705042RMAH
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17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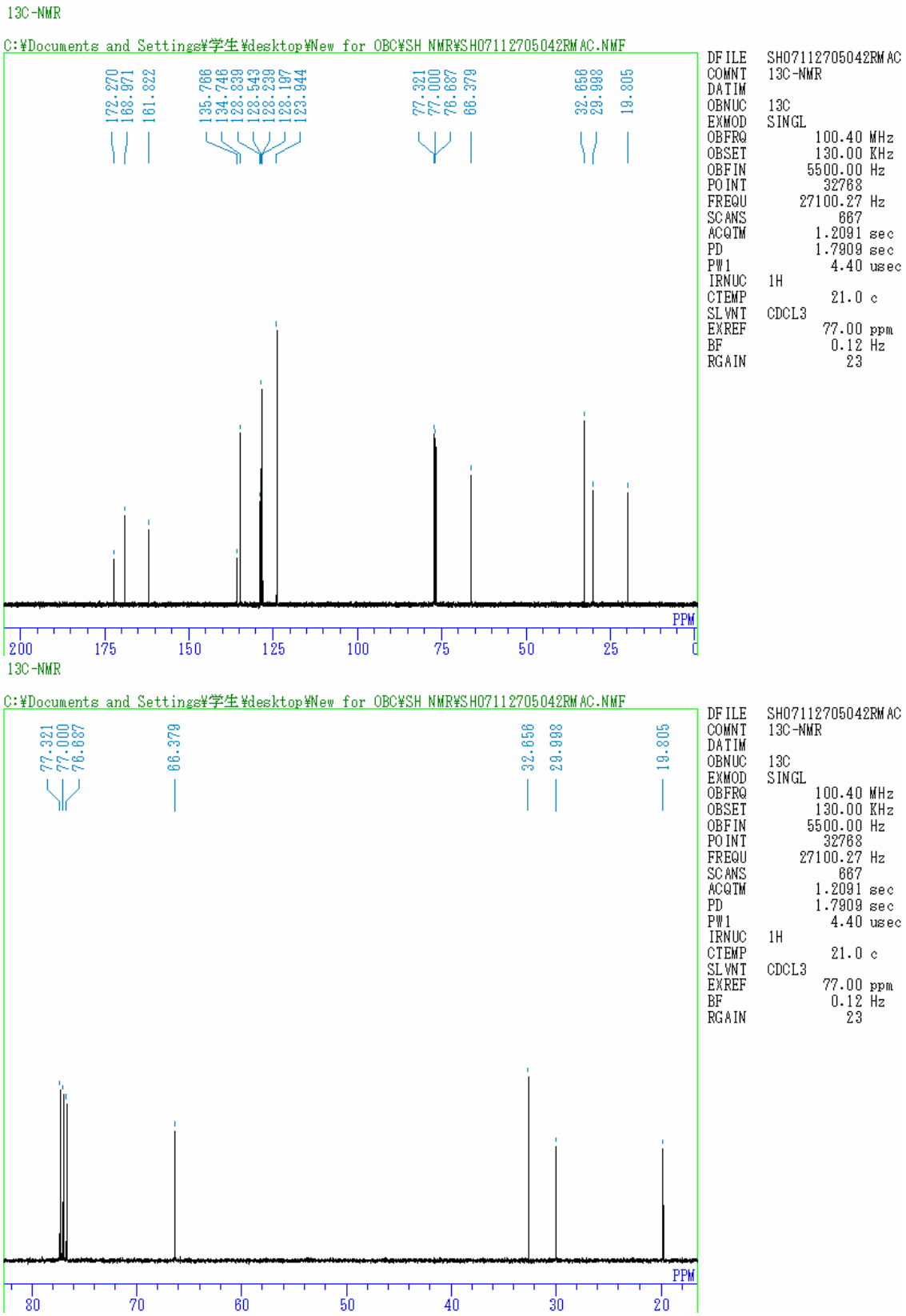
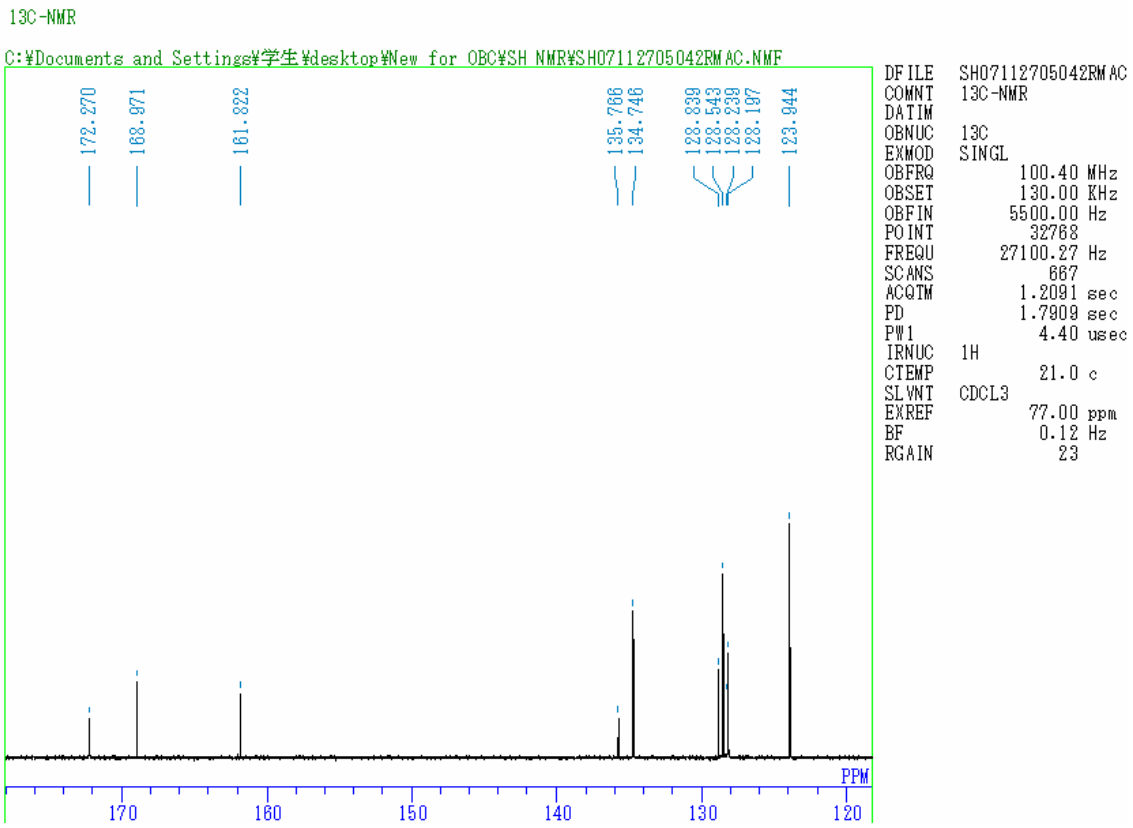
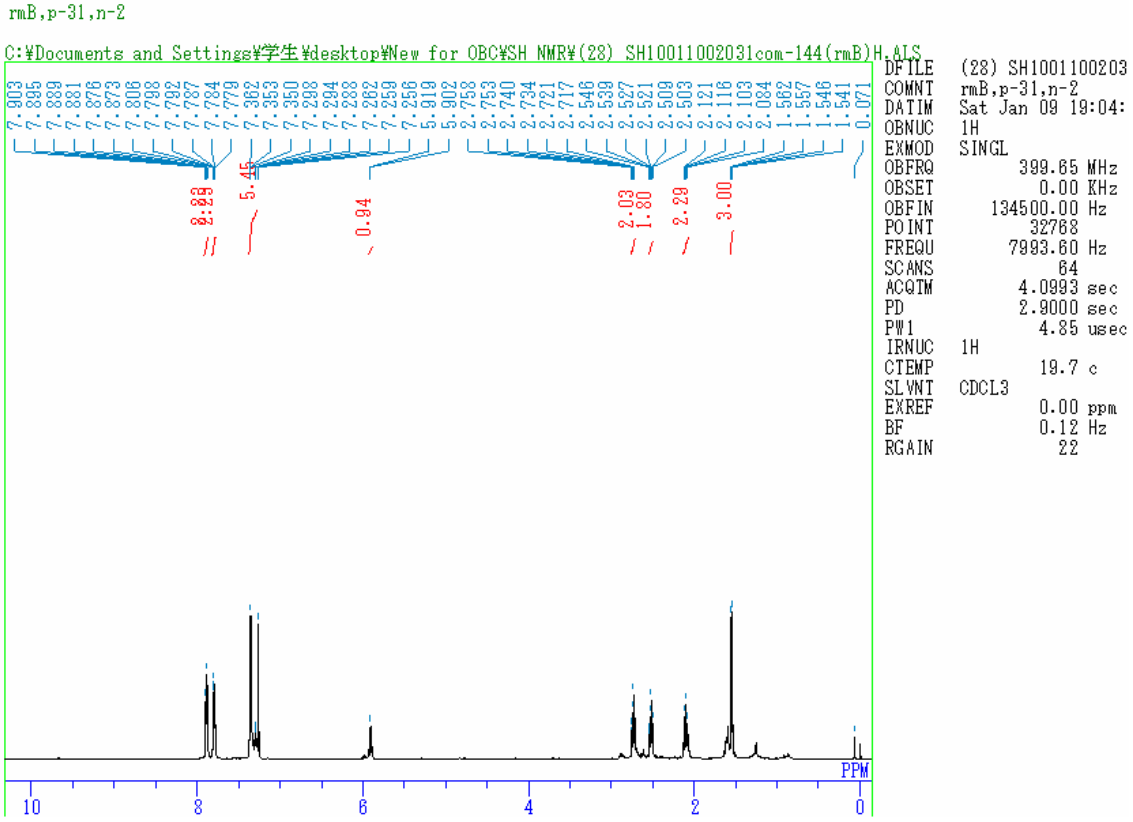
7.871
7.861
7.858
7.809
7.807
7.797
7.789
7.783
7.775
7.766
7.372
7.361
7.351
7.341
7.334
7.329
7.323
7.320
7.314
7.307
7.261
1.97
2.03
4.88
PPM
8.2
8.0
7.8
7.6
7.4
7.2
DFILE SH07112705042RMAH
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

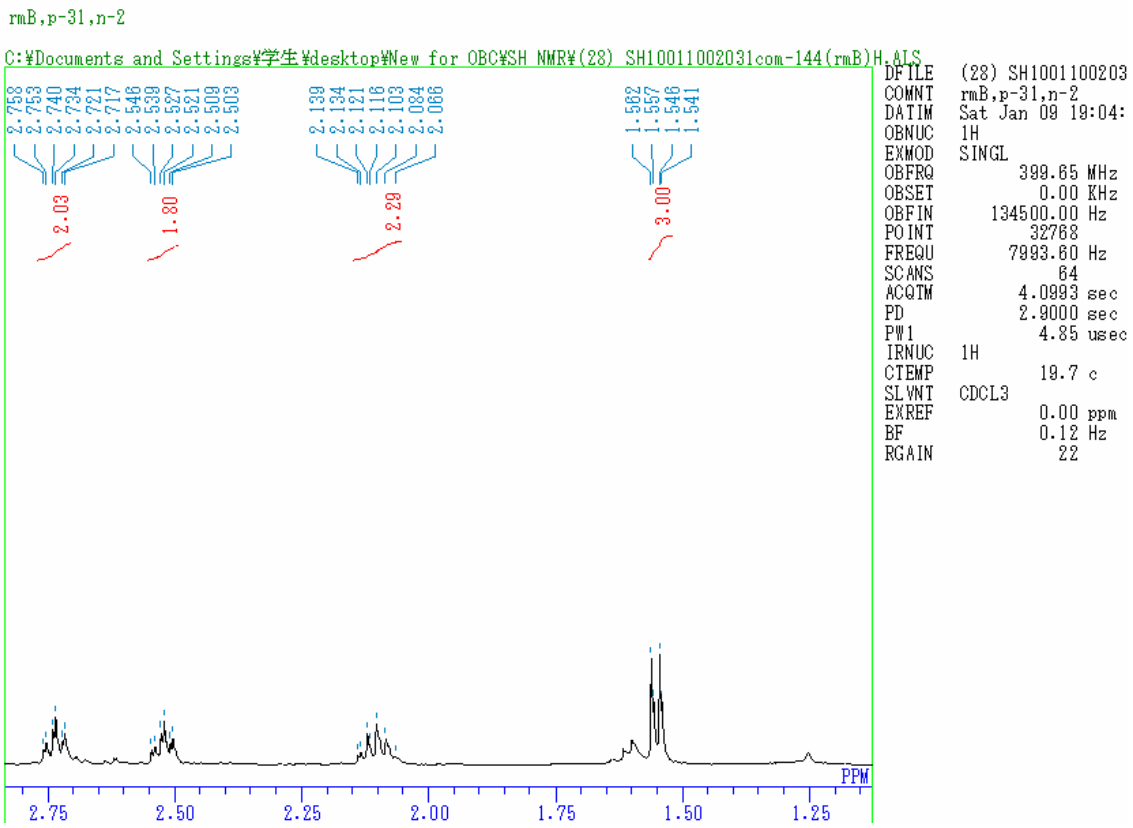
Phthalimido 4-benzyloxycarbonylbutanoate (27) (^{13}C–NMR)





Phthalimido 4-(4-methylbenzyloxycarbonyl)butanoate (28) (^{1}H–NMR)

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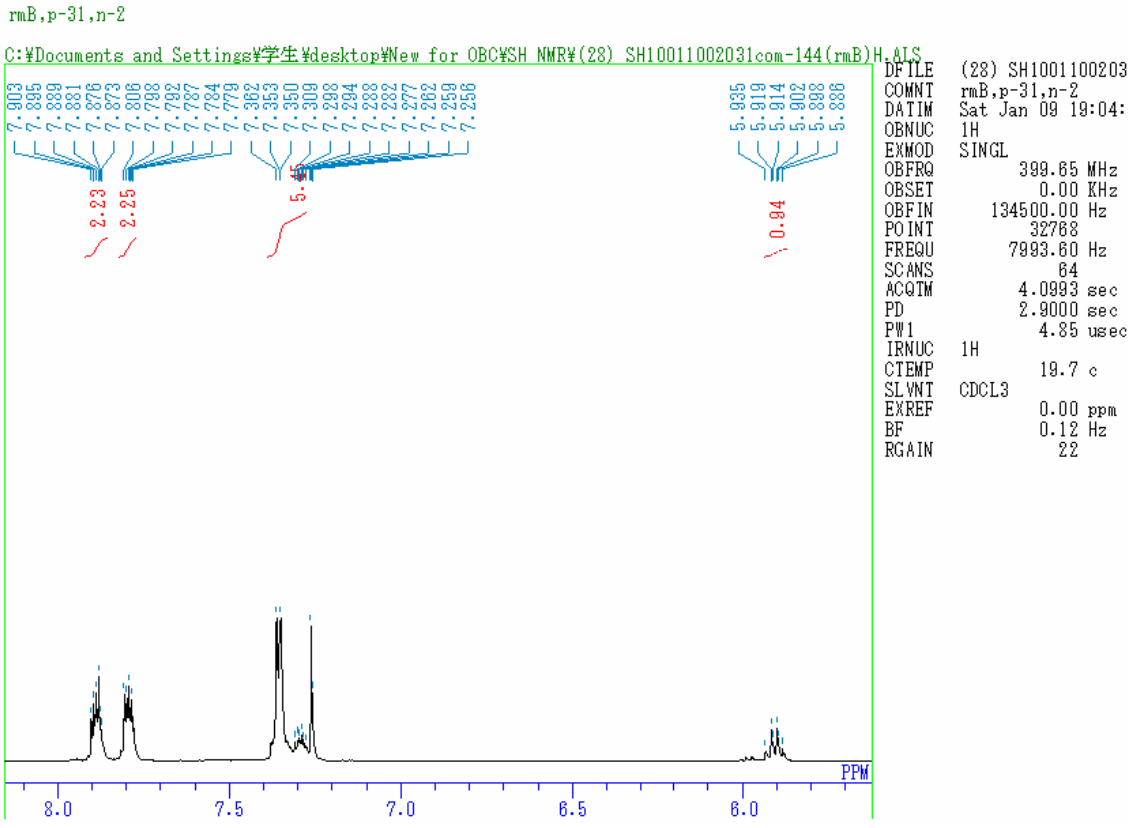
2.139 2.134 2.121 2.116 2.103 2.084 2.066
1.562 1.557 1.546 1.541
2.03
1.80
2.29
3.00
PPM
2.75 2.50 2.25 2.00 1.75 1.50 1.25
DFILE (28) SH1001100203
COMNT rmB,p-31,n-2
DATIM Sat Jan 09 19:04:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22


rmB,p-31,n-2
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5.935 5.919 5.914 5.902 5.898 5.886
2.23
2.25
5.
0.94
PPM
8.0 7.5 7.0 6.5 6.0
DFILE (28) SH1001100203
COMNT rmB,p-31,n-2
DATIM Sat Jan 09 19:04:
OBNUC 1H
EXMOD SINGL
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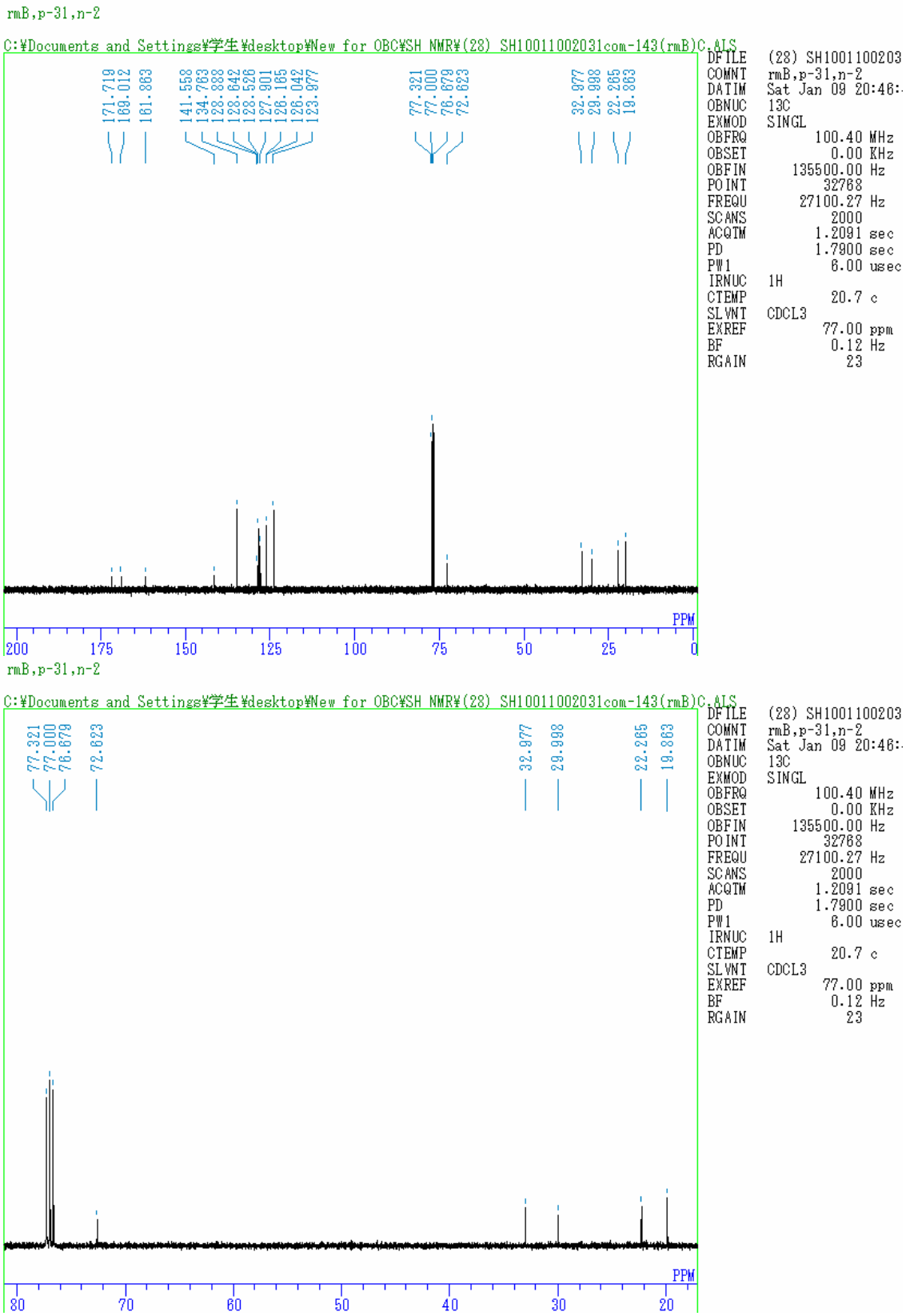
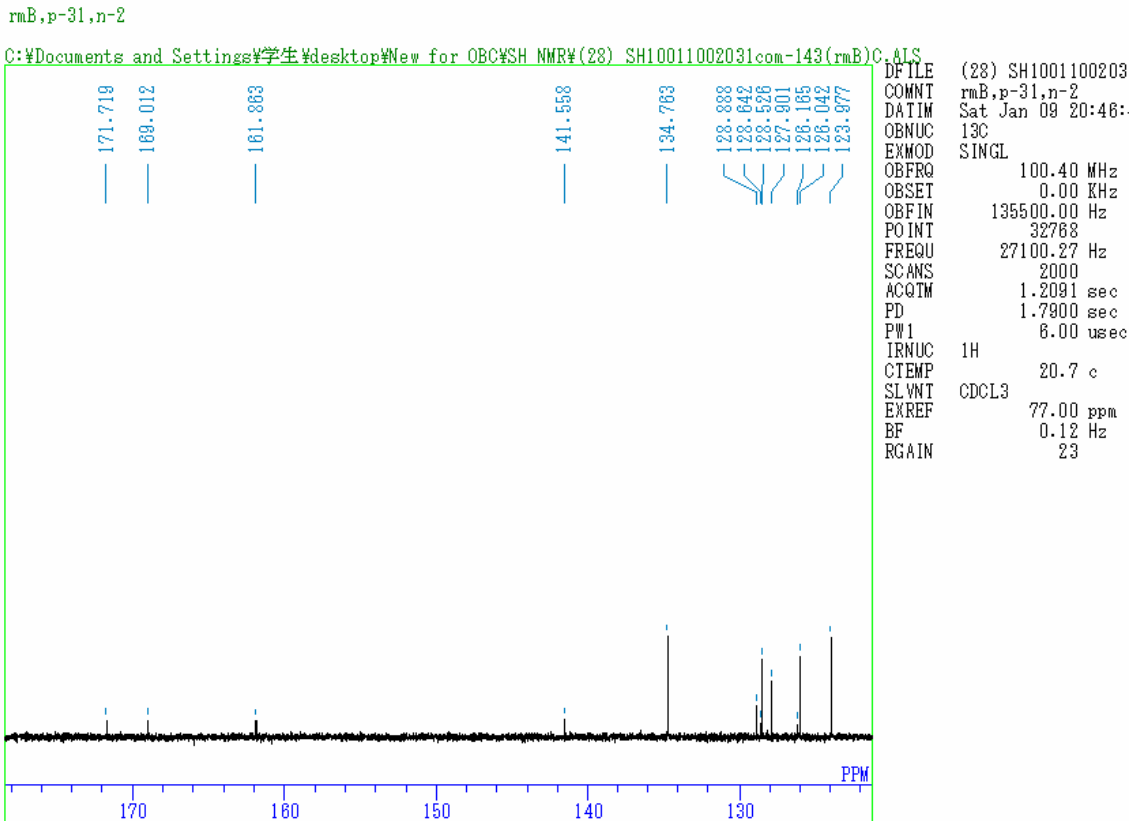
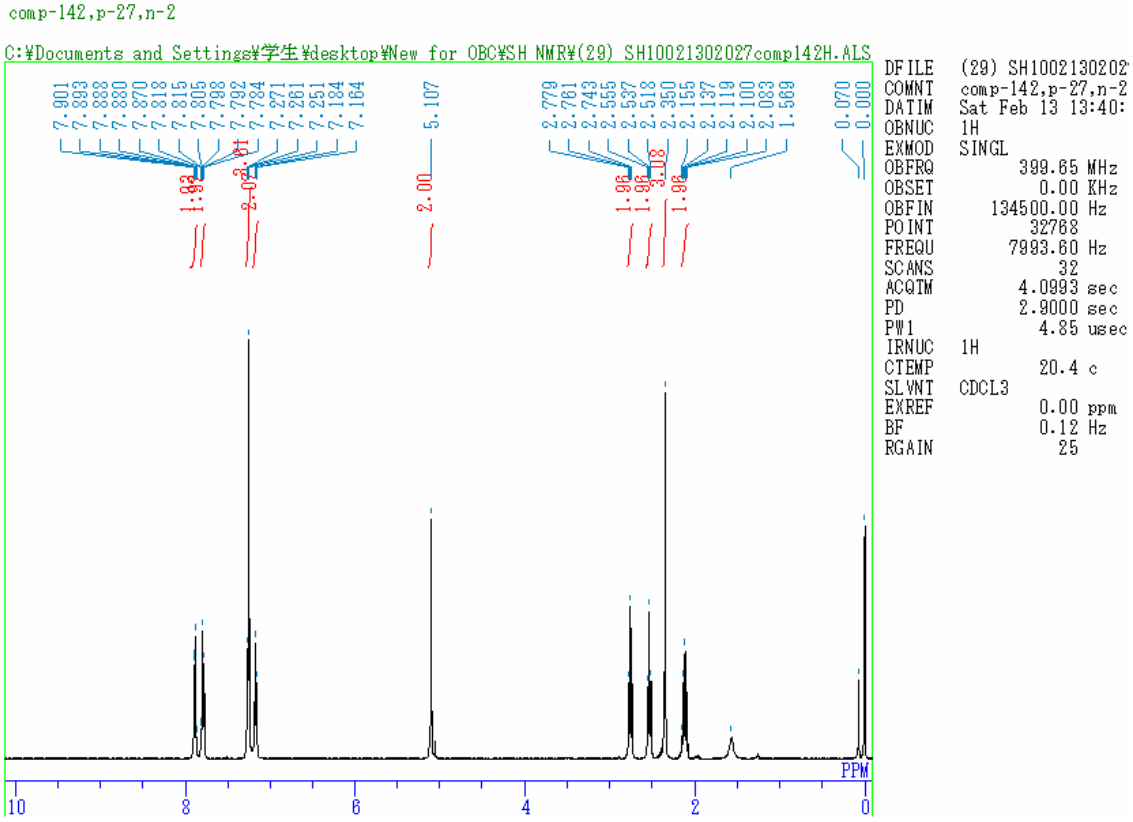
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22

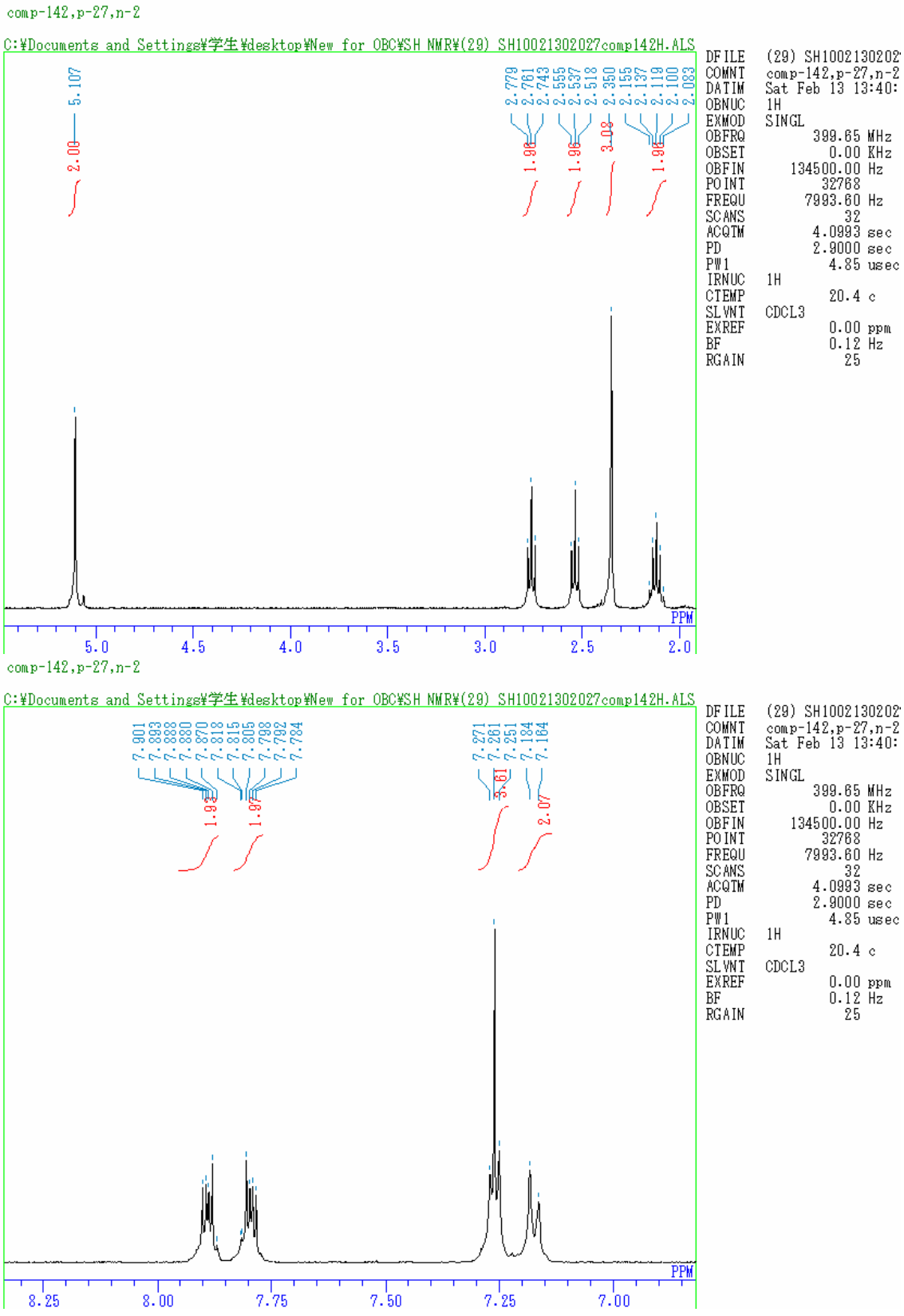
Phthalimido 4-(4-methylbenzyloxycarbonyl)butanoate (28) (^{13}C–NMR)





Phthalimido 4-(1-methylbenzyloxycarbonyl)butanoate (29) (^{1}H–NMR)




comp-142,p-27,n-2
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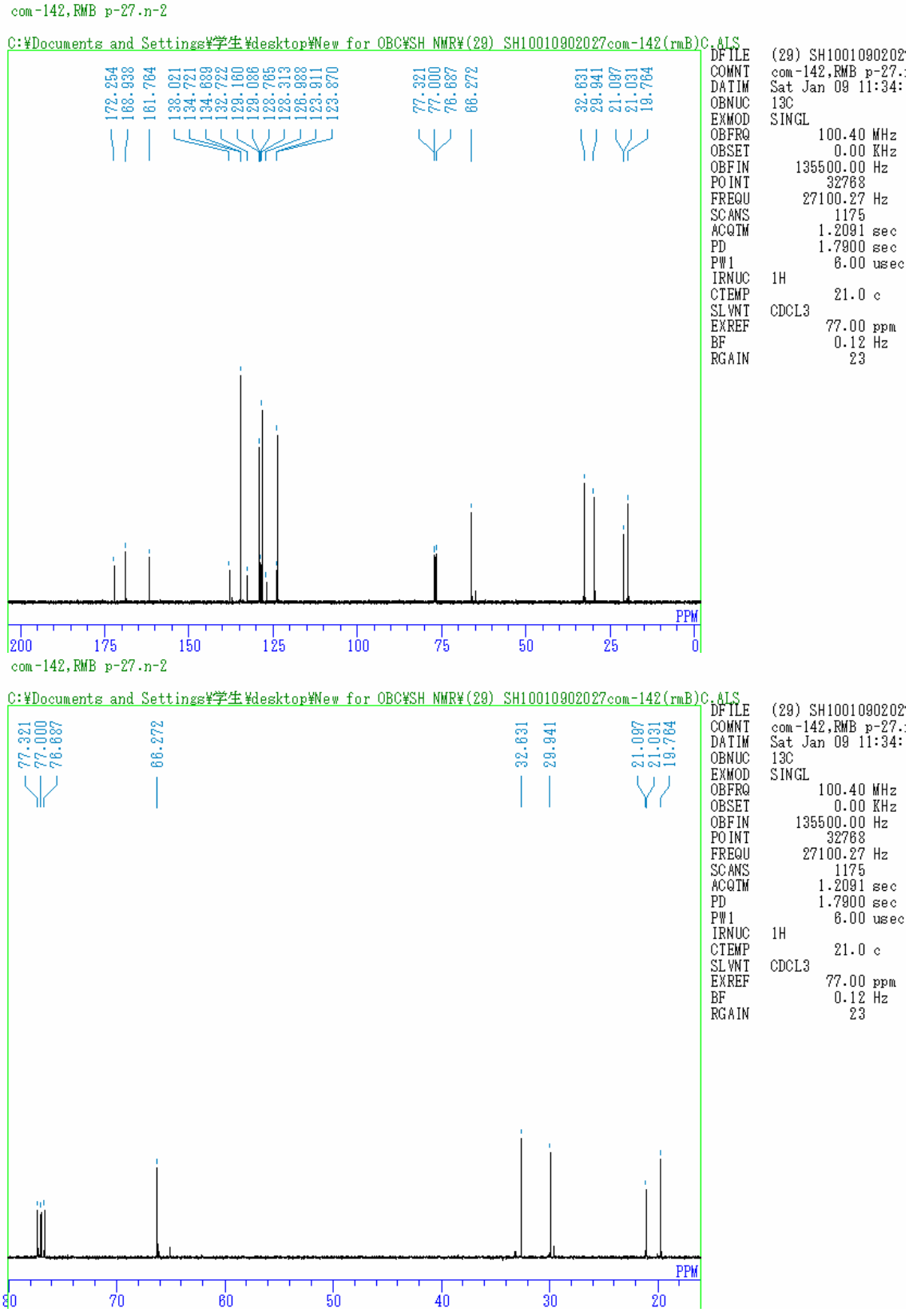
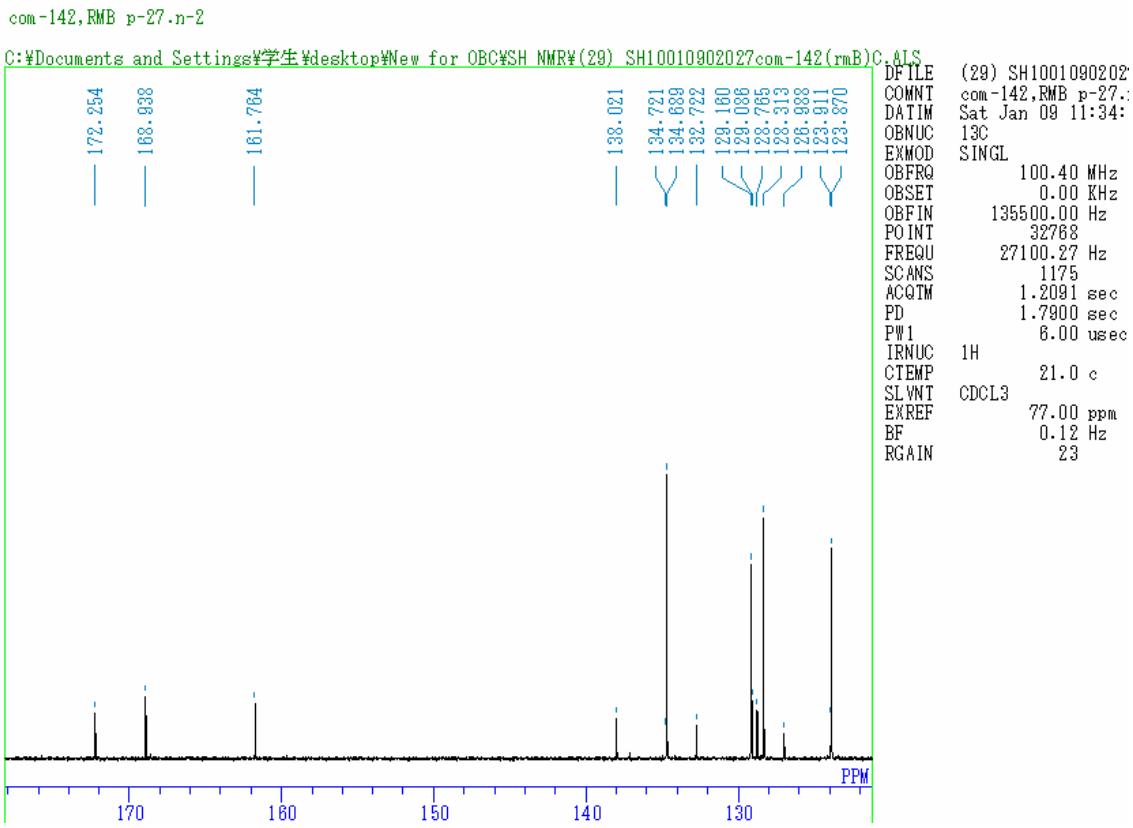
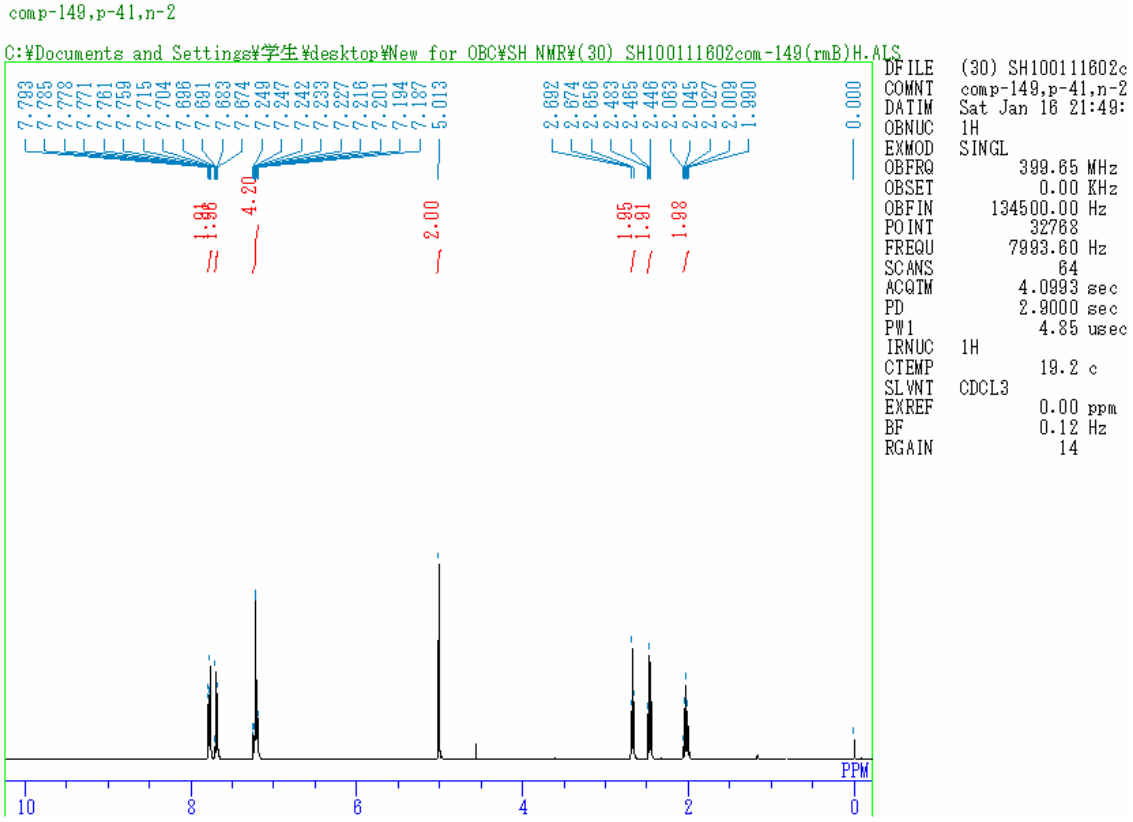
5.107
2.00
2.779
2.761
2.743
2.555
2.537
2.518
2.350
2.155
2.137
2.119
2.100
2.083
1.96
1.96
3.03
1.96
PPM
5.0
4.5
4.0
3.5
3.0
2.5
2.0
DFILE (29) SH10021302027
COMNT comp-142,p-27,n-2
DATIM Sat Feb 13 13:40:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 25
comp-142,p-27,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(29) SH10021302027comp142H.ALS
7.901
7.893
7.888
7.880
7.870
7.818
7.815
7.805
7.798
7.792
7.784
7.271
7.261
7.251
7.184
7.164
1.93
1.97
3.61
2.07
PPM
8.25
8.00
7.75
7.50
7.25
7.00
DFILE (29) SH10021302027
COMNT comp-142,p-27,n-2
DATIM Sat Feb 13 13:40:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 25

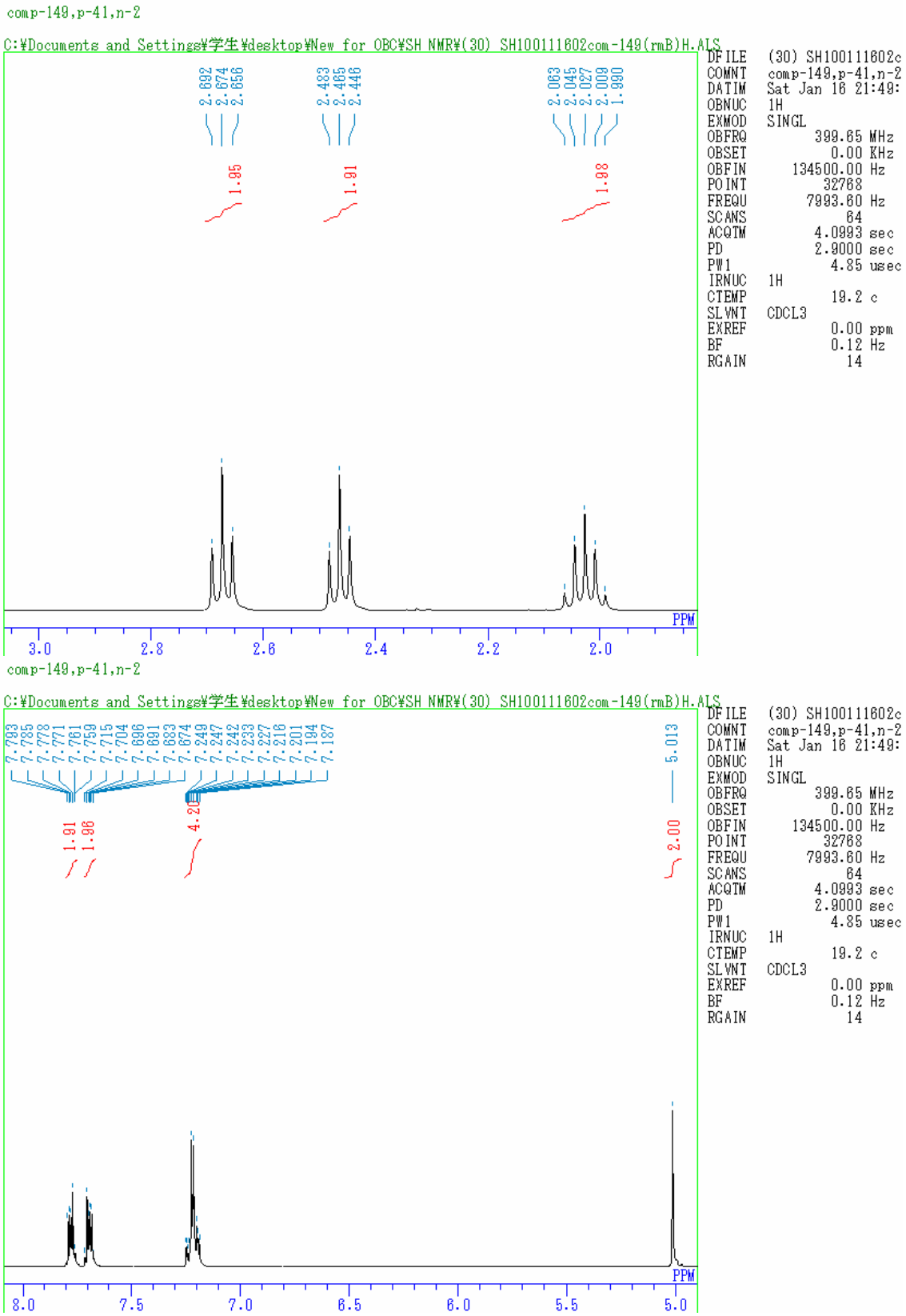
Phthalimido 4-(1-methylbenzyloxycarbonyl)butanoate (29) (^{13}C–NMR)



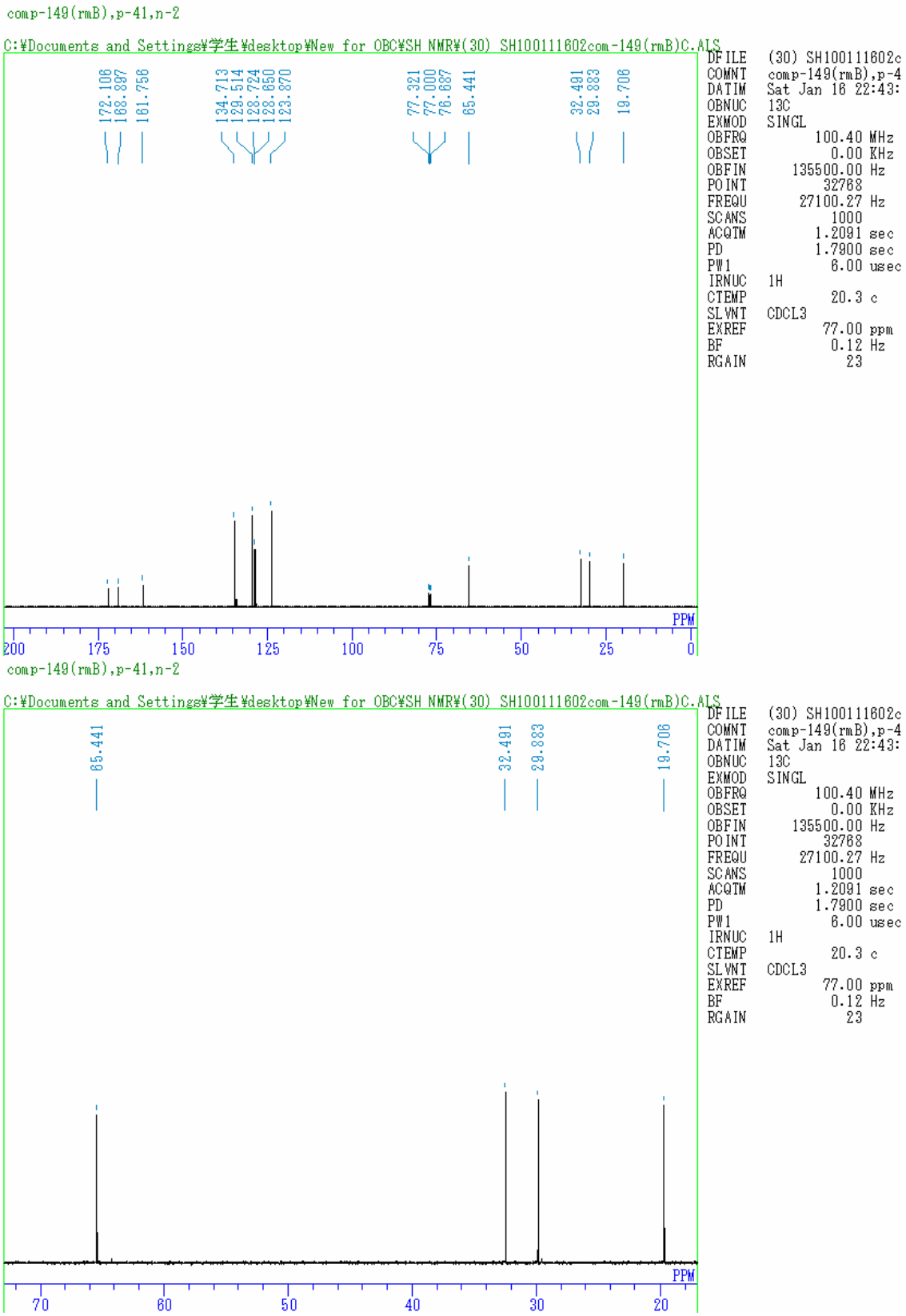


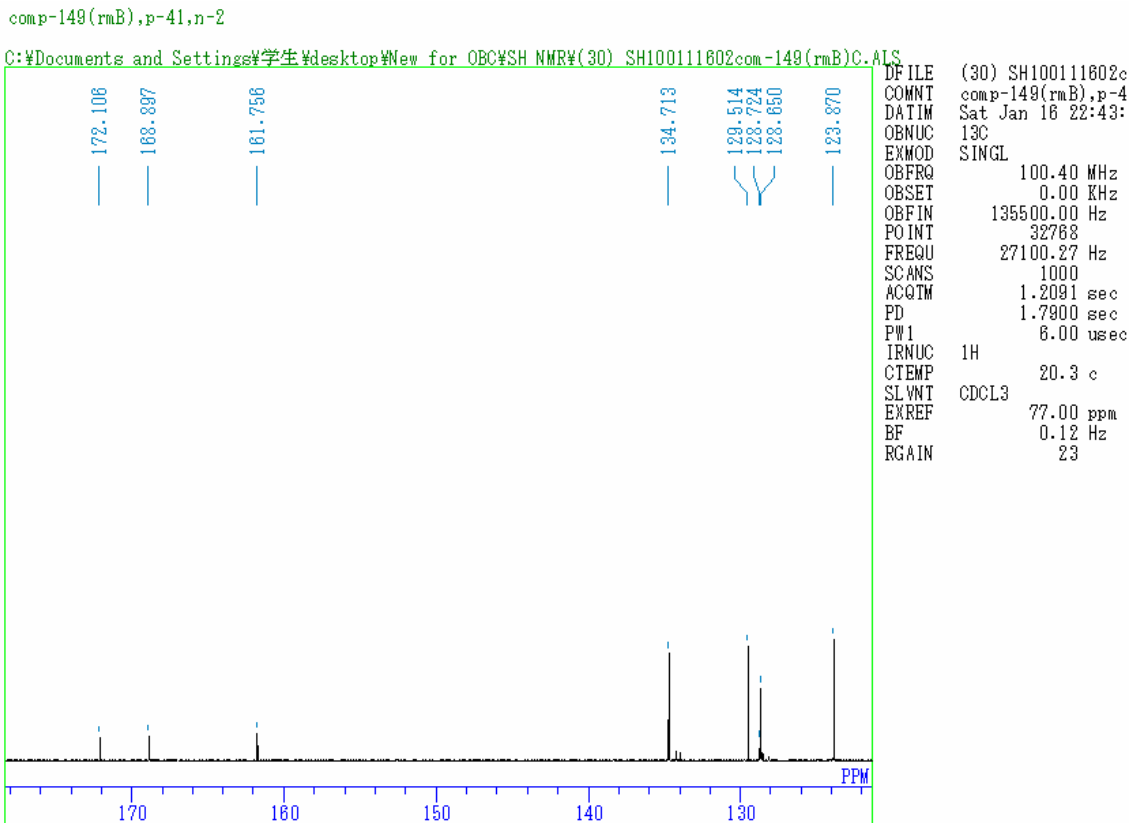
Phthalimido 4-(4-chlorobenzyloxycarbonyl)butanoate (30) (^{1}H–NMR)



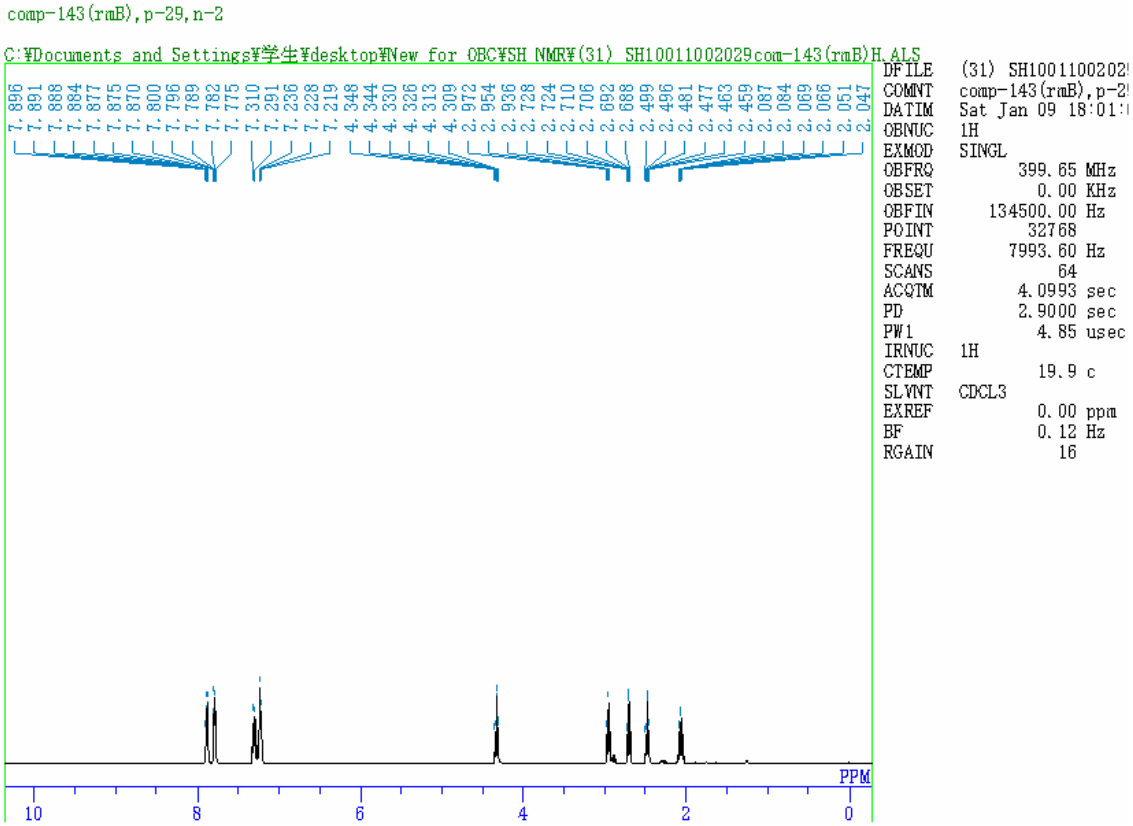

comp-149,p-41,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(30) SH100111602com-149(rmB)H.ALS
2.692
2.674
2.656
2.483
2.465
2.446
2.063
2.045
2.027
2.009
1.990
1.95
1.91
1.98
PPM
3.0
2.8
2.6
2.4
2.2
2.0
DFILE (30) SH100111602c
COMNT comp-149,p-41,n-2
DATIM Sat Jan 16 21:49:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14
comp-149,p-41,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(30) SH100111602com-149(rmB)H.ALS
7.793
7.785
7.778
7.771
7.761
7.759
7.715
7.704
7.696
7.691
7.683
7.674
7.249
7.247
7.242
7.233
7.227
7.216
7.201
7.194
7.187
5.013
1.91
1.96
4.20
2.00
PPM
8.0
7.5
7.0
6.5
6.0
5.5
5.0
DFILE (30) SH100111602c
COMNT comp-149,p-41,n-2
DATIM Sat Jan 16 21:49:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14

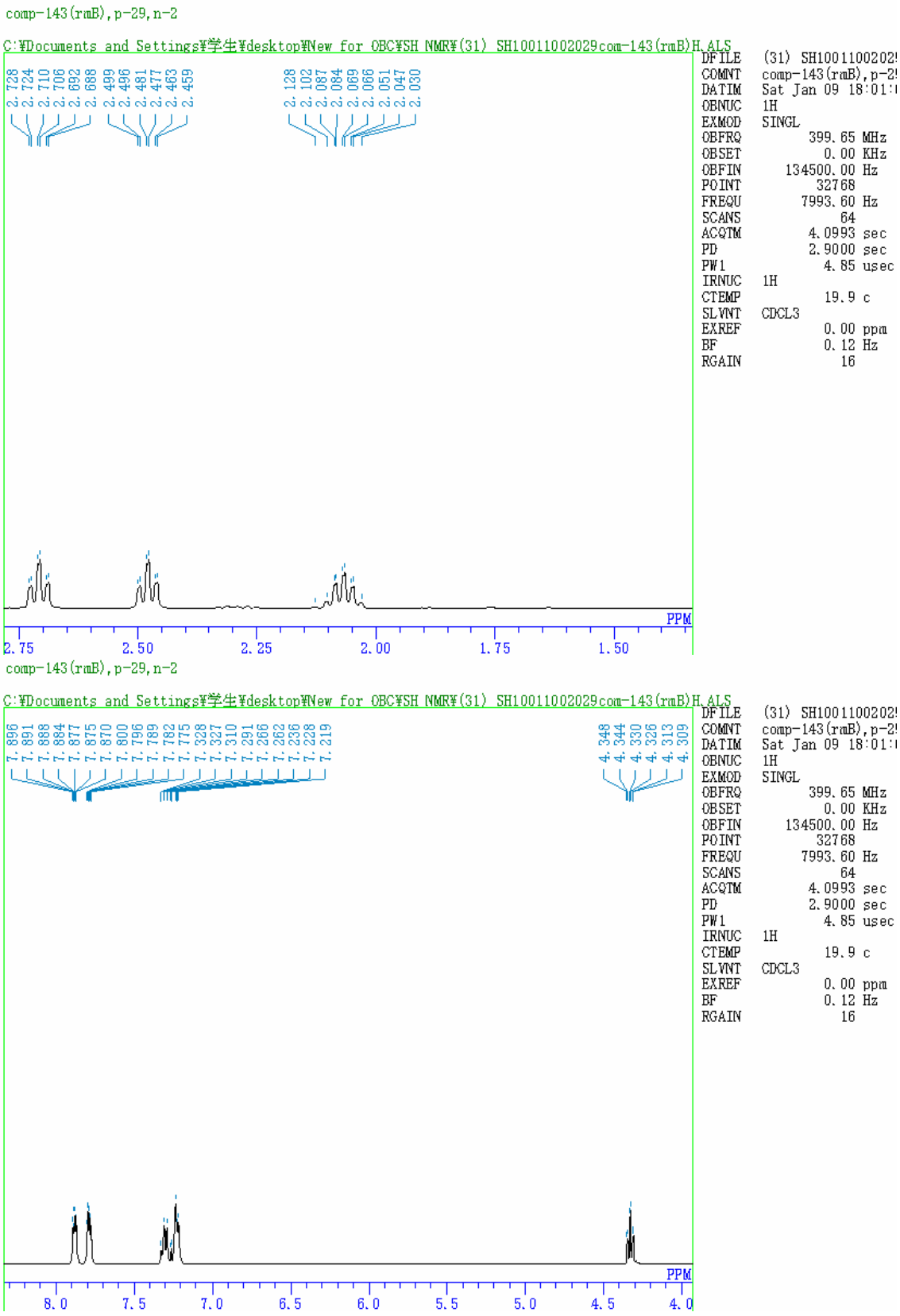
Phthalimido 4-(4-chlorobenzyloxycarbonyl)butanoate (30) (^{13}C–NMR)



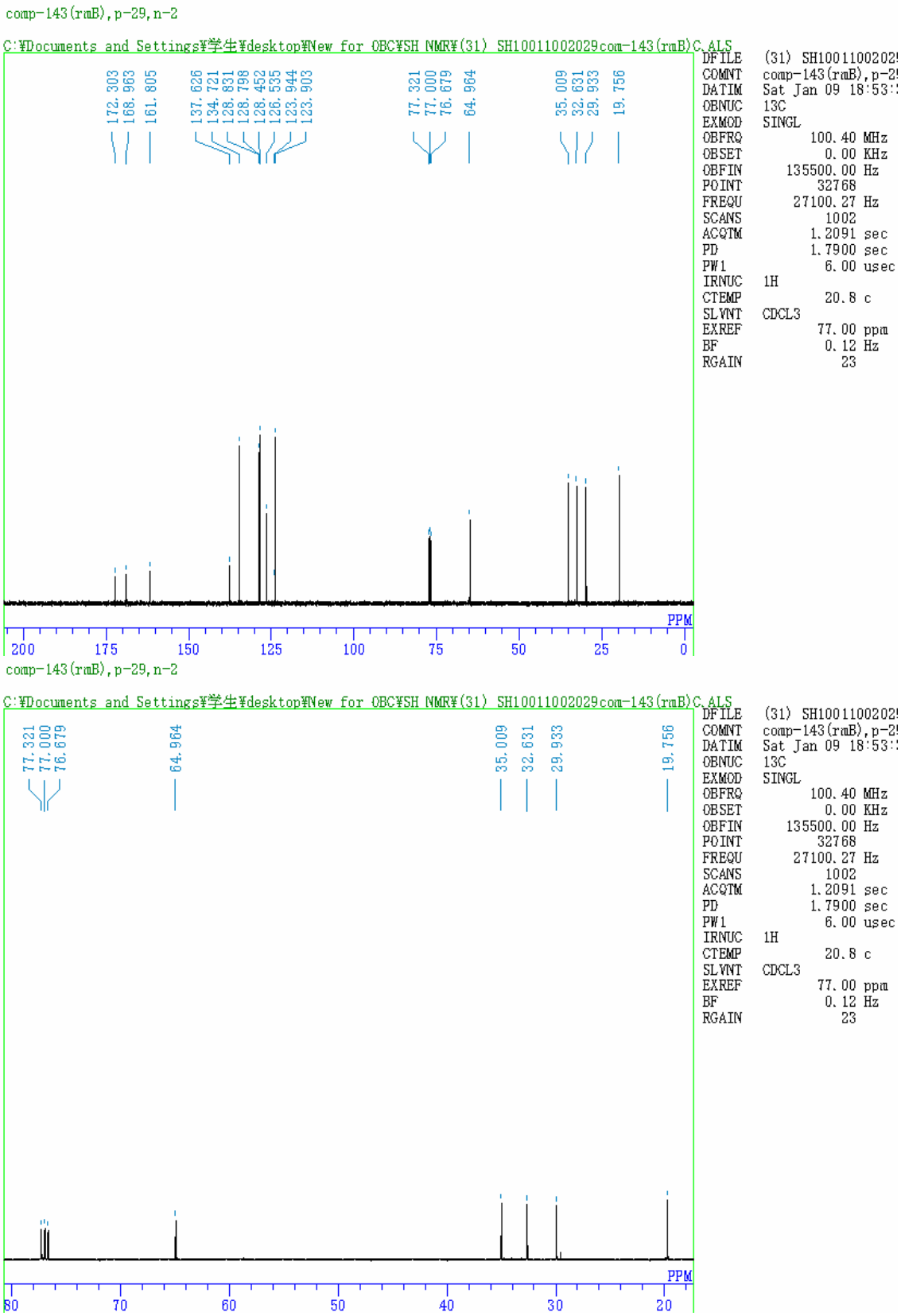


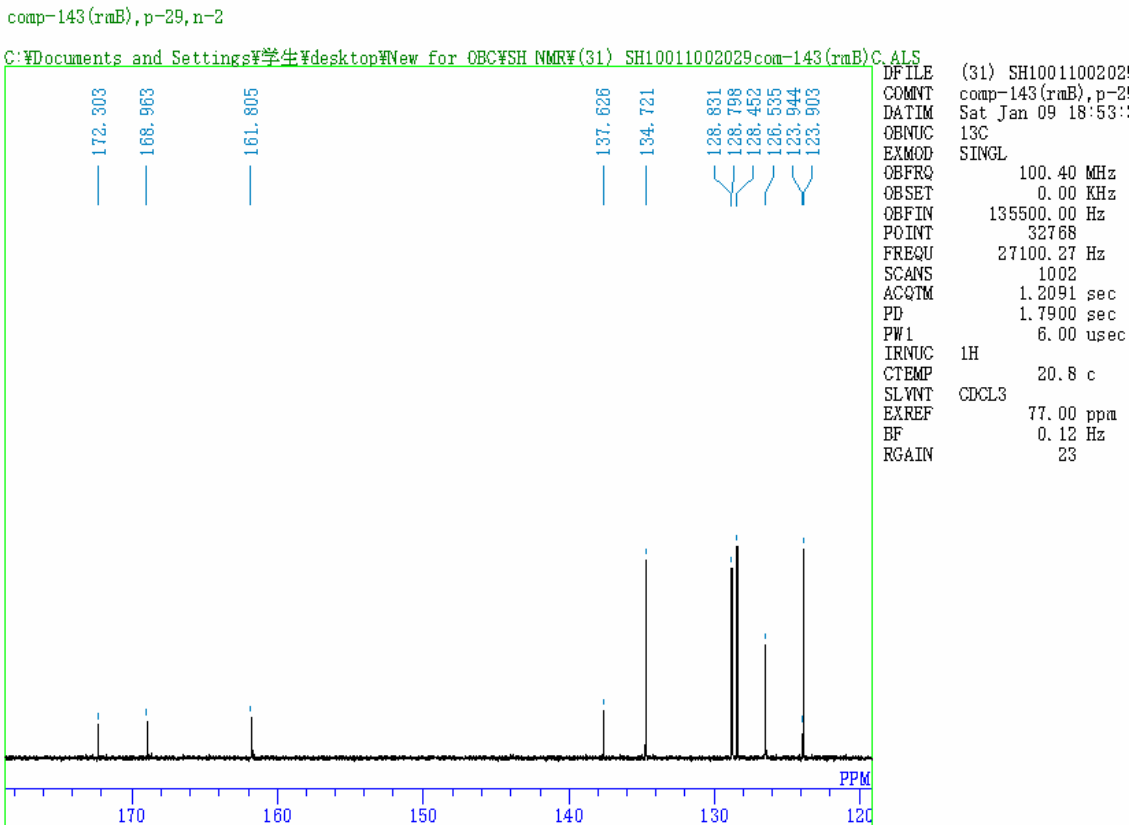
Phthalimido 4-phenylethyloxycarbonylbutanoate (31) (^{1}H–NMR)



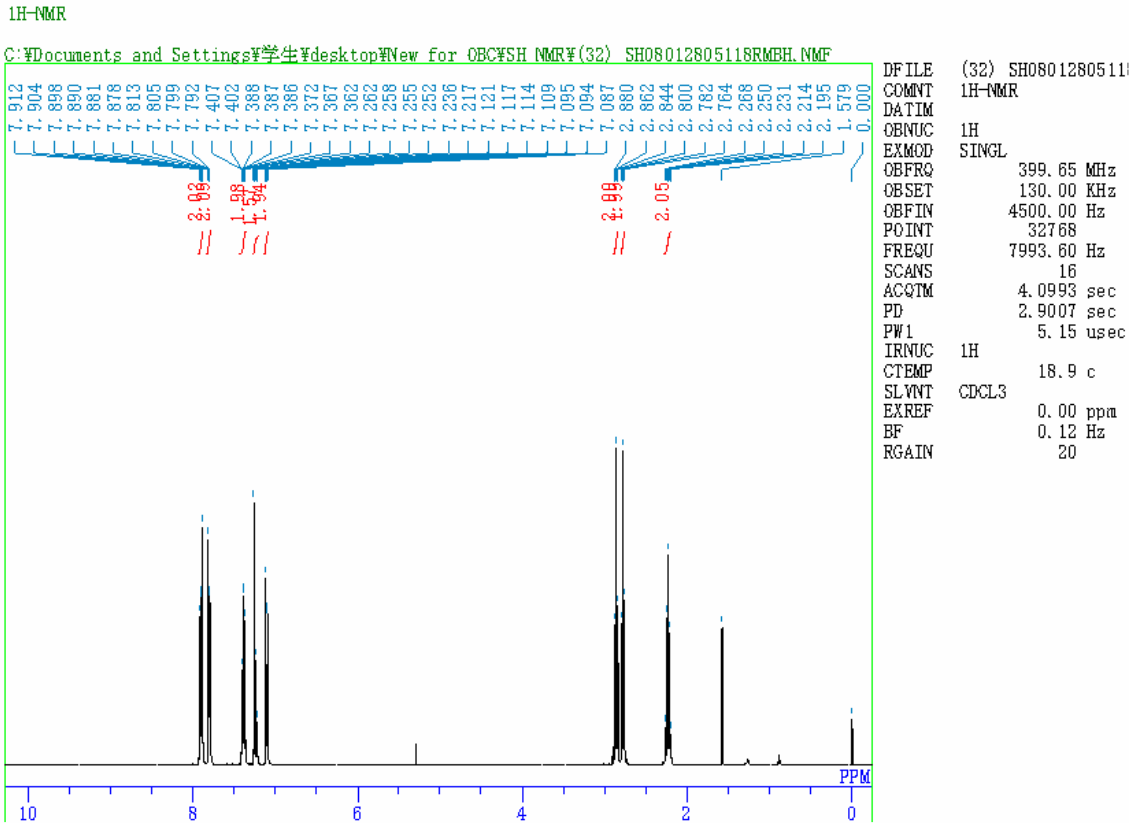

comp-143(rmB),p-29,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(31) SH10011002029com-143(rmB)H.ALS
DFILE (31) SH1001100202
COMNT comp-143(rmB),p-2
DATIM Sat Jan 09 18:01:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16
PPM
2.75
2.50
2.25
2.00
1.75
1.50
comp-143(rmB),p-29,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(31) SH10011002029com-143(rmB)H.ALS
DFILE (31) SH1001100202
COMNT comp-143(rmB),p-2
DATIM Sat Jan 09 18:01:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16
PPM
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0

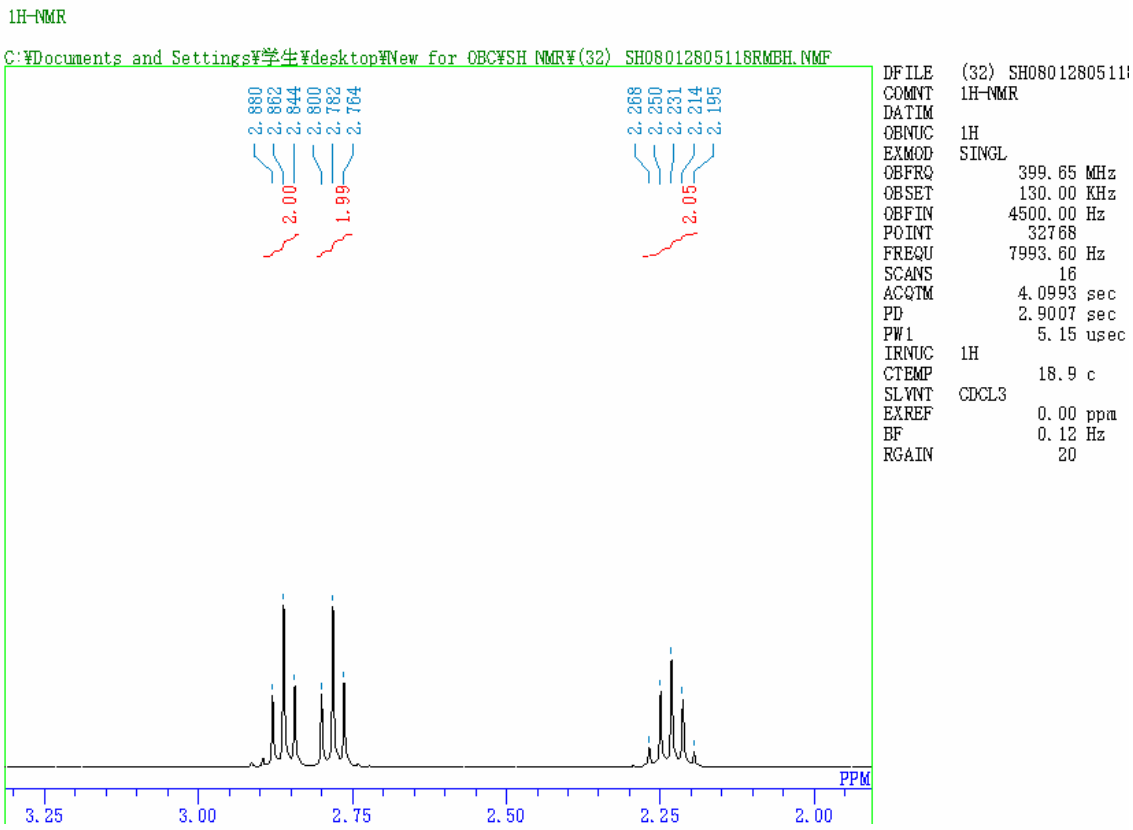
Phthalimido 4-phenylethyloxycarbonylbutanoate (31) (^{13}C–NMR)





Phthalimido 4-phenyloxycarbonylbutanoate (32) (^{1}H–NMR)

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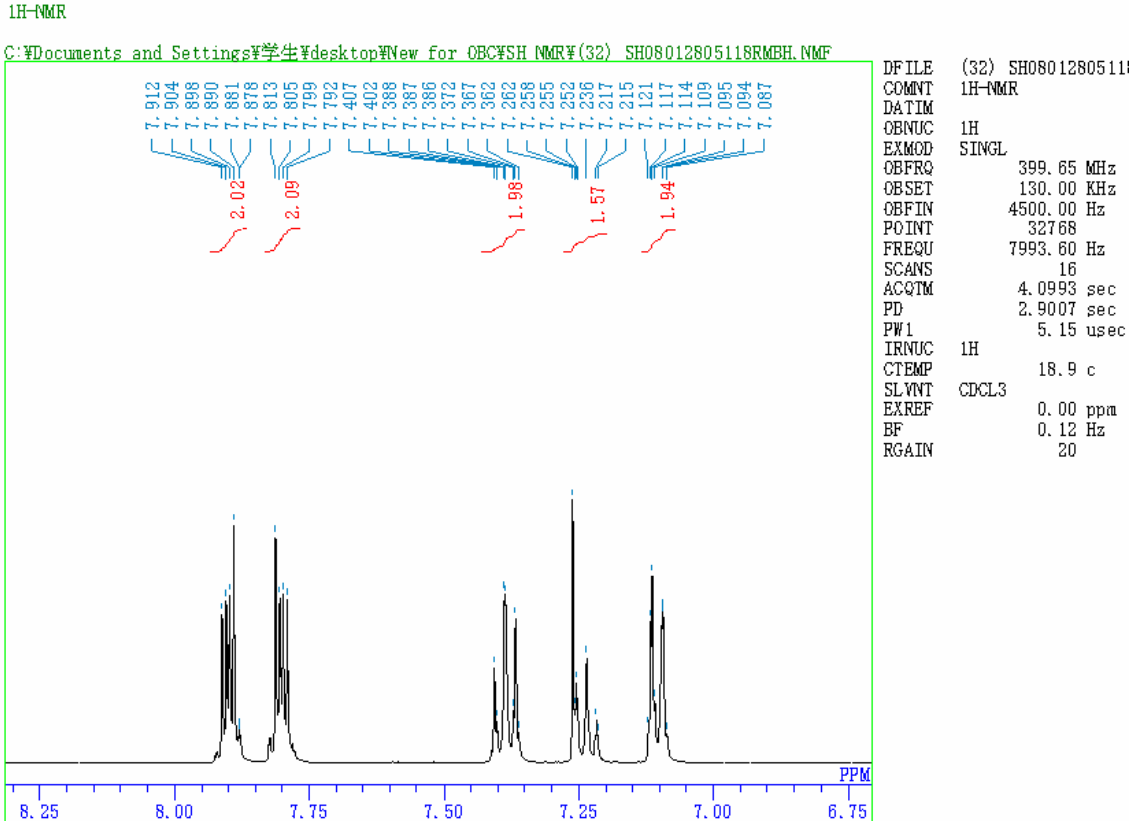
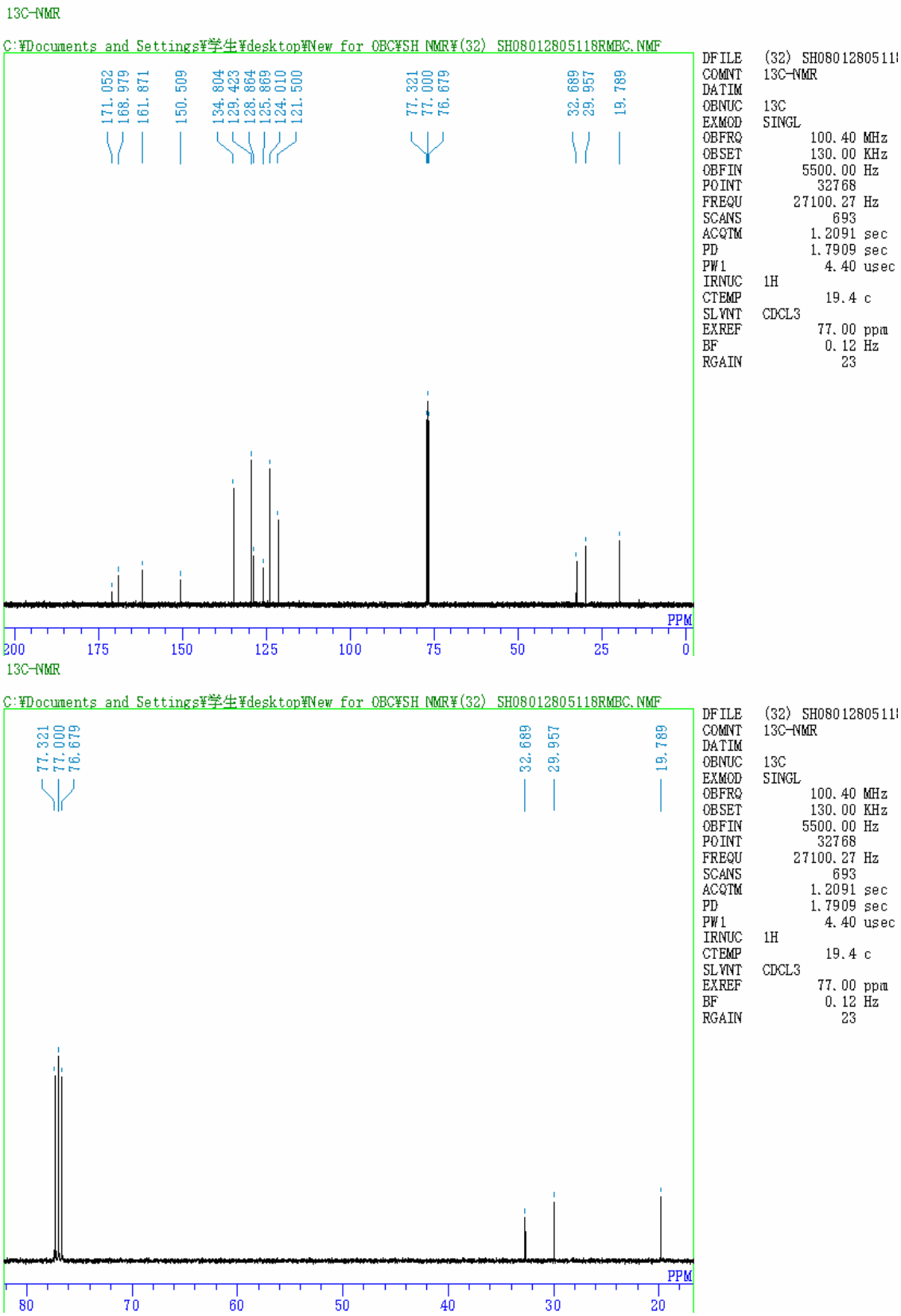
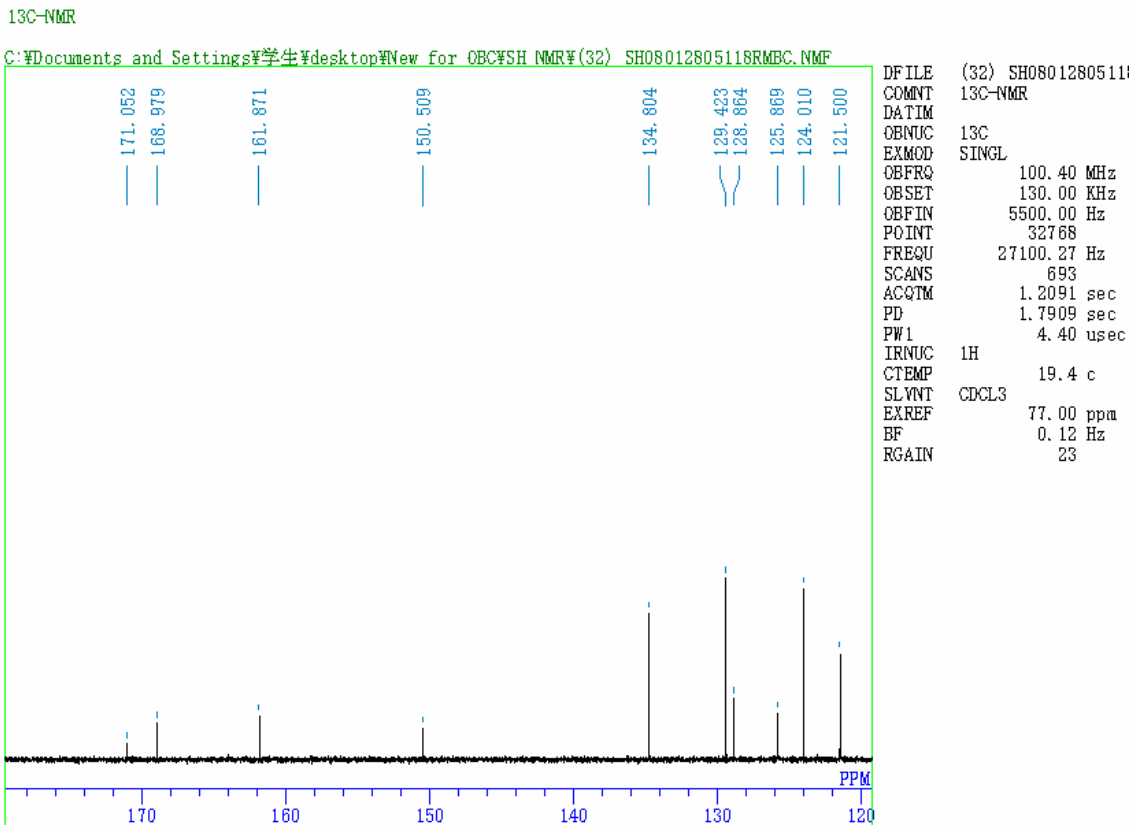
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(32) SH08012805118RMBH.NMF
2.880
2.862
2.844
2.800
2.782
2.764
2.268
2.250
2.231
2.214
2.195
2.00
1.99
2.05
PPM
3.25
3.00
2.75
2.50
2.25
2.00
DFILE (32) SH0801280511
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20

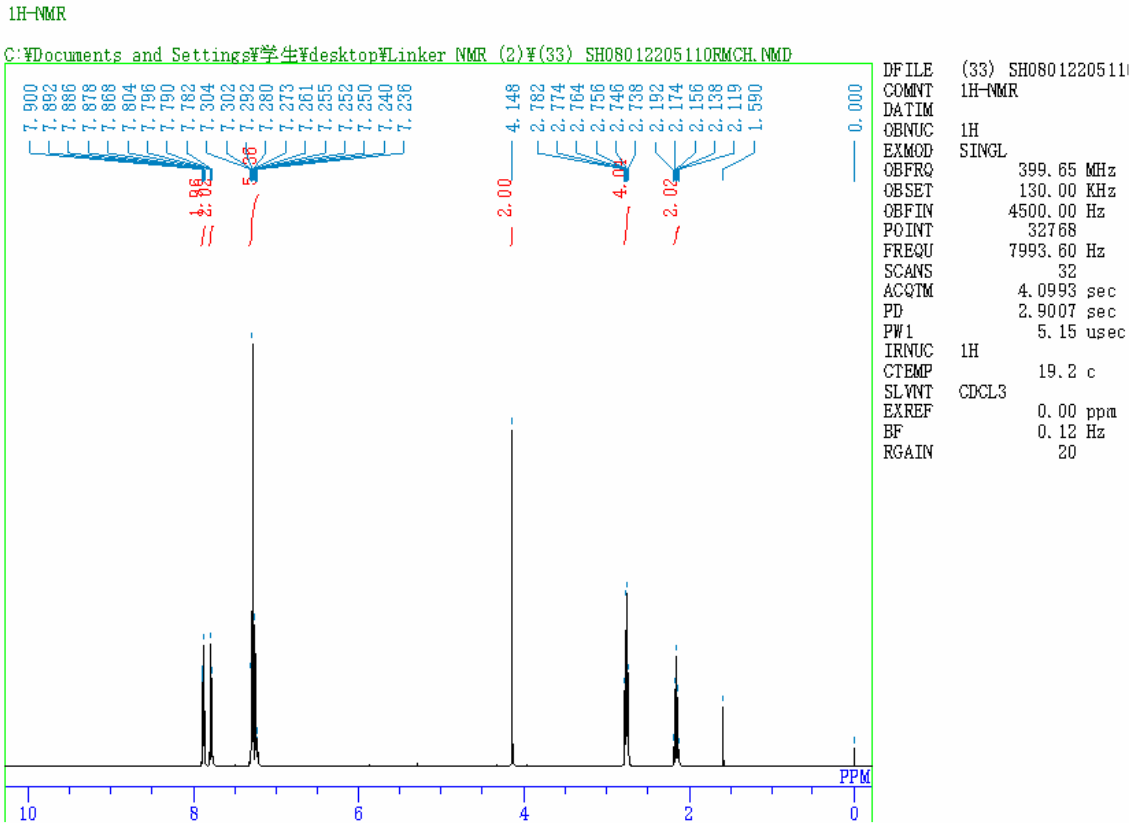

1H-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(32) SH08012805118RMBH.NMF
7.912
7.904
7.898
7.890
7.881
7.878
7.813
7.805
7.799
7.792
7.407
7.402
7.388
7.387
7.386
7.372
7.367
7.362
7.262
7.258
7.255
7.252
7.236
7.217
7.215
7.121
7.117
7.114
7.109
7.095
7.094
7.087
2.02
2.09
1.98
1.57
1.94
PPM
8.25
8.00
7.75
7.50
7.25
7.00
6.75
DFILE (32) SH0801280511
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20

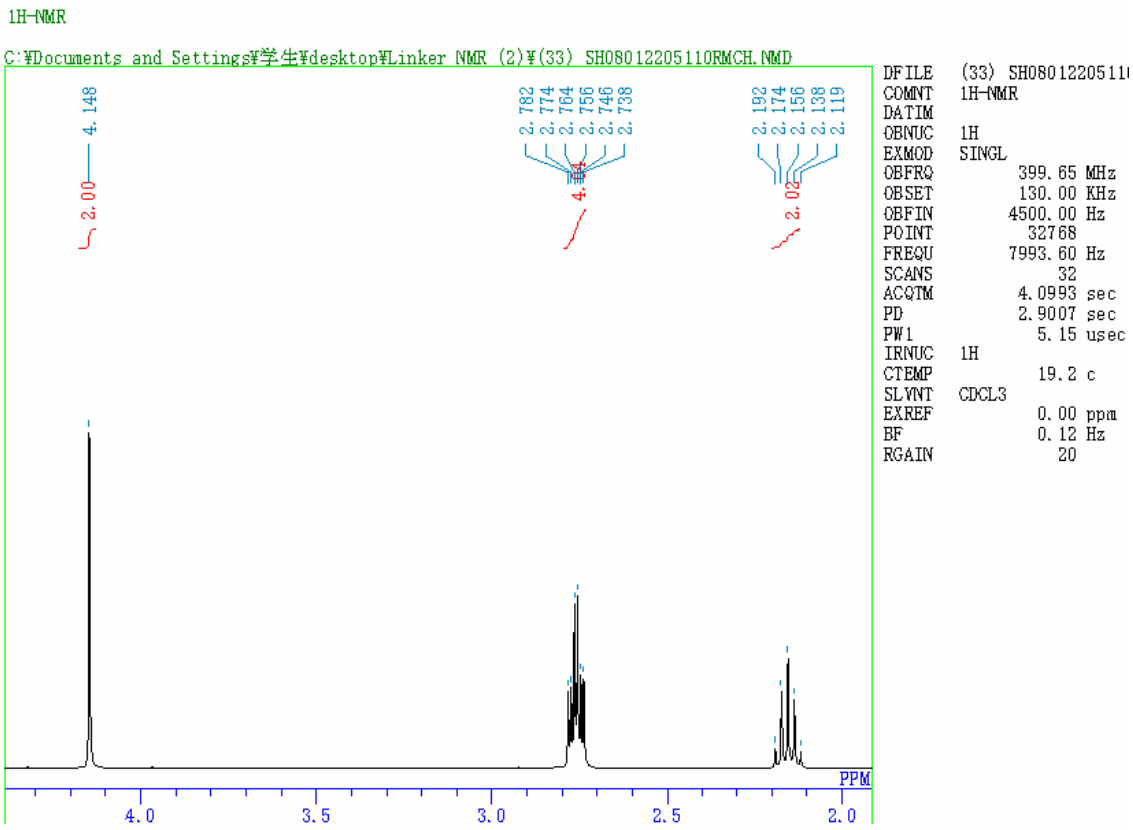
Phthalimido 4-phenyloxycarbonylbutanoate (32) (^{13}C–NMR)

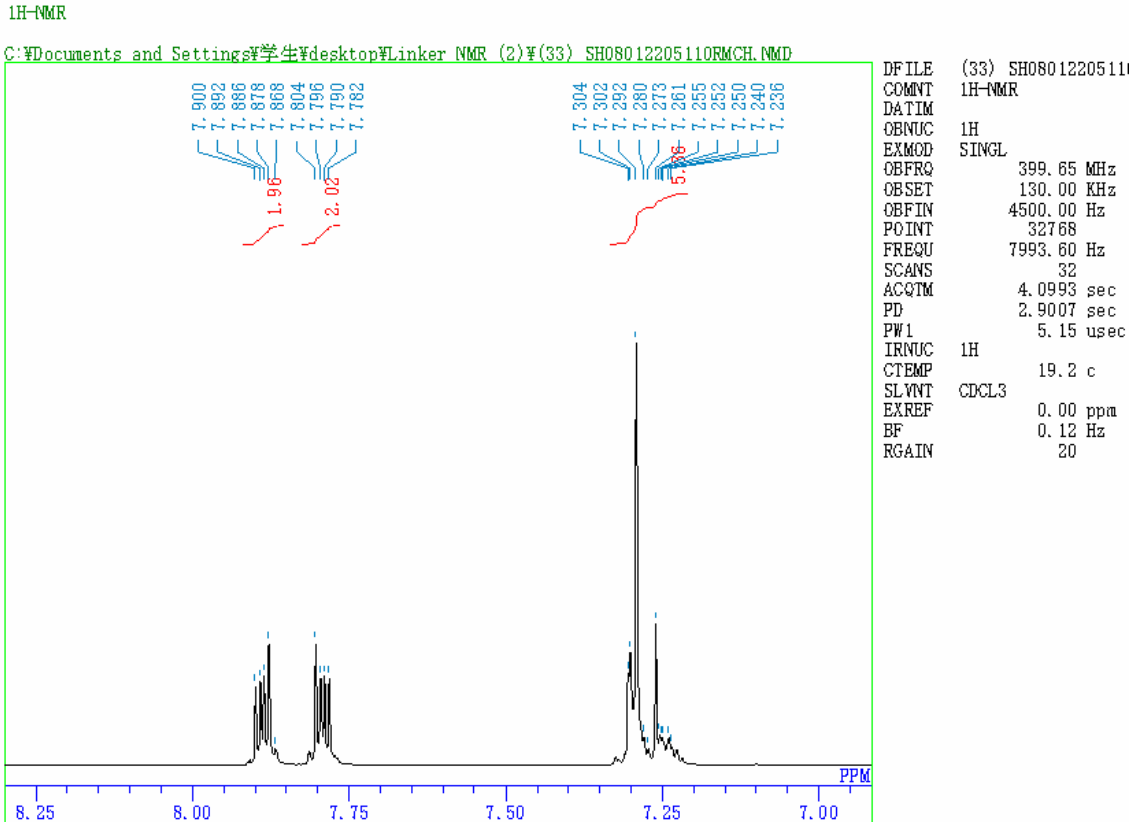




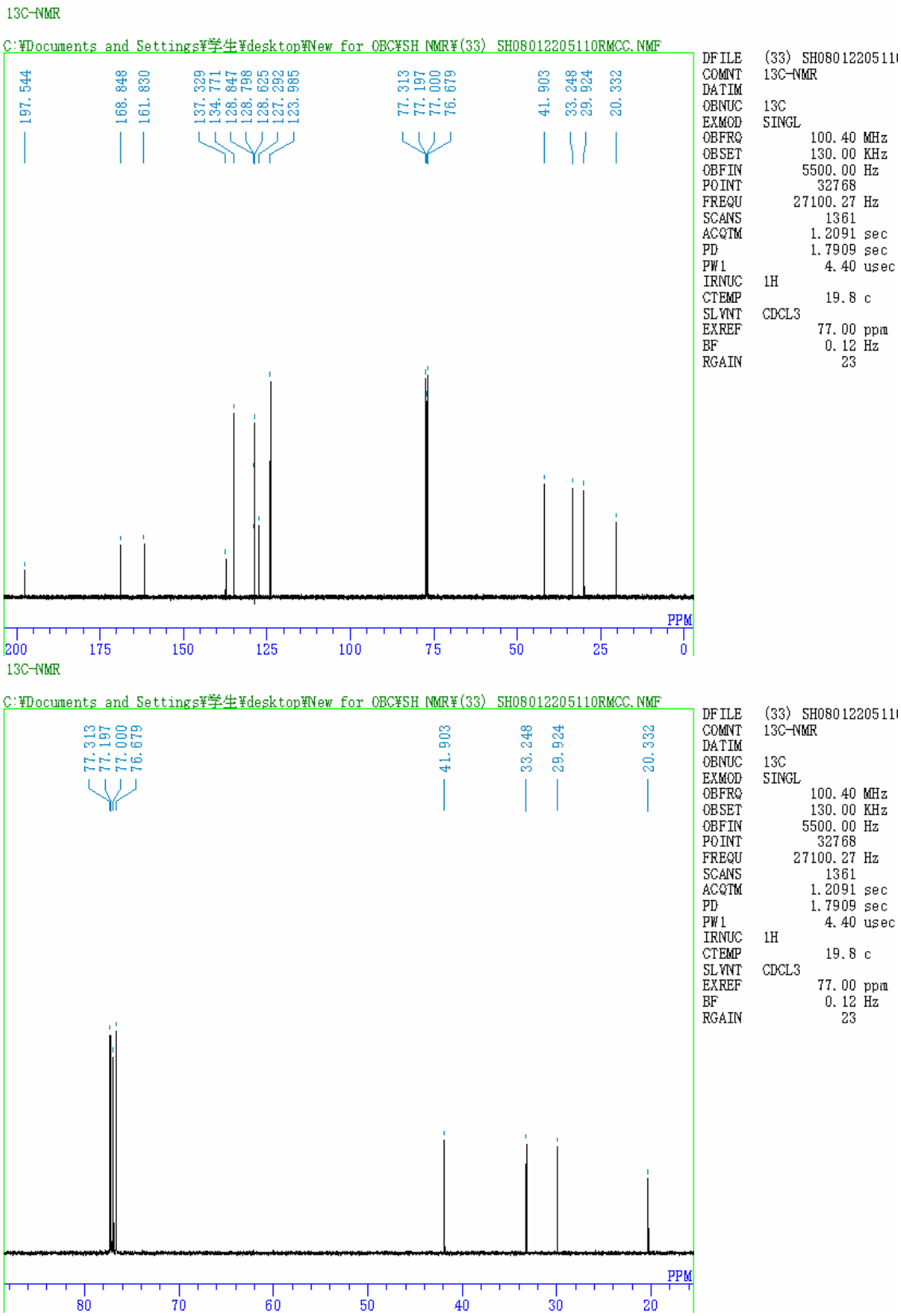
Phthalimido 4-(*S*-benzyloxycarbonyl)butanoate (33) (^{1}H–NMR)

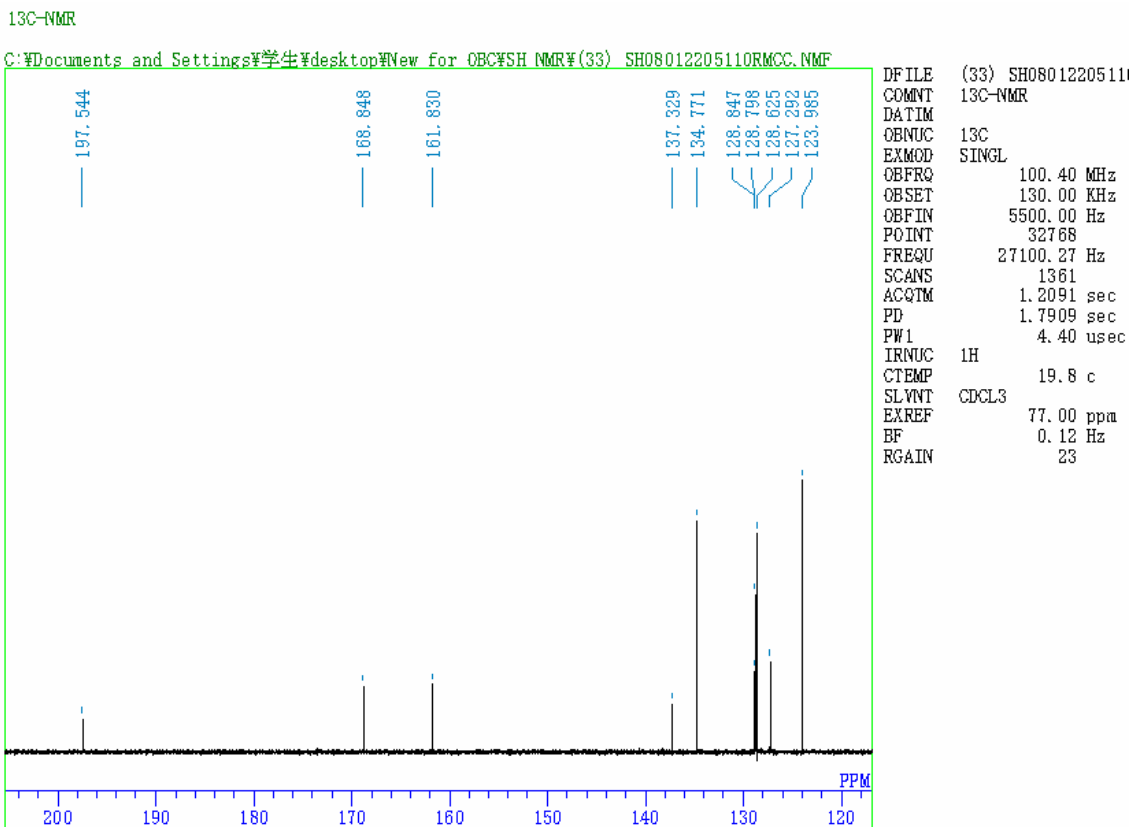



1H-NMR
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(33) SH08012205110RMCH.NMD
4.148
2.782
2.774
2.764
2.756
2.746
2.738
2.192
2.174
2.156
2.138
2.119
2.00
4.04
2.02
PPM
4.0
3.5
3.0
2.5
2.0
DFILE (33) SH08012205110
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20


1H-NMR
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(33) SH08012205110RMCH.NMD
7.900
7.892
7.886
7.878
7.868
7.804
7.796
7.790
7.782
7.304
7.302
7.292
7.280
7.273
7.261
7.255
7.252
7.250
7.240
7.236
1.96
2.02
5.86
PPM
8.25
8.00
7.75
7.50
7.25
7.00
DFILE (33) SH08012205110
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20

Phthalimido 4-(*S*-benzyloxycarbonyl)butanoate (33) (^{13}C–NMR)



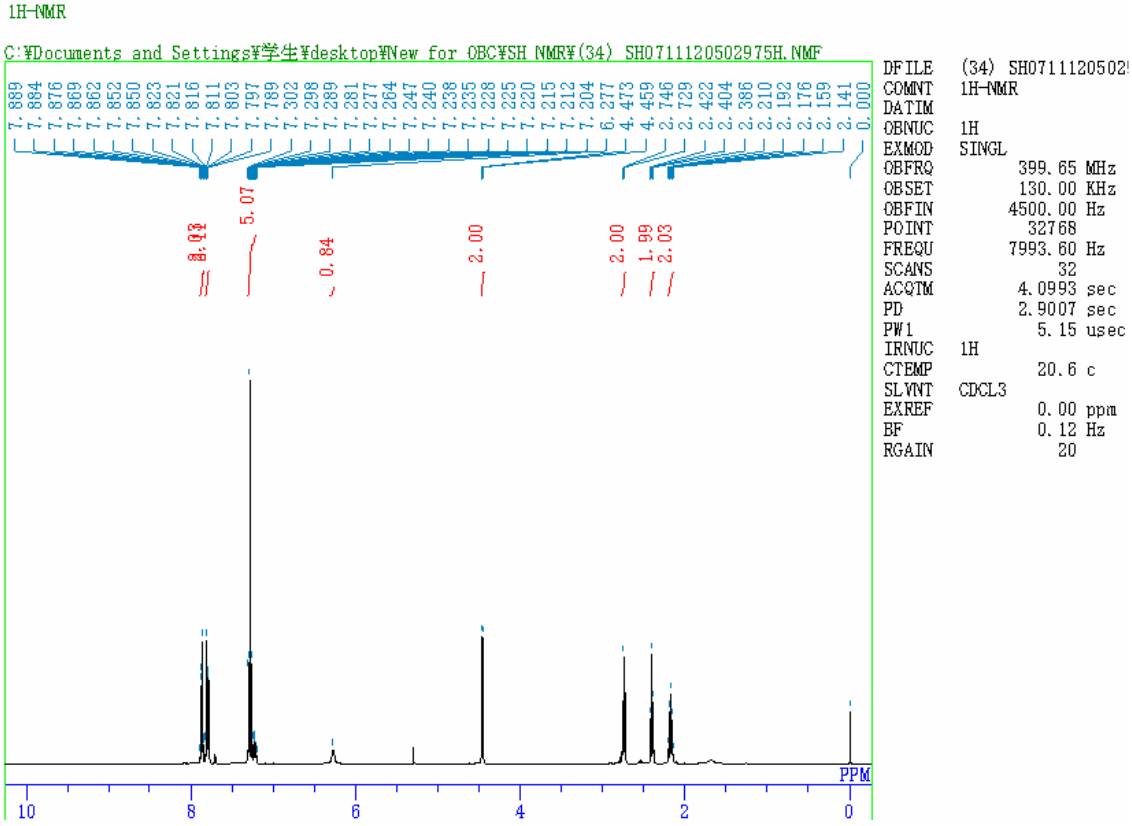

13C-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(33) SH08012205110RMCC.NMF
197.544
168.848
161.830
137.329
134.771
128.847
128.798
128.625
127.292
123.985
PPM
200
190
180
170
160
150
140
130
120
DFILE (33) SH0801220511
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 1361
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.40 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

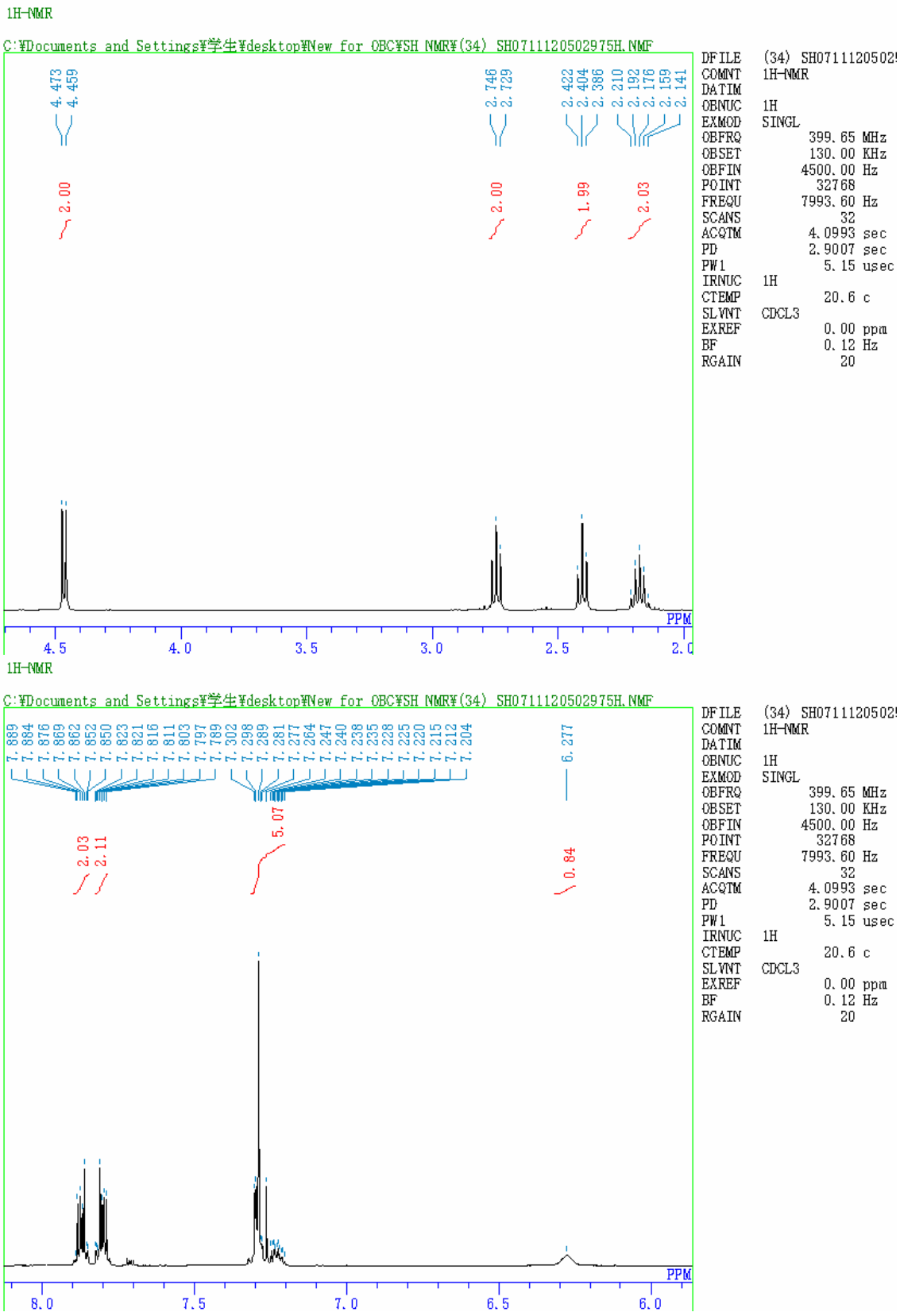
(v) Supporting Information for the manuscript:

Title: “Syntheses and reactivities of non-symmetrical "active ester" bi-dentate cross-linking reagents having a phthalimidoyl and acid chloride, 2-benzothiazole, or 1-benzotriazole group”

(*^{1}H –NMR and ^{13}C–NMR)*

Phthalimido 4-(*N*-benzyloxycarbonyl)butanoate (34) (^{1}H-NMR)




1H-NMR
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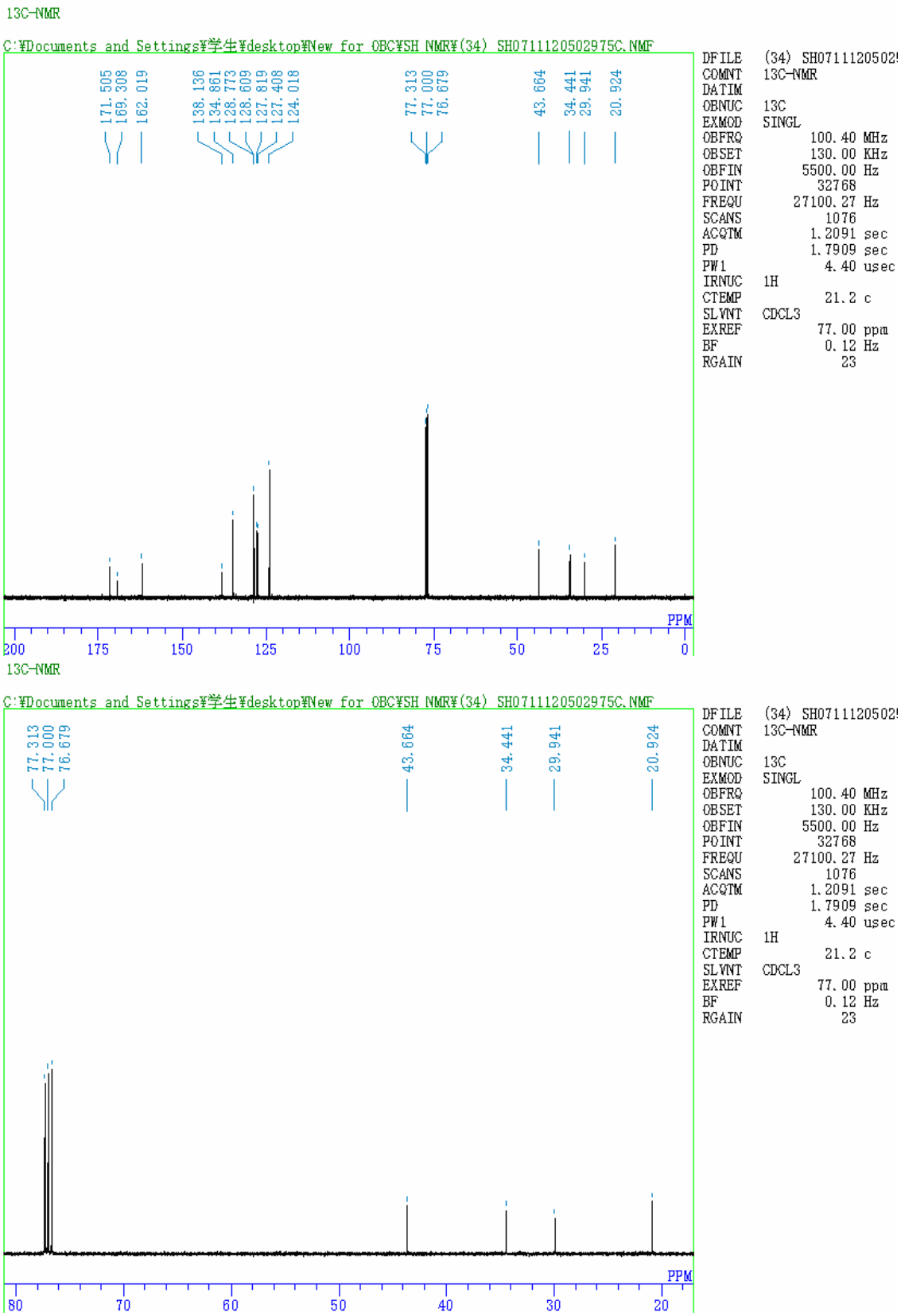
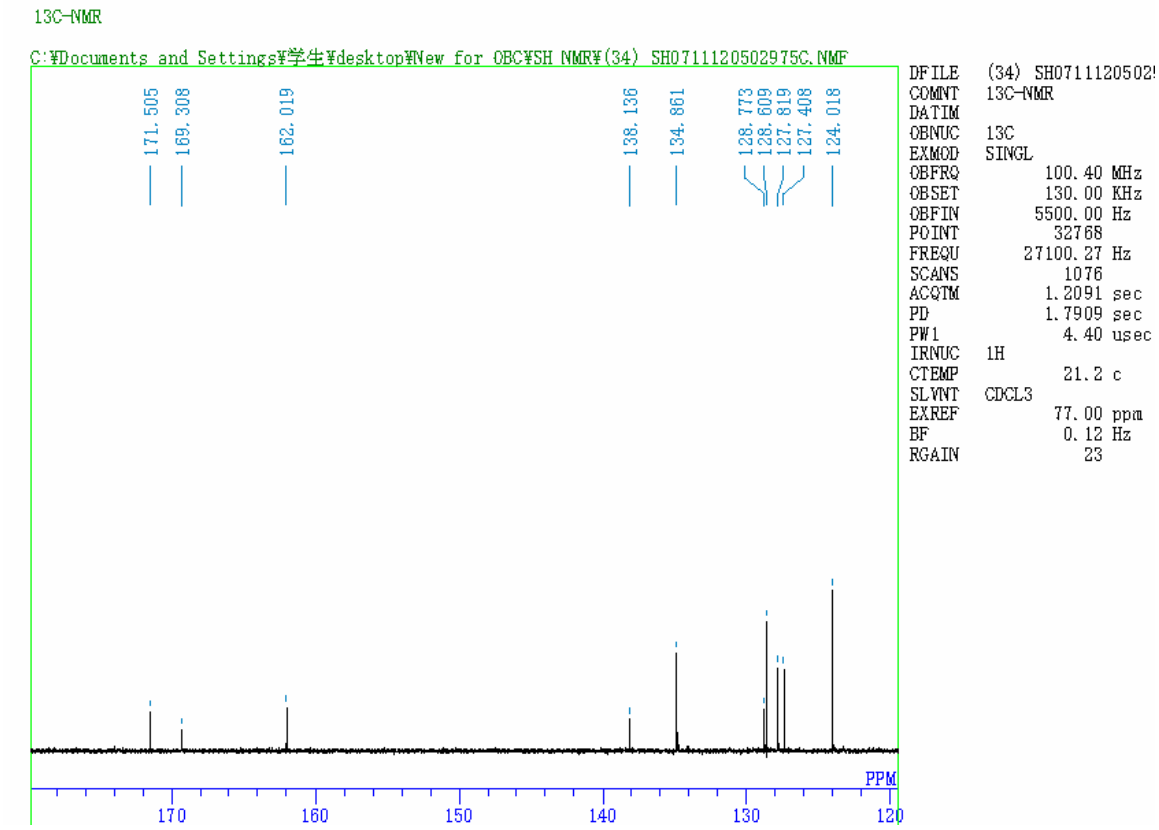
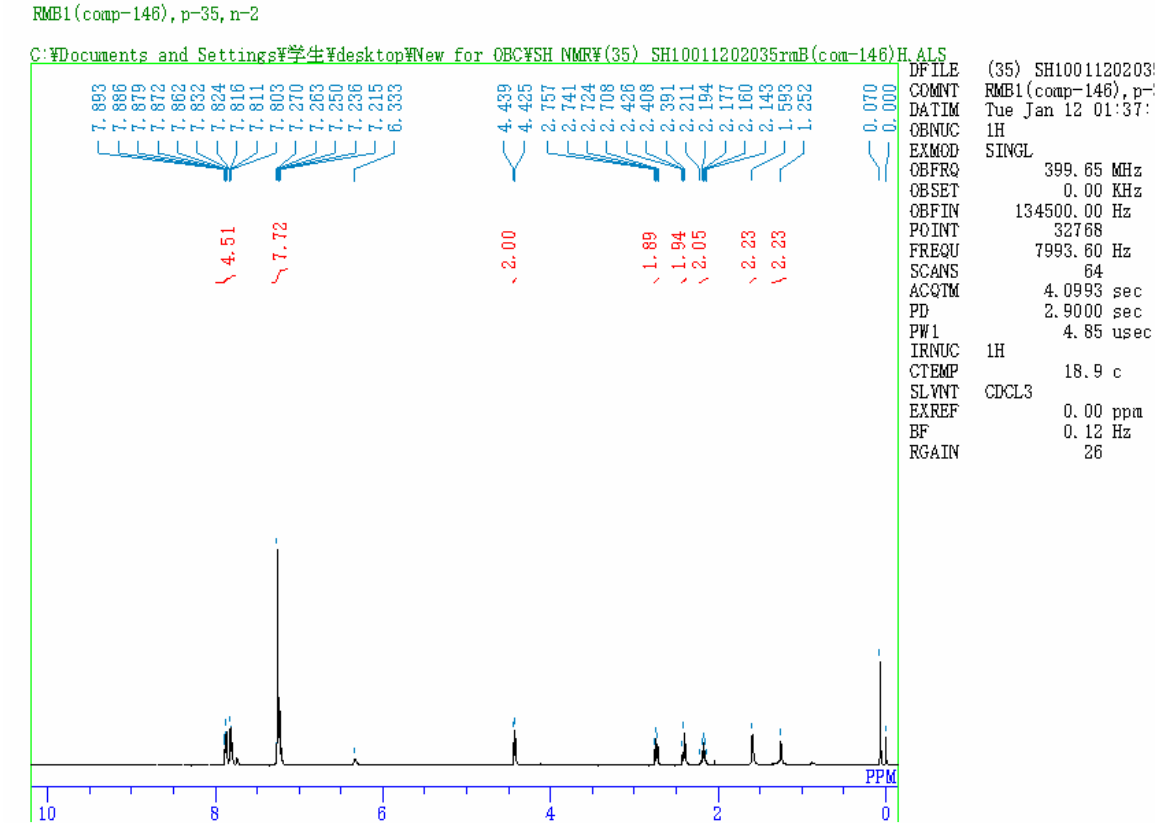
4.473
4.459
2.746
2.729
2.422
2.404
2.386
2.210
2.192
2.176
2.159
2.141
2.00
2.00
1.99
2.03
PPM
4.5
4.0
3.5
3.0
2.5
2.0
DFILE (34) SH0711120502
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20
1H-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(34) SH0711120502975H.NMF
7.889
7.884
7.876
7.869
7.862
7.852
7.850
7.823
7.821
7.816
7.811
7.803
7.797
7.789
7.302
7.298
7.289
7.281
7.277
7.264
7.247
7.240
7.238
7.235
7.228
7.225
7.220
7.215
7.212
7.204
6.277
2.03
2.11
5.07
0.84
PPM
8.0
7.5
7.0
6.5
6.0
DFILE (34) SH0711120502
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20

Phthalimido 4-(*N*-benzyloxycarbonyl)butanoate (34) (^{13}C-NMR)



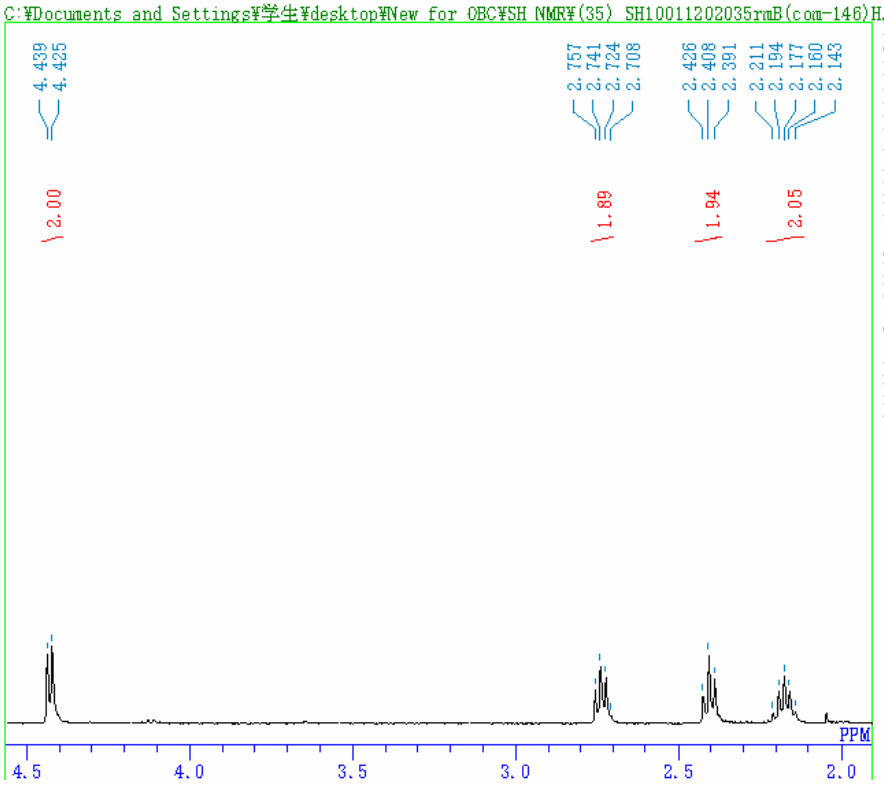


Phthalimido 4-(*N*-4-cholorobenzyloxycarbonyl)butanoate (35) (^{1}H-NMR)



RMB1(comp-146),p-35,n-2

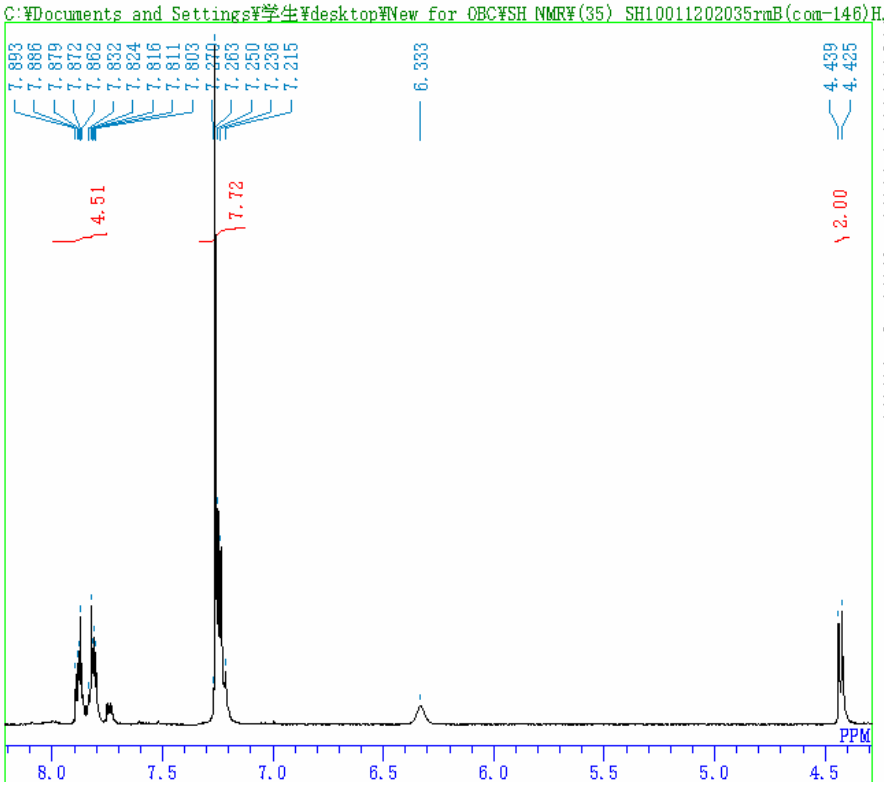
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(35) SH10011202035rmB(com-146)H.ALS



DFILE	(35) SH10011202035
COMNT	RMB1(comp-146),p-3
DATIM	Tue Jan 12 01:37:
OBNUC	1H
EXMOD	SINGL
OBFRQ	399.65 MHz
OBSET	0.00 KHz
OBFIN	134500.00 Hz
POINT	32768
FREQU	7993.60 Hz
SCANS	64
ACQTM	4.0993 sec
PD	2.9000 sec
PW1	4.85 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26

RMB1(comp-146),p-35,n-2

C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(35) SH10011202035rmB(com-146)H.ALS



DFILE	(35) SH10011202035
COMNT	RMB1(comp-146),p-3
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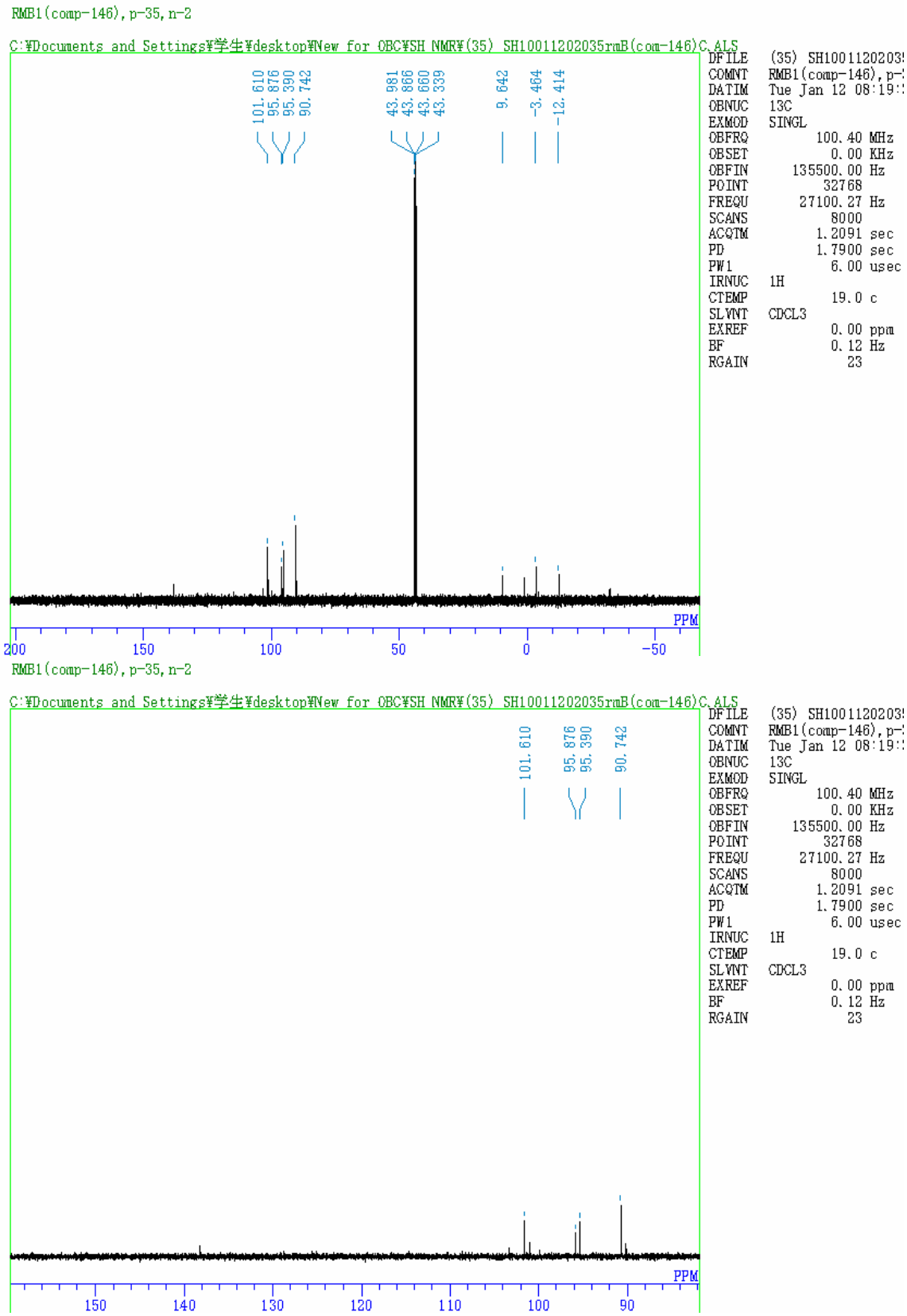
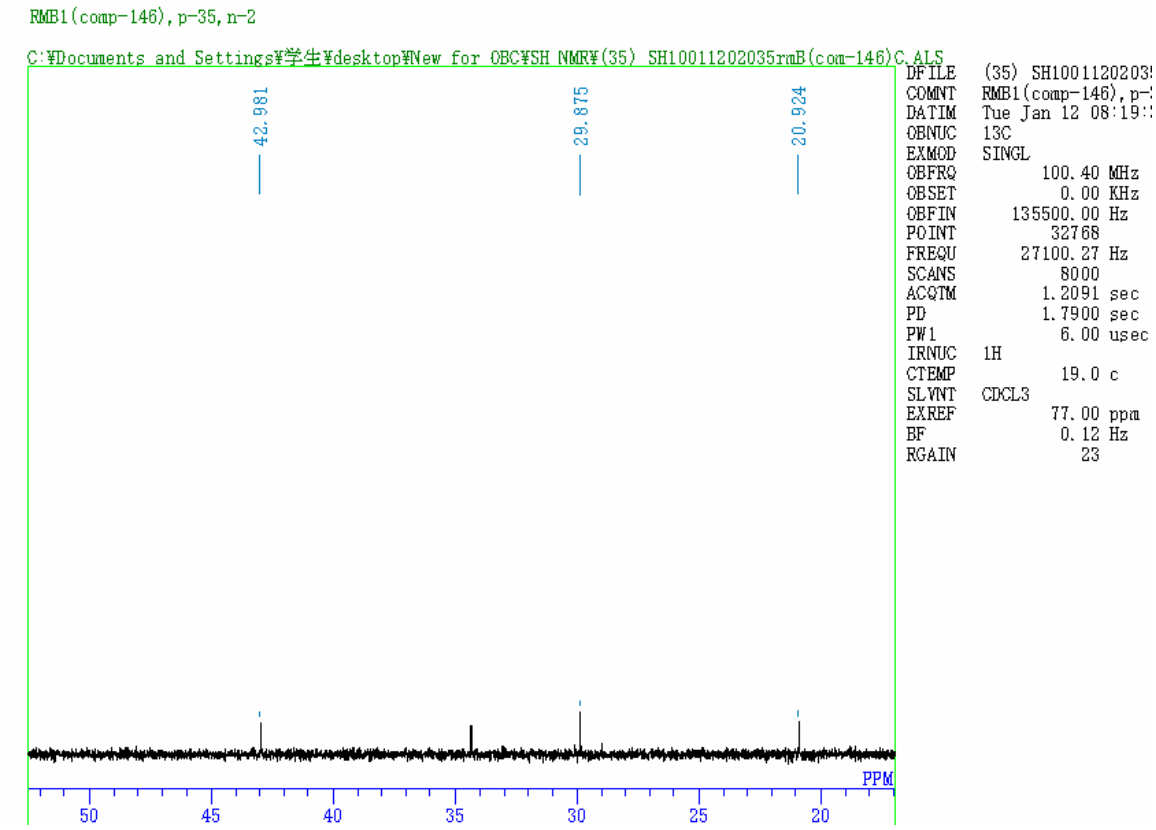
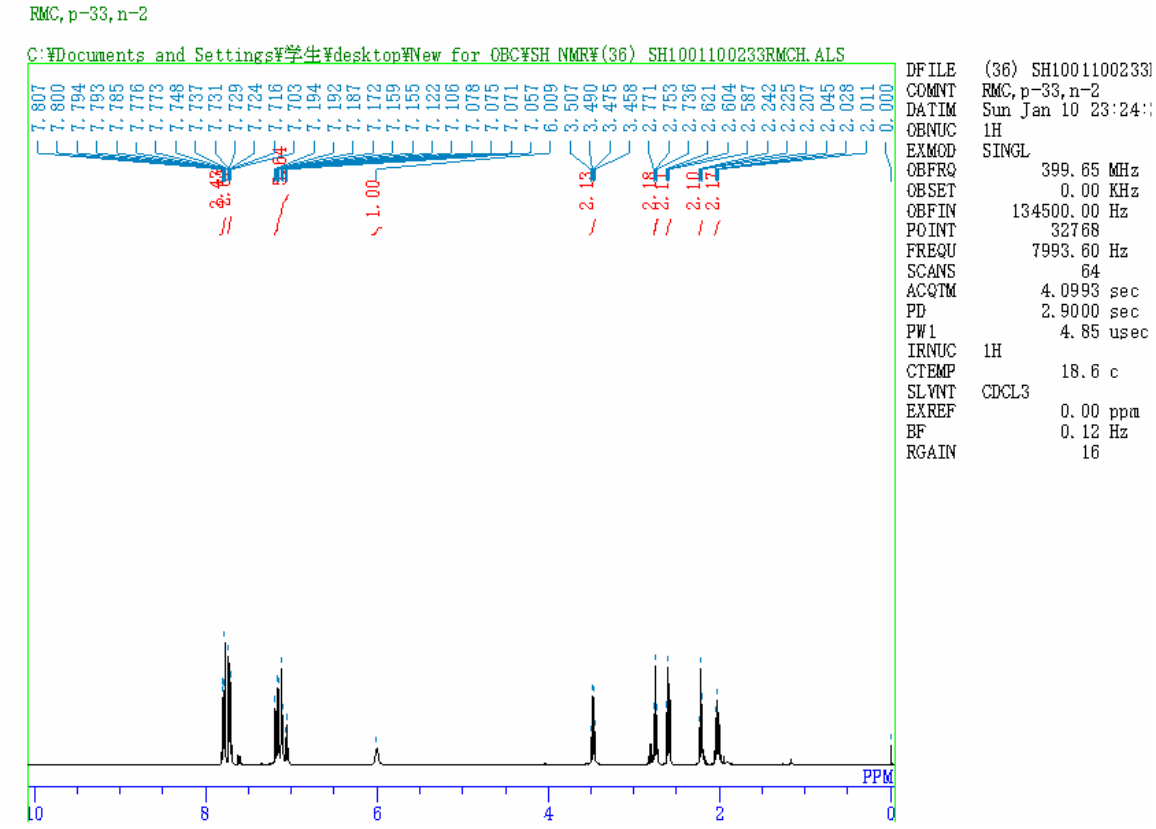
EXMOD	SINGL
OBFRQ	399.65 MHz
OBSET	0.00 KHz
OBFIN	134500.00 Hz
POINT	32768
FREQU	7993.60 Hz
SCANS	64
ACQTM	4.0993 sec
PD	2.9000 sec
PW1	4.85 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26

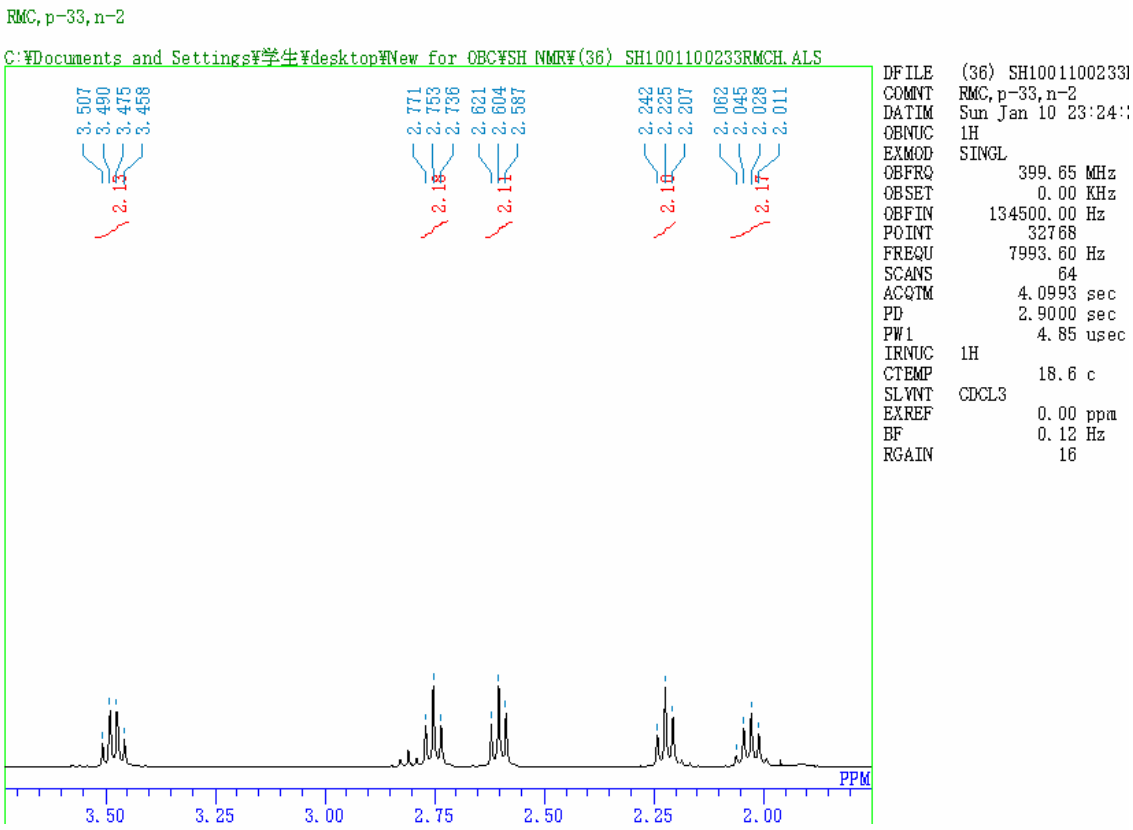
Phthalimido 4-(*N*-4-cholorobenzyloxycarbonyl)butanoate (35) (^{13}C-NMR)

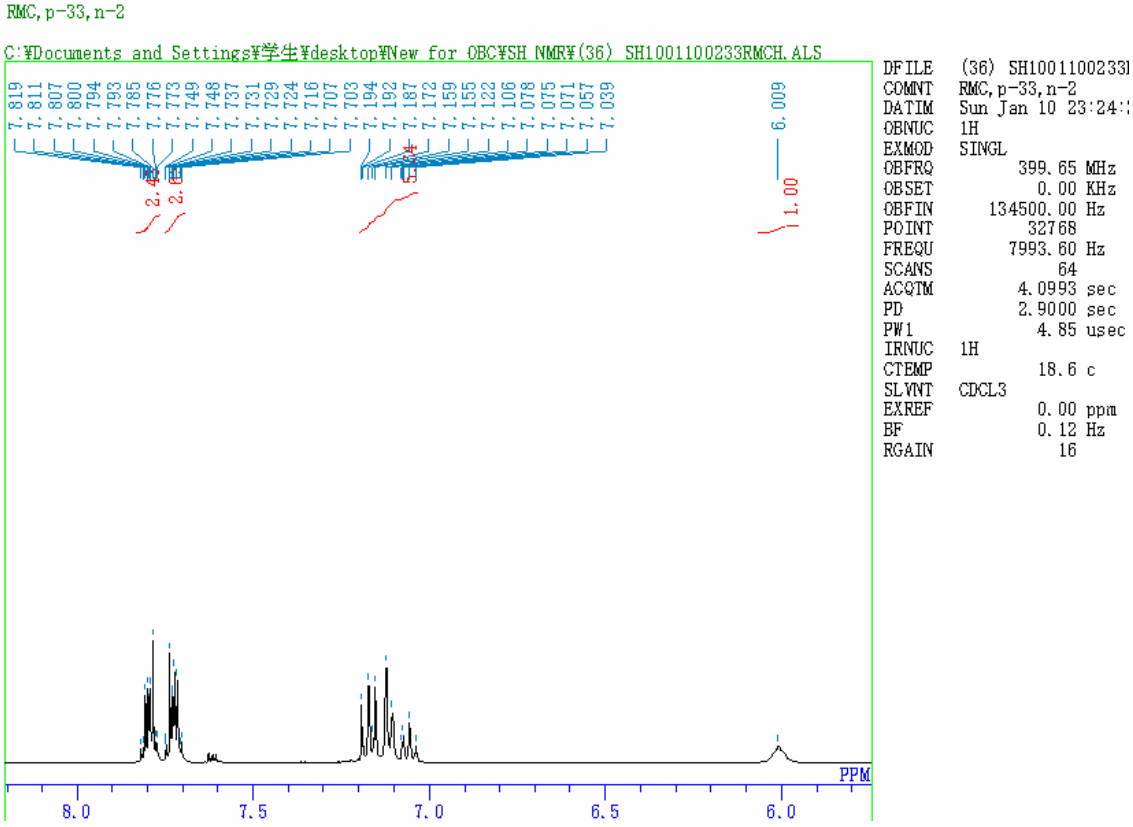




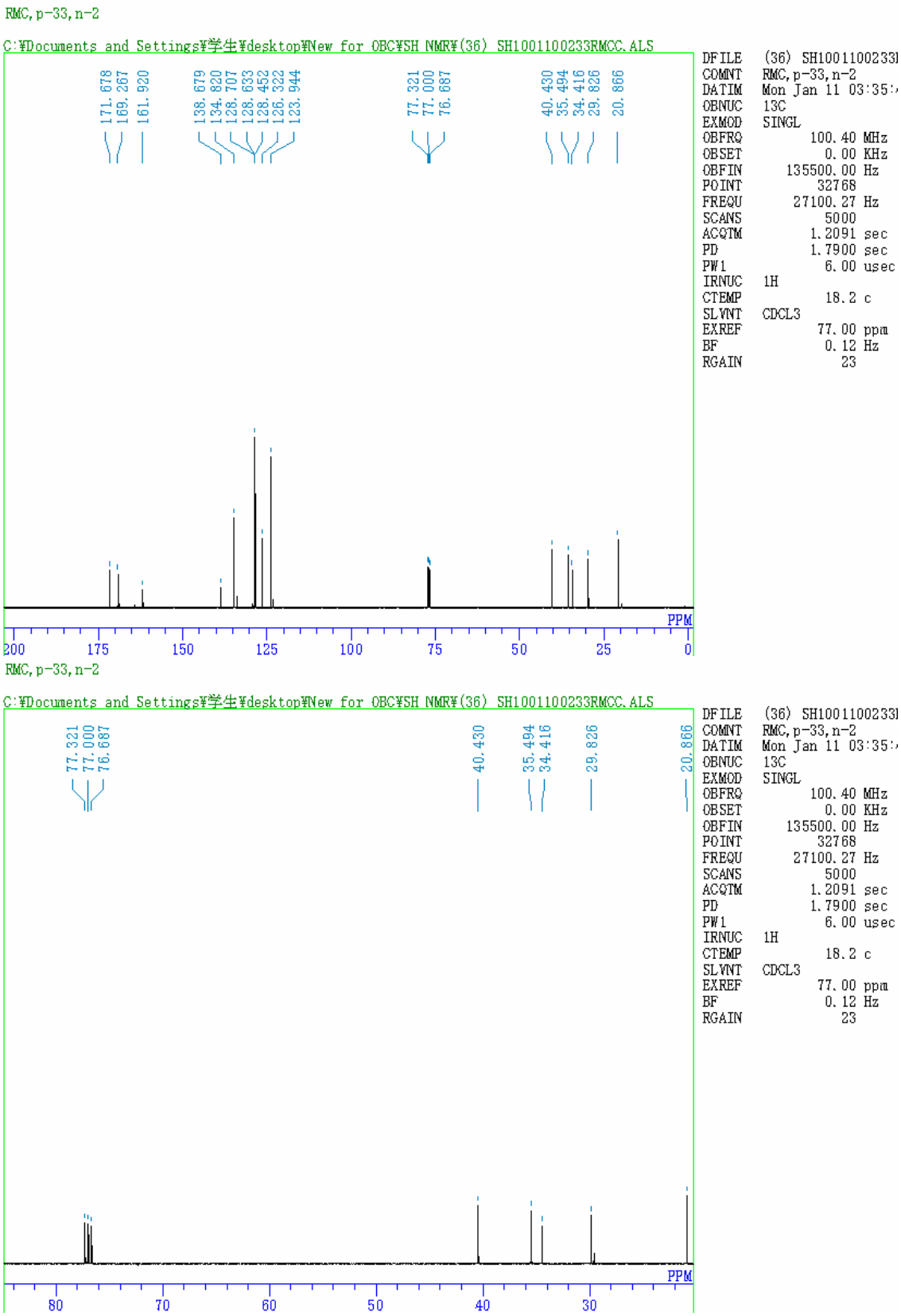
Phthalimido 4-(*N*-phenylethyloxycarbonyl)butanoate (36) (^{1}H-NMR)

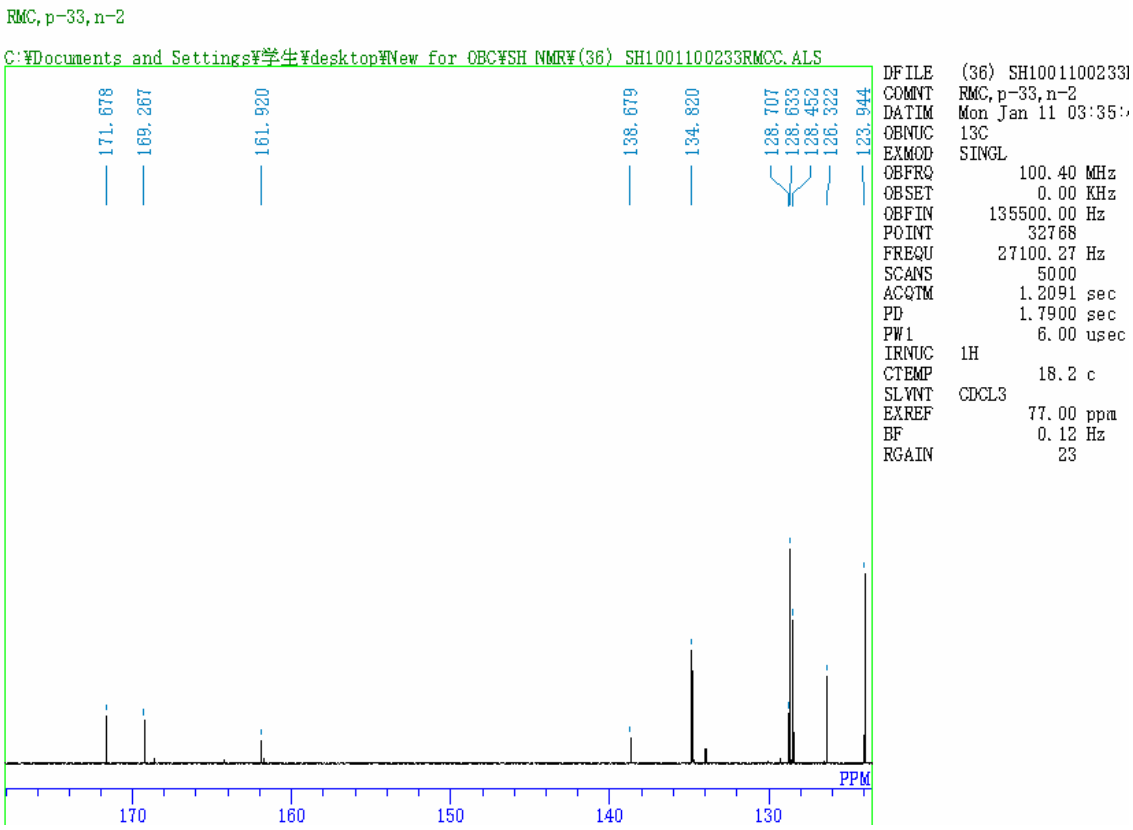



RMC,p-33,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(36) SH1001100233RMCH.ALS
3.507
3.490
3.475
3.458
2.771
2.753
2.736
2.621
2.604
2.587
2.242
2.225
2.207
2.062
2.045
2.028
2.011
2.13
2.18
2.11
2.10
2.17
PPM
3.50
3.25
3.00
2.75
2.50
2.25
2.00
DFILE (36) SH1001100233
COMNT RMC,p-33,n-2
DATIM Sun Jan 10 23:24:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16

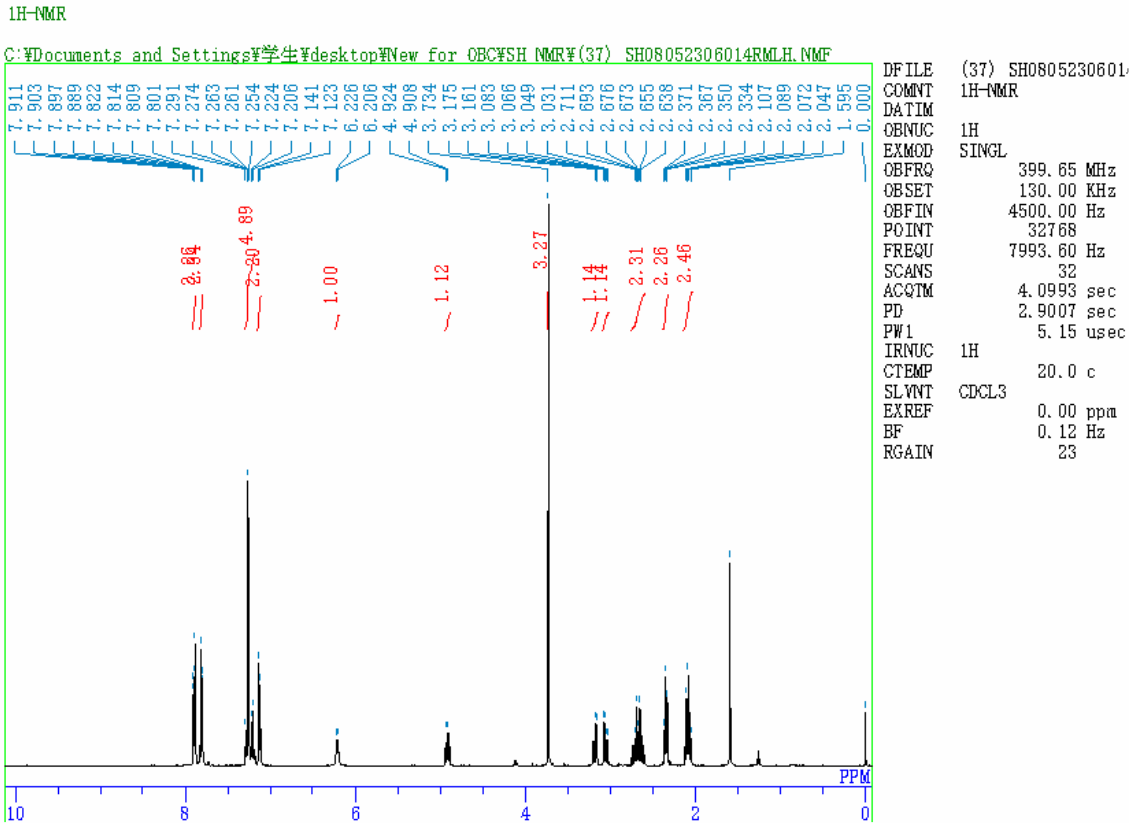

RMC,p-33,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(36) SH1001100233RMCH.ALS
7.819
7.811
7.807
7.800
7.794
7.793
7.785
7.776
7.773
7.749
7.748
7.737
7.731
7.729
7.724
7.716
7.707
7.703
7.194
7.192
7.187
7.172
7.159
7.155
7.122
7.106
7.078
7.075
7.071
7.057
7.039
6.009
1.00
PPM
8.0
7.5
7.0
6.5
6.0
DFILE (36) SH1001100233
COMNT RMC,p-33,n-2
DATIM Sun Jan 10 23:24:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16

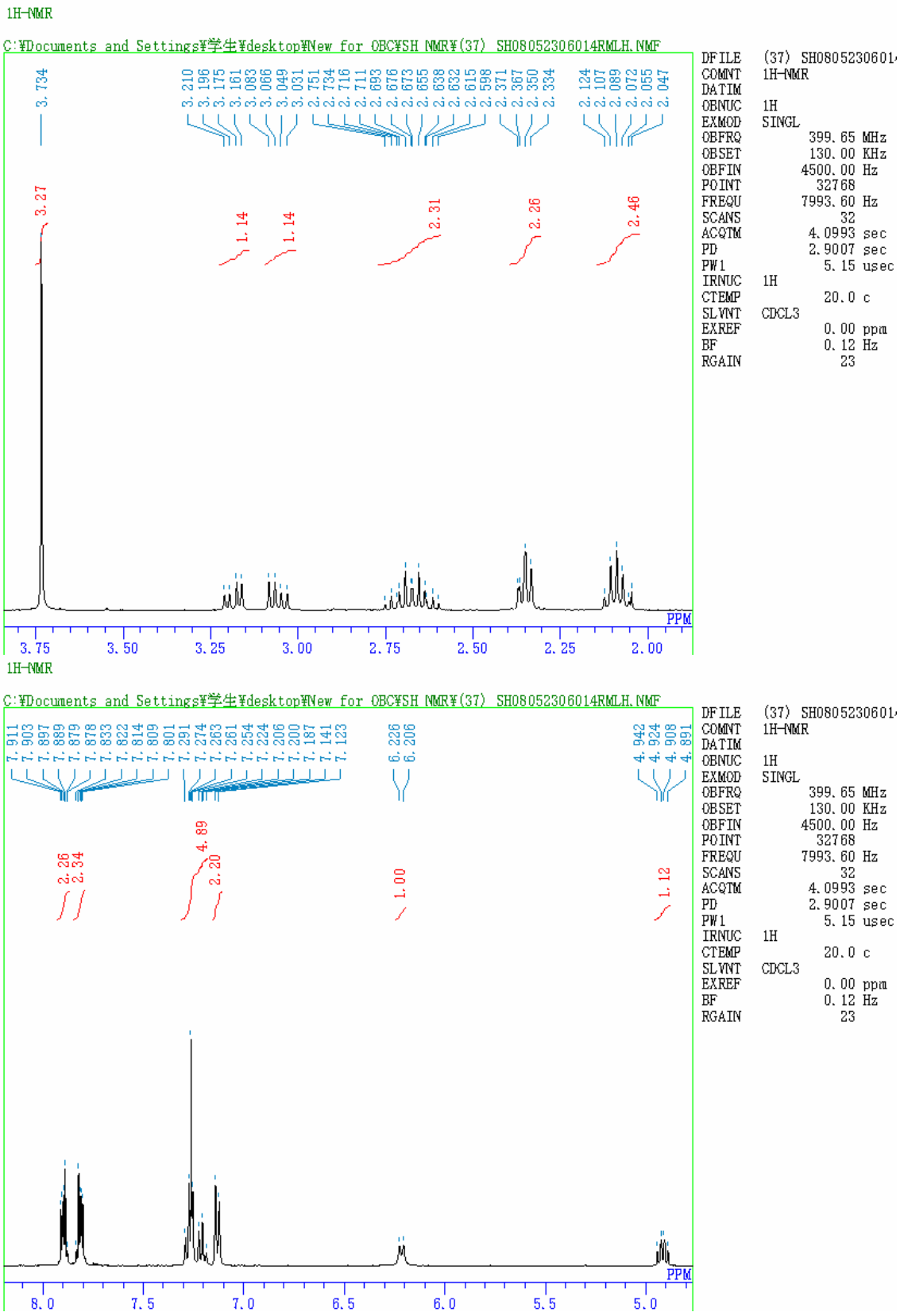
Phthalimido 4-(*N*-phenylethyloxycarbonyl)butanoate (36) (^{13}C-NMR)



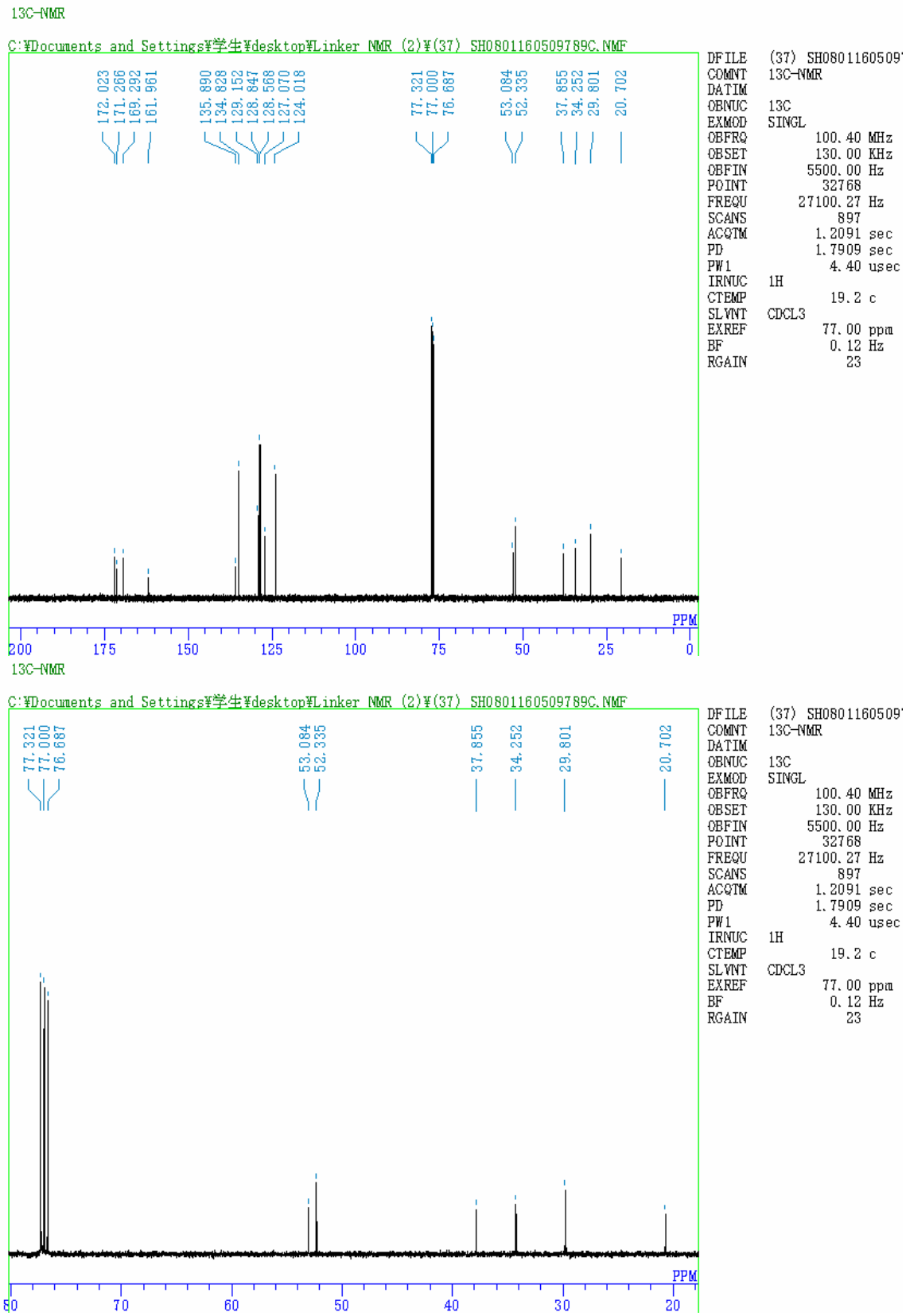


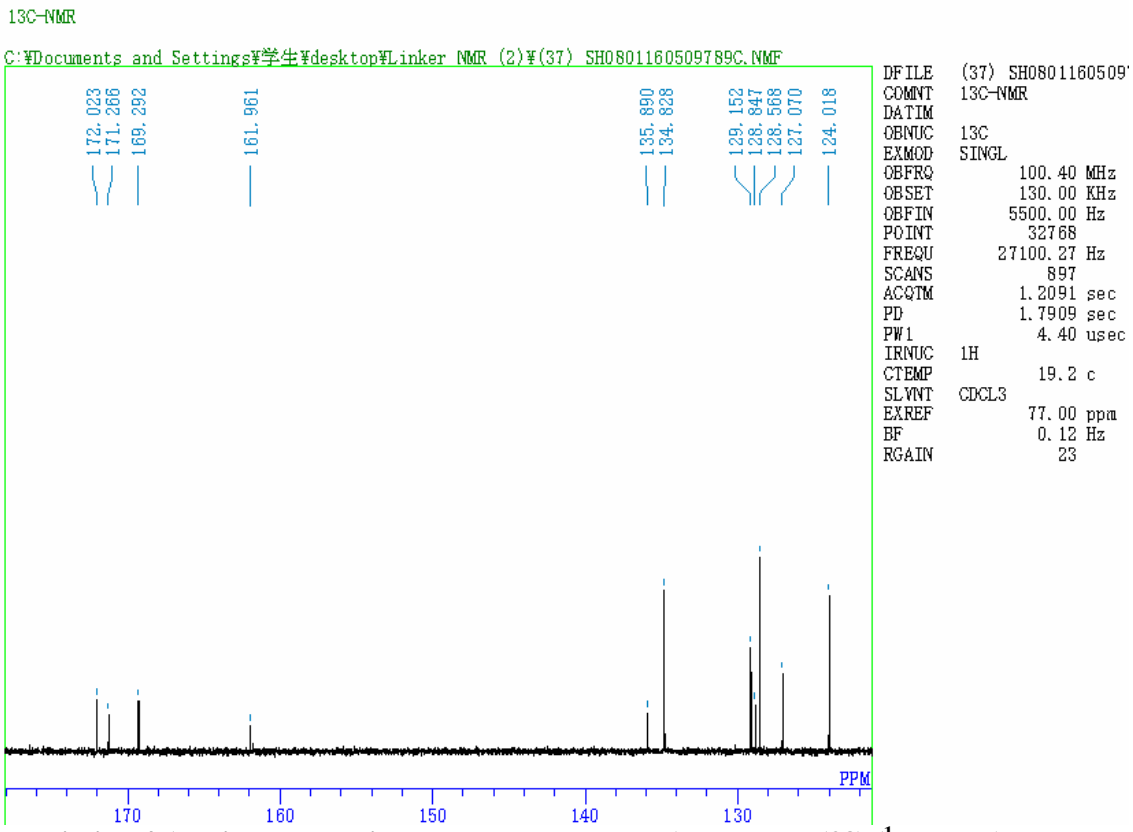
Phthalimido 4-(*N*-phenylalaninocarbonyl methyl ester)butanoate (37) (^{1}H-NMR)



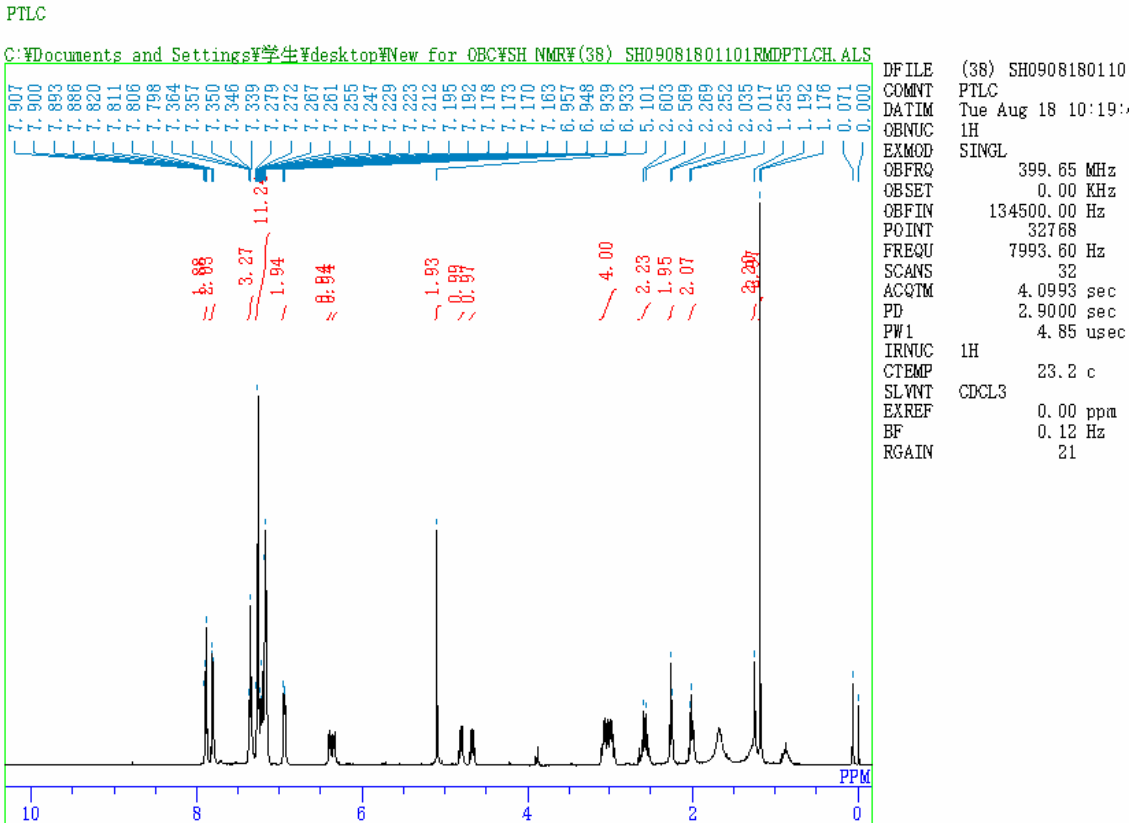

1H-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(37) SH08052306014RMLH.NMF
3.734
3.210
3.196
3.175
3.161
3.083
3.066
3.049
3.031
2.751
2.734
2.716
2.711
2.693
2.676
2.673
2.655
2.638
2.632
2.615
2.598
2.371
2.367
2.350
2.334
2.124
2.107
2.089
2.072
2.055
2.047
3.27
1.14
1.14
2.31
2.26
2.46
PPM
3.75
3.50
3.25
3.00
2.75
2.50
2.25
2.00
DFILE (37) SH0805230601
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23
1H-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(37) SH08052306014RMLH.NMF
7.911
7.903
7.897
7.889
7.879
7.878
7.833
7.822
7.814
7.809
7.801
7.291
7.274
7.263
7.261
7.254
7.224
7.206
7.200
7.187
7.141
7.123
6.226
6.206
4.942
4.924
4.908
4.891
2.26
2.34
4.89
2.20
1.00
1.12
PPM
8.0
7.5
7.0
6.5
6.0
5.5
5.0
DFILE (37) SH0805230601
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 23

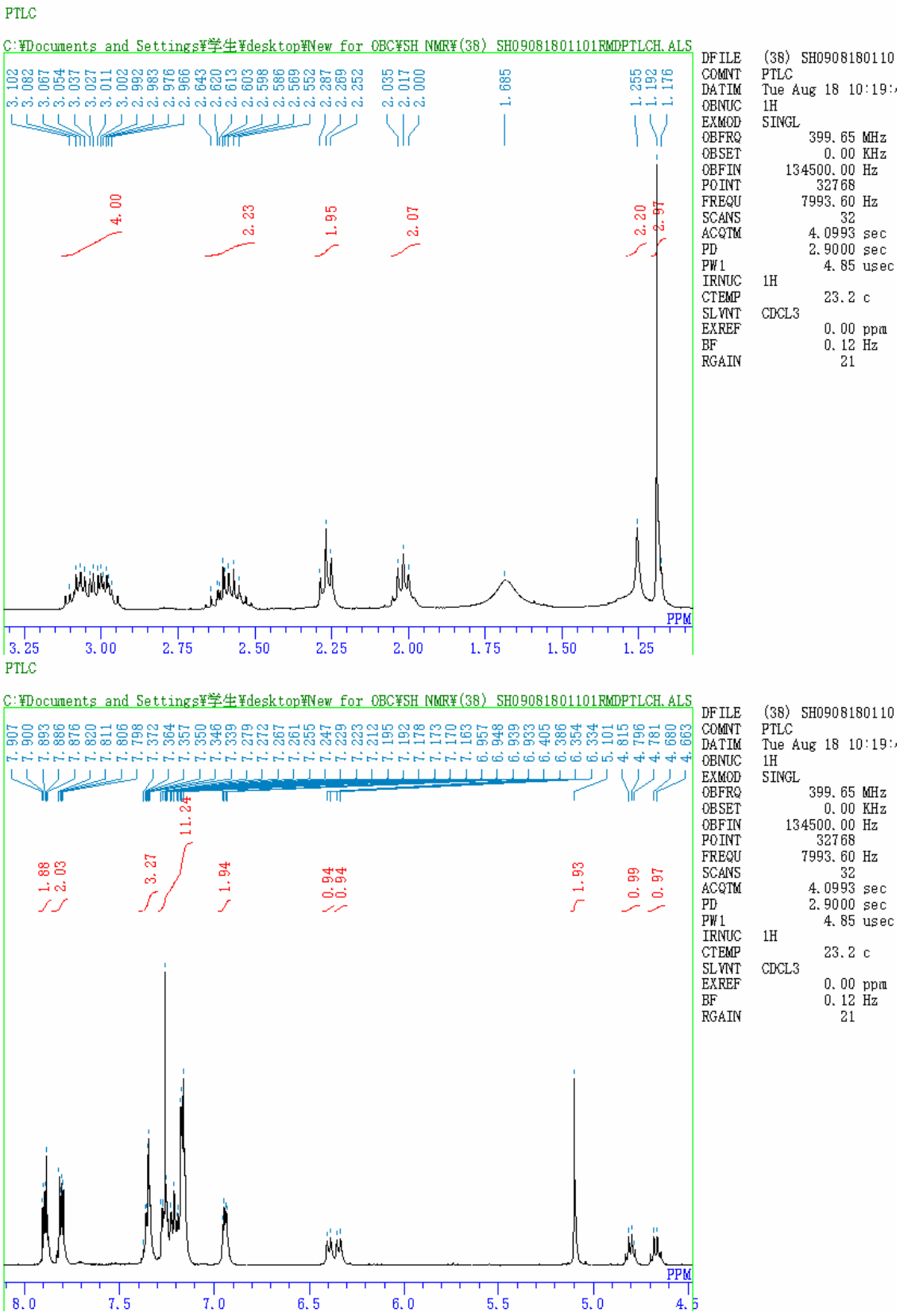
Phthalimido 4-(*N*-phenylalaninocarbonyl methyl ester)butanoate (37) (^{13}C-NMR)



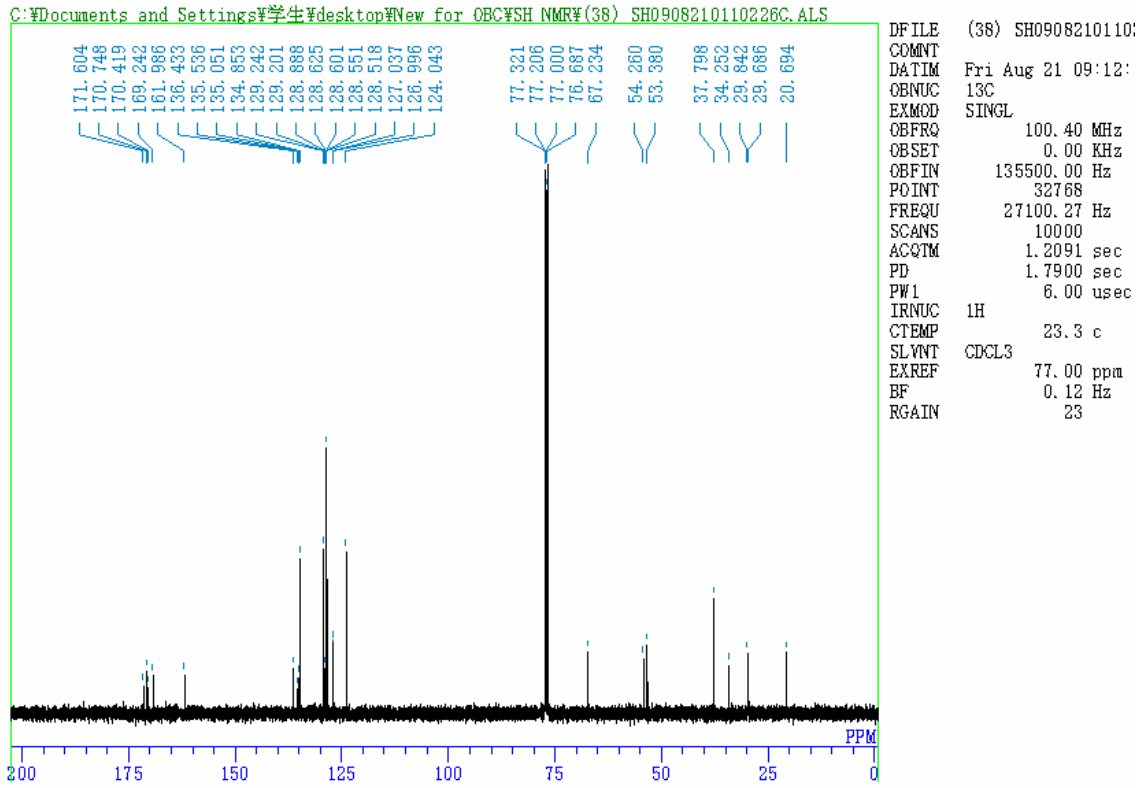


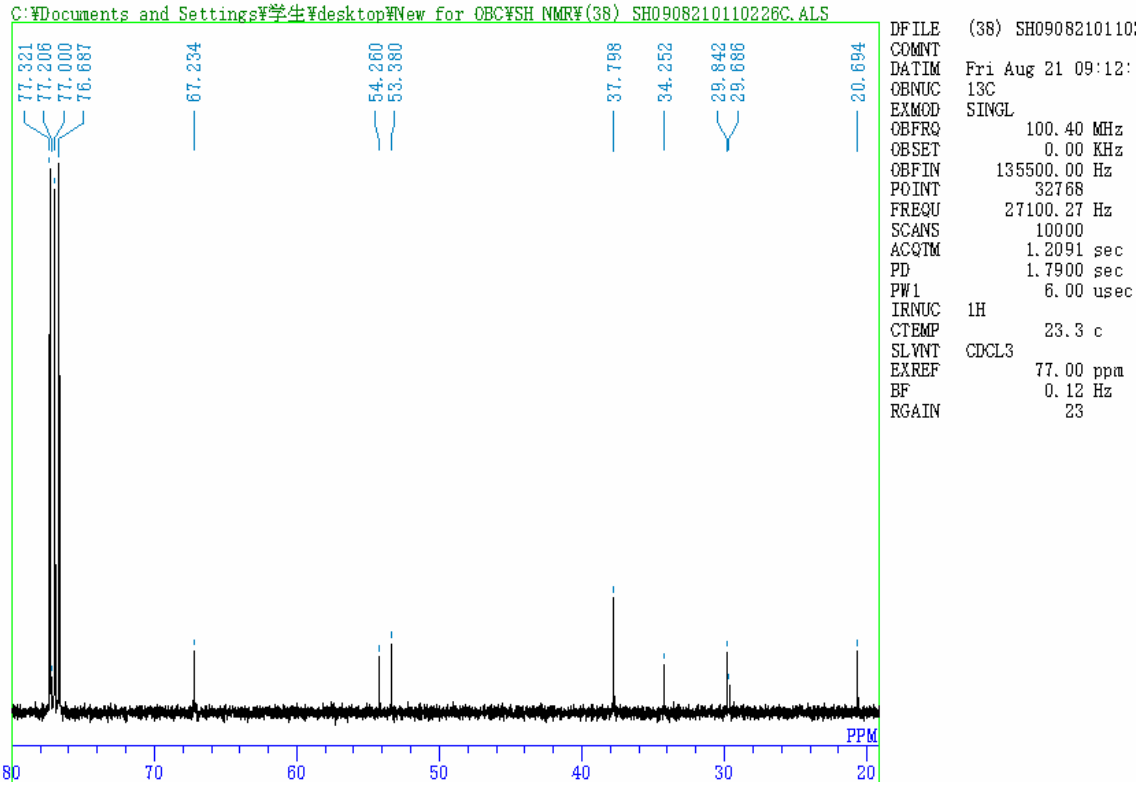
Phthalimido 4-(*N*-diphenylalaninocarbonyl benzyl ester)butanoate (38) (^{1}H-NMR)

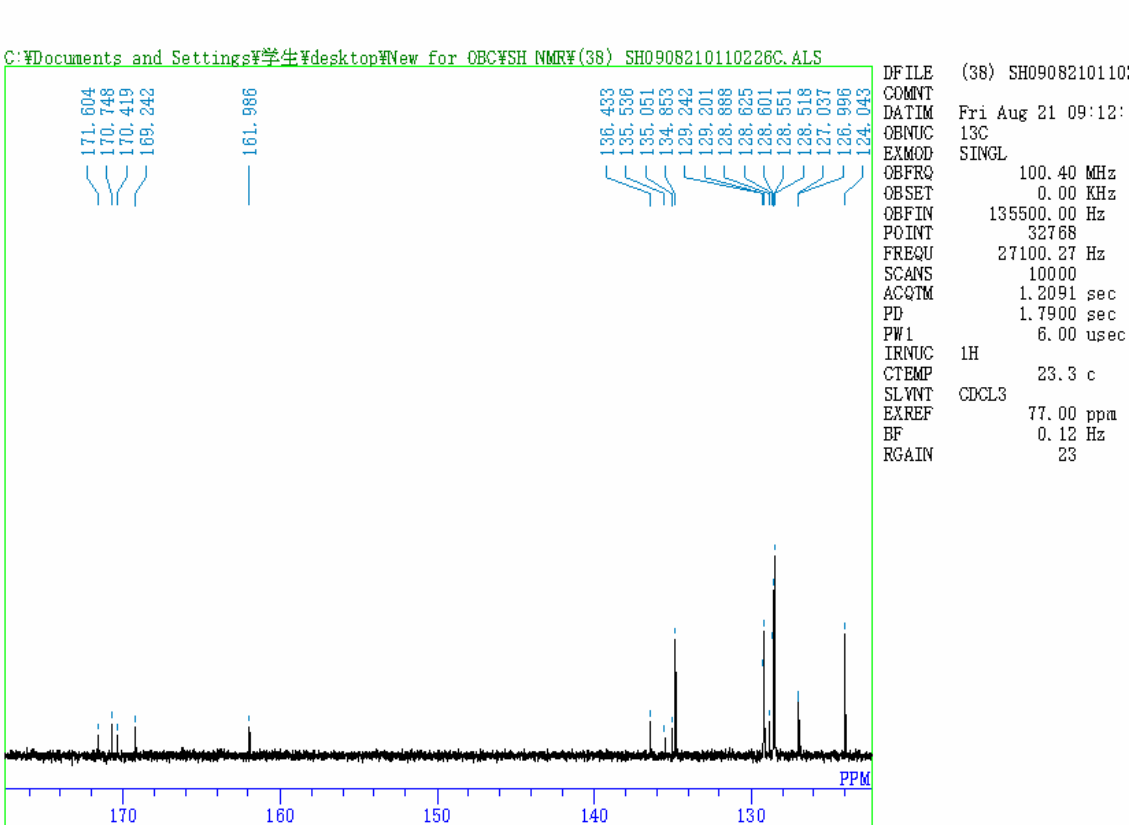



PTLC
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(38) SH0908180110RMDPTLCH.ALS
3.102 3.082 3.067 3.054 3.037 3.027 3.011 3.002 2.992 2.983 2.976 2.966 2.643 2.620 2.613 2.603 2.598 2.586 2.569 2.552 2.287 2.269 2.252 2.035 2.017 2.000 1.685 1.255 1.192 1.176
4.00 2.23 1.95 2.07 2.20 2.97
PPM
3.25 3.00 2.75 2.50 2.25 2.00 1.75 1.50 1.25
DFILE (38) SH0908180110
COMNT PTLC
DATIM Tue Aug 18 10:19:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 21
PTLC
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(38) SH0908180110RMDPTLCH.ALS
7.907 7.900 7.893 7.886 7.876 7.820 7.811 7.806 7.798 7.372 7.364 7.357 7.350 7.346 7.339 7.279 7.272 7.267 7.261 7.255 7.247 7.229 7.223 7.212 7.195 7.192 7.178 7.173 7.170 7.163 6.957 6.948 6.939 6.933 6.405 6.386 6.354 6.334 5.101 4.815 4.796 4.781 4.680 4.663
1.88 2.03 3.27 11.24 1.94 0.94 0.94 1.93 0.99 0.97
PPM
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5
DFILE (38) SH0908180110
COMNT PTLC
DATIM Tue Aug 18 10:19:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 21

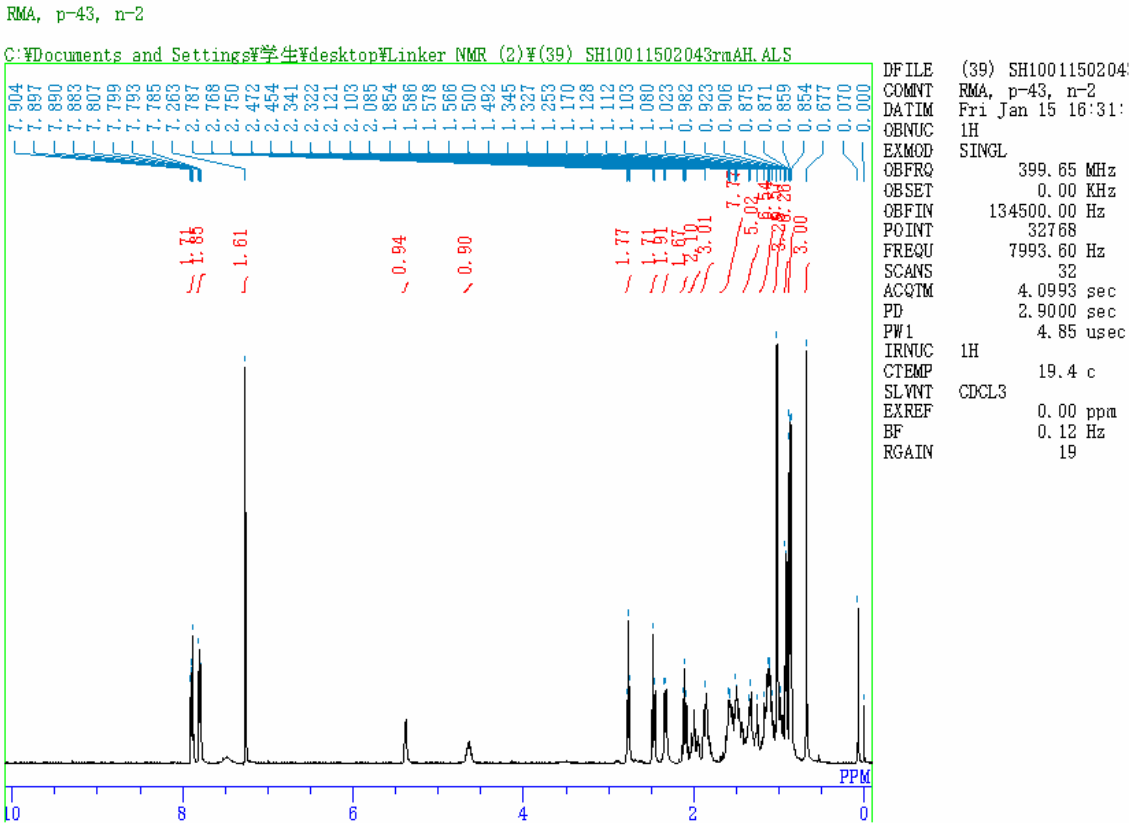
Phthalimido 4-(*N*-diphenylalaninocarbonyl benzyl ester)butanoate (38) (^{13}C-NMR)

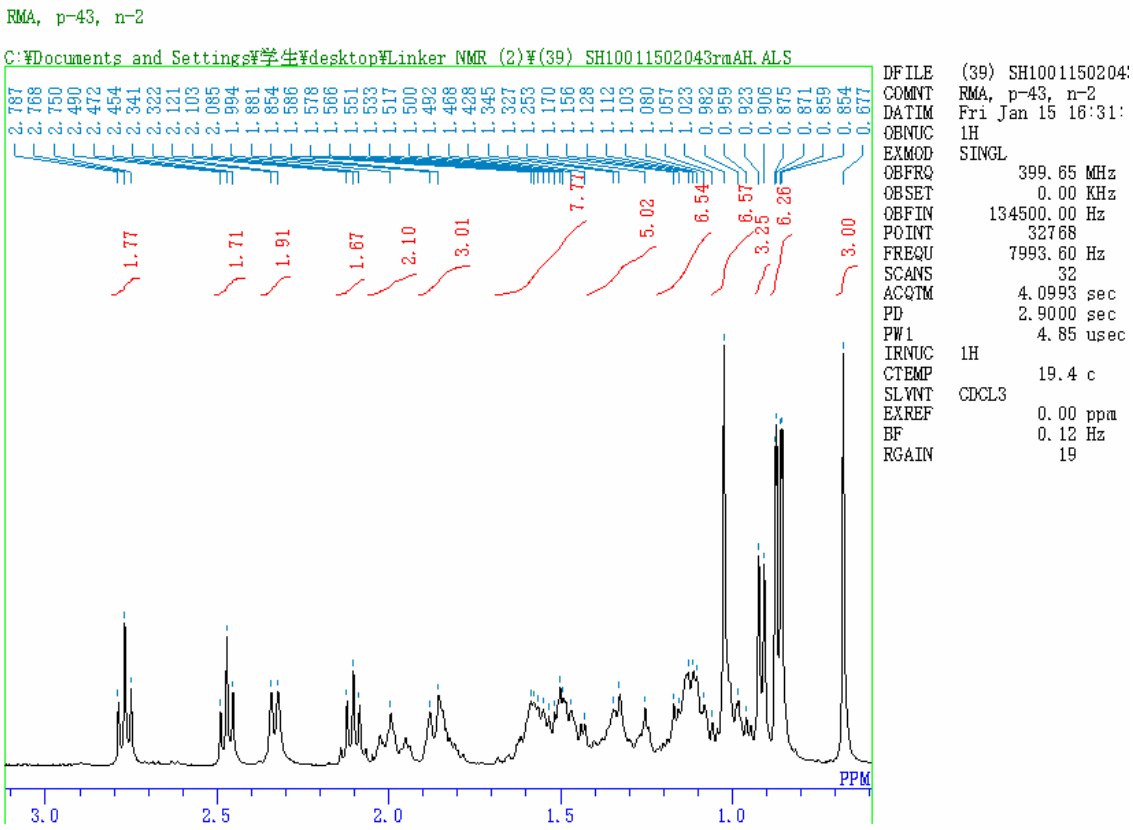


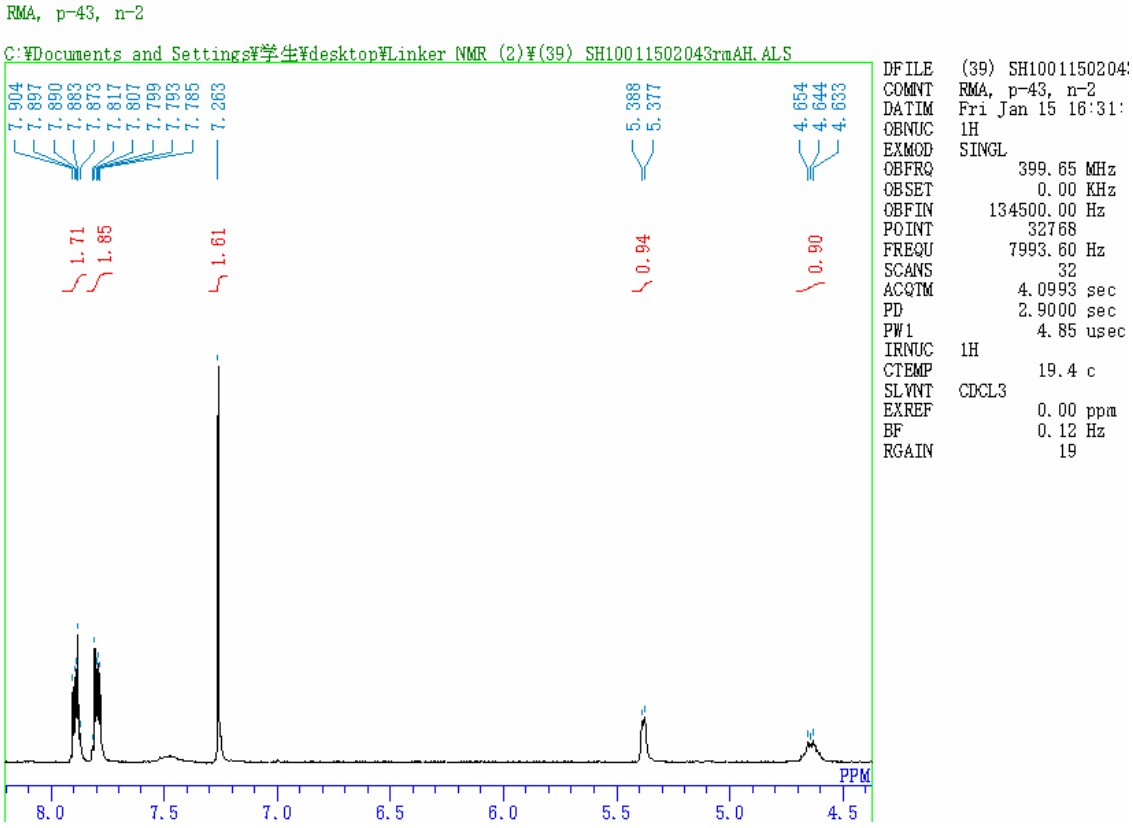




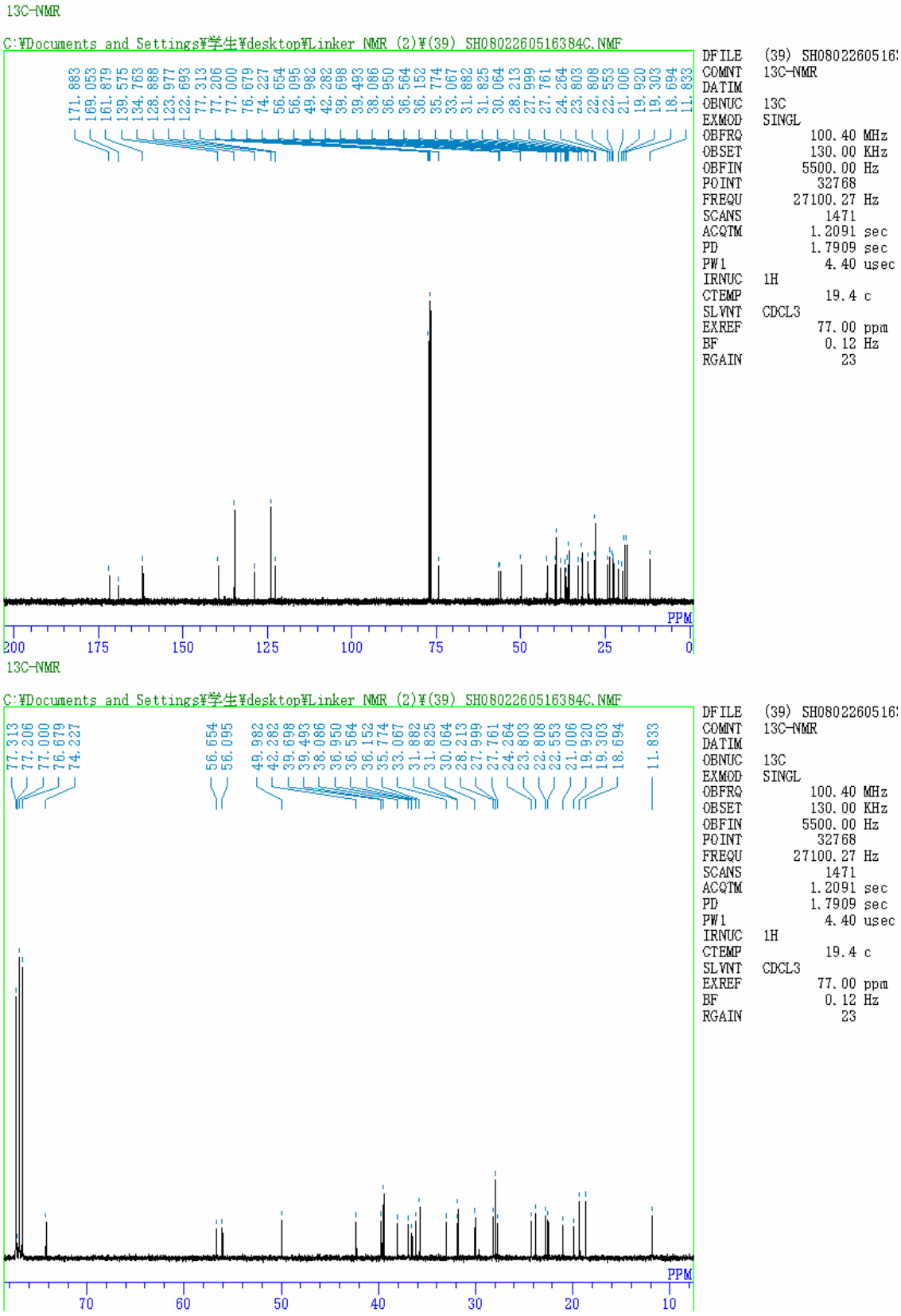
3β-cholesteryl 4-(phthalimidoyloxycarbonyl)butyrate (39) (^{1}H-NMR)

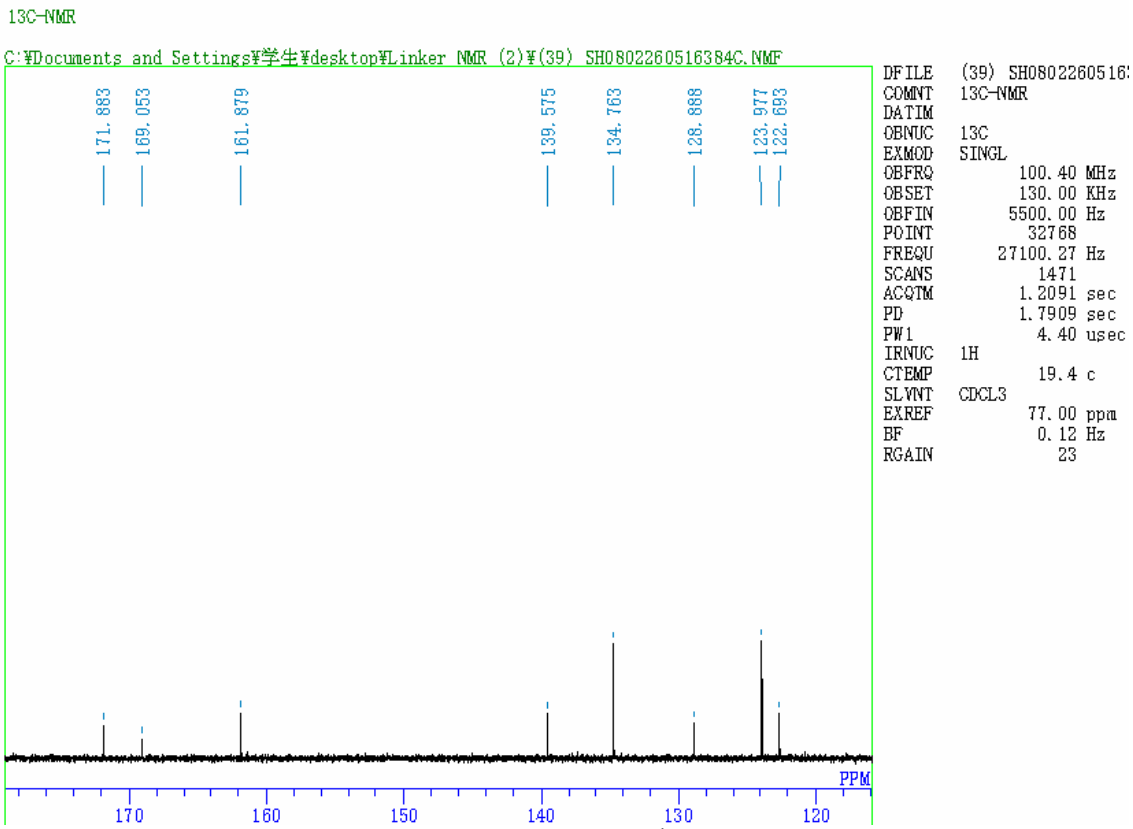



RMA, p-43, n-2
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(39) SH10011502043rmAH.ALS
DFILE (39) SH1001150204
COMNT RMA, p-43, n-2
DATIM Fri Jan 15 16:31:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
PPM

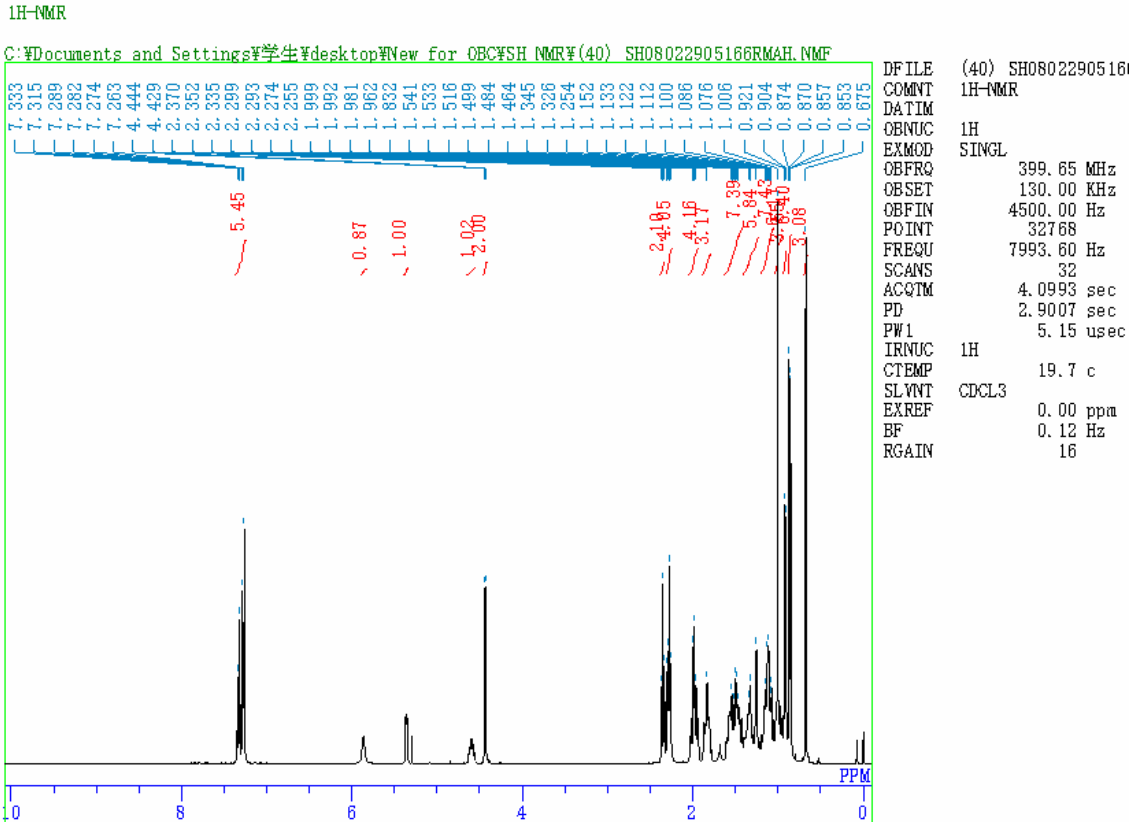

RMA, p-43, n-2
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(39) SH10011502043rmAH.ALS
DFILE (39) SH1001150204
COMNT RMA, p-43, n-2
DATIM Fri Jan 15 16:31:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19
PPM

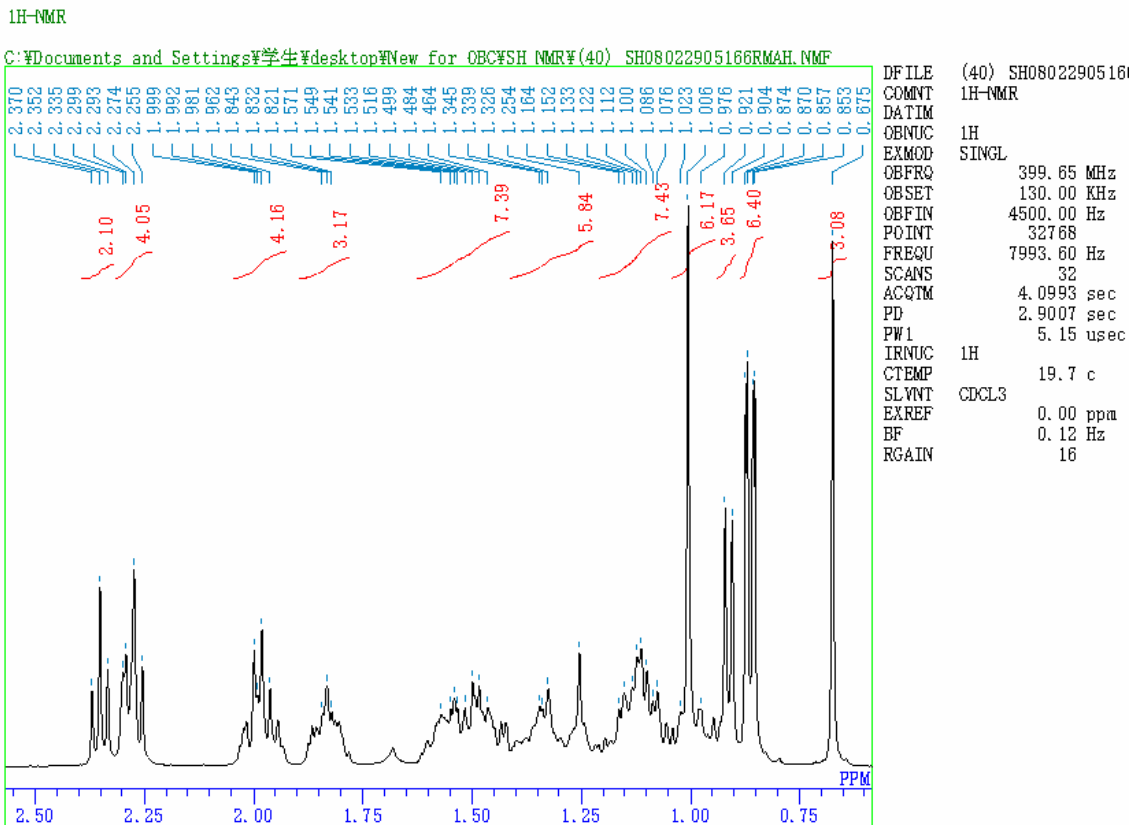
3β-cholesteryl 4-(phthalimidoyloxycarbonyl)butyrate (39) (^{13}C-NMR)

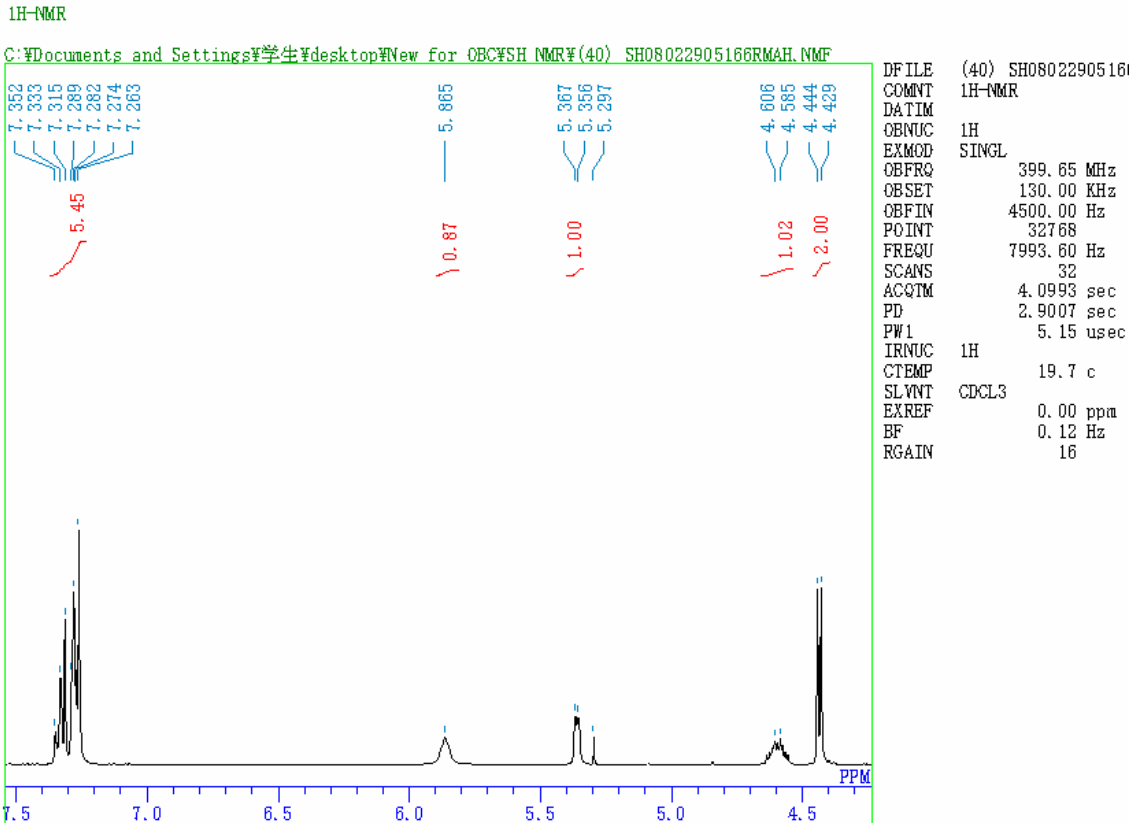




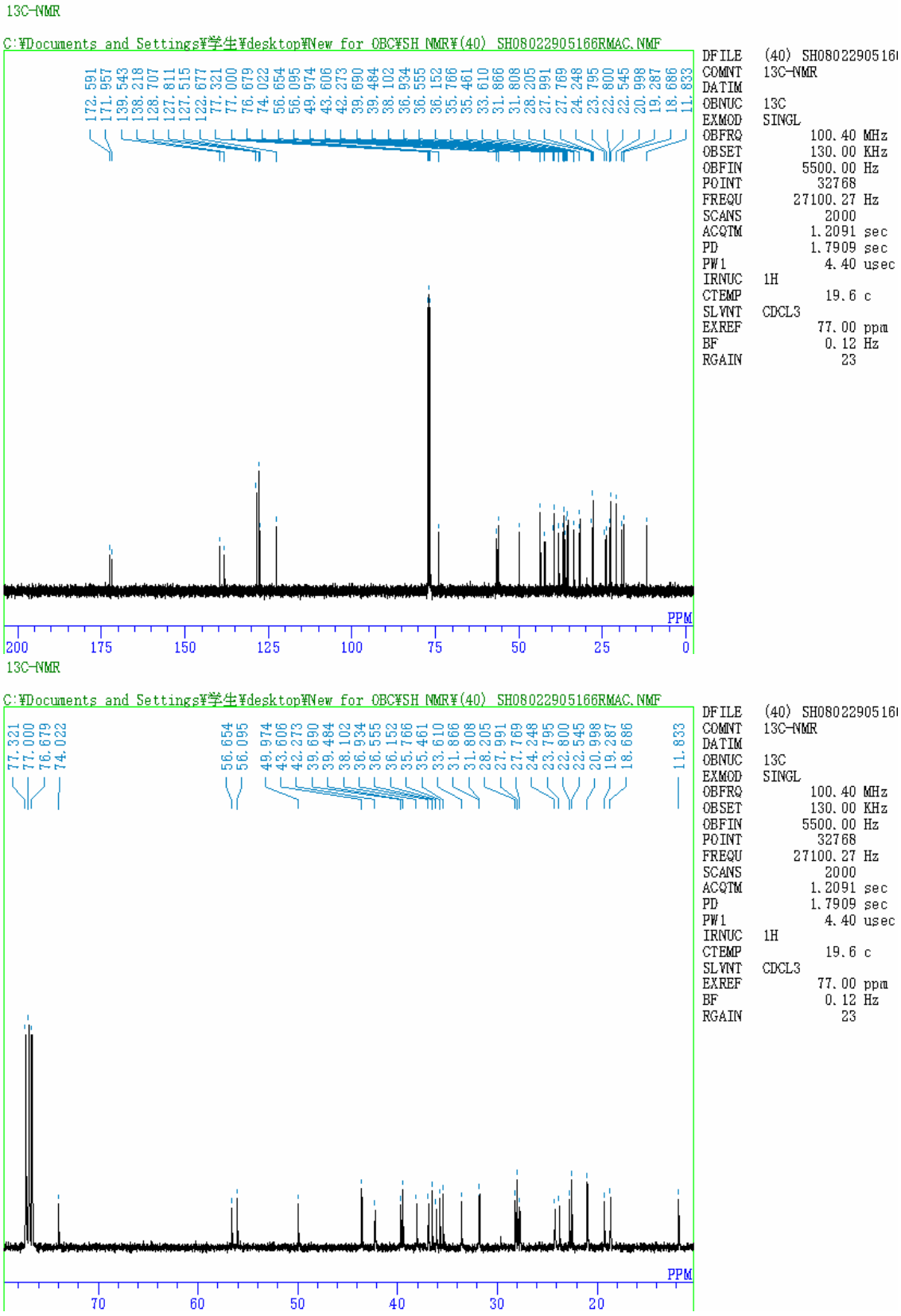
3β-cholesteryl 4-(benzylaminocarbonyl)butyrate (40) (^{1}H-NMR)

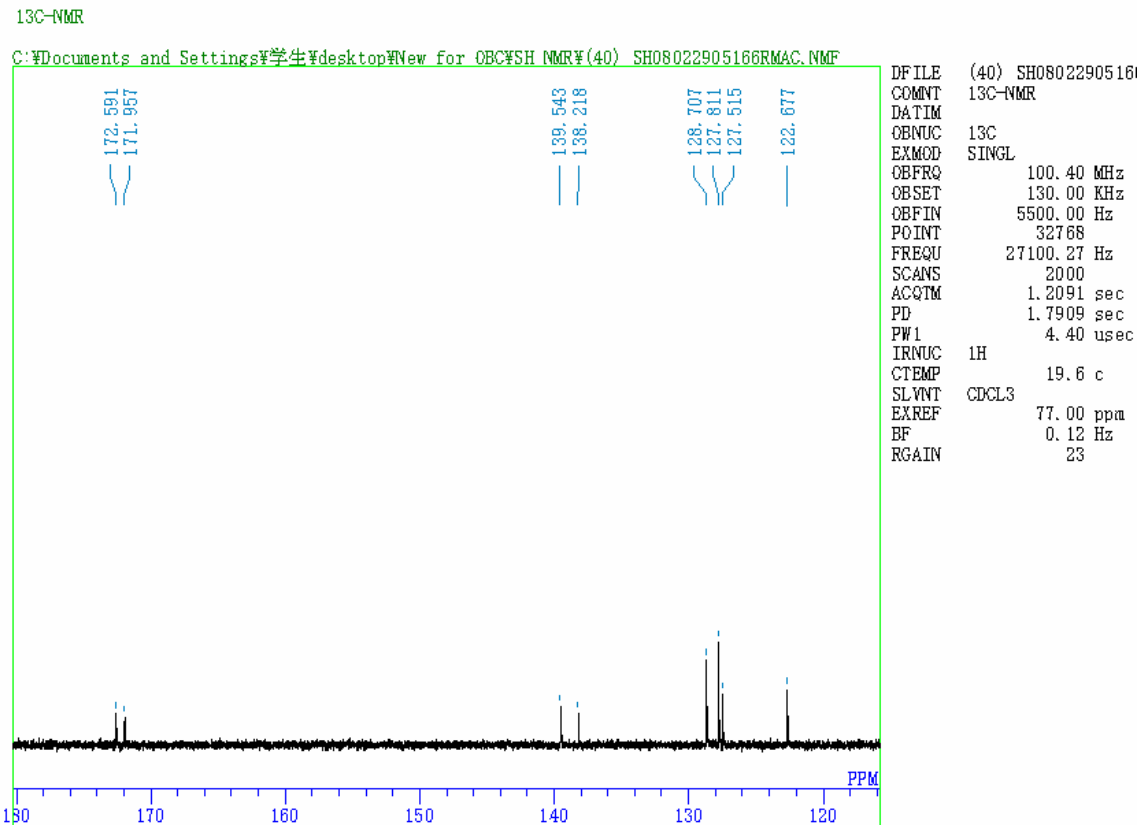



1H-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(40) SH08022905166RMAH.NMF
2.10
4.05
4.16
3.17
7.39
5.84
7.43
6.17
3.65
6.40
3.08
PPM
2.50
2.25
2.00
1.75
1.50
1.25
1.00
0.75
DFILE (40) SH08022905166
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16

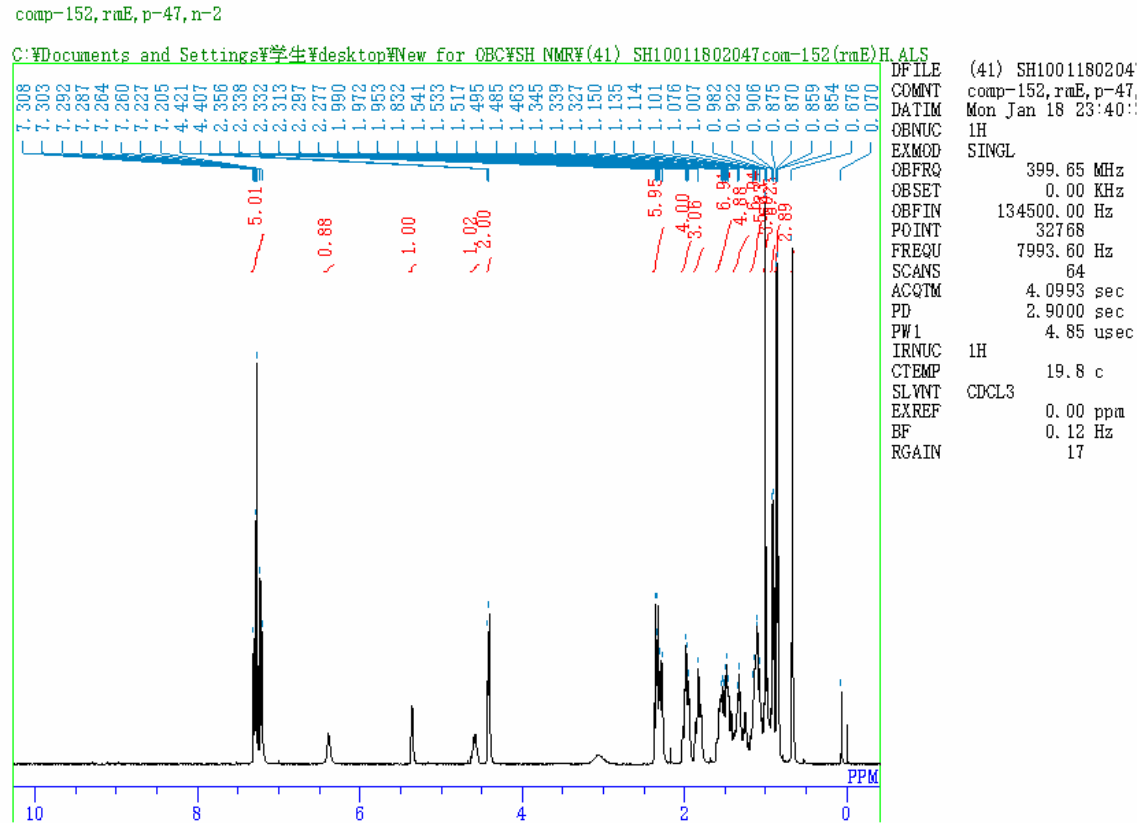

1H-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(40) SH08022905166RMAH.NMF
5.45
0.87
1.00
1.02
2.00
PPM
7.5
7.0
6.5
6.0
5.5
5.0
4.5
DFILE (40) SH08022905166
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16

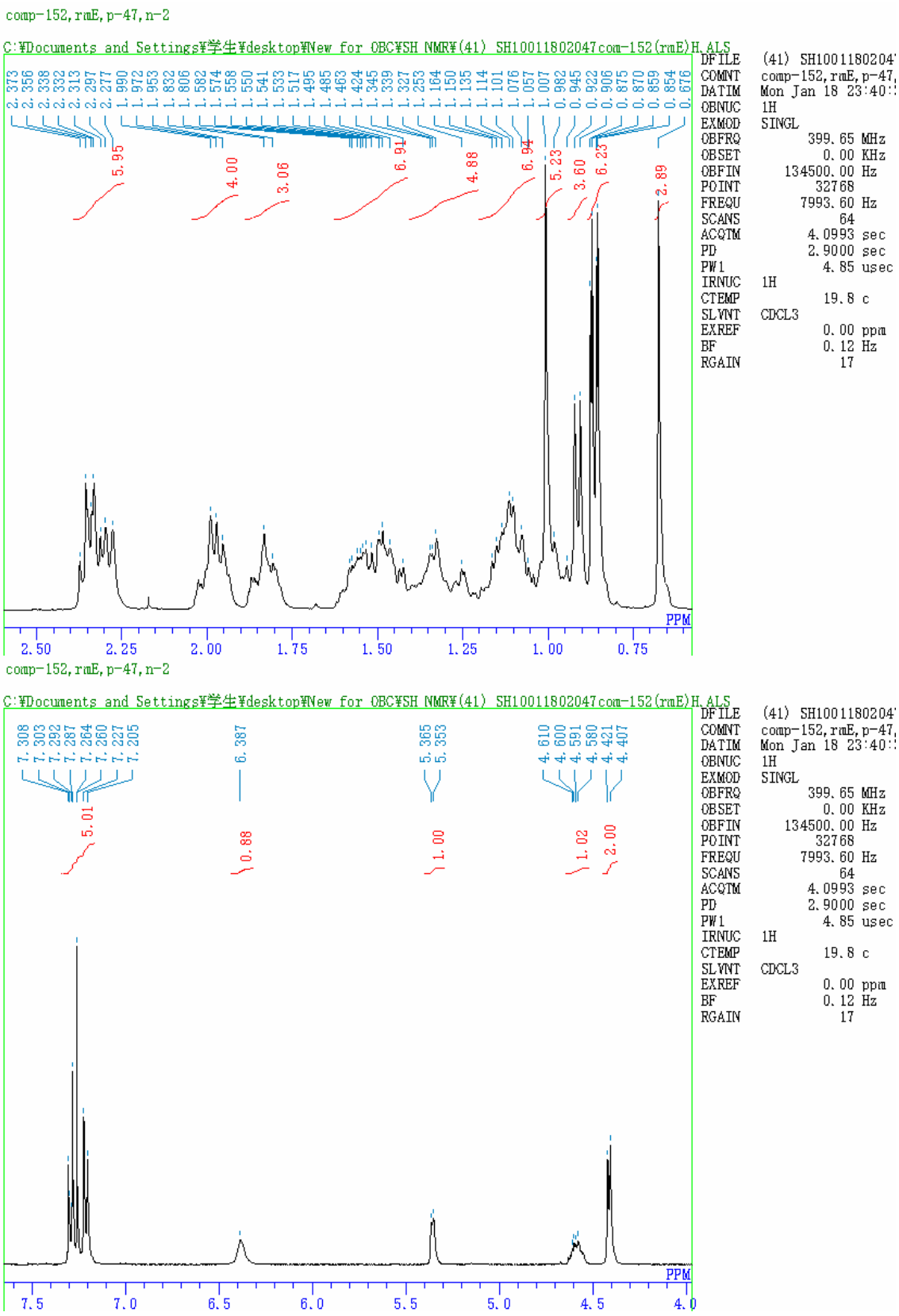
3β-cholesteryl 4-(benzylaminocarbonyl)butyrate (40) (^{13}C-NMR)



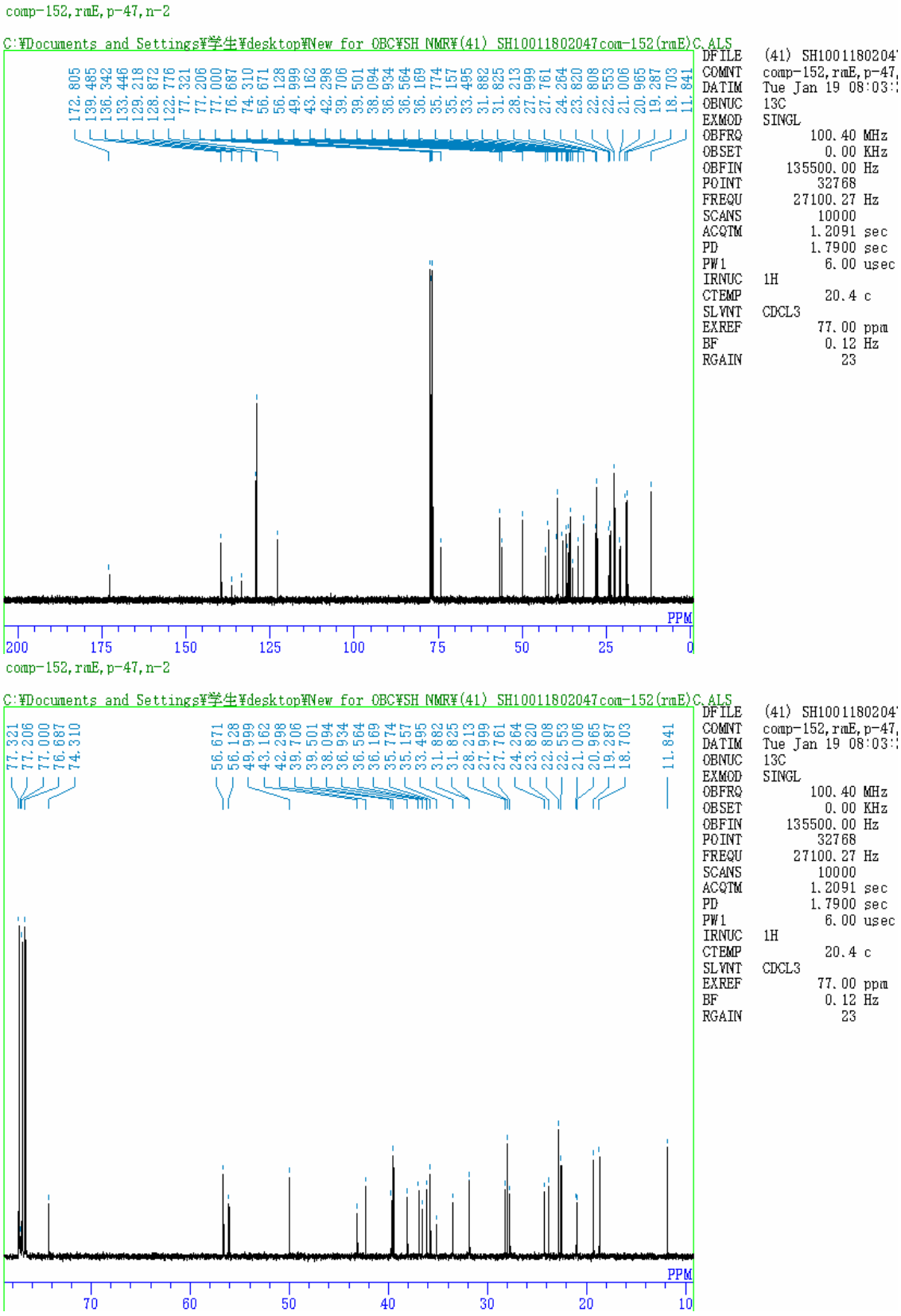


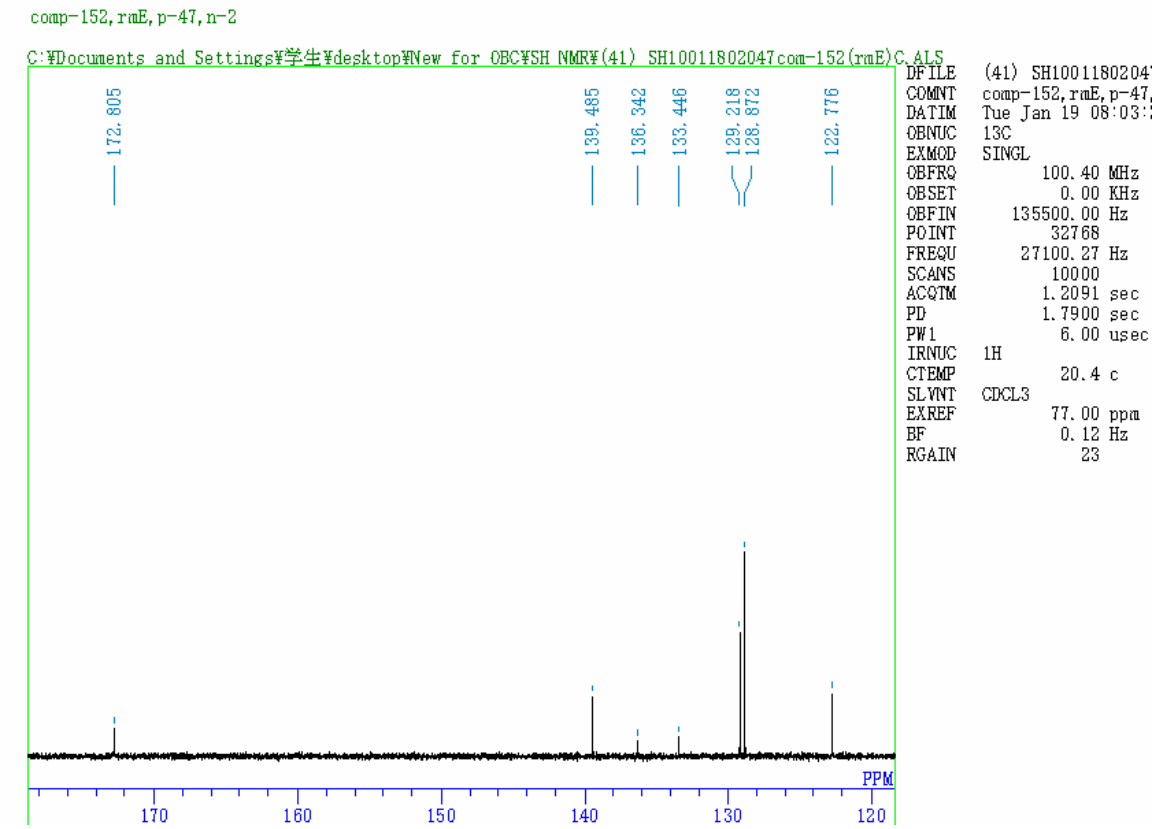
3β-cholesteryl 4-(4-cholorobenzylaminocarbonyl)butyrate (41) (^{1}H-NMR)




comp-152,rmE,p-47,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(41) SH10011802047com-152(rmE)H.ALS
DFILE (41) SH1001180204
COMNT comp-152,rmE,p-47,
DATIM Mon Jan 18 23:40:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17
PPM
comp-152,rmE,p-47,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(41) SH10011802047com-152(rmE)H.ALS
PPM

3β-cholesteryl 4-(4-cholorobenzylaminocarbonyl)butyrate (41) (^{13}C-NMR)



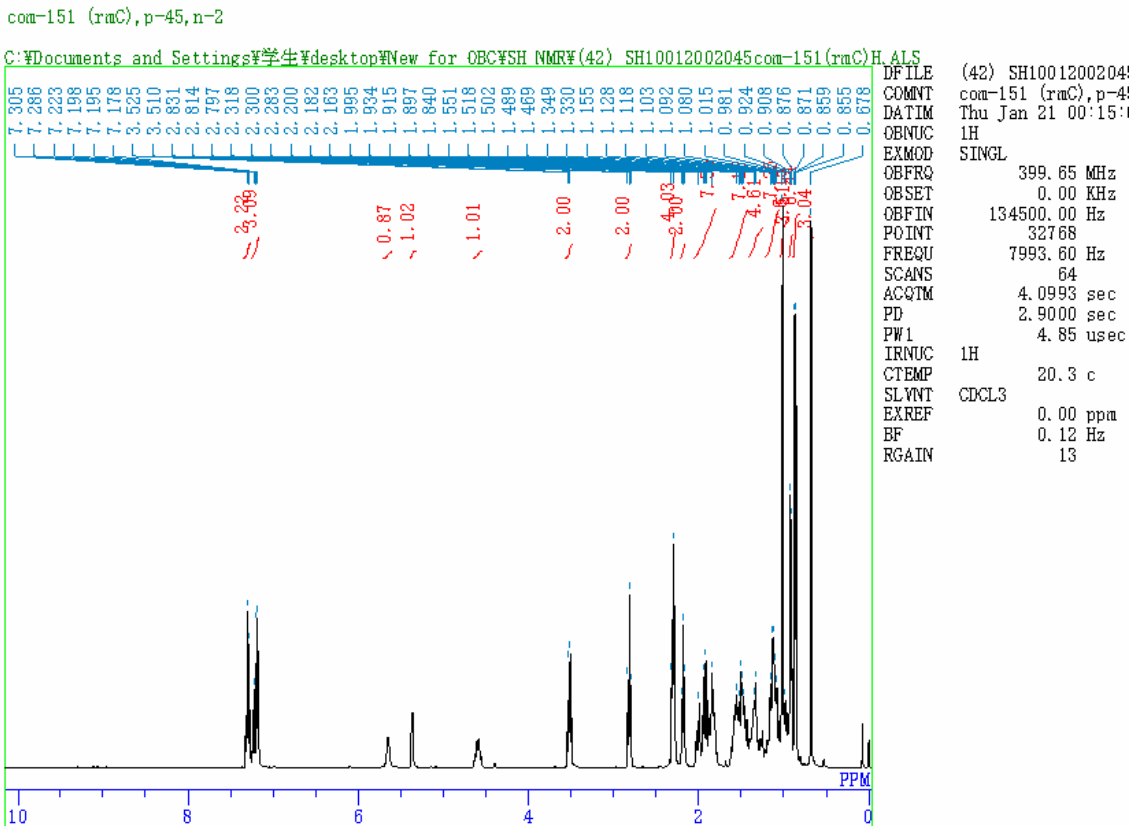

comp-152,rmE,p-47,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(41) SH10011802047com-152(rmE)C.ALS
172.805
139.485
136.342
133.446
129.218
128.872
122.776
DFILE (41) SH1001180204
COMNT comp-152,rmE,p-47,
DATIM Tue Jan 19 08:03:
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 10000
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
PPM
170
160
150
140
130
120

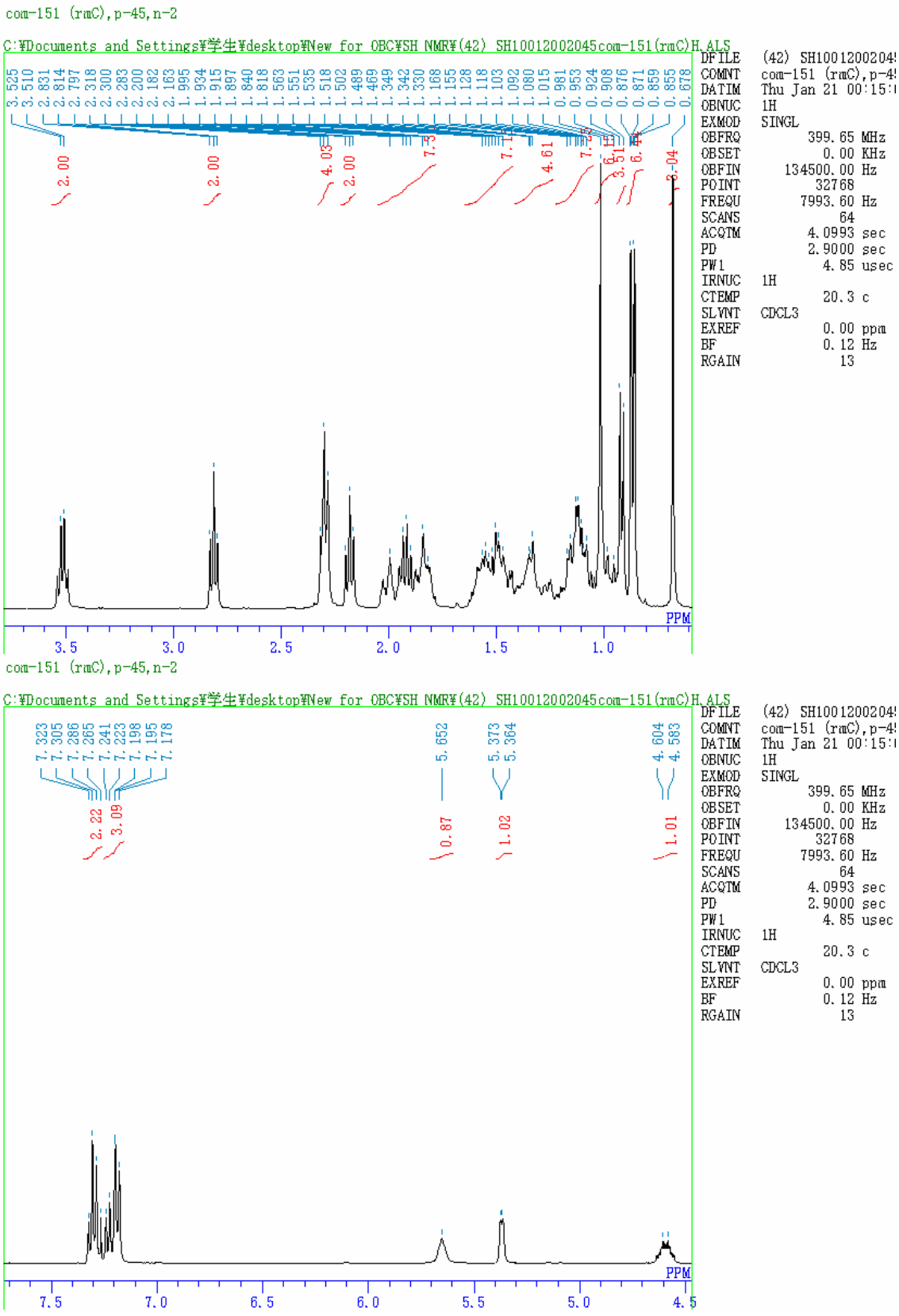
(vi) Supporting Information for the manuscript:

Title: "Syntheses and reactivities of non-symmetrical "active ester" bi-dentate cross-linking reagents having a phthalimidoyl and acid chloride, 2-benzothiazole, or 1-benzotriazole group"

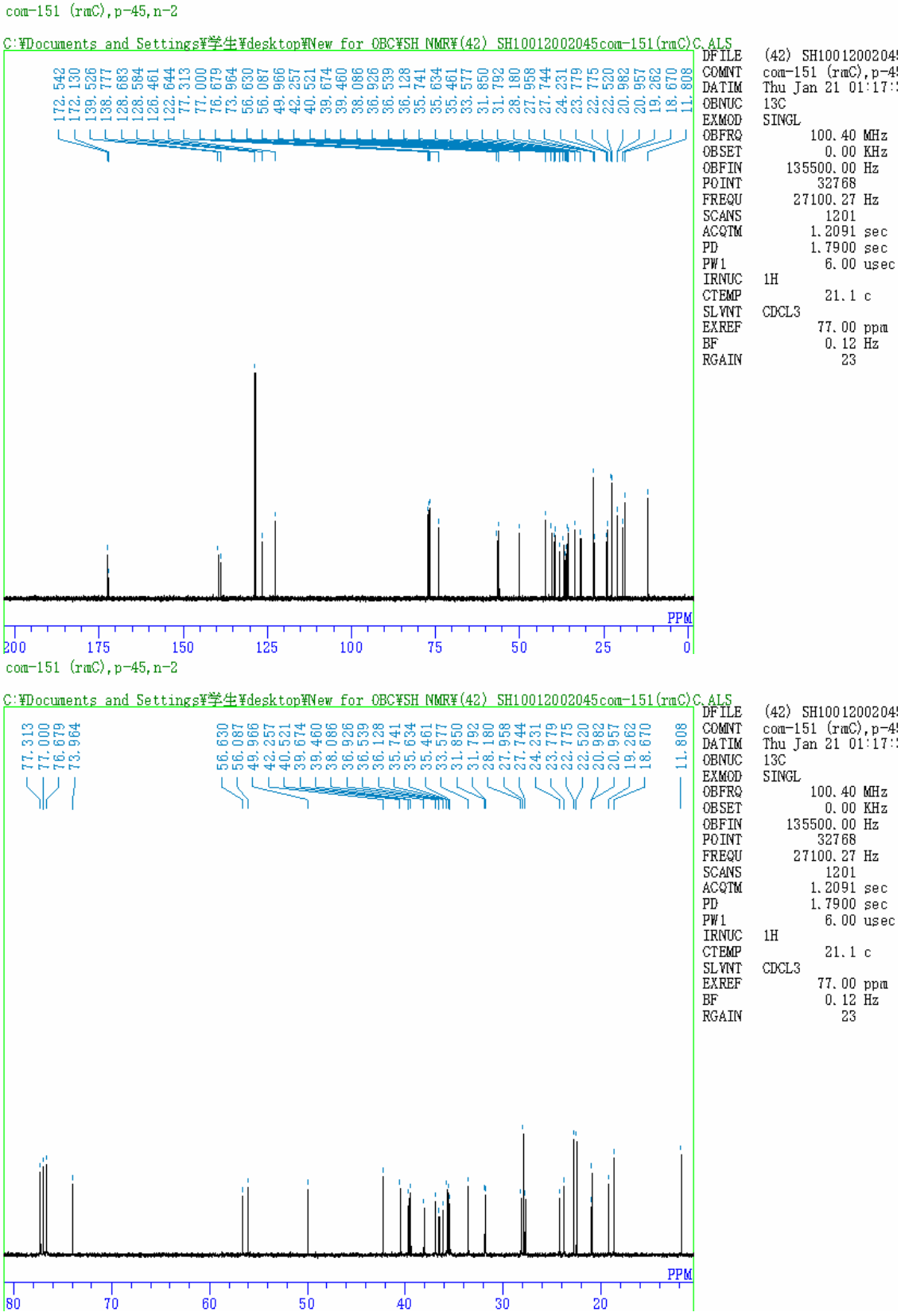
(^{1}H –NMR and ^{13}C–NMR)

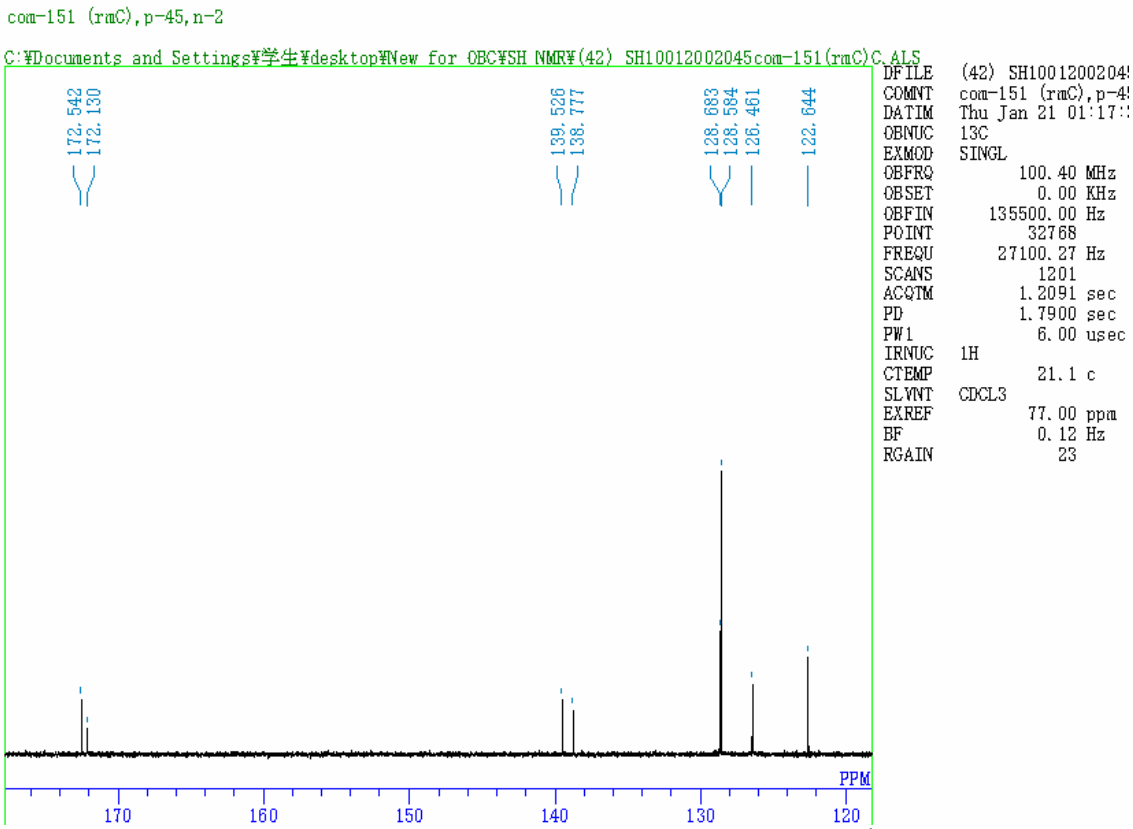
3β-cholesteryl 4-(2-phenylethylaminocarbonyl)butyrate (42) (^{1}H-NMR)



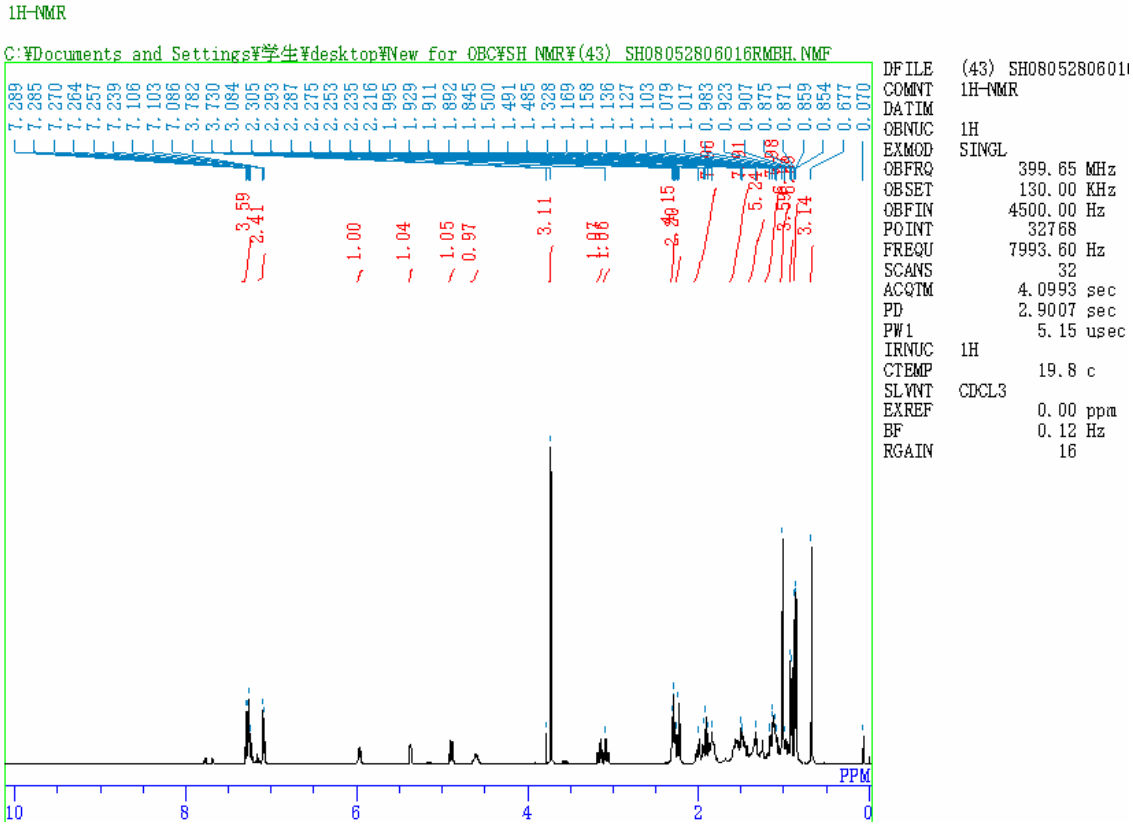

com-151 (rmC),p-45,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(42) SH10012002045com-151(rmC)H.ALS
DFILE (42) SH1001200204
COMNT com-151 (rmC),p-4
DATIM Thu Jan 21 00:15:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13
PPM
3.5
3.0
2.5
2.0
1.5
1.0
com-151 (rmC),p-45,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(42) SH10012002045com-151(rmC)H.ALS
DFILE (42) SH1001200204
COMNT com-151 (rmC),p-4
DATIM Thu Jan 21 00:15:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13
PPM
7.5
7.0
6.5
6.0
5.5
5.0
4.5

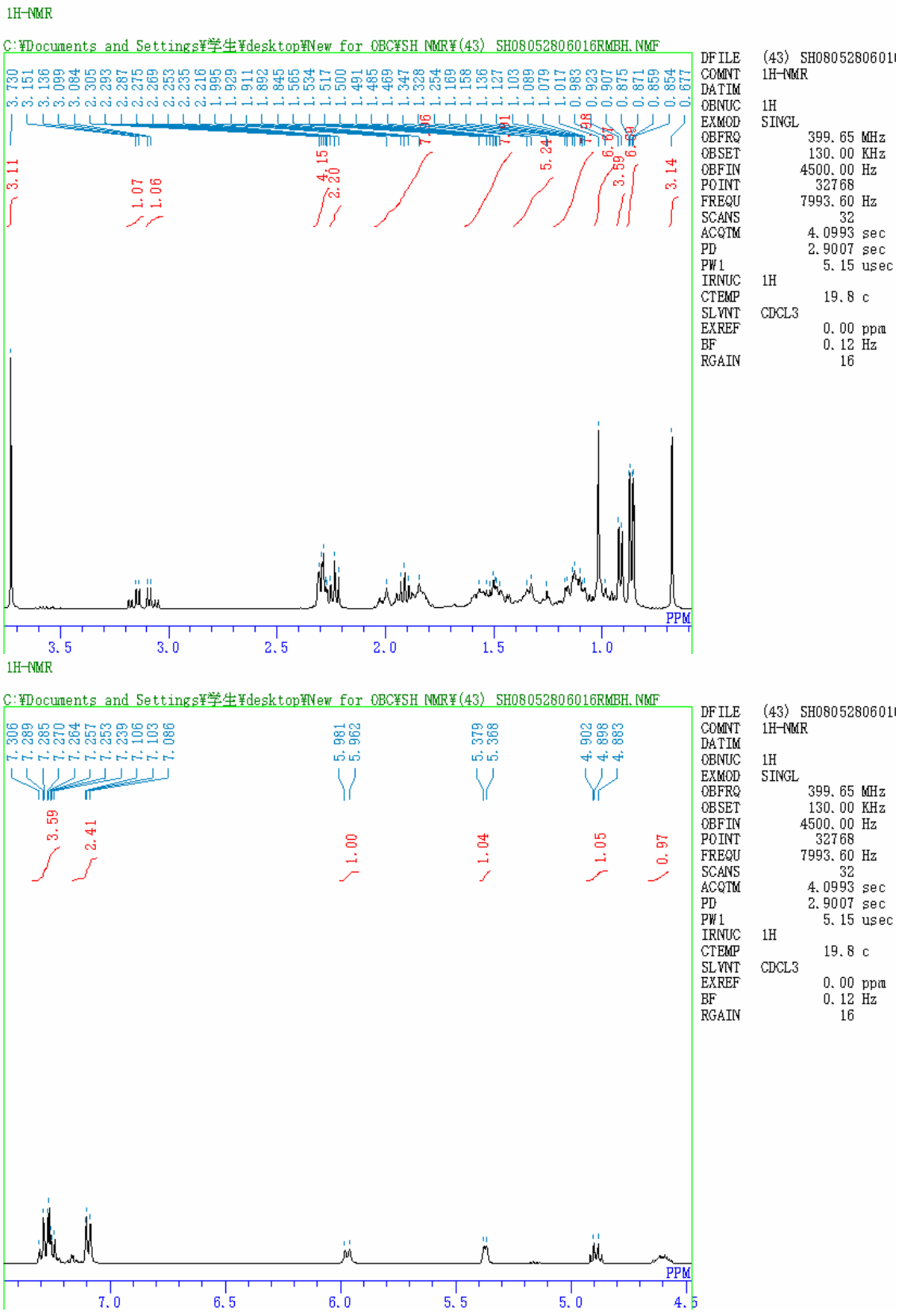
3β-cholesteryl 4-(2-phenylethylaminocarbonyl)butyrate (42) (^{13}C-NMR)



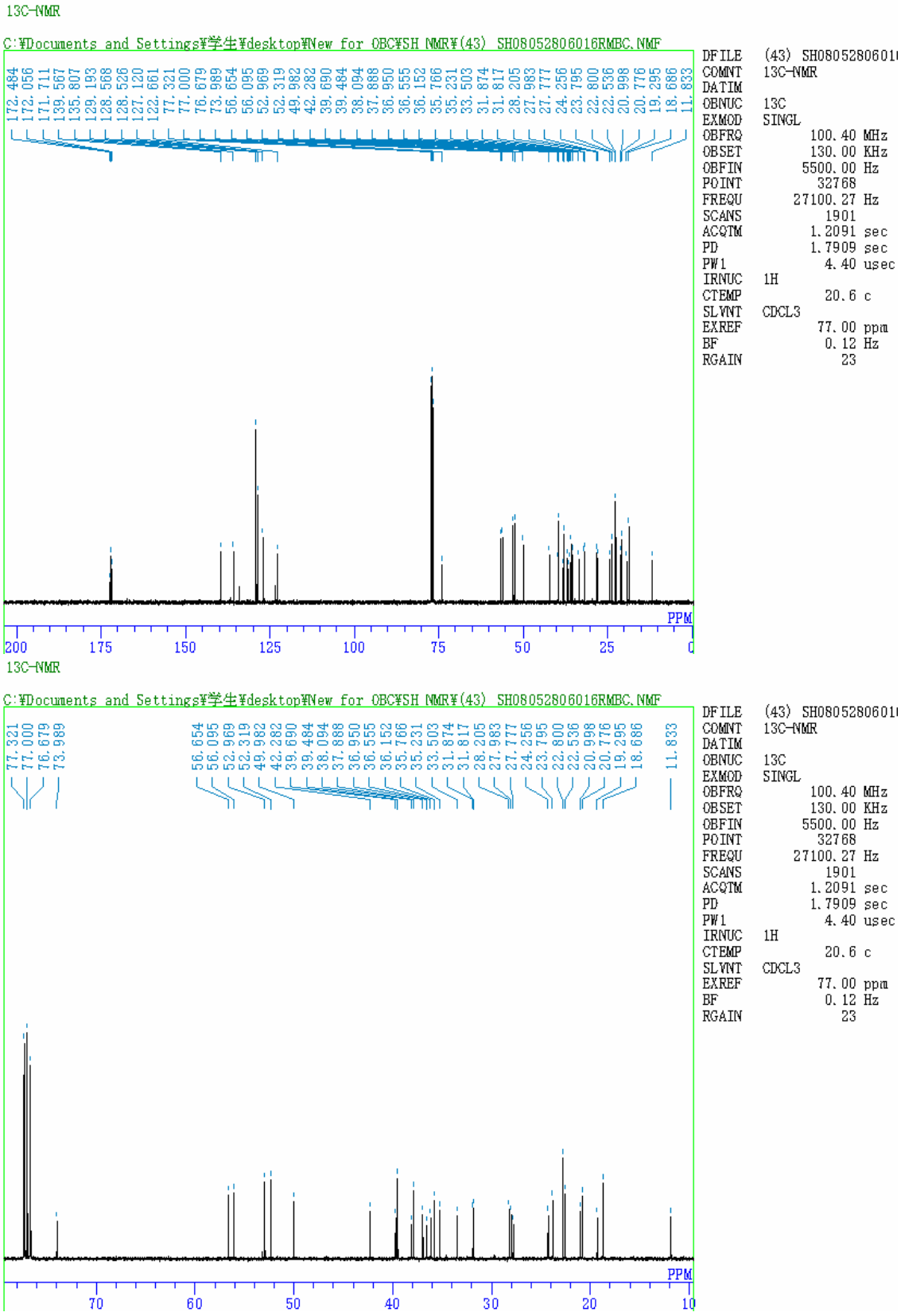


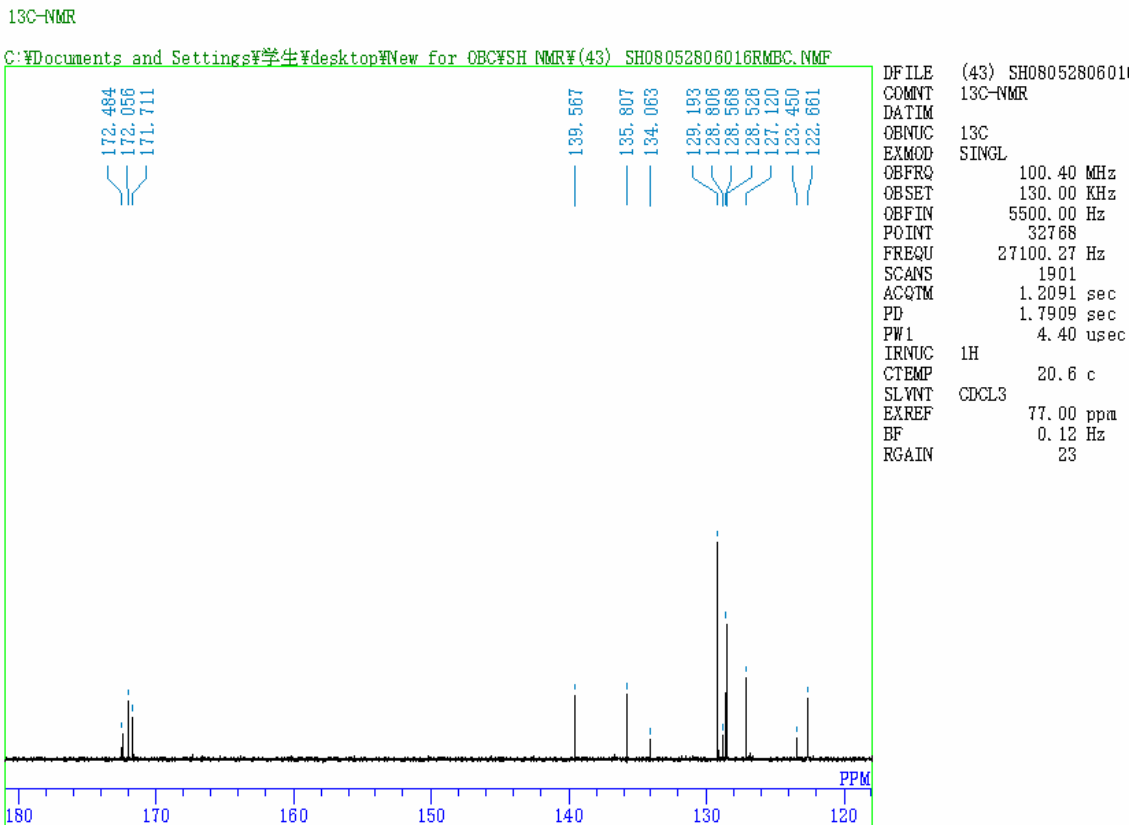
3β-cholesteryl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (43) (^{1}H-NMR)



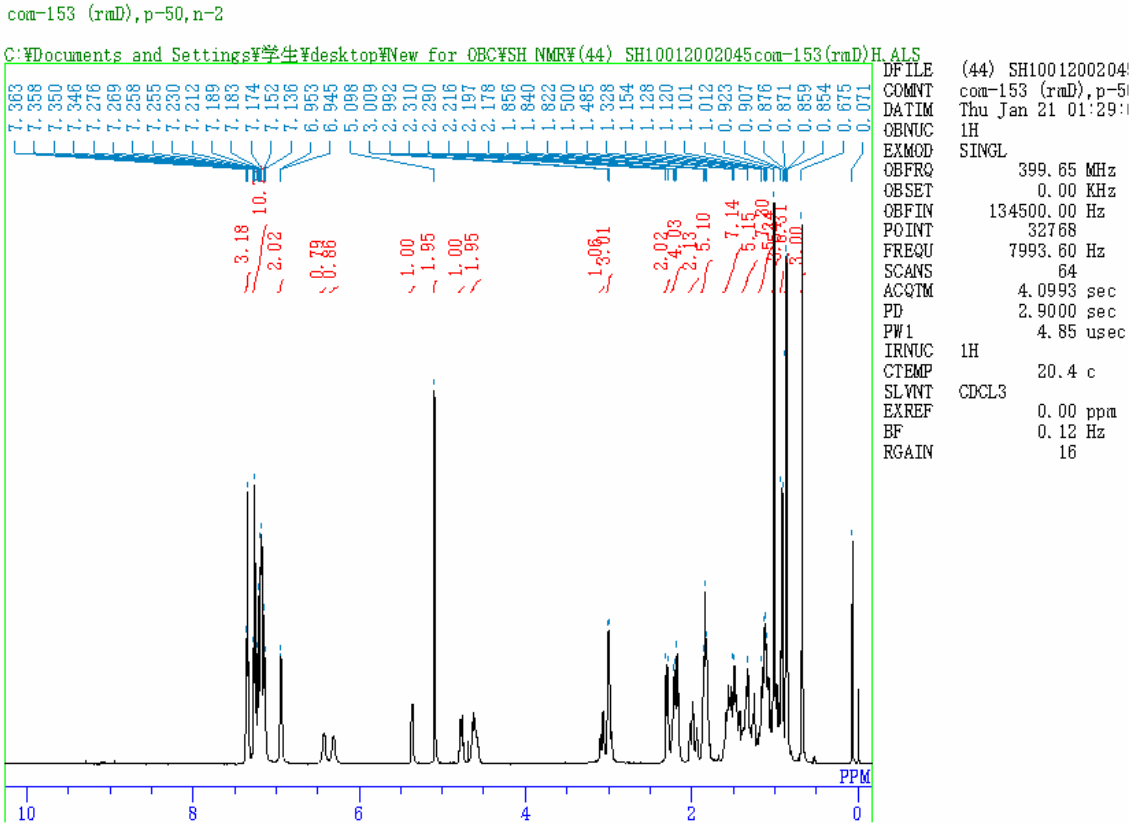

1H-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(43) SH08052806016RMBH.NMF
DFILE (43) SH0805280601
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16
PPM
1H-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(43) SH08052806016RMBH.NMF
DFILE (43) SH0805280601
COMNT 1H-NMR
DATIM
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 130.00 KHz
OBFIN 4500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 32
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16
PPM

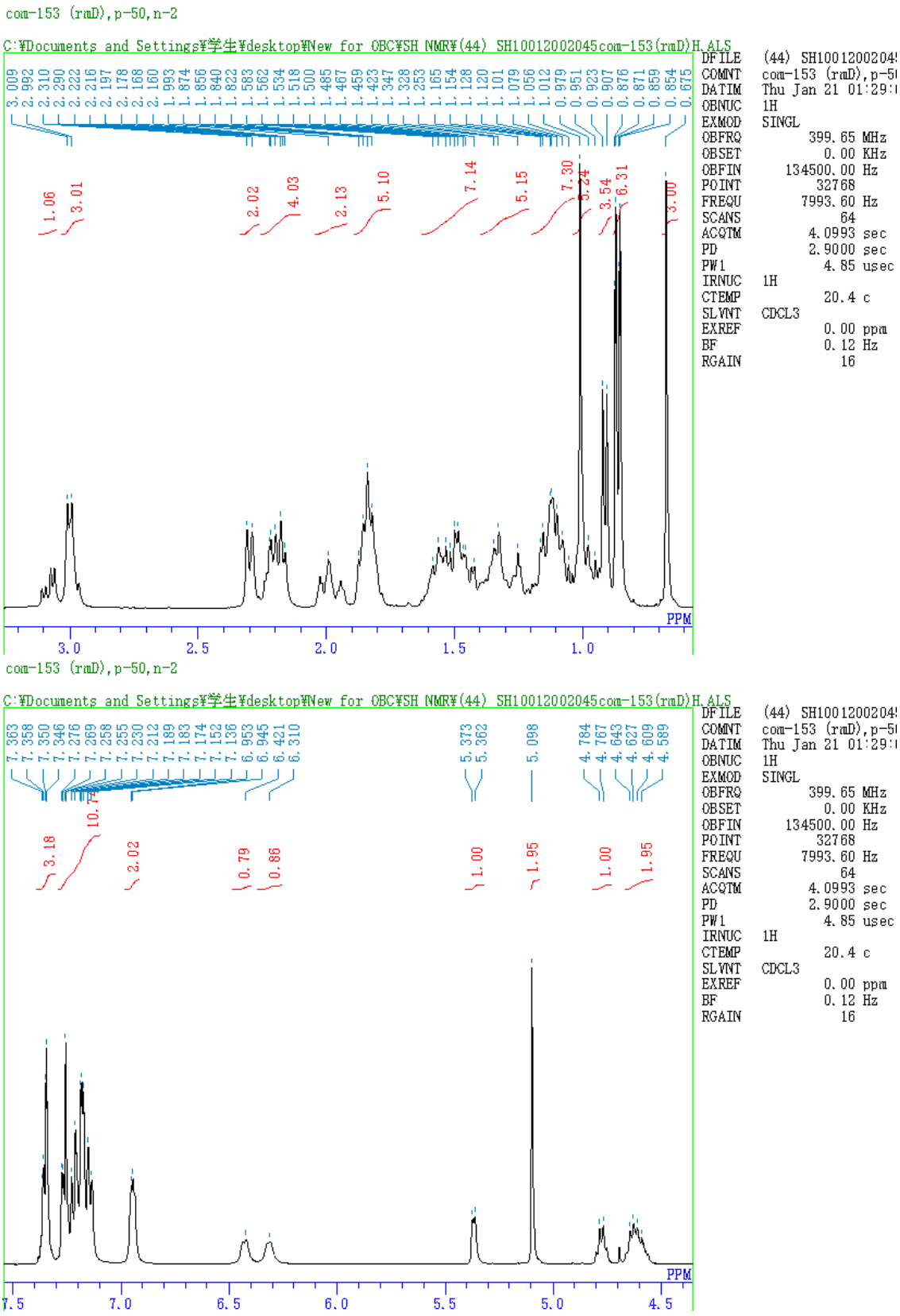
3β-cholesteryl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (43) (^{13}C-NMR)



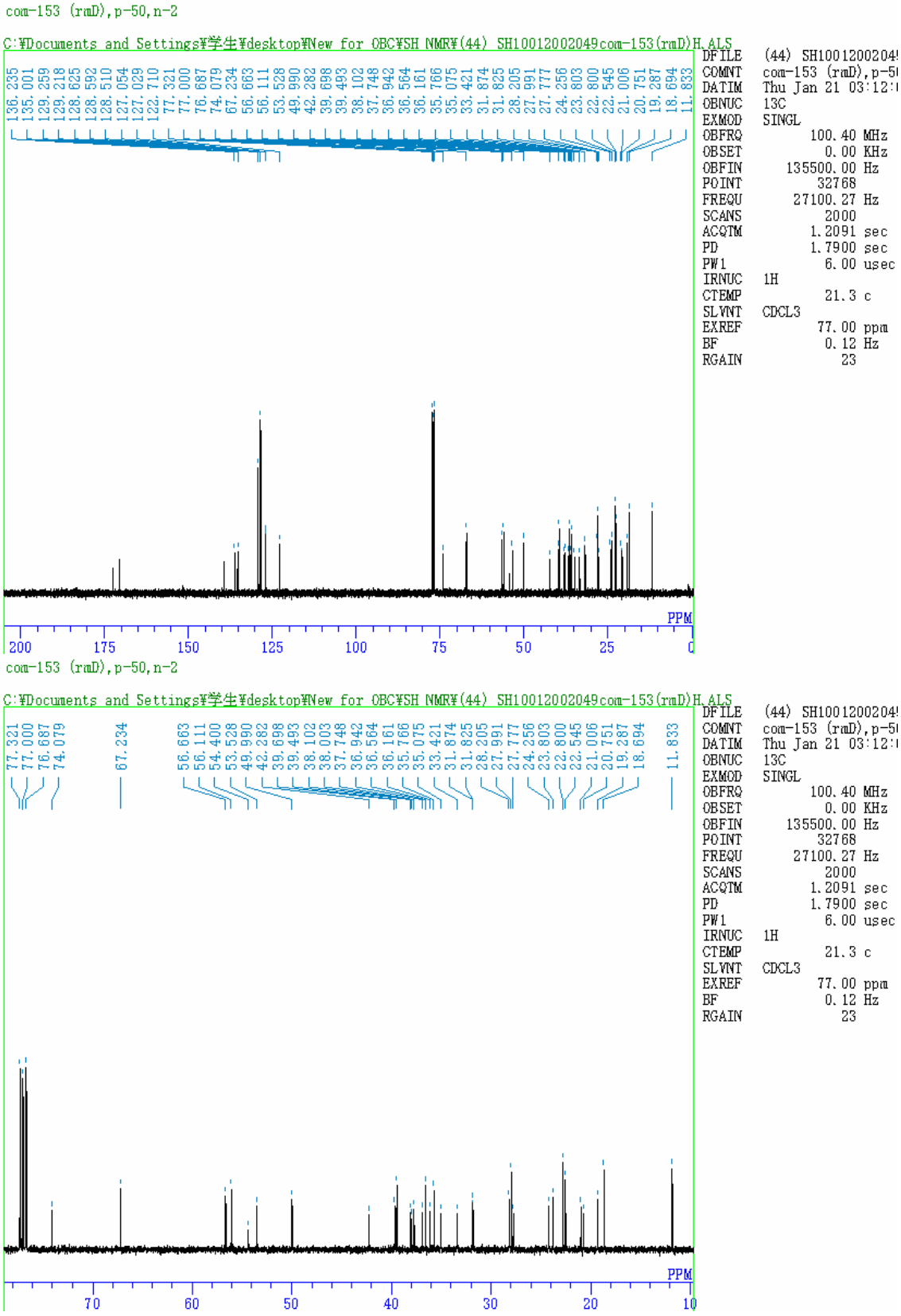


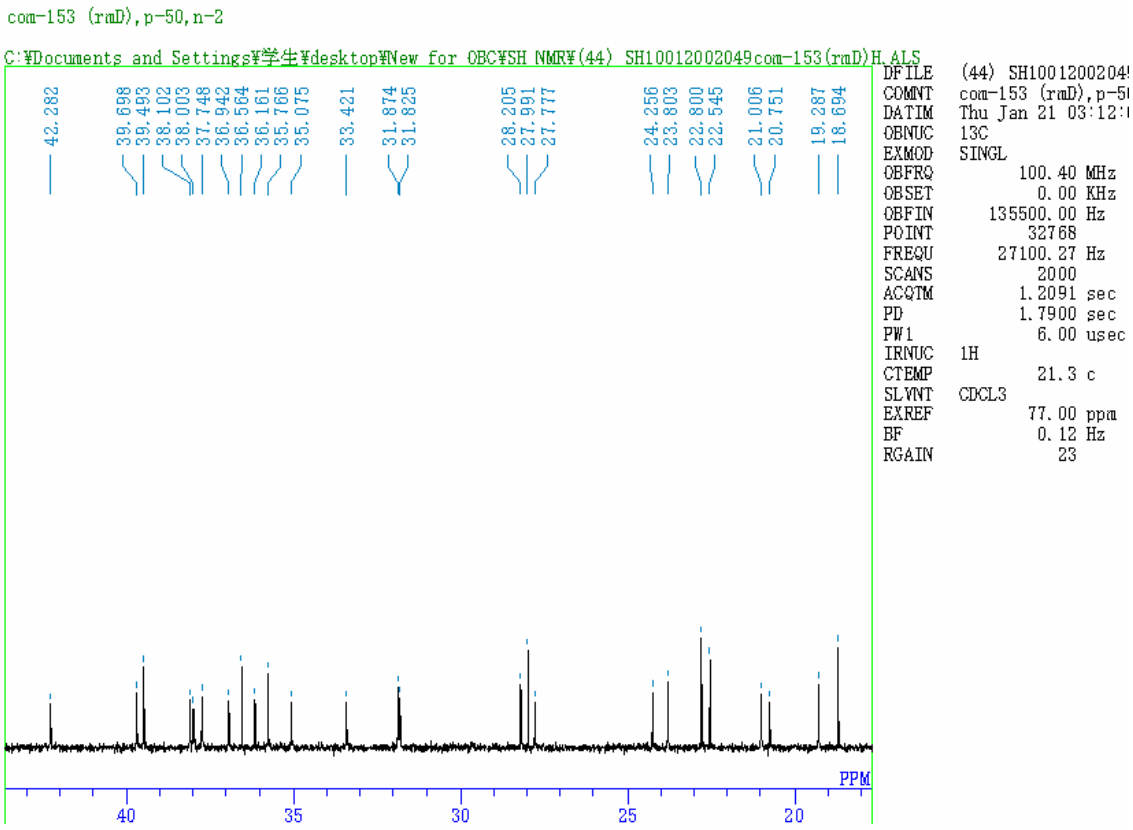
3β-cholesteryl 4-(diphenylalaninocarbonyl benzyl ester)butyrate (44) (^{1}H-NMR)

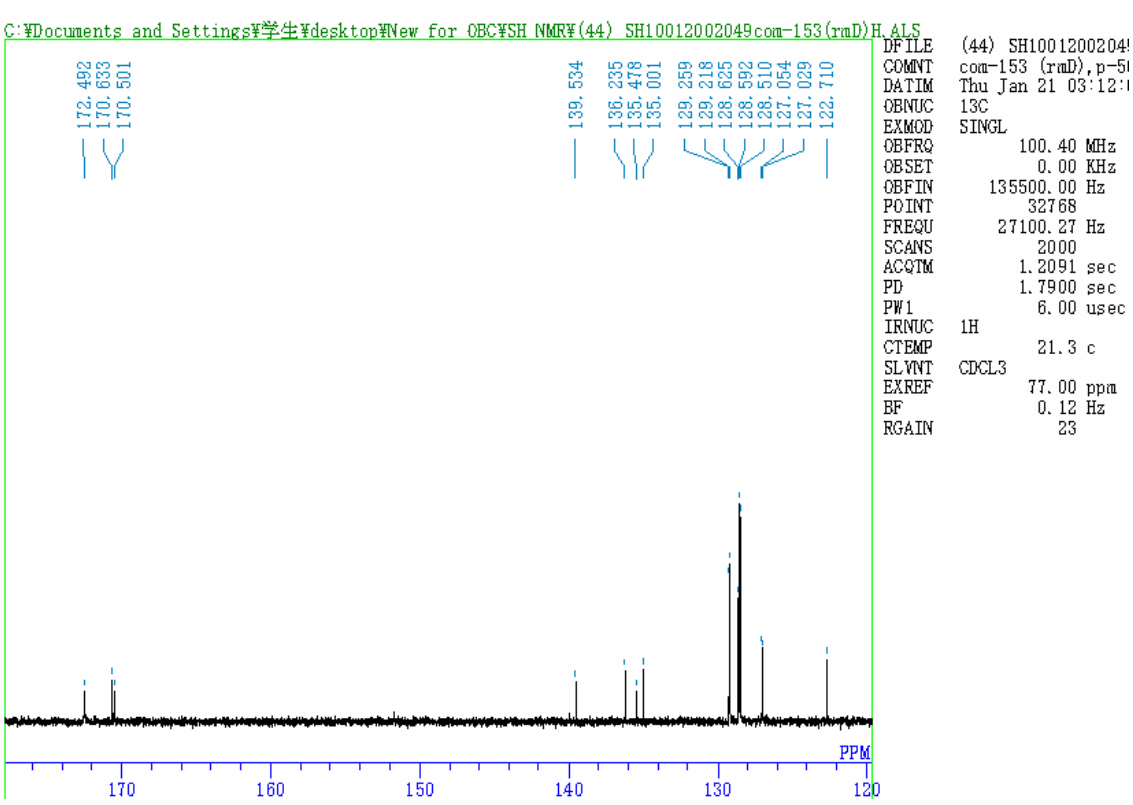



com-153 (rmD),p-50,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(44) SH10012002045com-153(rmD)H.ALS
DFILE (44) SH1001200204
COMNT com-153 (rmD),p-5
DATIM Thu Jan 21 01:29:
OBNUC 1H
EXMOD SINGL
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 64
ACQTM 4.0993 sec
PD 2.9000 sec
PW1 4.85 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16
PPM
com-153 (rmD),p-50,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(44) SH10012002045com-153(rmD)H.ALS
PPM

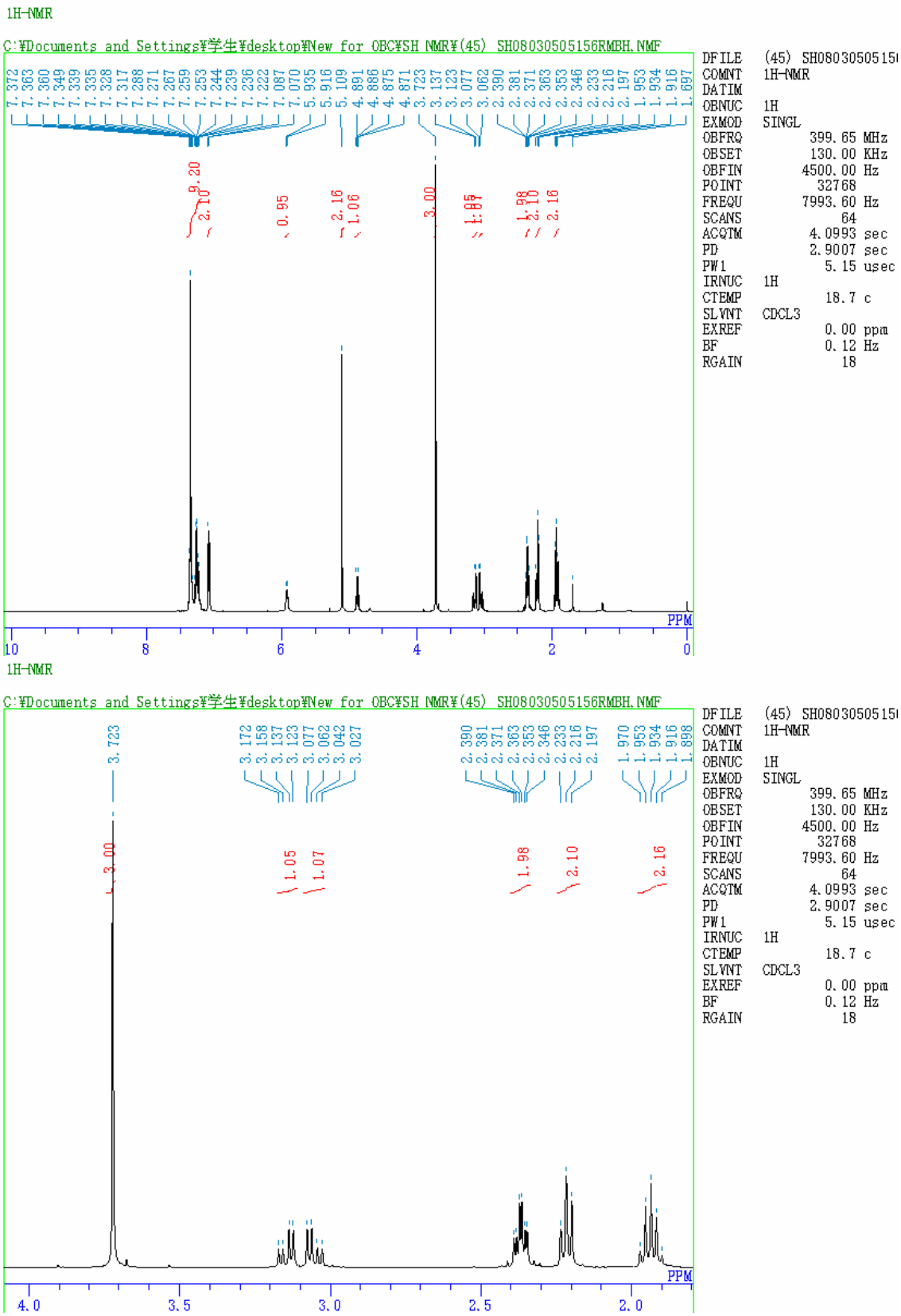
3β-cholesteryl 4-(diphenylalaninocarbonyl benzyl ester)butyrate (44) (^{13}C-NMR)

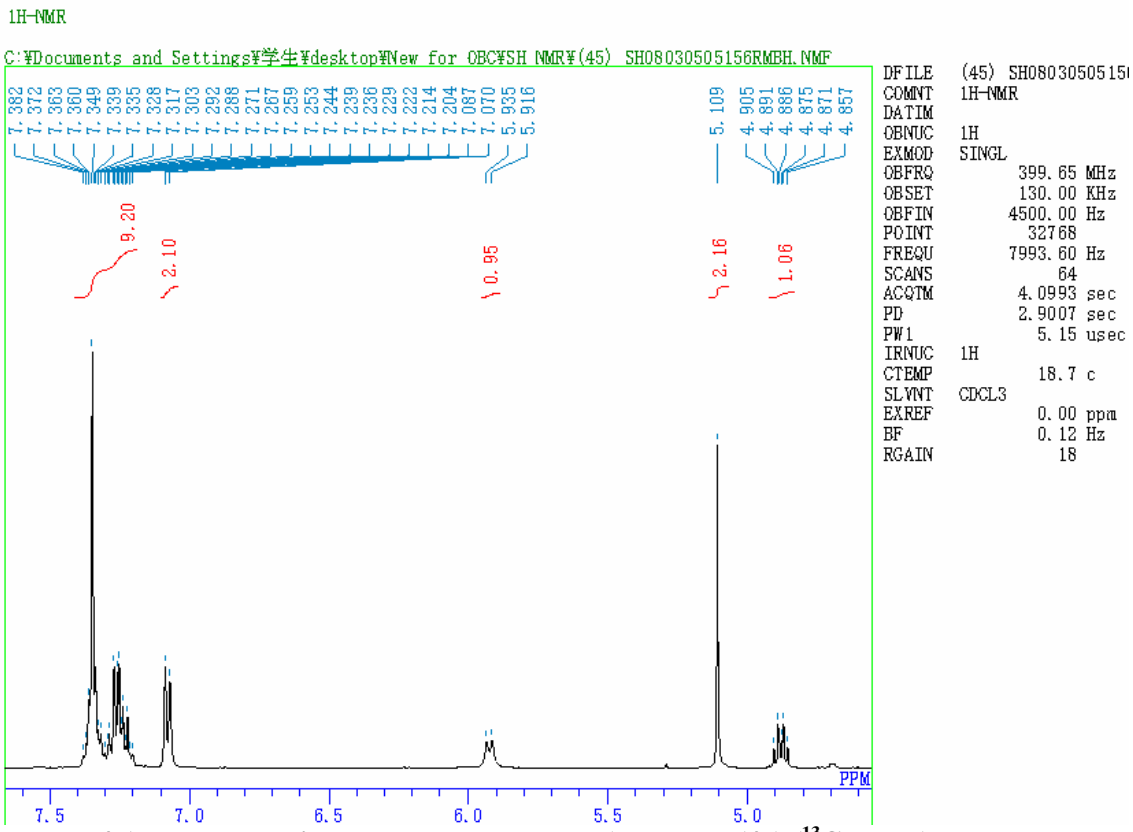



com-153 (rmD),p-50,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(44) SH10012002049com-153(rmD)H.ALS
42.282
39.698
39.493
38.102
38.003
37.748
36.942
36.564
36.161
35.766
35.075
33.421
31.874
31.825
28.205
27.991
27.777
24.256
23.803
22.800
22.545
21.006
20.751
19.287
18.694
PPM
40
35
30
25
20
DFILE (44) SH1001200204
COMNT com-153 (rmD),p-5
DATIM Thu Jan 21 03:12:
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 2000
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

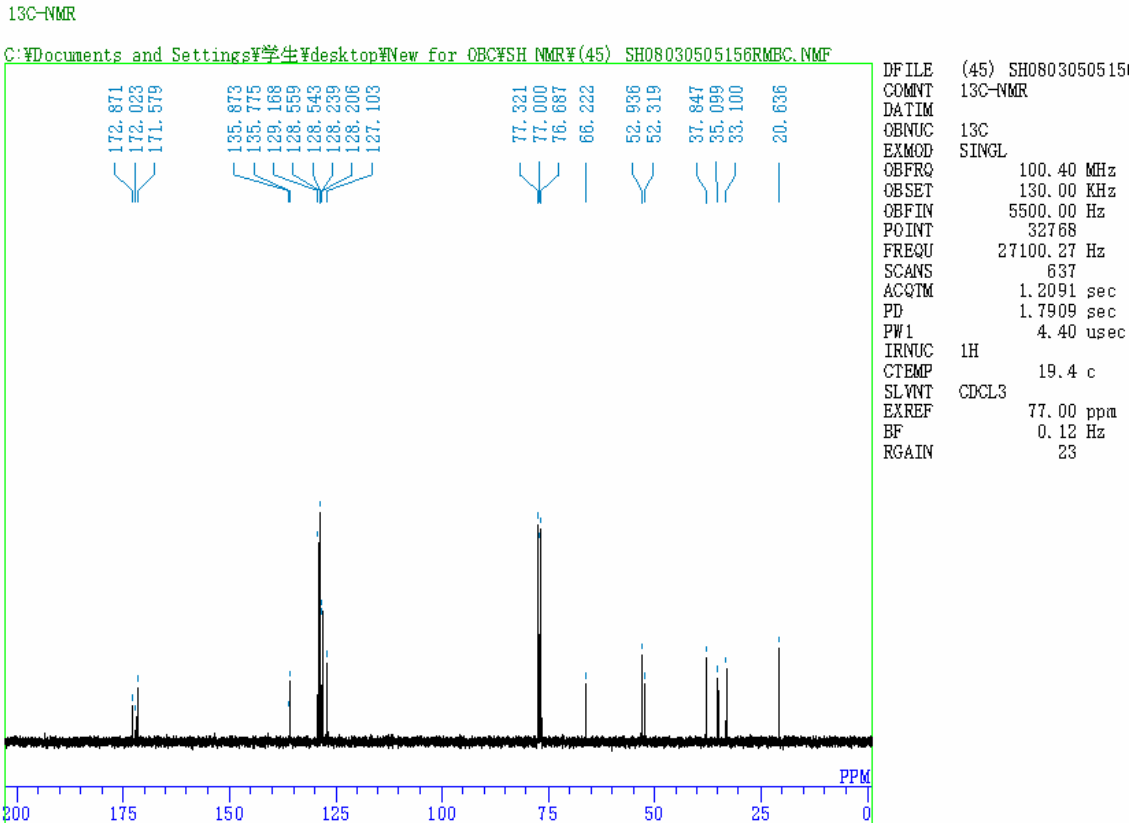

com-153 (rmD),p-50,n-2
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(44) SH10012002049com-153(rmD)H.ALS
172.492
170.633
170.501
139.534
136.235
135.478
135.001
129.259
129.218
128.625
128.592
128.510
127.054
127.029
122.710
PPM
170
160
150
140
130
120
DFILE (44) SH1001200204
COMNT com-153 (rmD),p-5
DATIM Thu Jan 21 03:12:
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 2000
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

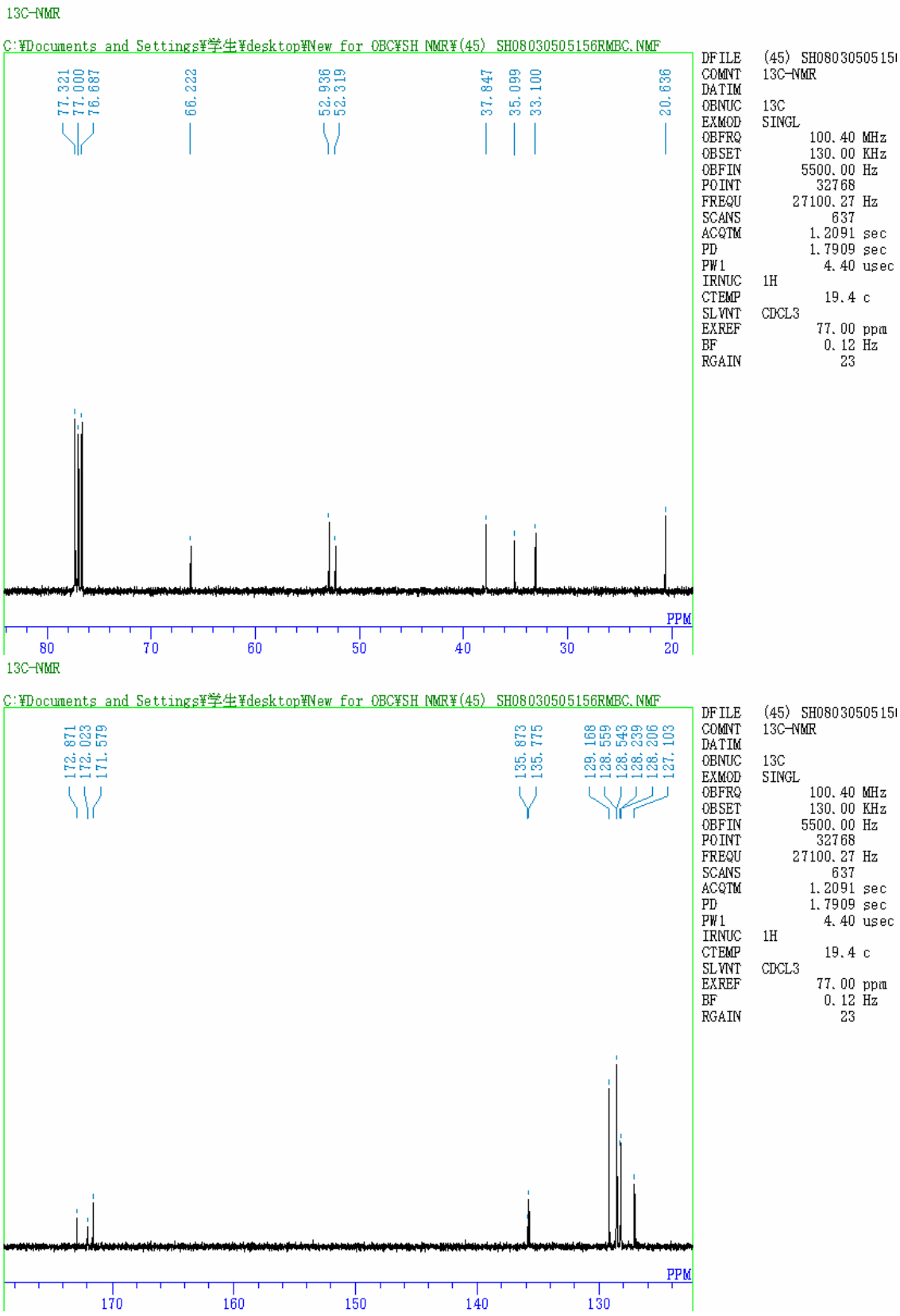
O-benzyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (45) (^{1}H-NMR)



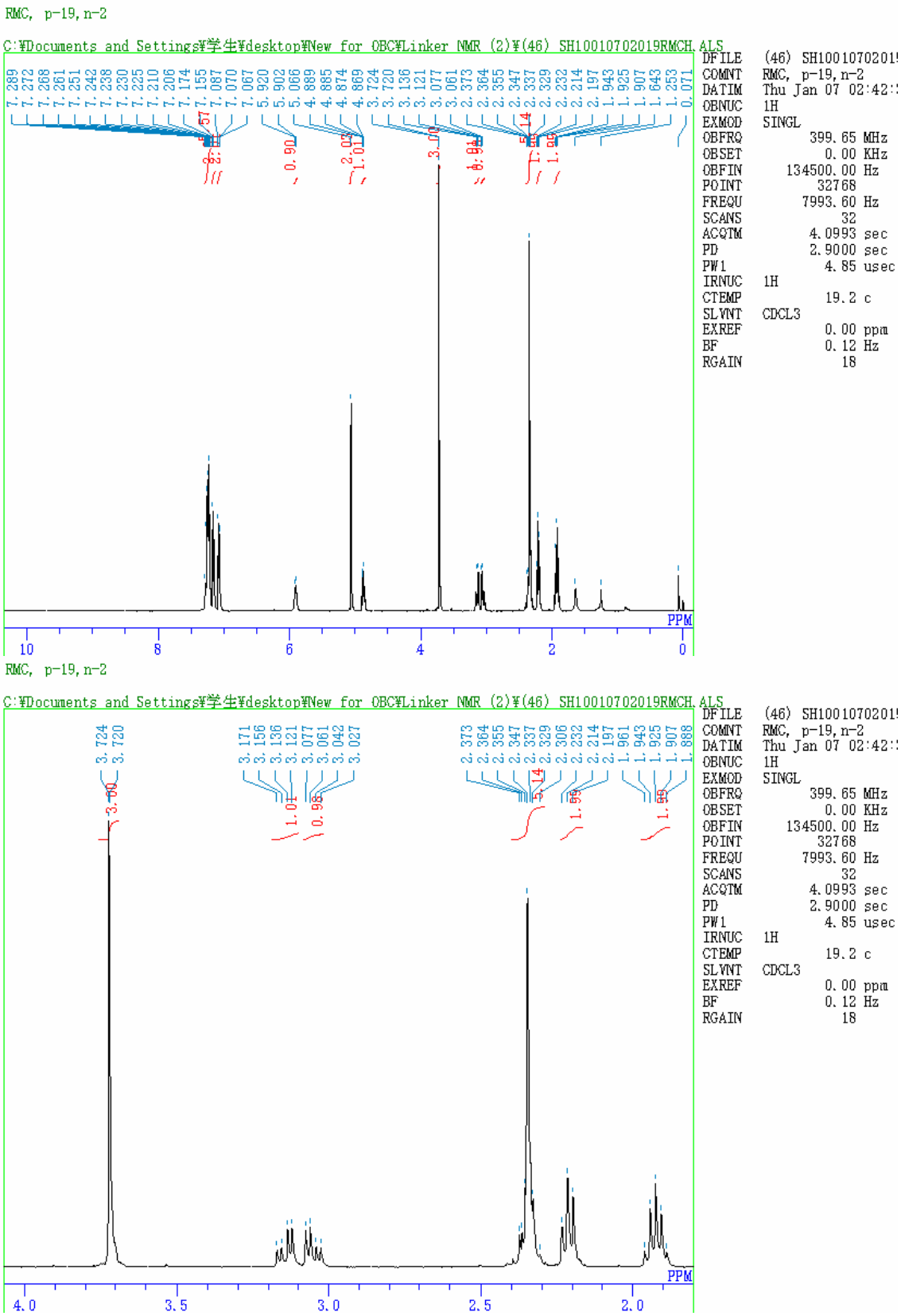


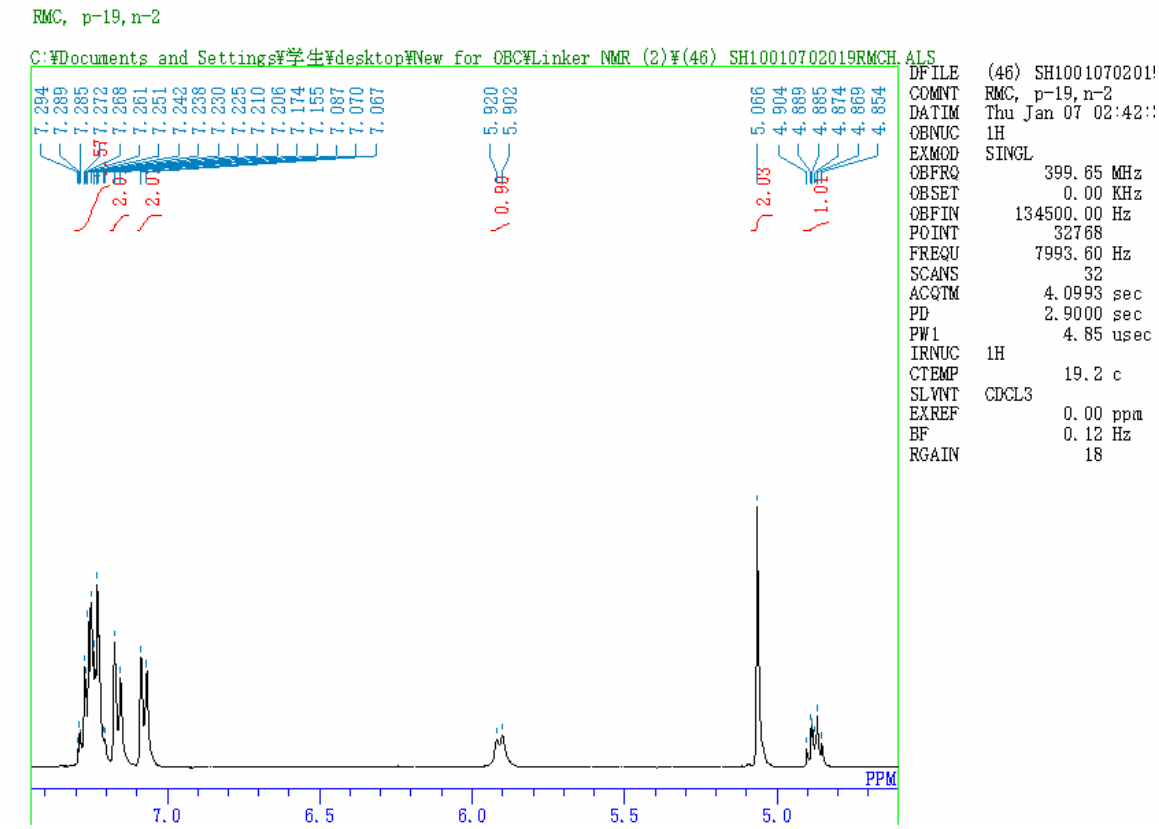
***O*-benzyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (45) (^{13}C-NMR)**



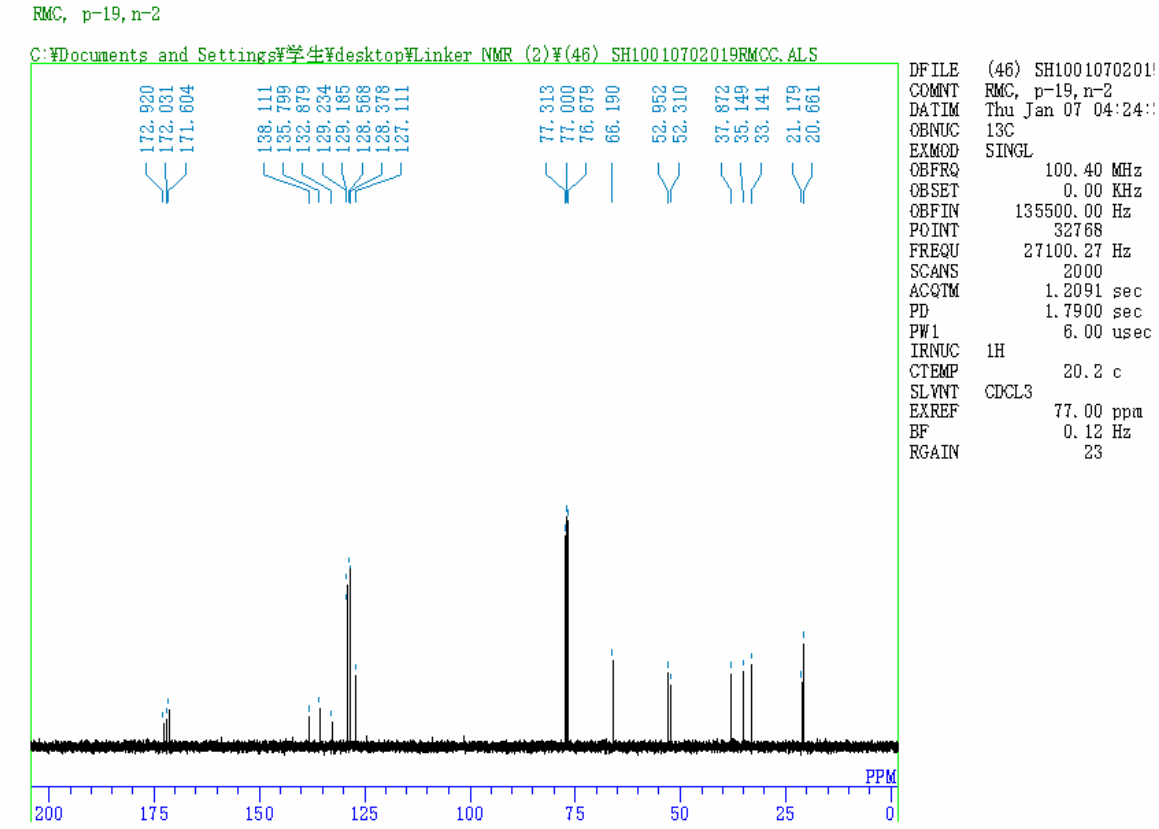

13C-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(45) SH08030505156RMBC.NMF
77.321
77.000
76.687
66.222
52.936
52.319
37.847
35.099
33.100
20.636
PPM
80
70
60
50
40
30
20
DFILE (45) SH08030505151
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 637
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.40 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
13C-NMR
C:¥Documents and Settings¥学生¥desktop¥New for OBC¥SH NMR¥(45) SH08030505156RMBC.NMF
172.871
172.023
171.579
135.873
135.775
129.168
128.559
128.543
128.239
128.206
127.103
PPM
170
160
150
140
130
DFILE (45) SH08030505151
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 637
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.40 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

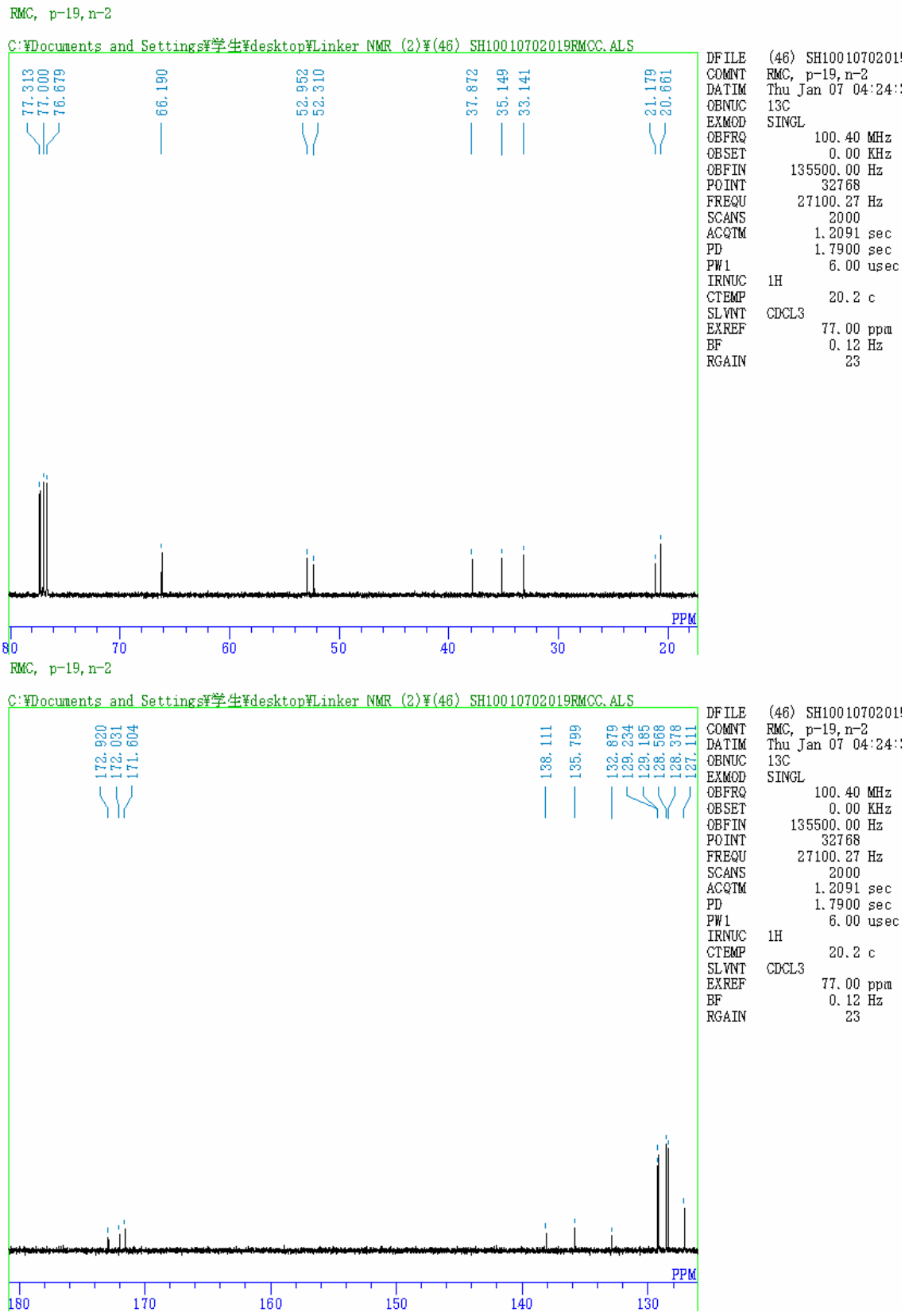
O-4-methlbenzyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (46) (^{1}H-NMR)



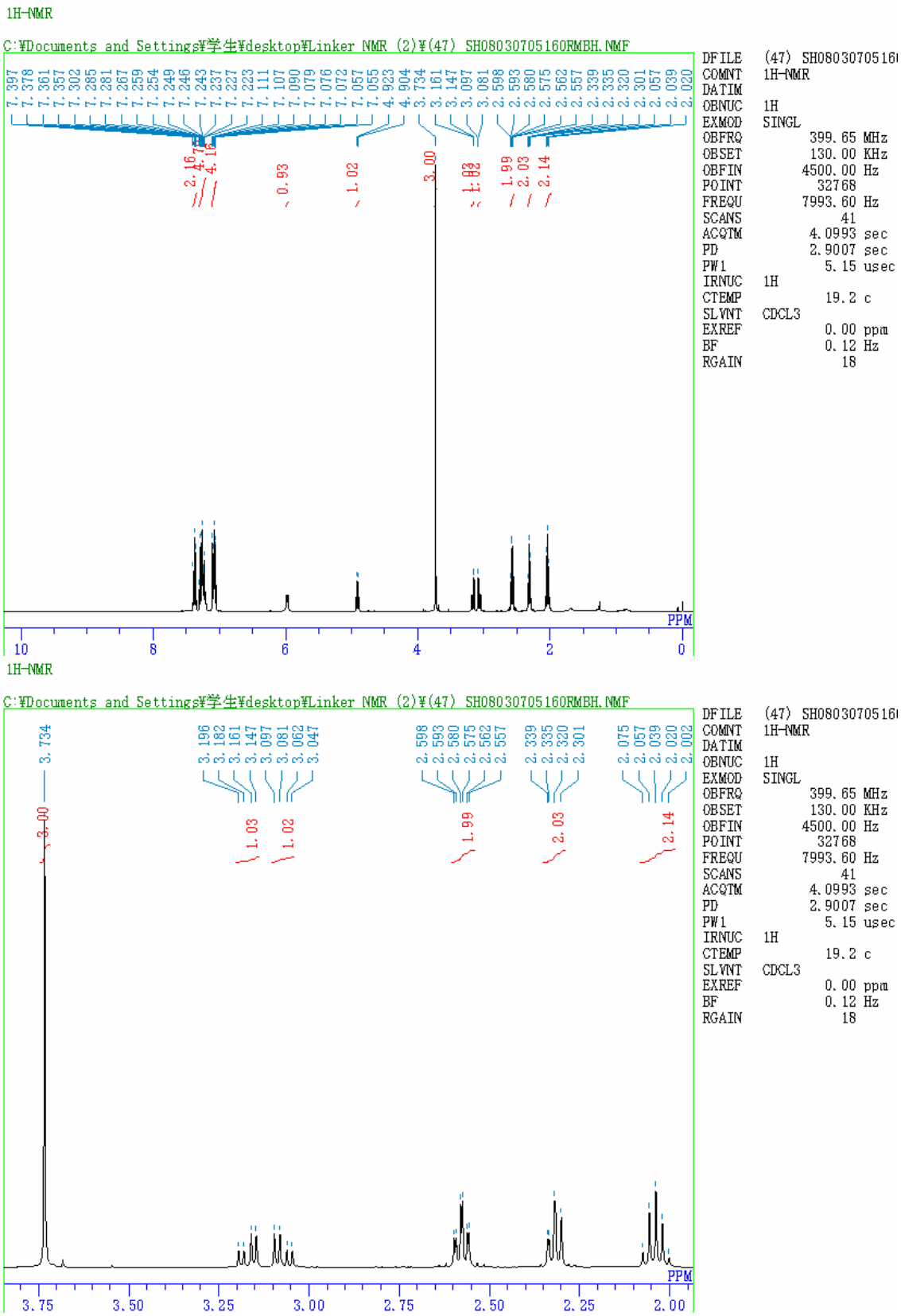


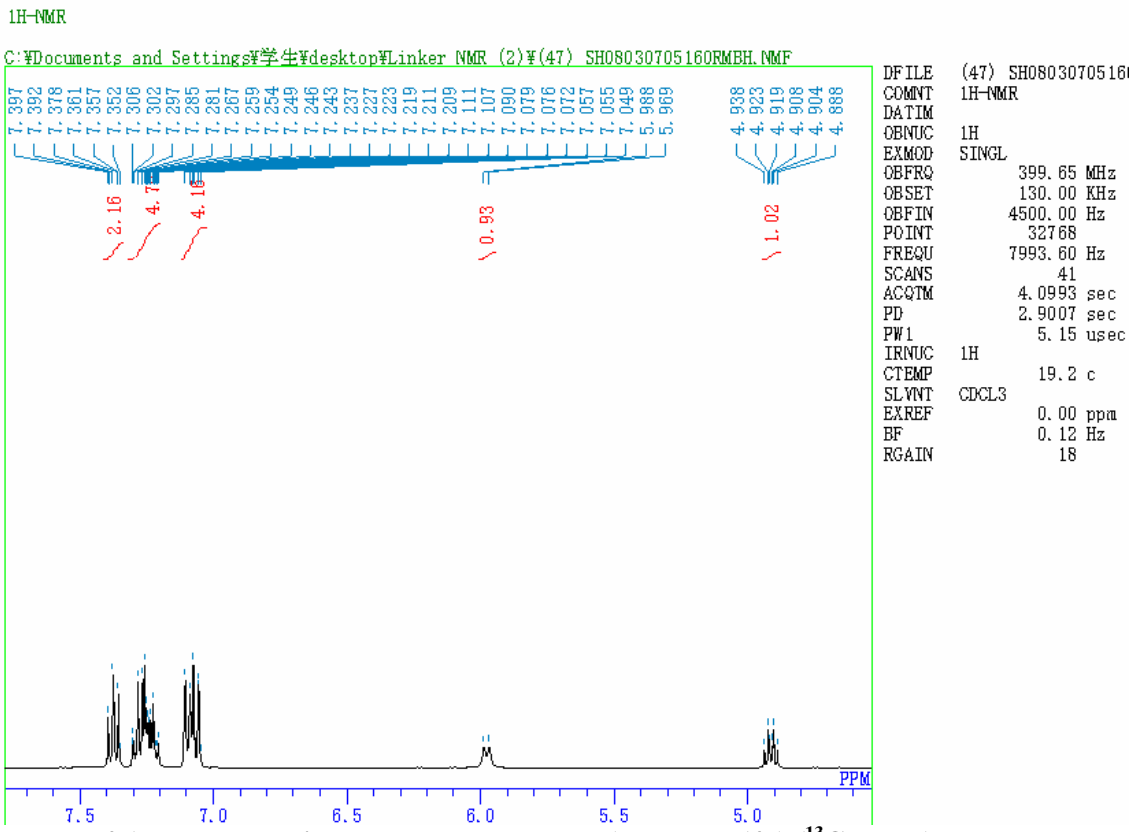
***O*-4-methlbenzyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (46) (^{13}C-NMR)**



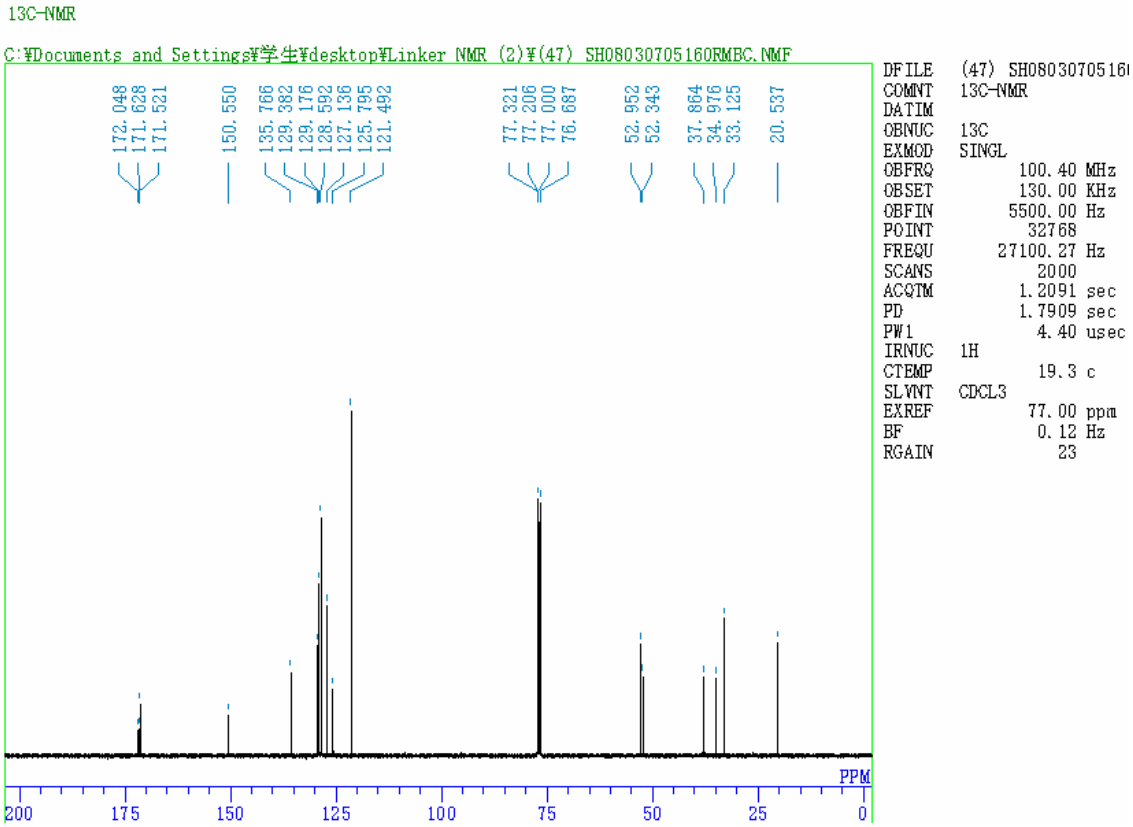

RMC, p-19,n-2
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(46) SH10010702019RMCC.ALS
77.313
77.000
76.679
66.190
52.952
52.310
37.872
35.149
33.141
21.179
20.661
PPM
80
70
60
50
40
30
20
DFILE (46) SH1001070201
COMNT RMC, p-19,n-2
DATIM Thu Jan 07 04:24:
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 2000
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
RMC, p-19,n-2
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(46) SH10010702019RMCC.ALS
172.920
172.031
171.604
138.111
135.799
132.879
129.234
129.185
128.568
128.378
127.111
PPM
180
170
160
150
140
130
DFILE (46) SH1001070201
COMNT RMC, p-19,n-2
DATIM Thu Jan 07 04:24:
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 2000
ACQTM 1.2091 sec
PD 1.7900 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

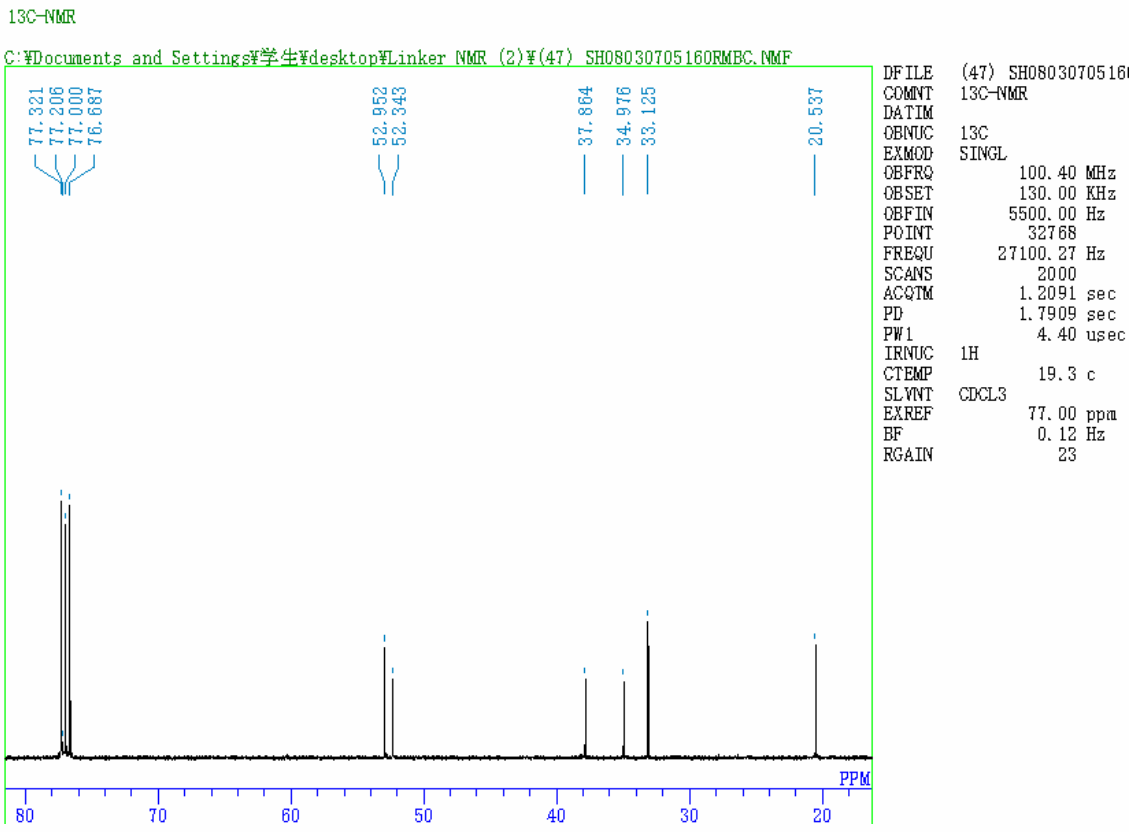
O-phenyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (47) (^{1}H-NMR)

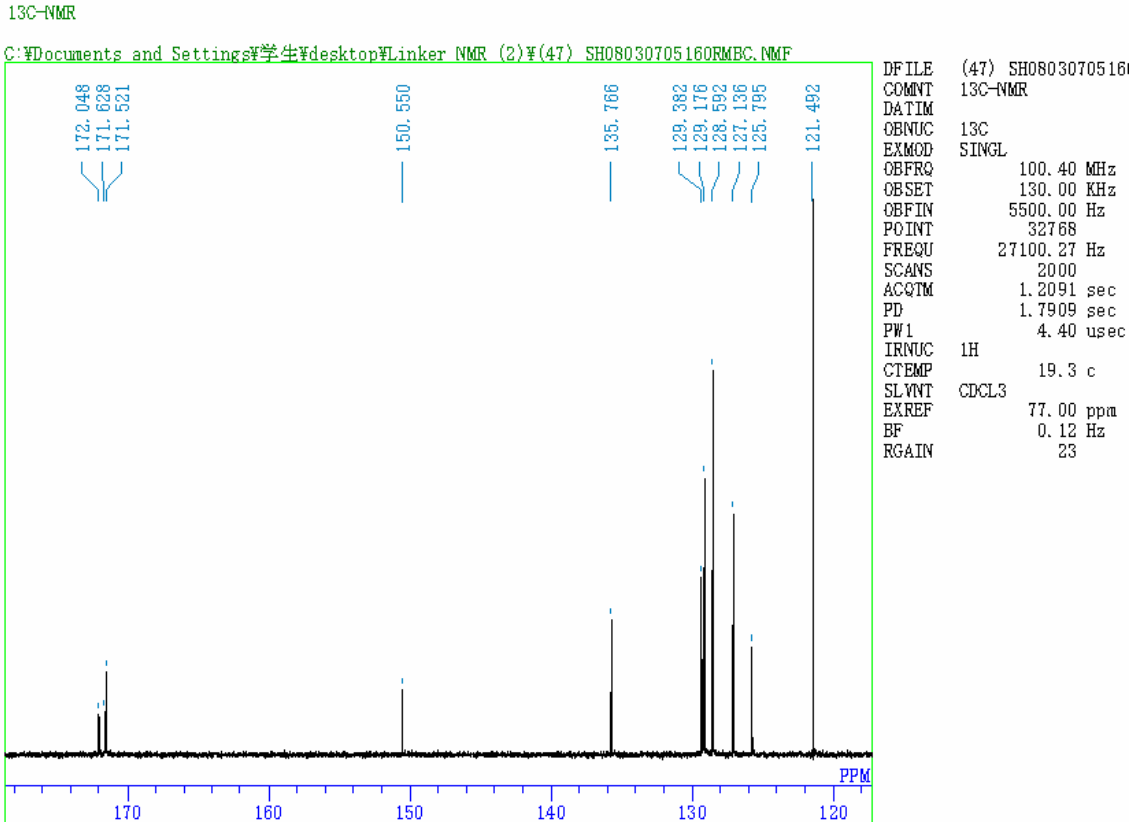




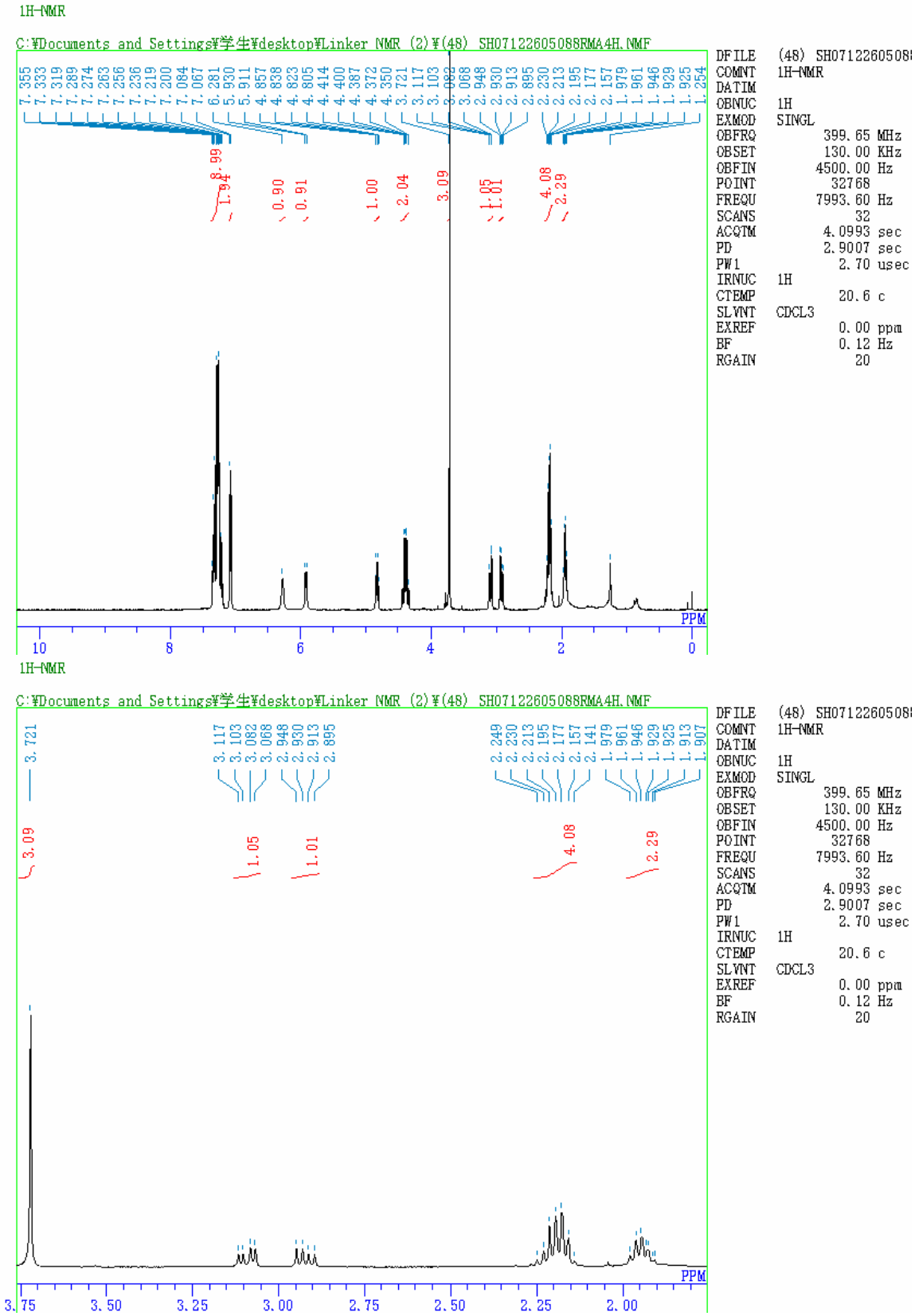
***O*-phenyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (47) (^{13}C-NMR)**

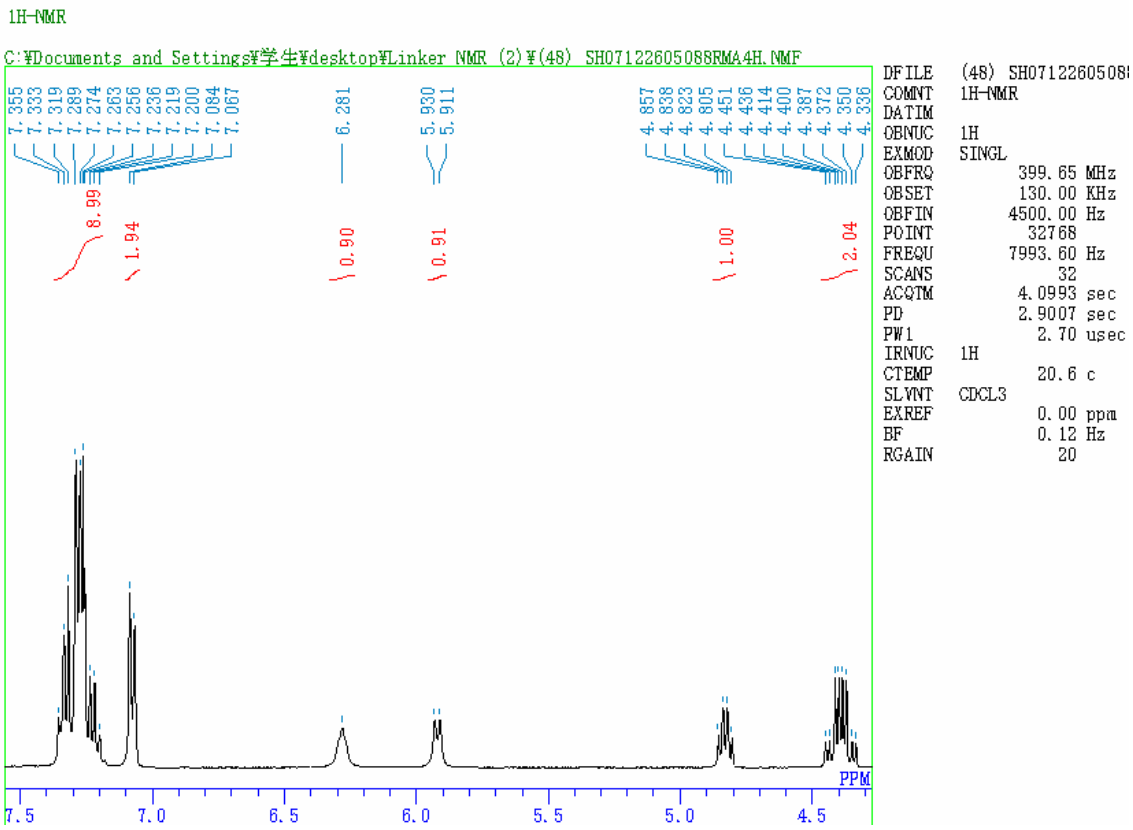



13C-NMR
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(47) SH08030705160RMBC.NMF
77.321
77.206
77.000
76.687
52.952
52.343
37.864
34.976
33.135
20.537
PPM
80
70
60
50
40
30
20
DFILE (47) SH08030705160
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 2000
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.40 usec
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

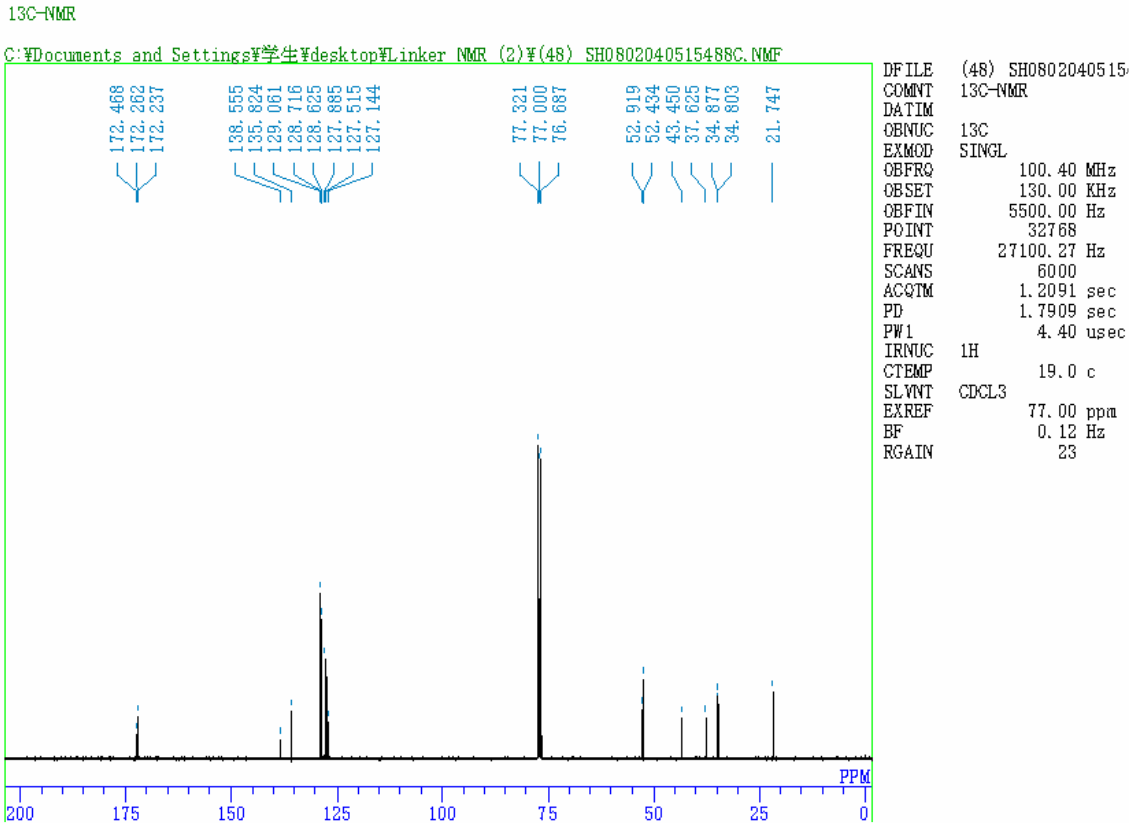

13C-NMR
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(47) SH08030705160RMBC.NMF
172.048
171.628
171.521
150.550
135.766
129.382
129.176
128.592
127.136
125.795
121.492
PPM
170
160
150
140
130
120
DFILE (47) SH08030705160
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 2000
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.40 usec
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

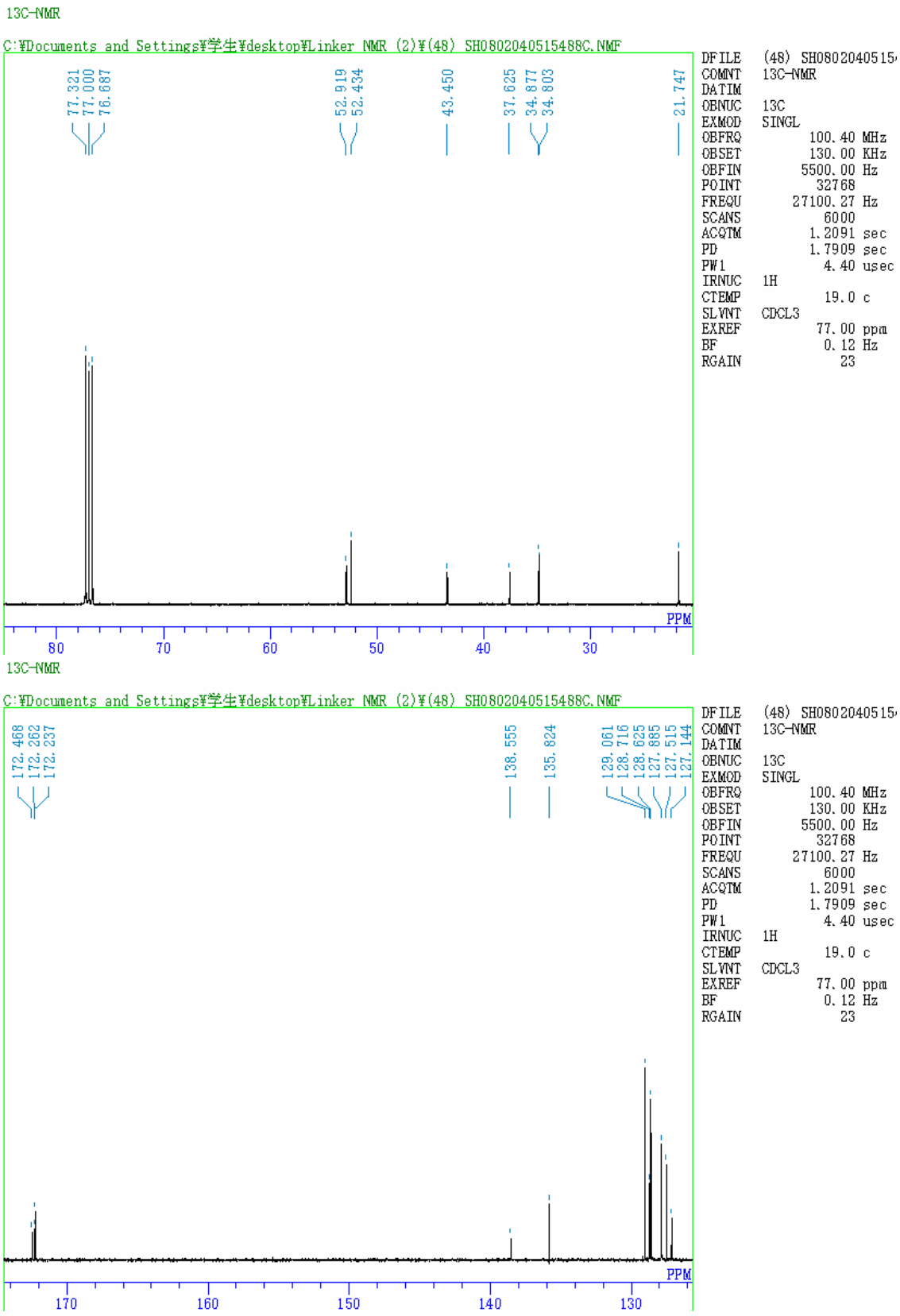
N-benzyl 4-(L-phenylalaninocarbonyl methyl ester)butanamide (48) (^{1}H-NMR)



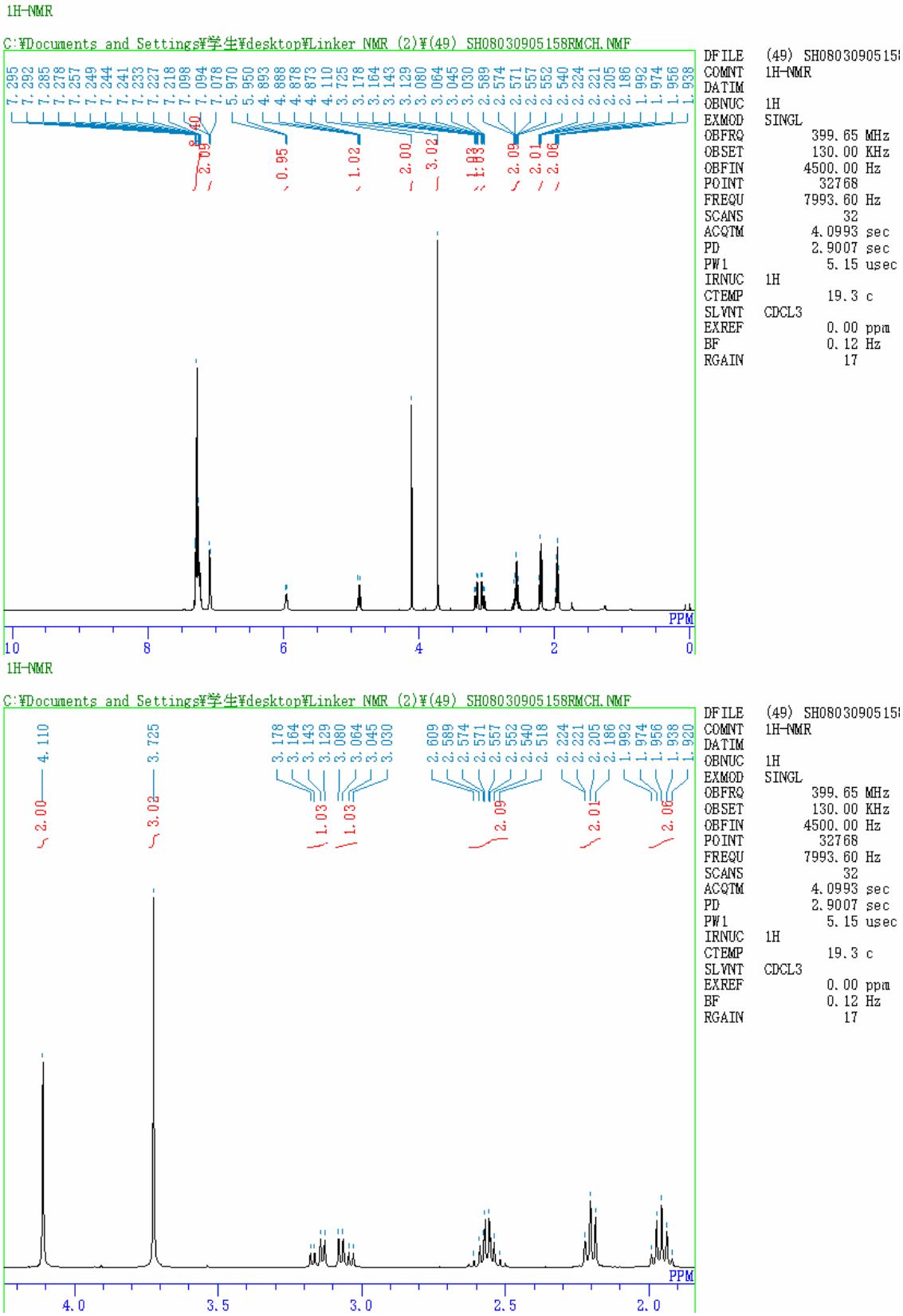


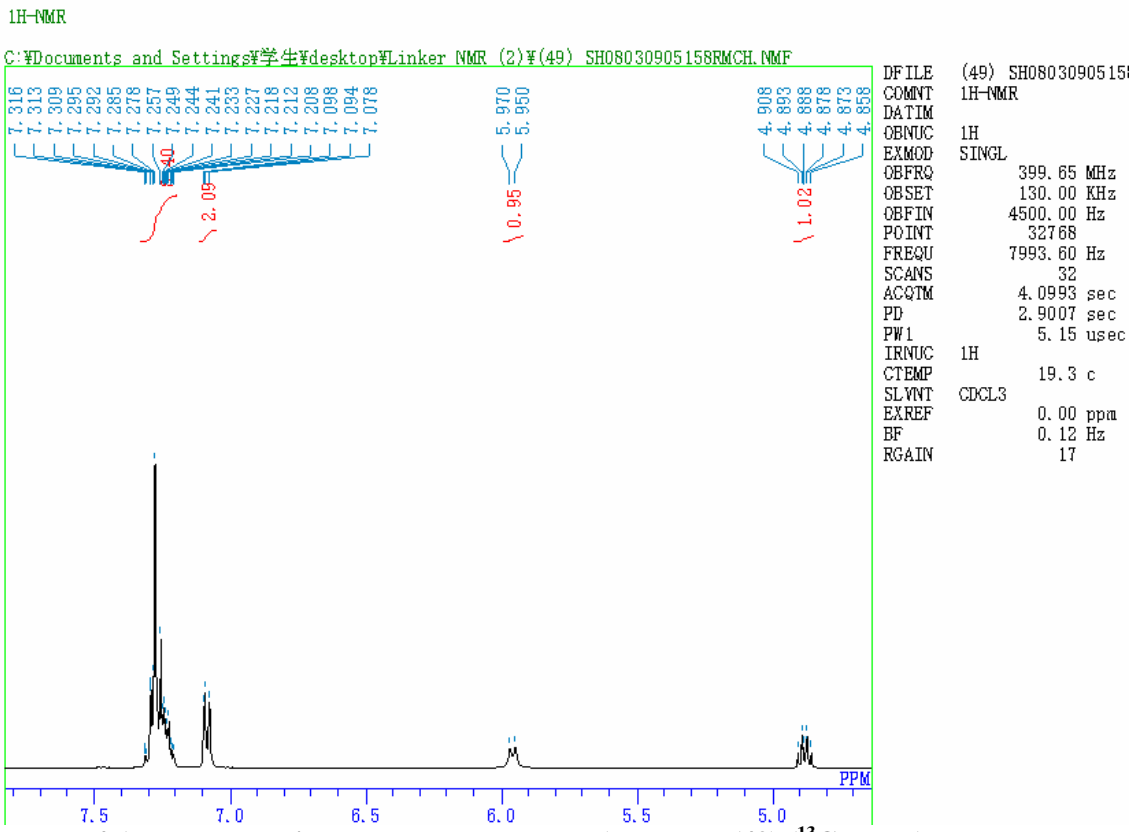
***N*-benzyl 4-(L-phenylalaninocarbonyl methyl ester)butanamide (48) (^{13}C-NMR)**



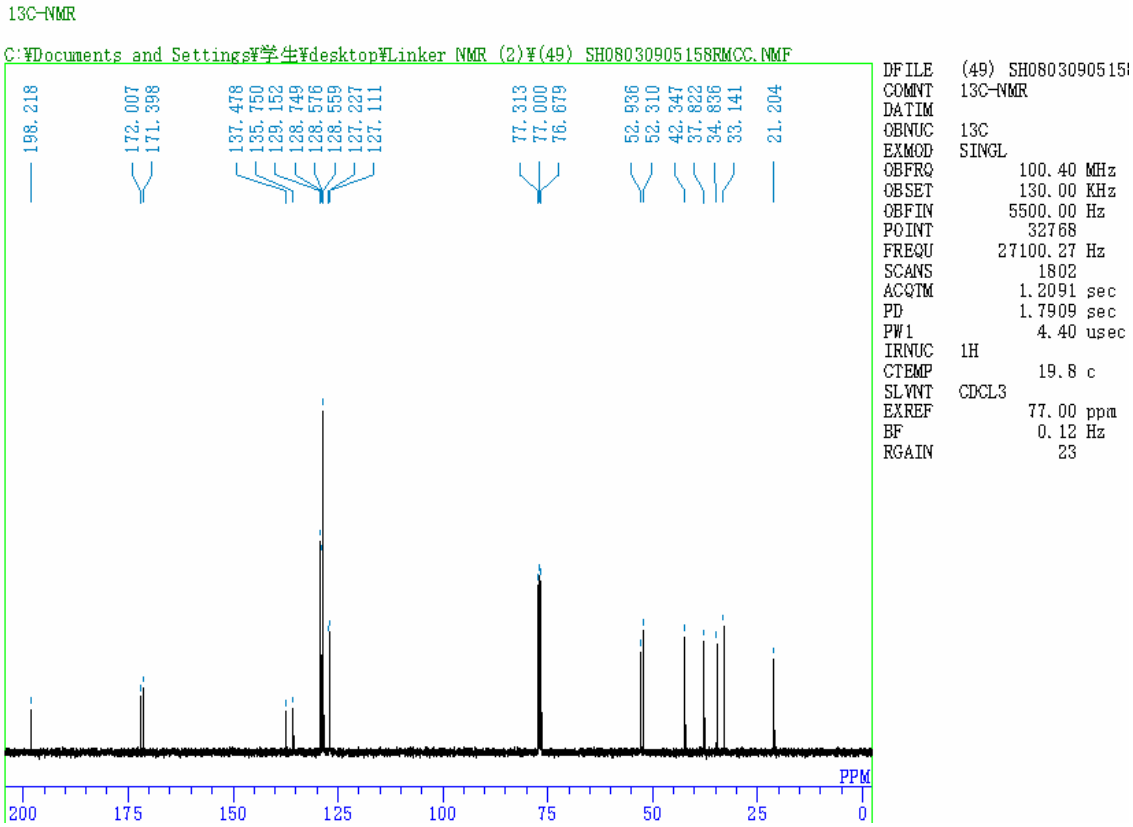

13C-NMR
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(48) SH0802040515488C.NMF
77.321
77.000
76.687
52.919
52.434
43.450
37.625
34.877
34.803
21.747
PPM
80
70
60
50
40
30
DFILE (48) SH0802040515
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 6000
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.40 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
13C-NMR
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(48) SH0802040515488C.NMF
172.468
172.262
172.237
138.555
135.824
129.061
128.716
128.625
127.885
127.515
127.144
PPM
170
160
150
140
130
DFILE (48) SH0802040515
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 6000
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.40 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23

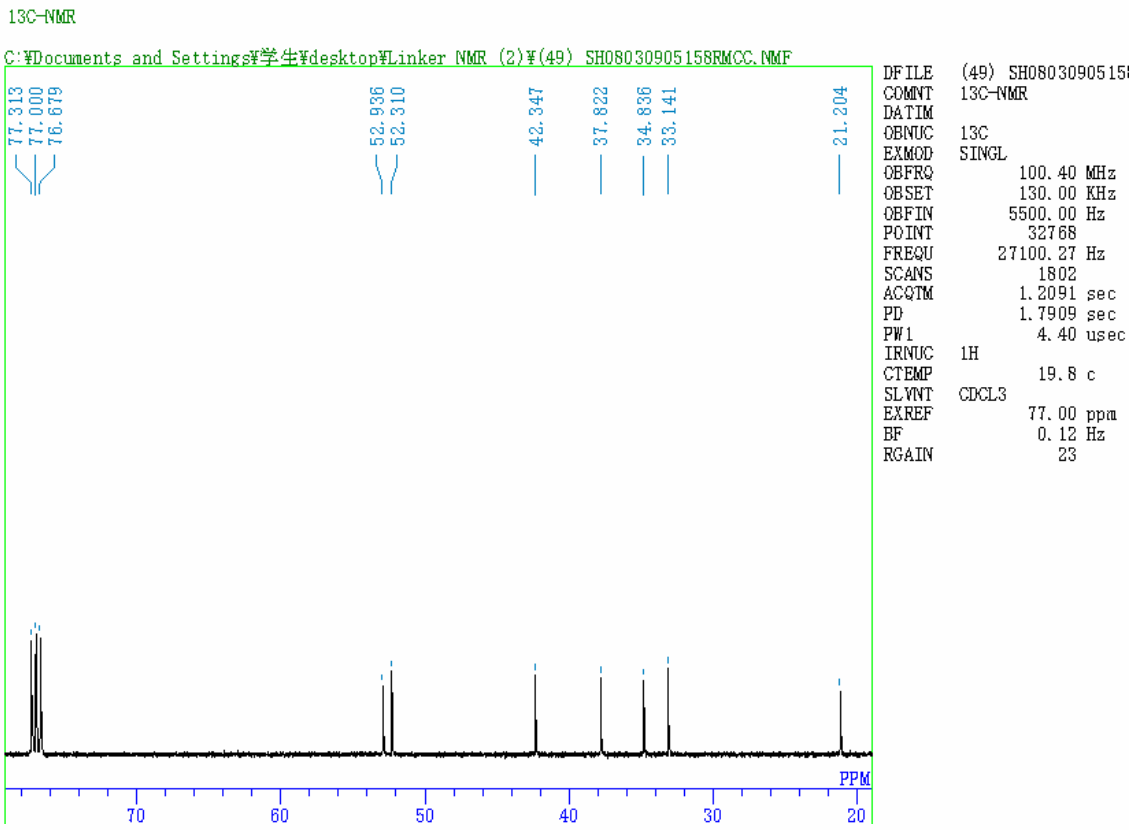
S-benzyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (49) (^{1}H-NMR)

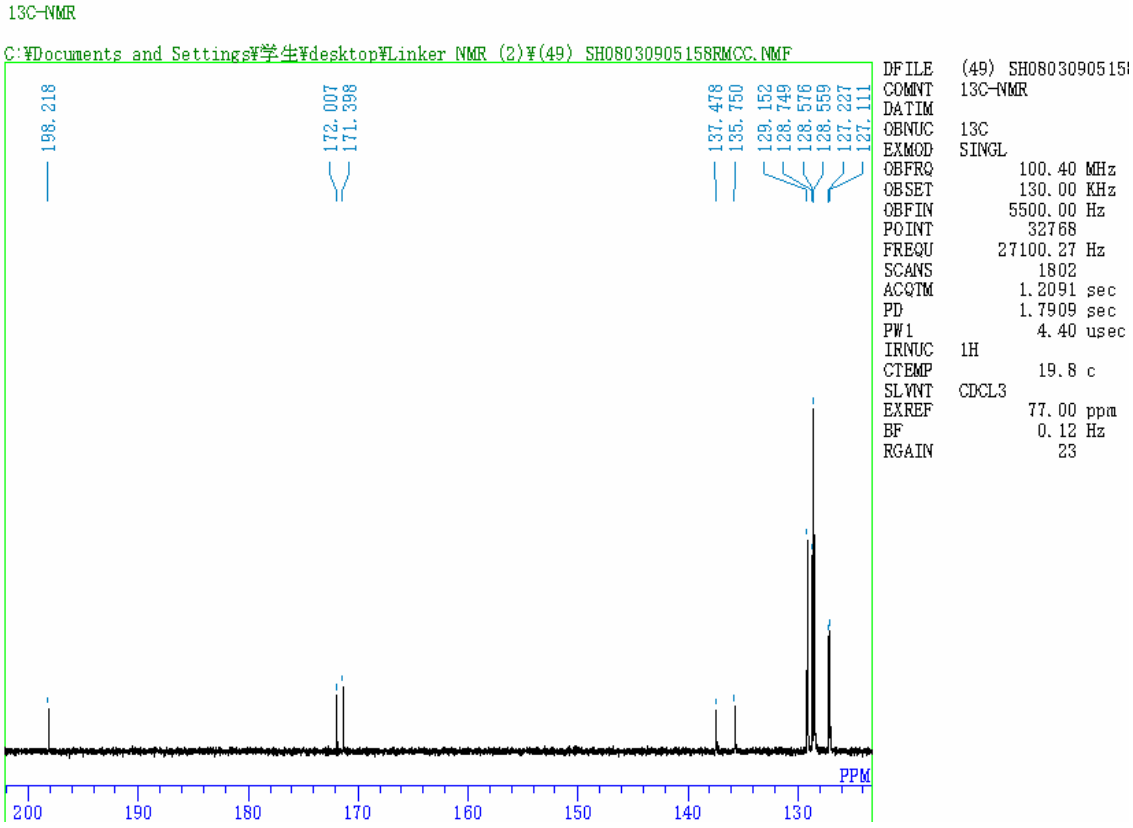




***S*-benzyl 4-(L-phenylalaninocarbonyl methyl ester)butyrate (49) (^{13}C-NMR)**




13C-NMR
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(49) SH08030905158RMCC.NMF
77.313
77.000
76.679
52.936
52.310
42.347
37.822
34.836
33.141
21.204
PPM
70
60
50
40
30
20
DFILE (49) SH0803090515
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 1802
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.40 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23


13C-NMR
C:¥Documents and Settings¥学生¥desktop¥Linker NMR (2)¥(49) SH08030905158RMCC.NMF
198.218
172.007
171.398
137.478
135.750
129.152
128.749
128.576
128.559
127.227
127.111
PPM
200
190
180
170
160
150
140
130
DFILE (49) SH0803090515
COMNT 13C-NMR
DATIM
OBNUC 13C
EXMOD SINGL
OBFRQ 100.40 MHz
OBSET 130.00 KHz
OBFIN 5500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 1802
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.40 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23